

SPM SmartSPM™-1000 Instruction Manual

software version: 3.3.68

version: 01.2013



CONTENTS:

| | |
|--|-----------|
| 1. SYSTEM OVERVIEW | 5 |
| 1.1. Smartspm main parts and design | 6 |
| 1.1. Overview of the software interface | 8 |
| 1.2. Scanning probe microscopy techniques overview | 9 |
| 2. TECHNICAL SPECIFICATIONS OF SMARTSPM 1000..... | 11 |
| 2.1. Measuring modes | 11 |
| 2.2. Scanner and Base | 11 |
| 2.3. AFM Head HE001 | 11 |
| 2.4. Liquid cell with temperature control (optional)..... | 12 |
| 2.5. Conductive AFM unit (optional) | 12 |
| 2.6. Controller electronics | 12 |
| 2.7. Software | 12 |
| 3. PERFORMING MEASUREMENTS..... | 13 |
| 3.1. Switching on the system | 13 |
| 3.2. Sample installation..... | 15 |
| 3.3. Selecting a probe and measuring mode | 17 |
| 3.4. Probe installation | 18 |
| 3.5. Adjustment of an optical registration system..... | 19 |
| 3.6. Adjusting the instrument for the Semicontact / Non-contact AFM mode | 23 |
| 3.6.1.1. Automatic adjustment to the resonance frequency | 24 |
| 3.6.1.2. Setting the amplitude of the cantilever | 25 |
| 3.6.1.3. Manual selection of the operating frequency | 25 |
| 3.6.1.4. Manual setting of amplitude and magnitude of the Mag signal..... | 26 |
| 3.6.1.5. Difficulties at tuning to the resonant frequency | 26 |
| 3.6.2.1. Preliminary approach | 28 |
| 3.6.2.2. The approach with automatic distance control | 28 |
| 3.6.2.3. Fine tuning of the oscillation frequency, phase and amplitude near the sample surface | 29 |
| 3.6.2.4. Landing to the sample surface..... | 29 |
| 3.6.3. Feedback adjustment | 30 |
| 3.7. Adjusting the instrument for the Contact AFM mode..... | 36 |
| 3.8. Automatic instrument adjustment..... | 36 |
| 3.9. Scanning..... | 37 |
| 3.10. Adjusting scanning parameters | 37 |
| 3.11. Changing AFM mode..... | 38 |
| 3.12. Saving and processing scanning results | 39 |
| 3.13. Sample or probe replacement, shutting down | 39 |
| 3.14. Measurements of curves | 39 |

| | |
|--|----|
| 4. MEASUREMENT TECHNIQUES | 41 |
| 4.1. Low voltage mode | 42 |
| 4.3. Two-pass magnetic force microscopy and electric force microscopy techniques | 42 |
| 4.4. Single-pass MFM technique | 43 |
| 4.5. Kelvin probe force microscopy (KPFM) | 44 |
| 4.6. Piezo Response Force Microscopy | 46 |
| 4.6.1. Choice of PFM technique | 46 |
| 4.6.2. Choice of cantilever | 46 |
| 4.6.3. Sample Requirements | 46 |
| 4.6.4. Installing the Sample | 46 |
| 4.6.5. Contact PFM mode control panel | 47 |
| 4.7. Top mode | 55 |
| 4.8. Nanolithography | 56 |
| 4.8.4 Macro Litho lines | 60 |
| 4.9. "Moto Scan" mode | 62 |
| 4.10. Obtaining atomic resolution in the AFM mode | 63 |
| 4.10.1. Measurement conditions | 63 |
| 4.10.2. Preparing a sample | 63 |
| 4.10.3. Estimation of noise conditions | 63 |
| 4.10.4. Conducting measurements | 65 |
| 4.10.5. Setpoint selection | 66 |
| 4.11. Dynamic Force Microscopy (DFM) using cantilever as a probe | 67 |
| 5. SAMPLE POSITIONING | 69 |
| 5.1. The continuous movement of the sample | 70 |
| 5.2. Moving the sample at a specified distance | 71 |
| 5.3. Moving to a defined point | 71 |
| 5.4. Setting the positioning system to the central position | 71 |
| 6. FAST SCANNING | 72 |
| 6.1. Switching to the fast scanning mode | 74 |

| | |
|---|------------|
| 7. OPERATION WITH ADDITIONAL EQUIPMENT | 75 |
| 7.1. Conductive AFM..... | 75 |
| 7.1.6. Getting series of volt-ampere characteristics | 81 |
| 7.2. Scanning Tunneling Microscopy (STM) | 83 |
| 7.3. Operation in liquid | 89 |
| 7.3.6.1. Selecting the cantilever drive frequency in liquid | 93 |
| 7.3.6.2. Adjust the drive frequency | 94 |
| 7.3.6.3. Tip-to-sample approach | 95 |
| 7.4. Working with the quartz oscillator (Tuning Fork mode) | 96 |
| 7.4.1. Assembling the system to work with the quartz oscillator | 98 |
| 7.4.2. Making a probe | 99 |
| 7.4.3. Probe mounting | 99 |
| 7.4.4. Preparing the device for measurements | 99 |
| 7.4.5. Switching on the Tuning fork mode | 101 |
| 7.4.6. Adjusting the resonance..... | 101 |
| 7.4.7. Setting the oscillation amplitude | 102 |
| 7.4.8. Preliminary approach | 103 |
| 7.4.9. Automatic approach | 103 |
| 7.4.10. Selecting a feedback signal | 103 |
| 7.4.11. Landing to surface | 103 |
| 7.4.12. Setting up Z feedback | 104 |
| 7.4.13. Scanning | 104 |
| 7.4.14. Image quality optimization..... | 105 |
| 7.5. Signal Access Module | 106 |
| 8. APPENDICES..... | 110 |
| 8.1. Troubleshooting and maintenance | 110 |
| 8.2. Scanner sensors calibration | 112 |
| 8.3. Central position setup of the XY sample positioning system | 114 |
| 8.4. Focus adjustment of the optical registration system of the HE001 head..... | 115 |
| 8.5. External Input/Output connector specification | 117 |
| 8.6. Position of the scanner for transportation..... | 117 |

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 ambient, liquid and controlled atmosphere environment. 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.
- 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).

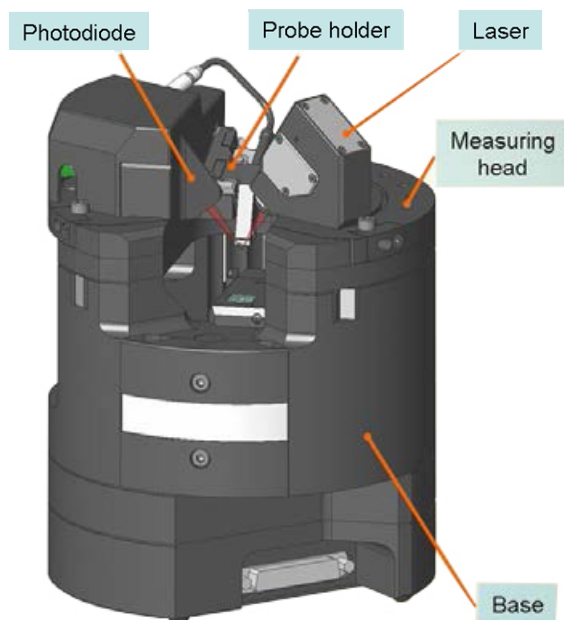


Figure 1.1.1. SmartSPM main parts.



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). The tip sharpness is one of the most important parameter that determines the maximum resolution of the microscope.

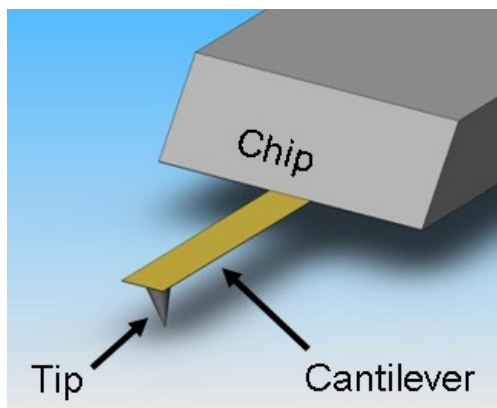


Figure 1.1.3. Cantilever type probe for atomic-force microscopy.

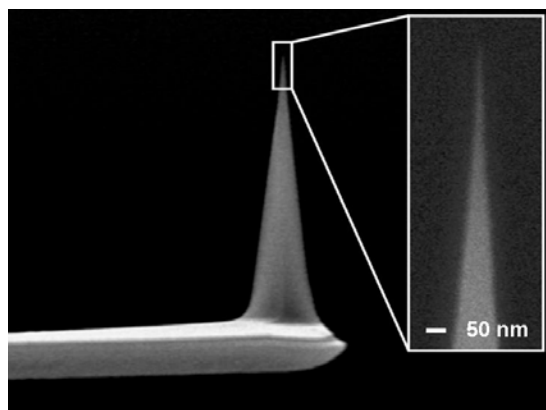


Figure 1.1.4. SEM image of the probe.

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.

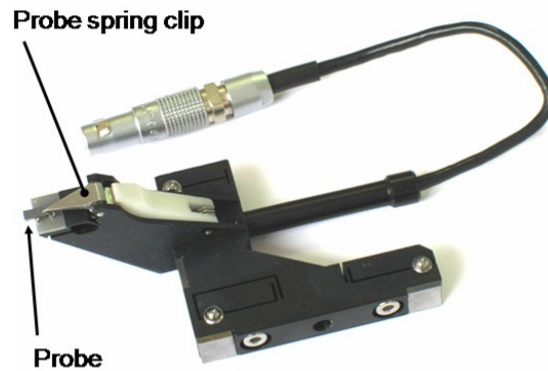


Figure 1.1.5. The probe holder with the installed probe.

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 18 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.

1.1 OVERVIEW OF THE SOFTWARE INTERFACE

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

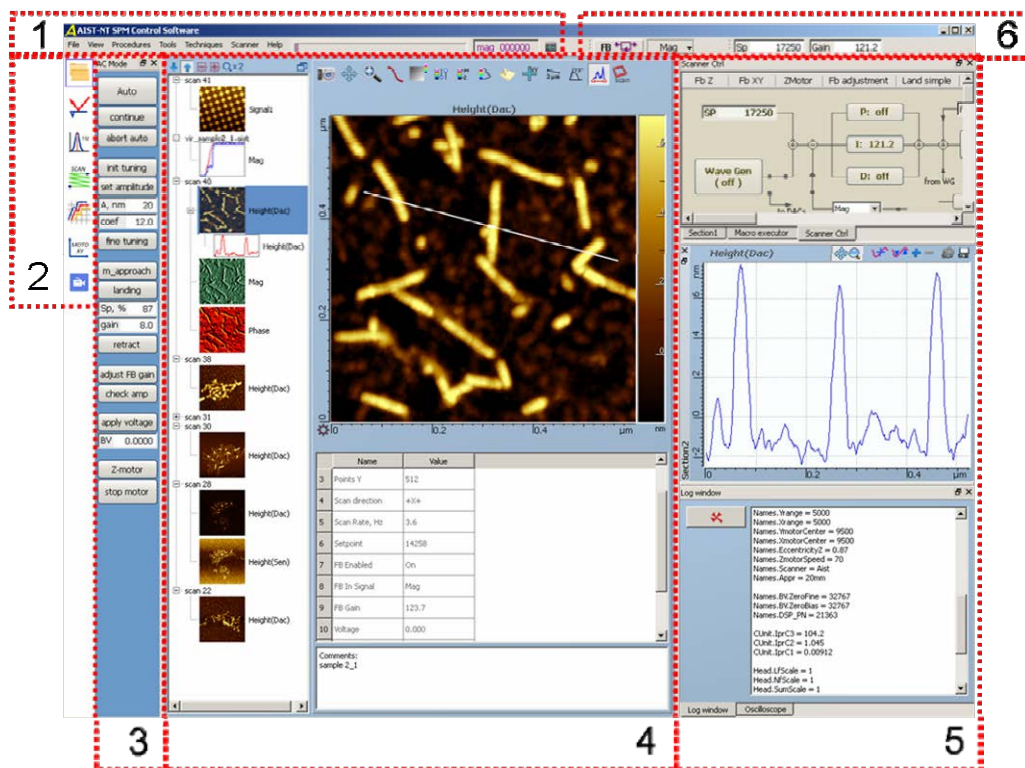


Figure 1.2.1. User interface of AIST-NT SPM Control Software.

The numbers corresponds 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".

1.2 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

2.1. Measuring modes

- Contact AFM in air/(liquid optional);
- Semicontact AFM in air/(liquid optional);
- 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).

2.2. 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 range 5x5mm;
- Positioning resolution 1um.

2.3. AFM Head HE001

- 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 (10x, NA=0.28 and 20x, NA=0.42 respectively).

2.4. Liquid cell with temperature control (optional)

- Sample size 2mm thickness, 25mm diameter;
- Heating up to 60°C;
- Cooling below room temperature down to 5°C;
- Sample positioning range 5x5mm;
- Positioning resolution 1µm;
- Cell size 40x40x12mm;
- Volume of liquid 3ml;
- Capability of liquid exchange;
- Autoclave and ultrasonic cleaning of cell parts.

2.5. 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.

2.6. 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.

2.7 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).

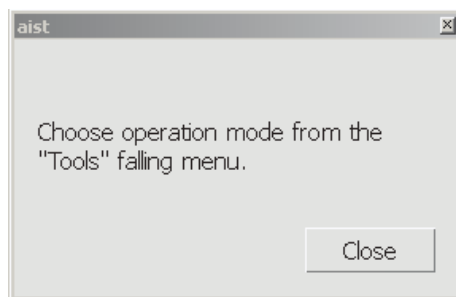


Figure 3.1.2 Information window

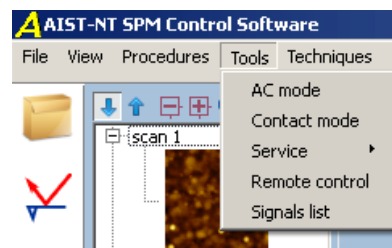


Figure 3.1.3 “Tools” pull-down menu

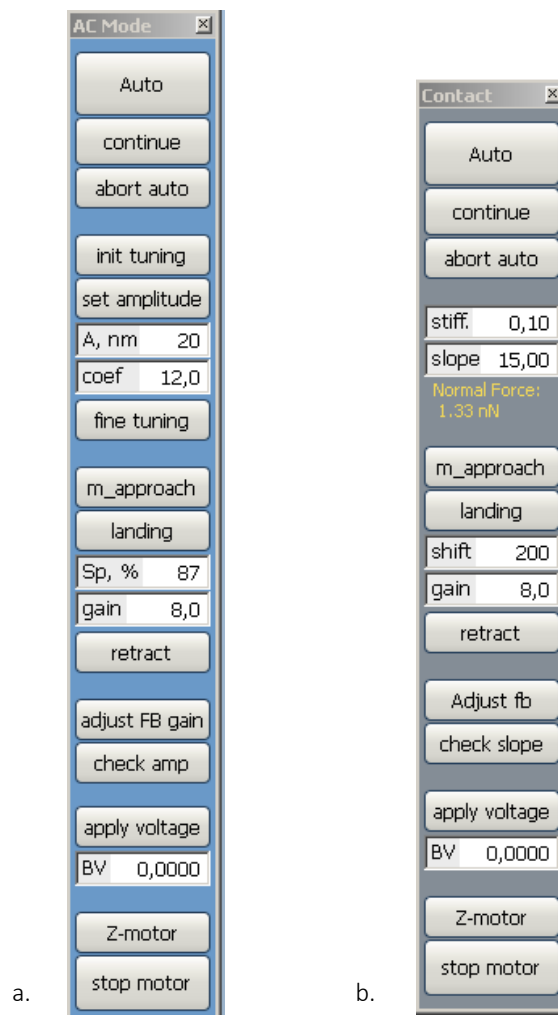


Figure 3.1.4 Control panels: **(a)** semi-contact/non-contact modes, **(b)** contact mode.

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).

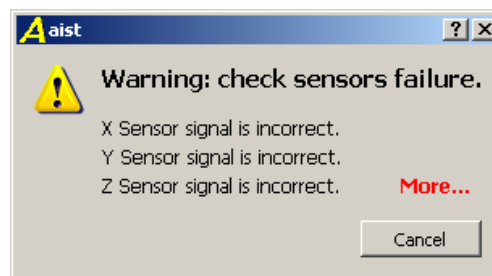


Figure 3.1.5 Warning window of scanner sensors calibration

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 **part 8.2 of Appendix**.

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.

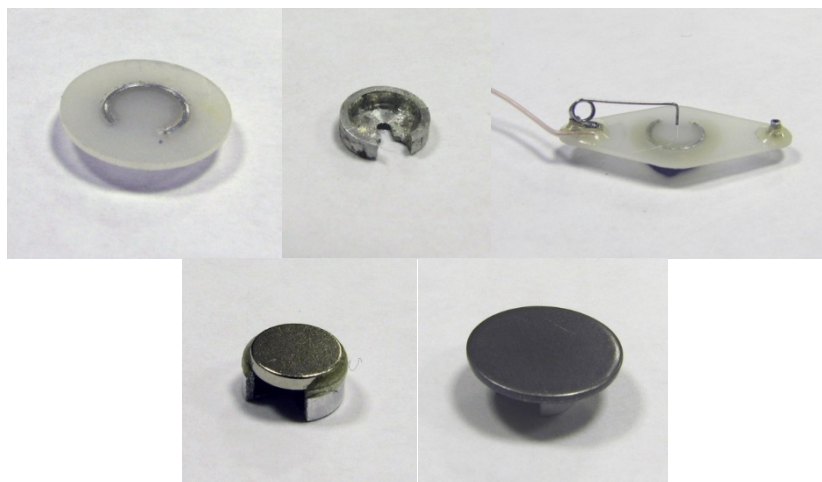


Figure 3.2.1. Sample holders: **1**—basic holder, **2**—holder for fast scanning, **3**— holder with an electrical contact, **4** and **5**— magnetic holder.

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).

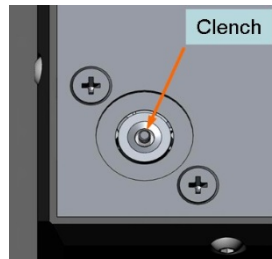


Figure 3.2.2 Sample holder fixing clench

To install a sample holder, do the following steps:

- Press the **Z-motor** button in the bottom of the **AC mode** 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. Z-motor Ctrl control panel

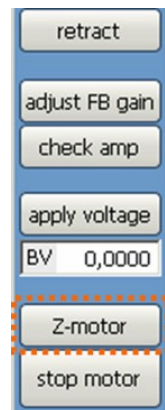


Figure 3.2.4. Position of the Z-motor button on the AC mode control panel

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

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



Figure 3.2.5 Fixing clench handle.



Figure 3.2.6 Sample holder installation.

- 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 holder by pressing the “New tip” button on the control panel.
- Disconnect the probe holder electric connector from the measuring head.
- Remove the probe holder from the carriage by pulling and then turning holder’s latch clockwise up to the stop (fig. 3.4.1, 3.4.2).

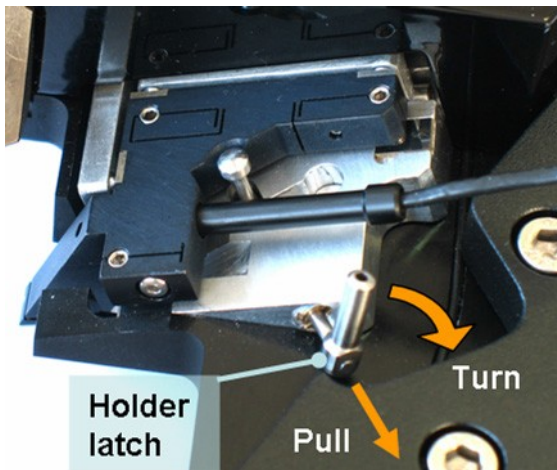


Figure 3.4.1 Probe holder fixed by its latch.

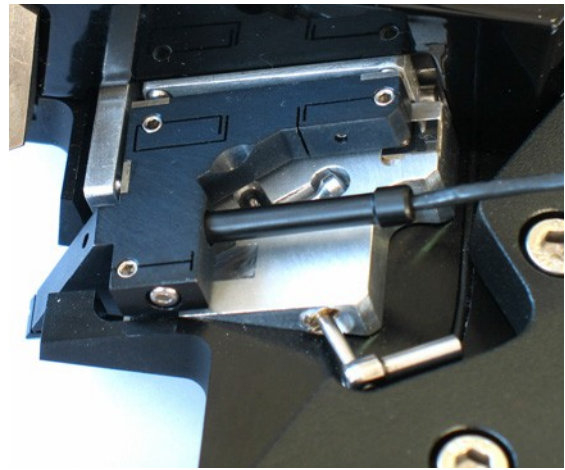


Figure 3.4.2 Loose probe holder.

- Place the probe holder on the table in upside-down position.
- Take one probe from the box using the 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 hinge.
- Push the white key to open probe's spring clip (fig. 3.4.3).
- Using tweezers place the probe under the clip as shown on fig. 3.4.4.

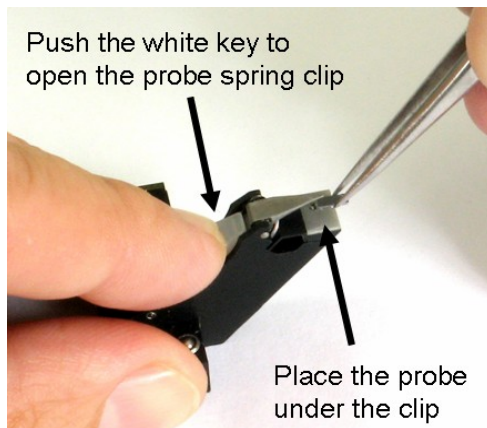


Figure 3.4.3 Installing the probe holder into the probe holder.

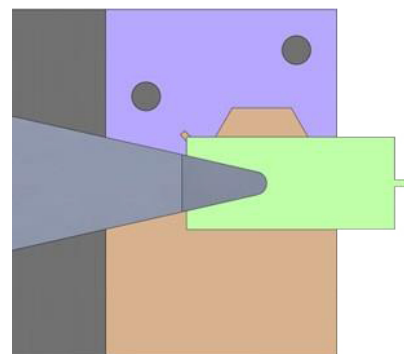


Figure 3.4.4 Proper probe position in the

- Install the probe holder into the carriage, fix it with the latch and attach the electric cable, so that the red stripe mark on the connector is opposite 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  icon 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.

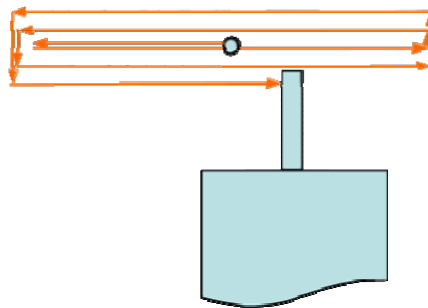


Figure 3.5.1 The trajectory of the probe holder at finding of the cantilever

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).

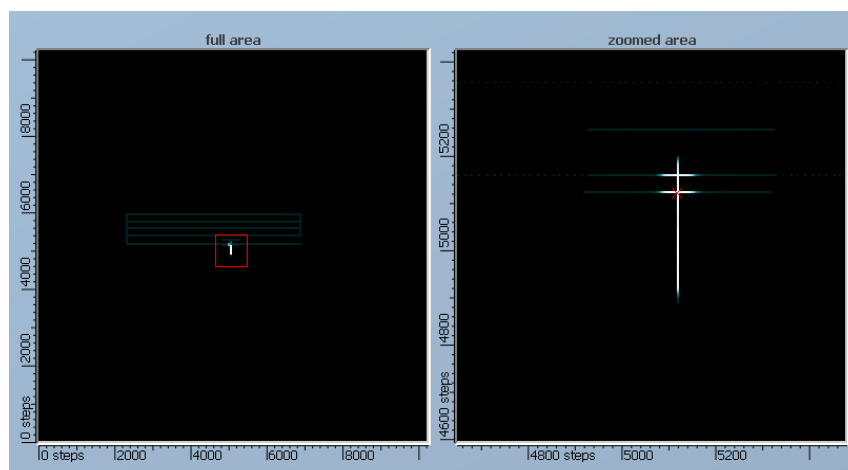


Figure 3.5.2 The image of the rectangular cantilever in the Laser Adjustment window, obtained after looking for a probe using the find tip procedure.

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.

