SOLVER platform

SOLVER NEXT

Instruction Manual

Scanning Probe Microscope
SOLVER NEXT

Instruction Manual
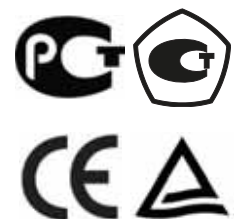
2012

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Read me First!

Observe safety measures for operation with devices containing sources of laser radiation. Do not stare into the beam. A label warning about the presence of laser radiation is attached to the laser sources (Fig. 1).

Fig. 1

Before you start working with the instrument, get acquainted with the basic safety measures and the operation conditions for the instrument!

If you are a beginner in scanning probe microscopy, we recommend you to familiarize with basic SPM techniques. "Fundamentals of Scanning Probe Microscopy" by V.L. Mironov gives a good introduction to the subject. This book is available free of charge at http://www.ntmdt.com/manuals.

Some equipment, which is described in the manuals, may not be included into your delivery set. Read the specification of your contract for more information.

Feedback

Should you have any questions, which are not explained in the manuals, please contact the Service Department of the company (support@ntmdt.ru) and our engineers will give you comprehensive answers. Alternatively, you can contact our staff on-line using the ask-on-line service (http://www.ntmdt.com/online).

The manuals are updated regularly. Their latest versions can be found in the site of the company, in the section "Customer support" (http://www.ntmdt.com/support).

SOLVER NEXT SPM. Instruction Manual

Table of Contents

1. Overview

SOLVER NEXT is a multifunctional Scanning Probe Microscope of general purpose. It is a state-of-the-art instrument with highly automated operations. Its digital controller enables a wide range of SPM techniques. Automatic adjustment of cantilever deflections detection provides ease-of-operation even for an unexperienced user.

The key distinction of the SOLVER NEXT SPM from its analogues is availability of two built-in measuring heads (AFM and STM) that are automatically adjusted to their working position.

Besides, the design of the instrument provides insertion of auxiliary measuring heads. Currently, the kit of available auxiliary heads includes those for liquid and nanosclerometric measurements.

The sample can be heated up to 150 °C with the use of the mountable heating stage.

Focusing and zooming of the built-in optical videomicroscope are motorized. Selection of the scan area is motorized as well.

Scanning Probe Microscope is capable to perform measurements with the following techniques depending on working environment:

In air and in liquid

Atomic Force Microscopy (AFM) (contact; semicontact; non-contact); Lateral Force Imaging; Phase Imaging Mode; Force Modulation Mode; Adhesion Force Imaging; AFM Lithography (Force), AFM and STM spectroscopies.

In air only

Scanning Tunneling Microscopy (STM); Magnetic Force Microscopy (MFM); Electric Force Microscopy (EFM); Scanning Capacitance Microscopy (SCM); Kelvin Probe Microscopy; Spreading Resistance Imaging; Piezoresponse Force Microscopy, Nanosclerometry; AFM Lithography (force and current), STM Lithography, AFM and STM spectroscopies.

1.1. Design

General view of the SOLVER NEXT SPM is shown in Fig. 1-1.
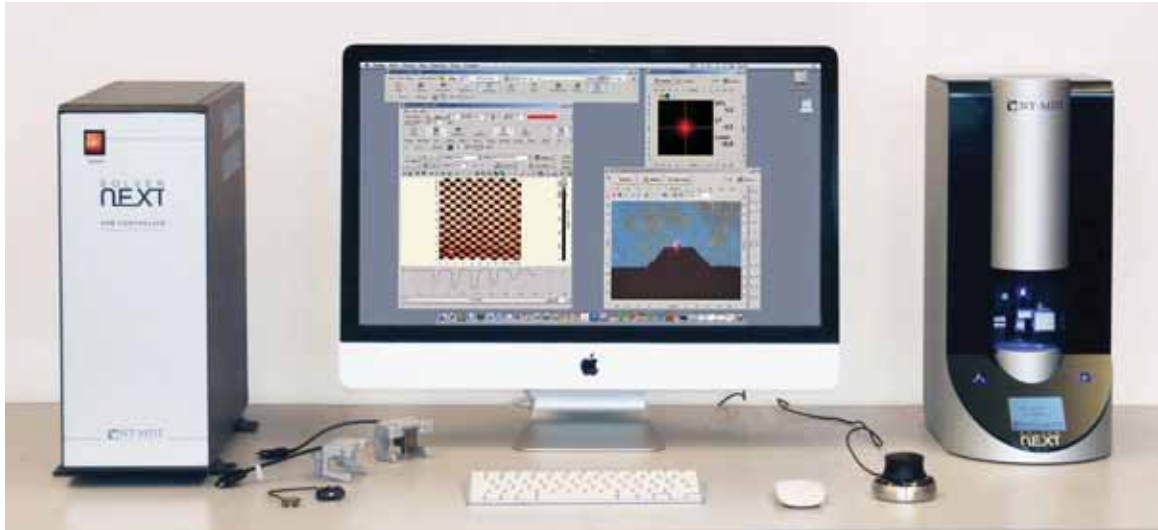
Fig. 1-1. General view of the SOLVER NEXT SPM

The basic delivery set of SPM SOLVER NEXT contains:

- measuring unit;
- SPM controller;
- CD-ROM with software, manuals, and supporting materials
- Installation disk with drivers of the video camera;
- Set of sample holders and supporting substrates.

