Advanced Integrated Scanning Tools for Nano-Technology

SPM SmartSPM™-1000

Instruction Manual

HE002

version: 01.2013

software version: 3.3.68

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1. SYSTEM OVERVIEW

Scanning Probe Microscope (SPM) SmartSPM-1000 is a modern multifunctional measuring system, designed for surface studies of various objects with a nanometer spatial resolution. The design of SmartSPM-1000 provides an opportunity for measurements in air and in a controlled atmosphere. The software for SmartSPM-1000 includes all the standard techniques of atomic force microscopy (AFM), scanning tunneling microscopy (STM), and a wide range of additional and special techniques. The high level of automation makes easier the operation of the microscope.

The SmartSPM-1000 key features:

- High scanning speed.
- Maximum scan range - 100x100x15 microns.
- Positioning accuracy of the probe and no distortion of the image achieved by using the scanner with capacitive closed loop sensors.
- Qscan scanning mode, which has a wide range of settings for scanning.
- Low-noise optical registration system with infrared laser allows for studying samples which are sensitive to the visible light.
- Top and side simultaneous optical access with planapochromat objectives (NA=0.7 and NA=0.28 respectively).
- Automation of measurements including automatic laser and photodiode positioning and setting of measurement parameters.
- Motorized XY sample positioning in the range of 5x5 mm.
- Expandable digital modular controller.

1.1 SmartSPM main parts and design

The SmartSPM scanning probe microscope consists of the base and measuring head mounted on it (**fig. 1.1.1**). The measuring head includes a cantilever probe holder, laser and photodiode. The base includes a scanner, sample holder, tip-to-sample approach system and sample positioning system. The instrument is operated by a digital controller (**fig. 1.1.2**).

Figure 1.1.1 SmartSPM main parts

Figure 1.1.2 SPM controller

The SmartSPM is designed around a stationary probe. That is, samples are scanned back and forth beneath the probe. The probe is basically a sharp silicon tip located on the end of the flexible lever, which is fixed on the chip (**fig. 1.1.3**). The typical curvature radius of the tip is in the range of several nanometers (**fig. 1.1.4**).

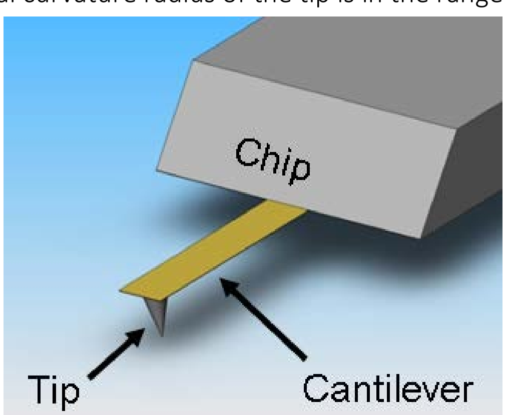


Figure 1.1.3 Cantilever type probe for atomic-force microscopy

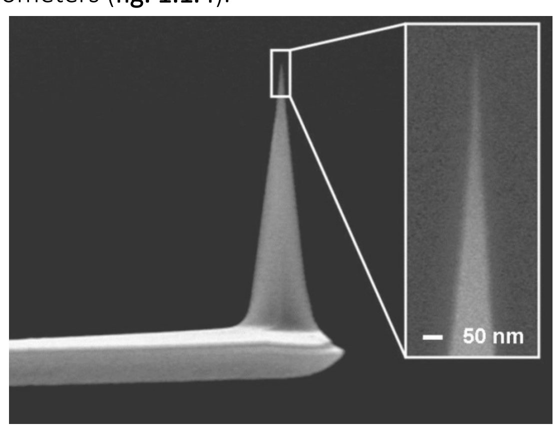


Figure 1.1.4 SEM image of the probe

The probe is an AFM consumable that can be installed inside the probe holder and fixed with the spring clip (**fig. 1.1.5**). The probe holder can be easily removed from the measuring head for convenience of replacement of the probe.

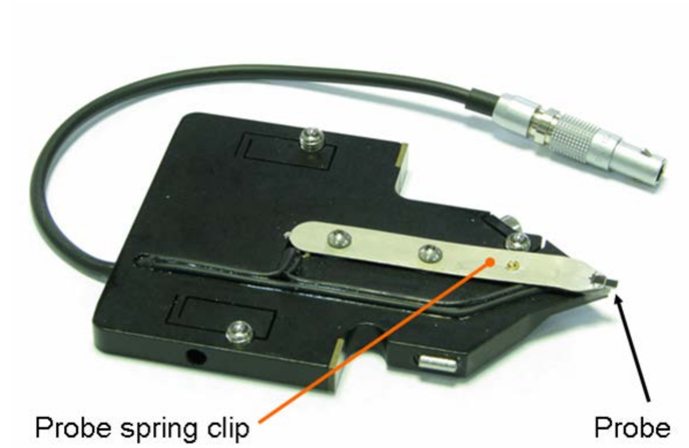


Figure 1.1.5 The probe holder with the installed probe

The laser and four-sectional photodiode are the main parts of the optical registration system of cantilever deflection, which changes during the scanning process. In the properly adjusted system the laser beam, being reflected from the cantilever backside, falls on the photodiode. In the SmartSPM head all required adjustment of the registration system is made automatically by moving the probe holder and the photodiode by stepper motors.

The scanner is made of a set of piezo actuators, which linear size can be changed by applying different potential. Using this effect the scanner moves the sample relatively to the probe. To have the precise scanner motion control, the feedback loop based on a set of capacitive sensors connected to the scanner is used.

The sample can be fixed by glue or double-sided scotch stick tape on the sample holder, which then is to be installed on the scanner. The SmartSPM is designed for measuring samples, which dimension does not exceed 40x50x15 mm.

Motorized approach system is used for approach of the sample to the probe after the probe or the sample is changed. The vertical translation range of the motorized approach system is 20 mm.

Motorized sample positioning system is used for roughly selecting a place on the sample surface which is to be scanned. The XY sample translation range is 5x5 mm provided by the stepper motors, which are controlled from the SmartSPM control software.

1.2 Overview of the software interface

The AIST-NT SPM Control Software consists of the following main components shown in the **fig. 1.2.1.**

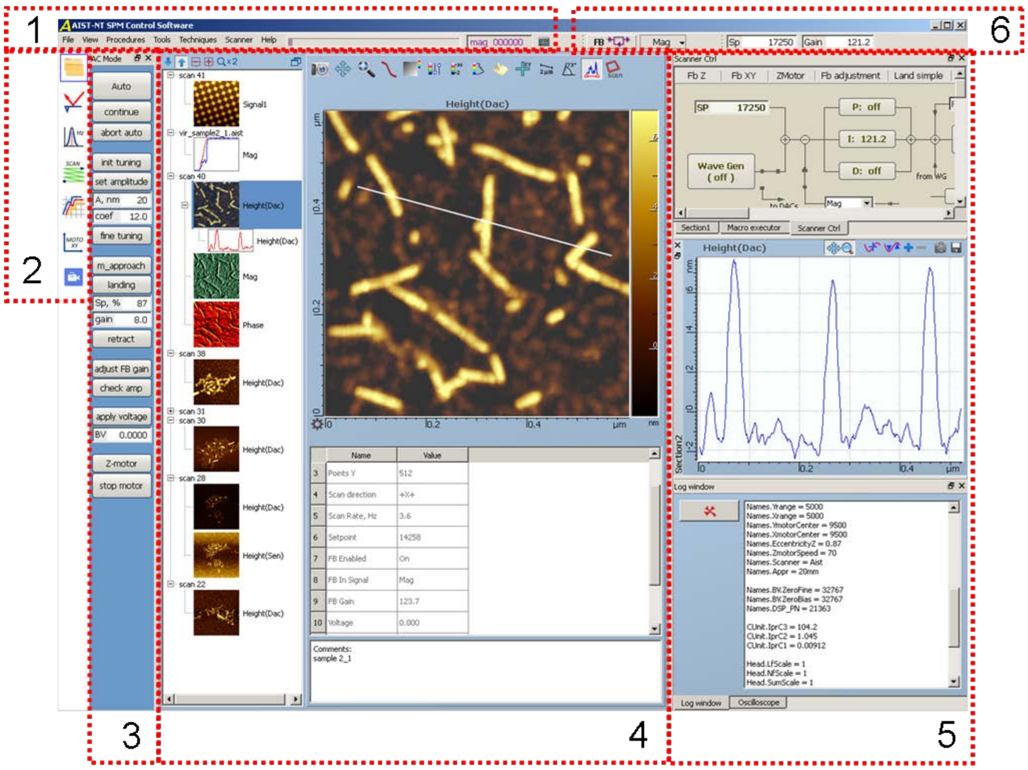


Figure 1.2.1 User interface of AIST-NT SPM Control Software

The numbers in the fig. 1.2.1 correspond to the following elements:

1. Main menu;
2. Main procedures selection window;
3. SPM mode control panel;
4. Main window. There are a number of the main windows in the program, each of them corresponds to a certain system operation stage:
    - **Data view** – data processing;
    - **Laser adjustment** – alignment of the deflection detection system;
    - **Resonance** – probe resonance frequency search;
    - **Scan** – scanning;
    - **Curves view** – viewing curves;
    - **XYZ motors** – sample positioning;
    - **Video window** – video microscope view;
5. Auxiliary windows;
6. Measurements toolbars.

The more detailed description of the program can be found in the corresponding sections of the "Control software reference manual".

1.3 Scanning probe microscopy techniques overview

AIST-NT SPMs carry all modern SPM modes and techniques in a single instrument.

Contact AFM

In contact mode, the cantilever is scanned over a surface at a fixed deflection. Provided that the PID feedback loop is optimized, a constant force is applied to the surface while scanning. If the PID feedback parameters are not optimized, a variable force is exerted on the surface by a probe during a scan. Contact AFM mode is typically used for measuring topography of hard samples. The cantilevers used for contact mode may be constructed from silicon or silicon nitride. Resonant frequencies of contact mode cantilevers are typically around 50 KHz and the force constants are below 1 N/m.

Semicontact AFM

This technique measures topography by tapping the surface with an oscillating tip. When the oscillating probe hits the sample, its short-time interactions with minimal shear are less destructive than the tip-sample forces in contact mode. Stable oscillations of the tapping probe are possible when the probe has enough energy to overcome adhesive and capillary forces of the samples in air. Traditionally, Si probes with stiffness in the 5-20 N/m, such as fpN10, are used for tapping mode experiments. Depending on the type of the sample and problem to solve, probes with stiffness ranging from 0.3 N/m to 400 N/m may be required. Probes with low stiffness should be applied to soft materials or particles with low adhesion to surface. Stiff probes are used for imaging at the elevated forces needed for the visualization of mechanically-different components of multicomponent samples.

Non-contact AFM

In non-contact mode, the long-distance Van der Waals forces are sensed by an oscillating probe, which is excited at its resonance frequency by a piezoelectric transducer and brought in close proximity to a sample. The probe oscillation amplitudes are typically in the range of 5 nm or less.

Phase Imaging

Together with either Semicontact AFM or Non-contact AFM your SPM can provide image contrast caused by differences in surface adhesion and viscoelasticity.

Lateral Force Microscopy (LFM)

The cantilever in an AFM can twist or rotate as it is scanned across a surface. The amount of motion is in some way related to the differences in the chemical/physical properties of a surface. The chemical/physical property could be the nano-roughness or it could be differences in chemical composition. In LFM the probe torsional movement is monitored with a four section photodetector having a left and right section to measure frictional forces between the probe tip and sample surface.

Conductive AFM

The spatial map of the electrical conductivity of a surface is measured. A bias is placed between a conductive sample and probe. Then the sample's surface is scanned. Monitoring the current flow between the probe and the surface yields a conductivity map of the surface.

Magnetic Force Microscopy (MFM)

The AFM technique used for the characterization of the magnetic field distribution over a sample surface is known as magnetic force microscopy. This is a two-pass mode, where the surface topography is measured in tapping mode during the first pass and magnetic forces are mapped on the second pass with respect to the topography data.

Cantilevers with a magnetic Co-Cr coating should be used for MFM research. The coating is formed on both sides of the cantilever to prevent the bending from intrinsic stress and increase the reflection of the laser beam. The spring constant and resonant frequency of the cantilever should be chosen to provide stability in semicontact mode, as well as high sensitivity to weak magnetic forces during the second pass.

Kelvin Probe (Surface Potential Microscopy)

In the Kelvin probe mode, the probe is scanned across a surface, typically in vibrating mode, and a feedback loop is used to keep the voltage between the probe and surface equal. This is a quantitative two-pass technique and measures topography on the first pass and the absolute potential of surface on the second.

Electric Force Microscopy (EFM)

This mode is used for measuring electric field gradient distribution above sample surfaces. Traditionally, the probes of stiffness ~ 5 N/m with conducting coatings are used for such measurements. In some cases, doping of the Si wafer, which was used for microfabrication of the probes, is sufficient to provide reasonable force response to electrostatic interactions with a sample.

Lithography

One can use a probe tip to mechanically scratch or indent a sample surface. It is also possible to use AFM to electrochemically pattern metal surfaces in the nanometer range. By applying a voltage between the probe tip and the sample substrate an electrochemical reaction is induced. This technique based on an electrochemical process is called tip-induced local anodic oxidation.

2. TECHNICAL SPECIFICATIONS OF SMARTSPM 1000

Measuring modes

- Contact AFM;
- Semicontact AFM;
- Non-contact AFM;
- Phase Imaging;
- Lateral Force Microscopy (LFM);
- Magnetic Force Microscopy (MFM);
- Kelvin Probe (Surface Potential Microscopy);
- Electric Force Microscopy (EFM);
- Piezo Response Force Microscopy;
- Force curve measurements;
- Nanolithography;
- Conductive AFM (optional);
- STM (optional);
- Volt-ampere characteristic measurements (optional).

Scanner and Base

- Scanning range 100um x 100um x 15um (+/-10%);
- Scanning type by sample;
- XY non-linearity 0.05%;
- Z non-linearity 0.05%;
- Noise:
  - 0.1nm RMS in XY dimension in 200Hz bandwidth with capacitance sensors on;
  - 0.02nm RMS in XY dimension in 100Hz bandwidth with capacitance sensors off;
  - <0.04nm RMS Z capacitance sensor in 1000Hz bandwidth;
- Digital closed loop control for X,Y,Z axes;
- XY resonance frequency 7 kHz (unloaded);
- Z resonance frequency 15 kHz (unloaded);
- Active elimination of XY phase lag, overshooting and ringing results in fast scanning without any dynamic image distortion;
- Motorized approach range 20 mm;
- Maximum sample size: 40x50 mm, 15 mm thickness;
- Motorized sample positioning range5x5mm;
- Positioning resolution 1um.

AFM Head HE002

- Laser wavelength 1300nm;
- No registration laser influence on biological sample;
- No registration laser influence on photovoltaic measurements;
- Registration system noise <0.03nm.

- Fully motorized: 4 stepper motors for cantilever and photodiode automated alignment;
- Free access to the probe for additional external manipulators and probes;
- Top and side simultaneous optical access with planapochromat objectives (NA=0.7 and NA=0.28 respectively).

Conductive AFM unit (optional)

- Current range 100fA ÷ 10uA;
- 3 current ranges (1nA, 100na and 10uA) switchable from program;
- Conductive to Kelvin mode switchable from program.

Controller electronics

- Modular fully digital expandable controller;
- High speed DSP 190 MHz;
- USB 2.0 interface;
- High speed 500 kHz 18-bit ADC, 20 channels;
- 5 MHz frequency range registration system;
- 2 lock-in amplifiers with 5 MHz frequency range;
- 6 digital 32–bit generators 5 MHz frequency range, 0.01 Hz resolution;
- Software controlled modulation possibilities for probe, X, Y and Z scanners, Bias voltage and two external outputs;
- HV amplifiers -5 … +120v, 0.4 ppm HV noise;
- AC, DC Bias Voltage -10 … +10v, 2 MHz frequency range;
- 7 stepper motors control;
- Digital inputs/outputs for integration with external equipment,
- Analog input/outputs for integration with external equipment.

Software

- Automatic alignment of registration system;
- Automatic configuration and presetting for standard measuring techniques;
- Automatic cantilever resonance frequency adjustment;
- Capability to work with force curves;
- Macro language Lua for programming user functions, scripts and widgets;
- Capability to program controller with DSP macro language in real time without reloading control software;
- Capability to process images in coordinate space including making cross-sections, fitting and polynomial smoothing up to 13 degree;
- Processing up to 5000x5000 pixel images.

3. PERFORMING MEASUREMENTS

3.1 Switching on the system

WARNING! Before starting the controller for the first time, make sure that the power switch 110/220V on the rear panel of the controller corresponds to your power supply voltage.

To start working with the scanning probe microscope system:

- Turn on the PC;
- Turn on the controller;
- Run the AIST-NT software;

Right after you run the AIST-NT software, the popup window (**fig. 3.1.1**) appears from which you may choose either "**Initialize SPM**" to start working with the instrument or "**View only mode**" to work with previously acquired data offline.

Figure 3.1.1 Device initialization popup window

Press "**Initialize SPM**". After successful initialization, the information window appears (**fig. 3.1.2**) with the hint to select one of the AFM modes from the "**Tools**" pull-down menu (**fig. 3.1.3**). Select "**Contact mode**" or "**AC mode**" to work either in contact or semicontact AFM modes. A corresponding control panel appears right after you select one of the modes (**fig. 3.1.4**).

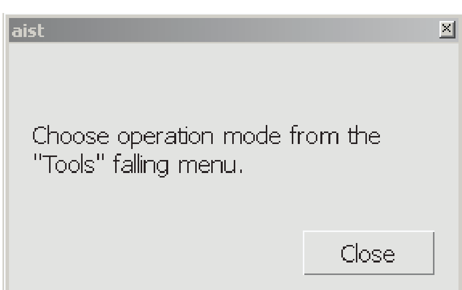


Figure 3.1.2 Information window

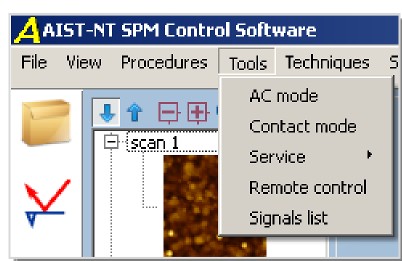


Figure 3.1.3 "Tools" pull-down menu

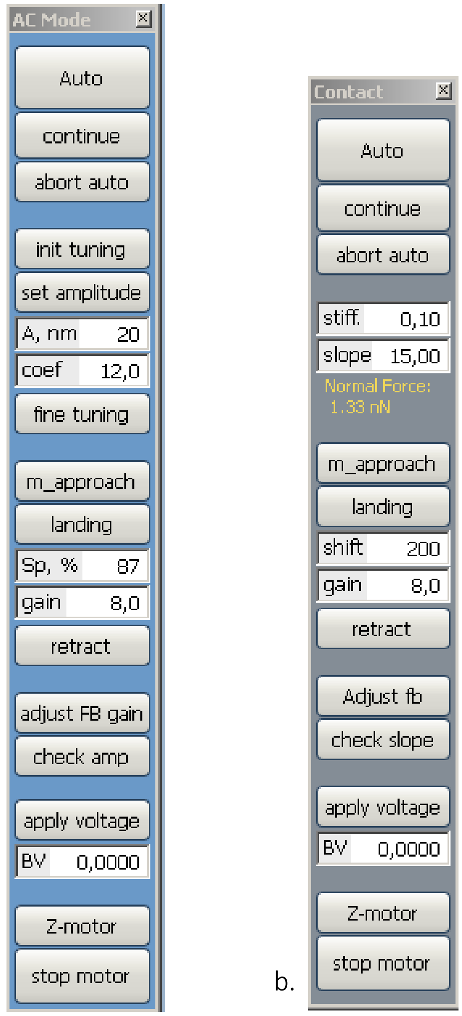


Figure 3.1.4 Control panels: (a) semi-contact/non-contact modes, (b) contact mode.

During controller initialization the program checks the scanner sensors calibration. In case there is any error instead of the information window (**fig. 3.1.2**) the following warning dialog window appears (**fig. 3.1.5**).

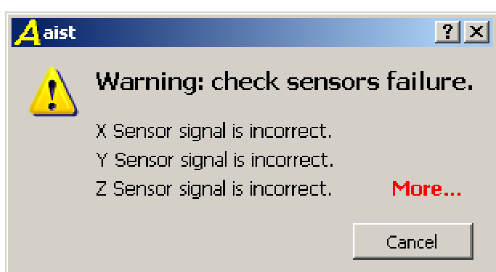


Figure 3.1.5 Warning window of scanner sensors calibration

Once such window appears, click the **More...** button and follow the relevant instructions:

1. Make sure that the scanner is properly connected to the controller.
2. Make sure that the current **default.scn** file corresponds to the scanner number.
3. Calibrate your scanner sensors following the instructions described in Appendix 8.2.

3.2 Sample installation

Typical samples viewed by SPM are small chips of material, the surface of which you are interested in investigating. The SmartSPM is designed to work with samples, which size does not exceed 40x50 mm and 15 mm in height.

Before loading the sample into the microscope, you should fix it on the sample holder. There are a few types of sample holders provided with the microscope (**fig. 3.2.1**): (**1**) holders with polycor substrate on top, (**2**) round metal holders 4 mm in diameter, holder with the electrical contact (**3**), magnetic holder with a set of metal discs (**4 and 5**). A slit in the bottom part of the sample holder is used for mounting it on the scanner.

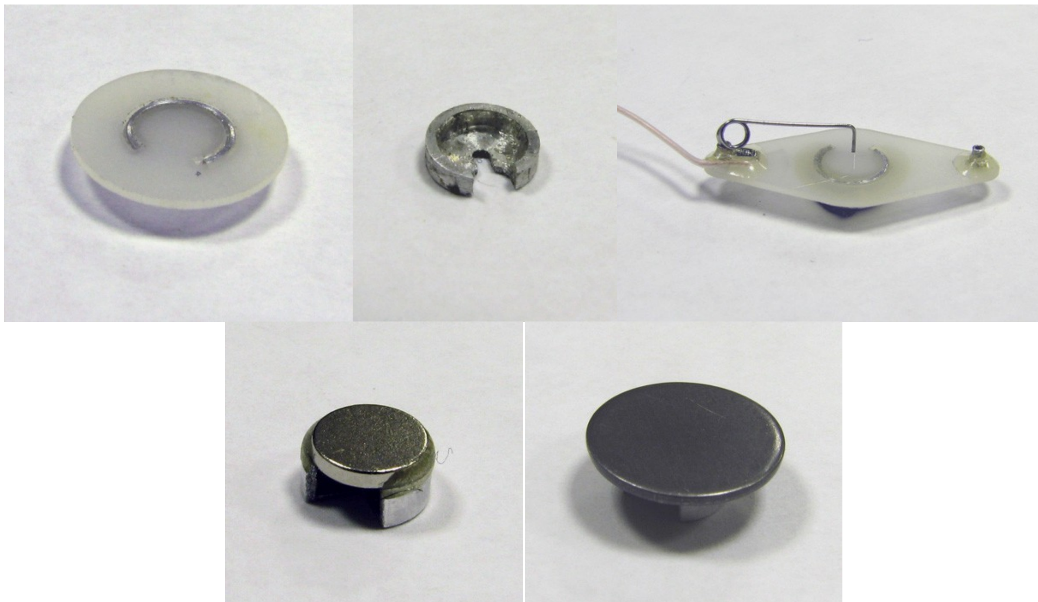


Figure 3.2.1 Sample holders: **1** - basic holder, **2** - holder with an electrical contact, **3 and 4** - magnetic holder, **5** - holder for fast scanning.

For AFM or other non-conductive measurements use the holder with a polycor substrate (**1**), or a magnetic plate (**4 and 5**). Holder with the contact is to ground the sample or to apply voltage to the sample for working in electrical modes of SPM (Kelvin mode, current spreading microscopy, STM).

To mount the sample, use any double-sided scotch tape or "Superglue" (cyanoacrylate "Superglue" adhesive). Keep in mind that mounted on the double-sided adhesive tape sample may drift on the substrate. Therefore, to minimize drift is preferable to use glue. Use a blade to remove your sample from the holder.

The best performance is achieved with a minimum weight of the sample, therefore, for scanning on high speed, use small-size samples and small holder (**2**). Use "Superglue" to glue samples to this holder. Squeeze out a drop of “Superglue” on some surface. Then put your adhesive in 2-3 points on the holder using either a thin needle or a piece of wire. Finally, place your sample on the holder and press on it. Make sure that your adhesive has dried out before installing your sample into the microscope.

You can install any standard sample holder on the microscope scanner the following way:

There is a fixing clench (rod with head) in the center of the upper side of the scanner (**fig. 3.2.2**). The fixing clench has a spring, so the rod's head is pressed to the scanner's surface. During the sample holder's installation, the fixing clench rod is lifted up and the holder is slid into under the rod's head through its slit. When the sample holder is installed, it is fixed to the scanner with the fixing clench's head (**fig. 3.2.6**).

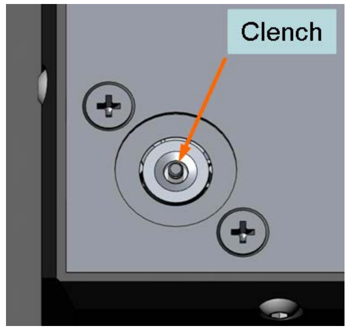
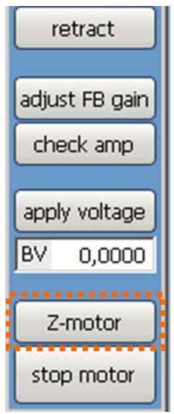


Figure 3.2.2 Sample holder fixing clench

To install a sample holder, do the following steps:

- Press the **Z-motor** button in the bottom of the **AC mode** (**Fig 3.2.3**) or **Contact mode** control panel to open the **Zmotor Ctrl** control panel (**fig. 3.2.4**). Move the scanner up from its lowest position to a few millimeters (e.g. 5-10 mm) using the **move** button on the **Zmotor Ctrl** panel (**fig. 3.2.3**). You can specify a required moving distance in millimeters using the **dist** input field. Positive values means moving up, negative down.



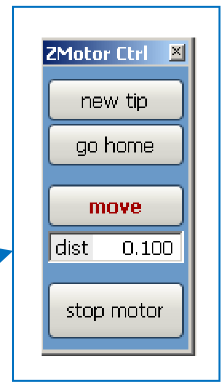
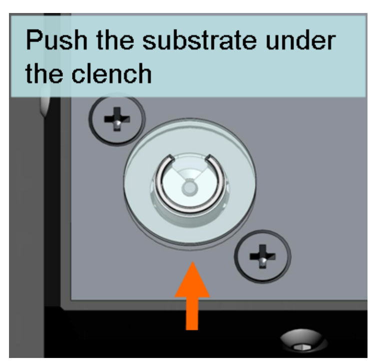


Figure 3.2.3 Position of the **Z-motor** button on the **AC mode** control panel

Figure 3.2.4 **Z-motor Ctrl** control panel



NOTE: The motion of the scanner can also be carried out using tools of the **XYZ motors** window (see Chapter 5).

Figure 3.2.5 Fixing clench handle

Figure 3.2.6 Sample holder installation

- Turn the fixing clench handle down to 90 degrees clockwise to lift the fixing clench rod up (**fig. 3.2.5**).

While holding the handle in this position, slide your sample holder under the clench head, so the slit sets against the rod till its end (**fig. 3.2.6**).

- Release the handle. Make sure that your sample is fixed and the holder is in the scanner's central position.

NOTE: When the scanner is in its lowest position, the fixing clench is open, so you do not need to turn the fixing clench handle. To move the scanner to its lowest position, use the
go home button of the **ZMotor Ctrl** control panel.

3.3 Selecting a probe and measuring mode

There are special types of probes for each measuring mode. You should select one of the measuring modes depending on your sample type.

There are three basic AFM measuring modes provided with the microscope for measuring surface topography – contact AFM, semicontact AFM and non-contact AFM.

In the contact AFM mode the probe is kept in constant contact with the sample surface during the whole scanning process due to the feedback loop, which maintains the loading force at some constant level. Use this mode for system calibration on test gratings or for measuring solid samples as the typical loading force is comparatively high. We recommend using soft long cantilevers with the spring constant less than 0.3 N/m in this mode.

In the semicontact AFM and non-contact AFM modes the probe oscillates very close to the sample surface at its resonant frequency. The feedback loop maintains the oscillation amplitude at some constant level. In this mode the lateral component of the interactive force between the probe and sample surface is substantially decreased in comparison with the contact AFM mode. The normal component of the interactive force is determined by the amplitude of free oscillation of the probe and by the level of its limitation.

The amplitude of free probe oscillation in the semicontact AFM is normally in the range of 50 – 200 nm. The working amplitude is typically set to half of it. In this case repulsive forces play the main role in the probe-surface interaction. We recommend using this mode for measuring samples with a great number of topographic features and for phase imaging of polymers.

The difference between the semicontact AFM and non-contact AFM is that the amplitude of free probe oscillation is about 2 – 10 nm in the latter and the working amplitude is set to 80-95% from the initial one. In this case attraction forces play the main role in the probe-surface interaction, so the probe does not directly contact the sample surface during the scanning process. We recommend using this mode for measuring delicate samples such as carbon nanotubes, different kinds of nanoparticles, biological macromolecules, polymer monolayers etc.

We recommend using relatively hard cantilevers for the semicontact or non-contact AFM modes with spring constant 5 – 40 N/m and resonance frequency 70 – 500 kHz.

3.4 Probe installation

To install or replace the probe, do the following steps:

- Remove the scanner from the probe by pressing the "**new tip**" button on the **ZMotor Ctrl** panel (**fig. 3.2.3**).
- Disconnect the probe holder electric connector from the measuring head.

- Pull the handle of a support of the probe holder up and turn it clockwise on 180 degrees (**fig. 3.4.1**). After turning, the handle stays in its up position.
- Carefully remove the probe holder from the head.

Figure 3.4.1 Removing the probe holder from the AFM measuring head HE002

- Put the probe holder upside-down on the table.
- Open up the probe's spring clip by pressing the probe holder to table surface (**fig. 3.4.2**).
- Take one probe from the box using tweezers. Please notice that each probe lies inside the gel box, so its tip is up and reflecting side is down. One-cantilever probe chip lies oriented to the box hinges.
- Using tweezers place the probe under the clip as shown on **fig. 3.4.3**.