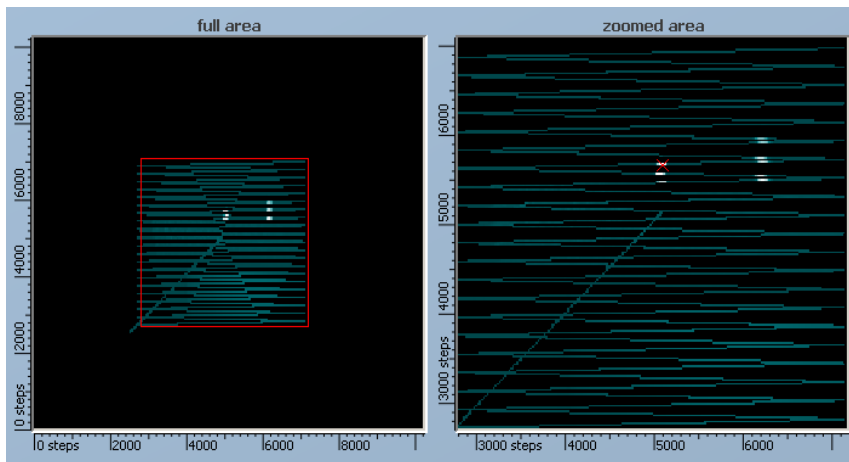


Figure 3.5.3 Manual adjustment on the probe with the two levers. The laser beam is focused on the short lever.

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:

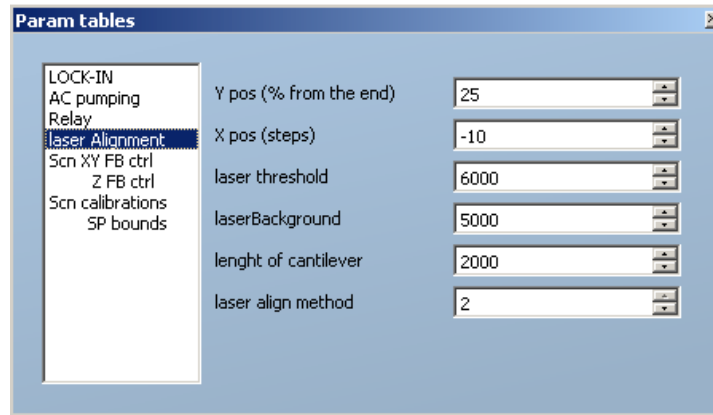


Figure 3.5.4 The Laser Adjustment bookmark in the Param tables window

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. 3.5.5).
- 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.

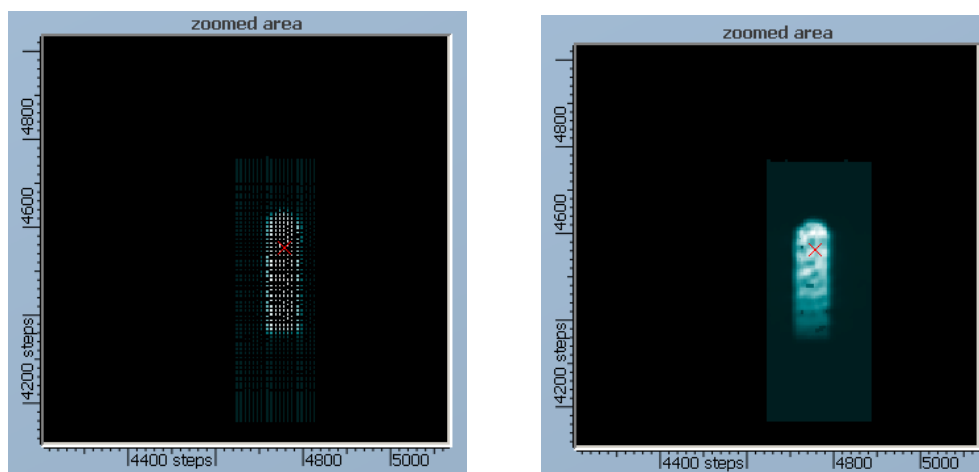


Figure 3.5.5 **Left:** Image of the cantilever obtained by using show tip command.

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

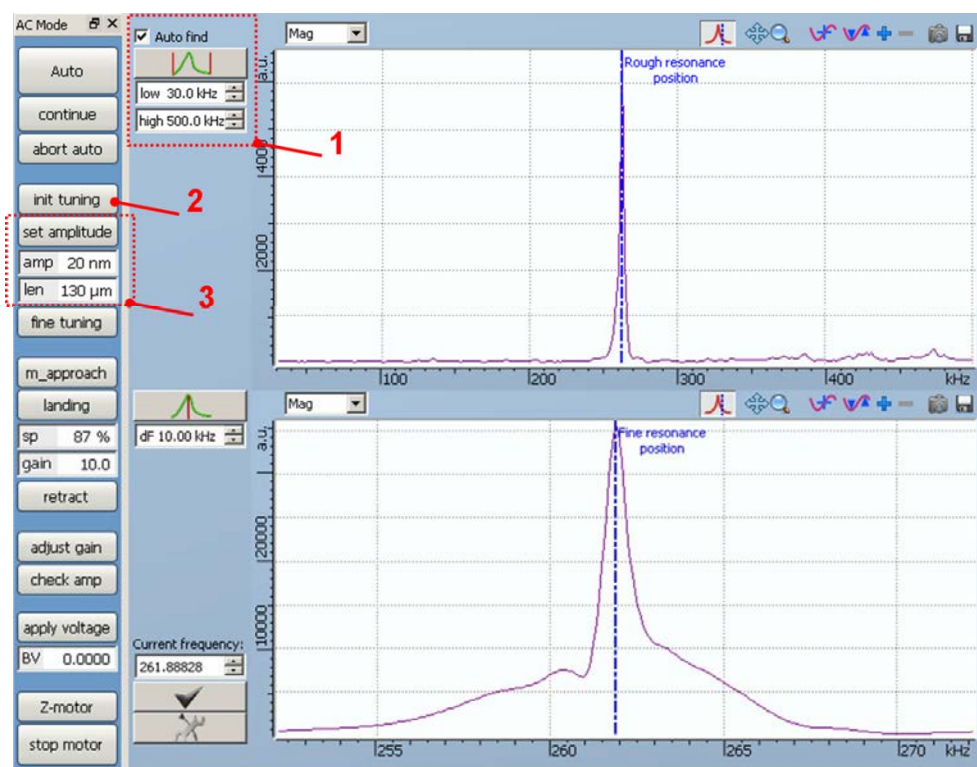


Figure 3.6.1.1 The Resonance window that displays resonance curve of the probe in defined range (upper field) and around specified frequency (lower field). To the left of the window, the AC mode control panel is shown.

The numbers designate: **1** - low and high fields, **2** - the init tuning button, **3** – the set amplitude button and amp and len fields.

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 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.

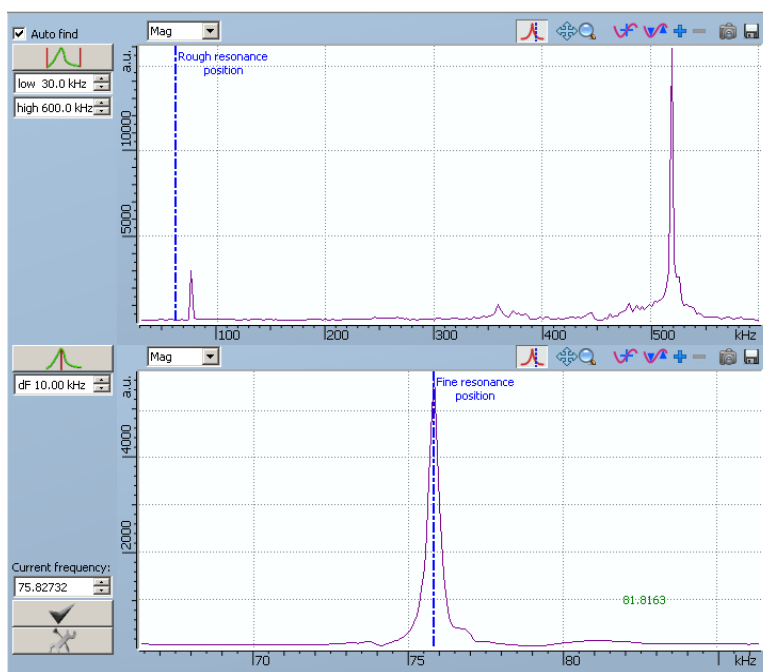

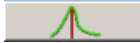



Figure 3.6.1.3 Resonance curve with two peaks Follow the procedure below:

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).

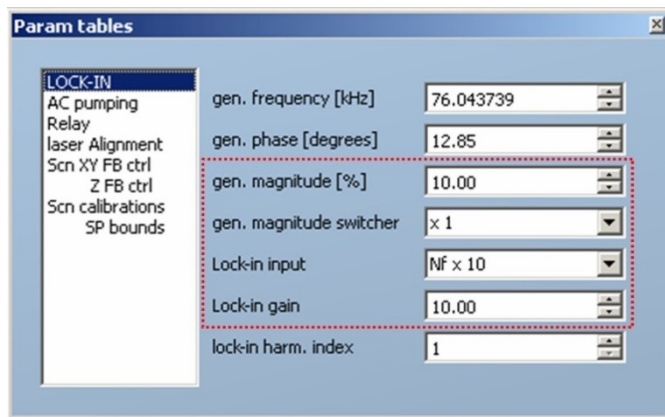


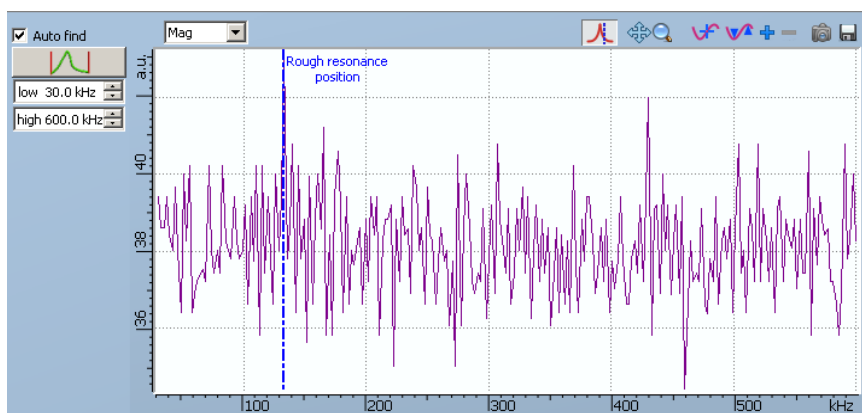
Figure 3.6.1.4 The LOCK-IN bookmark in the Param tables window

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.

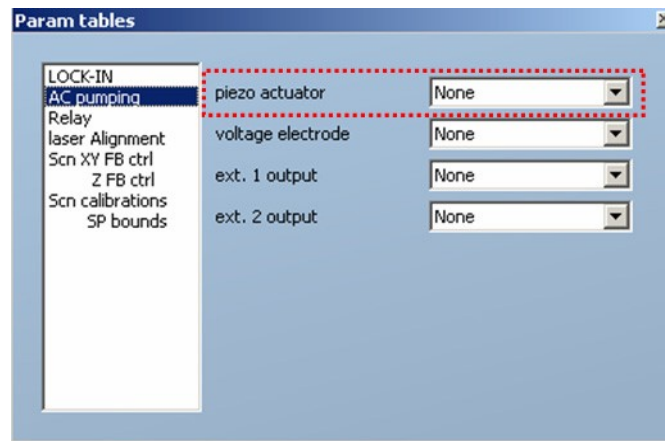
3.6.1.5 Difficulties at tuning to the resonant frequency

The complete absence of resonances:

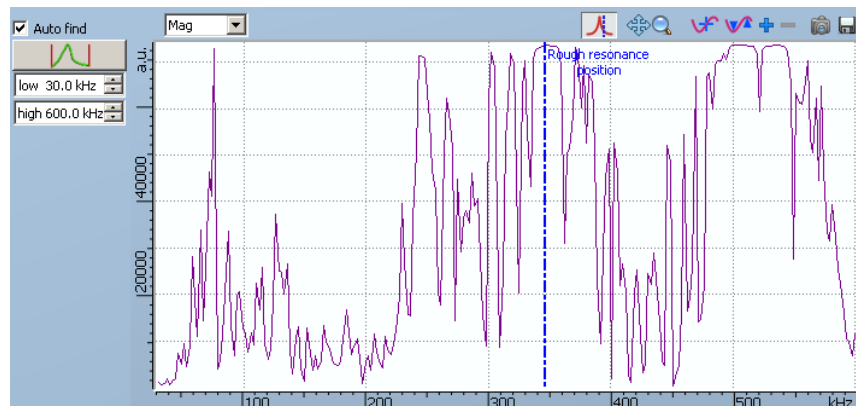


The following are some possible reasons:

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:

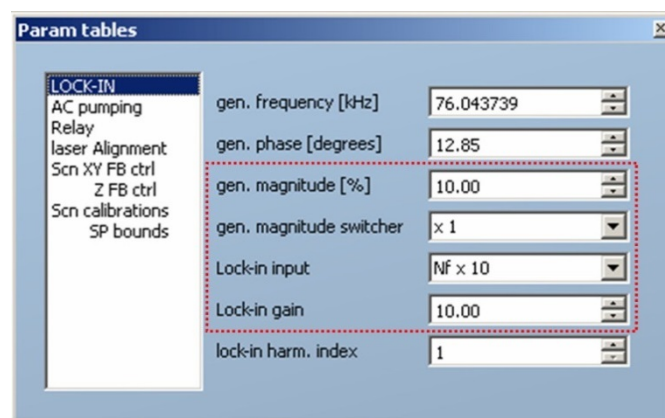


For applying voltage, set the piezo actuator = Gen.2. Supersaturated frequency response:



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.

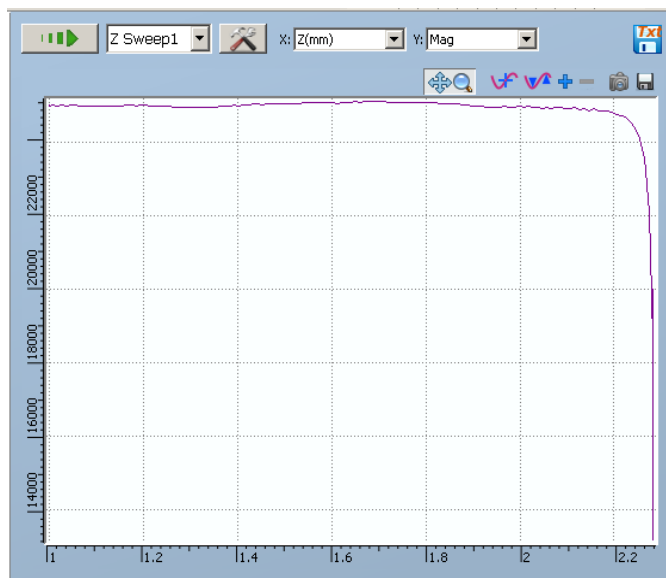


Figure 3.6.2.2 Mag(Z) curve – dependence of the probe amplitude on the vertical position of the scanner during the approach procedure

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.

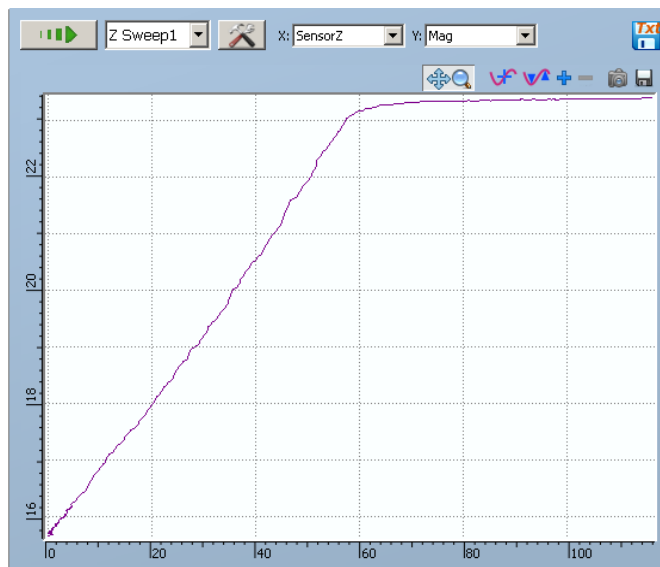


Figure 3.6.2.4 Mag(Z) dependence during the landing

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.

3.6.3. Feedback adjustment

When operating in the AC mode, the Z-axis feedback system maintains the probe oscillation amplitude at a constant level preset by the Sp parameter (setpoint), by moving the sample to and away from the probe.

For correct operation of feedback, you must properly set a feedback gain value. In case of insufficient gain, the system doesn't process changes of surface roughness correctly, and obtained images are blurred, and in case of too high gain, system's self-oscillations occur, so called parasitic oscillations or "generation", and periodical noise appears on images. The feedback gain value is set by the feedback gain parameters. The major parameter is the integral control parameter Gain (or I). In some cases, a proportional control parameter P can be additionally used.

After landing is performed using the Landing operation, minimal Gain is automatically set. Minimal Gain depends on the cantilever initial oscillation amplitude: if it is below 10 nm, Gain is set to 10 arbitrary units (a. u.); if it is above or equal to 10 nm, Gain is set to 20 a. u. Such value of the Gain parameter, on the one hand, is sufficient for maintaining the amplitude at the preset level in the absence of scanning, and on the other hand, it provides no generation as it is initially lower than values used during the scanning process.

Before switching to the scanning mode, you must adjust feedback: set optimal values of the feedback parameters. Feedback gain parameters depend on the cantilever parameters and measurement conditions and are determined experimentally.

The software provides the following options of feedback adjustment:

1. Automatic adjustment using the adjust gain button.
2. Manual adjustment using the oscilloscope.
3. Automatic adjustment using the meander.

4. Manual adjustment using the meander.

Automatic adjustment using the adjust gain button is a basic way of adjustment, and other methods are optional.

3.6.3.1 Automatic adjustment using the adjust gain button

This procedure of automatic adjustment is performed as follows:

The software increases the gain integral parameter Gain until the noise (rms) of the Mag signal is increased by half. The operating parameter Gain is set to be one half of the obtained value. In case of low cantilever resonance frequencies, a proportional parameter P is added, which is defined in the same way. Specific value P is equal to one hundredth from 1% of Gain.

The automatic adjustment is started by the adjust gain button on the AC mode control panel. After pressing the button, a message box appears where the maximum possible Gain for current conditions appears (fig. 3.6.3.1 to the left). Maximum Gain depends on the cantilever resonance frequency and initial amplitude and is defined under the empirical formula as $\text{amp}[\text{nm}] * 8 * (1 + (\text{freq}[\text{kHz}] - 100)/900)$. For example, for a cantilever with resonance frequency 280 kHz and amplitude 20 nm, the maximum Gain is 191 units, and with amplitude 100 nm – 959 units.

As soon as the adjustment is finished, the message box shows the parameters found (fig. 3.6.3.1 to the right). In case of successful adjustment, the Gain found is almost equal to the maximum one.

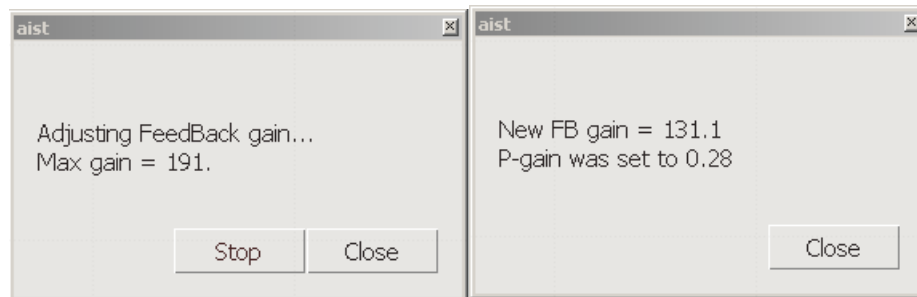


Figure 3.6.3.1 Message boxes of the adjust gain procedure.

3.6.3.2 Problems with automatic adjustment

Sometimes it occurs that the Gain found is substantially lower than the maximum possible one, or after starting the adjustment, the following message appears: **“Warning! Too big initial noise”**.

It may happen if the adjustment procedure was affected by an external noise- for example, someone slapped the door during the adjustment or knocked on the floor where the unit is installed, etc. In this case, you must restart the adjust gain procedure.

If after several tries of the auto-adjustment procedure, the Gain can't be adjusted, you must place the probe to another place of the sample, or increase the initial oscillation amplitude of the cantilever.

3.6.3.3 Manual adjustment using the oscilloscope

Proceed as follows:

- Set P to zero.
- Turn the oscilloscope on, and then switch the signals Mag and Phase on.
- When monitoring the signal in the oscilloscope, increase the Gain until parasitic oscillations occur (fig. 3.6.3.2).
- Set the Gain equal to half of the value, which is present when the parasitic oscillations occur.

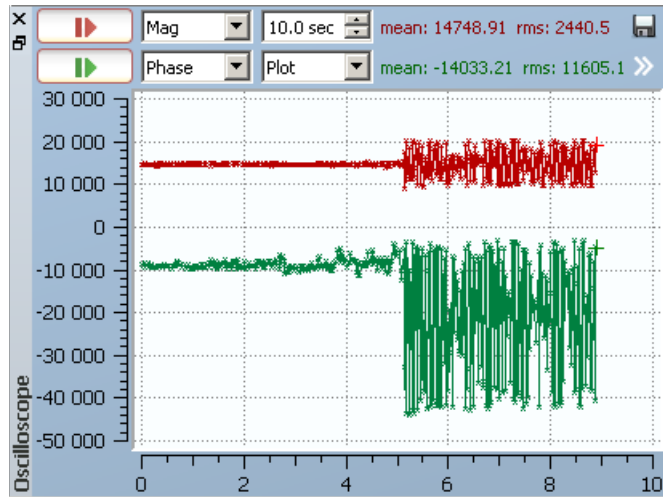


Figure 3.6.3.2 Generation in signals Mag and Phase in the oscilloscope.

NOTE: This adjustment method must be used carefully as in the “generation” mode, uncontrolled movement of the sample along the vertical axis occurs, which may result into the probe or sample damage.

3.6.3.4 Adjustment using the meander

One of the standard ways of feedback adjustment is to obtain a transient response of the system. For this purpose, a square wave signal (called “meander”) is fed to the feedback input, and how the behavior of the regulated signal depends on the gain parameters is monitored.

To make adjustment using the meander, use the feedback adjustment panel (fig. 3.6.3.4), which is opened using the FB adjustment button in the Scanner Ctrl window (fig. 3.6.3.3).

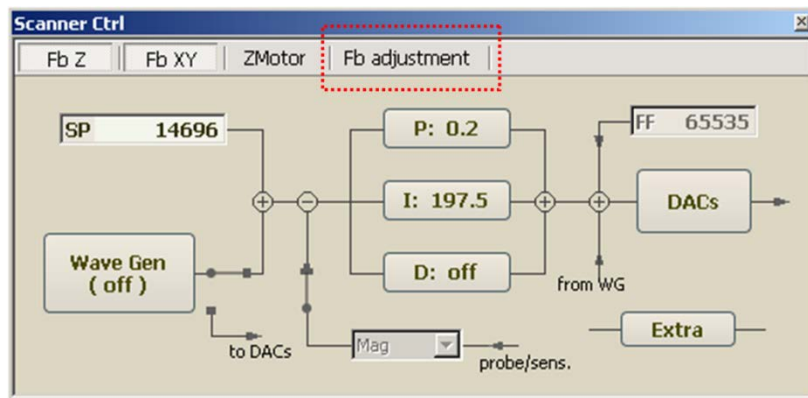


Figure 3.6.3.3 Location of the FB adjustment button in the Scanner Ctrl window.

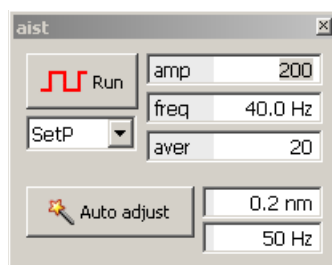


Figure 3.6.3.4 Feedback adjustment panel using the meander.

The panel includes the following elements:



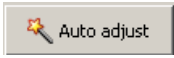
— meander input button

amp — meander amplitude, in ADC units

freq — meander frequency

aver — number of periods used for averaging

SetP/HvZ — selection where to feed the meander: to the feedback input / to the control voltage channel of the scanner's Z-axis

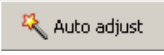


— automatic adjustment start button

0.2 nm — meander amplitude for automatic adjustment

50 Hz — meander frequency for automatic adjustment

3.6.3.5 Automatic adjustment using the meander

To perform automatic adjustment, use the  button.

The procedure obtains a transient response of the system for the meander fed to the feedback input and increases the integral parameter Gain until the response becomes strongly oscillating (fig. 3.6.3.5). After that, if it's necessary, the proportional gain parameter P is adjusted. To convert the meander amplitude from nanometers to ADC units, the meander is fed into the HvZ channel before adjustment, and its response is measured in ADC units. During the adjustment process, respective messages are shown in the Log window (fig. 3.6.3.6).