Fig. 1-2 shows basic components of the measuring unit of SOLVER NEXT.
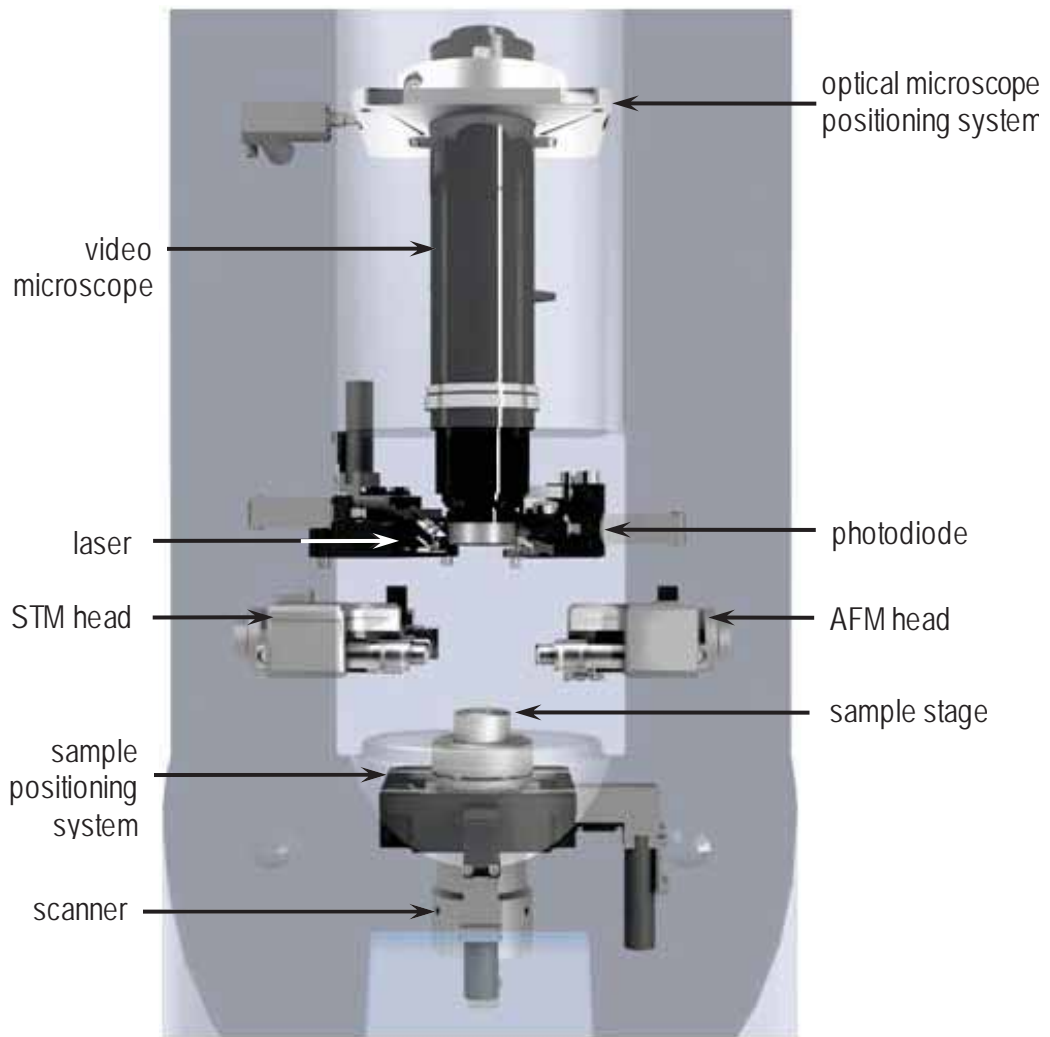
Fig. 1-2. Basic components of the measuring unit of SOLVER NEXT

The sample to study is fixed on a metal substrate that is mounted on the sample holder. The sample holder is fastened to the piezotube of the scanner.

The positioning system allows moving the sample in the XY plane to select the investigation area as well as displacing the sample vertically to approach it to the probe.

The SOLVER NEXT measuring unit is equipped with two measuring heads, one of which is for atomic-force microscopy (AFM head) and the other is for scanning tunneling microscopy (STM head). The system of switching the measuring head HEADHIPEX™ enables quick exchange between experimental techniques and does not need special training of the operator.

The measurement area is protected from external noises, electromagnetic interference, and temperature fluctuation with the protective shield (ISOSHIELD™ system). In its lower position, the protective shield isolates the measuring head with the sample inside, thus improving quality of the detected image. Besides, the ISOSHIELD™ system automatically disables the laser detection system when the protective shield is in upper position to prevent exposure of the operator by laser irradiation.

Capabilities of the SPM SOLVER NEXT can be extended with auxiliary measuring heads, a nanosclerometric head (provides measurements of hardness and modulus of elasticity as well as modification of the sample surface) and a head for measurements in liquid.

1.2. Positioning the Built-in Measuring Heads

The built-in measuring heads can stay in one of three basic stationary position:

- Inoperative position (Fig. 1-3);
- Working position (Fig. 1-4, Fig. 1-5);
- Position for exchanging the probe (Fig. 1-6, Fig. 1-7).
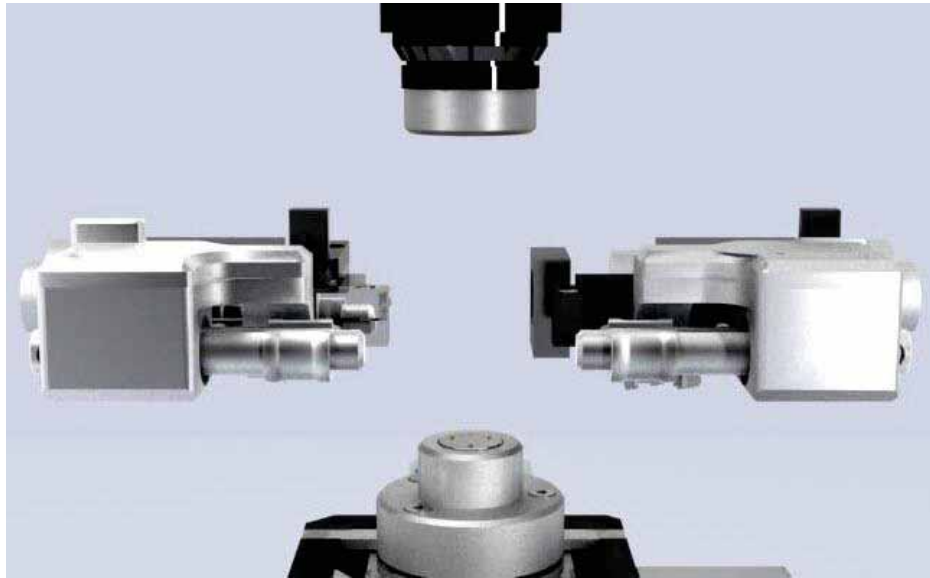
Fig. 1-3. The measuring heads are in inoperative positions
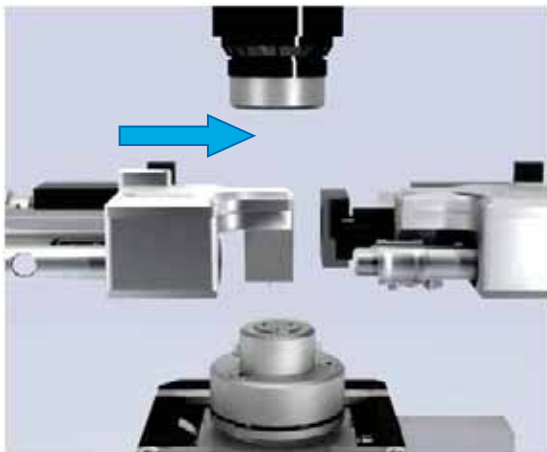
Fig. 1-4. STM head in working position
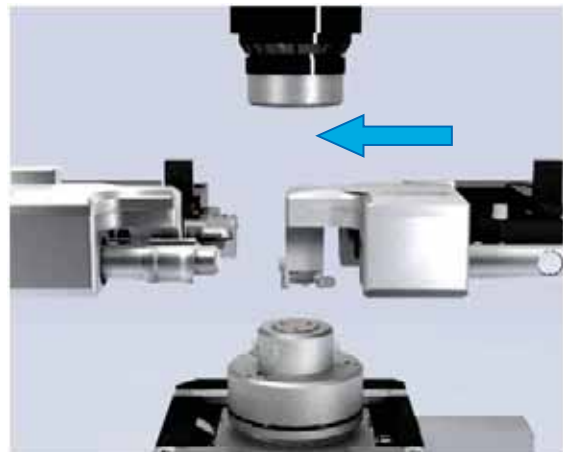
Fig. 1-5. AFM head in working position

Fig. 1-6. STM head in position for exchanging the probe

Fig. 1-7. AFM head in position for exchanging the probe

When a head is not involved in the current operation, it stays in its inoperative position inside the body of the measuring unit.

The working positions are used in the course of measurements with a measuring head being oriented so that the probe is above the sample.

A measuring head is switched from its working position to the position for exchanging the probe by manual rotation through 180º.

A head is switched from its position for exchanging the probe to the working position with the use of the Measurement Heads Control panel (Fig. 1-8) of the Control program.

Fig. 1-8. Measurement Heads Control panel

Designation of the heads positioning buttons is following:

 – moves the protective shield up and down;

 – turns on/off illumination of the measuring heads (enabled only when the protective shield is in its lower position).

 – moves the STM head to its working position and the AFM head to its inoperative position;

 – moves the AFM head to its working position and the STM head to its inoperative position;

 – moves both heads to their inoperative positions.

1.3. Technical Specification

Measuring system	
Built-in measuring heads	AFM, STM
Mountable measuring heads	liquid, nanosclerometric
System for detecting the cantilever deflection	automated alignment
Laser wavelength of cantilever deflection system	850 nm
Sample	
Size	up to ø20×10 mm
Positioning range, in XY plane	5×5 mm
Positioning method	automated with the use of optical image
Minimum positioning step	0.3 μm
Sample weight	up to 40 g
Heating capabilities	up to 150 °C
Scanning system	
Scanning mode	by sample
Scan area	100×100×10 μm (±10 %) 2.2×2.2×10 μm (±10 %) (in the high resolution mode)
Relative error of distance measurement: XY plane Z direction	 less 1 % (less 0.1 % with the use of the feedback sensors) less 5 %
Non-linearity, in XY plane (with the use of feedback sensors)	less 0.1 %
RMS noise in 1 ÷ 200 Hz frequency range, XY plane	less 0.3 nm (0.2 nm typically) less 0.001 nm (in the high resolution mode)
RMS noise in 10 ÷ 1000 Hz frequency range, Z direction: with the use of the feedback sensors in the high resolution mode	 less 0.04 nm (less 0.02 nm in the high resolution mode)
Videomisroscope	
Positioning range,	5×5 mm
Positioning step	0.3 μm
Resolution	2 μm
Field of view	3.4 ÷ 0.53 mm
Dimensions and weight	
Overall dimensions	321×214×480 mm
Weight	30 kg
Power characteristic	
Voltage	110/220 V
Power consumption	less 400 W

2. Basic Safety Measures

General Safety Measures

- **Ground the instrument before operation!**

- Do not disassemble any part of the instrument. Disassembling of the product is permitted only to persons certified by NT-MDT.

- Do not connect additional devices to the instrument without prior advice from an authorized person from NT-MDT.

- This instrument contains precision electro-mechanical parts. Therefore protect it from mechanical shocks.

- Protect the instrument against the influence of extreme temperature, the direct impact of sun radiation, and moisture.

- For transport, provide proper packaging for the instrument so as to avoid its damage.

Electronics

- To reduce possible influence of power line disturbances on the measurements, we recommend supplying the units instrument with a surge filter.

- Before operation, set the power switch of the SPM controller to the position corresponding to value of the local electrical power line (this is only done with the controller being off!).

- Switch the SPM controller off before connecting/disconnecting its cable connectors. Disconnecting or connecting the cable connectors during operations may cause damage to the electronic circuit and disable the device. A warning label is attached to the SPM controller (Fig. 2-1).

A t t e n t i o n !

The electronic module has to be switched off before connecting or disconnecting any cable. Otherwise electronic components will be damaged.
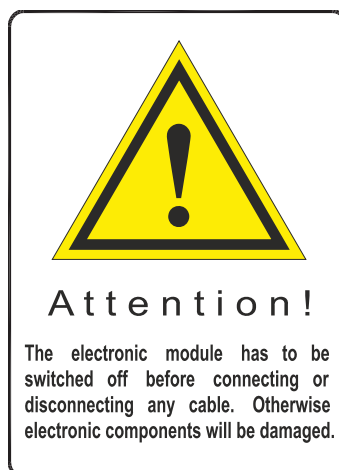
Fig. 2-1

Laser

- Observe safety measures for operation with devices containing sources of laser radiation. Do not stare into the beam. A label warning about the presence of laser radiation is attached to the rearing panel of the measuring unit.