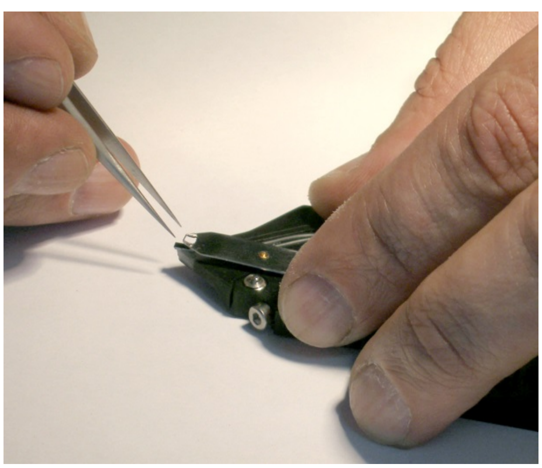


Figure 3.4.2 Installing the probe into the holder

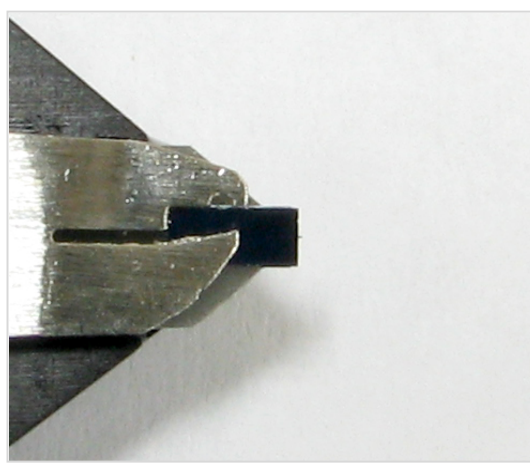


Figure 3.4.3 Proper probe position in the holder

- Install the probe holder into the measuring head, fix it with the latch by turning it one more time clockwise to 180°.
- Looking to the side of the microscope make sure that the spring of the latch has come to the corresponding hole on the holder. (**fig. 3.4.4, left**).

Moving of the holder to the left and to the right has to return it to its initial position by the spring. If the spring support is missed the deepening (**fig. 3.4.4, right**), slightly rotating the handle of the support, move it up and down until it is placed into deepening on the probe holder.

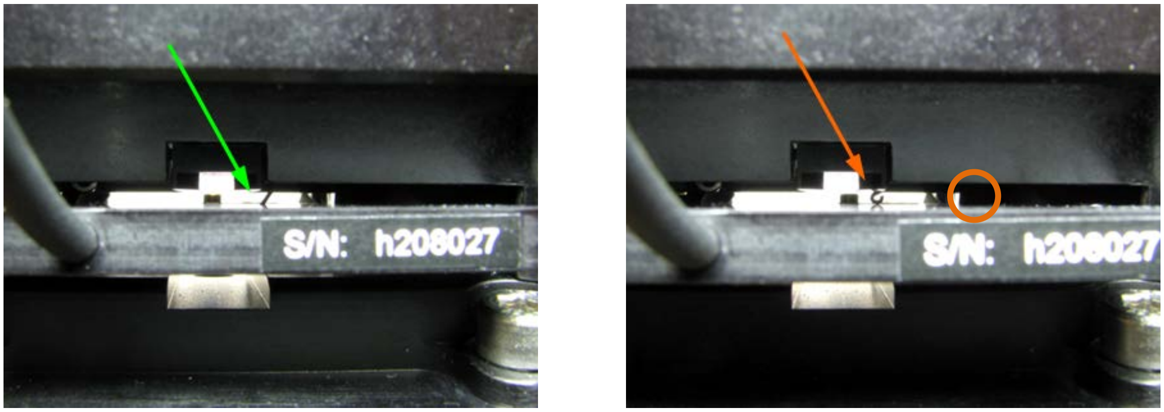


Figure 3.4.4 Mounted on the microscope probe holder. **Left:** the holder is installed correctly; the green arrow points to the spring support, which placed into the deepening on the holder. **Right:** the holder is not installed correctly, the red arrow points to the end of the spring support, which is not placed into the deepening in the holder.

- Attach the electric cable, so that the red stripe mark on the connector is opposite to the same mark on the jack.

3.5 Adjustment of an optical registration system

After installing the probe holder on the microscope, adjust an optical registration system that detects the cantilever deflection. The system is considered to be adjusted when a laser beam is directed on certain spot of the back side of the cantilever and a reflected from the cantilever laser beam is onto the center of a photodiode. In the measuring head of the **SmartSPM** microscope, the position of the laser in the registration system is fixed and the positioning of the laser beam on the cantilever is accomplished by moving the probe holder, i.e. cantilever is brought to the laser beam.

Adjustment of the registration system consists of two stages:

1. Adjusting the position of the cantilever.
2. Adjusting the position of the photodiode.

NOTE: The registration system has a lens, focusing a laser beam on the surface of the cantilever. The focusing is carrying out by changing the length of support screw of the probe holder. The probe holder by default is set to work with the probes of the 0.4 mm chip thickness (probes of NSC and CSC series produced by MikroMash and probes of fpN, fpC series by NIIFP). Using the probes with the chip thickness far from 0.4 mm, the laser spot on the cantilever will be out of focus causing the loss of the image quality. In order to work with chips of other thickness, adjust the focus of the optical registration system of the measuring head.

Before setting up the registration system, make sure that the distance between the probe and a sample is more than 1 mm. The need for such a gap is that during the adjustment procedure the motion of the probe holder with set cantilever can result in a collision of the cantilever with a sample. Therefore, if the gap is less than 1 mm, move the scanner down by pushing the **New tip** button (see **figure 3.2.3**) on the **Z motor Ctrl** control panel (motorized sample positioning in vertical direction). The **Z motor Ctrl** control panel can be opened by clicking on the **Z motor** button on the **AC mode** or **Contact mode** panels.

To adjust the registration system, select the **Laser Adjustment** window (click on the  on in the main procedures selection window).

The functionality of this window described in the section 5.2 of the "Control software reference manual".

3.5.1 The basic procedure of the adjustment of the cantilever position

The basic procedure intends for adjusting the registration system at using single-lever rectangular cantilevers.

To adjust the position of the cantilever, perform the following steps:

- Set the probe holder to its initial position by pressing the **Init position** button. (The initial position is the central position of the holder.)
- Start the search of the cantilever by clicking on the **find tip** button.

By default, the search is performed in the following way:

The holder starts moving from the current position and moves on of the expanding trajectory until the laser beam comes onto the cantilever (**fig. 3.5.1**). After that, the laser beam is passed across and along the cantilever to determine its length and width. In the end, the holder is set that the laser beam is on the specified place of the back-side of the cantilever.

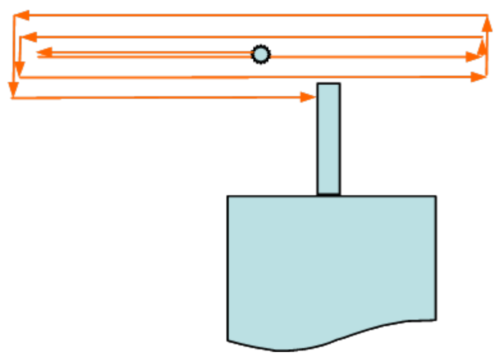


Figure 3.5.1 The trajectory of the probe holder at finding of the cantilever

The process for finding the cantilever is shown in the **Laser Adjustment** window. The **full area** field, which is in the bottom-left part of the window, shows the full trajectory and the **zoomed area**, which is to the right of the **full area**, displays the area inside of the red frame. When the adjustment on the rectangular cantilever is completed, the image looks like as it shown in fig. 3.5.2. Lines show the trajectory of the probe holder motion during the search of the cantilever. The brightness of the line depends on the intensity of the reflected laser beam: dark blue lines correspond to the background level (the laser beam does not shine on the cantilever) and bright white lines correspond to the cantilever (the laser beam shines on the cantilever). At adjusting system on the rectangular cantilever a cross consisted of white lines is obtained: a vertical line displays to the length, the horizontal lines - the width of the cantilever. The free end of the cantilever with a tip is on the top. The red cross indicates the position of the laser beam (in this case approximately above the tip).

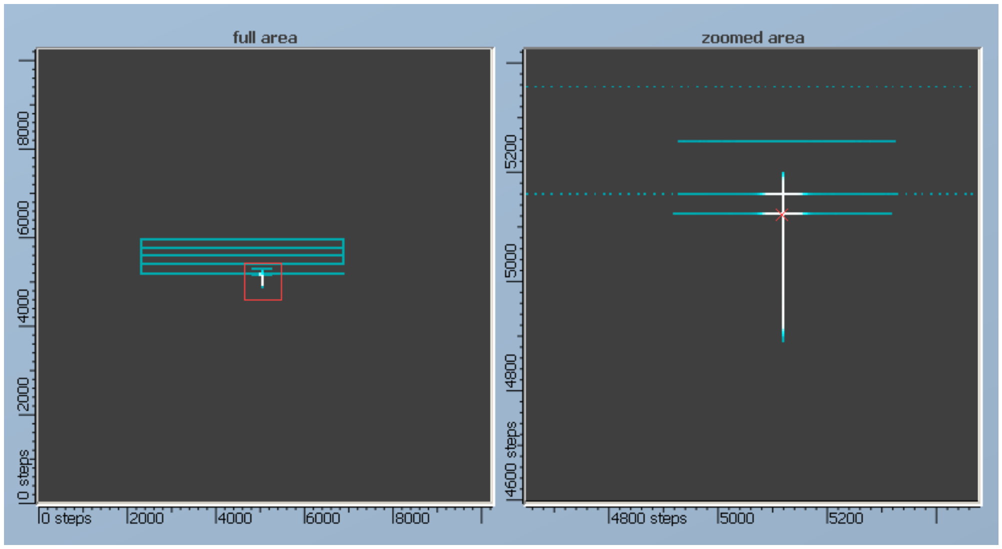


Figure 3.5.2 The image of the rectangular cantilever in the **Laser Adjustment** window, obtained after looking for a probe using the **find tip** procedure.

3.5.2 Looking for a cantilever around

If during the operation the holder was taken out and then was placed back without changing the probe (for example to replace the sample), it is useful to perform the slight readjustment of the position of the laser beam on the cantilever using the **look around** command. This command starts the search for the cantilever in the immediate vicinity of the current position of the cantilever. The search approach is similar to the **find tip** method.

3.5.3 Manual adjustment of the cantilever position

Perform the manual adjustment of registration system when the cantilever can not be find using the **find tip** procedure or when use non-standard cantilevers (multi-lever probes, probes with triangular levers, probes with a small reflection coefficient, and i.e.).

To accomplish the adjustment manually, follow steps:

- Set the probe holder to the center position by clicking on the **init position** button**.**
- In the **full area** field, click and drag the pointer to surround the desired selection and then, release the mouse button to finish the selection. The result is shown in **figure 3.5.3**.
- Click on the **go corner** button - the holder with a cantilever will be moved that the laser beam will be directed to the lower left corner of the selected area.
- Start scanning a selected area with the holder by clicking **Scan X**. In the **zoomed area** the image of the cantilever(s) will appear (see to the right field in **fig. 3.5.3**).
- Set the laser approximately on the selected lever by clicking, in the **zoomed area** on the desired spot (the red cross will appear) and click the **go** button.
- Perform the **look around** procedure in order to accurately determine the position of the laser beam on the cantilever.

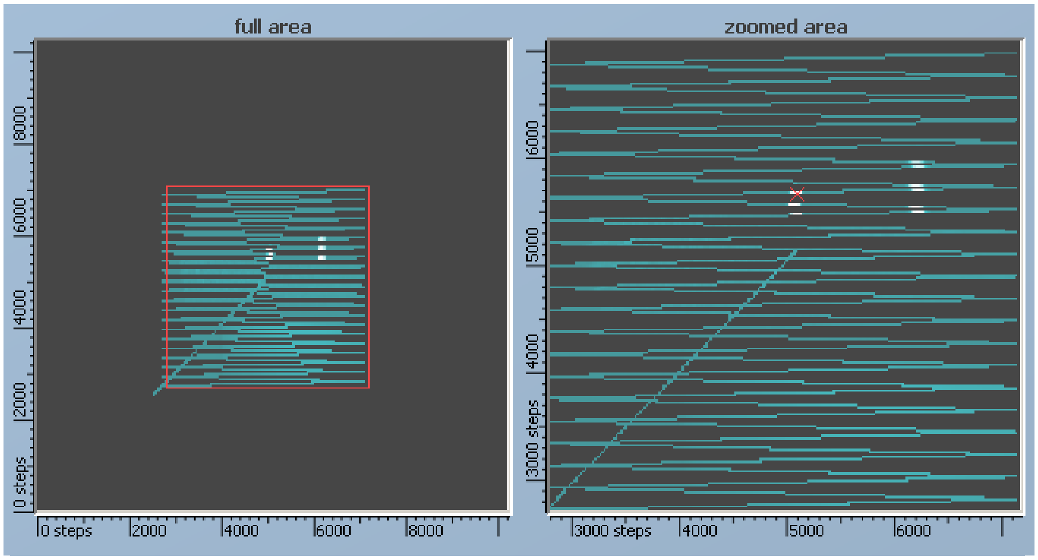


Figure 3.5.3 Manual adjustment on the probe with the two levers.
The laser beam is focused on the short lever.

3.5.4 The position of the laser beam on the cantilever

The position of the laser beam on the cantilever is set by **Y pos** and **X pos** in the **Laser Alignment** tab of the **Param tables** window:

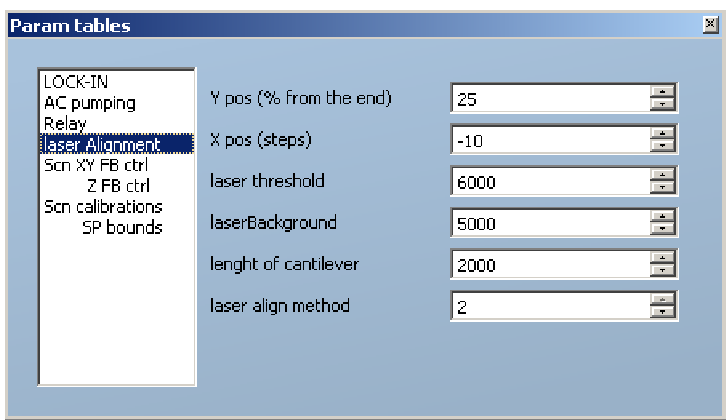


Figure 3.5.4 The **Laser Adjustment** bookmark in the **Param tables** window

Y pos - specifies the location of the laser spot along the cantilever. **Y pos** is in percentage from the cantilever length which is measured at the adjustment. Percentages counts from the free end of the cantilever.

X pos - specifies the location of the spot across the cantilever. **X pos** is in steps of the motor which moves the holder in the X direction. One step is approximately 0.5 microns.

The program uses the set parameters for accomplishing the **find tip** and the **look around** procedures.

The laser spot is set by default on the point which is in the center of the cantilever on the width, and on 25% of the length from the free end of the cantilever. The value of -10 steps for **X pos** is entered to compensate the backlash of the motor.

Set **Y pos** = 50% for **triangular cantilevers** in order to establish the laser spot on the center of the reflecting area.

Set **Y pos** in the range of 30 - 60% for **long rectangular cantilever** which are preferred for the contact measurements because at lower values the reflected laser beam can go beyond the detection area of the photodiode with a strong deflection of the cantilever (for example, when approaching to the charged surface of the sample).

The functionality of other parameters in the **Laser Alignment** tab is described in the section 6.8.4 of the "Control software reference manual".

3.5.5 The adjustment of the photodiode position

At the adjustment of the photodiode position, the photodiode is moved that the laser beam is reflected from the back-side of the cantilever to its center.

To adjust the position of the photodiode use the **adjust diode** button. After starting the adjustment procedure, the **"Please wait, photodiode adjustment is in progress ..."** message will appear.

When adjustment is completed, the **"Photodiode setting completed"** message will appear.

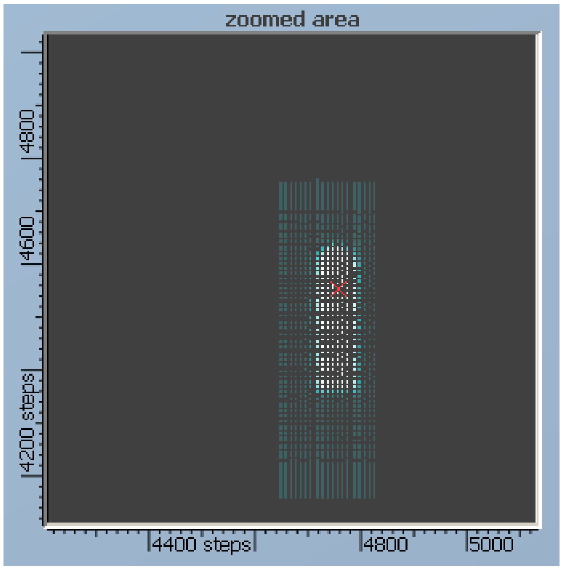
NOTE: The adjustment of the photodiode can be monitored on an oscilloscope. Vertical cantilever deflection signal corresponds to **Nf** (short for Normal force), torsion cantilever deflection signal corresponds to **Lf** (short for Lateral force). When adjustment is finished, the signals **Nf** and **Lf** should be in between of ± 350 ADC units.

3.5.6 Additional features: manual adjustment of the laser beam on the cantilever

The program provides the ability to manually correct the position of the laser beam on the cantilever. Using manual adjustment the laser spot can be set to maximize the reflected signal or to maximize the signal of the cantilever amplitude. This option may be necessary, for example, when working with the probes having a non-uniform reflective coating.

For manual adjustments, follow steps:

- Adjust the registration system.
- For the **AC mode**: Tune on the resonant frequency of the cantilever, by clicking the **init tuning** button on the **AC mode** control panel (see section 3.6.1).
- Obtain an image of the cantilever using the **show tip** command, or an image of the distribution of the **Mag** signal using the **show mag** command (**fig. 3.5.4**).
- In the **zoomed area** field, set the red cross at a desired point on the back-side of the cantilever and click the **go** button. The system will move the holder with the cantilever that the laser beam shines on a given point.
- After adjusting the position of the cantilever, adjust the position of the photodiode using the **adjust diode** command.



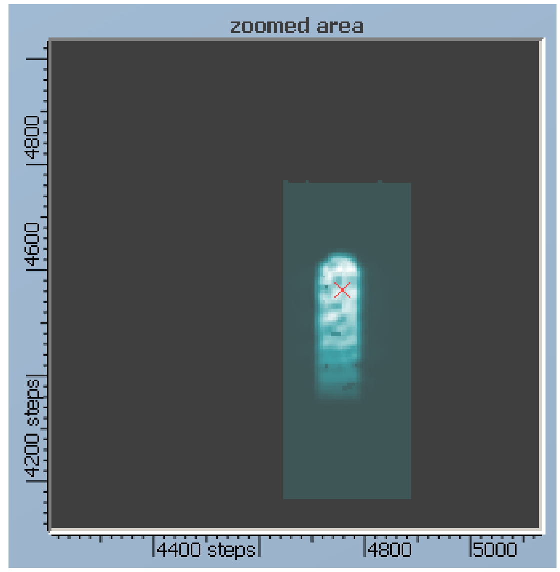


Figure 3.5.4 Left: Image of the cantilever obtained by using **show tip** command. **Right:** The distribution of the **Mag** signal on the cantilever obtained by using **show mag** command.

Control the signal level by the oscilloscope. Intensity of the reflected on the photodiode laser beam corresponds to the **Sum** signal and the amplitude of the cantilever oscillation signal corresponds to **Mag**.

NOTE: The manual adjustment procedure can be performed using the **Moto Scan** scan mode (section 4.9). In this mode, recorded images of the cantilever surface are more detailed compare to images obtained using **show tip** or **show mag** commands**.**

3.6 Adjusting the instrument for the Semicontact / Non-contact AFM mode

Select "**AC mode**" from the "**Tools**" pull-down menu. The "**AC mode**" control panel appears (**fig. 3.1.4**).

3.6.1 Tuning in to the resonant frequency of the probe and setting its amplitude

After setting up the optical registration system it is necessary to find the resonant frequency of the cantilever and set the amplitude of vibrations at this frequency. The oscillations of the probe are carried out by applying the signal from one of the built-into the controller generators to the piezoelement of the probe holder.

Select the **Resonance** window to tune to the resonant frequency of the probe. Set the **low** and **high** frequency boundaries of the scanning range so that the resonant frequency of the probe is within the range (area 1 in **fig. 3.6.1.1**). Approximate resonant frequencies of the cantilevers are usually specified on a label on the box.

3.6.1.1 Automatic adjustment to the resonance frequency

Click the **Init tuning** button (area 2 in **fig. 3.6.1.1**) on the **AC mode** control panel to search for the resonant frequency and to set the operation frequency. After clicking on this button, the "**Setup resonance**" message is appeared.

The program performs following actions:

1. Measures the amplitude signal in the frequency range between specified **low** and **high** values and displays the result (frequency response) in the upper field of the **Resonance** window. The highest in the specified frequency range peak is automatically selected as the resonance peak.

2. After that starts more detailed scanning near the resonant peak in the range specified in **dF**. The result is displayed in the lower field.
3. The program sets the **Mag** amplitude signal is equal to 25,000 ADC units by varying the power of the generator that excites the cantilever.

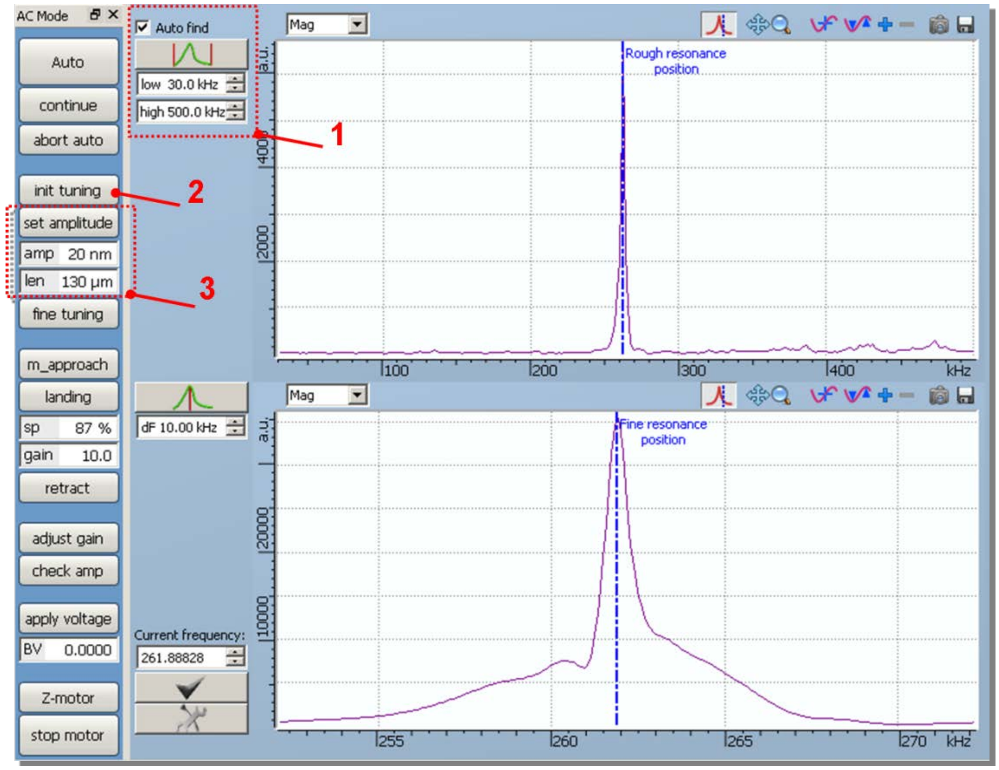


Figure 3.6.1.1 The **Resonance** window that displays resonance curve of the probe in defined range (upper field) and around specified frequency (lower field). To the left of the window, the **AC mode** control panel is shown. The numbers designate: 1 - **low** and **high** fields, 2 - the **init tuning** button, 3 – the **set amplitude** button and **amp** and **len** fields.

4. Displays the **"Resonance setup completed"** message after completing the adjustment.

In the cases when the resonant peak is not found or the **Mag** signal can not be set to 25000, the "**Failed to set resonance or amplitude**" message is appeared.

NOTE: After the **init tuning** procedure, the amplitude of the cantilever remains unset because this procedure uses a fixed gain of the synchronous detector (**Lock-in input** = Nfx10, **Lock-in gain** = 10) and sets the **Mag** level independently of parameters of the cantilever.

3.6.1.2 Setting the amplitude of the cantilever

Once the operating frequency has been set, it is necessary to set initial amplitude of the cantilever (the amplitude of vibrations far away from the surface of the sample). Selection of the the amplitude value is determined by the type of cantilever and the used method: for semi-contact method set it to 50-200 nm, non-contact method – to 5-20 nm.

To set the amplitude:

Enter the length of the cantilever in the **len** box on the **AC mode** control panel (area 3 in **fig. 3.6.1.2**). The length of the cantilever is written on the label of a box with the probes.

Enter the amplitude in the **amp** text box and press the **set amplitude** button.

3.6.1.3 Manual selection of the operating frequency

In cases when it is necessary to set the operating frequency manually (for example, if there are a few resonant peaks), use the settings located in the **Resonance** window.

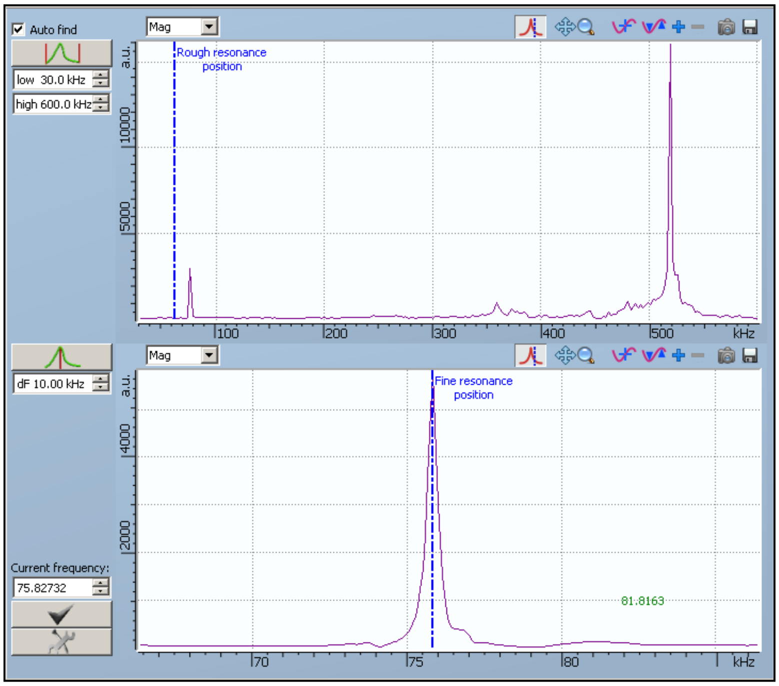
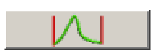
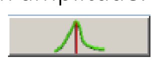


Figure 3.6.1.3 Resonance curve with two peaks

Follow the procedure below:

Scan the frequency by pressing the  button. The frequency response of the cantilever will appear in the upper field (**fig. 3.6.1.3**). Vertical blue line indicates the approximate position of the resonance. In case of multiple peaks, the program automatically selects the peak with a maximum amplitude.

Set a marker approximately at the desired peak and then click the  button that starts the scanning around the frequency set with a marker. After scanning in the lower field, the program draws the frequency response around the selected peak and sets the marker at the exact position of the top of this resonant peak.

To adjust the frequency, change the position of the marker. Set the selected frequency using the  button. The selected frequency is displayed in the **Current frequency** text box located above the button.

3.6.1.4 Manual setting of amplitude and magnitude of the Mag signal

To manually adjust the amplitude of the cantilever and magnitude of the **Mag** signal, use the **LOCK-IN** bookmark in the **Param tables** window (see **fig. 3.6.1.4**).

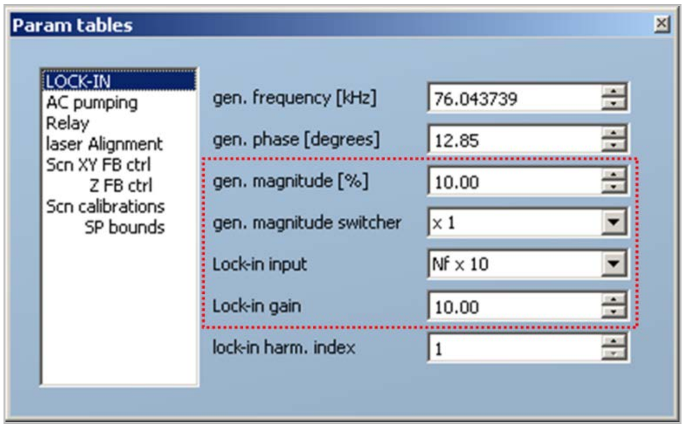


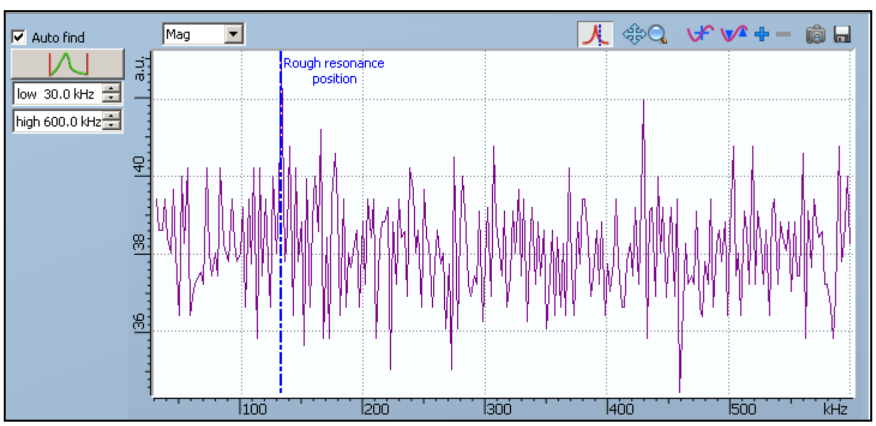
Figure 3.6.1.4 The **LOCK-IN** bookmark in the **Param tables** window

To change the ***amplitude of the cantilever***, change the voltage on the generator. The voltage value is set by **gen. magnitude** (smooth regulation of the voltage) and **gen. magnitude switcher** (switching ranges of the voltage).

To change the ***magnitude of the Mag signal***, change the gain of the synchronous detector. The **Lock-in gain** parameter is for smooth adjustment of the gain and The **Lock-in input** (Nf, Nfx10) parameter is to turn the pre amplification on or off .

3.6.1.5 Difficulties at tuning to the resonant frequency

The complete absence of resonances:

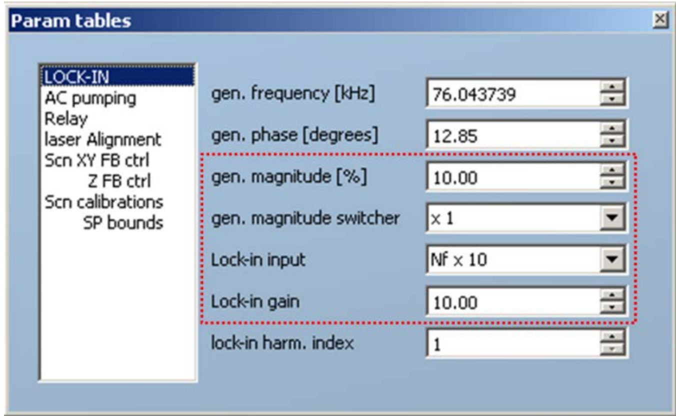


The following are some possible reasons:

1. The optical registration system is not adjusted, there is no cantilever.

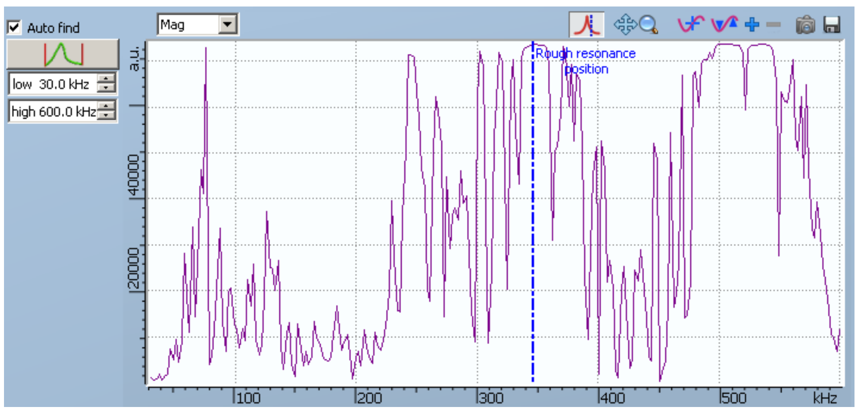
2. The electric cable of the probe holder is not connected to the measuring head.