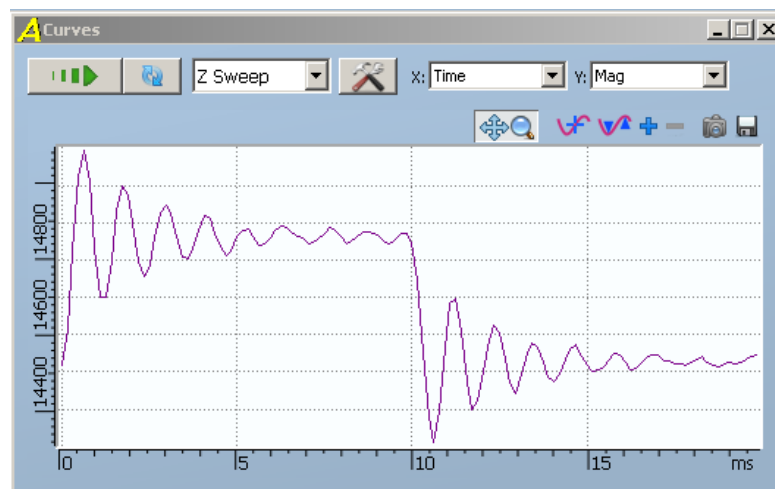


Figure 3.6.3.5 Transient response during automatic adjustment using the meander.

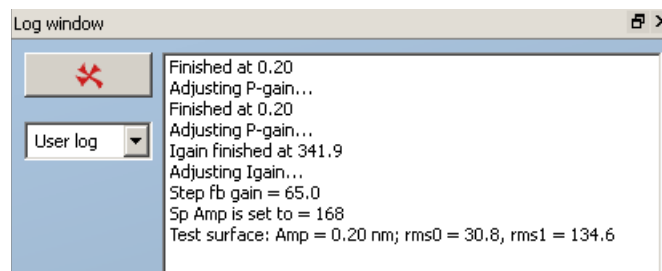


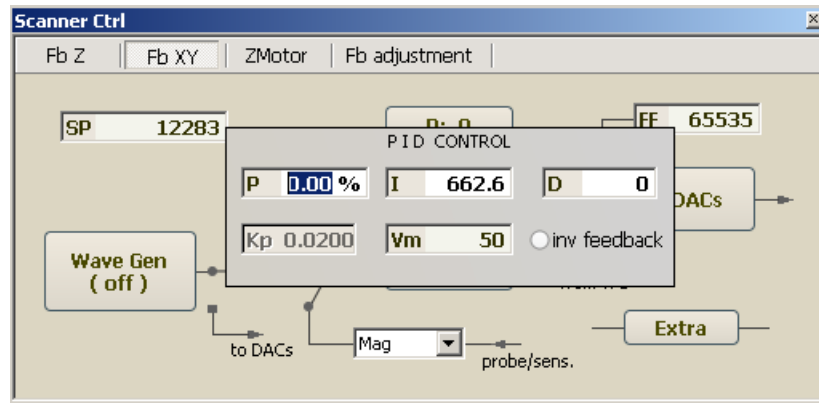
Figure 3.6.3.6 Auto adjust messages.


3.6.3.6 Manual adjustment using the meander

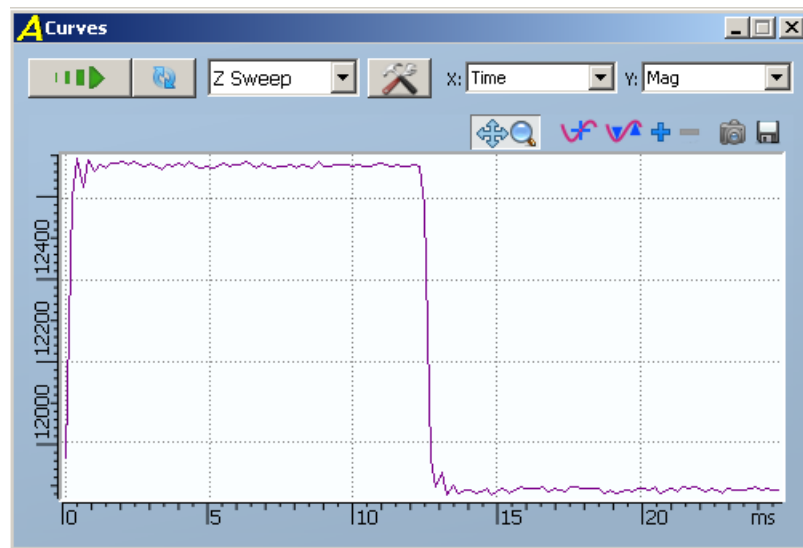
To perform manual adjustment use the feedback adjustment panel (Fig. 3.6.3.4).

Proceed as follows:

- Press P, I, D buttons of the Scanner Ctrl window to open the PID control panel:



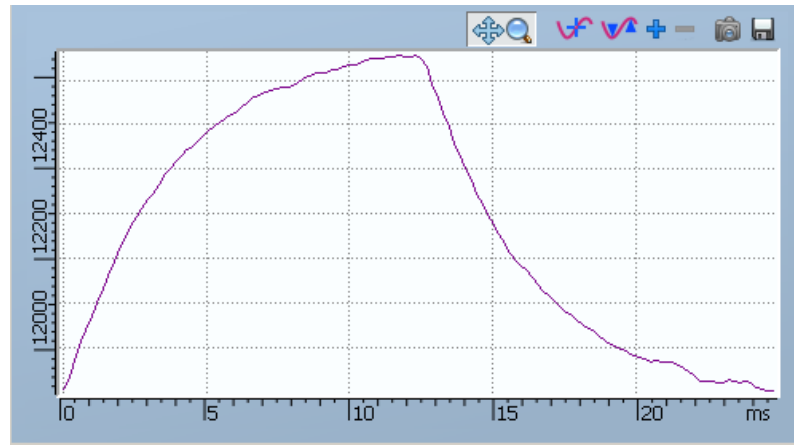
- Set P to zero.
- Turn on the meander feed-in using the  button on the feedback adjustment panel Fb Adjustment.
- Go to the Curves View window. This window shows the system transient response:



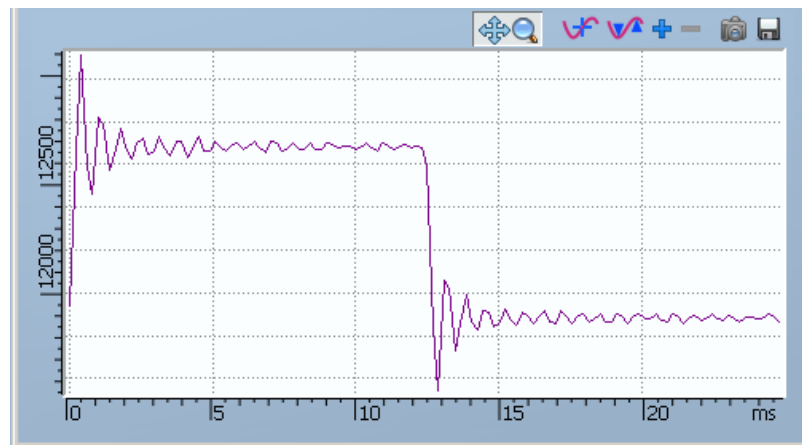
If the response is noisy and can be hardly read, increase the meander amplitude using the apm parameter, or increase averaging using the aver parameter.

The response shape depends on the feedback gain value.

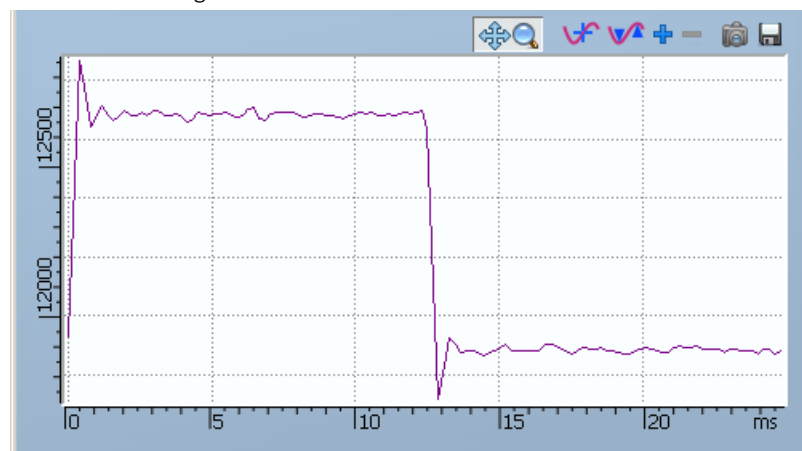
In case of insufficient integral parameter I, the response shape is almost triangular:



In case of too high I , you may see signal over-oscillations on steps:

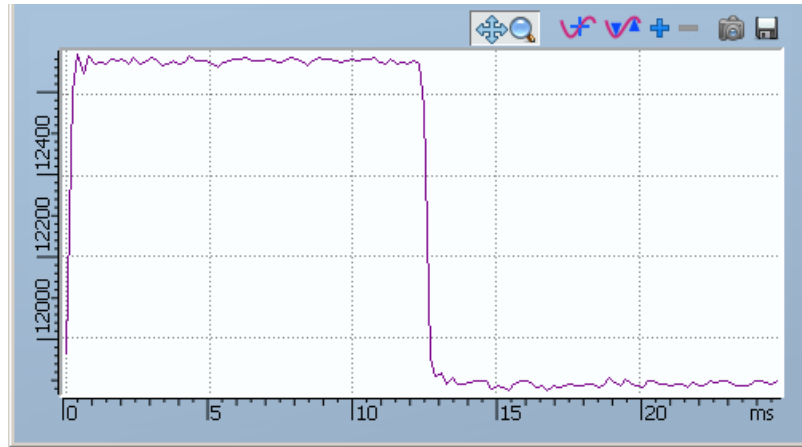


By changing I , try to achieve small overshooting:



After that, increase P until overshooting disappears. The proportional parameter P value is usually equal to several fractions of a percent from the integral parameter I value.

In case of correct adjustment, the signal must have a square shape:



After the adjustment is complete, turn off the square wave signal generator.

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 scanning any delicate samples 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).

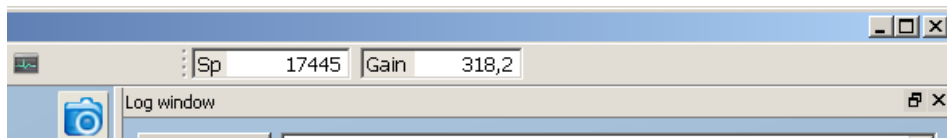


Figure 3.10.1 Fb- Toolbar1 toolbar

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).

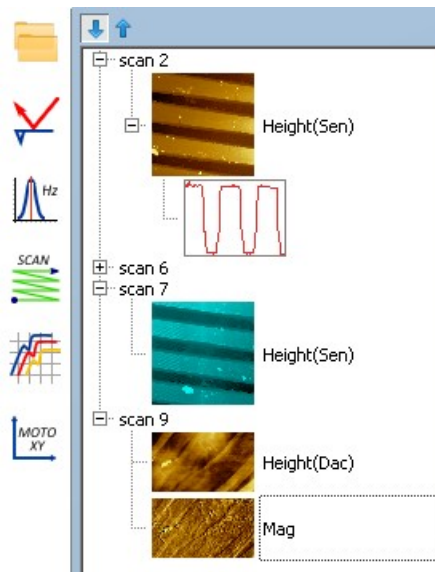


Figure 3.12.1 Data file structur

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.

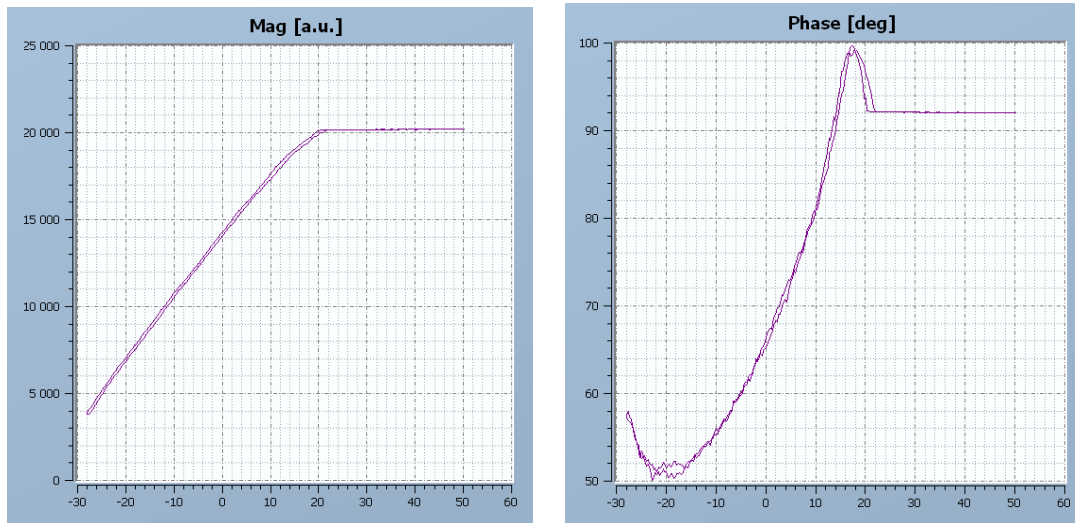


Figure 3.14.1 Amplitude and phase curves

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.

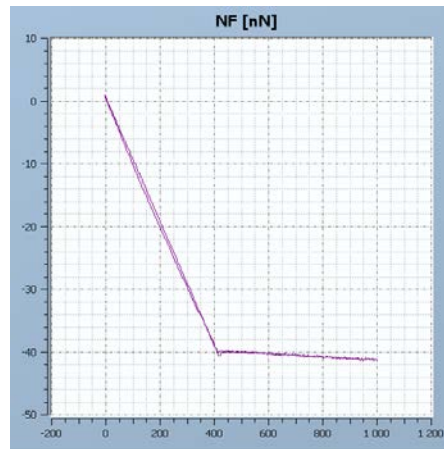


Figure 3.14.2 Force-distance curve

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".

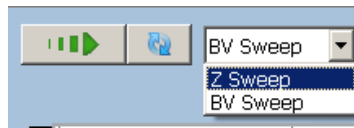




Figure 3.14.3 Spectroscopy type selection combo box in the Curves view window

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.

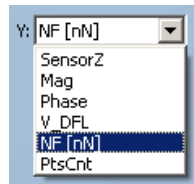



Figure 3.14.4 SPM spectroscopy Z Sweep signal list

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).

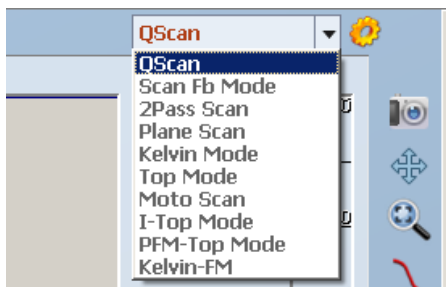


Figure 4.0.1 Available techniques the Scan menu.

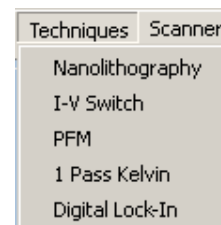


Figure 4.0.2 Available techniques in the drop-down list of Techniques drop-down list of the main window.

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 μm in the center of the initial 100 μm 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):

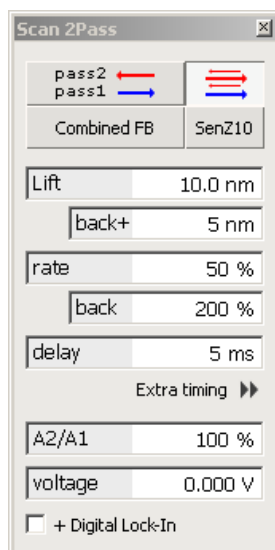


Figure 4.3.1 The 2Pass Scan panel for using two-pass techniques.

- 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.

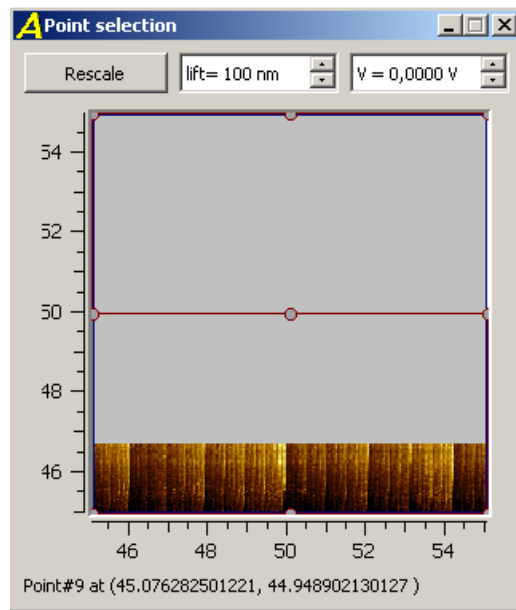



Figure 4.4.1 Surface estimation window

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

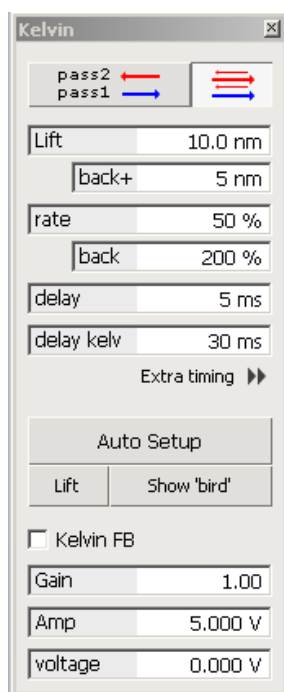


Figure 4.5.1 Kelvin mode control panel

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 $\text{mag} \cdot \sin$ feedback loop during the second pass. The feedback loop maintains 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 $\text{mag} \cdot \sin$.

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 there are two methods of Piezo Response:

1. Contact PFM mode
2. Point-by-point Measurement 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-Top mode. 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

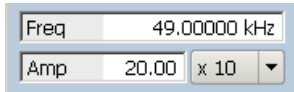
Open contact PFM mode control panel from Techniques menu:



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:



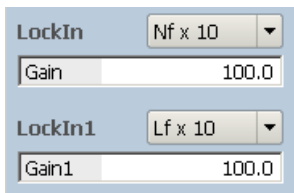
The voltage provided by built-in generator controller Gen.1, which is automatically connected to the probe (voltage electrode) when the contact PFM mode is on. For safety reasons, the control panel at the opening of the voltage amplitude of the modulation are always set to zero.

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);

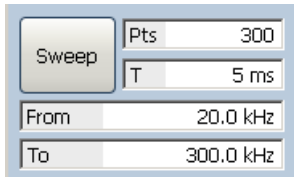
Parameters of the synchronous detector Lock-in1:

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:



The screenshot shows a control panel with a 'Sweep' button. To its right are two input fields: 'Pts' with the value '300' and 'T' with the value '5 ms'. Below these are two more input fields: 'From' with the value '20.0 kHz' and 'To' with the value '300.0 kHz'.

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:



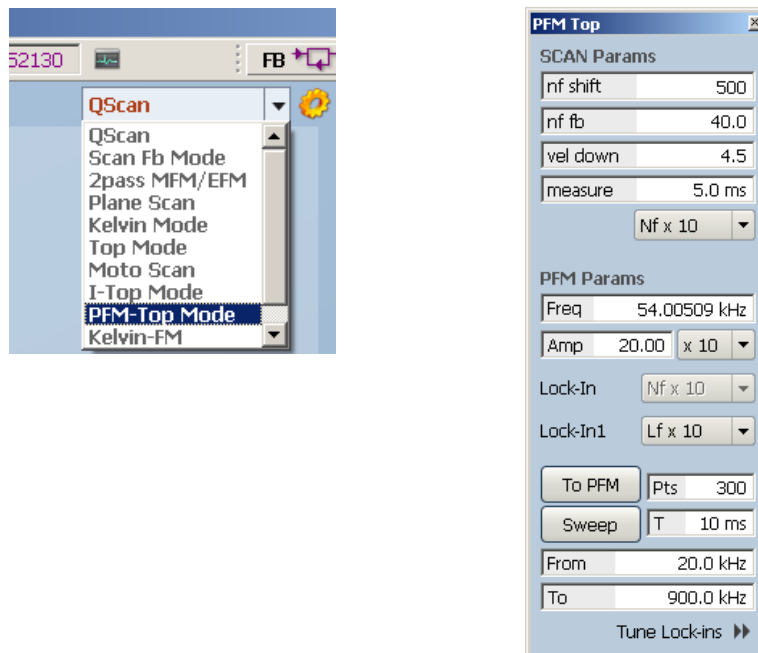
The screenshot shows a button labeled 'Tune Lock-ins' with a double arrow icon to its right.

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:



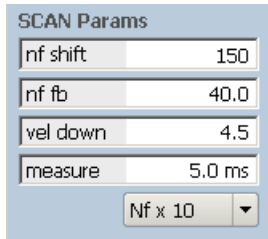
The figure consists of two screenshots. The left screenshot shows a list of scanning methods with 'PFM-Top Mode' highlighted. The right screenshot shows the 'PFM Top' control panel with the following parameters:

- SCAN Params:**
 - nf shift: 500
 - nf fb: 40.0
 - vel down: 4.5
 - measure: 5.0 ms
 - Nf x 10 (dropdown)
- PFM Params:**
 - Freq: 54.00509 kHz
 - Amp: 20.00 x 10 (dropdown)
 - Lock-In: Nf x 10 (dropdown)
 - Lock-In1: Lf x 10 (dropdown)
 - To PFM: Pts 300
 - Sweep: T 10 ms
 - From: 20.0 kHz
 - To: 900.0 kHz
 - Tune Lock-ins (button)

Figure 4.6.2 Contact PFM-Top mode control panel

The panel contains the following elements:

Scan Settings Scan Params:



| SCAN Params | |
|-------------|--------|
| nf shift | 150 |
| nf fb | 40.0 |
| vel down | 4.5 |
| measure | 5.0 ms |
| Nf x 10 ▼ | |

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 Nfx10 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:



| PFM Params |
|------------|
|------------|

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 turned 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.

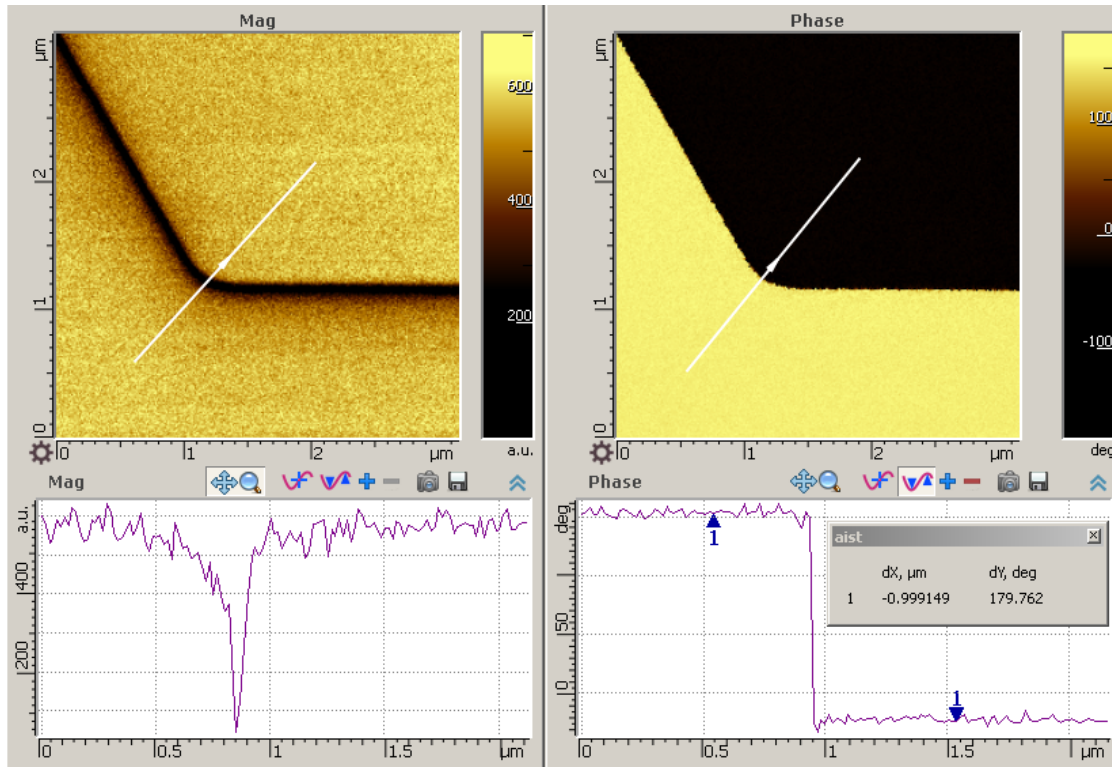


Figure 4.6.3 Images of the amplitude and phase of the normal vibrations of the cantilever obtained at the junction between the two domains on the sample of lithium niobate .

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.