CAUTION
INVISIBLE LASER RADIATION
AVOID DIRECT EXPOSURE TO BEAM

CLASS II LASER PRODUCT
1 mW   850 nm

Fig. 2-2

Scanner

- Do not apply to the scanner forces bigger than it is necessary for installation of a probe or a substrate with a sample. Avoid impacts on the scanner and its lateral displacement. Remember that thickness of the scanner piezotube walls makes only 0.5 mm.

3. Operating Conditions

The SPM should be installed in a room of total area larger 6 sq. meters with range of purity better 8 (by ISO 14644-1-2002).

To provide for the qualitative and safe operation of the instrument, it is recommended to observe the following conditions:

- environment temperature:
    - ultimate range   (15–35) °C;
    - recommended range   (20–25) °C.
- temperature drift: less 1 °C per hour;
- relative humidity:
    - ultimate range   (20–80) % (moisture condensation prevented);
    - recommended range   (30–50) % (moisture condensation prevented).
- vibration amplitude
  in the band 1 ÷ 1000 Hz   less 0.5 μm;
- atmospheric pressure   (760±25) mm Hg/(100±4) kPa;
- the work area should be supplied with a protective grounding circuitry and with grounded electric mains (110/220 V);
- electric mains:
    - voltage   (110±10)/(220±22) V;
    - frequency   (50.0±0.4)/(60±0.5) Hz;
- the room should be protected from mechanical vibrations and acoustic noises, either internal or external;
- vibration criterion VC-C, 12.5 μm/s (one-third octave band criterion);
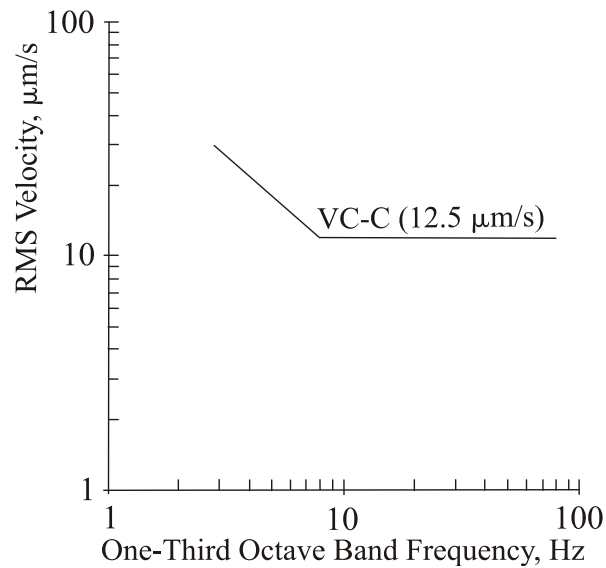
- the table intended for installation of the measuring unit must be stable and, whenever possible, massive;

- to provide solid stability against vibrations, SPM modules should be placed on self-leveling floor with marble covering alongside bearing components of the building (pillows, beams etc.);

- the measuring unit of the SPM (the approach unit with the measuring head) should be placed on a separate table away from computers and monitors, to eliminate electromagnetic interference;

- the instrument should be protected from the direct impact of sun radiation;

- the operation of the instrument is susceptible to heat flows, draughts and sudden alternations in temperature and humidity.

4. Packing, Storage and Transportation

4.1. Storage Conditions

- Storage in regions of the Far North and equated localities should comply with requirements of the standards GOST P 52931-2008 and GOST 15846.

- Location of the instrument in a warehouse must provide easy access to it and free space for personnel.

- Instrument and their parts should be stored on shelves.

- At least 100 mm distance between the stored parts and walls and ceiling of the premises should be reserved.

- Distance between the stored parts and heating facilities of the premises should be at least 0.5 m.

4.2. Packing and transportation rules

- Packing of the instrument should be performed indoors in a ventilated room at ambient temperature of 15° C – 40 °C and relative humidity at most 80 % with no corrosive agent in the atmosphere. Storage conditions should comply with the standard GOST 15150.

- The instrument should be carried by one or several modes of enclosed transport according to transport regulations applicable to the mode(s) in service.

- Only transportation of the instrument in proper package is permitted to prevent transportation damage.

- The instrument in its package should be placed and secured in a vehicle in a way that provides steady position and excludes shocks of the deliverable parts by one another or by walls of the vehicle.

- Transportation conditions should comply with requirements of the standards GOST P 52931-2008 and GOST 15150.

- Transportation in regions of the Far North and equated localities should comply with requirements of the standards GOST P 52931-2008 and GOST 15846.

- Before transporting the measuring instrument, it should be fixed with transport brackets. For installation of the brackets refer to Appendix.

5. Setup and Installation

Usually installation of the SPM SOLVER NEXT is carried out by a field engineer. To install the SPM on your own, perform the following steps:

- Unpack SPM SOLVER NEXT. Remove transportation locks.
- Connect the electromechanical units of the SPM.
- Install the software as follows:
    - Copy the folder with the Nova Px program to the hard drive of your computer;
    - Install drivers of the SPM controller;
    - Install drivers of the video camera.
- Calibrate stepper motors.
- Adjust the video system.

These procedures are explained in details in Appendix.

6. Preparing for Measurements

This chapter explains the preparatory procedures required for AFM measurements. The sequence of those procedures includes the following steps:

1. Powering Sequence (see i. 6.1 on p. 19).
2. Mounting Sample (see i. 6.2 on p. 20).
3. Installing the Probe (see i. 6.3 on p. 24).
4. Initial Approach (see i. 6.4 on p. 30).
5. Selecting Relevant Area (see i. 6.5 on p. 33).
6. Adjusting the Optical Cantilever Deflection Detection System (see i. 6.6 on p. 35).