3. There is no voltage applied to the piezoelectric probe holder:



For applying voltage, set the **piezo actuator** = **Gen.2**.

Supersaturated frequency response:



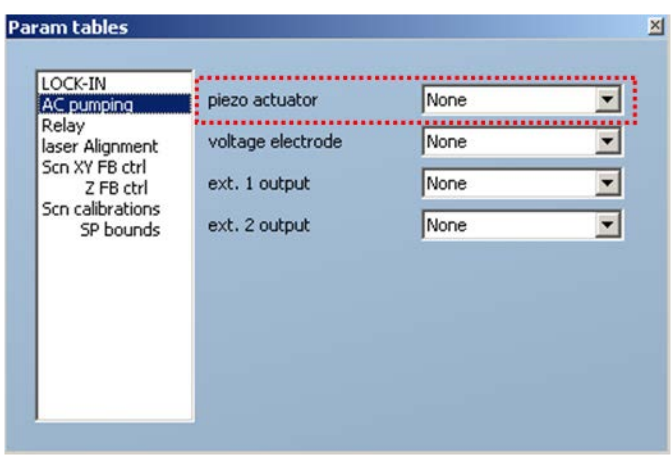
The most like reasons are in too strong oscillations of the cantilever or in too high magnification of the **Mag** signal.

To return to the normal oscillations and magnification either start the **init tuning** procedure or manually set the following options in the **LOCK-IN** bookmark:

3.6.2 Approaching the sample to the tip

After tuning onto the resonant frequency and setting the amplitude of the cantilever, it is necessary to bring together the probe and the sample so that the cantilever tip is in the vicinity of the sample surface. In SmartSPM measuring head is fixed, therefore the change of the gap between the tip and the sample is accomplished by moving the sample to the probe.

The approach of the sample to the probe is divided into three stages:



1. The preliminary approach with visualization distance control to the gap up to 1-2mm.

2. The approach with automatic distance control to the gap of 1 micrometer.

3. Landing on the surface.

3.6.2.1 Preliminary approach

Move the sample to the probe on a distance of 1 - 2 mm by entering the number in the **dist** textbox and pressing the **move** button, on the **Zmotor Ctrl** panel (**fig. 3.2.3**). Control the distance between the probe and the sample by looking at the side of the microscope.
After that, if it's necessary to measure in some particular place on the sample surface, you may roughly position your sample relatively to the probe (see Chapter 5).

3.6.2.2 The approach with automatic distance control

The procedure of the automatic distance control is organized as follows:
After starting the approach procedure, scanner is extended all the way to the probe.
If in the scanner up position the sample does not touch the probe, the scanner is set to the middle position and the motor starts moving the sample up. During the movement the electronic system measures the following signals: the amplitude, phase, normal deflection of the probe and the intensity of the reflected laser beam. At the moment of reaching surface, the sharp change(s) occurs in one of these signals, the motor stops, and the scanner moves the sample 1 micron from the probe.

To start automatic approach procedure, click **m_approach** on the **AC mode** control panel. After starting the approach procedure, the program will go to the **Curves View** window, which displays the dependence of the amplitude of the cantilever (**Mag** signal) on the vertical position of the scanner (**fig. 3.6.2.2**). The scanner position is measured in millimeters, the origin – in the lowest position. Once the approach is completed, the "**motor approach completed, surface was found**" message is appeared.

Figure 3.6.2.2 Mag(Z) curve – dependence of the probe amplitude on the vertical position of the scanner during the approach procedure.

Press the **stop motor** button in case of any emergency stop. Press the **m_approach** button to continue the approach procedure.

3.6.2.3 Fine tuning of the oscillation frequency, phase and amplitude near the sample surface

After approaching to the surface, the amplitude of the probe oscillations may be slightly less compare to initial oscillations due to additional probe rubbing against the air leading to the frequency down shift.
Therefore, before landing on the surface it is desirable to adjust the working frequency, amplitude, and phase.
Press the **fine tuning** button on the control panel to return the working frequency on the top of the resonance peak and then press the **set amplitude** button to set the **Mag** signal to 25000. The **Phase** signal is set automatically to 0 ADC units, i.e. 90° phase shift.

3.6.2.4 Landing to the sample surface

Press the **landing** button on the control panel to start the landing procedure. The "**landing is running**" message appears. The scanner starts to approach the sample to the probe tip. The operation stops when the "**Mag**" signal reaches the "**SP**" parameter value. This parameter can be set in the corresponding input field on the control panel. If the amplitude is set to less than or equal 20nm, then the "**SP**" parameter is set to 85% from the initial amplitude of probe oscillation, otherwise this parameter is set to 70%.
After landing, the **Curves View** window displays the so-called "landing curve" – the Mag (Z) dependence (**fig. 3.6.2.4**) and the "**landing completed**" message appears in the information box.

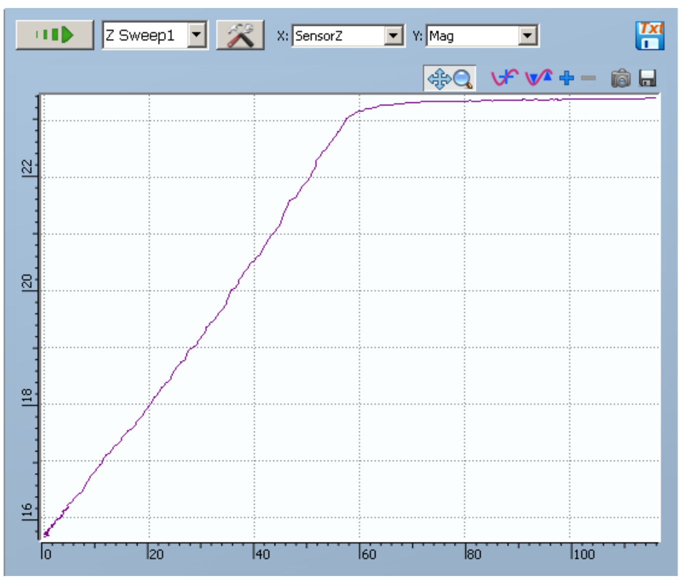


Figure 3.6.2.4 Mag(Z) dependence during the landing

3.6.3 Setting feedback gain

During the landing procedure, to avoid any possible undesirable oscillation caused by feedback (parasitic oscillations) **Gain** parameter of 8 units is used for the amplitudes less than 20 nm and 20 units for the others.
However, a higher gain is typically required for the correct scanning results. Press the **adjust gain** button on the control panel to find the optimal FB gain. A new value appears in the information window. The **Gain** value depends on the initial oscillation amplitude. The typical value is about 100 units for 20nm amplitude and 700 units for 200nm amplitude.

3.7 Adjusting the instrument for the Contact AFM mode

Select "**Contact mode**" from the "**Tools**" pull-down menu. The Contact AFM mode control panel appears (**fig. 3.1.4**).

The procedure of the manual instrument adjustment for the contact method is similar to the procedure for semi-contact mode described in paragraph 3.6, except for the tuning on the resonant frequency and for setting the amplitude of the initial amplitude of the cantilever.

3.8 Automatic instrument adjustment

The program has an option to automatically configure the device for working in different modes that allows saving time when routine operations must be performed.

At starting the automatic instrument adjustment, the program by itself accomplishes the sequence of operations that are used to manually configure the device. During automatic adjustment, the program switches between windows and displays different messages in the pop-up message box.

Automatic adjustment is begun with an analysis of the current state of the device. The program checks which of the parameters of the system are already set up and then starts configuring the device from the necessary step.

After switching on the device for the first time and also after replacing the probe, the procedure of the automatic instrument adjustment is performance from the first step.

The automatic adjustment device is started by clicking the **Auto** button on the **AC mode** or **Contact mode** control panels.

3.8.1 Automatic instrument adjustment for Semicontact / Non-contact AFM mode

For automatic adjustment of the instrument in the semicontact and the non-contact modes the following steps are sequentially completed:

1. Setting the position of the cantilever; program uses the **find tip** or **look around** procedures
2. Setting the position of the photodiode - **adjust diode**
3. Setting on the resonant frequency - **init tuning**
4. Setting the amplitude - **set amplitude**
5. Pause
6. Automatic approach - **m_approach**
7. Adjusting working frequency and phase - **fine tuning**
8. Setting the amplitude - **set amplitude**
9. Landing on the surface - **landing**
10. Selecting the feedback gain coefficient - **adjust gain**

Before running the automatic adjustment, set the initial amplitude of the cantilever. Set approximately the initial probe oscillation amplitude in nanometers in the input field below the "**set amplitude**" button on the control panel. For the non-contact AFM mode set the amplitude in the range of 5 – 20 nm. For the semicontact AFM mode set the amplitude in the range of 50 – 200 nm. By default the amplitude is 20 nm.

After entering the value of the amplitude, run auto setup procedure by pressing the **Auto** button, located on top of the control panel.

3.8.2 Automatic instrument adjustment for Contact AFM mode

The procedure of automatic instrument adjustment for the contact mode is similar to the procedure for semi-contact method described in paragraph 3.7.1, except for the tuning on the resonant frequency and for setting the amplitude of the initial amplitude of the cantilever.

3.9 Scanning

To start scanning, open the **Scan** window.
This window has the following default settings:

Scan mode:	**QScan** (closed loop scanning in XY direction).
Scan area:	10 microns. The scan area is situated in the center of the full area.
Scan rate:	1 Hz (one line per second).
Number of points:	256x256.
Measuring signals:	Height(Dac), Height(Sen).

Check these parameters and change them if necessary. Press to run  the scanning process. The detailed description of all **Scan** window controls can be found in the Chapter 5.4 «Control software reference manual».
During the scanning process the current profiles of the selected measuring signals are displayed in the **Section 1** and **Section 2** windows. These two windows are automatically opened and their positions are user-defined.

3.10 Adjusting scanning parameters

The main scanning parameters are the scanning rate "**rate**", feedback gain "**Gain**" and setpoint "**Sp**". For the semicontact/non-contact modes there is also the **initial probe oscillation amplitude**. The preset parameters are approximate and can be used for scanning some "average" sample, but as a rule each sample is unique. So, in order to achieve the best results, you need to experiment with parameters directly during the scanning process.

Scanning rate "rate"

In the program, the scan speed is measured in hertz, in other words this is the number of lines scanned per second. Generally speaking, the slower the scanning rate is the more accurate the feedback loop tracks the surface profile. Consequently, the resulting image is closer to the real surface topography. On the other hand, it takes more time, so try to set some optimal rate based on the viewed image, which should not have any kind of distortions.

Feedback gain – "Gain" parameter

The feedback loop response depends on this parameter. The lower the gain is the more blurred image is obtained. On the other hand, very high gains can cause some parasitic oscillations. It's recommended to set the "**Gain**" parameter a bit lower than the one when you start to observe parasitic oscillations (undesirable oscillation caused by feedback).

Setpoint and initial probe oscillation amplitude in semicontact/non-contact AFM modes

The "**Sp**" is amplitude of probe oscillation that the feedback loop maintains in the semicontact and non-contact AFM modes. The "**Sp**" may vary from the initial amplitude to zero. The lower the "**Sp**" is the stronger the interaction is between the probe and sample surface.
Set the initial probe oscillation amplitude in the range of 5-20 nm and the setpoint amplitude about 0.8-0.9 from the initial one (non-contact AFM mode) during <u>scanning any delicate samples</u> in order to minimize the influence of the probe onto the sample surface.

Set the initial probe oscillation amplitude in the range of 50-200 nm and the setpoint amplitude about 0.5-0.7 from the initial one (semicontact AFM mode) during scanning any rough samples or in case of the high speed scanning in order to make the instrument work more stable.

Setpoint in contact AFM mode

The "**Sp**" parameter is shift of the **Nf** signal in the contact AFM mode, which is proportional to the loading force the probe applies to the sample surface. So, the bigger the shift is the higher the interaction force becomes.
You may vary scanning **rate**, **Gain** and **Sp** during the scanning process. To change scanning rate, use the corresponding input field in the **Scan** window. To change feedback gain **Gain** and setpoint **Sp**, use the **Fb-Toolbar1** toolbar in the top of the main program window (**fig. 3.10.1**).

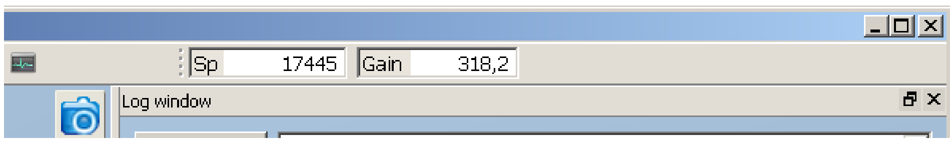


Figure 3.10.1 Fb-Toolbar1 toolbar

Change the **initial probe oscillation amplitude** only when the scanner is withdrawn from the probe.

Do the following steps to change the initial probe oscillation amplitude:

- Press the "**check amp**" button on the control panel to verify the "**coef**" parameter required for converting the amplitude from DAC units into nanometers. The setpoint amplitude and **coef** parameter measuring procedure runs. The information window appears showing the current amplitude and **coef** parameter after the procedure is completed.
- Press the "**Retract**" button on the control panel to retract the scanner to its lower position. The "**HV was set to 0**" message appears. The distance between the probe and sample is about 7 microns in such position.
- Input the required amplitude and corrected "**coef**" parameter in the corresponding input fields on the control panel and press the "**set amplitude**" button.
- Run **Landing** and **adjust gain** to continue or just press **Auto.**

3.11 Changing AFM mode

Switch between semicontact/non-contact and contact AFM modes ONLY when the scanner is withdrawn from the probe. To switch between "AC mode" and "Contact mode", press the "**Retract**" button and select the required mode from the "**Tools**" pull-down menu. Once the control panel appears, adjust the instrument following clause 3.6 - 3.8 of this manual.

3.12 Saving and processing scanning results

Use the "**Data view**" window to open, save and process your results.
All data obtained during one session are stored in one file. Inside such file you can find each measurement in a separate folder. Each folder contains not only scans of different signals, but also a table of parameters which were used during the measurement. It is also possible to include any oscilloscope data, spectroscopy and resonance curves in the same file (**fig 3.12.1**).

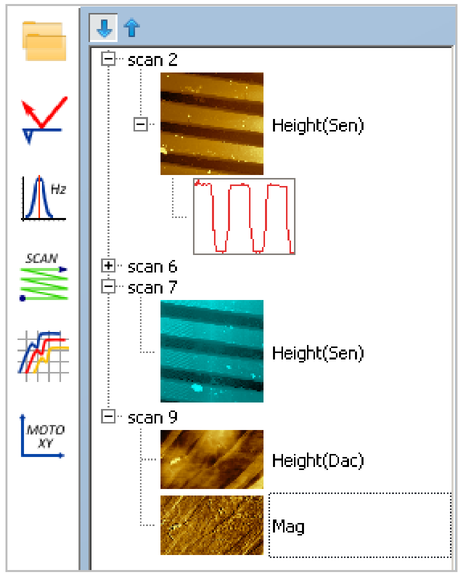


Figure 3.12.1 Data file structure

All data in the **Data view** window obtained either during scanning or loaded from file is automatically saved into a temporary file **aist3.db**. If you close the program with some data in the **Data view** window and open it again, it will open with the same data. This provides some certain level of your data protection in case you accidentally close the program or the program hangs.

Press **File → Save all..** in the main menu to save all data from the Data view window into one file. Use the **Save selection** command to save only the selected data into file.

Press **File → Open...** in the main menu to open your data.

Click on data name and then click it again to change its name.

To select a few data folders or images, use the standard key sequences: Ctrl+A, left mouse button + Shift, left mouse button + Ctrl.

Use the **Delete** button to delete data.

The detailed description of the **Data view** window controls can be found in the Chapter 5.1 "Control software reference manual".

3.13 Sample or probe replacement, shutting down

Either use the **move** button and the **dist** input field in the **ZMotor Ctrl** control panel or just the **New tip** button in the same panel to retract your sample from a probe before changing either of those.

NOTE: The **New tip** button moves a sample down to 2 mm.

Quit the program before shutting down the controller. If you quit the program, the current settings are saved and the sample is automatically retracted to 2 mm from the probe with the approach mechanism.

3.14 Measurements of curves

Using this machine, it is possible to record different signals dependencies on the distance between the tip and the surface. Depending on the selected mode, various curves are useful to obtain the correct data, keep the probe in good condition, and so on.

For example, in the semi-contact or non-contact AFM mode amplitude or phase curves are usually used to obtain the dependence between the amplitude or phase of probe oscillation and the gap between the probe and sample surface (**fig. 3.14.1**). From the amplitude curve one can obtain the information about the initial amplitude of oscillation, probe sticking or about the static charge on the sample surface. The phase curve gives the information about the interaction conditions between the probe and the sample surface. You can use these curves to properly select the initial amplitude or set point.

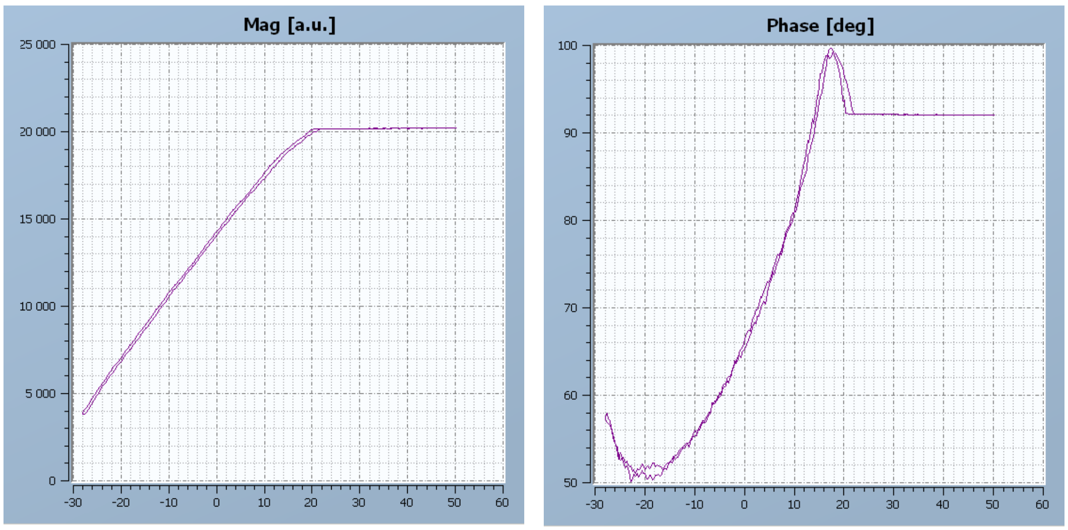


Figure 3.14.1 Amplitude and phase curves

In the contact AFM mode Nf(Z) curves are used to obtain the dependence between the cantilever deflection and the gap between the probe and sample surface (fig. 3.14.2). Another common name of such curves is “force-distance curves”. They are used to correct the setpoint (loading force). Also the force spectroscopy method is based on this dependence, which gives the information about a number of surface physical properties such as adhesion and elasticity.

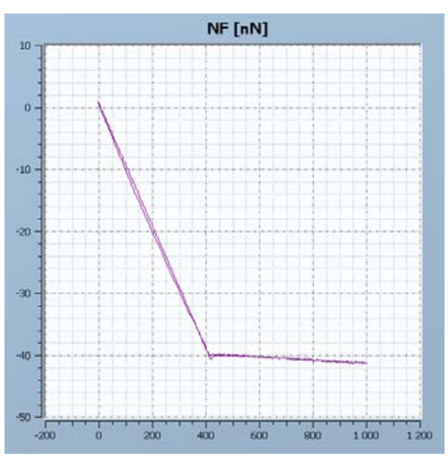


Figure 3.14.2 Force-distance curve

The **Curves view** window is designed to display the curves. Select the type of spectroscopy in the drop-down menu (Figure 3.14.3):

Z Sweep – spectroscopy with the advanced list of signals and additional options for the Z movement

BV Sweep – electric spectroscopy

The **Z Sweep** and **BV Sweep** control panels can be opened using the  button.

The detailed description of all the control elements can be found in the Chapter 5.5 of "Control software reference manual".

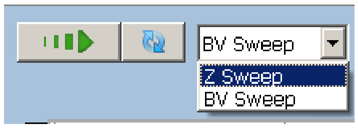
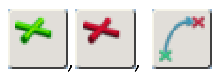


Figure 3.14.3 Spectroscopy type selection combo box in the **Curves view** window

To measure the curve, select the necessary settings and click .

A curve is obtained in one point where the probe is situated (please notice that right after the scanning process is complete, the probe is moved to the scan center). Use  buttons in the **Scan** window to assign the point where you want to obtain a curve.

During one spectroscopy cycle, all the signals allocated in the list are obtained (**fig. 3.14.4**). Depending on the selected SPM mode the corresponding signal list is automatically loaded. Set the required signals in the X and Y combo-box controls to make the corresponding curve shown on the screen.

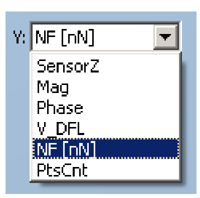


Figure 3.14.4 SPM spectroscopy **Z Sweep** signal list

NOTE: The **Z Sweep** curve measured in the contact mode displays force curve, in which the deviation of the probe is converted to units of force. For correct conversion, calibrate the signal NF (nN) first using the check slope procedure and then enter the **slope** and **stiffness** parameters in the corresponding input fields of the control panel for the contact method.

Using the  button you can attach the curve to the data tree or save it into a separate text file.

4. MEASUREMENT TECHNIQUES

The list of basic techniques can be opened from the drop-down menu in the top right corner of the **Scan** window (fig. 4.0.1).

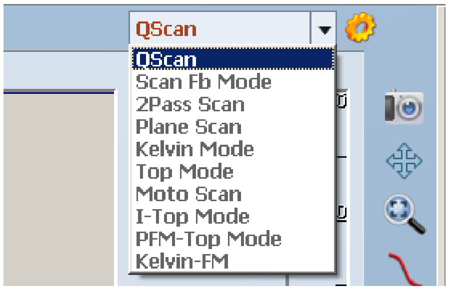


Figure 4.0.1 Available techniques in the drop-down list of the **Scan** window.

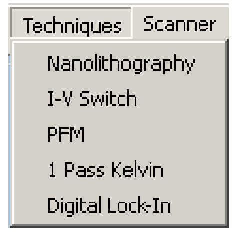


Figure 4.0.2 Available techniques in the **Techniques** drop-down list of the main menu.

The following techniques can be selected from this list:

Qscan –	high speed scanning;
Scan Fb Mode –	XY closed loop scanning;
2Pass Scan -	two-pass MFM/EFM;
Plane Scan –	single-pass MFM/EFM;
Kelvin Mode –	Kelvin Probe microscopy;
Top Mode –	"Top" mode;
Moto Scan –	scanning by stepper motors;
I-Top Mode –	conductive AFM in the Top mode (conductive unit is required);
PFM-Top mode –	piezoelectric response microscopy in the Top mode;
Kelvin-FM –	two pass Kelvin probe force gradient microscopy.

The SPM techniques such as **Nanolithography, IV-Switch** (conductive unit control window) and **PFM** (Piezoelectric Force Microscopy) can be opened from the **Techniques** drop-down list of the main menu (**fig. 4.0.2**).
The special techniques such as friction force measurements, calibration of spring constants can be run from the **Macro executor** window.

4.1 Low voltage mode

The low voltage mode is designed with the purpose of decreasing the noise level in the XY plane at scanning of small fields (less than 1 micron). In this mode the high voltage amplifiers are turned off from the X and Y axis of the scanner. As a result, the maximum scanning area is shrunk down to 5 um in the center of the initial 100 um scanning area. Low voltage mode is used for obtaining an atomic resolution and for working in the STM mode.

Follow the steps below to switch to the low voltage mode:

- Press the **"Retract"** button to withdraw your sample from the probe.
- Select the **"Relay"** tab in the **"Param table"** window and switch **"XY"** to the **"Low"** position.
- To continue the measurements press the "**Auto**" button to automatically engage your sample with the probe or press "**Landing**" to do it manually.

4.3 Two-pass magnetic force microscopy and electric force microscopy techniques

For each scan line, the height profile is recorded during the first pass. At the end of the first pass the probe is lifted to some certain distance above the surface. Then it is moved along the just-learned height contour at a constant lift height. During this second pass, the changes of the probe oscillation phase, which are caused by the long-distant magnetic or electric forces, are measured. In this way the height image is recorded in the first pass and the phase image recorded in the second pass that corresponds to the distribution of magnetic or electric field normal gradient component across the sample surface.

To use the Magnetic Force Microscopy technique, follow the steps below:

- Use a non-contact cantilever with a magnetic coating.
- Adjust your system to work in the semi-contact mode. Set up the initial probe oscillation amplitude to 20 – 100 nm.
- Make a test scan, i.e. obtain a topography image to ensure that all settings are valid.
- Select **2Pass Scan** in the list of the scanning modes to switch the instrument to the two-pass MFM/EFM technique. The MFM/EFM control panel appears (**fig. 4.3.1**):

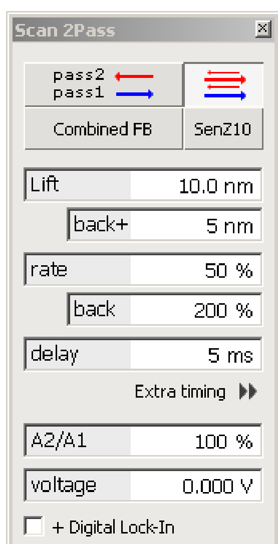


Figure 4.3.1 The **2Pass Scan** panel for using two-pass techniques.

- Set the height of the second pass in the **Lift** field. When scanning samples with unknown topographical properties, set deliberately high **Lift**, for example, 100 nm.

- Select **“Phase”** in the measuring signals list.
- Set the scan resolution to 256x256 pixels and the scanning speed to 1 s.
- Start the scanning process.

You can monitor the MFM image on the second pass, signal **“Phase[2]”**.

During the scanning, reduce **Lift**, so that on the second pass, the contrast is the highest, but at the same time the probe does not touch the surface of the sample.

To use the Electric Force Microscopy technique, follow the steps below:

- Use a non-contact cantilever with a conductive coating.
- Make the same steps to adjust the instrument as for the Magnetic Force Microscopy technique.
- Change the **Voltage** parameter in the **Scan 2Pass** control panel to increase your phase image contrast on the second pass by changing the voltage applied to your probe.

The functionality of the rest of the **Scan 2Pass** panel settings is discussed in the paragraph 5.4.8 of "Control software reference manual".

4.4 Single-pass MFM technique

With any of AIST-NT SPMs it’s possible to perform the MFM scanning in one single pass (**“Plane Scan”** mode). This mode is useful for scanning flat, uniform, soft-magnetic materials. The distribution of magnetic field normal gradient component in this mode is measured at some lift height and in the plane parallel to the sample surface. This way the possibility that the magnetic field of the probe can cause any magnetic reversal process on the sample surface is highly reduced. Before scanning the sample, the measuring plane is determined by nine points inside the selected scanning area.

To work in the **“Plane Scan”** mode, follow the steps below:

- Use a non-contact cantilever with a magnetic coating.
- Adjust your system to work in the semi-contact mode. Set up the initial probe oscillation amplitude to 20 – 100 nm.
- Once the landing is successfully done, select **“Plane Scan”** from the list of the scanning modes to switch the instrument to the **“Plane Scan”** mode.
- The sample surface estimation window appears (**fig. 4.4.1**). Set lift to 100-300 nm for the first scan.

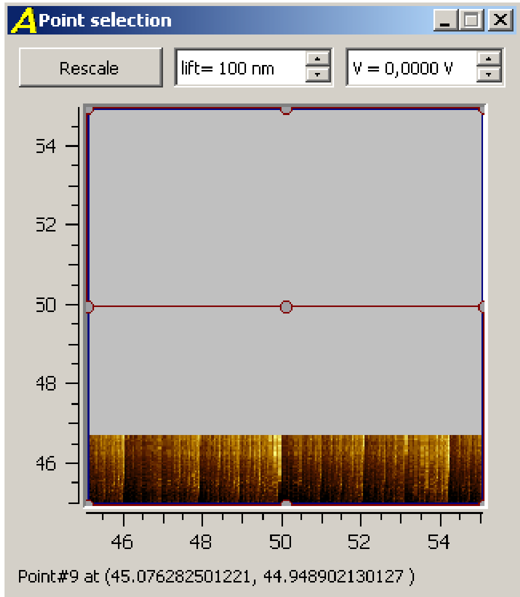


Figure 4.4.1 Surface estimation window

- Switch to the **"Scan"** window, select a scanning area and switch to the scanning area view mode using .
- Press the **Rescale** button in the sample surface estimation window to load the selected scanning area into this window.
- Leave only the **"Phase"** signal in the signals field.
- Start the scanning process.

The instruments starts estimating the sample surface by moving the probe around 9 selected points one by one. The scanning process starts right after the surface is successfully estimated. You may apply high scan rates due to the fact that the scanning is performed without tapping the surface. To increase the image contrast, decrease the lift height. Reload the scanning area into the sample surface estimation window once it's been changed.

NOTE: The **Plane Scan** mode can also be used for the single-pass EFM technique. Use non-contact cantilevers with some conductive coating for this technique. Change applied voltage using the **V** input field in the **Plane Scan** control panel.

4.5 Kelvin probe force microscopy (KPFM)

KPFM is a scanned probe method where the contact-potential difference between a probe tip and a surface can be measured. The measurement is performed in the two-pass technique. In other words, for each scan line, the height profile is recorded during the first pass. At the end of the first pass the probe is lifted to some certain distance above the surface and the potential offset is measured.

Follow the steps below:

- Make sure that your sample is grounded. Use a special sample holder with grounding contact. Place the fixing spring on your sample surface. Plug your sample holder contact into the grounding socket on the scanner's cover.

- Use a non-contact cantilever with a conductive coating.
- Adjust the instrument to work in the semi-contact mode. Set up the initial probe oscillation amplitude to 20 – 100 nm.
- Make a test scan, i.e. obtain a topography image to ensure that all settings are valid.
- Select **Kelvin mode** from the list of the scanning modes to switch on the Kelvin Probe Force Microscopy technique. The KPFM control panel appears (fig. 4.5.1).
- Press the **Auto Setup** button to automatically adjust the instrument to work in the KPFM mode.
- The **cpd** and **slope** parameter values are output in the **Log Window** right after the successful adjustment. The **cpd** parameter is a value of contact-potential difference in the point where the probe is during the adjustment.
- Turn on the **CPD** signal in the measuring signals list. Turn on the voltage feedback by marking the **Kelvin FB** checkbox.