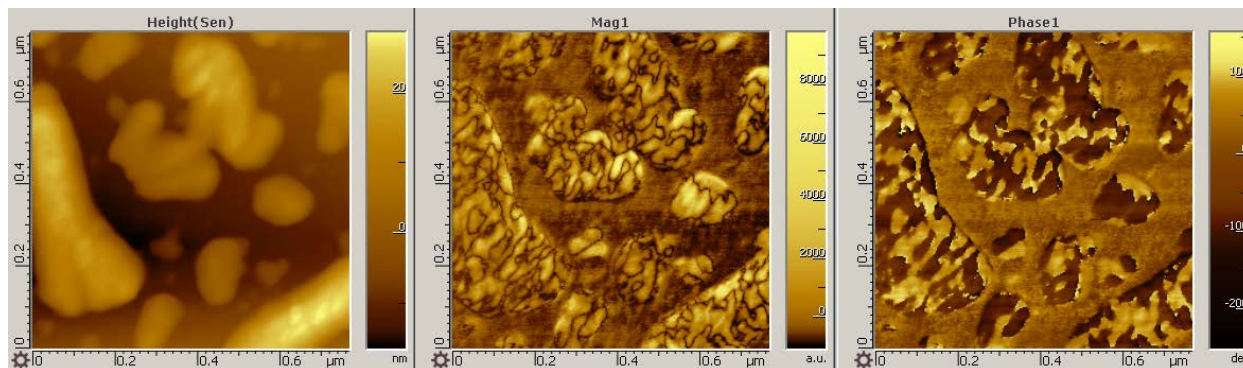


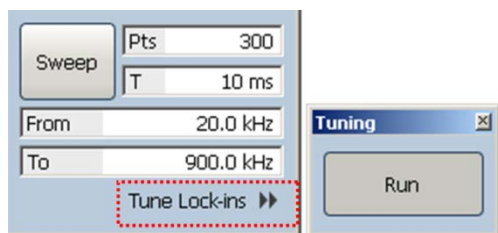
Figure 4.6.4 Topography and lateral piezoelectric response obtained in the PFM-Top fashion.

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”.

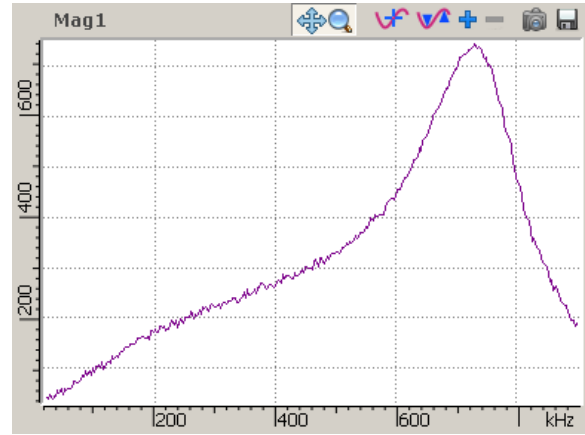
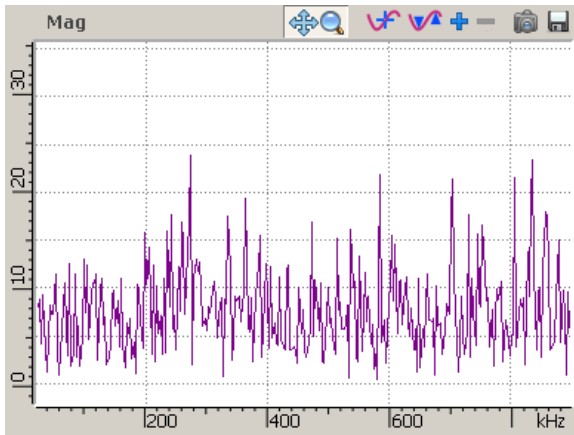
Setting synchronous detectors are required only once in this session. There is no need to repeat procedure after switching methods or change cantilever/sample.

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

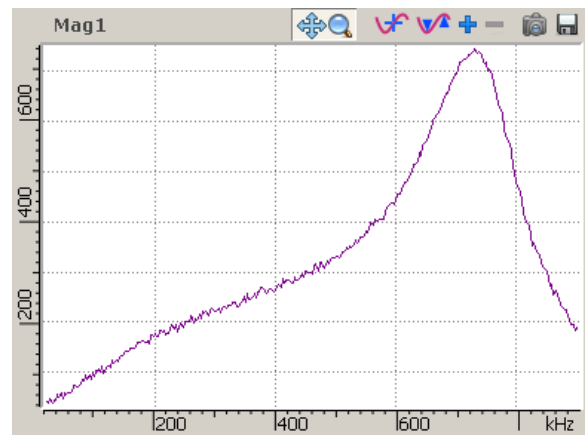
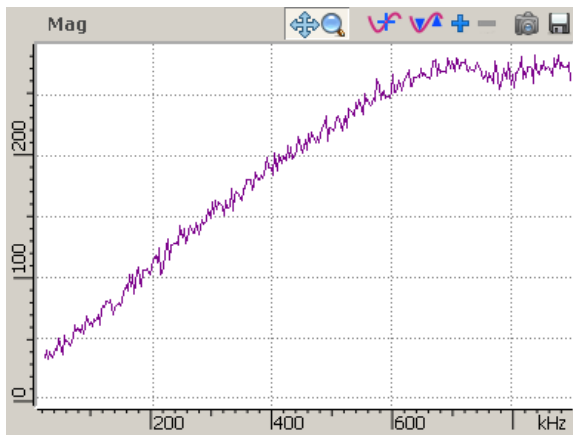
Do the following:

- 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.

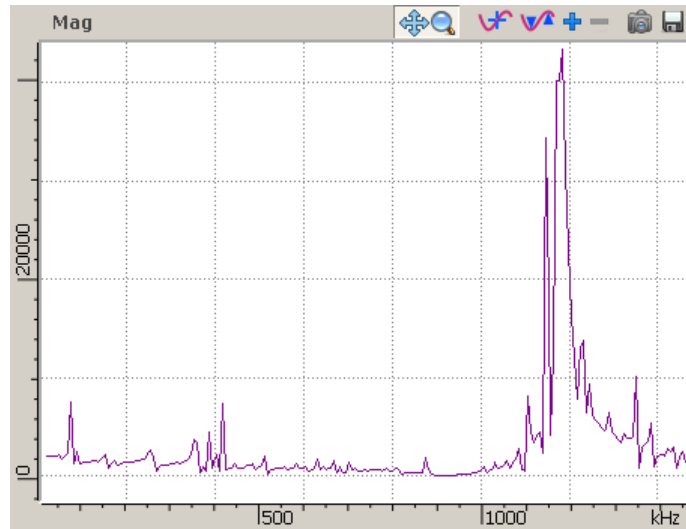
4.6.11 Frequency selection

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 is 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 are.

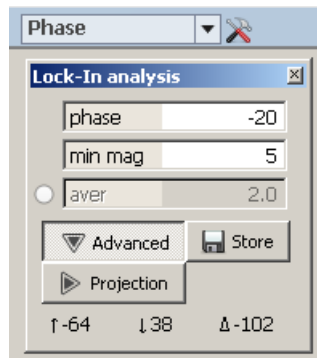
When installing the frequency resonance peak levels measured signals will be higher, but the resonance peak may change a little due to scanning due to changes in contact cantilever to surface according to the relief of the sample. This change leads to artifacts in the signals piezoelectric response.

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

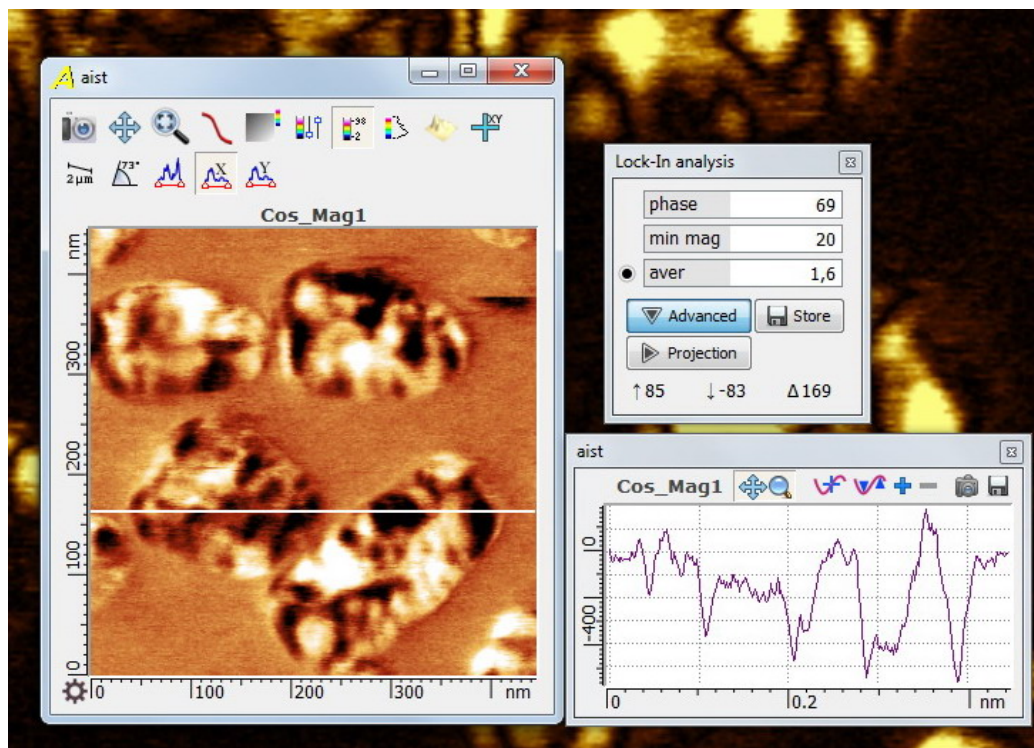
Threshold computation phase "min mag" is intended to reduce the phase noise in the areas in which no piezoelectric response. If the amplitude is less than the "min mag", then the phase at this point is not calculated and sets in 0 degrees.

4.6.12.3 Signal Filtering

For filtering signals used parameter "aver". Band filter is given by: $2 \text{ Pi Bandwidth} = 1 / (\text{aver} * T)$, 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 $2 \text{ Pi Bandwidth} = 1 / T$ (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

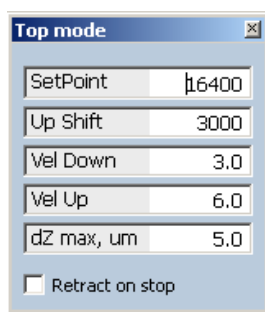
button "Projection" in a floating window displays the result of this processing raster - raster "Cos_Mag". This can be done both after and during the scan (when new data arrives from the controller the image will be updated) .

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.

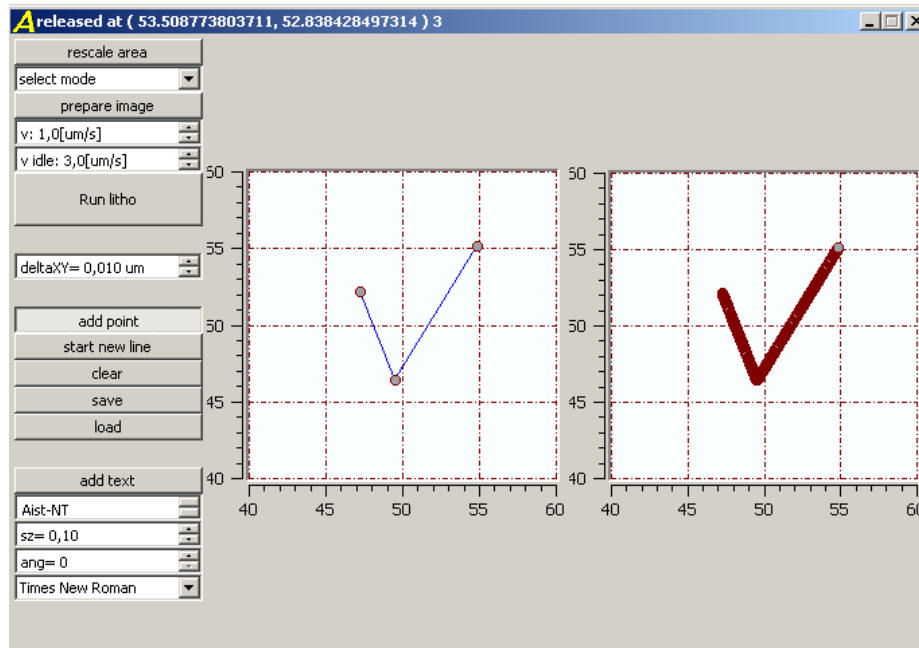


Figure 4.8.1 Nanolithography control panel for vector modes

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.

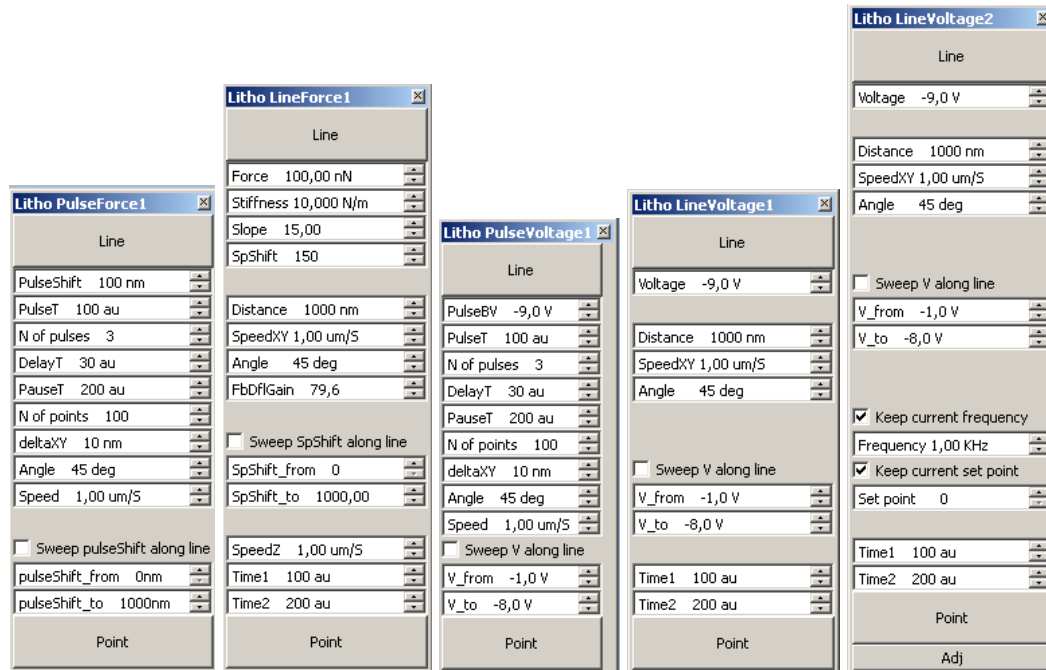


Figure 4.8.2 Settings panels for vector modes

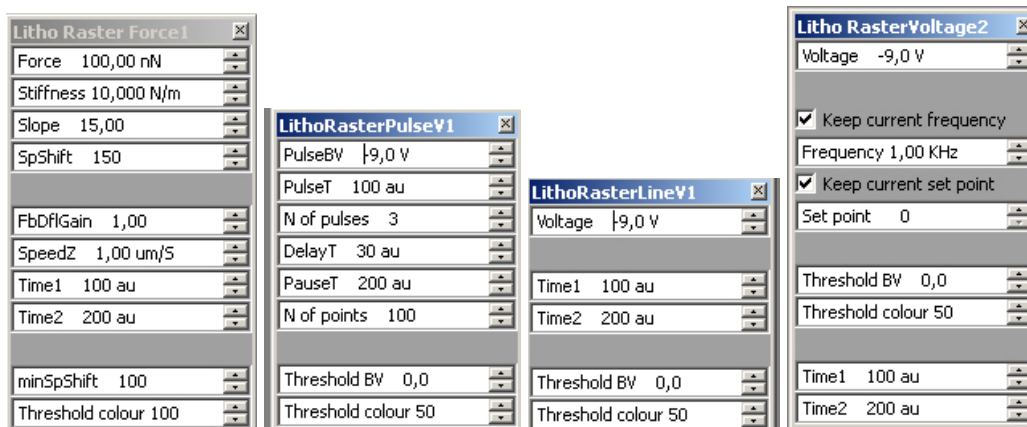


Figure 4.8.3 Settings panels for raster modes

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.

Threshold 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.

Threshold 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.

litho lines

Run X

X0 5.00 μm

Y0 5.00 μm

Length 5.0 μm

Step 1000 nm

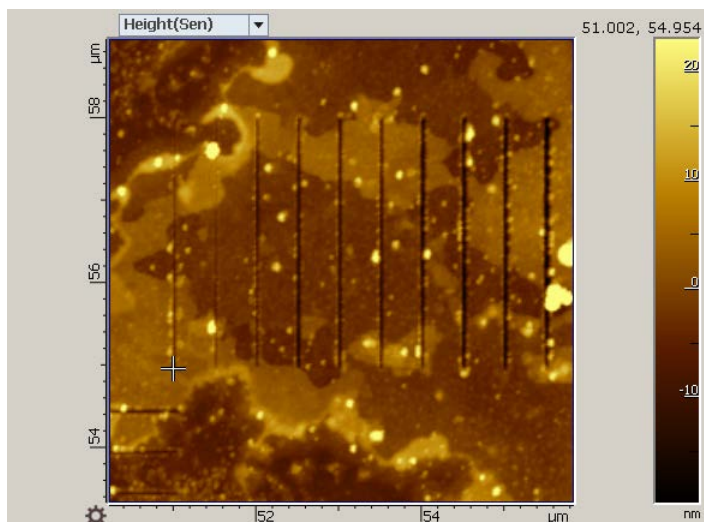
Rate 5 $\mu\text{m/s}$

Lines 10 10

Nf shift 100

Dshift 50 1

fbgain 500.0



Parameters:

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 of 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 params 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).

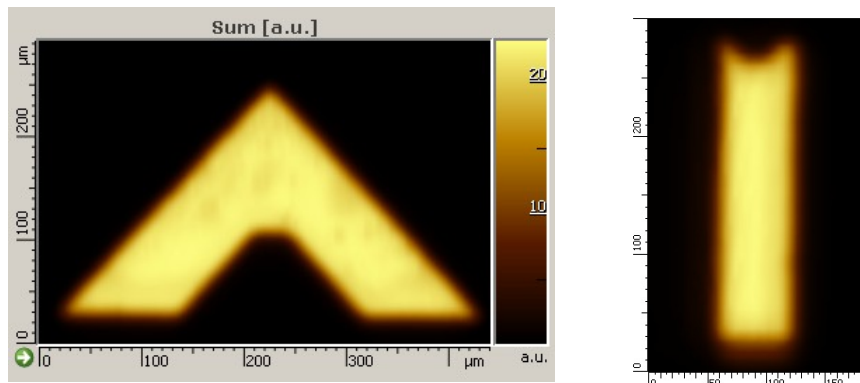





Figure 4.9.1 Photodiode signal distribution across the backside of the triangular and rectangular cantilevers obtained in the "Moto Scan" mode.

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.

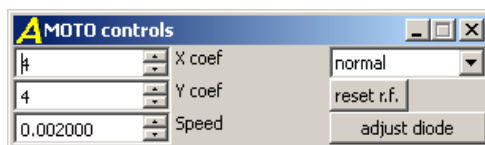


Figure 4.9.2 The MOTO controls panel to control the Moto Scan mode.

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 Obtaining atomic resolution in the AFM mode

Obtaining atomic resolution means getting images of the crystal lattice of the highly-oriented pyrolytic graphite (HOPG) or mica. Measurements are conducted by contact method with observing the crystal lattice in the signal of the lateral probe deviation. Obtaining atomic resolution is considered as a good test of system suitability for conducting measurements with high resolution.

4.10.1 Measurement conditions

1. The microscope must be isolated from any vibrations.
2. The microscope must be isolated from any wind/sound noise (hood-covered).
3. There must be no wind and sound noise in the room.
4. Temperature in the room must be stable.

4.10.2 Preparing a sample

Stick a piece of graphite to the sample holder using a superglue. Use of adhesive tape is not desirable as after sticking the sample may drift for quite a long time.

Just before the measurement start, clean the sample – peel off graphite surface layers using adhesive tape to obtain clean surface.

4.10.3 Estimation of noise conditions

Before starting to obtain atomic resolution, estimate the level of noise. It can be done using the graphite image by measuring a small smooth surface area.

Proceed as follows:

- Switch the device to the low voltage mode upon XY. To do it, in the Param tables window, in the Relay tab, set the parameter XY Voltage to LOW.
- Use non-contact cantilever.
- Adjust the device for semi-contact method of operation in the repulsion mode. To do it, set the initial oscillation amplitude at 50-100 nm. After approaching, set the setpoint Sp to be half of the initial oscillation amplitude. Adjust feedback.
- For scanning, use the Qscan mode.
- Obtain a test scan with the size of about 1 micron that is located in the center of the scanning area. Ensure that the device operates sustainably in the repulsion mode: phase signal is negative; no phase jumps to the area of negative values during the scanning process. If there are some phase jumps, either decrease the setpoint or increase the initial amplitude.
- On the test scan, find a smooth area with the size of 100-300 nm, with changes of surface roughness on steps of about 1-2 nm. Scan this area using the following parameters:

Scanning rate rate = 1 Hz;

Number of points = 256x256;

Measured signal Height (Dac).

As an example, figure 4.10.1 shows two images obtained by this method. When obtaining the first image, the microscope was placed on the laboratory table without any vibration protection, and without a hood. When obtaining the second image, the microscope was covered with a hood, and passive vibration protection was used.

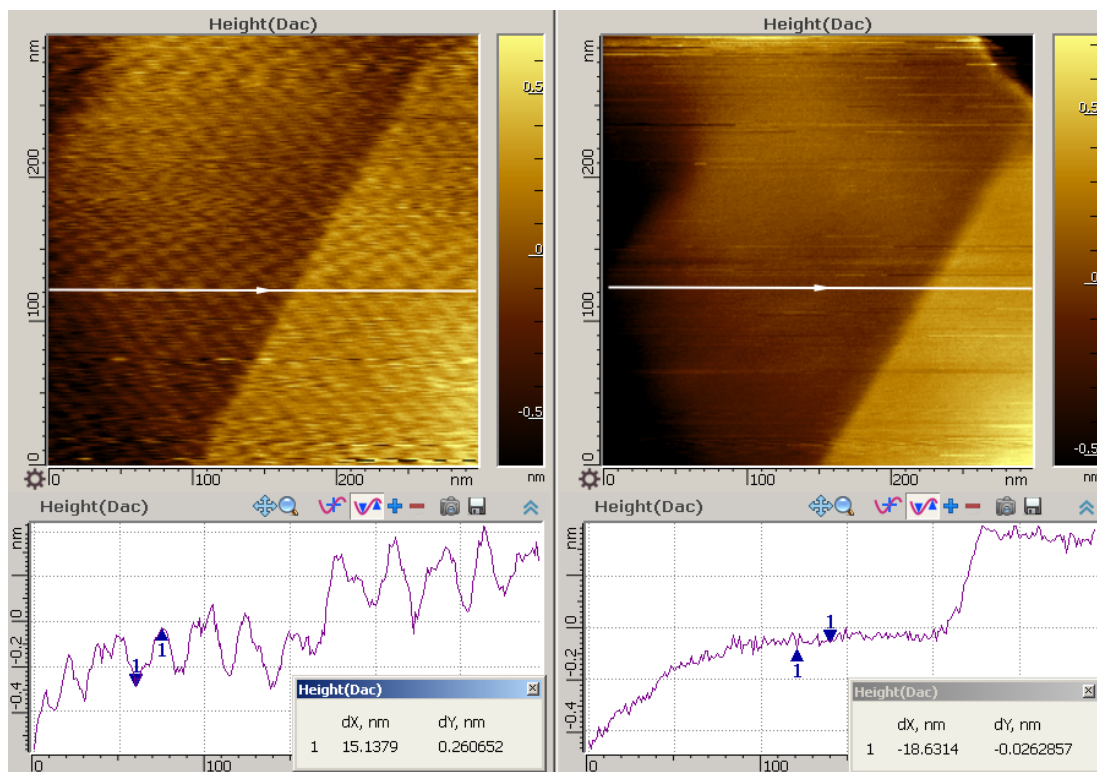


Figure 4.10.1 Images and profiles of graphite surface obtained on the SmartSPM microscope: on the left is the device on the laboratory table and on the right is the device with passive vibration protection.

The left image contains quasi-periodical noise with frequency 20 Hz, which amplitude differs depending on the line. From the profile given below the image, it can be seen that the changes within one oscillation was 0.2-0.3 nm. Probably, this frequency corresponds to the resonance frequency of the laboratory table where the microscope is located. The table transfers vibrations from the building to the device.

The right image is much better – no periodical noise, and the noise level is lower.

If you managed to obtain an imaged, which quality is close to the right image in fig. 4.10.1, then, most likely, you will succeed in obtaining atomic resolution. If you images look like those to the left in fig. 4.10.1, you have no chances to get atomic resolution.

4.10.4 Conducting measurements

If your estimation of noise conditions was positive, you may go on and proceed with obtaining atomic resolution.