6.1. Powering Sequence

1. Turn the computer on.
2. Turn the vibration protection system on.
3. Turn on the SPM controller.

> ⚠️ **ATTENTION! All connectors should be tightened up before turning the instrument on. Disconnection of connectors during operation may cause damage to the electronic components.**

4. Start the Nova Px Control Program. The Program Main window appears on the screen (Fig. 6-1).
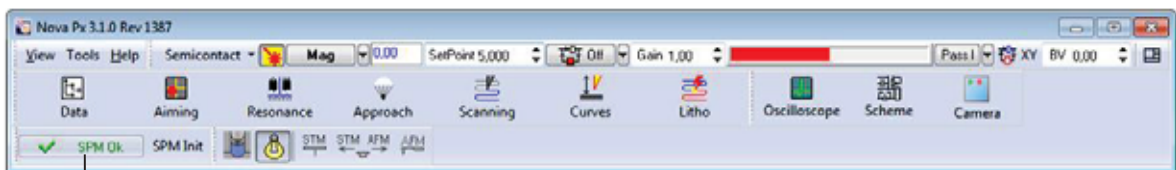
SPM state indicator

Fig. 6-1. The Program Main window

5. Operation of the Control program starts with initialization of the instrument. If initialization is successful, the [⬚ Initializing] indicator in the Initialization panel will change its appearance to [✓ SPM Ok].

If the initialization has not run automatically, launch it by clicking the [SPM Init] button.

6.2. Mounting Sample

The procedure on mounting the sample is very important. A special care should be paid to reliable fixation of the sample as it determines quality of the image.

Mounting Sample on Holder

The SPM SOLVER NEXT is equipped with a set of substrates and sample holders (Fig. 6-2). The substrates are of different design for specific research purposes. This section explains how to mount the samples on substrates of different types.
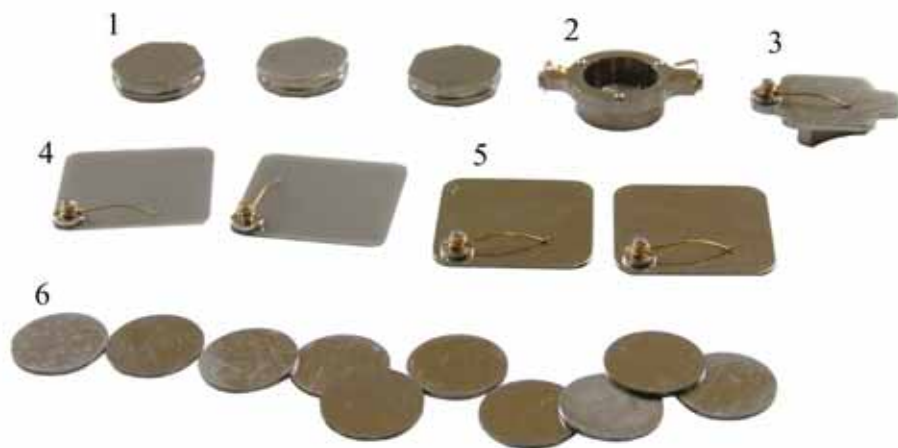
Fig. 6-2. Set of substrates and sample holders for SOLVER NEXT
1 – hexagonal magnetic sample holder; 2 – non-magnetic sample holder;
3 – STM sample holder; 4 – nonconducting substrate with a spring contact;
5 – conducting substrate with a spring contact; 6 – round metal substrate

In most cases, the round metal substrate can be applied. The sample is fixed on the round substrate either with a double-sided adhesive tape or with conductive paste (the latter is used for STM or electric AFM measurements). If the adhesive tape is used, do not start measurements for several minutes after fixation until sliding of the sample stops.

The round metal substrate with the sample on it is placed on the magnetic holder (Fig. 6-3). If the sample is too thin (thickness less 3 mm), a stack of two holders should be used.

Fig. 6-3. Round substrate with the sample (fixation with conductive paste) on a stack of two magnetic sample holders

Another option is to stick the sample immediately on the magnetic holder; however, this will need to unstick the sample every time when exchanging the sample.

Substrates with a spring contact (see 4, 5 in Fig. 6-2) are used in measurements on electric techniques (STM, Kelvin Probe Microscopy etc.). With these substrates, the sample can be fixed with a double-sided adhesive tape or pressed with the spring (if rear surface of the sample is smooth). It should be borne in mind that fixation with conductive paste or with the spring provides electrical contact between the sample and the substrate from both the top and the bottom of the sample.

A substrate with a spring contact is placed on a non-magnetic sample holder 2 (Fig. 6-4). As well, the metal substrate with a spring contact can be placed on a hexagonal magnetic sample holder 1.

Fig. 6-4. Sample fixed on a substrate with a spring contact with a double-sided adhesive tape, substrate placed on a non-magnetic holder

The STM sample holder 3 is designated for STM measurements with atomic resolution. In this case, the sample can be attached immediately to the holder, thus reducing noises.

Mounting Holder with Sample in SOLVER NEXT

1. If any of the measuring heads is in its working position at the moment of mounting/exchanging the sample, that head should be moved to its inoperative position by clicking the [STM AFM] button in the Measuring heads Control panel (Fig. 6-5).
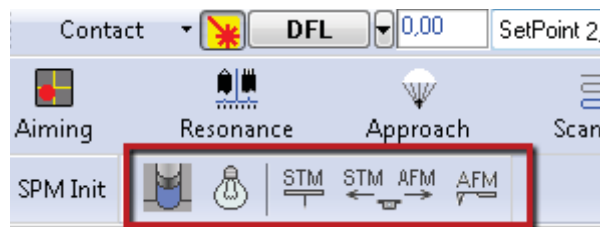
Fig. 6-5. Measurement Heads Control panel

2. Place the holder with the sample fixed on it on the sample stage of the scanner (Fig. 6-6, Fig. 6-7). If a substrate with a spring contact is used, check that the contact is at the side distant from you, as shown in Fig. 6-7.
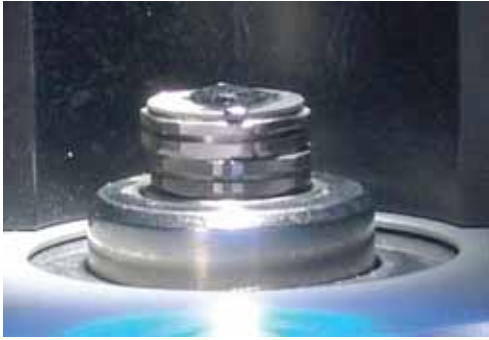
Fig. 6-6
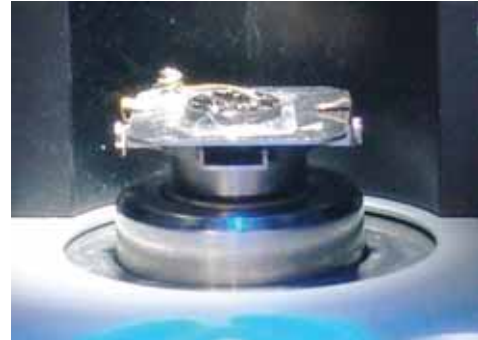
Fig. 6-7

Setting the sample height value

As the measurement procedure moves the sample in the course of scanning, distance between the sample surface and the sample stage of the scanner should be accounted for accuracy of X and Y data.