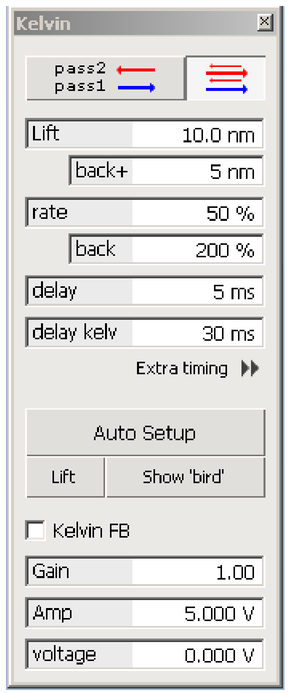


Figure 4.5.1 Kelvin mode control panel

- Start the scanning process. The surface potential signal is imaged in the **CPD[2]** signal in the second pass.

Parameters in the upper part of the panel correspond to the two-pass scanning and they are described in paragraph 5.4.8 of "Control software reference manual".

Settings of the Kelvin mode:

Kelvin FB – switches on the mag*sin feedback loop during the second pass. The feedback loopmaintains the constant potential between the probe and sample surface during the scanning process.

Gain – **Kelvin FB** feedback gain value during the second pass. The feedback signal is mag*sin.

Amp – AC voltage applied to the probe during the second pass.

Voltage – bias voltage applied to the probe during the second pass in the case the feedback loop is switched off.

Lift – switching the device on the second pass.

Show "bird" – shows the dependence between the probe oscillation amplitude and applied AC voltage (dependence appears in the **Curves view** window).

4.6 Piezo Response Force Microscopy

The Piezo Response Force Microscopy (PFM) is a technique used to investigate the mechanical response of a piezoelectric material under the influence of electric voltage. The probe is scanned over the sample surface in hard contact. An AC voltage is applied between the probe tip and sample surface. Due to the piezoelectric effect some features on the sample surface (i.e. grains or domains) change their dimensions under the applied voltage and therefore make the probe oscillate. Amplitude and phase of such oscillations are detected by two synchronous detectors. One detects normal cantilever oscillations and another detects lateral ones. The oscillation amplitude signal gives the information about a value of piezo response while the oscillation phase signal about a direction of polarization.

In the AIST-NT software ther is two methods of Piezo Response:

1. Contact PFM mode

2. Point-by-point Measurment of Piezo Response, i.e. PFM-Top mode.

In contact PFM mode scan performed in contact mode.

In PFM-Top mode the movement between points of the scan is carried out in tapping mode, and at each point of measurement for Piezo Response made the transition to the contact mode.

4.6.1 Choice of PFM technique

Choice of technique depends on the properties of the sample. If the sample contact method allows the study, it can be used as a contact PFM and PFM-Topmode. For those samples for which contact is inappropriate, use PFM-Top mode.

Drawback regime PFM-Top mode is a low scanning speed, because at each point of the scan the transition between contact and semi-contact mode, which takes a lot of time.

4.6.2 Choice of cantilever

To contact PFM mode can be used contact and non-contact cantilevers . In PFM-Top mode used noncontact cantilevers .

NOTE: The use of non-contact short cantilever provides a higher level of the normal response . This is due to the fact that at one and the same normal vibration amplitude of a piezoelectric the short cantilever is deflected at larger angle than the long cantilever.

For PFM mode used cantilevers with the conductive coating. However it is possible to use uncoated cantilevers, because the conductivity of silicon is usually sufficient to bring the voltage to the sample.

4.6.3 Sample Requirements

A non-conductive piezoelectric material should be disposed on a conductive substrate which forms the lower electrode . In the absence of a conductive layer on the sample as a lower electrode , use any metal plate on which the sample is secure .

4.6.4 Installing the Sample

To install the sample use the sample holder with a spring contact . Set contact so it will be connected to the lower electrode of the sample. Contact wire connect to the earth socket on the unit .

4.6.5 Contact PFM mode control panel

Open contact PFM mode control panel from Techniques menu:

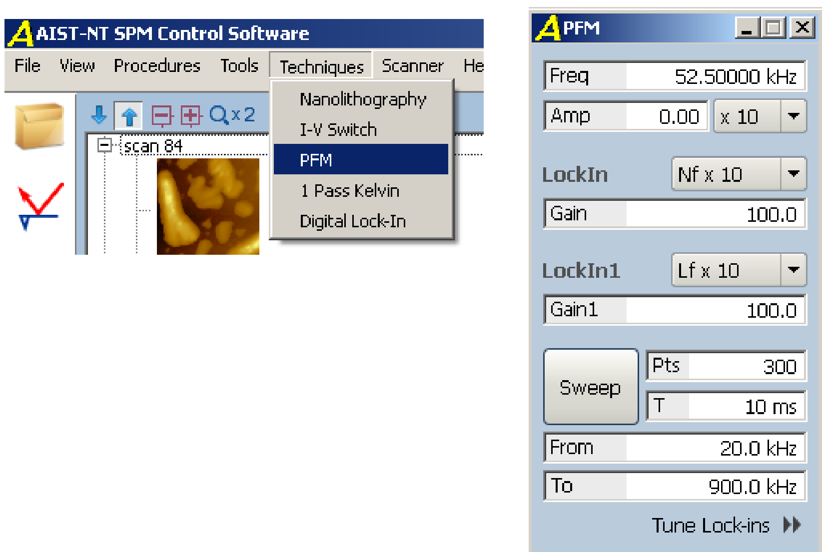
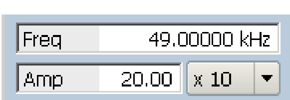


Figure 4.6.1 PFM control panel

The parameters that you can find on the PFM control panel are the following:

Voltage parameters applied to sample:



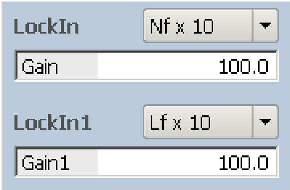
The voltage provided by built-in generator controller Gen.1, which is automatically connected to the probe (voltage electrode) when the contact PFM mode in on. For safety reasons, the control panel at the opening of the voltage amplitude of the modulation are always set to zero.

Freq – Frequency

Amp – Amplitude

x10, x1, x0.1 – Range switch of the generator. The maximum amplitude of 100x10 equals to 10 volts.

Oscillation detection parameters:



For the detection of the cantilever oscillation using controller synchronous detectors. For the detection of the normal vibrations of the cantilever used Lock In (another name - Lock In 2) and for lateral oscillations - Lock-In1.

Parameters of the synchronous detector Lock-in:

Nfx10 - input synchronous detector is supplied pre-amplified 10 times the normal cantilever deflection signal Nf;
Gain - Gain (gain maximum use of 100);

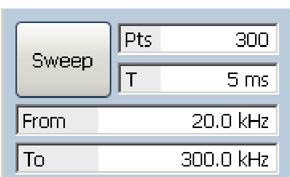
Parameters of the synchronous detector Lock-in1:

Lfx10 - input synchronous detector is supplied pre-amplified signal 10 times the lateral deflection of the cantilever Lf;

Gain1 - then the maximum gain of 100;

Parameters for obtaining an amplitude-frequency characteristic (AFC):

PFM control panel provides an opportunity to get AFC of the cantilever pressed against the surface. Such an AFC is needed in order to correctly set the frequency of the AC voltage applied to the sample. AFC displays in the Curves view window:



Sweep – scan by frequency;

Pts - number of points in the curve;

T - the measurement time in point;

From, To - scanning range;

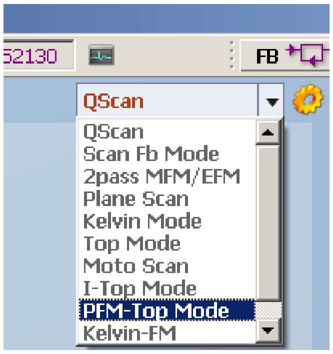
Setting the synchronous detectors:

Tune Lock-ins - starting the setup procedure zeros synchronous detectors (see paragraph 4.6.9).

NOTE: Contact PFM mode control panel can not be closed during working - with the closing panel PFM mode is turned off.

4.6.6 Contact PFM-Top mode control panel

Contact PFM-Top mode control panel can be opened from the list of methods in the Scan window:



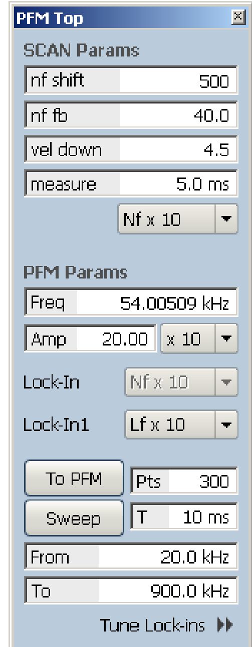
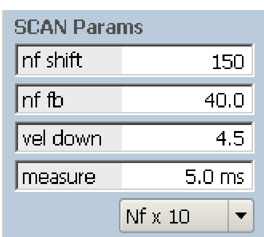


Figure 4.6.2 Contact PFM-Top mode control panel

The panel contains the following elements:

Scan Settings Scan Params:



Scanning parameters determine the measurement signals PFM switching speed between the semi-contact and contact methods, as well as the operating point and the feedback gain for the contact method.

Nf shift - parameter defining the force exerted by the tip to the sample in contact mode : after entering the contact method Nf signal should be reduced by the amount of Nf shift;

Nf fb - feedback gain in the contact mode;

Vel down - closing speed of the probe and the sample during the transition from semi-contact in the contact mode;

Measure - measuring time PFM signal;

Nfx10 - preamp signal Nf 10 times before digitization;

Preamplifier Nfh10 should be used when working with short non-contact probes to increase stability in the contact mode . Nf shift parameters and Nf fb scaled automatically when disconnected gain Nfx10.

PFM parameters:

Options "PFM Params" within the parameters of the contact panel PFM mode.
Button "To PFM" switches the system in tapping mode and contact mode involves measuring PFM signals.

4.6.7 Work in contact PFM mode

- Set the sample as described in paragraph 4.6.4.
- Set the cantilever. Recommendations for choosing a cantilever given in paragraph 4.6.2.
- Turn the control panel contact method Contact mode on and configure the device to work in contact mode.
- Run to the surface and get a test scan of the surface topography , in order to verify the accuracy of the scan settings.
- Turn on the piezoelectric response measurements by selecting PFM in Techniques windows. The Contact PFM-Top mode control panel should be opened (Fig. 4.6.1).
- The first time the PFM is turne on in this session make the setting of synchronous detectors using procedures Tune Lock-ins (see paragraph 4.6.9).
- Set the voltage and frequency in accordance with the recommendations in paragraphs 4.6.10 and 4.6.11.
- To scan in the contact PFM mode can be used QScan or Scan Fb mode.
- In the scan window in addition to the height turn signals Mag, Phase (normal amplitude and phase) and Mag1, Phase1 (lateral amplitude and phase) .
- Start scanning. Scanning speed is selected based on the magnitude of the measured signal amplitude : the smaller the signal level the slower the scan is necessary, since the accumulation of weak signal requires more time . Commonly used small speed is less than 1 Hz.
- During the scanning process adjust the phase in accordance with paragraph 4.6.12.1. If necessary, use filtering signals (4.6.12.3).

As an example how the contact PFM mode works in Figure 4.6.3 shows images of the amplitude and phase of the normal vibrations of the cantilever obtained at the junction between the two domains on the sample of lithium niobate . Used short proximity cantilever fpN10. Operating frequency was chosen in the nonresonant region . From these images one can say that the domains have the same response of normal and opposite directions of polarization.

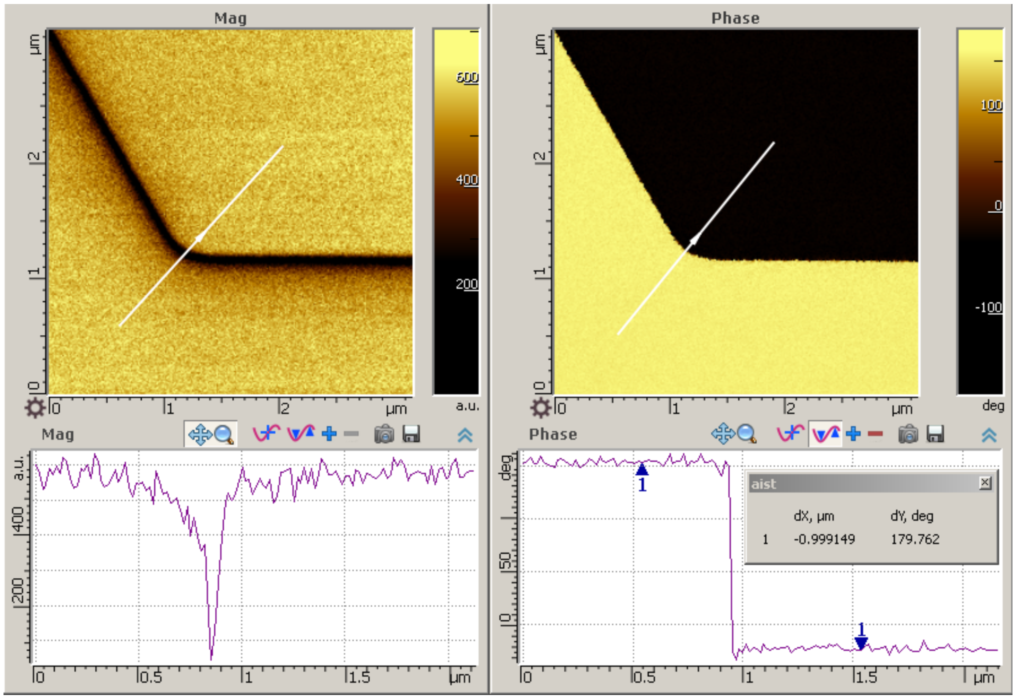


Figure 4.6.3 Images of the amplitude and phase of the normal vibrations of the cantilever obtained at the junction between the two domains on the sample of lithium niobate .

4.6.8 Operation in PFM-Top mode

- Set the sample as described in paragraph 4.6.4.
- Set the cantilever. Recommendations for choosing a cantilever given in paragraph 4.6.2.
- Turn on AC mode control panel and configure the device to work semicontact.
- Run to the surface and get a test scan of the surface topography , in order to verify the accuracy of the scan settings.
- Turn on the poin-by-point measurement piezoelectric response by selecting PFM-Top Mode from Scan window. PFM-Top mode control panel should be opened (fig.4.6.2).
- The first time the PFM is turne on in this session make the setting of synchronous detectors using procedures Tune Lock-ins (see paragraph 4.6.9).
- Set the voltage and frequency in accordance with the recommendations in paragraphs 4.6.10 and 4.6.11.
- In the scan window in addition to the height turn signals Mag, Phase (normal amplitude and phase) and Mag1, Phase1 (lateral amplitude and phase).
- Start scanning.
- In contrast to the contact PFM mode in PFM-Top mode scanning speed is not a parameter governing the accumulation time signals, speed change only affects the transition between points. Time spent at point (signal acquisition time) is determined by the parameter "measure".
- During the scanning process adjust the phase in accordance with paragraph 4.6.12.1. If necessary, use signals filtering (4.6.12.3).

Example scans obtained in PFM-Top mode is shown in Figure 4.6.4. The sample represents islands of piezoelectric material disposed on the metallized substrate. From left to right are shown: topography, lateral amplitude and lateral phase. On the amplitude signal are clearly visible domains boundaries, and phase signal shows that these domains have opposite polarization.

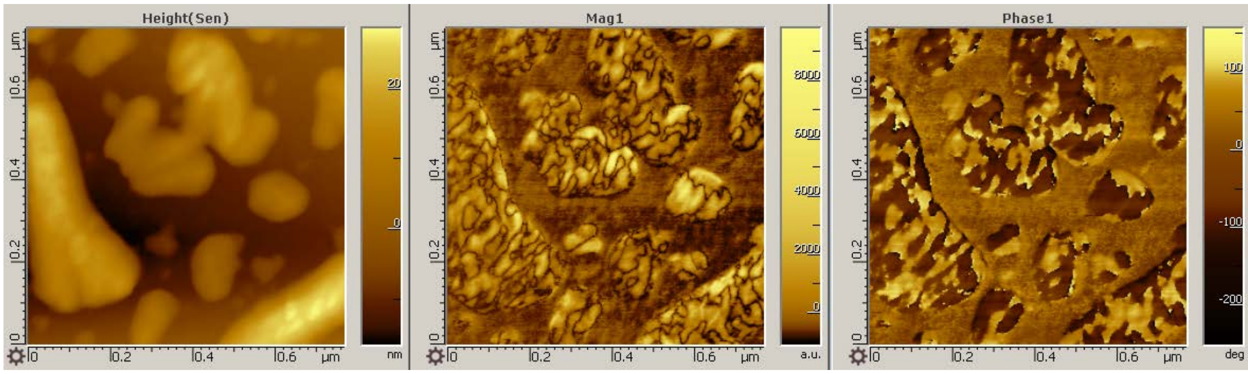


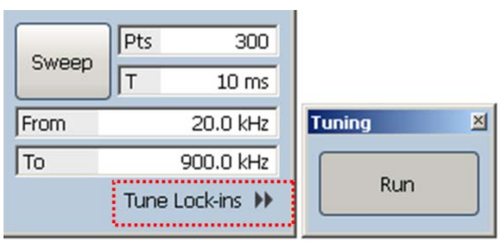
Figure 4.6.4 Topography and lateral piezoelectric response obtained in the PFM-Top fashion.

4.6.9 Setting the synchronous detectors

Before the measurements in PFM or PFM-Top modes, you must configure the synchronous detectors. When setting is done compensation of zeros of the synchronous detector is made, so in the operating frequency band the output signal is minimized due to the absence of an input signal.

Do the following:

- Open the PFM or PFM-Top mode control panel.
- Make sure that signals of Nfx10 and Lfx10 are received by synchronous detectors.
- By clicking on the arrows next to Tune lock-ins, open the Tuning window:



- On the panel that appears, click Run.

After starting the scanner settings will take a sample from the probe . Setup takes about half a minute . During setup window displays Curves View AFC synchronous detectors, recorded at different amplitudes of modulation.

Upon completion of the settings panel Tuning automatically closes. For landing on the surface, follow the procedure “landing”.

Setting synchronous detectors are required only once in this session. There is no need to repeat procedure after switching methods or change cantilever/sample.

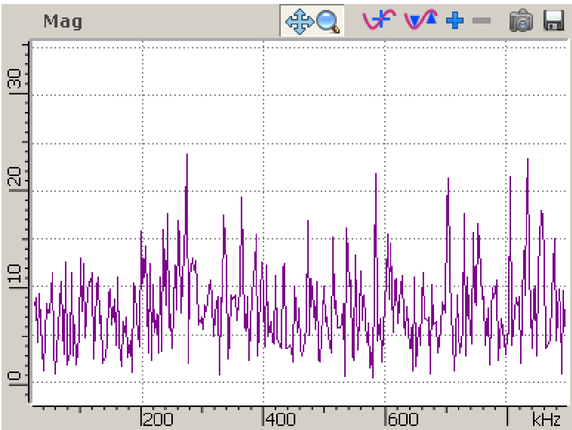
NOTE: To see the results of adjustment necessary to record AFC of the device.

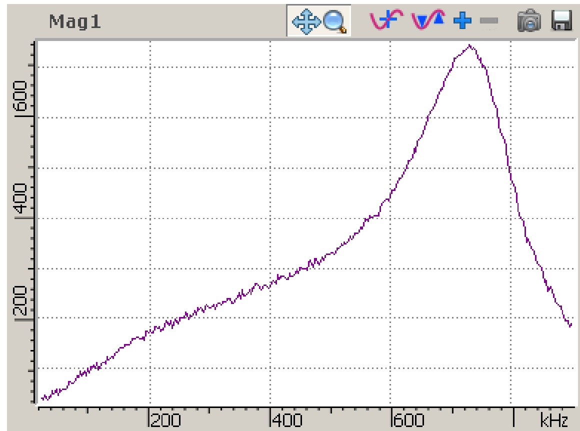
Do the following:

- Set the instrument blank probe holder (without the cantilever). Connect the holder to the measuring head.

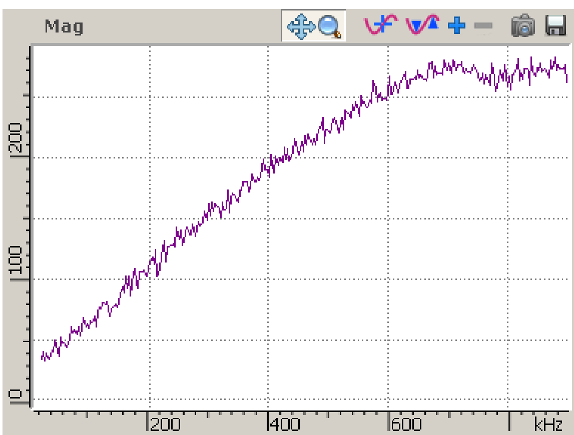
- Set the modulation voltage of 10 volts, set fairly wide range of scanning by using parameters From and To, and run the scan by pressing Sweep button.

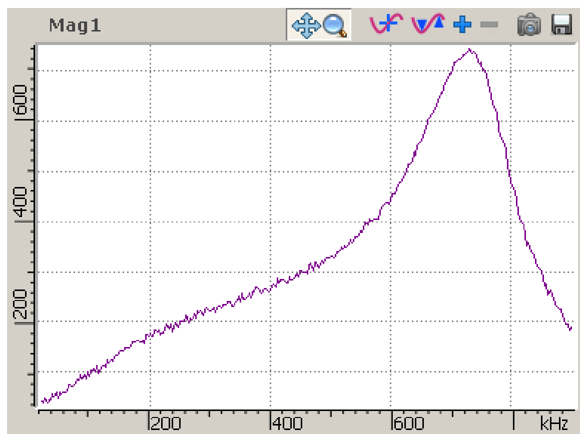
In the Curves view window should look something such curves for signals mag (corresponding to the synchronous detector Lock-in) and mag1 (corresponds to the synchronous detector Lock-in):





To get the AFC without compensation zeros, Right click on Sweep button and uncheck Zeros compensation. Then record the AFC again. Curves without compensation zeros (i.e. before the configure of synchronous detectors) look like this:





4.6.10 Voltage selection

PFM typically used for large enough amplitude modulation of the voltage of a few volts.

As the voltage increases the magnitude of the response .

NOTE: If the front side of the sample are exposed areas of the lower electrode, the conductive coating of needle of probe in contact with these areas may be burned at high voltages.

4.6.11 Frequensy selection

In order to select the operating frequency AFC of cantilever pressed against the sample surface must be recorded.

To do this, follow these steps:

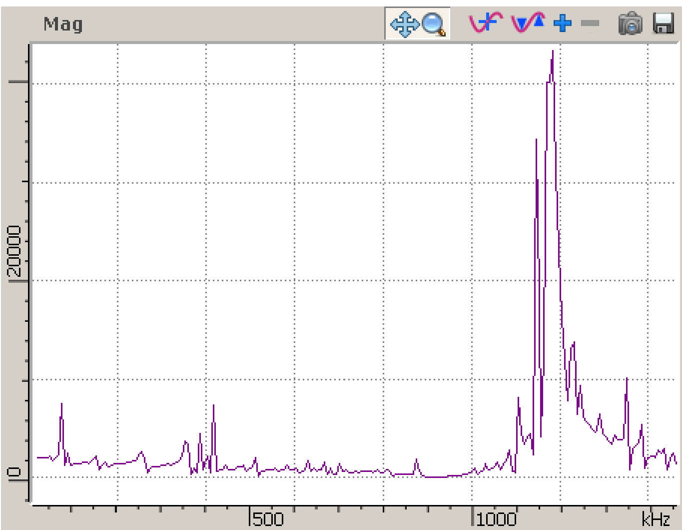
- Install the sample, set cantilever and move to the sample surface .

- Open the appropriate PFM mode control panel.

- Set the modulation voltage of 2 volts, set fairly wide range of scanning by using parameters From and To, and run the scanning by pressing the Sweep button.

- In the Curves view window AFC will be displayed.

As an example of AFC resulting in a sample of lithium niobate by noncontact cantilever fpN10 with a resonance frequency of about 200 kHz at a modulation amplitude of 10 volts:



On this curve, the resonance peak at 1200 kHz corresponds to the resonance of cantilever landed on surface of the sample (the frequency of this peak in the 7 times bigger than the resonance frequency of the free cantilever). Other minor resonant peaks are most likely the resonances of the sample.

Operating frequency PFM can be installed on the resonance peak and in areas where no resonances .

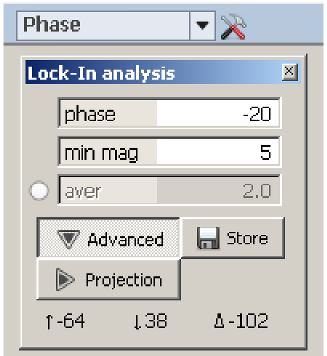
When installing the frequency resonance peak levels measured signals will be higher, but the resonance peak may change a little due to scanning due to changes in contact cantilever to surface according to the relief of the sample. This change leads to artifacts in the signals piezoelectric response .

Therefore, if the signal levels are high enough (a few hundred a.u.), the frequency should be set to no resonances below resonance of cantilever landed on the surface . This frequency setting provides the most reliable data.

4.6.12 Additional signal processing of PFM

Additional features of signal processing provides a form of Lock-In analysis. Access to this form is only possible after opening panels PFM or PFM-Top.

If the scan window is displayed the selected one of the PFM signals (Mag, Phase, Mag1, Phase1), then right field signal selection button appears . Clicking on it opens a Lock-In analysis window:



ATTENTION! Process signals using the form Lock-in analysis and save the results in a tree data is only available until the next start the scanning procedure. Using this form for processing images downloaded from the data tree program is not impossible. To overwrite the image data into a tree after scanning you must click on "Store" button.

4.6.12.1 Setting phase

Phase signal in SmartSPM has a scale from 0 to 360 degrees. If the phase becomes larger than 360 degrees, it starts counting again, and the image phase jump occurs. Phase should be adjusted in order to avoid such jumps within the domain of the phase. To adjust the phase change the parameter "phase".

Also, when you click "Advanced" at the bottom of the form displays the current phase of the two opposite vectors maximum response and maximum phase difference. Optimally adjust the phase so that one of the layers was about -90, another 90 degrees.

4.6.12.2 Threshold phase calculation

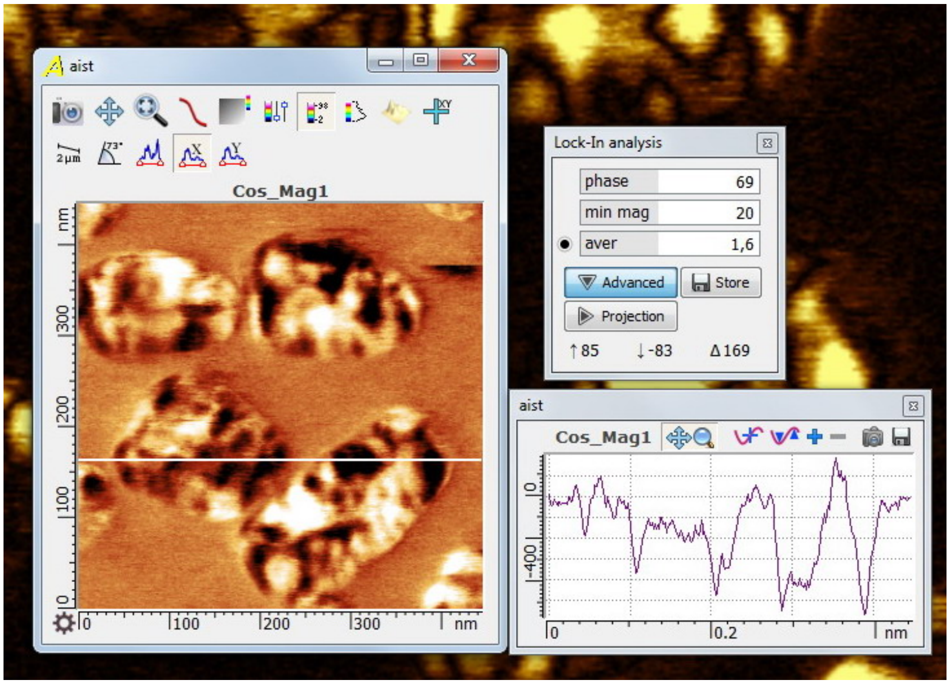
Threshold computation phase "min mag" is intended to reduce the phase noise in the areas in which no piezoelectric response. If the amplitude is less than the "min mag", then the phase at this point is not calculated and sets in 0 degrees.

4.6.12.3 Signal Filtering

For filtering signals used parameter “aver”. Band filter is given by: 2 Pi Bandwidth = 1 / (aver * T), where T - time per point when scanning. This filter replaces the standard low-pass filter applied to the output and lockin MagCos MagSin (standard filters - external synchronous detectors) . Processing functions simultaneously and raster Mag, and raster Phase. Unlike the standard version, this filtering is applied to the entire raster at any time. Since the filter is applied to the lines already obtained data when filtering occurs parasitic phase shift image (In the case of the standard filter with decreasing filter bandwidth , the picture moves to the right) .

If the filter is switched off , it can be assumed that the band of the measured signals is given by 2 Pi Bandwidth = 1 / T (this filtering algorithm is provided in the scan DSP).

4.6.12.4 Projecting piezoelectric response



PFM- response can be represented by one image , instead of two (amplitude and phase). To do this, at each point of the image output lockin (vector with components and MagSin MagCos) projected on the direction of maximum response . If you click on

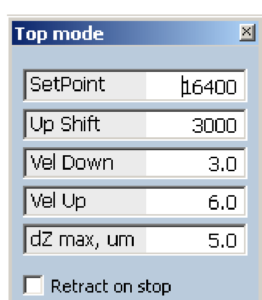
button "Projection" in a floating window displays the result of this processing raster - raster "Cos_Mag". This can be done both after and during the scan (when new data arrives from the controller the image will be updated) .

If the window was open during the scan , the raster "Cos_Mag" remain in the data tree . After scanning raster can be saved by clicking on the "Store" button.

4.7 Top mode

The **Top Mode** scanning mode is used when scanning in the standard AFM modes (semicontact or non-contact) becomes difficult due to some parasitic oscillations in the feedback loop. Parasitic oscillations usually appear when the tip is stuck in the surface at some low driving amplitude. It's also possible for parasitic oscillations to appear when scanning is done at some sharp edges (holes, cracks, sharp spikes etc.). In this case the tip starts touching the sample by its lateral surface. In the **Top Mode** the probe is lifted up above the sample surface between two scanning points. In each scanning point the probe is approached back to the surface. The scanning signal is measured right after the tip oscillation amplitude reaches the set threshold. This way it makes it possible to avoid any parasitic oscillations.

Select **Top Mode** from the list of the scanning modes to switch on the **Top Mode** technique. The **Top Mode** control panel appears:



You can see the following parameters:

SetPoint –	**Mag** signal value at which the measurement is done. When start scanning, the program writes the SP value, which was established for the conventional scan to the **Top mode** panel.
Up Shift –	Setpoint shift-up value, i.e. **Mag** signal change before measurement in the next point.
Vel Down –	Speed when the tip moves down
Vel Up –	Speed when the tip moves up
dZ max –	Limitation on the maximum distance between the probe and the surface.
Retract on stop –	Retraction of the probe from the surface at the end of the scan.