Do as follows:

- In the View tap of the software main menu, open the following additional windows Macro executor, Filters, Params Tables.
- Move the sample away from the probe by pressing Retract.
- Switch to the contact mode.
- In the Filters window, set the digitization frequency ctrl rate at 50 kHz and load the signal table lf & bv:



- Approach the sample using the landing button and setting shift of 500 units.
- Using the multNfLf.lua macro, set the signal multiplier of the signal Lf to be equal to 10, and then to 100:



- In the oscilloscope, turn on the Lf signal and ensure that its value is close to zero.
- Set Gain to 100 units.
- To conduct scanning, go to the Qscan mode. Adjust the following settings:
 - Scanning mode: dacs.
 - Measured signal: Lf.
 - Scanning area – 3x3 nm. This area is located in the center of the full field.
 - Number of points: 256x256.
 - Scanning rate: 10 Hz.
- Start scanning in the continuous mode.

Most probably, after the scanning starts, strong drift will occur. Wait for the drift attenuation and do not stop scanning. In case of substantial drift, increase the scanning rate.

4.10.5 Setpoint selection

The contrast of the image obtained greatly depends on the level of force pressing the probe to the surface, which is adjusted by the setpoint Sp.

To select an optimal setpoint, change Sp during the scanning process. When Sp is decreased, shift (pressing force) is increased.

Figure 4.10.2 shows the image obtained at different values of the shift parameter. Initial shift was 200 units (lower quarter of the image), then the force was increased with increment 500 units, e.g. shift was 700, 1200, 1900 units.

It can be seen in the image that for 200 units, the lowest contrast was obtained, and the crystal lattice looks smoothly and without defects. When the force increases, so does the contrast. At 1900 units, a gap in the beginning of the line appears, and it relates to the fact that the probe is turned when the scanning direction is changed. The optimum force for this probe was 200-700 units.

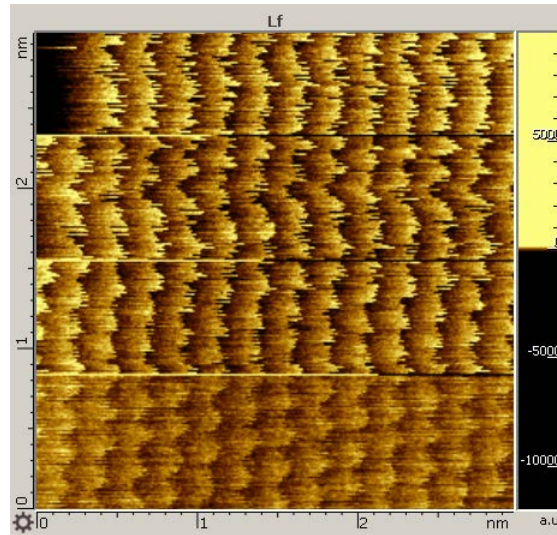


Figure 4.10.2 Selecting a setpoint during scanning.

To filter high-frequency noise, use the Lf filter in the Filters window.

In practice you should try to obtain an image that looks as the one shown in figure 4.10.3.

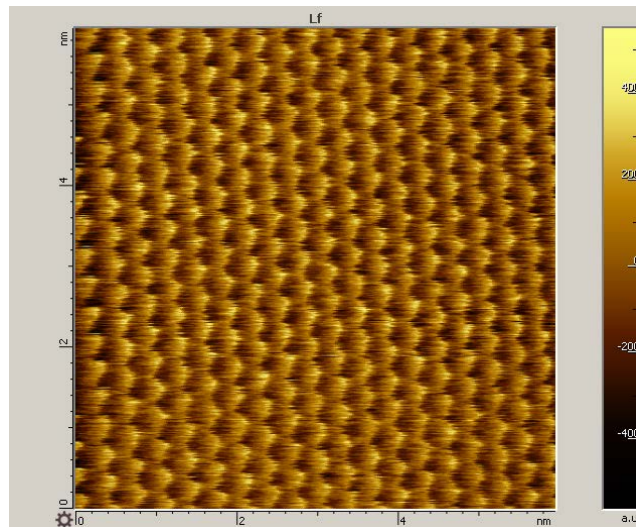
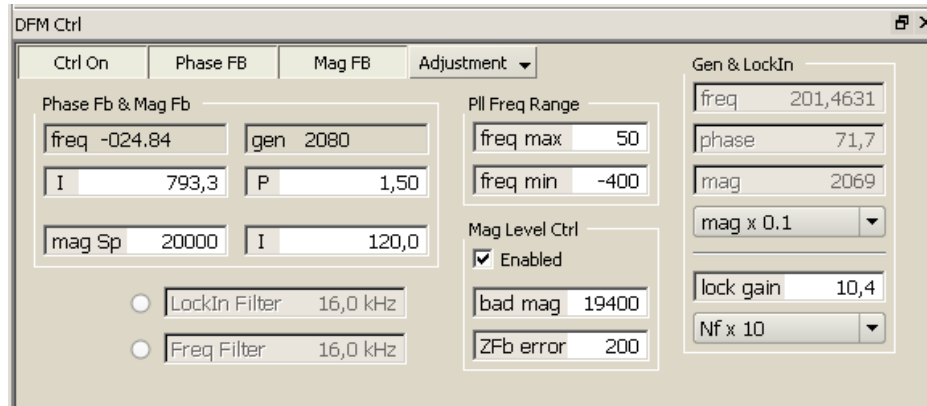


Figure 4.10.3 Atomic resolution obtained on highly-oriented pyrolytic graphite.

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.

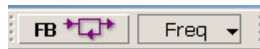
NOTE: Please learn more details about the feedback loop adjustment procedure in the Chapter 6 Fast Scanning, clause 6.2.

- 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.

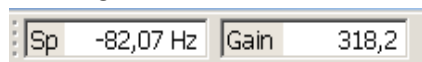
Now the oscillation amplitude and phase are maintained at the specified level by the corresponding feedback loops.

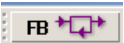
Land to the surface:

- 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

1.1 - 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.

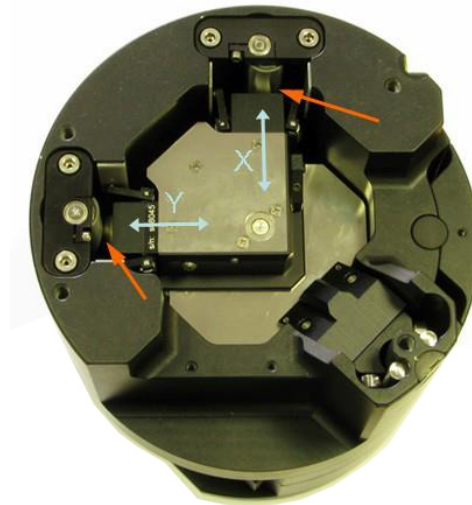



Figure 5.1 The photo shows the lower part of the SmartSPM microscope without the measuring head. Orange arrows show the location of the eccentrics of the motorized sample positioning. Blue arrows show the direction of the travel.

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.

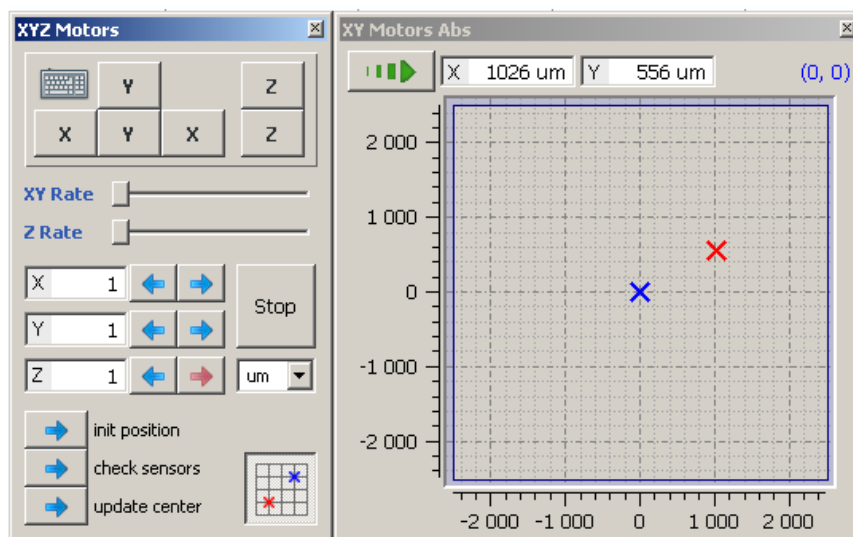


Figure 5.2 The XY motors window with the opened coordinate field


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.

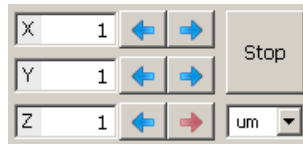
The program has an option to move the sample using keyboard that can be activated by clicking on the  button. X and Y buttons correspond to the arrows on the keyboard, the Z buttons correspond the "+" and "Enter", located on the right side of the keyboard.

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  **init position** 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

In this document, “fast scanning” means scanning with maximum possible rate in given conditions. The maximum scanning rate depends on many factors, particularly, on the sample topography, scanning area size, and cantilever parameters. Depending on the conditions, the maximum rate can be rather low (0.5 -1 Hz), or really high (50-80 Hz).

The maximum scanning rate is determined by the rate of feedback operation upon the Z axis, e.g. by the rate, at which the scanner processes changes of surface roughness on the sample when scanning. The rate of feedback operation upon the Z axis, in its turn, is correlated with the feedback gain parameter Gain – the higher it is the quicker is the feedback.

Gain depends on the cantilever resonance frequency – a cantilever with high resonance frequency has higher Gain. In the semi-contact mode, Gain also depends on the initial oscillation amplitude of the cantilever – higher Gain is obtained at higher oscillations.

By default the device is adjusted for the “standard scanning”. “Standard scanning” means scanning of small fields (1-10 microns) with small rate (1-2 Hz). The feedback band upon the Z axis is limited by the filter to provide the minimal level of noise. Limitation of the feedback band leads to the decrease of the maximum value of the feedback gain parameter Gain, which decreases the maximum feedback operation rate.

To switch to the fast scanning, change the device settings. To do it, use the FastScan.lua macro, then turn on the Fast mode (see 6.1 below). The macro deactivates all feedback filters, increases signal digitization frequency and performs a number of switches that are aimed to provide the maximum scanning rate.

After the settings for the fast scanning have been set, adjust feedback upon the Z axis using the manual adjustment procedure described in 3.6.3.6. The obtained feedback gain parameter Gain will be probably higher than the parameter found when making adjustments for the standard scanning.

For scanning, use the QScan mode. At first, start the scanning at a small rate (1-2 Hz). Pay attention to the actual scanning rate that is shown on the indicator located in the left lower corner of the scanning window in the QScan mode (fig. 6.1). When switching to the Fast settings, the Adaptive scanning mode is on, where the scanning rate is changed when scanning, depending on the feedback error value. This is why the actual rate is slightly less than the set rate.

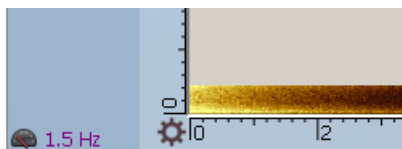


Figure 6.1 Actual scanning rate indicator.

Turn on the cyclic scanning. Smoothly increase the rate and keep watching the image.

When determining the maximum possible rate, you must be guided by the quality of image obtained. The signs of too high rate are:

1. Regular noise appears on images (“noisiness”), which is absent at lower rates. Such noise can be better seen on the feedback error signals (Mag, Nf) or on the phase signal.
2. When increasing the set rate, the actual rate doesn’t increase.

As an example we give some surface images obtained with different rates (fig. 6.2). Both images are obtained using settings for the fast scanning. Non-contact cantilever fpN10 with resonance frequency 324 kHz was used. Initial cantilever oscillation amplitude was set to 100 nm. The left image was obtained at rate 18 Hz, the right one – at rate 30 Hz.

You may see the following distortions in the right image:

1. It can be seen on the topography that separately located particles are blurred – stretched along the X axis.
2. On the image of the Mag signal, it can be seen that in general, the image is less distinct than the right one. Also, over-oscillations are seen along the X axis.
3. The cross section taken along one of the lines shows that in the right image, the error value during the processing of topography irregularities is several times higher than in the left image.

Thus, the image analysis shows that for these conditions, the rate 18 Hz is the most optimal rate whereas 30 Hz rate is too high.

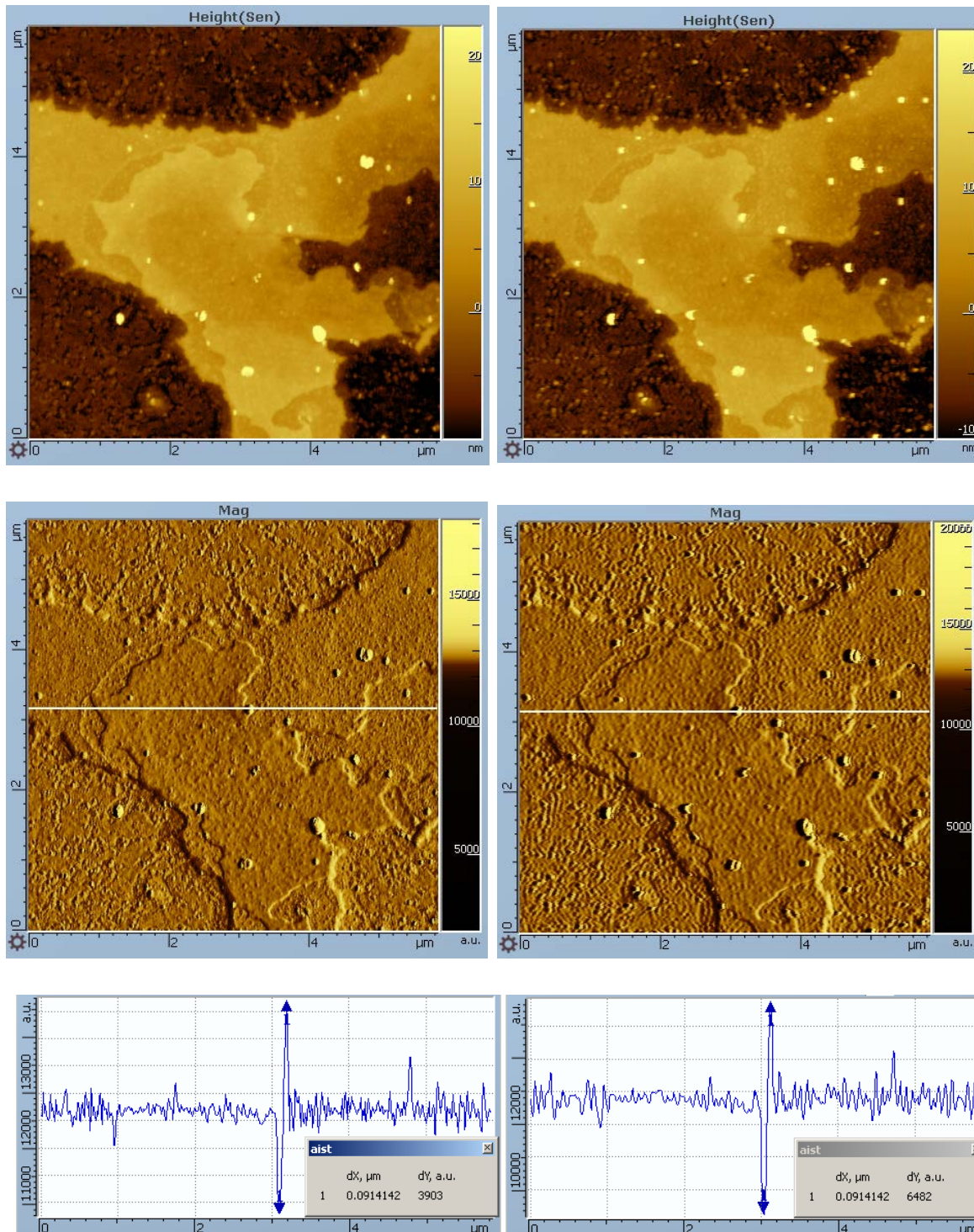

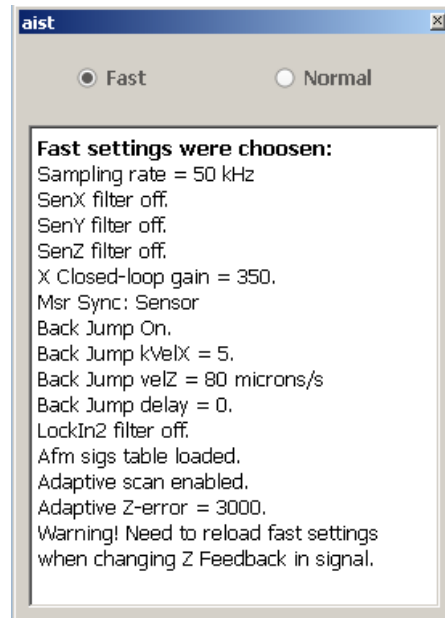


Figure 6.2 Surface images obtained with different rates. Both images are obtained using settings for fast scanning. The left image was obtained at rate 18 Hz, the right one – at rate 30 Hz.

6.1 Switching to the fast scanning mode

To switch to the fast scanning mode, use the FastScan.lua macro. To start the macro, open the Macro Executor window, select the macro from the list and press  button.

After starting the macro, turn on the Fast mode:



When switching to the Fast mode, the following changes occur:

1. Increased rate of signal digitization is set:
Sampling rate = 50 kHz
2. Maximum feedback band for scanner sensors is set – filters of corresponding signals are off:
SenX filter off
SenY filter off
SenZ filter off
3. Gain parameter is increased in the feedback loop of the scanner sensor along the fast scanning direction:
X Closed-loop gain = 350
4. Scanning synchronization is switched to the mode “upon scanner sensors”:
Msr Sync: Sensor
5. Back Jump mode is on; its parameters are set so that the quickest possible return to the beginning of the next line is provided:
Back Jump On.
Back Jump kVelX = 5
Back Jump velZ = 80 microns/s
Back Jump Delay = 0
6. Feedback band along the Z axis is expanded – the filter of the signal applied to the feedback input is off:
LockIn2 filter off – for AC mode
Nf filter off – for Contact mode

7. A signal table is loaded where a signal applied to the feedback input is digitized each cycle:
 - Afm sigs table loaded – for AC mode
 - Afm sigs table 2 loaded – for Contact mode
8. Adaptive scanning mode is on. The value of the error when the rate is reduced, is reset:
 - Adaptive scan enabled.
 - Adaptive Z-error = 3000 - for AC mode
 - Adaptive Z-error = 1000 – for Contact mode

When selecting the Normal mode, the settings corresponding to the standard scanning, e.g. scanning at rate 1-2 Hz, are set.

ATTENTION! To avoid generation, before switching from fast scanning to standard scanning, set the proportional feedback gain parameter $P=0$, and integral parameter $I=20$. After switching, re-adjust feedback.

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 sample.



The sample holder with spring for electrical contact to the

Figure 7.1.1 A set of hardware required for Conductive AFM

7.1.2 Installing the conductive unit

Install the conductive unit on the HE001 measuring head to work in the conductive AFM mode:

- Switch off the controller.
- Drive two special screws into the holes in the measuring head (fig. 7.1.2, 7.1.3).
- Install the conductive unit onto these two special screws and fix it using the fixing screws. Position of the fixing screws indicated by the arrows in fig. 7.1.4.
- Plug the conductive unit cable into the "Head" connector on the controller (position 1 in figure 7.1.5).
- Connect the measuring head cable to the connector of the conductive unit cable (position 2 in figure 7.1.5).



Figure 7.1.2 Screws to install the unit

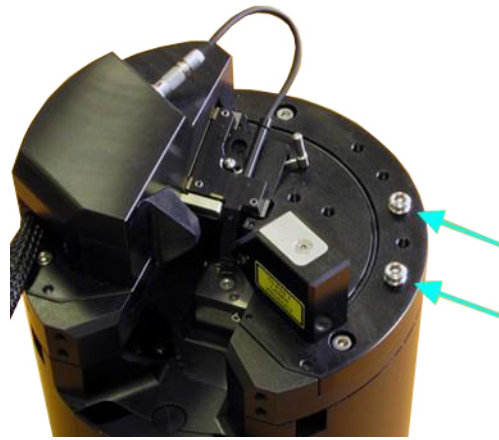


Figure 7.1.3 Position of the screws

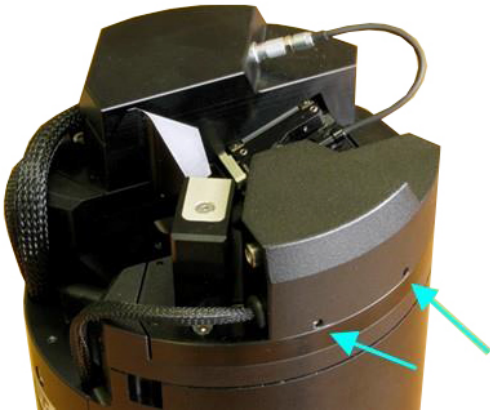


Figure 7.1.4 Position of the fixing screws

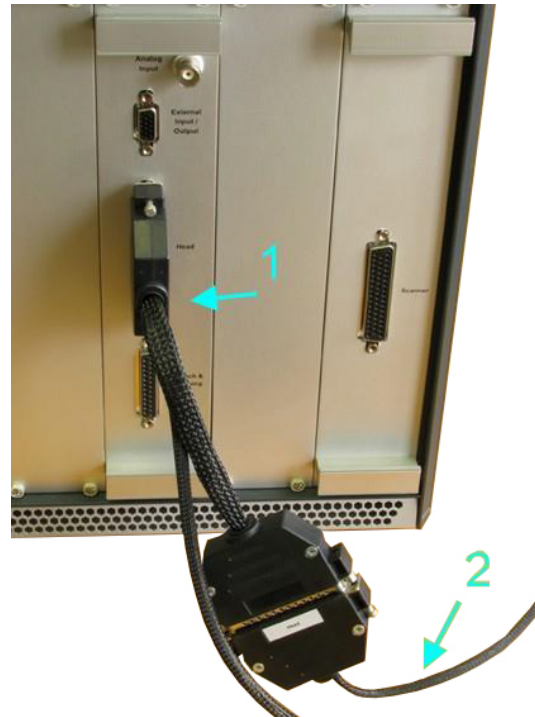


Figure 7.1.5 Cable connection of the conductive unit to the controller.

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.

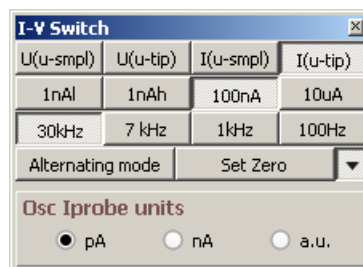


Figure 7.1.7 Conductive unit control panel

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-s mpl – bias voltage is applied to the sample, the probe is grounded

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

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

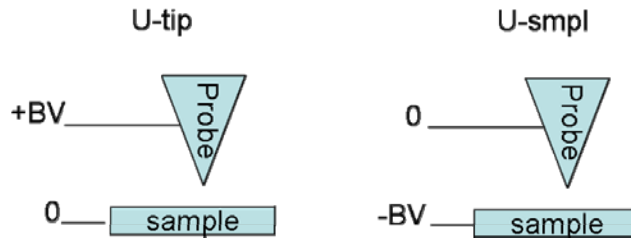


Figure 7.1.8 The schemes of applying bias voltages in U-tip and U-smpl modes.

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.

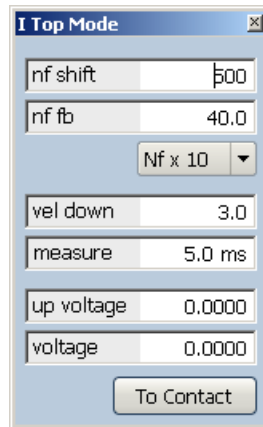
Use the BV Sweep mode in the Curves View window to obtain volt-ampere curves.

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

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 probe 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 (below) 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.