To this purpose, define the composite (including the substrate and the holder) sample height. For your convenience, heights of substrates and holders are given in the table below. A relevant value should be used in the Control program as the sample height.

Table 6-1. Heights of substrates and sample holders

Substrate/sample holder	Height
	3 mm
	6 mm
	4.5 mm
	0.5 mm
	0.5 mm

For setting the sample height in the program, perform the following steps:

1. Open the **ScanSettings** window by clicking the ![Settings...] button in the Scanning window (Fig. 6-8).
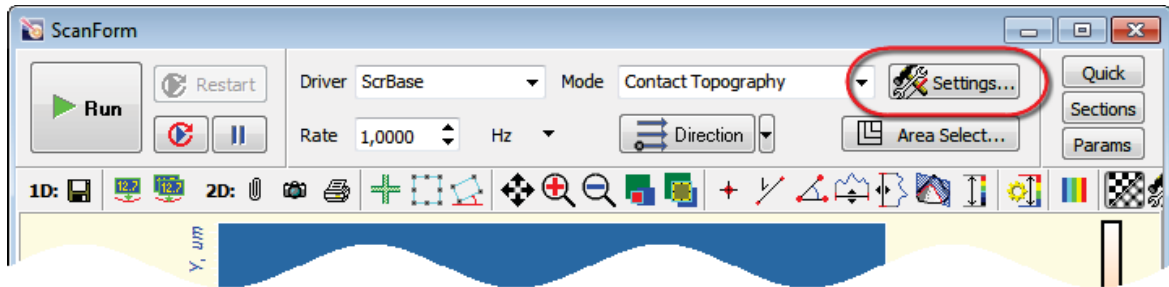
Fig. 6-8. Settings

2. In the **SampleHeight** input field (Fig. 6-9), enter the desired composite sample height.
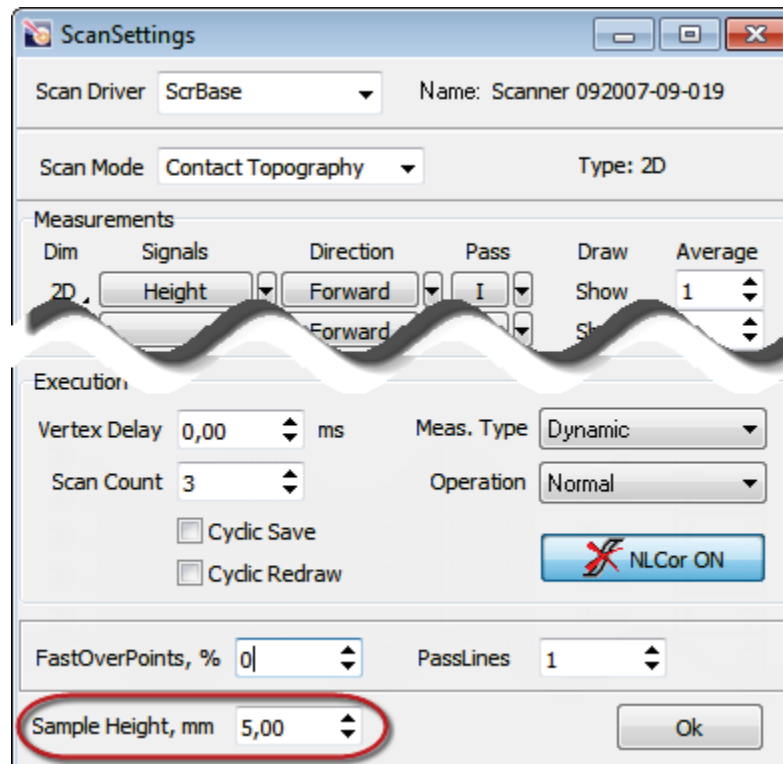
Fig. 6-9. Setting the sample height

3. Click the **OK** button to apply your modifications and to close the **ScanSettings** window.

The **SampleHeight** parameter should be modified every time when another value of the sample height is required.

6.3. Installing the Probe

ATTENTION! Before installing/exchanging the probe move the scanner down so that the sample does not obstruct advancing the measuring head.

For moving the scanner down, perform the following steps:

1. Open the **Approach** window (Fig. 6‑10) by clicking the [Approach] button in the Main Operations panel of the Main Program window.
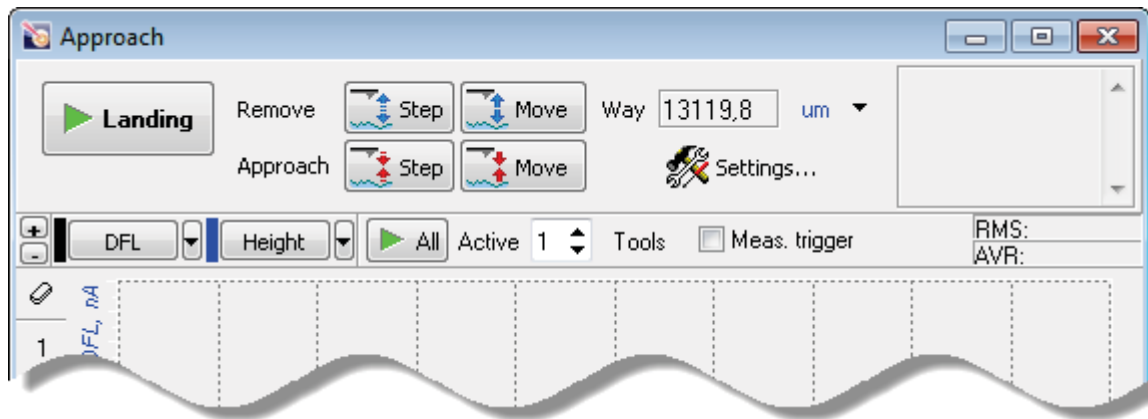
Fig. 6‑10. **Approach window**