The Top Mode algorithm is described as follows:
In the initial point the probe tip moves towards the surface until the **Mag** signal drops down to the **SetPoint** parameter value. Once the **SetPoint** parameter value is reached, the system measures the height and other signals if they are set in the scanning window. While the tip moves to the next point, it's removed from the surface with the **Vel Up** speed until the **Mag** signal exceeds the **SetPoint + Up Shift** threshold. In the next point the tip moves again towards the surface until the **Mag** signal drops down to the **SetPoint** parameter value.

To run the system in the Top Mode, do the following steps:

- Set either the semicontact or non-contact AFM mode. You may set rather small initial driving amplitude, i.e. a few nanometers.
- Select **Top Mode** in the list of the scanning modes to switch on the Top Mode technique.
- Set **SetPoint**, so if it's increased by **Up Shift**, the probe tip doesn't lose the contact with the surface.
- Select the **Height(Sen**) from the signal list. The **Height(Dac)** is not used in the **Top Mode**.
- Do not set too many points per scan line as the system work slower in the **Top Mode** than in the standard mode. We recommend to set 256x256 points.
- Run the scanning process.
- To finish the operation and to return to the normal scanning, close the **Top Mode** control panel.

4.8 Nanolithography

The force and electric nanolithography modes are used for the fabrication of different patterns on your sample surface. Any of the AIST-NT's scanning probe microscopes allows the user to use the tip as a tool to mechanically extrude or scratch the surface (i.e. force nanolithography), or chemically modify it (i.e. electric nanolithography), for instance, using the local anodic oxidation method. The modification is done accordingly to some pre-defined pattern (vector or raster).

There are the following nanolithography modes available:
Vector modes (i.e. to create a pattern a number of geometrical primitives such as points, lines, curves, and shapes or polygons are used):

Vector pulse force – vector pulse force nanolithography. One or a number of force pulses (i.e. the tip is moved towards the surface to some pre-defined distance) are applied to the surface either in one point or in each point along a line.

Vector constant force – vector force nanolithography. In the initial point the contact AFM mode is turned on and the tip is pressed into the surface with some pre-defined force. The pressing force in the process of nanolithography is kept constant due to the feedback loop.

Vector pulse voltage – vector pulse electric nanolithography. One or a number of electric pulses (i.e. some voltage is applied to the tip) are applied either in one point or in each point along a line.

Vector constant voltage1 – vector electric nanolithography, variant 1 (for contact or semicontact AFM modes). The constant voltage is applied to the tip in the process of nanolithography along a line.

Vector constant voltage2 – vector electric nanolithography, variant 2 (for semicontact AFM mode only). The constant voltage is applied to the tip in the process of nanolithography along a line, but it's also possible to set the tip driving frequency and setpoint.

Raster modes (i.e. to create a pattern a data structure (bitmap) representing generally a rectangular grid of pixels is used) are available in four variants similar to vector modes in terms of the way the sample surface is modified:

Raster constant force – raster force nanolithography;

Raster pulse voltage – raster pulse electric nanolithography;

Raster constant voltage1 – raster electric nanolithography, variant 1;

Raster constant voltage2 – raster electric nanolithography, variant 2;

Notice that during jumping between lines in vector modes and between points in pulse and raster modes, the feedback loop is on according to the settings of the AFM mode selected earlier.

4.8.1 Nanolithography control panel

Select **Nanolithography** in the **Techniques** submenu of the main program menu to open a nanolithography control panel. The control panel includes a number of tools which allow creating either vector or raster patterns (**fig. 4.8.1**). It also includes the tool to add some text as a pattern.

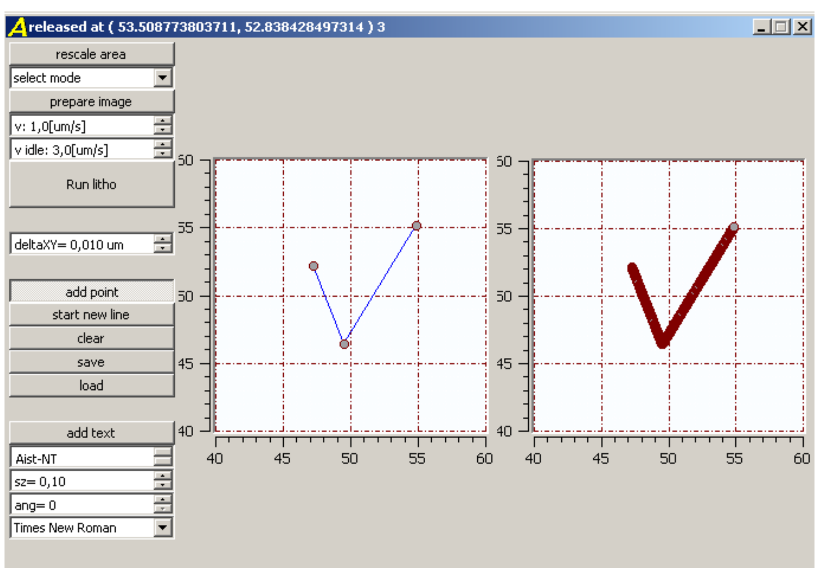


Figure 4.8.1 Nanolithography control panel for vector modes

Below is the description of each control item:

Rescale area –	loads the scanning area set in the scanning window into the nanolithography control panel.
Select mode –	you can select a nanolithography mode from this combo box. The corresponding settings panel appears once the mode is selected.

If no mode is selected, but the nanolithography process is started, the scanner will move accordingly to the settings of the AFM mode selected earlier without any influence on the surface.

Prepare image –	prepares the pattern for nanolithography, i.e. sets the points array according to which the tip moves during the nanolithography process. Use the **deltaXY** parameter to set the distance between two points;
v –	tip movement speed during the nanolithography process, set in microns per second;
Run Litho –	runs the nanolithography process.

Vector nanolithography tools:

deltaXY –	distance between the points where nanolithography is made;
add point –	adds a new point to the line;
start new line –	starts a new line;
clear –	deletes pattern;
save –	saves pattern;
load –	loads pattern from file;
add text -	adds text;
Aist-NT -	text input field;
sz -	font size;
ang –	text angle;
Arial -	text font.

Raster nanolithography tools:

open raster –	loads a raster pattern from file.
clear raster –	deletes raster pattern;
left-top X –	initial point and direction selection combo box.

4.8.2 Nanolithography settings panels for different nanolithography modes

Use the nanolithography settings panels to adjust and check the selected nanolithography mode parameters (**fig. 4.8.2, 4.8.3**). Each mode has its own settings panel.

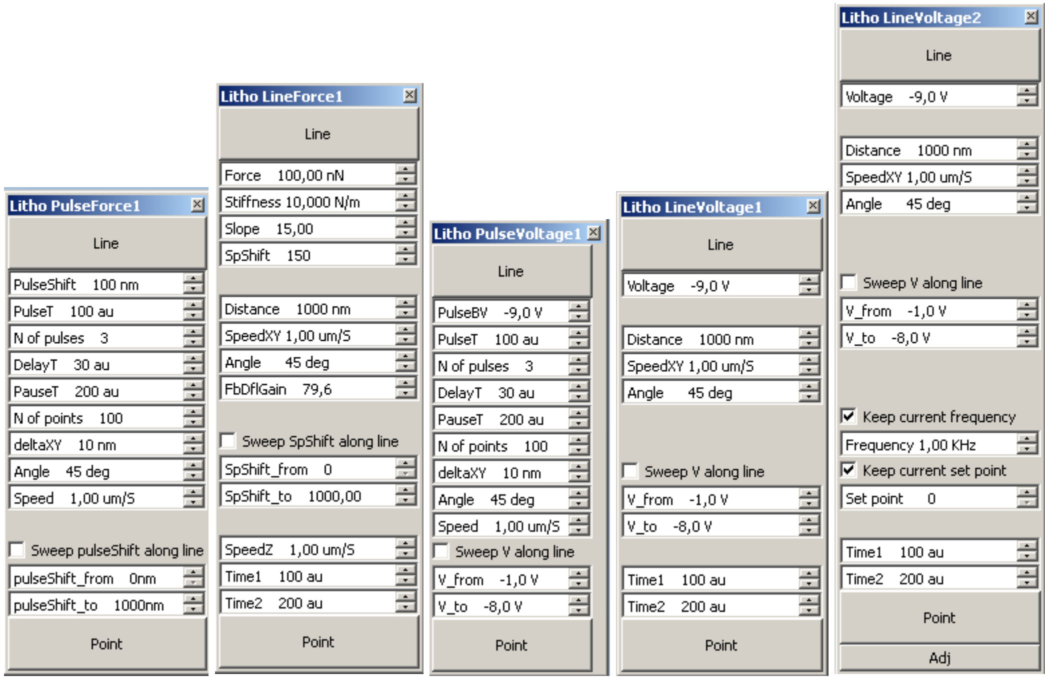


Figure 4.8.2 Settings panels for vector modes

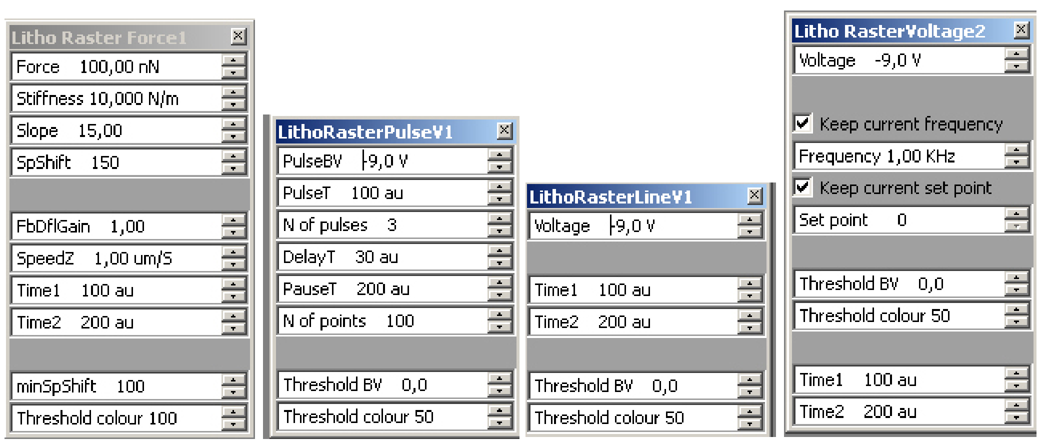


Figure 4.8.3 Settings panels for raster modes

Below is the description of each control item:

Buttons:

Line—runs the single line nanolithography. The initial point is the current tip position. The single line nanolithography parameters are **N of points**, **DeltaXY**, **Angle**, **SpeedXY**, **Distance**.

Point—runs the single point nanolithography.

Parameters:

PulseShift — tip shift distance value in vertical dimension during one pulse, in nanometers (nm);

PulseBV— voltage applied to the tip during one pulse, in volts (V);

PulseT—pulse duration, in arbitrary units (au*****);

Delay T—time delay before switching on the feedback loop after one pulse;

Pause T—pause duration before the next pulse;

N of pulses—number of pulses in one point;

N of points—number of points, in which the nanolithography pulses are performed;

DeltaXY —distance between the points;

Angle—tilt angle of the line made of the points. Zero tilt angle means the X axis. The tilt angle is measured counter-clockwise.

SpeedXY—jump speed between two points;

Distance—line length;

Force—pressing force value during the force nanolithography;

Stiffness—probe stiffness;

Slope—ratio between the scanner vertical displacement and cantilever deflection;

SpShift—setpoint shift value for the force nanolithography;

FbDFLgain—feedback loop coefficient calculated based on the cantilever deflection (Nf signal) and used for performing the force nanolithography;

SpeedZ—tip movement speed in Z dimension in the initial point of each line;

Time1—voltage change duration (in electric nanolithography), duration between switching off the driving oscillation applied to the probe and the beginning of nanolithography process in the first point in the line (in force nanolithography);

Time2—duration of the nanolithography in one point called using the **Point** command;

Voltage—voltage applied to the tip during the electric nanolithography;

Sweep voltage along—linear change of voltage along the line during the electric nanolithography process.

V_from—voltage value in the initial point;

V_to—voltage value in the end point;

Keep current frequency—check this check box to use the current tip driving frequency (i.e. driving frequency which is set in the scanning parameters) for the nanolithography.

Frequency—tip driving frequency used for nanolithography

Keep current set point—check this check box to use the current setpoint (i.e. setpoint which is set in the scanning parameters) for the nanolithography.

Set point—setpoint used for nanolithography.

In case of the raster nanolithography the level of pressing force or applied voltage corresponds to the shades of gray color.

Treshhold BV—voltage value that corresponds to the minimum exposure during the raster electric nanolithography. The maximum exposure is determined by the **Voltage** parameter.

MinSpShift—setpoint shift value that corresponds to the minimum exposure during the raster force nanolithography. The maximum exposure is determined by the **SpShift** parameter.

Treshhold colour—template pattern color that corresponds to the minimum exposure during the raster nanolithography. The maximum exposure corresponds to white color.

***** one arbitrary unit corresponds to one DSP cycle (approximately 50 µs).

4.8.3 Performing nanolithography

Follow the steps below to perform nanolithography:

- Force nanolithography:
  - Use relatively soft polymer samples. We recommend using a DVD disk surface as the best test sample.
  - Use a stiff non-contact cantilever (≥10 N/m).
- Electric nanolithography:
  - Use samples coated with some GaAs, CoCr or Ti layer.
  - Ground the sample. For this, use some conductive tape and ground contact.
  - Use either a contact or non-contact cantilever with a conductive coating.
  - The higher the humidity level is the more effectively you can run the local anodic oxidation.
- Adjust the instrument to be used either in the semicontact or contact AFM mode. In case of the semicontact AFM mode, set the initial tip driving amplitude to 20-70 nm.
- Make a test scan. Select a uniform and clear area on the sample surface to run nanolithography.
- Set the scanning area according to the one to be allocated for the nanolithography pattern. Press  to select the scanning area view mode.
- Select **Nanolithography** in the **Techniques** submenu of the main program menu to switch the instrument to the nanolithography mode.
- Press the **rescale area** button to load the selected scanning area into the control panel.
- Select the required nanolithography mode from the **select mode** combo box.
- The corresponding settings panel appears. All settings are pre-defined, but you may correct them if necessary.
- Use the corresponding tools from the control panel to create a nanolithography pattern, load it from file or add some text.
- Change the pre-defined speed (**v**) and distance (**DeltaXY**) parameters if necessary.
- Press the **prepare image** button. The nanolithography pattern divided into points appears in the right pane.
- Press the **Run litho** button to run the nanolithography operation.

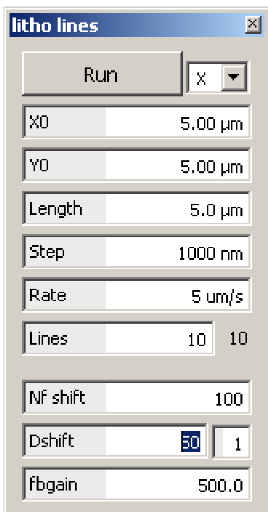
- A number of complete points and lines are shown in the control panel title bar during the nanolithography process. The **Finished.True!** string appears right after the nanolithography process is finished.
- Switch to the Scanning window to obtain a scanned image of your nanolithography pattern.

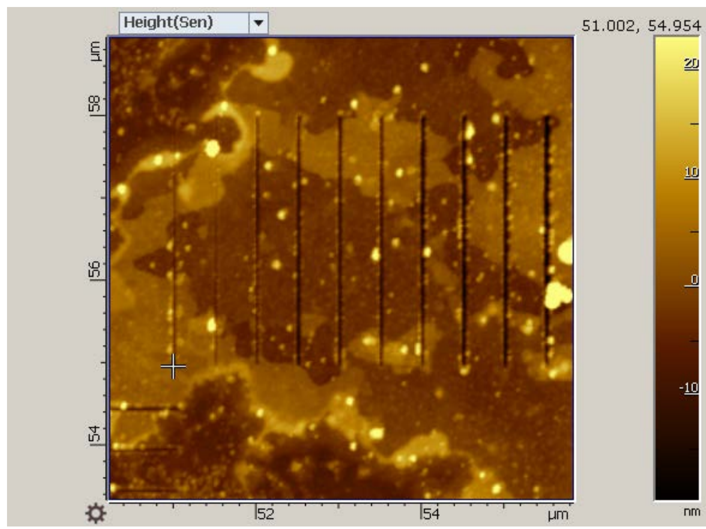
4.8.4 Macro Litho lines

This macro is intended to form a pattern on the sample in the form of a set of lines by force lithography. To conduct lithography system goes into contact method. Movement between the lines is performed in tapping method.

The macro location is AIST\MACROS\USER2

To open this macro from another folder use  button in Macro Executor window.





Parametrs:

X, Y – directions of lines;
X0, Y0 – absolute coordinates of starting point of pattern. Starting point is a left bottom corner of pattern;
Length – the length pf lines;
Step – space between lines;
Rate – speed of lithography;
Lines – number of lines;
Nf shift – shift of setpoint (clamp) during lithography;

D shift – shift of setpoint from line to line (change of clamp). Number 1 to change every line by setpoint, number 2 to change every second line etc.

fb gain – amplifier feedback gain during lithography;

Work order:

Adjust the instrument for semi-contact method.

- Scan the selected area.
- Open the macro litho lines.

-Set the initial position of the grid using parameters X0, Y0. In order to find the coordinates of the selected point, use the marker  (pre-switch to the absolute scale of coordinates using  button).

- Set the paramets of lines: the direction, length, spacing, number of lines.

- The impact is determined by the clamp - parameter Nf shift. Optimum clamping chosen experimentally. For the selection of clamping set Nf shift to 100 and make 10 lines, increasing the clamp for each 100 units by setting Dshift to 100.
- Start lithography by pressing Run button. During the lithography on the toolbar at the top of the program will change the feedback signal with Mag on Nf. At the end of lithography the Run button will overcome.
- To see the result, scan the selected field again.

4.9 “Moto Scan” mode

"**Moto Scan**" is a mode used for obtaining distribution maps of different signals across the reflective surface of the cantilever (fig. 4.9.1). Scanning is realized by the stepper motors mounted on the cantilever holder and on the AFM photodiode.

This mode can be used to estimate the quality of the reflective coating of cantilevers and for accurate positioning of the laser beam on the cantilever. Also, the "**Moto Scan**" mode is used for focus adjustment of the optical registration system of the measuring head (see Annex 8.4).

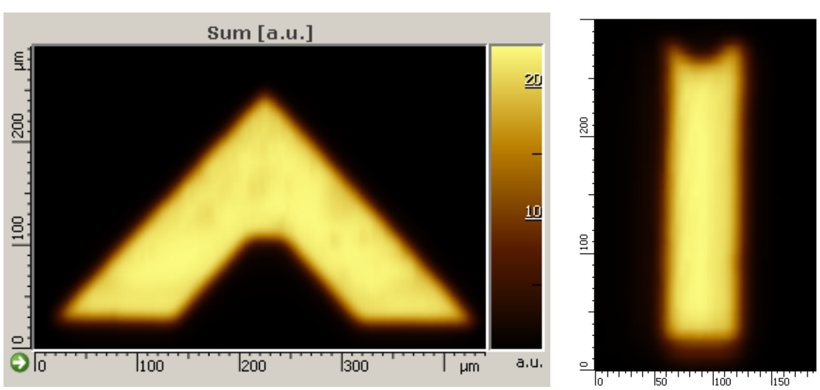


Figure 4.9.1 Photodiode signal distribution across the backside of the triangular and rectangular cantilevers obtained in the "**Moto Scan**" mode.

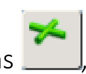
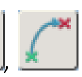
4.9.1 Working in the “Moto Scan” mode

Follow steps below to start working in the Moto Scan **mode:**

- Install your probe into the AFM measuring head.
- Remove your sample to 2 mm from the probe (in case of **CombiScope** system, remove the head to the service position).

- Adjust the optical registration system of the SPM measuring head using the **Laser Adjustment** window.
- To obtain a distribution map of probe oscillation amplitude (signal **Mag**) across the reflective surface of your cantilever, switch on the **AC mode**, adjust the working frequency to the cantilever's resonance frequency and set the oscillation amplitude to 20 - 100 nm.
- Select the **"Moto Scan"** scan mode in the **Scan** window; the control panel will appear (see **Figure 4.9.2**).
- Select **Sum, Nf, Lf, Mag** signals from the signal list.
- Set scanning area to 500x500 steps in the center of the full scanning area. (Note that in the "**Moto Scan**" mode scanning area is measured by a number of stepper motor steps).
- **X coef**, **Y coef** and **Speed** in the "**Moto Scan**" mode settings window are number of points per scanning line and scanning speed correspondingly. Use the default values at the beginning. Decrease a number of points **X coef** and **Y coef** to obtain more detailed images.
- Run the scanning process. Your results can be saved into file as standard scans.

NOTE: After finishing scanning, the probe holder is returned to its original position. If the scanning was interrupted, the holder stops at the current position and does not return to the starting position. This leads to the shift of the scanning area with respect to the cantilever. To return to the specific field, readjust the optical registration system and press **reset r.f.**

You can correct the laser position on the cantilever's reflective surface using the following buttons , ,  in the **Scan** window. Adjust the photodiode each time after changing the position of the laser beam.

4.9.2 "Moto Scan" scanning mode control elements

Click on the  button, which is located next to the list of the scan modes in the **Scan** window, to open a control panel.

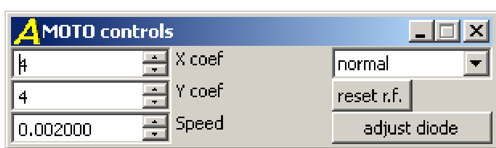


Figure 4.9.2 The **MOTO controls** panel to control the **Moto Scan** mode.

The panel contains the following elements:

X coef, **Y coef** —factors that define the number of points on the line. The number of points is determined by the formula: number of motor steps per line / coefficient = number of points.

Speed—the speed of scanning.

Scanning modes:

Normal—scanning with the probe holder;

Diode—scanning with the photodiode;

diode f.a. (full area)—scanning with the photodiode over the full area (service mode);

reset r.f. (reference frame)—setting the position of the scanning area in appliance with the position of the cantilever. Use this button after setting the position of the cantilever.

adjust diode—Adjusting the photodiode position. This button duplicates the same button in the **Laser adjustment** window.

4.10 Atomic resolution AFM imaging

The Lateral Force Microscopy (LFM) technique (Contact AFM mode) can be used for imaging atomic lattice of HOPG or mica in ambient environment. How low drift and noise level of the whole AFM system is can be checked using such imaging.

Measurement conditions

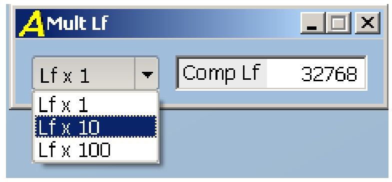
1. The SPM unit must be installed on a vibration isolation table.
2. The SPM unit must be isolated from any acoustic and electrostatic noise (i.e. special enclosure must be used).
3. There must no air flows (e.g. air conditioning systems) and noise in the room.
4. The room temperature must be stable.

Sample preparation

Stick a piece of HOPG to the sample holder using double-sided scotch tape or glue. If you use a double-sided scotch tape, stick your sample as well in advance as possible before starting your measurements, otherwise it will drift causing a certain amount of instability.
Right before starting your measurements, peel off the top layer(s) from your sample using scotch tape in order to have a smooth and clean surface.

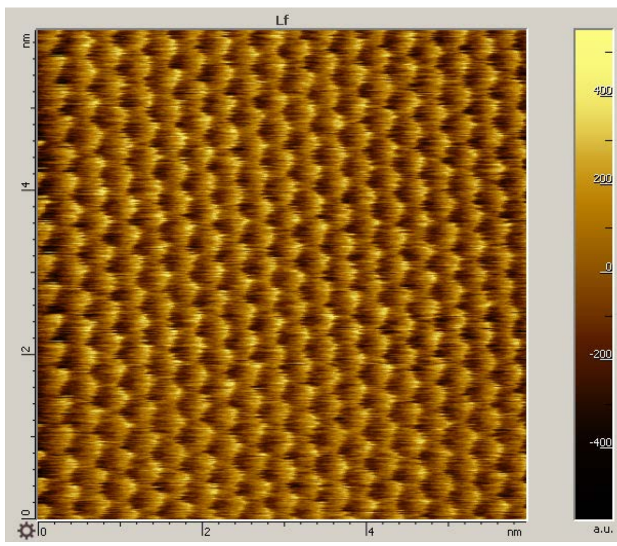
Obtaining atomic resolution LFM images

- Switch on the instrument and initialize the controller in advance (no later than 1 hour before starting your measurements) in order to allow them to warm up to their working temperature.
- Set the **high/low XY voltage** parameter to **LOW** in the **Relay** tab of the **Param tables** window to switch the instrument to the low voltage mode in XY direction:
- Use a **non-contact cantilever** of the fpN10 series or similar.
- Adjust the instrument to work in the **semi-contact AFM mode** (AC mode) using the initial amplitude of oscillation of 20-50 nm.
- Engage the probe tip with the sample surface and make 1um x 1 um test scan in the center of the full scanning area. Make sure that your HOPG surface in this area is clean and smooth, and there are no periodic parasitic noise caused by any vibration. Select scan area of 1-2 nm on the smooth part of the surface as close to the center of the full scanning area as possible. Scan this area.
- Press the **Retract** button to retract your sample from the probe.
- Switch to the DC mode (Contact AFM mode).
- Set the **shift** parameter to 500 and press the **landing** button to start the landing procedure.
- Use the **Mult-Lf** macro to set the gain of the **Lf** signal to 10 and then to 100:



To keep the **Lf** signal about 130000 units, change slightly the **Comp Lf** parameter. Select the **Lf** signal in the **Oscilloscope** window to watch how it changes.

- Select the **QScan** mode in the **Scan** window. Use the following settings:
  - Scanning mode: **dacs**;
  - Signal: **Lf**;
  - Number of points: 256x256;
  - Scanning rate: 10Hz;
- Run the scanning procedure in the cyclic mode to start obtaining images of atomic lattice of HOPG:

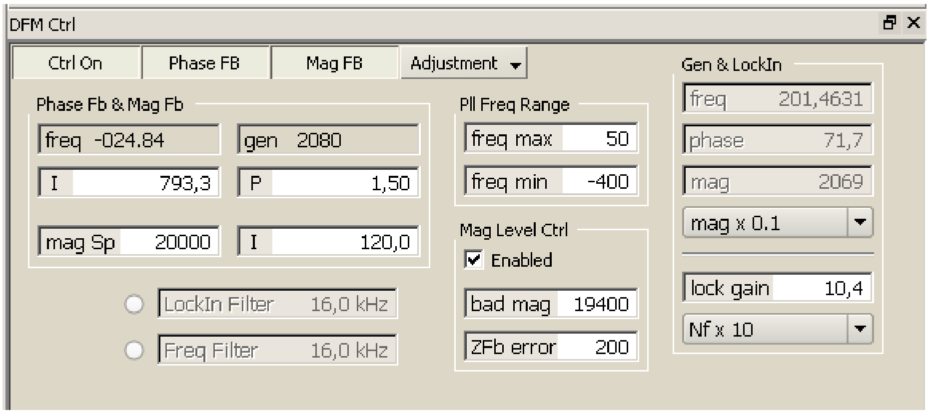


To increase the image quality:

1. Change the **Sp** and **Gain** parameters. Normally the best images are obtained when the **shift** parameter is in the range of 200-2000 and the **gain** parameter is in the range of 20-200.
2. To enable scanning at high rates, switch the **ctrl rate** digitization frequency to 100 kHz in the **Filters** window.
3. Change the **Lf** filter value depending on scanning rate.

4.11 Dynamic Force Microscopy (DFM) using cantilever as a probe

In DFM mode changes in the force gradient cause instantaneous changes in the oscillator frequency which are detected by a Frequency Modulation (FM) demodulator (i.e. Phase Locked Loop - PLL) and used as the feedback signal. Typically DFM mode is used for smooth samples which have the average roughness in the order of few nanometers. In case of samples with rough topography the height changes must not be very sharp.



To start working in DFM mode, open the **DFM Ctrl** window from the **View** tab of the main program menu:

The detailed description of the DFM Ctrl window elements can be found in the Chapter 6.6 of the "Control software reference manual".

To work in the DFM mode, do the following steps:

- Use a non-contact cantilever.
- Set the instrument for the semicontact AFM mode and set the initial oscillation amplitude to about 10 nm.
- Engage your tip with the sample surface and make a test scan in the semicontact AFM mode using the **QScan** mode.
- Press the **Retract** button on the **Auto** control panel to retract your tip from the sample surface.

Adjust DFM:

- Open the **DFM Ctrl** window.
- Press the **Init** button – phase signal will be set to the initial value (130070 a.u.)
- Switch on and adjust the phase feedback loop:
    - Set the initial values of the gains: **P** = 0, **I** = 100.
    - Press the **PhaseFB** button to switch on the phase feedback loop.
    - Select **Phase Fb Adjustment** from the **Adjustment** drop down menu to switch to the feedback loop adjustment mode: the **Wave Generator** panel will appear and the corresponding settings will be made in the **QScan** window.
    - Switch on **Wave Generator** and run the scanning procedure. Select the values of I and P parameters, so that the cross-section of the **Phase** signal gets the rectangular shape.
    - Stop the scanning procedure and **Wave Generator** after the **I** and **P** parameters have been adjusted.

- Press the **Restore QScan pars** button from the **Wave Generator** settings panel.

NOTE: Please learn more details about the feedback loop adjustment procedure in the Chapter 6 Fast Scanning, clause 6.2.

- Set **mag Sp** in the **Phase Fb& Mag Fb** groupbox to the current **mag** value, which was set during the semicontact AFM mode adjustment above.
- Switch on and adjust the oscillation amplitude feedback loop:
  - Set **I** = 30.
  - Press the **MagFb** button to switch on the oscillation amplitude feedback loop.

Now the oscillation amplitude and phase are maintained at the specified level by the corresponding feedback loops.
Land to the surface:

- Activate the **FB toolbar 2** toolbar (the list of all available toolbars can be displayed by right-clicking anywhere on the top part of the main window). Set **Freq** as the feedback signal:

- Activate the **FB toolbar 1** toolbar. Set **Sp** in the range from -100Hz to -30Hz and **Gain** in the range of few hundreds:

- Activate the **freq** signal in the oscilloscope.
- Press  to switch on the feedback loop. The Z scanner position indicator must show the scanner extension and the frequency must decrease in accordance with the setpoint.

Scanning:

- Select the **QScan** mode in the **Scan** window. Switch on the "adaptive scanning" in the scanning parameters.
- Set the scanning speed to 1 Hz, scan size to ~5 microns. You may also select **Mag**, **Freq**, **Gen Mag** as additional signals.
- Run the scanning procedure. If the instrument is properly adjusted, the actual scanning speed will appear 10-20% less than the specified one.
- To exit from the DFM mode, switch off the Z feedback loop and press the **Ctrl on** button to switch off the DFM mode.