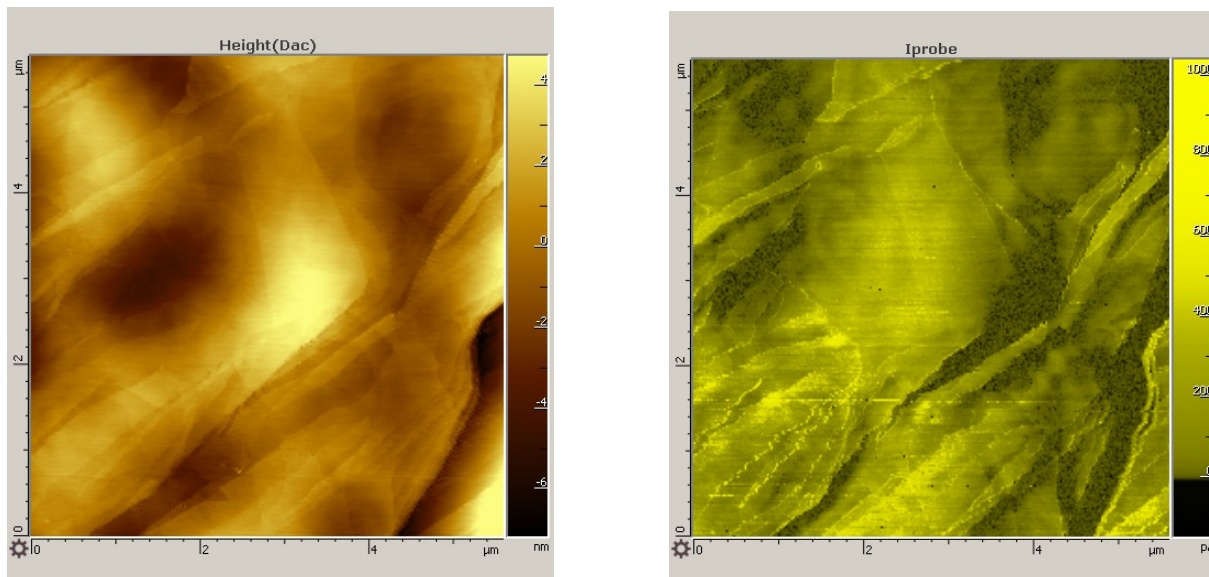
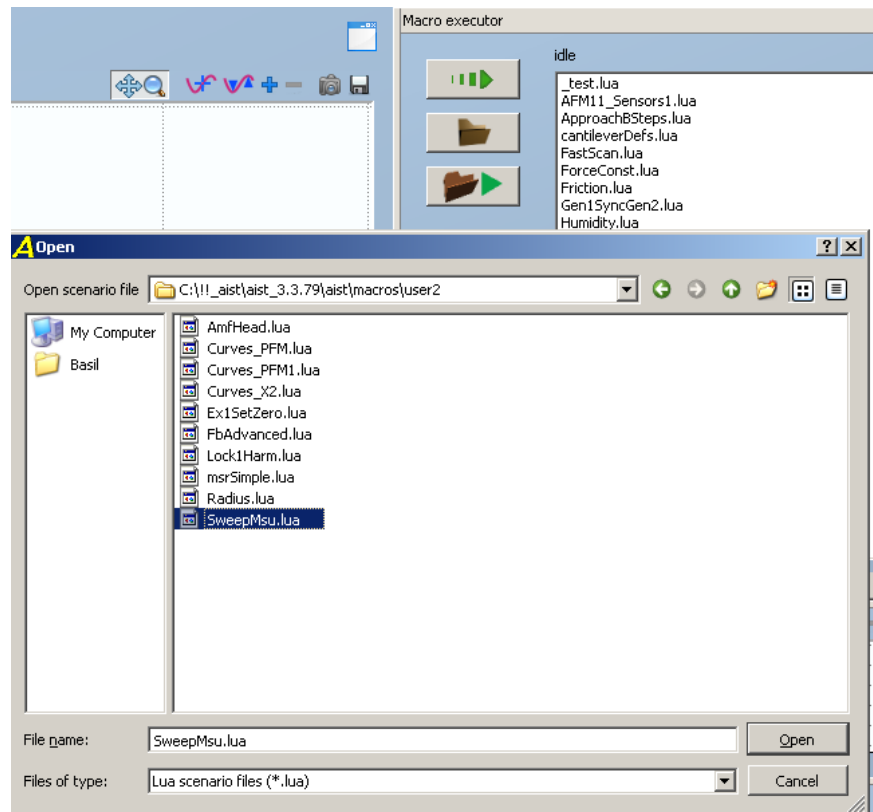


Figure 7.1.9 Topography (left) and spatial map of the electrical conductivity (right) of HOPG. The topography image was obtained beforehand in the Qscan semicontact AFM mode. The electrical conductivity map was obtained in the I-top mode. Conditions: fpN10Pt cantilever, Nf shift = 1000, voltage = 5 mV.

7.1.6 Getting series of volt-ampere characteristics

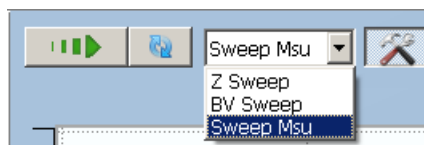
To remove the volt-ampere characteristics (VAC) on the grid, or with sweep by setpoint use the macro SweepMSU.

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:

Sweep Msu

Iprobe

From -0.005 V

To 0.005 V

points 200

passes 2

pointT 2.00 ms

delay 30 %

turn delay 0.0 ms

many delay 0.0 ms

fbXY 10 %

☐ From current V

☐ Auto save

☐ Save file

☐ Map XY

nx 3 ny 3

xy step 1.0 μm

☐ Map setpoint

step 50 pA

N 5

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 scanner, 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):



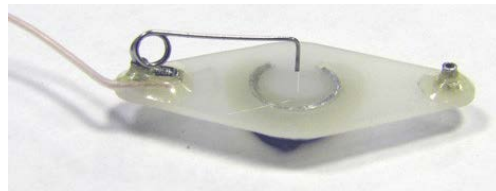
STM tipholder platform



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.

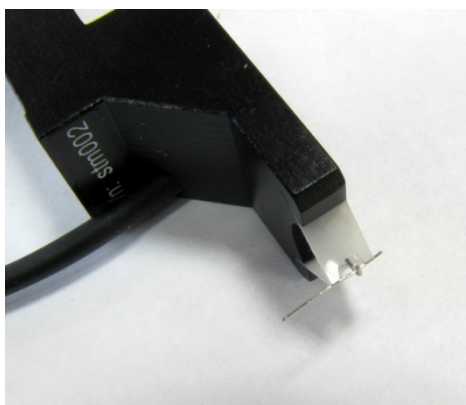


Figure 7.2.3 STM probe installation

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.

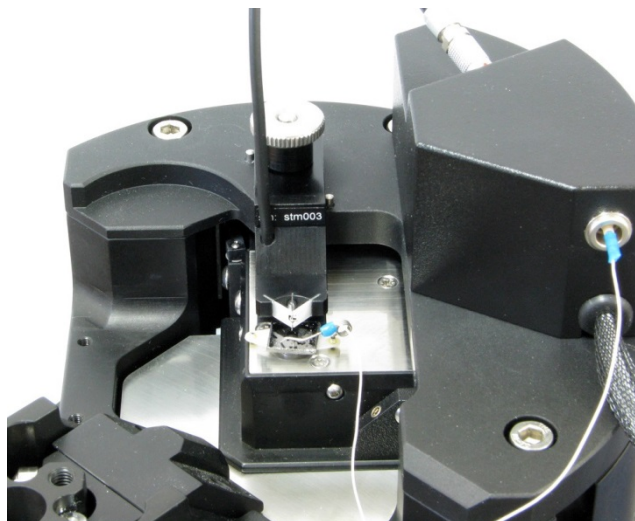


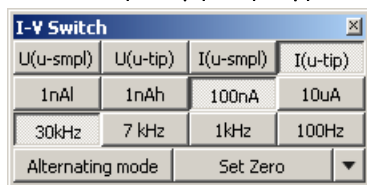
Figure 7.2.4 STM tipholder in its working position

7.2.2 Measurements in the STM mode

ATTENTION! Before starting measurements, check the following:

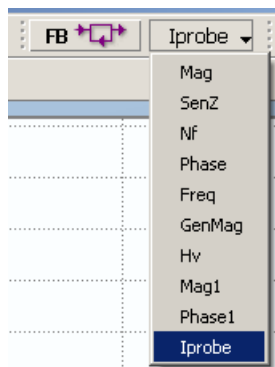
1. Spring clip touches your sample;
2. The spring clip wire is connected to the conductive unit;
3. The STM tipholder is connected to the conductive unit. Do the following:
 1. Open the Oscilloscope window and select the Iprobe signal.
 2. Select I-V Switch from the Techniques menu to open the conductive 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.

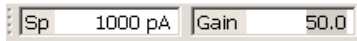
3. Set the zero current by pressing Set Zero.
4. Open the Fb ToolBar2 control panel. To open the list of control panels, right click anywhere on the empty gray background of the top part of the main program window. Select Iprobe as the feedback signal:



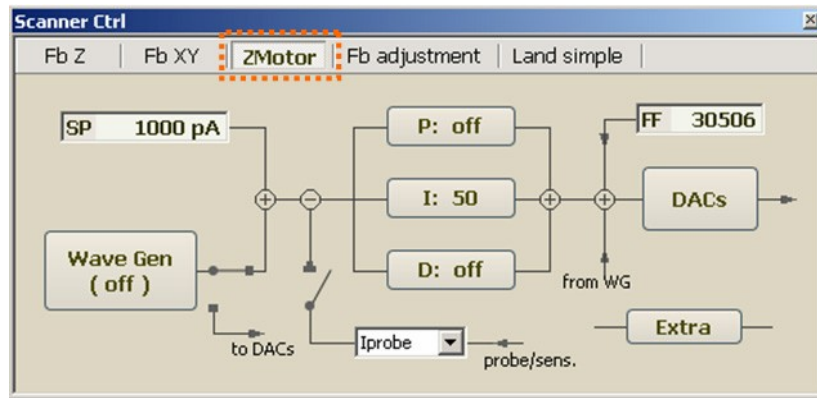
5. Open the Bias V control panel. Set bias voltage to 0.1V:



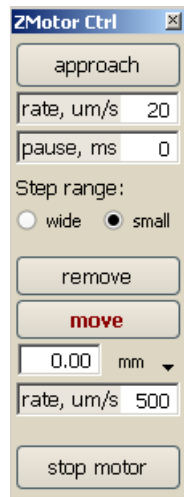
6. Open the Fb ToolBar1 control panel and set setpoint Sp = 1000 pA, feedback gain Gain = 200:



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:

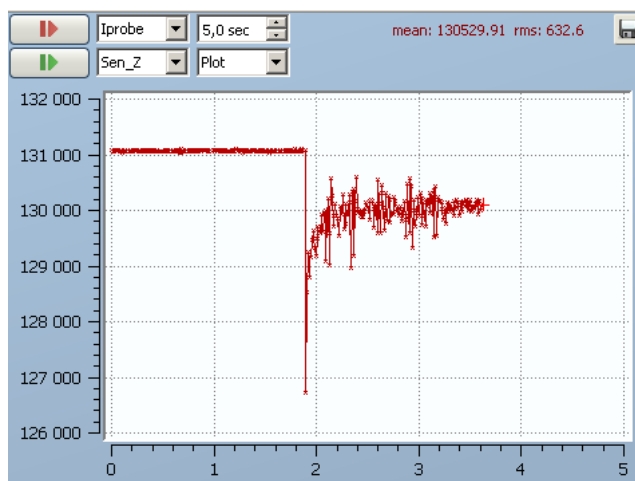


9. Set the rate parameter (approach rate) to 10-20 microns per second.

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 μm in the center of the whole scanning range
- Signals: Height(Dac), Iprobe
- Number of points: 256x256
- Scanning speed: 1 Hz

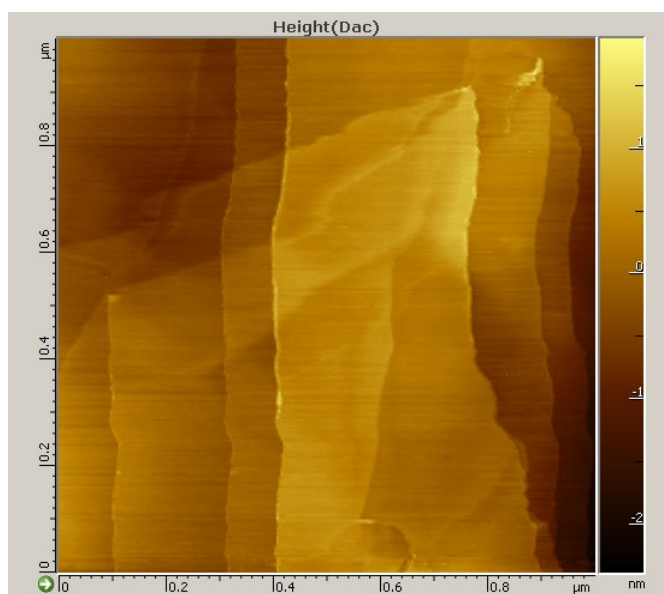


Figure 7.2.5 Typical image of graphite surface in STM mode.

7.2.3 Obtaining atomic resolution images in STM mode

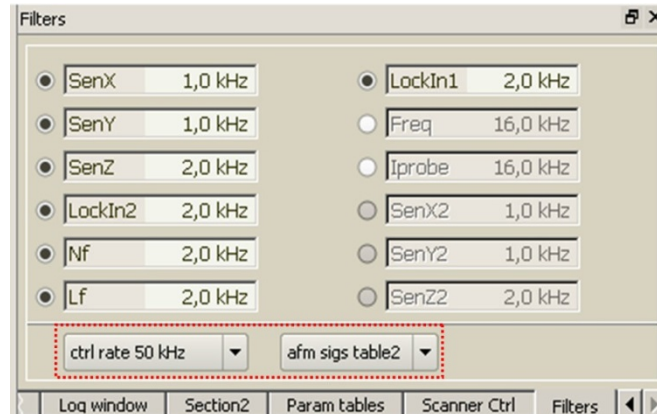
Use a piece of highly oriented pyrolytic graphite (HOPG) to obtain the atomic resolution imaging of its crystalline structure. How to prepare the sample and probe is described above.

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.

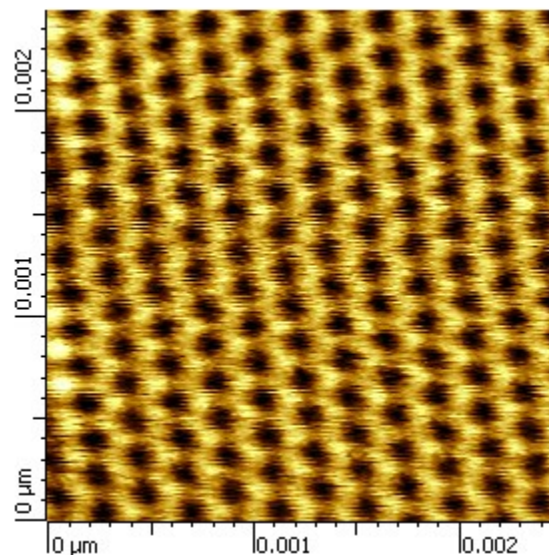
Measurement procedure:

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.

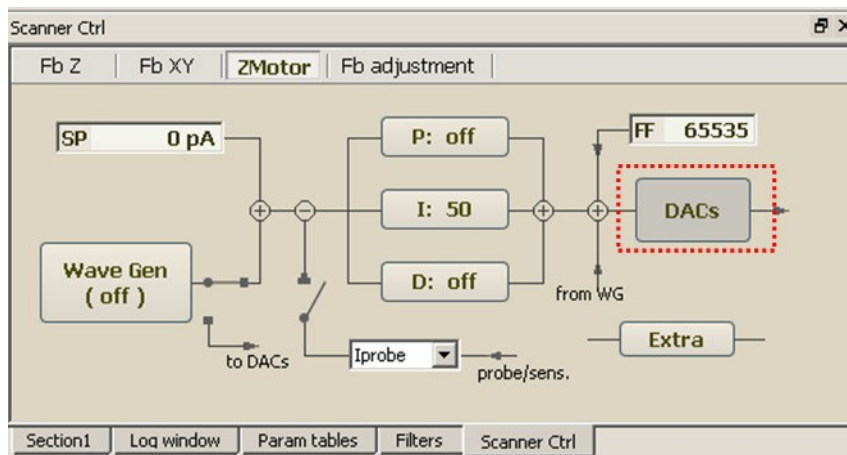
Run the scanning process. Right after you run the scanning process, most likely some drift will be observed. That's why we recommend to use the cyclic scan. After some time the system will stabilize and it will be possible to obtain images similar to the following one:



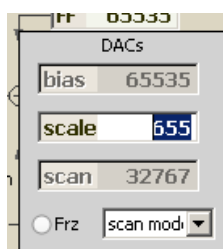
5. Change the tunneling current Sp in the range from -500 to -10000 pA and feedback loop gain Gain to achieve the optimal image contrast.

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.
3. Press the DACs button in the Scanner Ctrl window:



The DACs dialog box appears. Set Scale to 655:



7.3 Operation in liquid

The set for operation in liquid includes a liquid cell with a sample holder, special guide for mounting the liquid cell onto the microscope and a probe holder (fig. 7.3). The liquid cell has the internal and external parts connected with a membrane. Sample is mounted on the internal part using a spring clip. During the scanning process, only the internal part of the cell moves. The external part lies on the scanner cover and remains fixed. The liquid cell is mounted on the guide, which lies on the scanner cover.

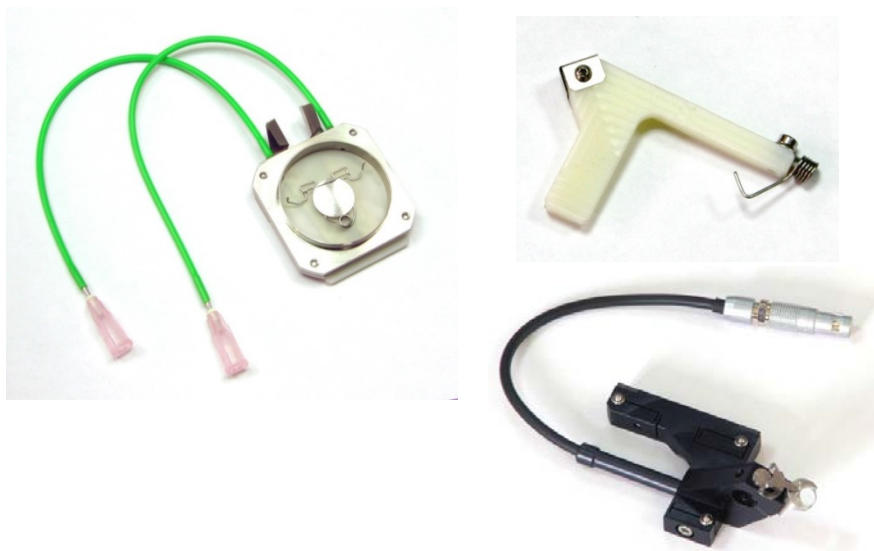


Figure 7.3 Set for operation in liquid

7.3.1 Liquid cell installation

- Remove the scanner to its bottom position and mount the guide for the liquid cell on the scanner (fig.7.3.1). Place your sample in the liquid cell and fix it by the sample holder (fig. 7.3.2). Make sure that your sample thickness is less than 1.5 mm.



Figure 7.3.1 Installation of the guide liquid cell

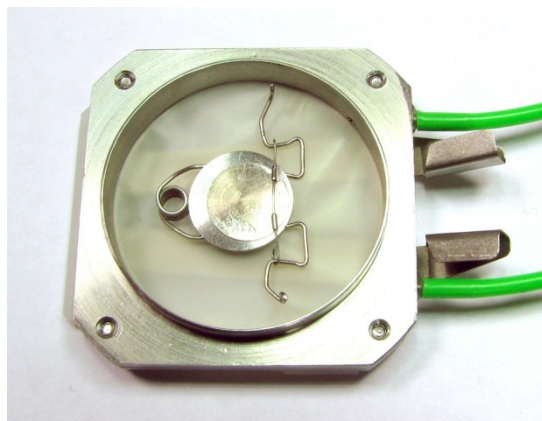


Figure 7.3.2 Sample installation for mounting the

- Install the liquid cell on the microscope, so the internal part of the cell slides into the scanner clench and the plastic ledge on the front side of the liquid cell gets under the guide.
- Move the scanner up to 2 mm in order to fix the liquid cell.
- Fill the cell with liquid, so it covers your sample.

7.3.2 Probe selection

To investigate your delicate samples (e.g. biological samples, polymers) in liquid environment, we recommend to use silicon nitride PNP-DB cantilevers made by NanoWorld (or similar type). Both sides of such chip have cantilevers.

Use the short lever of NanoWorld PNP-DB probe to work in semicontact AFM mode (lever A, length 100 μm , force constant 0.48 N/m, resonance frequency 67 kHz in air). The resonance frequency of the short lever in liquid is in the range of 9 – 12 kHz.

To work in contact AFM mode, you can use both short and long levers.

For rigid samples you can use standard non-contact Si cantilevers (e.g. fpN), but don't forget to re-adjust your cantilever holder in accordance with the cantilever's chip thickness (see Appendix 8.4).

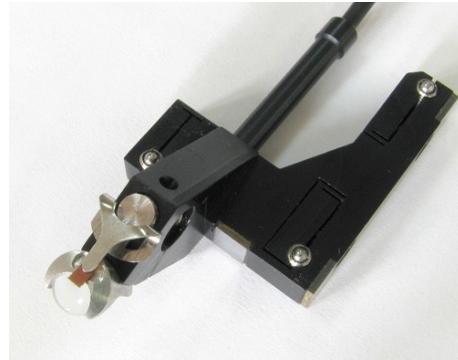
7.3.3 Probe installation

Do the following steps to install your probe into the holder:

1. Place upside-down the probe holder on the table.



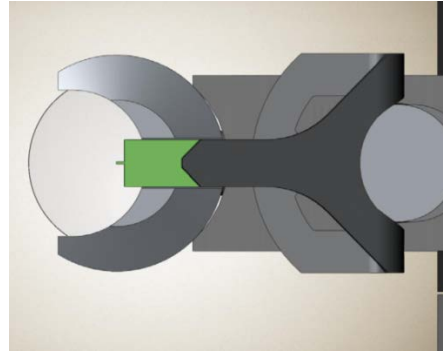
2. Lift the spring clip using tweezers, draw it aside and place on the edge of the probe holder.



3. Insert your probe into the groove.



4. Fix the probe by the spring clip



5. Correct probe position by moving it under the spring clip using tweezers. Make sure that the cantilever lies approximately on the probe holder lens axis.

- Put a 20 μ l drop of liquid between the probe and probe holder lens. Such drop will provide your probe moistening during the approach procedure and prevent from any air bubbles on the cantilever.
- Install the probe holder on the measuring head, fix it by the latch and attach the electric cable.
- Move the liquid cell up until the drop on the probe holder touches the liquid surface in the cell.
- Put a 20 μ l drop of liquid between the probe and probe holder lens. Such drop will provide your probe moistening during the approach procedure and prevent from any air bubbles on the cantilever.
- Install the probe holder on the measuring head, fix it by the latch and attach the electric cable.
- Move the liquid cell up until the drop on the probe holder touches the liquid surface in the cell.

7.3.4 Laser-photodiode adjustment

Make the laser-photodiode adjustment only when the probe is immersed in liquid.

Use commands “find tip” and “look around” the same way as you do in air if you use a single lever probe.

If you use a multilever probe do the following steps:

- Open the Laser calibration window.
- Press the init position button to set the probe holder carriage in the central position.
Select area on the full area window by left mouse button as shown in the figure 7.3.3.
- Press the go corner button to move the probe holder carriage, so laser beam is directed to the left bottom corner of the selected area.
- Press the Scan X button to start scanning the selected area. The image of your cantilever's chip and levers appears in the zoomed area window (fig. 7.3.3 right image).
- Set the red cross on the required lever and press the go button to position the laser beam on it.
- Press the look around button to finely tune the laser position.
- Press the adjust photodiode button to adjust the photodiode position.
- If you need to select another lever, you can use the acquired image or repeat the above procedure.

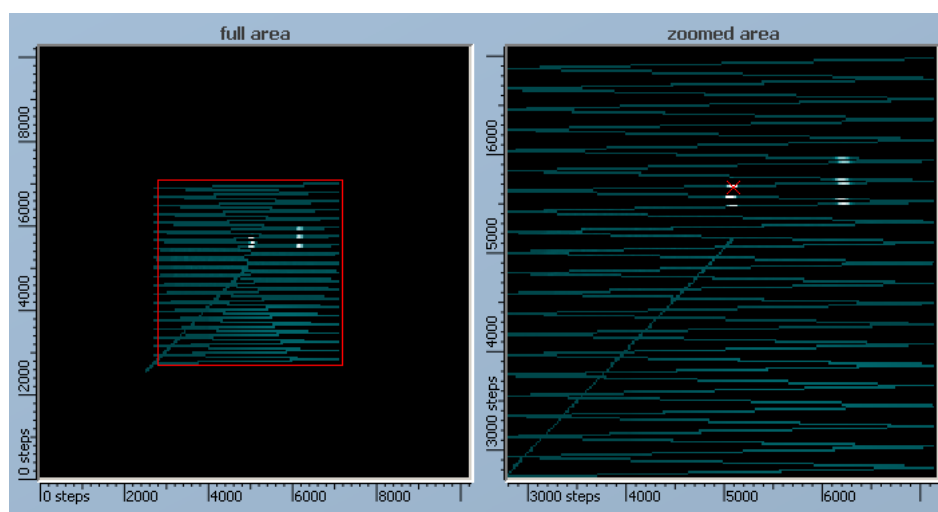


Figure 7.3.3 Multilever probe adjustment

NOTE: It is recommended to set the laser spot closer to the cantilever base while working with long contact cantilevers. To do so, before the laser-photodiode adjustment the AFM registration system set Ypos=60% in Param tables/ laser Alignment.

7.3.5 Operation in contact AFM mode

Once you finish the laser-photodiode adjustment, you can proceed your operation as if it is in air.