2. Click the [Move] button several times. This will start descend of the sample. When it reaches its lowest position, the stepper motor will turn off and the message box «Backward Limited Achieved» will appear.
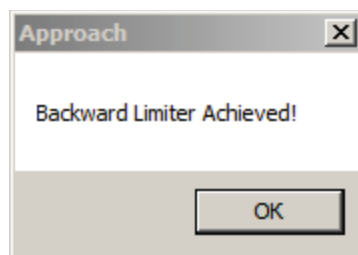
Fig. 6‑11

Regarding the selected measuring head, install an AFM or STM probe:

- Installing the AFM Probe – see p. 25;
- Installing the STM Tip – see p. 27.

6.3.1.   Installing the AFM Probe

For installing or exchanging an AFM probe, perform the following steps:

1. Move the AFM head to its position for exchanging the probe. To this purpose, perform the following steps:

    a) Click the ![AFM] button in the Measurement Heads Control panel (Fig. 6-5). This will move the AFM head to its working position (Fig. 6‑12).

    b) Turn the measuring head to you manually (Fig. 6‑13).
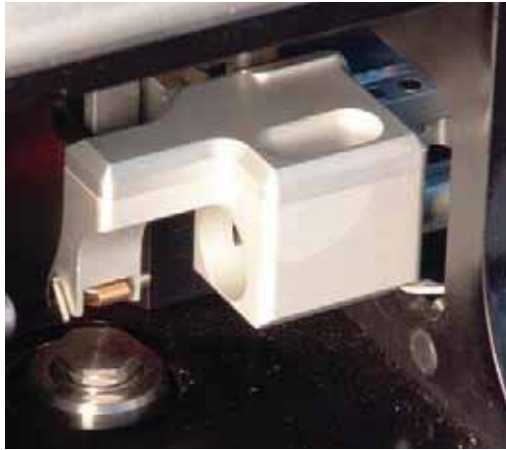
Fig. 6‑12
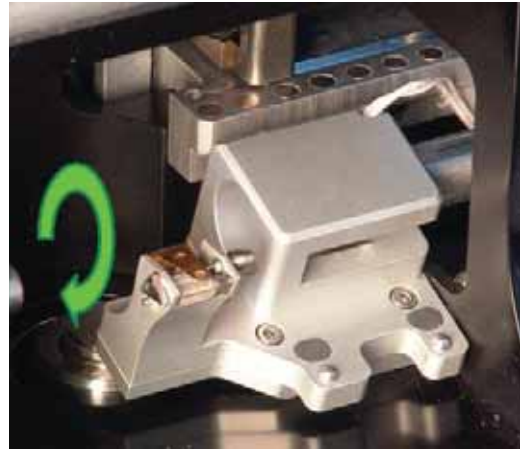
Fig. 6‑13

2. Release the probe clip spring by turning downward the trapezoidal lever, located on the outer side of the holder body, with a tweezers (see Fig. 6-14).

Fig. 6-14

Fig. 6-15

3. Using tweezers, take a probe from the container (see Fig. 6-15). Probes in the container are placed with their tips pointing upwards. Do not turn the chip over during the installation.

4. Place the probe on the sapphire pedestal to the left of the working position (see Fig. 6-16, Fig. 6-17) which is in the right corner under the clip.

Fig. 6-16
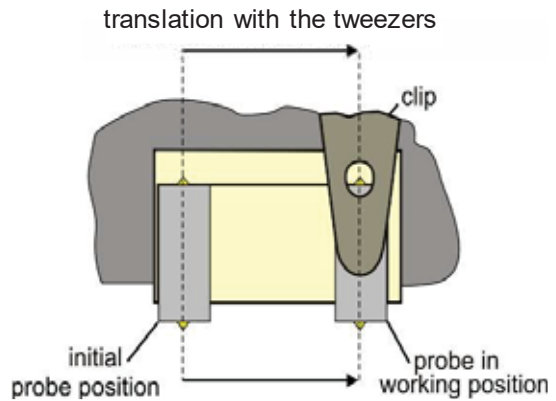
Fig. 6-17

5. Move the probe with the tweezers to the working position (see Fig. 6-18) as shown schematically in Fig. 6-17. The probe at its working position is shown in Fig. 6-19.

Fig. 6-18
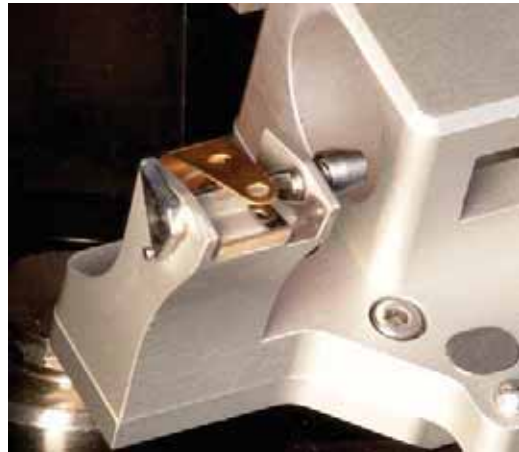
Fig. 6-19

6. Once the probe is in the working position, clamp it with the clip by turning the lever counterclockwise to extreme position with the tweezers (see Fig. 6-20).

7. Turn the measuring head to its working position manually (see Fig. 6-21).

Fig. 6-20

Fig. 6-21

AFM probe is now installed.

6.3.2. Installing the STM Tip

Manufacturing the Tip

The STM tip is the sharpened end of a piece of platinum-iridium (**PtIr**), or platinum-rhodium (**PtRo**) (with platinum content of about 80 %) or tungsten (**W**) wire, 8÷10 mm in length with a diameter of 0.25÷0.5 mm.