Taking into account the above settings the instrument is supposed to be set up in the attractive mode (i.e. the closer to the sample surface the less oscillation frequency is). With the same feedback loop sign in the repulsive mode the oscillation frequency starts to increase when the tip is coming closer to the surface, which will lead to the situation when the tip hits the surface. To avoid such situation, there are the limiting parameters of amplitude and frequency levels.

Amplitude limiting parameter:

The system automatically starts to retract the tip from the surface when the oscillation amplitude decreases down to the **bad mag** value.

Oscillation frequency limiting parameter:

Frequency changes are limited by the **Freq min** and **Freq max** parameters to keep the system near the resonance frequency in order to have the phase signal changed properly.

If during the scanning procedure the oscillation frequency reaches its upper limit **Freq max** and the scanning speed dramatically decreases (when the "adaptive scanning" option is on) down to 0.1 - 0.2 Hz, it means that the system switches to the repulsive mode. Typically such transition is observed on some topography bumps due to (a) too high initial oscillation amplitude or (b) too high loading force (too high frequency shift in the setpoint).

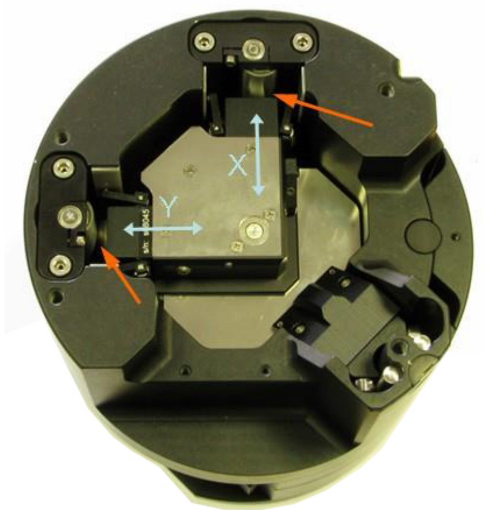
To fix this problem:

- Stop the scanning procedure;
- Switch off the feedback loop by pressing 

;
- Decrease the oscillation amplitude (**mag Sp** parameter);
- Decrease the frequency shift (**Sp** parameter).

5. SAMPLE POSITIONING

When there is a need to investigate specific surface area of the sample, the sample must be positioned relative to the probe of the microscope. For this purpose, the SmartSPM includes the motorized sample positioning system that



allows rough sample positioning in the XY plane in the range of 5x5 mm. Movement of the sample in the XY plane is provided by means of two eccentric mounted on the axes of stepper motors (**fig. 5.1**). The rotation of the motor with the eccentric on it cause the movement of the scanner due to the change of the distance from the axis of the motor and the plane of the eccentric.

Figure 5.1 The photo shows the lower part of the SmartSPM microscope without the measuring head. Orange arrows show the location of the eccentrics of the motorized sample positioning. Blue arrows show the direction of the travel.

To move the sample in the XY plane, open the **"XYZ Motors"** window, by selecting the **XYZ Motors** procedure from a list of procedures in the main menu or by clicking on the  icon in the list of procedures on the left side of the program window. The window is shown in Figure 5.2.

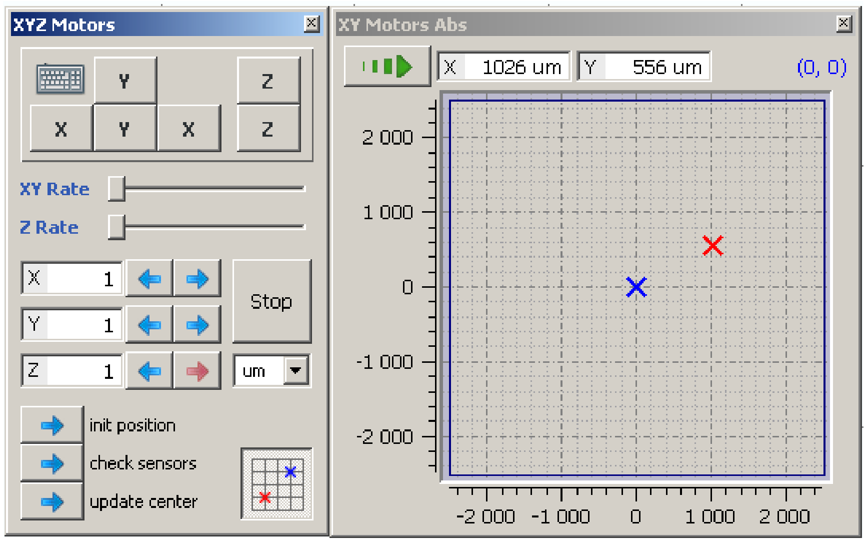
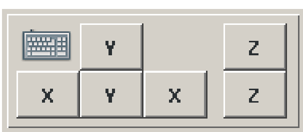


Figure 5.2 The **XY motors** window with the opened coordinate field

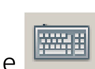
WARNING! Remove the sample from the probe to the distance of at least 100 microns before you start positioning of the sample to prevent damage to the probe or sample.

5.1 The continuous movement of the sample

For instantaneous movement of the sample, use the following group of buttons:



To move the sample, point the mouse cursor on the one of these buttons, left-click and hold on it. To stop the sample motion, release the left mouse button. Use a video microscope to control movements of the sample.

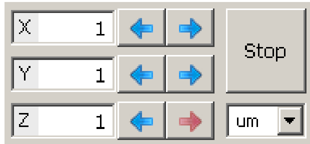
The program has an option to move the sample using keyboard that can be activated by clicking on the  button. X and Y buttons correspond to the arrows on the keyboard, the Z buttons correspond the "+" and "Enter", located on the right side of the keyboard.

XY Rate and **Z Rate** sliders set the speed of the sample motions. Left-most position of the sliders corresponds to the minimum speed and right-most – to the maximum speed.

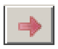
NOTE: Since the movement of the sample is provided by the rotation of the eccentrics, the direction of the movement of the sample is reversed for any of the axes when the movement is out of the range.

5.2 Moving the sample at a specified distance

To move the sample at specified distance, use the following elements:



To move the sample in x, y or z direction, enter the number in the corresponding X, Y or Z text box and press the button with the arrow.

For movements along the Z-axis blue arrow  indicates the movement of the sample from the probe, the red arrow  - movement to the probe.

The displacement can be set both in microns and steps of the positioning system motors. To switch the unit use the drop down menu 

.

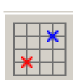
One step of the Z-motor corresponds to 100 nm. For XY-motors, the displacement that corresponds to one step varies depends on the position of the positioning system (the maximum step is 0.5 microns).

To stop motion of the positioning system, click on the 

button.

NOTE: Clicking on any of the displacement keys leads to halt an operation, and starts a new one. Therefore, before starting a new displacement of the sample using the positioning system, wait until the completion of the previous motion.

5.3 Moving to a defined point

To move the positioning system to the point with defined coordinates, opens the **XY motors Abs** window with coordinate field .

Coordinate field displays the total field of the positioning system. Blue cross indicates the current position of the positioning system. To set the point of destination, click on it at the coordinate field, or enter it coordinates in the text boxes at the top of the window. A red cross appeared after clicking shows the point of destination. To move to it, click on the green arrow .

5.4 Setting the positioning system to the central position

An initial position of the positioning system is its central position.

To move the positioning system to the central position, use the 

button.

ATTENTION! The **init position** command moves the positioning system through the entire XY range. Therefore, before proceeding it, make sure that the probe and/or the sample will not be damaged during the motion of the positioning system.

NOTE: The coordinates of the central position of the positioning system defines the accuracy of the conversion from steps to microns. The central position is specified in the file of the scanner parameters. By default, this file contains coordinates of the central position typical for this type of the instrument. To accurately determine the position of the center for a particular instrument follow the procedure described in Appendix 8.3.

6. FAST SCANNING

The "Fast scanning" mode is a mode when the scanning rate is higher than 5 Hz.

You can scan your sample at high rates either in Contact or Semicontact AFM mode. In Semicontact AFM mode you can work either in the field of attraction (attraction mode) or repulsion forces (repulsion mode). However, you can usually achieve much higher scanning rates working in the repulsion mode. To work in this mode, use cantilevers with some high resonance frequency (for example, fpN10 or fpN20 series). The initial amplitude of oscillations must be not less than 30-50 nm. The setpoint is about 0.5 - 0.7 from the initial amplitude. To check if you work in the repulsion mode, you should watch the phase shift during the landing procedure when the tip touches the sample surface. If the phase drops down, then you may conclude that you work in the repulsion mode.

Select the **QScan** mode in the **Scan** window to work in the Fast Scanning mode. Before running your instrument in the Fast Scanning mode, first adjust it in accordance with the Clause 6.1 and then adjust the Z feedback loop in accordance with the Clause 6.2.

To determine the maximum possible scanning rate, you should consider the quality of the image being obtained. The maximum scanning rate depends on your sample roughness, scanning area and cantilever's parameters.

The main features showing that the maximum scanning rate has been reached are the following:

1. Some parasitic oscillations appear on the image which you haven't seen at lower scanning rates. Such oscillations can be seen more clearly on the feedback error (Mag, Nf) or phase signals.

2. Feedback error signal becomes saturated when some sharp bumps are scanned.

6.1 Fast scanning setup

Set the following parameters in the **Filters** window:

- Switch off the **LockIn2** filter if you work in the Semicontact AFM mode (**Mag** - feedback signal) or the **Nf** filter if you work in the Contact AFM mode (**Nf** - feedback signal).
- Switch off the **SenZ** filter.
- The **SenX** and **SenY** scanner sensors filters. Increase the corresponding bandwidth for the fast scanning direction. For the **SmartSPM** it means that you should switch off this filter. For the **CombiScope** that you should increase the bandwidth several times.
- Switch **ctrl rate** to 100 kHz (digitalization rate).
- Load the second AFM signals table **(afm sigs table 2** in the **Filters** window) if you work in the Contact AFM mode.

After that adjust the feedback using the Square wave signal in accordance with the clause 6.2.
Select the **QScan** mode in the **Scan** window and set the following system parameters:

- Use the adaptive scanning mode (set the **adaptive** parameter to on).
- Use the **Back Jump** scanning mode. In this mode the actual scanning speed is less than in the standard mode at the same rate due to the fast scanner jump to a new scanning line. Use the following **Back Jump** parameters: **kVelX** = 4, **velZ** = 10.

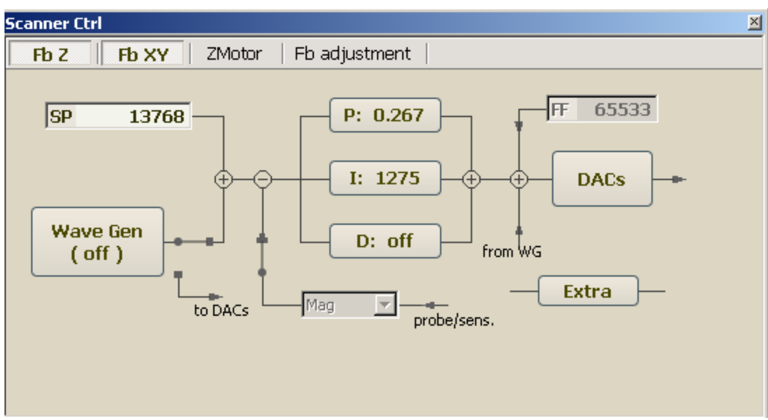
NOTE: Increase the feedback gain 2-3 times in the fast scanning direction when you use the **Back Jump** and **adaptive** scanning modes.

6.2 Manual feedback loop adjustment

To make the Z feedback work correctly, it's necessary to set the right integral and proportional feedback gain coefficients. One of the standard methods of feedback adjustment is a transition function test. To use this method, a square wave signal must be applied to the feedback input and a behavior of the control signal is investigated depending on the feedback gain coefficients.

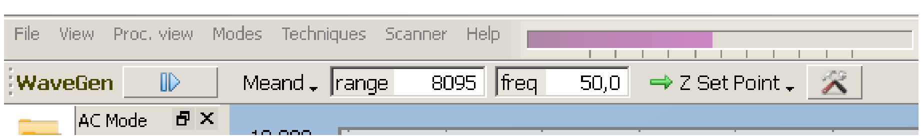
Do the following steps to manually adjust the feedback loop:

- Open the **Scanner Ctrl** window right after the approach and landing procedures are finished:



- Press the **FB adjustment** button in this window.

The **WaveGen** toolbar appears:



At the same time the **QScan** window will be displayed where the following parameters will be set accordingly to the feedback loop adjustment:

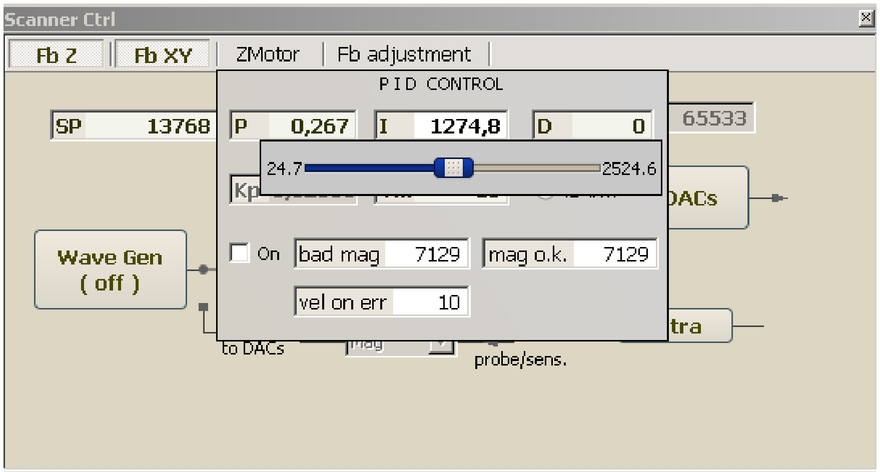
Measuring signal– **Mag**;

Move XY – switched off;

Scanning rate – 8.3 Hz;

The repeated scanning mode is on.

- Open the **PID control** from the **Scanner Ctrl** window by clicking on either **P**, **I** or **D**:

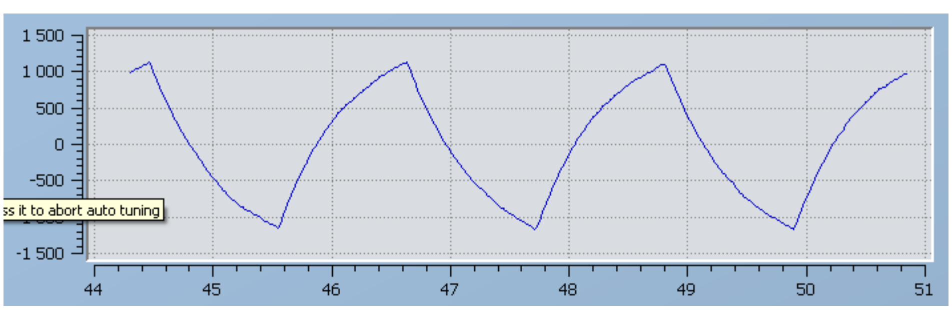


- Set **P** to zero.
- Run the square wave signal generator by pressing  on the **WaveGen** toolbar.

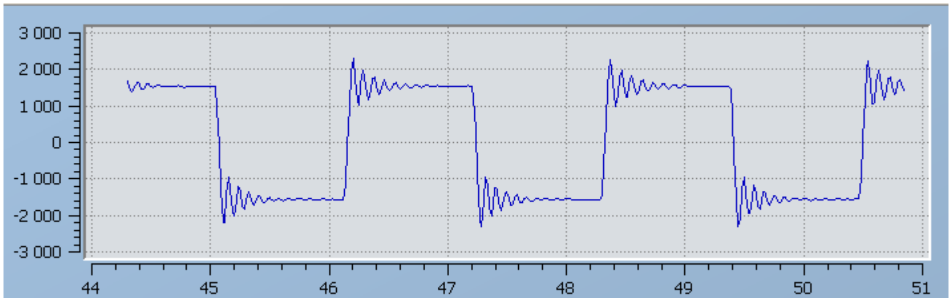
NOTE: The **WaveGen** toolbar has some default parameters. However, it is possible that the signal amplitude applied to the feedback input should be changed (**range** parameter). The default value of the **range** parameter can be too small, so it will be difficult to distinguish it from noise, or too big, so the probe will either retract from the surface or will press down too hard. The **range** parameter is measured in the same units as the **setpoint** for all input signals except **Freq** and **Iprobe**. For **Freq** 1Hz corresponds to 100 units of the **range**.

- Run the scanning process.
- Watch the signal form in its profile window:

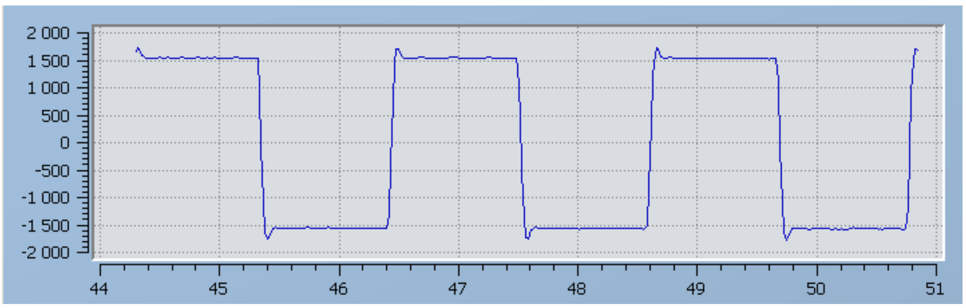
If the integral coefficient I is not enough, the signal form will be close to the triangle one shown below:



If the I coefficient is too big, there will be some oscillations after each square:

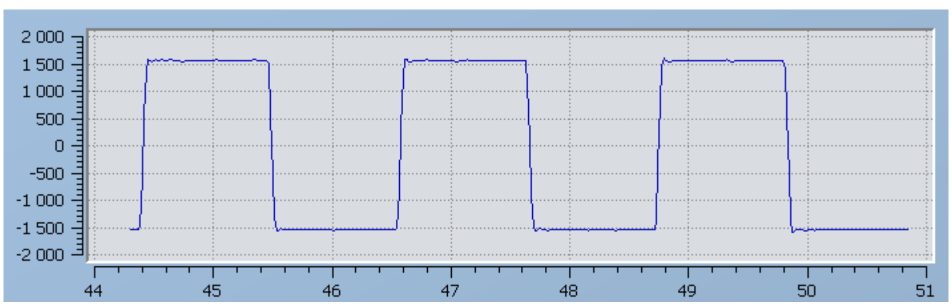


NOTE: The integral coefficient **I** of the feedback loop found during the **Adjust Fb Gain** automatic procedure usually appears a bit high.



Try to make the signal form as shown below by changing **I**:

After that increase **P** to make the form as square as possible:

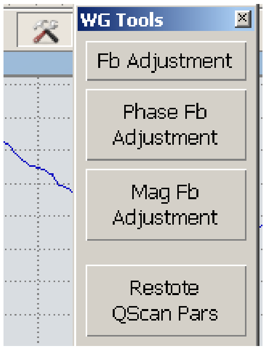


NOTE: Usually **P** is several hundred times less than **I**.

To start scanning after the successful adjustment described above, do the following:

- Switch off the square wave generator.
- Stop the scanning process.
- Restore the initial scanning parameters. To do so, open the **WG tools** panel by pressing  on the

WaveGen panel:



- Press the **Restore QScan Pars** button.

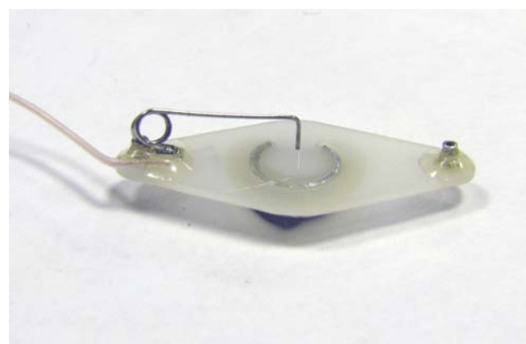
7. OPERATION WITH ADDITIONAL EQUIPMENT

7.1 Conductive AFM

7.1.1 Conductive unit

The conductive unit is a low noise I-V converter which is used to measure local current in AFM or STM mode. For using the conductive unit, use the sample holder with the spring clip and a 10M resistor (**fig. 7.1.1**).

Current unit



The sample holder with spring for electrical contact to the sample.

Figure 7.1.1 A set of hardware required for Conductive AFM

7.1.2 Installing the conductive unit

To install the module, follow below steps:

- Switch off the controller;
- Secure the conductive unit to the post and then, fix the post on an optical table with a clamp, as shown in **figure 7.1.2**.
- Plug the conductive unit cable into the "**Head**" connector on the controller (**position 1 in figure 7.1.3**).
- Connect the measuring head cable to the connector of the conductive unit cable (**position 2 in figure 7.1.3**).

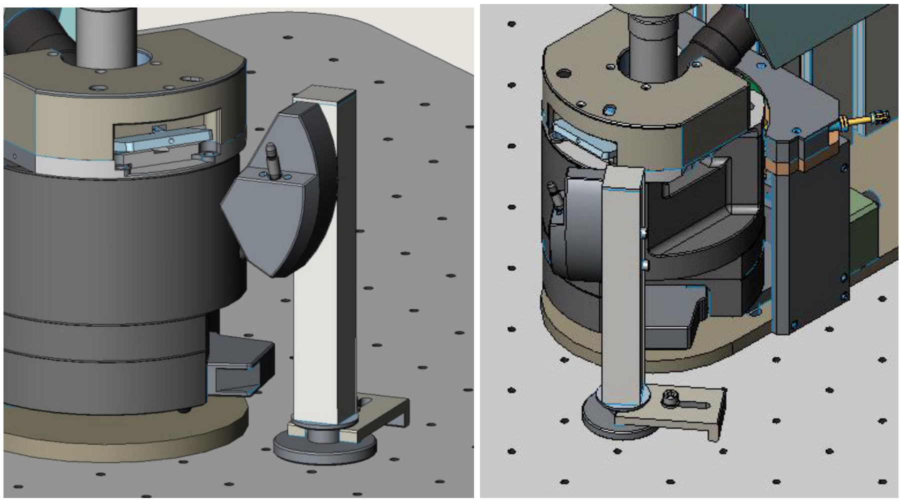


Figure 7.1.2 Installation of the conductive unit on the optical table.

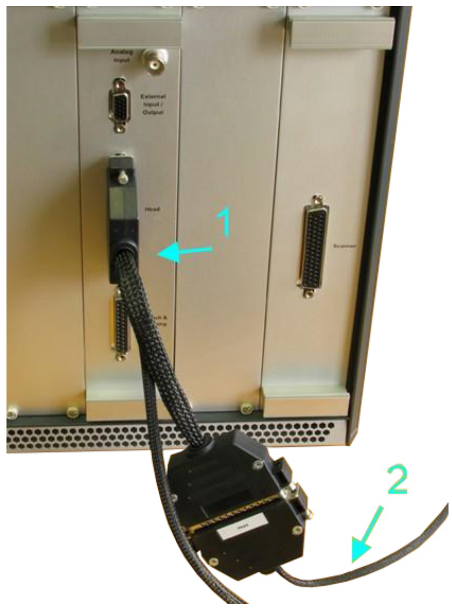


Figure 7.1.3 Cable connection of the conductive unit to the controller.

7.1.3 Controlling the conductive unit

To accomplish measurements with the conductive unit, use its control window (**Fig. 7.1.7**). To open the panel, select **I-V Switch** in the list of **Techniques** of the main menu.

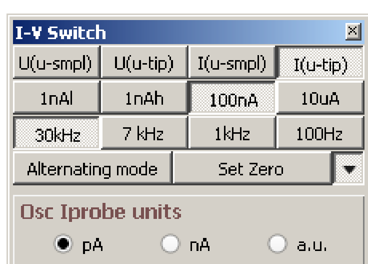


Figure 7.1.7 Conductive unit control panel

The buttons you can find on the control panel have the following functions:

U(u-smpl), U(u-tip) – applying voltage without measurements of the current.

I (u-smpl), I (u-tip) – applying voltage with measurements of the current.

Parameters **u-smpl** и **u-tip** set a scheme of applying bias voltage:

u-smpl – bias voltage is applied to the sample, the probe is grounded

u-tip – bias voltage is applied to the probe, the sample is grounded

Figure 7.1.8 shows schematic drawings of applying bias voltages (BV) at using of **U-tip** or **U-smpl** functions.

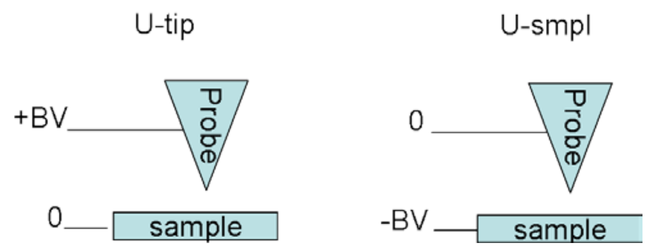


Figure 7.1.8 The schemes of applying bias voltages in **U-tip** and **U-smpl** modes.

The **30kHz**, **10kHz**, **1kHz**, and **100Hz** buttons are to filter the current on the output of the conductive unit.

The **1nAl, 1nAh, 100nA, 10uA** buttons are to switch over current ranges:

1nAl – low noise mode with 100 Hz bandwidth for measuring current up to 1 nA

1nAh – for measuring current up to 1 nA

100nA – for measuring current up to 100nA

10uA – for measuring current up to 10µA

Alternating mode – applies the AC driving voltage to the piezoelectric element of the probe holder. This function can be used for measuring current in the semicontact mode.

Set Zero – sets the zero level of the current signal. Before clicking on this button, retract the probe from the sample.

 - To select units for displaying the **Iprobe** signal on an oscilloscope. It can be displayed in pA, nA or in a.u. (ADC units).

7.1.4 Contact conductive AFM

Conductive Atomic Force Microscopy (CAFM) characterizes local conductivity variations across the sample surface. A conductive probe tip is required for CAFM. A DC bias voltage is applied between the probe and the sample. During the scanning process, the current passing through the probe/sample contact area is measured in each point. The resulting image is a local conductivity distribution map. To ensure a good contact between the surface and the probe the contact AFM mode is used.

To perform CAFM measurements, do the following:

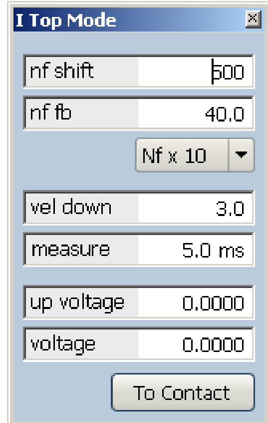
- Install the conductive unit following the instructions in the clause 7.1.2.
- For current measurements, use sample holder with spring providing an electrical contact. Fix your sample on the holder, so there is no electrical contact with its metal part. Insert the spring into the pin on the holder and press it to the surface of the sample (the surface has to be conductive). Connect another end of the wire to the current unit.
- Use a contact AFM cantilever tip with some conductive coating. Install the probe into the probe holder.
- Adjust the instrument to work in the contact AFM mode.
- Set the zero voltage applied to the probe before starting the approach procedure. You can adjust applied voltage using the **Apply Voltage** input field on the Contact AFM mode control panel.
- Select the **Iprobe** signal in the **Oscilloscope** window.
- Open the current unit control panel by selecting **I-V Switch** in the **Techniques** submenu of the main program menu.
- Press the **I(u-smpl)** button to apply the DC bias voltage to the sample and enable the current measurement. Select the **100nA** mode and the **30kHz** filter.
- Engage your probe with the sample surface. Watch the **Iprobe** signal in the oscilloscope as it must slightly change (depending on how conductive your sample is) when the tip just contacts the surface.
- Use the **Qscan** or **scan fb** modes for scanning of the surface.
- In the **Scan** window, check a box adjacent to **Iprobe** in the list of the available for recording signals.
- Run the scanning procedure. Monitor the current image as well as the **Iprobe** signal in the oscilloscope.
- Change setpoint **SP** (in other words the force that applied to the surface by the probe in the point of the contact) and the bias voltage using the **Apply Voltage** button on the Contact AFM control panel to optimize the current image quality during the scanning process.

ATTENTION! When using the probes with the conductive coating, make sure that the current value does not exceed 50nA by adjusting the bias voltage correspondingly. High currents can destroy your probe coating.

Use the **BV Sweep** mode in the **Curves View** window to obtain volt-ampere curves.

7.1.5 Point-by-point conductive AFM (I-top mode)

I-top mode is a scanning mode during which a spatial map of the electrical conductivity of a surface is measured. The unique feature of this mode is that the local current is measured in the contact AFM mode, but the movement between two scanning points is done in the semicontact AFM mode. This mode is extremely useful for those samples which cannot be scanned in the contact mode (e.g. nanotubes, nanoparticles).



The **I-top mode** control panel can be opened from the **Scan** window:

The control panel contains the following controls:

Nf shift –	working setpoint in contact mode.
Nf fb –	feedback loop gain value in contact mode.
Nfx10 –	10 times amplification of the **Nf** signal before digitization.
Vel down –	speed of engagement of the probe with the sample surface during the process of transition from semicontact to contact mode.
Measure –	time of current measurements at the point.
Up voltage –	sets the bias voltage during the process of movement between two scanning points.
Voltage –	sets the bias voltage during the process of local current measurement;
To contact –	switches the instrument to contact mode using the current parameters in the control panel.

Do the following steps to obtain an image in I-top mode**:**

- Install the conductive unit following the instructions in the clause 7.1.2.
- Use the sample holder with the spring. Fix your sample on the holder, so there is no electrical contact with its metal part. Insert the spring into the pin on the holder and press it to the surface of the sample to provide an electrical contact. Connect another end of the wire to the current unit.
- Select a non-contact cantilever with some conductive coating.
- Install the cantilever into the cantilever holder and then install it in the AFM head. Plug the cantilever holder connector to the conductive unit.
- Select **I-V Switch** from the **Techniques** menu. Press **I(u-smpl)** to apply the bias voltage to the sample. Select the **100nA** current range and **30 kHz** filter. Switch on **Alternating mode.**
- Select the **Iprobe** signal in the oscilloscope.
- Adjust the instrument to work in the semicontact AFM mode.
- Set the bias voltage to zero before approaching to the sample surface (use the **BV** input field and **apply voltage** button on the **AC mode** control panel).

- Execute the **Approach** and **Landing** procedure. Adjust Fb gain.
- Switch on the **I-top Mode** in the **Scan** window.
- Switch on the **Iprobe** signal in the signal list in the **Scan** window.
- Run the scanning procedure. Watch the **Iprobe** image in the **Scan** window and the **Iprobe** signal in the oscilloscope.
- Vary the **Nf shift** (pressing force) and **voltage** (bias voltage) parameter to improve image quality during the scanning process. Depending on your cantilever's stiffness and conductive coating the **Nf shift** parameter may vary from 50 to 2000 a.u. Change the **voltage** parameter, so the maximum current does not exceed 100 nA.

Figure 7.1.9 shows an image of the HOPG surface as an example of the method performance.

NOTE: The **I-top mode** is a quite slow mode due to the fact that there is a transition between contact and semicontact modes in each scanning point. Time per point is determined by the speed of movement in vertical direction **vel down** and measurement time at the point. The **rate** parameter in the **Scan** window determines only the speed of movement between scanning points.