7.3.6 Operation in semicontact AFM mode

There may be a problem to select the right resonance peak of the cantilever during the system adjustment for operation in liquid. The reason is that there are a lot of peaks on the amplitude frequency characteristic obtained in liquid (fig. 7.3.4), which are some acoustic resonances of the cantilever holder. Use only those peaks which are near the resonance frequency of your cantilever in liquid.

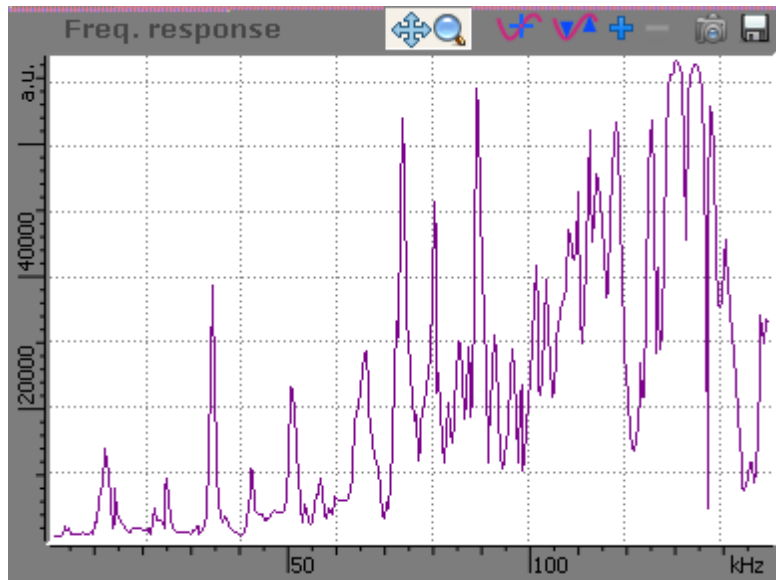


Figure 7.3.4 Amplitude frequency characteristic of Nanoworld PNP-DB probe, short cantilever, liquid environment, probe is driven by the piezoelement installed on the cantilever holder.

7.3.6.1 Selecting the cantilever drive frequency in liquid

Contact cantilevers

For soft (contact) cantilevers the resonance frequency in liquid is usually less than 4-6 times than the same in air. Thermal noise characteristic can be used to determine the resonance frequency of such cantilevers in liquid. To do so, switch off the driving voltage, set the maximum gain of the lock-in amplifier and obtain an amplitude frequency characteristic:

- Set Param tables/ AC pumping/ piezo actuator = None.
- Set the following parameters in Param tables/ lock-in: Lock-in input = Nfx10; Lock-in gain = 100.
- Set the following parameters in Param tables/ spectrum: Pts count = 500; Passes = 1;
Time per point = 2000; Time delay = 50.
- Change the digitization rate ctrl rate to 100 kHz in the Filters window.
- Open the Resonance window. Set the frequency range in accordance with the expected resonance frequency of your cantilever in liquid.
- Sweep the frequency.

You should obtain approximately the same characteristic as below:

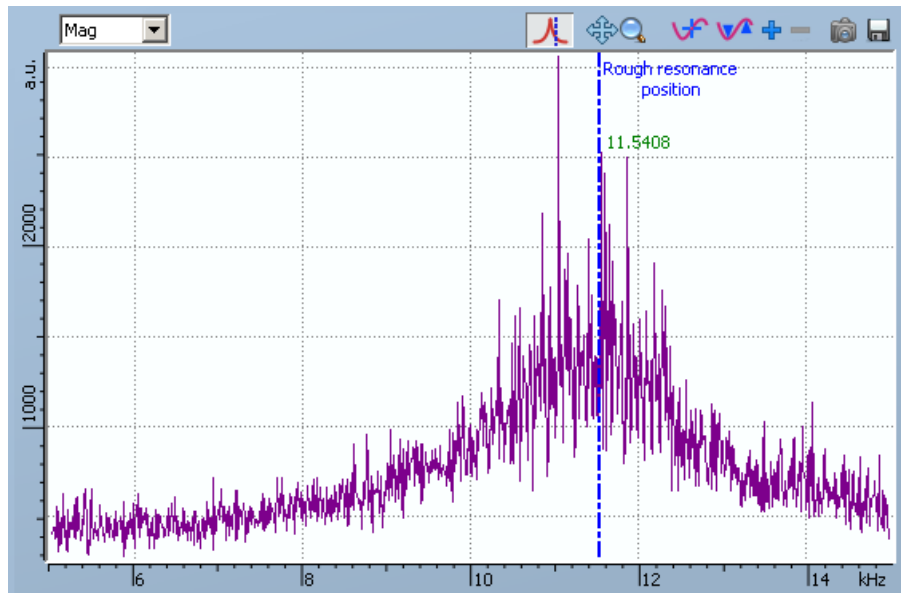


Figure 7.3.5 Thermal noise characteristic of Nanoworld PNP-DB probe, cantilever A, length 100 μm , force constant 0.48 N/m, nominal resonance frequency in air 67 kHz. The resonance peak in liquid is about 12 kHz.

Non-contact cantilevers

It has been determined that the peak of thermal noise characteristic of hard (non-contact) cantilevers is almost invisible. That's why we recommend to set the drive frequency about 2 times less in liquid than the resonance frequency in air.

7.3.6.2 Adjust the drive frequency

- Select the AC mode from the Modes menu.
- Set the cantilever's excitation and lock-in amp parameters in Param tables/ LOCK-IN: gen. magnitude = 50%
gen. magnitude switcher = x10

Lock-in input = Nfx10

Lock-in gain = 20

- Open the Resonance window. Set the frequency range in accordance with the expected resonance frequency of your cantilever in liquid.
- Sweep the frequency.
- Normally you can see a few peaks on the amplitude frequency characteristic nearby the expected resonance frequency (fig. 7.3.6). You can use any of those peaks as the drive frequency, but it's recommended to use the highest one in terms of amplitude and frequency.

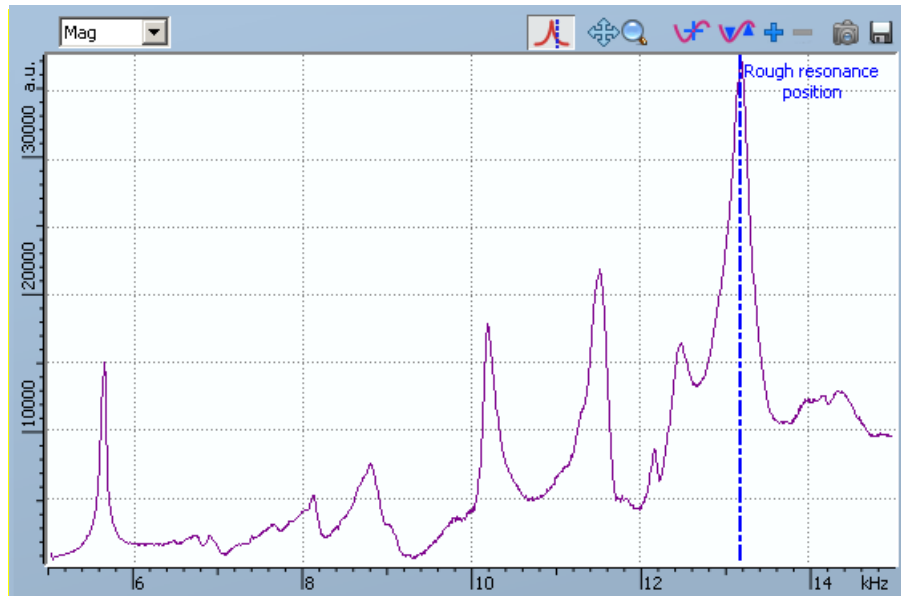


Figure 7.3.6 Amplitude frequency characteristic of the Nanoworld PNP-DB probe, short cantilever.

- Set the working frequency at the top on the selected peak.
- Switch on the Mag signal in the oscilloscope window.
- Set the Mag signal value in the range of 15000 – 30000 by changing the the Lock-in gain.

7.3.6.3 Tip-to-sample approach

- Set the lock-in filter parameter to 0.2 kHz in the Filters window.
- Press the `m_approach` button on the control panel to start the motorized approach procedure.
- Set the lock-in filter parameter to 2 kHz right after the motorized approach procedure is finished.
- Repeat the adjustment of the working frequency and the Mag signal as resonance peaks of the cantilever are changing their frequencies and amplitudes during the approaching process.
- Run the fine tuning procedure to set the phase signal to 90 degrees (i.e. 130000 arbitrary units).

Proceed with landing, feedback gain adjustment and scanning the same way as it is done in air.

Figure 7.3.7 (below) shows a typical curve of the cantilever amplitude as a function of distance. The specific feature of such curve is that during approaching to the surface the cantilever amplitude does not drop down to zero, but comes to some plateau. It's recommended to set the working setpoint amplitude not in the plateau part, but in the upper linear part of the curve.

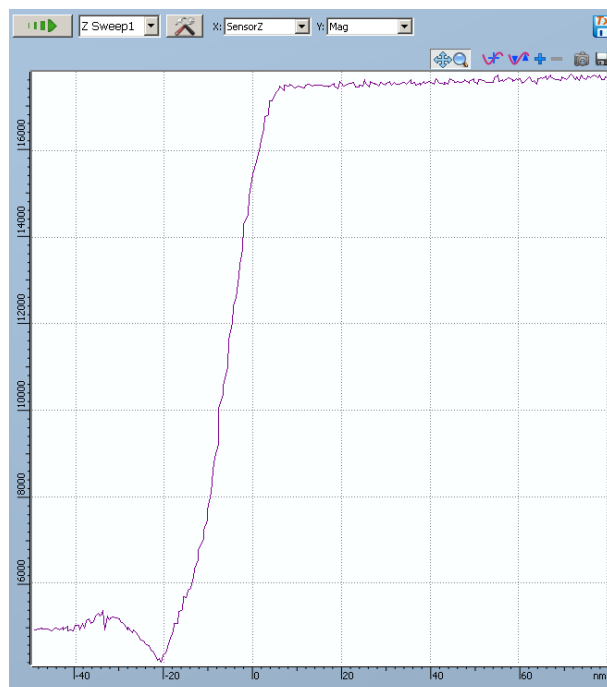


Figure 7.3.7 Typical curve of the cantilever amplitude as a function of distance between the probe and sample surface in liquid environment.

7.4 Working with the quartz oscillator (Tuning Fork mode)

As a sample surface force interaction sensor, a tuning-fork-shaped clock quartz oscillator may be used. Such tuning fork is a piezoelectric system: when oscillating, it generates electrical voltage. Oscillator excitation is performed mechanically: the tuning-fork is oscillated due to oscillations of a piezoelectric element embedded into the microscope probe holder. Excitation is performed at the resonance frequency. When interacting with the surface, the nature of tuning fork oscillations changes, which correspondingly leads to the change of the generated voltage parameters (amplitude, phase, frequency). These parameters are used as feedback signals.

Our device employs two basic methods of operation with a quartz oscillator: in semi-contact mode and in shear force mode (fig. 7.4.1).

In the semi-contact mode, a tuning-fork is positioned horizontally, and a stylus is attached in perpendicular to the tuning fork. When oscillating the tuning fork, the stylus oscillates in perpendicular to the sample surface.

In the shear force mode, a tuning-fork is positioned vertically, and a stylus (or any sensor) is attached along one of the tuning fork axes. When oscillating the tuning fork, the stylus oscillates in parallel to the sample surface.

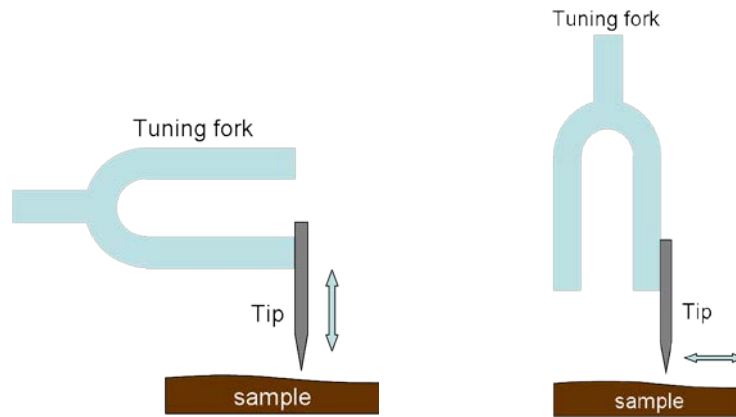


Figure 7.4.1 Operation scheme with horizontal and vertical positioning of the tuning fork.

In general, quartz oscillators are used as carriers of different sensors: fiber optic, SQUID, SET. Furthermore, using tuning forks is justified in experiments with photosensitive samples as in this case the probe-sample distance control scheme is non-optical (unlike AFM, it doesn't use laser).

High rigidity of tuning forks prevents probe tip sticking and allows working with extremely low amplitudes.

In most cases, due to high Q-factor of tuning forks, a dynamic force microscope mode is used (DFM), when Z-feedback controls tuning fork oscillation frequency shift using the PLL system.

7.4.1 Assembling the system to work with the quartz oscillator

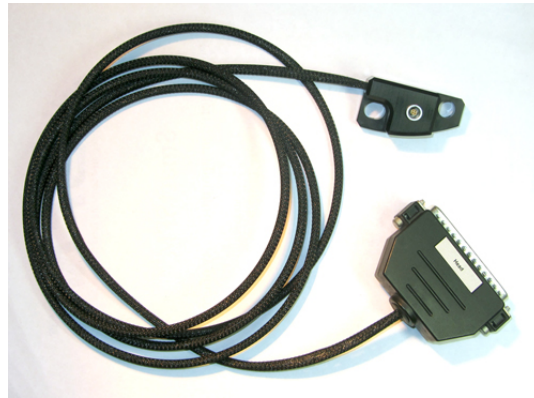
To work with the quartz oscillator, the following accessories are used:



h7 plate



Shf probe holder



Electrical cable adapter

Figure 7.4.2 Set of accessories to work with the quartz oscillator.

To assemble the system, proceed as follows:

- Turn off the controller.
- Remove the AFM measuring head from the microscope by unscrewing corresponding mounting screws. Detach the measuring head from the controller.
- Remove the adapter plate from the microscope (if available).
- Place h7 plate on the microscope.
- Screw in two special hex screws with side cone-shaped grooves into the h7 plate.
- Set the electrical cable terminal bracket on special screws. Fix it by tightening fixing screws located at the bracket side.
- Connect the cable to the controller's head connector.

7.4.2 Making a probe

To make a probe, use quartz oscillators with frequency 32768 Hz in a cylindrical body DT-38T with dimensions 3x8mm (fig. 7.4.3a). Remove the upper body from the oscillator so that the tuning fork becomes opened. Oscillator's terminal leads must be soldered to the circuit board supplied with the device (fig. 7.4.3 b, c). Attach a stylus or any corresponding sensor to one of the tuning fork sides using epoxy adhesive.

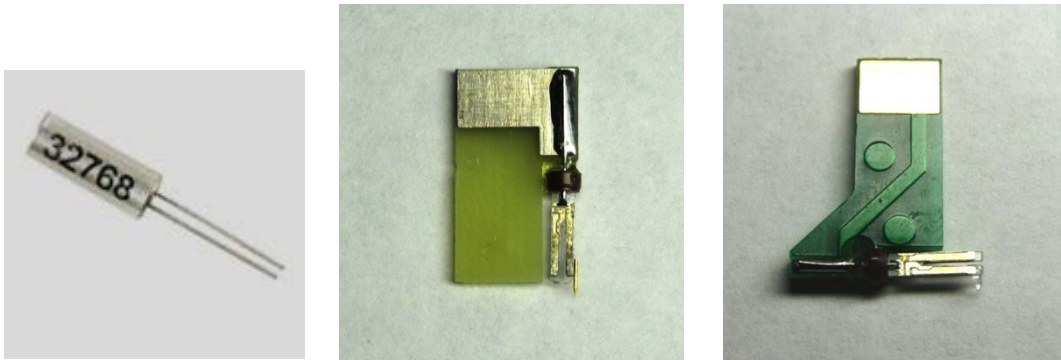


Figure 7.4.3 Quartz oscillator in the body (a), finished probe sensors: vertical (b) and horizontal (c).

7.4.3 Probe mounting

Figure 7.4.4 (below) shows the probe holders with sensors installed.

Horizontally positioned tuning fork:

Mount the sensor in such a way that the mounting screws must rest against round marks on the circuit board.

Vertically positioned tuning fork:

The lower edge of the sensor board must be at the level with the holder's lower plane.

Ensure that holder's spring contacts touch the sensor circuit board bonding pads. To fix the sensor in the holder, tighten screws on the holder using a 0.89 mm Allen key.

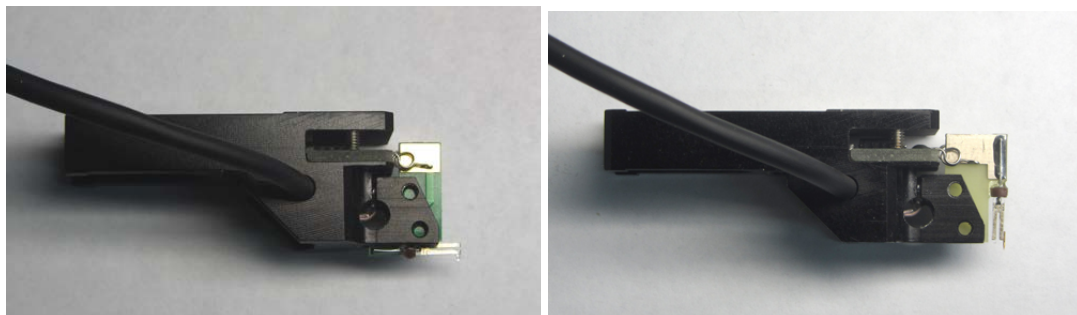


Figure 7.4.4 Quartz oscillator holder with the sensor installed.

7.4.4 Preparing the device for measurements

- Place a sample on the microscope as described in section 3.2.
- Set the holder with the sensor installed under a fixing screw; turn the holder anticlockwise so that it rests against the rods in the plate.
- Fix the holder by tightening the fixing screw on the plate.
- Holder connector must be connected to the terminal bracket.
-

Figure 7.4.5 shows the system assembly.



Figure 7.4.5 SmartSPM with the accessories mounted for working with the quartz oscillator.

7.4.5 Switching on the Tuning fork mode

To switch the system to the quartz oscillator mode, select Tuning fork in the tab Tools of the main menu (fig. 7.4.6). The software will make necessary switching and the T-Fork control panel will appear (fig. 7.4.7). The T-Fork control panel contains the buttons that allow adjusting the device to work with the quartz oscillator step-by-step.

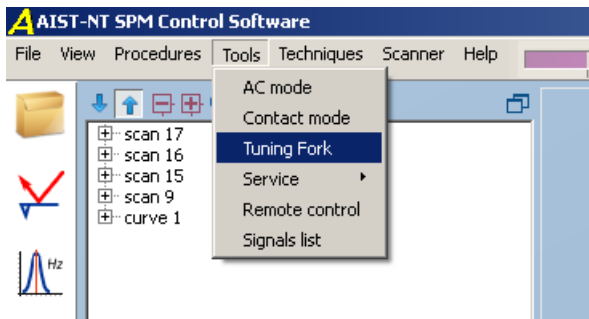


Figure 7.4.6 Switching on the Tuning fork mode.

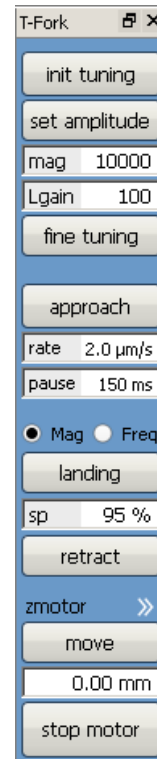


Figure 7.4.7 T-Fork control panel.

7.4.6 Adjusting the resonance

To adjust the excitation frequency to the resonance one, press the Init tuning button on the control panel. The software will proceed to the Resonance window and will perform frequency scanning in rough and fine ranges (fig. 7.4.8). Tuning fork frequency is 32,768 Hz, and the presence of the attached probe increases or decreases the frequency. The default rough scanning range is 24-38 kHz, fine scanning range is within 1 kHz.

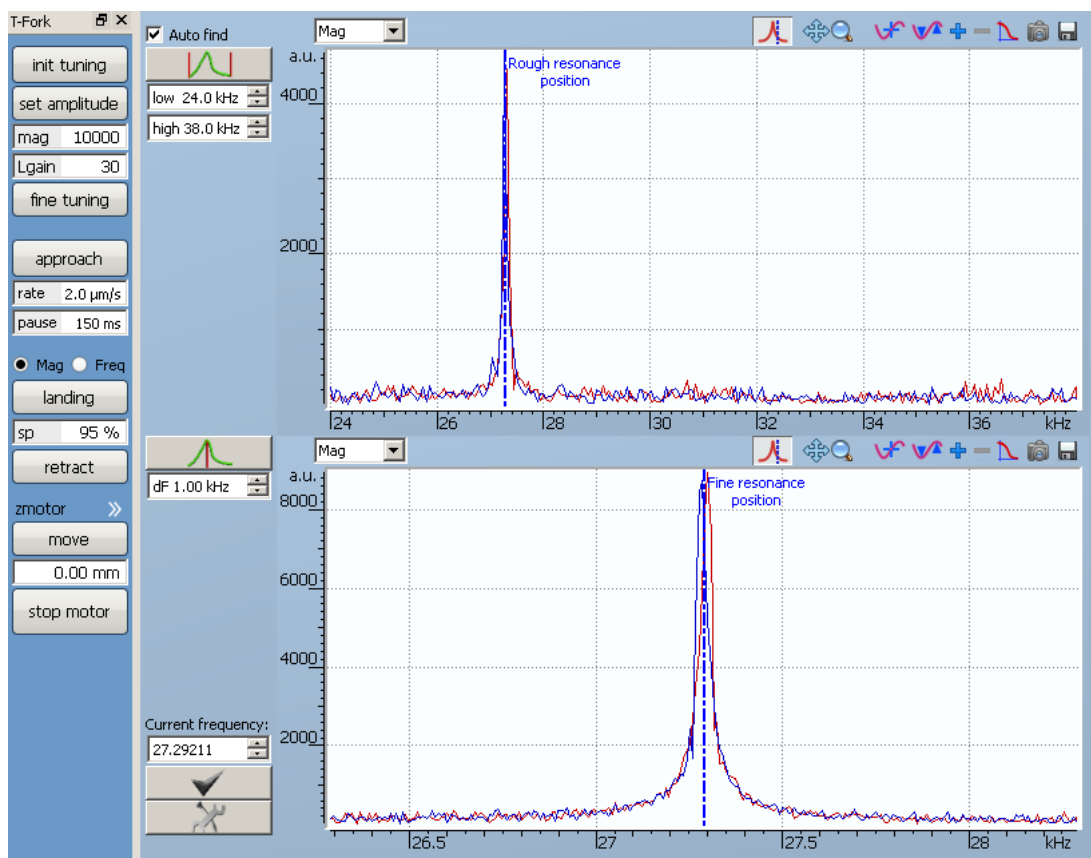


Figure 7.4.8 Resonance curve of the quartz oscillator with a stylus attached.

7.4.7 Setting the oscillation amplitude

To set the initial oscillation amplitude, use the set amplitude button and enter values in the mag and Lgain fields located below it:

| | |
|---------------|-------|
| set amplitude | |
| mag | 10000 |
| Lgain | 30 |

mag – oscillation amplitude in ADC units,

Lgain – synchronous detector gain parameter, lock-in gain.

After pressing the set amplitude button, the generator voltage is set in such a way that with the given Lgain, the set level of mag is provided.

Respectively, for the set level of mag, generator's voltage (and consequently, real oscillation amplitude) is increased with Lgain decrease. For example, for mag=10000 and Lgain=100 oscillation amplitude will be 10 times less than with the same mag and Lgain= 10.

Actual oscillation amplitude depends on the oscillator's Q-factor and is determined experimentally. For the purpose of probe and sample safety, it's recommended to begin with low amplitude by setting Lgain of around 30 -100.

7.4.8 Preliminary approach

By using the move button on the control panel, move the sample as close to the probe tip as possible - 0.2-0.3 mm from it. The necessity of such preliminary approach is connected with the slow automatic approach.

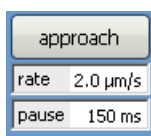
7.4.9 Automatic approach

To move the probe to the operating distance range from the surface, the following step-by-step algorithm is used:

Initially, the scanner is located in the lower position (as much as possible away from the probe). In the beginning of the approach, the scanner moves the sample towards the probe, and signals of the probe oscillation amplitude and phase are measured simultaneously. If signals do not change when the scanner is moved along the whole range (the probe is too far), the scanner moves the sample back to the lower position and makes a step towards the probe using the step Z motor. Then the process is paused, following which the cycle is repeated.

If quick changes of signal occur during scanner moving-out (the probe interacts with the surface), the movement stops and the scanner moves the sample by 1 micron away from the probe.