NOTE: There may be some additional noise on the topography image due to the transition between contact and semicontact mode.

To obtain an I-V curve, press the **To contact** button to switch the instrument to contact mode**.** To record curves, select **BV Sweep** in the **Curves View** window.

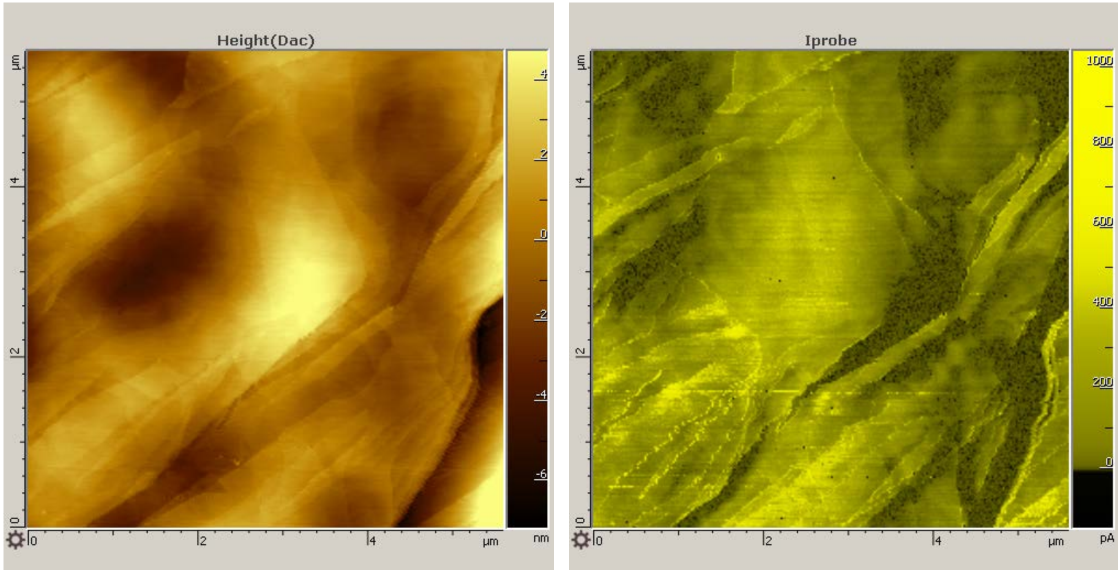
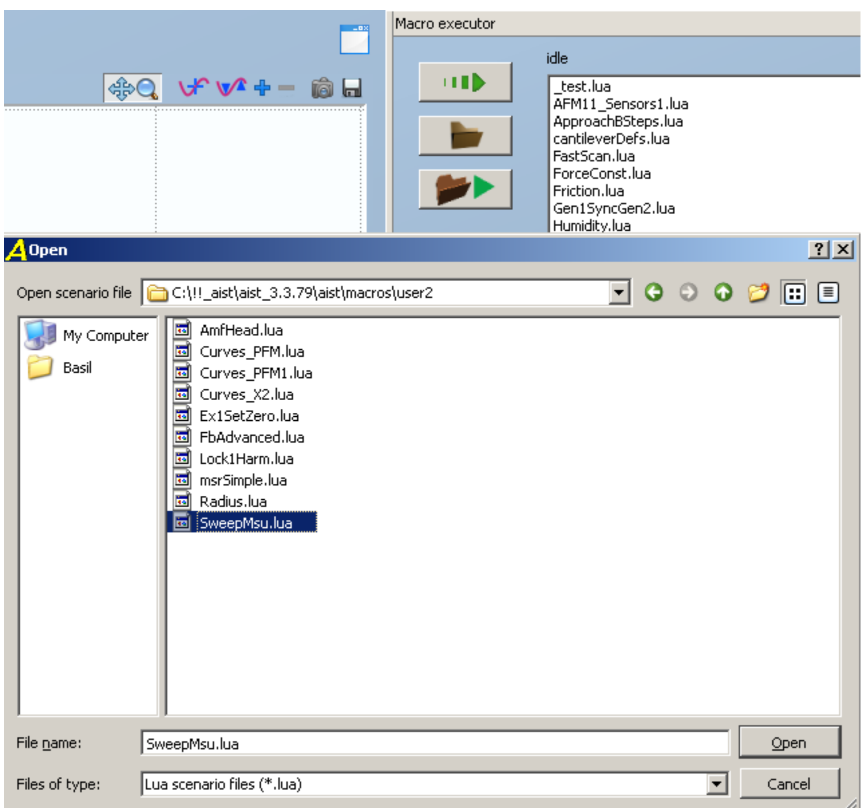


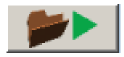
Figure 7.1.9 Topography (left) and spatial map of the electrical conductivity (right) of HOPG. The topography image was obtained beforehand in the **Qscan** semicontact AFM mode. The electrical conductivity map was obtained in the **I-top mode**. Conditions: fpN10Pt cantilever, Nf shift = 1000, voltage = 5 mV.

7.1.6 Getting series of volt-ampere characteristics

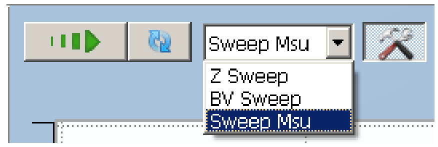
To remove the volt-ampere characteristics (VAC) on the grid, or with sweep by setpoint use the macro SweepMSU.

The macro location is AIST\MACROS\USER2:

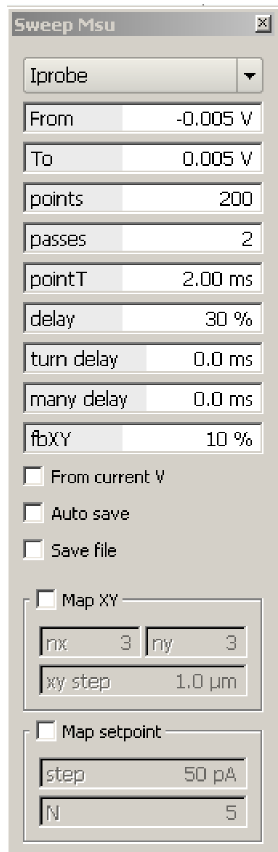


To open this macro from another folder use  button in Macroexecutor window.

After opening the macro you can find it from the list in Curves window.



Macros Control Panel:



Elements of the top of this control panel are similar to the elements of BVSweep control panel.

Map XY –obtain scan of curves by grid. The grid build around the current position of scaner, i.e. the current position of scanner is located in the center of current area.

Map setpoint – obtain scan of curves with changes of setpoint. The step value will be added to the current value of setpoint.

Save file –curves will be automatically saved in txt-file to this folder AIST\DATA\MSU numbered in order of receipt.

7.2 Scanning Tunneling Microscopy (STM)

The following components are required to work in STM mode: STM tipholder platform, conductive unit, STM tipholder and sample holder with spring clip (**fig. 7.2.1**):

STM tipholder platform

Conductive unit

STM tipholder

Sample holder with spring clip

Figure 7.2.1 Set of STM components

7.2.1 System preparation

The equipment installation procedure

Do the following step to set up the system:

- Switch off the controller;
- Unscrew the corresponding screws to remove the AFM measuring head and its mounting platform from the instrument;
- Install the STM tipholder platform on the instrument;
- Screw two special screws into the platform;
- Install the conductive unit onto these two special screws and fix it using the fixing screws;
- Plug the conductive unit cable into the "**Head**" connector on the controller.

Sample preparation

The STM mode is normally used only for conductive samples. As a test sample for STM a piece of highly oriented pyrolytic graphite (HOPG) is recommended to be used. Before any measurements, peel off the top layer from the

HOPG piece using a scotch tape in order to have a clean crystalline surface. To do so, stick a piece of scotch tape to the HOPG surface and smooth uniformly across the whole surface. Then peel off the scotch tape along with the graphite top layer. You may need to repeat this procedure a few times with some new pieces of scotch tape until you get a uniform layer peeled off.

Once your sample is ready, place it on some **non-conductive** double-sided scotch taped stuck to the sample holder and make the electrical contact using the spring clip (fig. 7.2.2). Place the sample holder onto the microscope, so the spring clip does not touch the STM tipholder. Connect the spring clip wire to the conductive unit.

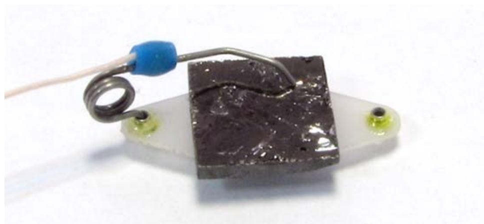


Figure 7.2.2 A piece of HOPG installed on the sample holder with spring clip

STM probe tip preparation

The STM probe is a sharp metal tip (in the best case, atomically sharp) usually made from either a platinum iridium (PtIr) alloy or tungsten wire. Wire probes for the SmartSPM STM must be less than 0.25 mm in diameter to fit into the tipholder.

The STM probe can be mechanically formed from a piece of wire using sharp scissors. To make a new STM probe, you need to have a sharp scissors and a strong grip wide-lever tweezers, so you can reliably hold the wire in your hands. It is more convenient if you grip the wire right from the wire hank without cutting it, so a few millimeters of it will stick out from the tweezers levers. Cut the wire end slightly at a very sharp angle and at the same time pull the scissors forward along the wire axis. Pulling is necessary in order not to touch the obtaining probe tip and to cut the wire. Once the probe tip is ready, cut it from the wire hank. The probe length must be about 7-8 mm.

STM probe installation

There is a special spring hook in the STM tipholder to fix your STM probe (**fig. 7.2.3**). Using thin tweezers hold the probe wire somewhere in the middle, then open the spring hook and place the probe wire in the STM tipholder groove as shown in the figure below. The sharp end of the tip must stick out from the holder no more than to 1 mm.

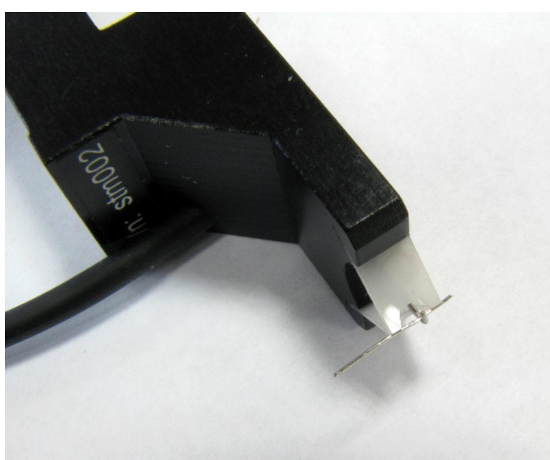


Figure 7.2.3 STM probe installation

NOTE: When working with 100x objective the probe end protruding from the top of the tube holder must be cut off or bent to 90 degrees.

Install the STM tipholder under the fixing screw on the platform and turn it anti-clockwise, so it sets against the special pins. Fix the tipholder by turning the fixing screw clockwise (**fig.7.2.4**). Plug the STM tipholder cable into the conductive unit.

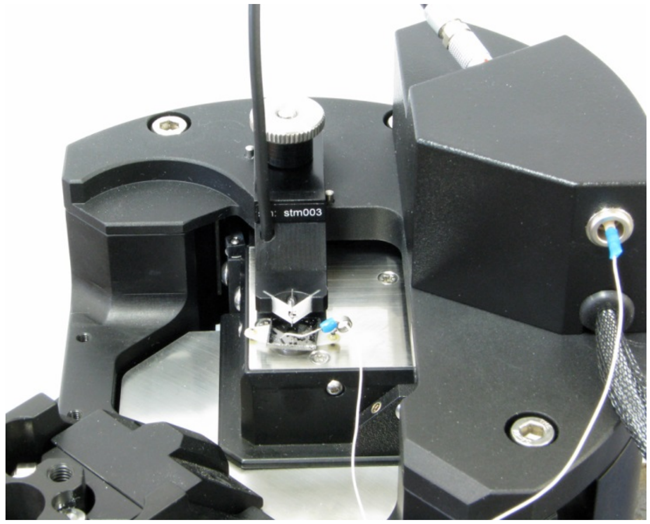


Figure 7.2.4 STM tipholder in its working position

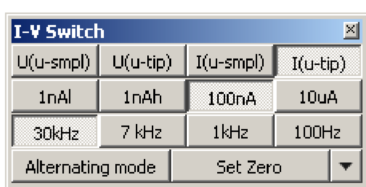
7.2.2 Measurements in the STM mode

ATTENTION! Before starting measurements, check the following:

1. Spring clip touches your sample;
2. The spring clip wire is connected to the conductive unit;
3. The STM tipholder is connected to the conductive unit.

Do the following:

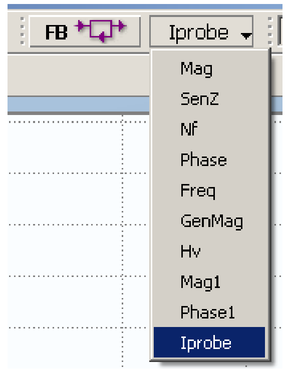
1. Open the **Oscilloscope** window and select the **Iprobe** signal.
2. Select **I-V Switch** from the **Techniques** menu to open the conductive unit control panel. Select the **I(u-smpl)** or **I(u-tip)** mode, **100 nA** range and **30kHz** bandwidth:



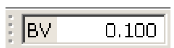
NOTE: While switching between the conductive unit modes the **Iprobe** signal must abruptly change. It means that the conductive unit is properly connected and functional.

3. Set the zero current by pressing **Set Zero**.

4. Open the **Fb ToolBar2** control panel. To open the list of control panels, right click anywhere on the empty gray background of the top part of the main program window.
   Select **Iprobe** as the feedback signal:

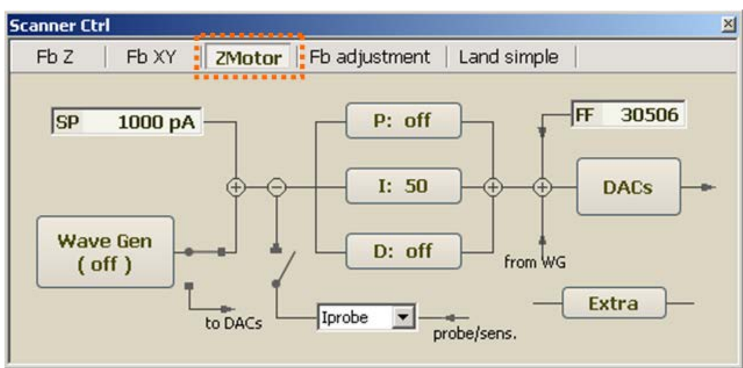


5. Open the **Bias V** control panel. Set bias voltage to 0.1V:

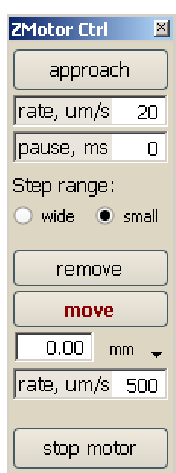


6. Open the **Fb ToolBar1** control panel and set setpoint **Sp** = 1000 pA, feedback gain **Gain** = 200:

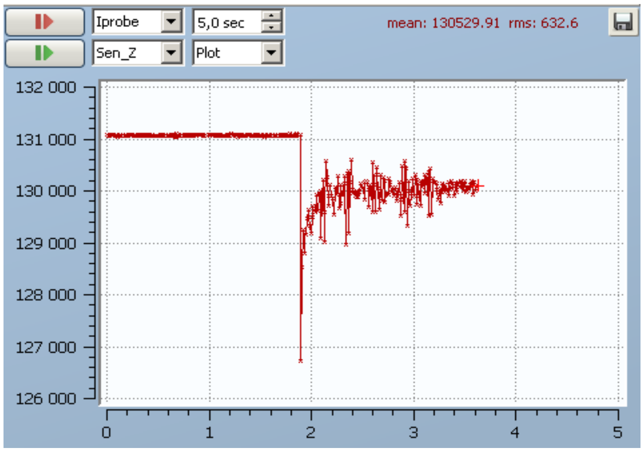
7. Press the **Z motor** button in the **Scanner Ctrl** window to open the **Z motor Ctrl** control panel:



8. Move the sample towards the STM probe tip, so the distance between them is about 0.5-1 mm, using the **move** button and the input field below:



9. Set the **rate** parameter (approach rate) to 10-20 microns per second.
10. Press the **Approach** button to run the approach procedure. During this procedure the scanner position bar indicator must show that the scanner position fluctuates in the middle of its range. Upon reaching the surface the stepper motor stops and the scanner slightly removes from the sample surface.
11. Use the **Land simple** button, at the top of the **Scanner Ctrl** window to land on the surface.
12. Watch the **Iprobe** signal in the Oscilloscope window. The signal must suddenly change upon touching the surface in accordance with the entered setpoint:



13. Set the appropriate feedback gain parameter. Typical values for the above settings is about 200-500 units.
14. Select the **scan fb mode** or **QScan** mode in the **Scan** window and set the following parameters:
    - Scan area: 1-3 um in the center of the whole scanning range
    - Signals: **Height(Dac)**, **Iprobe**
    - Number of points: 256x256
    - Scanning speed: 1 Hz

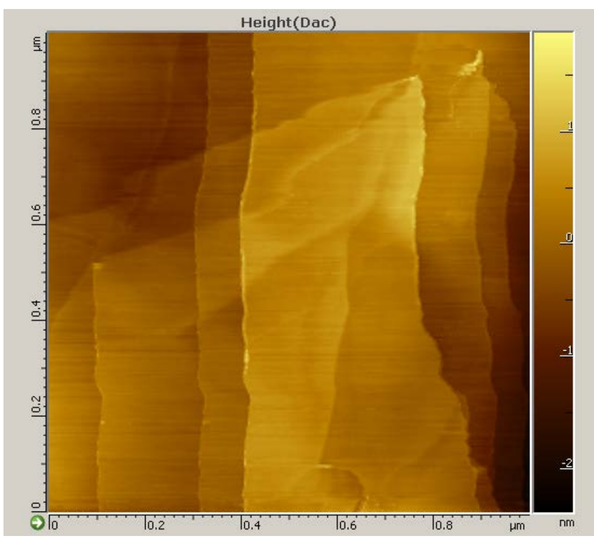


Figure 7.2.5 Typical image of graphite surface in STM mode.

7.2.3 Obtaining atomic resolution images in STM mode

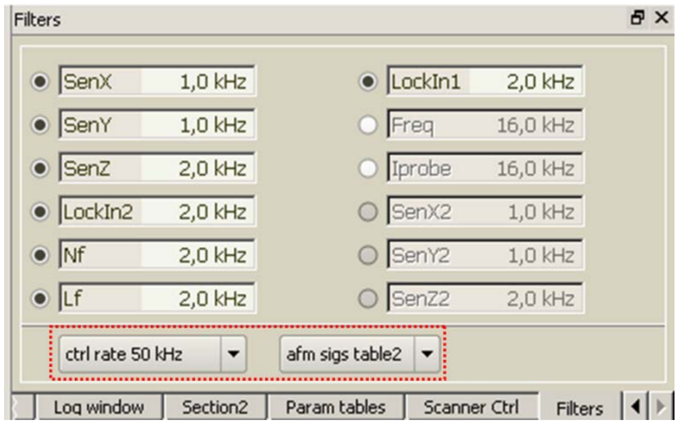
Use a piece of highly oriented pyrolytic graphite (HOPG) to obtain the atomic resolution imaging of its crystalline structure. How to prepare the sample and probe is described above.

Measurement environment requirements

- The SPM unit must be installed on a vibration isolation table.
- There must no air flows (e.g. air conditioning systems) and noise in the room.
- The room temperature must be stable.

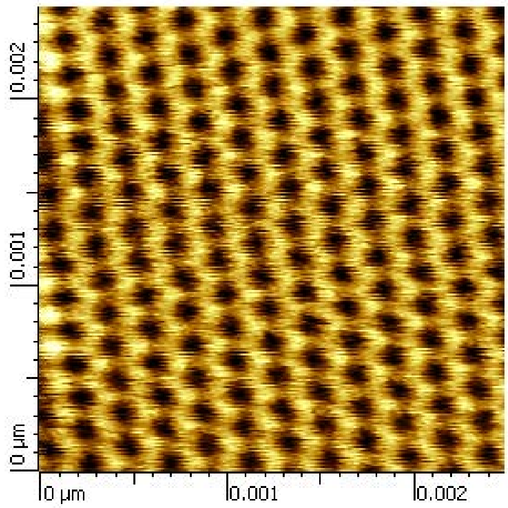
Measurement procedure

1. Make a test scan by doing the steps 1-14 above.
2. Select the **ctrl rate 50 kHz** digitalization rate and the **afm sigs table 2** signals table in the **Filters** window:



3. Decrease the **Gain** feedback gain parameter to 100.
4. In the **QScan** mode set the following parameters:
   - Scan area: 1-2 nm in the center of the whole scanning range;
   - Scanning mode: **dacs**;
   - Signal: **Iprobe**;
   - Number of points: 256x256;
   - Scanning speed: 10 Hz;
   - Cyclic scan.

Run the scanning process. Right after you run the scanning process, most likely some drift will be observed. That's why we recommend to use the cyclic scan. After some time the system will stabilize and it will be possible to obtain images similar to the following one:



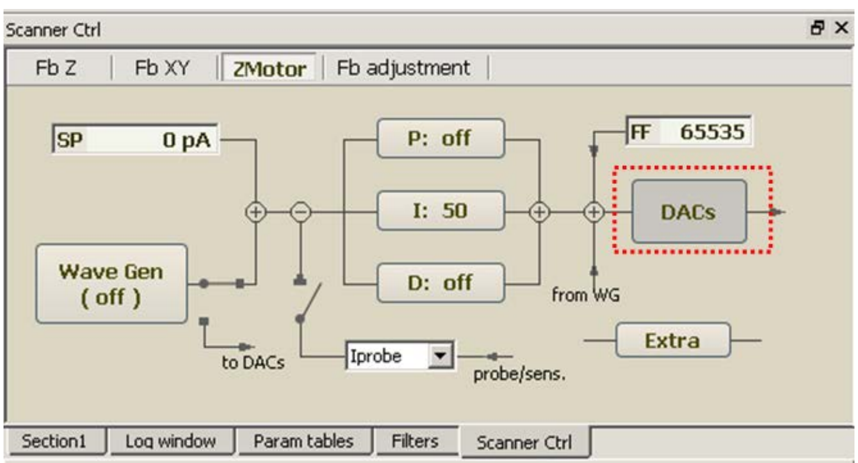
5. Change the tunneling current **Sp** in the range from -500 to -10000 pA and feedback loop gain **Gain** to achieve the optimal image contrast.

To obtain an image of atomic lattice in the height mode as well as to obtain an image of the surface with a very small height difference:

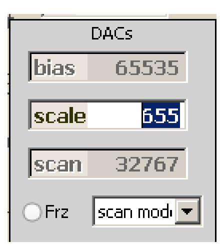
1. Select Height(DAC) in the signals list;
2. Do the following to decrease 10 times the **scale** parameter:

   Press the **Fb** button to switch off the feedback loop.

   Press the **DACs** button in the **Scanner Ctrl** window:



The **DACs** dialog box appears. Set **Scale** to 655:



7.4 Shear Force Microscopy (ShFM) using a tuning fork

Measurements are carried out in the dynamic force microscopy where an oscillation frequency of the tuning-fork is used as a feedback signal.

You need the following additional components to work in the ShFM mode:

1. Probe holder plate **h7** (for SmartSPM) or **h8** (for CombiScope),
2. Probe holder **shf**.

Figure 7.4.1 SmartSPM setup for measurements with quartz tuning-fork

7.4.1 Probe installation

Install your probe into the holder as shown in the **figure 7.4.2**. Make sure that the bottom part of the mounting plate of the probe matches the bottom part of the probe holder. The spring contacts of the holder must be securely clamped to the contact plates of the probe. To fix the probe in the holder, tighten the corresponding screws.

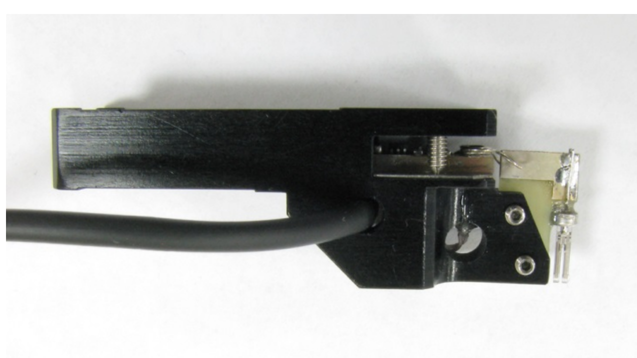


Figure 7.4.2 The holder with a quartz tuning-fork probe

Install your sample on the microscope's scanner.
Install the probe holder with the probe on the probe holder plate and rotate the holder anti-clockwise, so it rests against the corresponding poles. Fix the probe holder by tightening the screw on the probe holder plate.

7.4.2 Initial settings

Open the **Macro Executor**, **Scanner Ctrl** and **DFM Ctrl** windows.

Run the **tForkDefs.lua** macro from the **Macro executor** window**.** This macro will set the following parameters to be used in the ShFM mode using a tuning fork:

In the Resonance window:

Coarse range of frequency sweep: 20-36 kHz.

Fine range of frequency sweep: 1 kHz.

Frequency sweep parameters (you may press  to open the corresponding window):

Number of points **points** = 250;

Delay time per point **delay** = 30%;

Measurement time per point **pointT** = 2 ms,

Number of passes **passesCnt** = 2.

In the DFM Ctrl window:

Phase and amplitude feedback gains:

Phase (top string): **I** = 2, **P** = 50.

Amplitude (bottom string): **I** = 1.

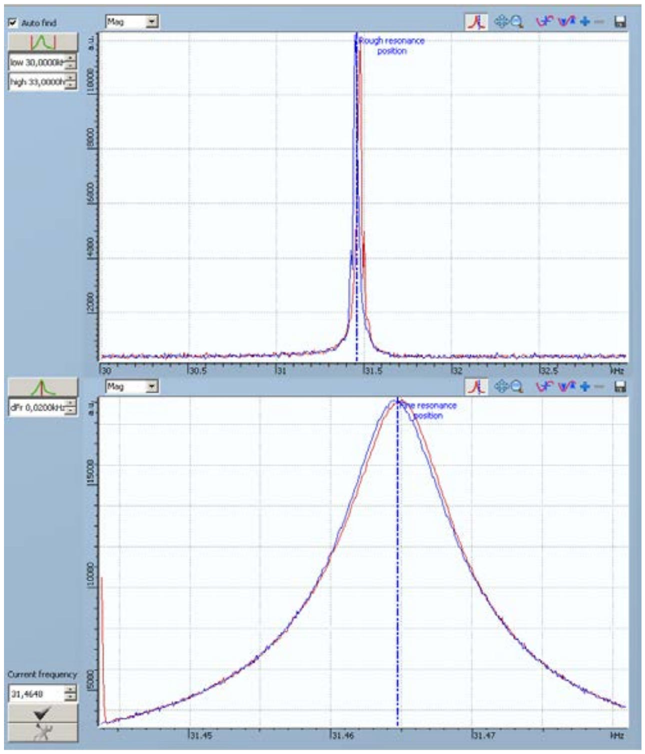
Oscillation amplitude setpoint **mag SP** = 20000.

In the Z-Motor Ctrl panel of the Scanner Ctrl window:

Motorized probe engagement parameters:
Approach speed **rate** = 2 um/s;
pause=150 ms;
Approach mode **Step range**: wide.

7.4.3 Setting resonance frequency

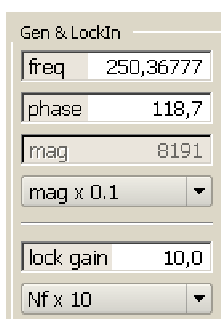
Open the **Resonance** window. Sweep the frequency in the coarse range and then sweep the frequency in the fine range. The resonance frequency should be ~32 kHz:



7.4.4 Setting probe's oscillation amplitude

In the **DFM Ctrl** window switch on the feedback loop with an amplitude signal at the input using the **Mag Fb** button. When the feedback, which has the **Mag** signal at the input is on, the amplitude of the **mag** signal is keep up at the **mag SP** = 20 000 au, and the physical amplitude of the tuning fork is set by changing the gain of the synchronous detector, **Lock gain**. The maximum **Lock gain** value corresponds to the minimum amplitude. At the coefficients of 100-10, the amplitude of the tuning fork is a few nanometers.

The recommended an initial value of **lock gain** is equal to 10:



7.4.5 Approach

Using the **move** button on the **Z-motor** panel which is opened in the **Scanner Ctrl** window, move the sample as close to the needle (up to a distance of 0.1 mm).

Turn the **Phase** signal in the oscilloscope.

Click and unclick the **Init** button in the **DFM Ctrl** window to set the **Phase** to 0.

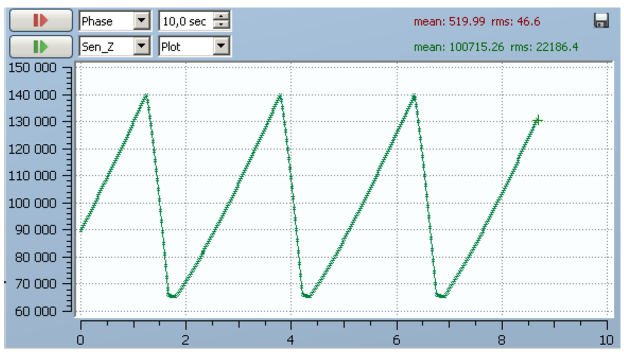
Open **Fb Tool Bar 1** and **2** control panels. Set **Phase** as the feedback signal, **SP** = -20000.
The feedback loop gain, **Gain** can be arbitrary:

Turn on **Phase** and **Sen_Z** in the oscilloscope.

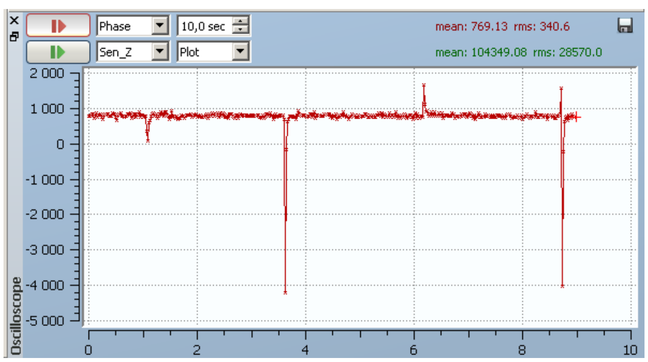
Press the **approach** button on the **Z-motor** panel of the **Scanner Ctrl** window.

Watch the selected signals in the oscilloscope during the approach procedure.

Sen_Z signal:



Phase signal:



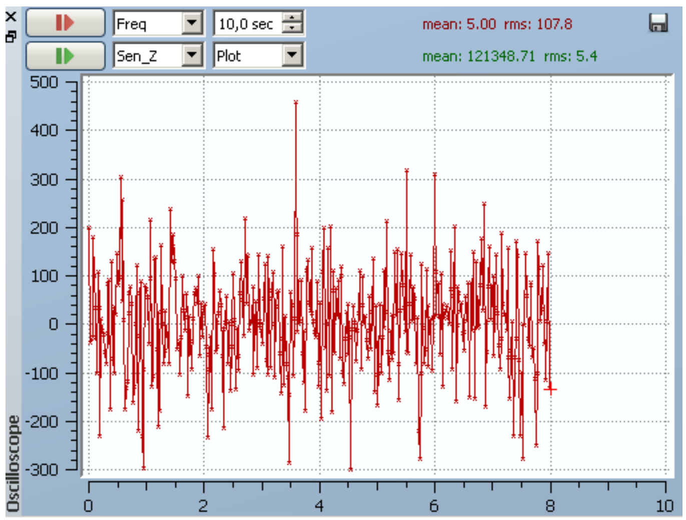
NOTE: There can be some false actuations of the approach mechanism due to some possible sudden changes of the phase signal caused by the stepper motor operation in the beginning and in the end of the scanner movement during each step. That's why you should run the approach procedure again right after it's finished for the first time to make sure that the phase change happens when the scanner moves forward.
The scanner moves backward to 1 micron after the approach procedure is complete.

7.4.6 Landing to the sample surface

Press the **Fb XY** button in the **Scanner Ctrl** window to switch on the scanner feedback in XY plane:

Press the **Phase Fb** and **Mag Fb** buttons in the **DFM Ctrl** window to switch on the corresponding feedback loops**:**

Select the **Freq** signal in the oscilloscope (one from the bottom of the list). The noise level of the **Freq** signal must be ~1 Hz (1 Hz is equal to 100 units at the oscilloscope scale):



Set **Freq** as the **FB** feedback signal, setpoint **Sp** = 2 Hz, **Gain** = 100:

Press the **land Simple** button in the **DFM Ctrl** window to run the landing procedure.

After landing, the Z-feedback has to be adjusted. In order to do that, monitor the **Freq** signal on the oscilloscope and gradually increase **Gain** on the **Fb Tool Bar 1** panel until parasitic oscillations appear. Set the **Gain** value to be equal to the half of the achieved value.

7.4.7 Scanning

After landing, go to the **Scan** window and select the **Qscan** mode. Make sure that in the settings of the **Qscan** mode, the **Adaptive** checkbox is marked. The quartz tuning fork has a very high quality factor. This fact appreciably limits the maximum scan rate at 0.5 - 1 Hz. In order to increase the scanning speed, use the **Back jump** mode.

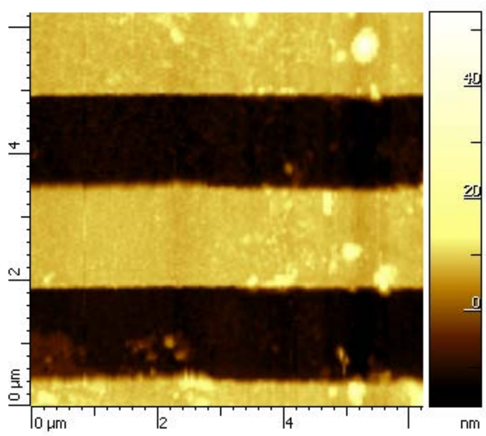
In addition to the height signals, check for recording **Phase** and **Freq** signals, which are the feedback error signals for this case.

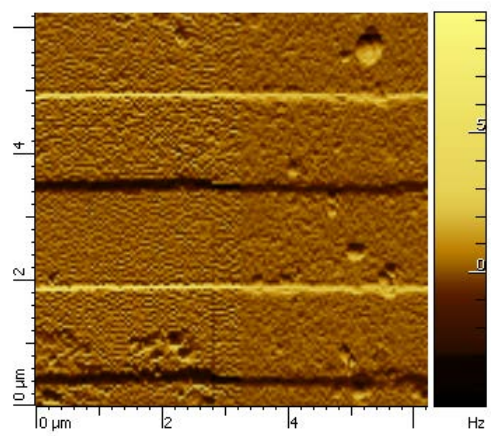
Set the scanning rate to 0.5 Hz and the scanning area to less than 5 microns.

Run the scanning procedure.

To adjust the quality of the image during the scanning process, change the scanning rate, the setpoint **Sp** within a few Hz and Z feedback gain **Gain**. Besides these parameters, you may change the oscillation amplitude of the probe by changing **mag SP** in the **Scanner Ctrl** window.

The parasitic oscillations in the Freq signal determine the maximum scanning speed. The scans below are images of the TGZ1 grating obtained by recording the **Freq** and **Phase** signals (scanning along the Y axis). The left sides for both of these scans were taken at the speed of 0.7 Hz and have "bouncing". It means that the speed of 0.7 Hz was too high for performance of these measurements. The right sides were taken at the speed of 0.3 Hz where noticeable "bouncing" disappeared.

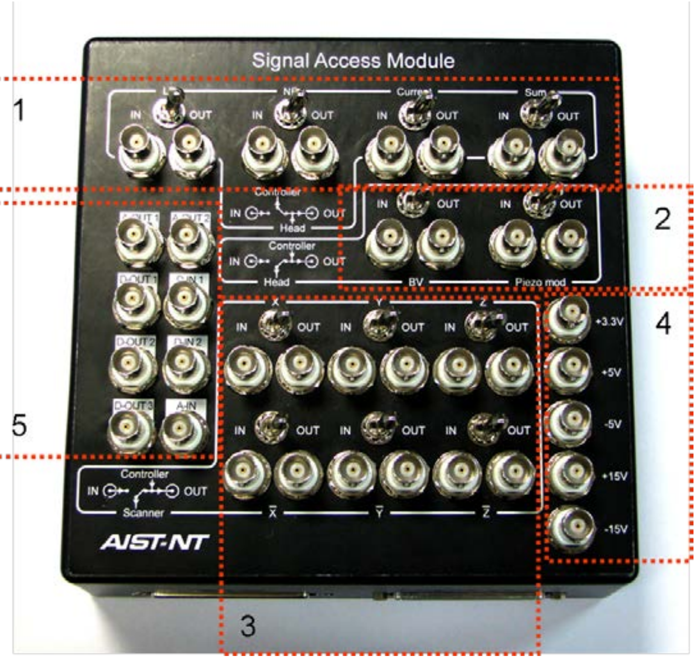




7.5 Signal Access Module

The Signal Access Module (SAM) allows the user to apply external signals to the instrument as well as output a number of internal instrument's signals to external devices.

7.5.1 Connectors on the top SAM panel



The groups of connectors are marked by corresponding numbers in the figure above:

Group #1

The Group #1 provides the access to the signals which comes to the controller from the measuring head:

- Lf - lateral cantilever deflection;
- Nf – normal cantilever deflection;
- Current – current signal (exists only if the conductive unit is available);
- Sum – total signal from the photodiode.

The I/O range of these four signals is from -12V to +12V in case the HE001 head is used. The range is limited by the buffer op amps.

For the HE002 head the I/O ranges are different:

LF, NF, Current: from -8.5V to +8.5V;

Sum: from -5V to +2V.

NOTE: The actual level of the signals is usually lower and depends on the registration system settings.
For each signal there are two connectors (IN and OUT) and switch.

ATTENTION! Shorting the OUT connector leads to the failure of the buffer op amp inside the SAM.

<u>OUT connector:</u> The corresponding signal from the measuring head always comes to this connector regardless of the switch position.

<u>IN connector:</u> Use this connector to connect the required external signal.

Switch in the OUT position (i.e. turned to the right):

The signal from the measuring head comes directly to the controller. This signal can be measured from the OUT connector. If any signal is applied to the IN connector, it doesn't come anywhere.

Switch in the IN position (i.e. turned to the left):

The signal from the measuring head comes to the OUT connector, but doesn't come to the controller. The signal applied to the IN connector comes to the controller. For instance, you may acquire the source signal from the measuring head, convert it and apply the converted signal to the controller.

Group #2

The Group #2 provides the access to the signals which come from the controller to the measuring head:

Bv - bias voltage applied to the cantilever;

Piezo mod - signal applied to the piezo element, which makes the cantilever oscillate.

For each signal there are two connectors (IN and OUT) and switch.

ATTENTION! Shorting the OUT connector leads to the failure of the buffer op amp inside the SAM.

ATTENTION! The Bv signal is provided without any buffer circuit, so shorting the OUT connector leads to the failure of the output controller amp.

OUT connector: The corresponding signal from the measuring head always comes to this connector regardless of the switch position.

IN connector: Use this connector to connect the required external signal.

Switch in the OUT position (i.e. turned to the right):

The signal from the measuring head comes directly to the controller. This signal can be measured from the OUT connector. If any signal is applied to the IN connector, it doesn't come anywhere.

Switch in the IN position (i.e. turned to the left):

The signal from the measuring head comes to the OUT connector, but doesn't come to the controller. The signal applied to the IN connector comes to the controller.

Group #3 (scanner)

The Group #3 provides the access to the signals which come from the controller to the scanner:

X, X – high voltage signals for the X axis;

Y, Y - high voltage signals for the Y axis;

Z, Z - high voltage signals for the Z axis.

The I/O range for all the signals: from -10V to +120V.

There are two piezo stacks in the SmartSPM which are used to move the scanner in X and Y directions. Due to this reason two signals are used per each axis: direct signal (X or Y) and opposite signal (X and Y), which works in antiphase regime (e.g. if the direct signal is 100V, the opposite signal is 10V). Both signals are the same (+55V) in the scanner's central position.
There is only one piezo stack in the Z direction, so the Z signal is not used.

The PI-517 scanner is used in the CombiScope system. For this system only the direct X, Y, Z signals are used.

ATTENTION! Shorting the OUT connector leads to the failure of the input high voltage amp on the HV controller's board, because no buffer circuit is used.

The principle of operation with these signals is the same as for the Group #2.

Group #4 (power supply connectors)

+3.3V, max load 100mA.

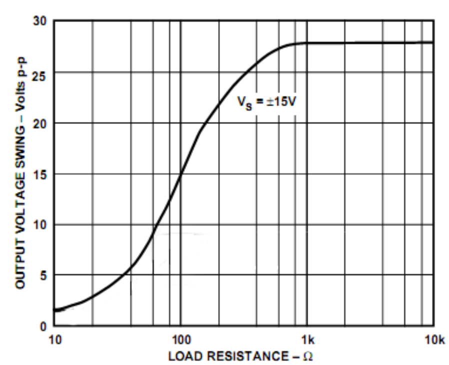
+/-5V, max load 100mA.

+/-15V, max load 70mA.

ATTENTION! Shorting the inner and outer contacts of any power supply connector leads to the failure of the controller power supply board.

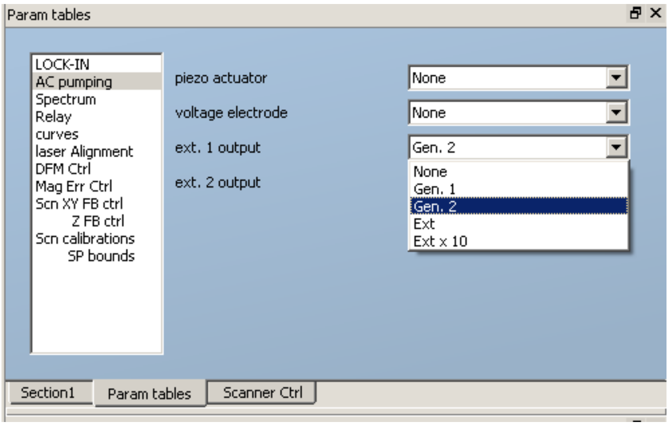
Group #5 (extension connectors)

Auxiliary I/O signals can be connected to these connectors.
To use these connectors, it is necessary to make a cable connection between the SAM-E connector on the SAM and the "External Input/Output" connector on the AFM board of the controller.

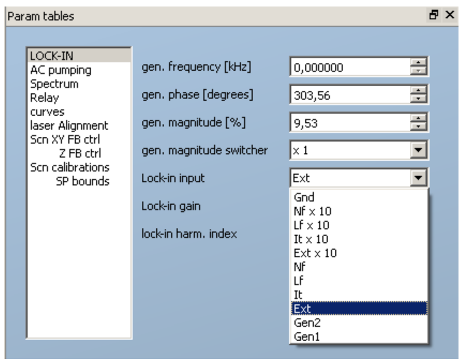
<table>
<tr><th>Name</th><th>Description</th><th>Voltage levels</th><th>Max. output current</th></tr>
<tr><td>D-OUT 1</td><td>Digital Output</td><td>0.0/+3.3 V</td><td>24mA</td></tr>
<tr><td>D-OUT 2</td><td>Digital Output</td><td>0.0/+3.3 V</td><td>24mA</td></tr>
<tr><td>D-OUT 3</td><td>Digital Output</td><td>0.0/+3.3 V</td><td>24mA</td></tr>
<tr><td>D-IN 1</td><td>Digital Input</td><td>0.0/+3.3 V</td><td>-</td></tr>
<tr><td>D-IN 2</td><td>Digital Input</td><td>0.0/+3.3 V</td><td>-</td></tr>
<tr><td>A-OUT 1</td><td>Analog output</td><td colspan="2" rowspan="2">

Output Voltage Swing vs. Load Resistance</td></tr>
<tr><td>A-OUT 2</td><td>Analog output</td></tr>
<tr><td>A-IN</td><td>Analog input</td><td>-12V to +12V</td><td>-</td></tr>
</table>

Open the **AC pumping** tab in the **Param tables** window to select the required signal from the **ext. 1 output** or **ext. 2 output** lists to be applied to the **A-OUT1** or **A-OUT2** connectors correspondingly:



The signal applied to the A-IN connector corresponds to the Ext signal in the control software. This signal can be applied to the lock-in amp input:



For more information about how to use the digital input/output signals please contact the AIST-NT developers.

7.5.2 Side connectors

Use the side connectors to connect the SAM to the instrument:

SAM-H – It's necessary to connect the 37-pin cable from the AFM board of the controller to this connector to enable the SAM, as it provides the power for the internal amps of the SAM. Once this cable is connected to the SAM, the connectors of the Group #1, #2, #4 (power supply) and the A-IN connector will be enabled.

SAM-S – Connect the 50-pin cable from the HV board of the controller to this connector to enable the connectors of the Group #3 (scanner).

Head - Connect the measuring head to this connector.

Scanner – Connect the scanner to this connector.

SAM-E – Connect the 15-pin "External Input/Output" cable from the AFM board of the controller to enable the extension connectors (Group #5).

You must connect only the SAM-H connector to the controller in order to enable the SAM. All other connectors can be left unplugged if the corresponding groups of connectors are not required.

7.5.3 Connecting the SAM to the instrument

To connect the SAM to the instrument, do the following:

1. Switch off the controller.
2. Unplug the measuring head cable **Head** from the controller and connect to the **Head** connector of the SAM. Connect the SAM and the **Head** connector of the controller with the **SAM-H** cable.
3. To use the Group #3 connectors, unplug the **Scanner** cable from the controller and connect it to the **Scanner** connector on the SAM. Connect the **Scanner** connector of the controller and the SAM with the **SAM-S** cable.
4. To use the extension connectors, connect the 15-pin cable to the **SAM-E** connector on the SAM and to the **External Input/Output** connector on the controller.

8. APPENDIXES

8.1 Troubleshooting and maintenance

GENERAL PROBLEMS:

Despite the fact that the program is still running (i.e. the program does not hang up or is not crashed), it does not perform any actions if you press any program buttons. The "**failed to get current signal value**" message appears in the **Log window**. The oscilloscope signal(s) has stopped.

Possible reason	Solution
Connection between the controller and computer is lost.	Press **the Reset Connection** button from the main program menu **Modes -> Service** to try to resume the connection.

LASER-TO-PROBE ADJUSTMENT

Cantilever cannot be found

Possible reason	Solution
The probe has no cantilever.	Replace the probe to a new one.
The probe is not correctly installed into the probe holder (e.g. slightly displaced).	Re-install the probe correctly.
Initial position of stepper motors is lost.	Press the "**New tip**" button to retract the sample from the probe tip to 2 mm. Press the "**Init position**" button in the "**Laser calibration**" window.
Incorrect search algorithm (for HE001 only)	By default the algorithm #2 ("search from the center") is used. Try to use the algorithm #1 ("search from the chip edges") **Param tables -> Laser alignment -> Laser alignment method=1.**
Incorrect search parameters (i.e. changed or deleted).	Restore the initial search parameters. You can find the search parameters in the **Laser Alignment** tab of the **Param tables**: Y pos = 25 X pos = 0 laser threshold = 8000 laser Background = 5000 length of cantilever = 2000 laser align method = 2

RESONANCE

Resonance peak cannot be found. The **“failed to set amplitude”** message appears.

Possible reasons	Solution
The probe holder is disconnected.	Connect the probe holder to the microscope head.
The resonance peak is out the specified frequency range.	Change the frequency range in the “**Resonance**” window, using the “**Low**” and “**High**” input fields, where you expect the system to find the resonance peak.
Incorrect amplitude of probe oscillation is set.	Set up the amplitude value in the range of 20 – 100 nm.

APPROACH

The approach procedure takes more than 10 minutes. A number of vertical segments appear on the approach curve.

Possible reason	Solution
A sudden jump of some signal occurs and makes the program slow down the approach speed as if the sample surface is very close to the probe tip.	Press the “**moto approach”** button to restart the approach procedure. The approach procedure interrupts and restarts from the current height.

LANDING TO YOUR SAMPLE SURFACE

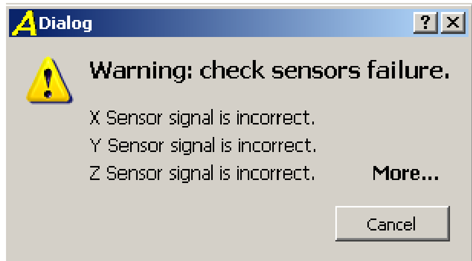
The system is unable to land. The “**Failed to land**” message appears.

Possible reason	Solution
Your sample surface is too far away from the probe, so the vertical (Z) range of the scanner is not enough.	Press the **Moto approach** button to restart the motorized approach procedure. Press the **Landing** button once the motorized approach procedure is completed.

8.2 Scanner sensors calibration

With any of AIST-NT's scanning probe microscopes the scanner motion control is realized based on the capacitive sensors closed loop feedback. To provide the efficient and accurate scanner operation, one should properly adjust and calibrate the capacitive sensors. The initial calibration is done during production, but from time to time it can appear that it's necessary to recalibrate the sensors. More often recalibration of the Z axis sensor is required.

If the scanner sensors calibration is invalid, then during controller initialization or during switching between the low and high voltage modes, the following warning message appears:

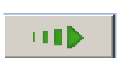


NOTE: Before calibrating the scanner sensors, make sure that the scanner is properly connected with the controller and your **default.scn** scanner settings file corresponds to your scanner number.

NOTE: One of the reasons why the scanner sensors calibration may fail can be your instrument temperature fluctuations. Therefore, before recalibrating the sensors make sure that you still see the warning message even if the room temperature is stabilized.

The scanner sensors calibration procedure is automatic.

To run this procedure, do the following:

- Select **Sensors** in the **Scanner** submenu of the main menu program to open **Sensors Ctrl** control panel (fig. 8.2.1).
- Tick off the corresponding checkbox to calibrate **X**, **Y** or **Z** sensor. To calibrate all three sensors together, tick off the **All** checkbox.
- Press  to start.
- Press **Yes** in the pop-up window to retract the scanner from the probe.

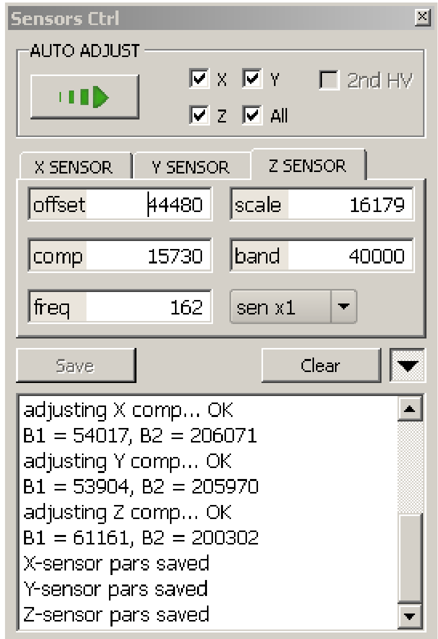
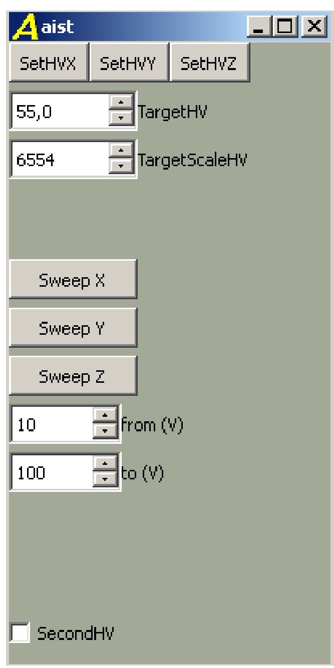


Figure 8.2.1 Sensors Ctrl scanner sensors calibration control panel

After that the sensors will be recalibrated. The new calibration parameters will be automatically saved in the **\aist\params\scanner\default.scn** scanner settings file after program shutdown.

To check if the scanner sensors calibration is correct use **AFM11_Sensors1.lua** macro. This macro allows applying saw-shaped voltage to each scanner's axis:

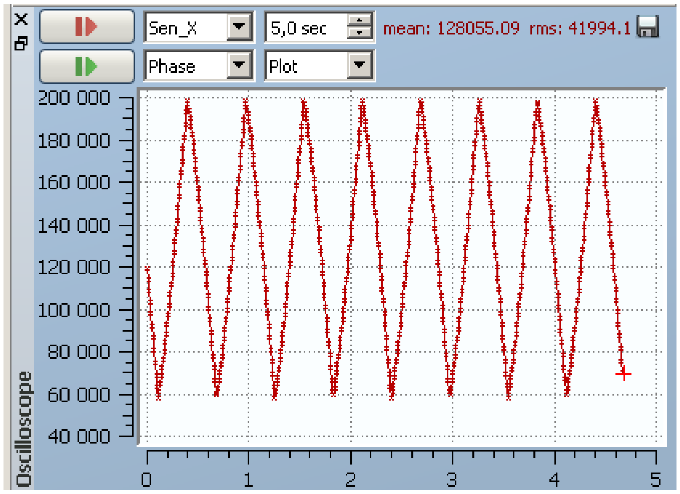


Remove the scanner to more than 100 µm from the probe tip.

- Press one after another **SetHVX, SetHVY, SetHVZ** to set the scanner in the center of the scanning range.

NOTE: All X, Y and Z scanner feedbacks are automatically switched off when pressing one of these buttons;

- Press one of the following buttons - **Sweep Z, Sweep X** or **Sweep Y** to apply the voltage sweep signal to one of the scanner axes. The sweep signal range is from 10 to 100 volts and can be set using the **from(V)** and **to(V)** input fields;
- Open the oscilloscope window and select the corresponding sensor signal (**Sen_Z**, **Sen_X** or **Sen_Y**). **Notice that**



the sensor signal should vary from 60000 to 200000 ADC units when the sweep signal is applied:

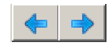
8.3 Central position setup of the XY sample positioning system

At the motion in the XY plane, the central position of the positioning system has to be accurately determined for correct conversion of motor steps in micrometers. In the production of the instrument, only the approximate central position is specified, therefore, for more precise positioning, the central position has to be clarified. The values of **XmotorCenter** and **YmotorCenter** parameters define the central position and can be found in the file with other parameters of the scanner. By default, both values are 9500.

The procedure for determining the central position of the positioning system is based on the fact that in the central position, the rotation of the eccentric on half of the turn moves the positioning system back in the same position. The complete turn of the eccentric corresponds to 10 240 steps of a stepper motor and a half turn - 5120 steps.

Do the following steps to determine the central position of the XY sample positioning system:

- Remove the cantilever holder from the instrument.
- Install some sample that has some noticeable features on its surface into the microscope.
- Open the **XYZ Motors** window.
- Move up the scanner to 10 mm to its working position where the sample is to be fixed.
- Press the **init position** button to move the XY sample positioning system to its central position.
- Set the minimum magnification of your optical microscope and focus the microscope on the surface of the sample.
- Find some noticeable point-like feature on the video image and mark its position on your monitor (for example, you can use a piece of paper sticker).
- In the **XYZ Motors** window check and if it is necessary select in the drop down menu the **steps** units for the sample displacement.
- Move the positioning system for 5120 steps on the X axis using the **X** text box and the  arrow.

- Mark the new position of the feature if it has moved.
- Move it to the middle point between its previous and new locations, using the  arrows and setting a small shift in the **X** textbox.
- Repeat above three steps using a higher optical magnification, so there is no shift of the point-like feature.
- Repeat all above steps for the Y axis.
- Press the **findSensor** button to read the information about XY coordinates of the central position. The sensors coordinates are show in the **Log window**.
- If any of the values appears to be less than 5120, move the positioning system for 5120 steps along the corresponding axis and read the sensors position once again.
- Save the found position in the file of scanner parameters using an **update center** command.

8.4 Focus adjustment of the optical registration system of the HE002 head

In order to provide the optimal size of the laser spot in the optical registration system of the measuring AFM head, laser beam is focused by a lens on the surface of the cantilever. Focus is adjusted by changing the distance between the cantilever and the lens.

The need to readjust the focus is related to the difference in the thickness of the probe chips from different manufacturers. The manufactures offer probes with chips of 0.3 mm, 0.4-0.45 mm, and 0.5 mm thicknesses.

In the HE002 head the change of the focus length is made by rotating of the front stop screw of the probe holder (see **Figure 8.4.1**). To ease the adjustment, the front set screw has a groove, and the probe holder has three marks corresponding to the chip thicknesses of 0.3, 0.4 and 0.5 mm (**Figure 8.4.2**).

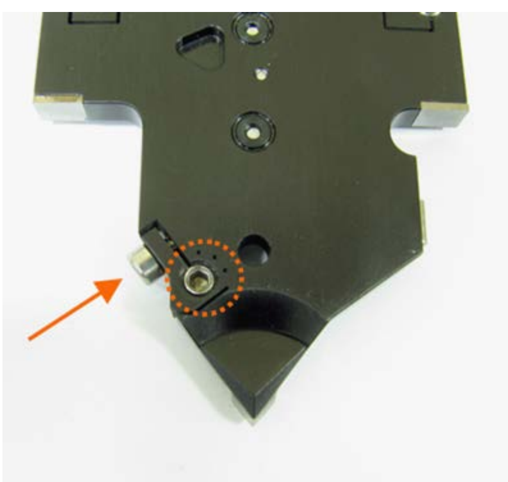


Figure 8.4.1 Probe holder. The front set screw and marks are inside of the circle; the arrow points to the locking screw.

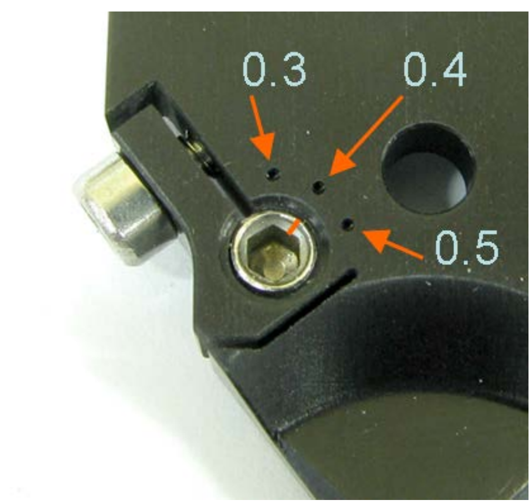


Figure 8.4.2 The marks on the probe holder

In order to readjust the probe holder on another chip thickness, follow to below steps:

Loosen the locking screw (indicated by the arrow in **Figure 8.4.1**), so that the stop screw could be rotated with a little resistance.

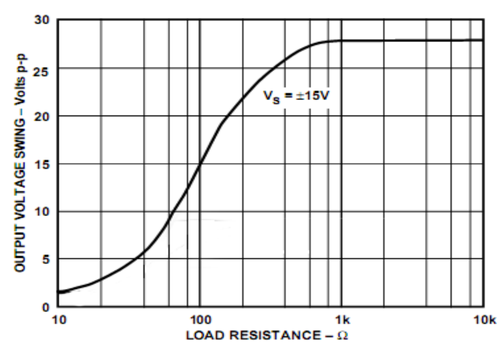
For adjustment onto the needed thickness, turn the front set screw so that the groove is in front of the mark corresponding to the thickness of the used probe (**Figure 8.4.2**).

After adjustment, tighten the locking screw.

8.5 External Input/Output connector specification

The controller has an **External Input / Output** connector which can be used to connect the external devices.

CONNECTOR SPECIFICATION:

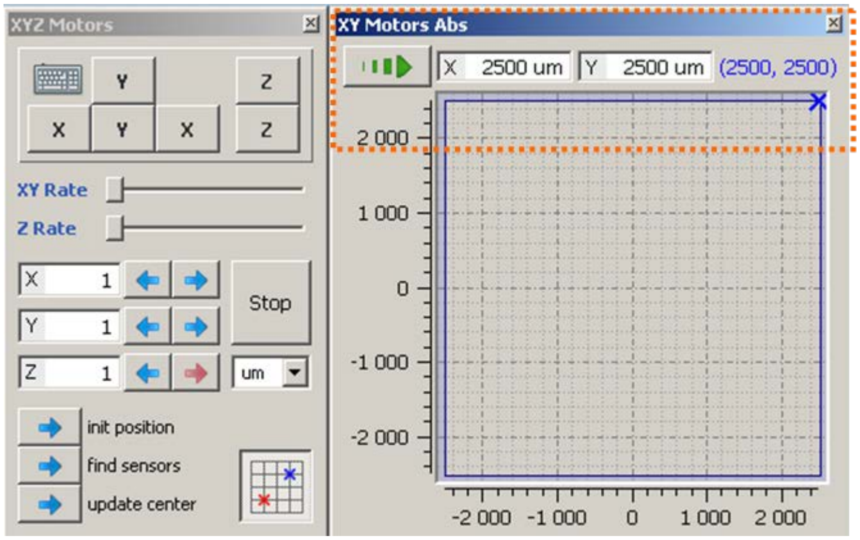
Pin #	Description	Voltage levels	Max. output current
1	+3.3 V		100mA
2	+15 V		100mA
3	digital output #2	0.0/+3.3 V	24mA
4	digital output #1	0.0/+3.3 V	24mA
5	Bias Voltage input	maximum input voltage cannot exceed 50V	
6	-5 V		100mA
7	+5 V		100mA
8	Gnd		
9	digital input #1	0.0/+3.3 V	
10	digital input #2	0.0/+3.3 V	
11	Gnd		
12	-15 V		100mA
13	digital output #3	0.0/+3.3 V	24mA
14	analog output (Ex2)	 *Output Voltage Swing vs. Load Resistance*	
15	analog output (Ex1)		

8.6 Position of the scanner for transportation

For the transportation of the probe microscope, scanner has to be set into its transport position.

In order to set the scanner into its transport position, follow below steps:

1. Move the positioning system of the scanner to 5-15 mm up from the lowest possible position.
2. Set the positioning system to its center position by clicking on the **init position** button in the **XYZ Motors** window.
3. Move the positioning system to the point with coordinates of X = 2500 and Y = 2500, using the **XY Motors Abs** coordinate field. This position corresponds to the upper right corner of the coordinate fields:



4. Move the positioning system of the scanner to the lowest position by pressing **Go home** on the **AC mode** control panel.