To start the approach, use the approach button. Parameters rate and pause set the approach rate and the pause between cycles, respectively:



7.4.10 Selecting a feedback signal

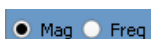
When working with the quartz oscillator, as a feedback input signal upon the height, the following signals may be selected:

Amplitude signal mag – feedback system maintains permanent oscillation amplitude (similarly to the AC-mode AFM technique).

Frequency signal Freq – feedback system maintains permanent oscillation frequency shift relative to the resonance frequency. For frequency determination, the PLL system is used.

The signal selection is determined by the sensor Q-factor. In case of low Q-factor (below 500), it's possible to use one of these signals, and in case of high Q-factor (above 1000), it's possible to work only with Freq.

To select a feedback signal, use the Mag/Freq switch on the control panel:

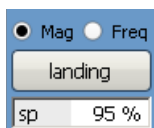


7.4.11 Landing to surface

Depending on the selected feedback signal, the landing is performed differently.

7.4.11.1 Landing to surface using the mag signal

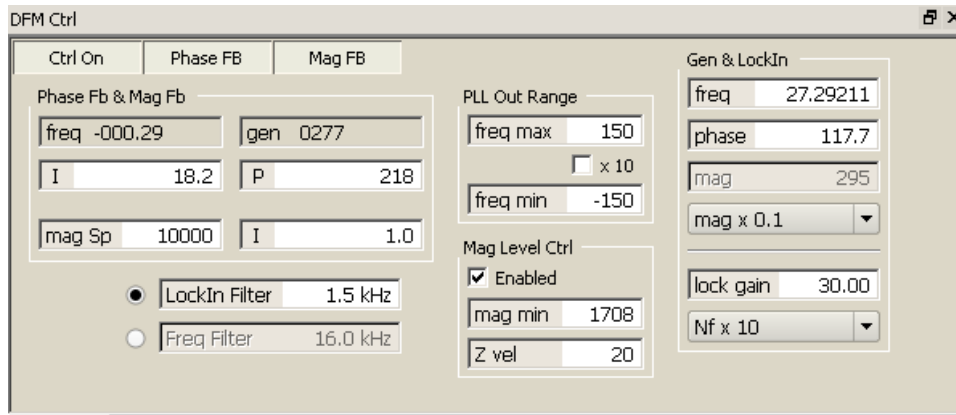
Set the mag signal as a feedback input and press landing:



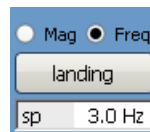
The landing is performed to the setpoint equal to 95% of the mag signal current value. It means that the probe and the sample will stop approaching to each other as soon as the oscillation amplitude drops by 5%. Thus, if before the landing the mag signal was 10000, then the Sp setpoint will be 9500.

7.4.11.2 Landing to surface using the freq signal

Before starting the landing, open the DFM Ctrl control window of the PLL system:



On the T-fork control panel, set the Freq signal as a feedback signal and press landing:



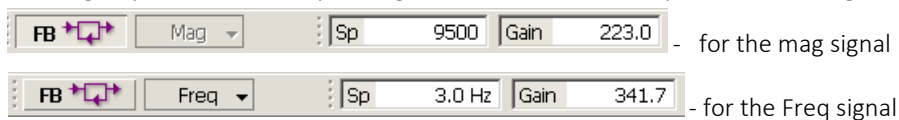
Sp= 3 Hz is used by default.

When starting the landing, in the DFM Ctrl window, the feedback using the oscillation phase Phase FB will be automatically on, which is necessary to determine oscillation frequency. The Phase FB feedback parameter (gain parameters I and P) is set automatically depending on the oscillator's Q-factor. It's not necessary to select them.

After the landing procedure, the oscillation amplitude may decrease. To maintain the amplitude at some constant level, turn on the Mag FB feedback by preliminarily setting mag Sp equal to the mag signal before the landing (10000 in our example).

7.4.12 Setting up Z feedback

The feedback gain parameter Gain operating value is set automatically after the landing:



If the gain parameter is insufficient, adjust it directly during the scanning process (see 7.4.14.3).

7.4.13 Scanning

1. After the landing, go to the Scan window and switch to the **Qscan mode**.
2. In the Qscan mode settings, ensure that the **Adaptive scanning mode is on**.

High Q-factor of the quartz oscillator substantially limits the maximum scanning rate.
To increase the average scanning rate, use the Back jump mode.

Use the Mag and Phase signals as additional signals to the height signals. When working with the feedback using frequency, these signals are complemented by the Freq signal that is a feedback error signal in this case.

3. Set initial scanning rate as 0.5 Hz and the scan size of 1-5 microns.
4. Start the scanning.

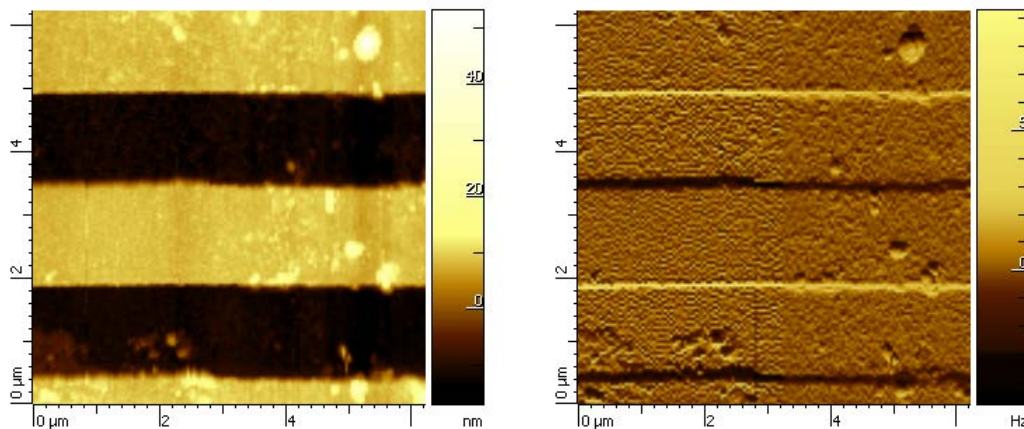
7.4.14 Image quality optimization

The following parameters mainly influence the image quality:

- Scanning rate rate;
- setpoint Sp;
- feedback gain parameter Gain;
- oscillator amplitude.

7.4.14.1 Adjusting the rate

Maximum scanning rate is defined upon the presence of “noisiness” in a feedback signal (Mag when working using the amplitude or Freq when working using the frequency). The images of the TGZ1 calibration grid are given below (topography and feedback error signal Freq); scanning was performed along the Y axis. At the start of scanning (in the left part of the scan), 0.7 Hz rate was set, as the “noisiness” can be seen in the Freq signal. In the right part of the scan, the rate was decreased to 0.3 Hz until the “noisiness” disappeared:



7.4.14.2 Adjusting the setpoint

You must use the minimal possible force, at which you obtain a stable image. When working using the amplitude, the setpoint usually lies within 90-98%, and when working using the frequency, it lies within a few hertz.

7.4.14.3 Setting feedback gain

You must use the maximum possible Gain value, at which parasitic oscillations (“generation”) are not yet started. To determine this value, smoothly increase Gain during the scanning process until generation occurs (it can be distinctly seen in the form of additional noise on the scanning line profile in the height signal). After that, slightly decrease Gain, for the generation to disappear.

7.4.14.4 Setting the oscillator amplitude

Use the least possible amplitudes of the oscillator.

When working using the amplitude, you can change the oscillation amplitude only when scanning is stopped and the probe is removed from the surface. To change the amplitude, proceed as described in 7.4.7.

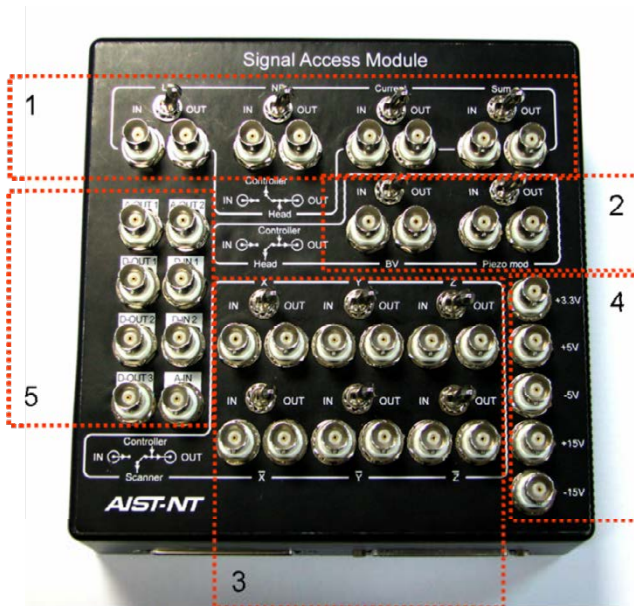
When working using the frequency, you can change the oscillation amplitude when scanning. To do it, preliminary turn on the

feedback Mag FB and change the value lock gain in the DFM Ctrl window. As it has been said above, when increasing lock gain, the oscillator amplitude decreases.

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.

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. 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.

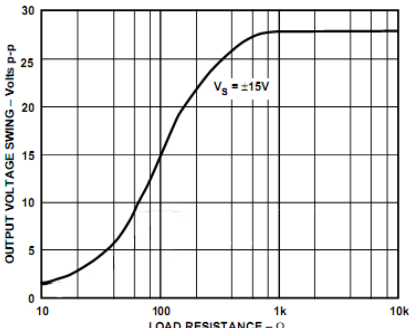
+/-15V, max load 70mA.

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

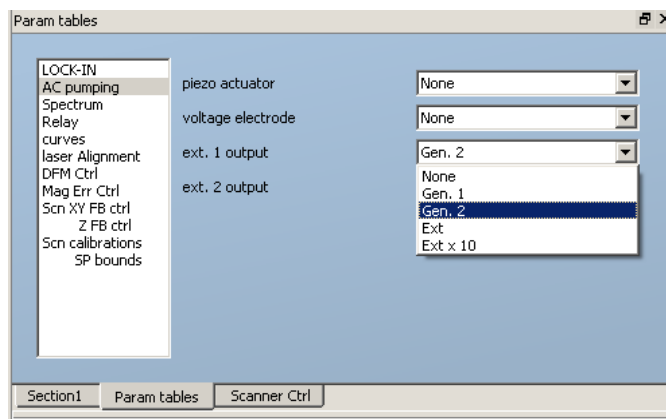
Group #5 (extension connectors)

Auxiliary I/O signals can be connected to these connectors.

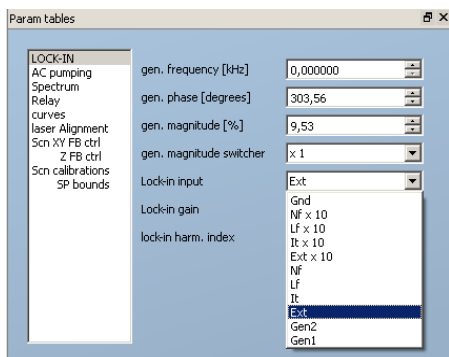
To use these connectors, it is necessary to make a cable connection between the SAM-E connector on the SAM and the "External Input/Output" connector on the AFM board of the controller.

| Name | Description | Voltage levels | Max. output current |
|---------|----------------|--|---------------------|
| D-OUT 1 | Digital Output | 0.0/+3.3 V | 24mA |
| D-OUT 2 | Digital Output | 0.0/+3.3 V | 24mA |
| D-OUT 3 | Digital Output | 0.0/+3.3 V | 24mA |
| D-IN 1 | Digital Input | 0.0/+3.3 V | — |
| D-IN 2 | Digital Input | 0.0/+3.3 V | — |
| A-OUT 1 | Analog output |  <p>Output Voltage Swing vs. Load Resistance</p> | |
| A-OUT 2 | Analog output | | |
| A-IN | Analog input | -12V to +12V | — |

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.

1. 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. |

2. 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). | <p>Restore the initial search parameters. You can find the search parameters in the Laser Alignment tab of the Param tables:</p> <p>Y pos = 25 X pos = 0 laser threshold = 8000 laser Background = 5000 length of cantilever = 2000 laser align method = 2</p> |
|---|--|

3. 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. |

4. 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. |

5. Landing to your sample surface:

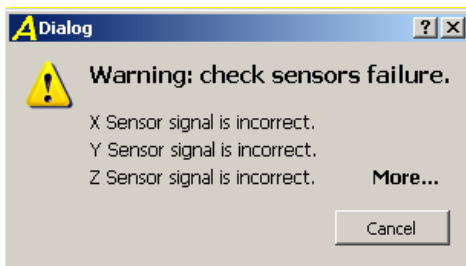
The system is unable to land. The “Failed to land” message appears.

| 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.

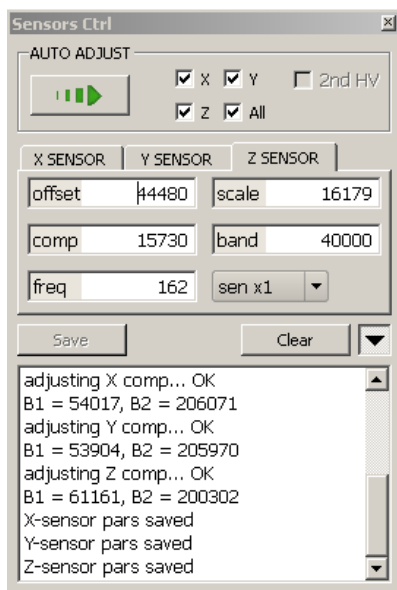
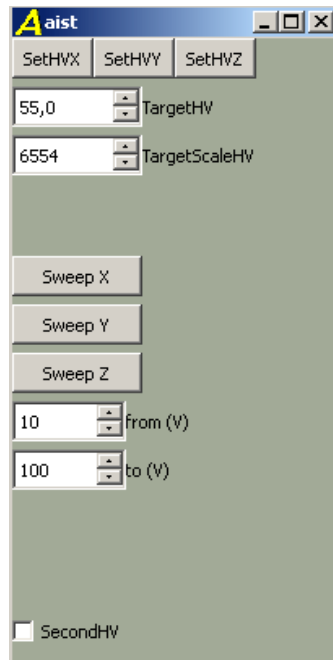


Figure 8.2.1 Sensors Ctrl scanner sensors calibration control panel

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.

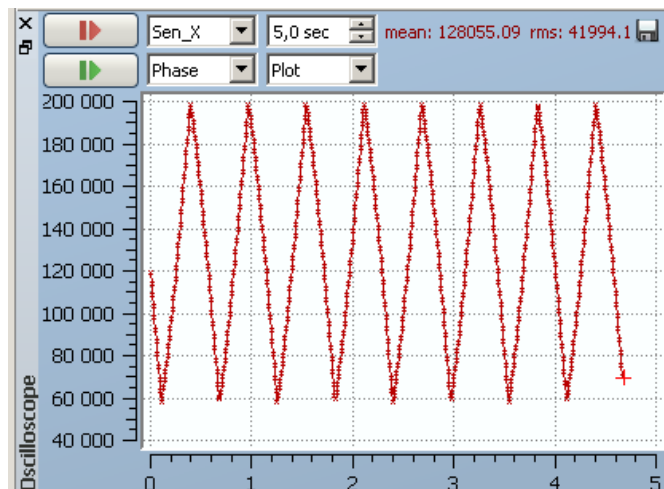
To check if the scanner sensors calibration is correct use `AFM11_Sensors1.lua` macro. This macro allows applying saw-shaped voltage to each scanner's axis:



- Remove the scanner to more than 100 μm from the probe tip.
- Press one after another SetHVX, SetHVV, 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 HE001 head

In the HE001 AFM head the laser is focused on the cantilever by means of the special lens. To change the focus, there is a rear support screw provided on the cantilever holder. This option is available due to the fact that cantilever chip thickness may vary from one supplier to another. By default the cantilever holder for operation in air is adjusted to work with cantilevers whose chip thickness is 0.4 mm (e.g. fpN series cantilevers) while the cantilever holder for operation in liquid is adjusted for 0.5 mm chip thickness (e.g. Nanoworld silicon-nitride cantilevers).

To adjust your cantilever holder to another chip thickness, do the following steps:

- Mark the initial position of the rear support screw using a mark pen (position 1 shown in Fig. 8.4.1);
- Loosen the fixing screw (position 2 shown in Fig. 8.4.1), so you can turn the rear support screw with a little resistance;

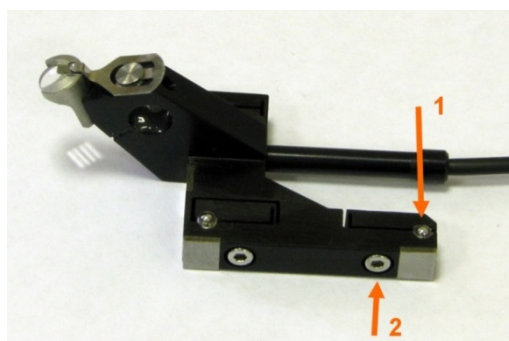


Figure 8.4.1 Cantilever holder. Rear support screw (1) and fixing screw (2).

- Install the cantilever into the holder and then mount the holder on the instrument;
- Use the find tip and adjust diode commands to adjust the AFM registration system;
- Use the show tip command to obtain an image of your cantilever's lever to check the laser focus adjustment (Fig. 8.4.3);
- Use the Motoscan mode to obtain a more detailed image (Fig. 8.4.4);
- Turn the rear support screw to adjust the laser focus (Fig. 8.4.2). Turn the screw clockwise to adjust the holder for cantilevers chip with higher thickness. You can perform this adjustment right on the instrument.

NOTE: To adjust the cantilever holder from 0.5 mm thickness to 0.4 mm, turn the rear support screw clockwise to 120 degrees.

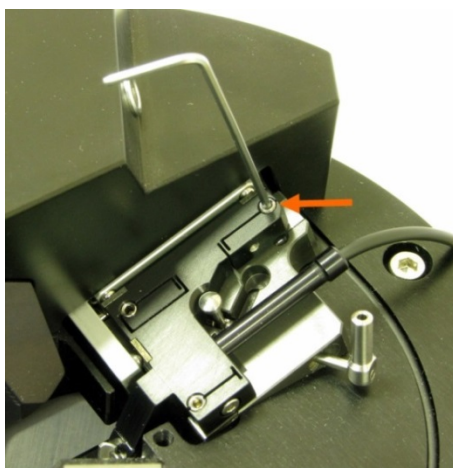


Figure 8.4.2 Adjustment of cantilever holder installed on the instrument.

- Check the laser focus again: use the look around command to align the laser to the cantilever and then use the show tip command to obtain a new image of the cantilever next to the previous one (Fig. 8.4.3);
- Obtained a new detailed image of the cantilever using the Motoscan mode (Fig. 8.4.4);
- Remove the cantilever holder from the instrument and tighten the fixing screw;

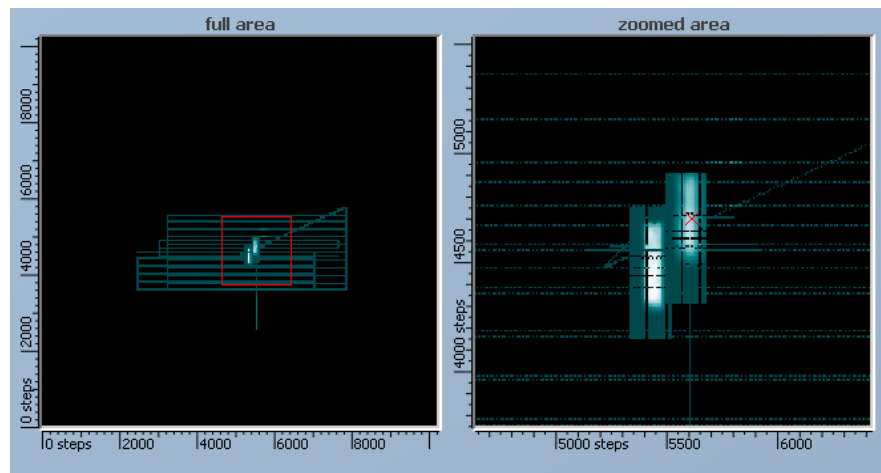


Figure 8.4.3 Images of the cantilever obtained using the show tip command in the process of the laser focus adjustment.

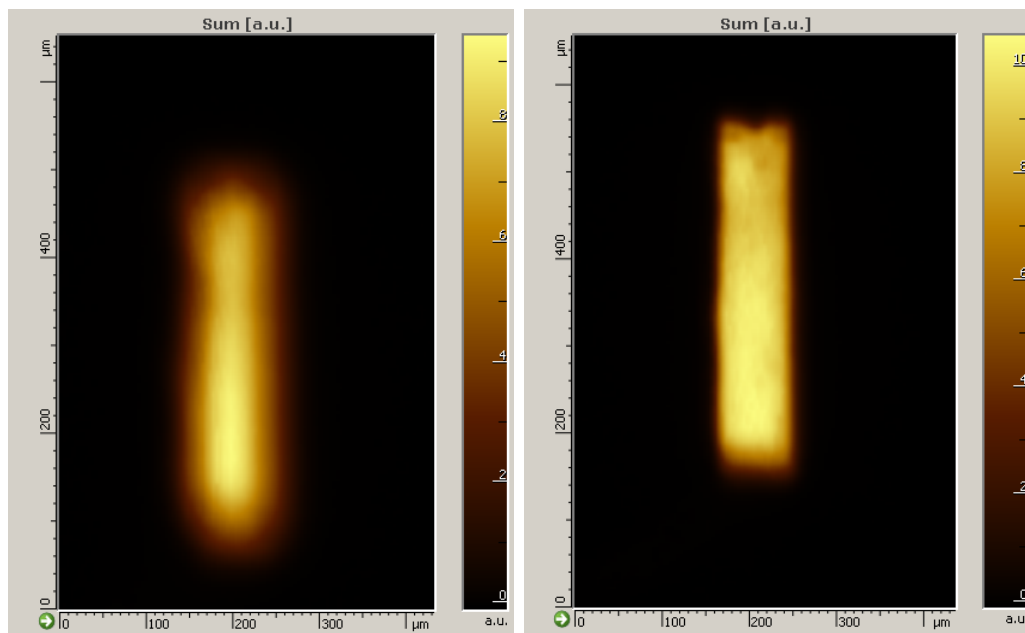


Figure 8.4.4 Images of the fpN03 cantilever obtained in the Motoscan mode before the adjustment (left) and after (right).

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:

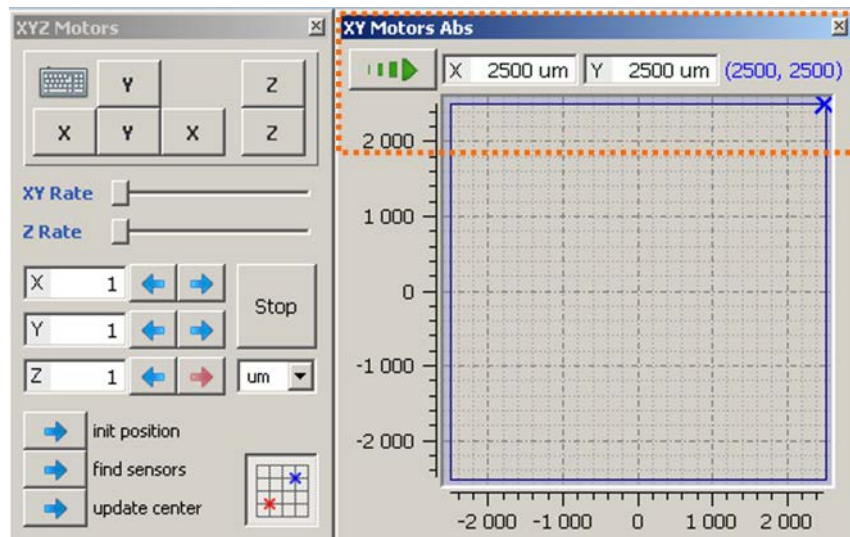
| Pin # | Description | Voltage levels | Load resistance | Max. output current | Freq. range (Generator) | Frequency of pulses |
|-------|---------------------|------------------------------|-----------------|---------------------|-------------------------|---------------------|
| 1 | +3.3 V | | | 100mA | | |
| 2 | +15 V | | | 100mA | | |
| 3 | digital output #2 | (0V..0.45V)/ (2.4V..3.3V) | | 8mA | | 30kHz (max) |
| 4 | digital output #1 | (0V..0.45V)/ (2.4V..3.3V) | | 8mA | | 30kHz (max) |
| 5 | Bias Voltage input | 50V (max) | | | | |
| 6 | -5 V | | | 100mA | | |
| 7 | +5 V | | | 100mA | | |
| 8 | Gnd | | | | | |
| 9 | unconnected | | | | | |
| 10 | unconnected | | | | | |
| 11 | Gnd | | | | | |
| 12 | -15 V | | | 100mA | | |
| 13 | unconnected | | | | | |
| 14 | analog output (Ex2) | ±10V | 1kOhm (min) | | 1kHz..5MHz | |
| 15 | analog output (Ex1) | ±10V | 1kOhm (min) | | 1kHz..5MHz | |

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.