The sharpness of the tip can be estimated by imaging a reference sample with known surface characteristics, for example, Highly Oriented Pyrolytic Graphite (HOPG).

There are two techniques of manufacturing an STM tip:

1. By cutting the wire apex with scissor (**PtIr**, **PtRo**) (see below).
2. By electrochemical etching (**W**, **Pt**, **PtIr**, **PtRo**).

The simplest STM tip manufacturing technique is cutting the wire apex with the scissors. This provides the apex radius of curvature less than 10 nm.

Sharp-edged scissors and tweezers with kinks on the interior surface (see the toolkit supplied with the SOLVER NEXT SPM) are used to cut the wire.

ATTENTION! Do not use the wire cutting scissors for other purposes.

Fig. 6-22

Apex forming procedure includes the following steps:

1. Clamp the wire with the tweezers so that it projects beyond its edge for 2÷3 mm (see Fig. 6-22).
2. Cut the wire at an angle of 10÷15 degrees as close to its apex as possible and simultaneously pull the scissors along the wire axis to separate the part being cut off. This is required to avoid the contact between the cutting edges of the scissors and the tip apex. Actually, the wire is rather torn off than truncated at the last moment of contact with the scissor. This results in a sharp apex, formed at the wire's end (see Fig. 6-23).

Fig. 6-23. Typical shape of a wire cutoff

3. Check the resulting cutoff shape using the optical microscope with 200-x magnification, if possible. Repeat the cutting process, if necessary.

ATTENTION! Avoid any contact with the tip apex in order not to damage it.

4. After cutting the wire you may anneal its apex in the flame of an alcohol lamp for 1÷2 seconds to remove organic substances. Check the apex of the tip using the optical microscope. The cutoff section should be seen bright, with no trace of carbon-black or dust.

   NOTE. The overall length of the tip should not exceed 10 mm.

Installing the Tip into the Holder

Use of two tweezers enables installing the STM tip into the holder.

To install or exchange the STM tip, perform the following steps:

1. Move the STM head to its position for exchanging the probe. To this purpose, perform the following steps:

   a) Click the STM button in the Measurement Heads Control panel (Fig.). This will move the STM head to its working position.

   b) Turn the measuring head to you manually (Fig. 6-24).
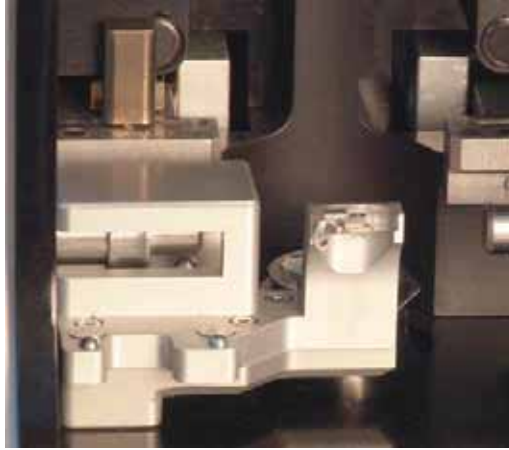
Fig. 6-24. STM head in position for exchanging the probe

2. Take a pair of tweezers. Hold the tip with the first tweezers and release the probe clip spring with the second tweezers (see Fig. 6-25).

ATTENTION! Avoid contacting the sharpened apex with any surfaces during installation.

3. Insert the tip's blunt end into the holder so that the sharpened end projects beyond the edge of the holder to distance of less 3÷4 mm (see Fig. 6-26).

Fig. 6-25
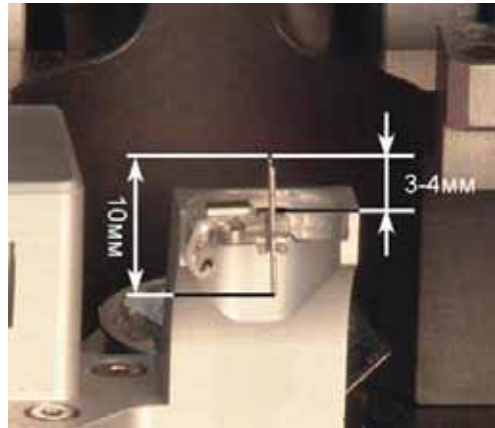
Fig. 6-26

4. Release the spring. Make sure that the tip is fixed firmly in the holder.

   NOTE. Tip sharpness and firmness of the tip fixation in the clamp are the primary factors determining the quality of STM results.

5. Turn the measuring head to its working position manually (see Fig. 6-27).
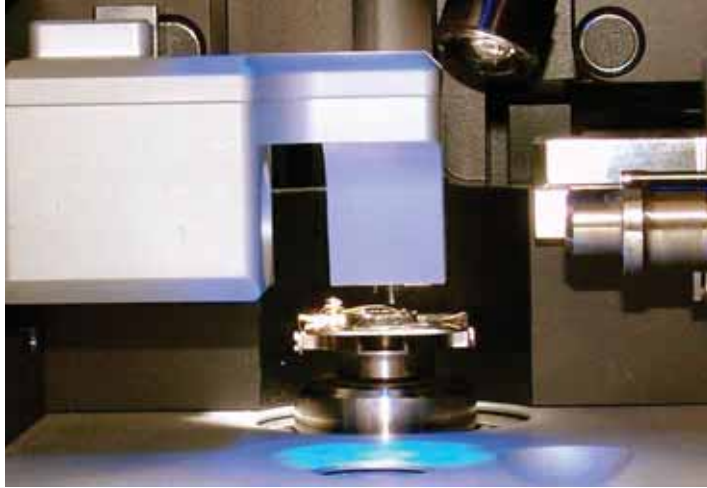
Fig. 6-27. STM head at its working position

STM tip is now installed.

6.4. Initial Approach

Preliminary approach is used for selecting a relevant scan area with the videomicroscope. The sample-probe distance at this stage should be 1÷2 mm.

To perform the initial approach, perform the following steps:

1. Open the **Approach** window (see Fig. 6-28) by clicking the [Approach] button.
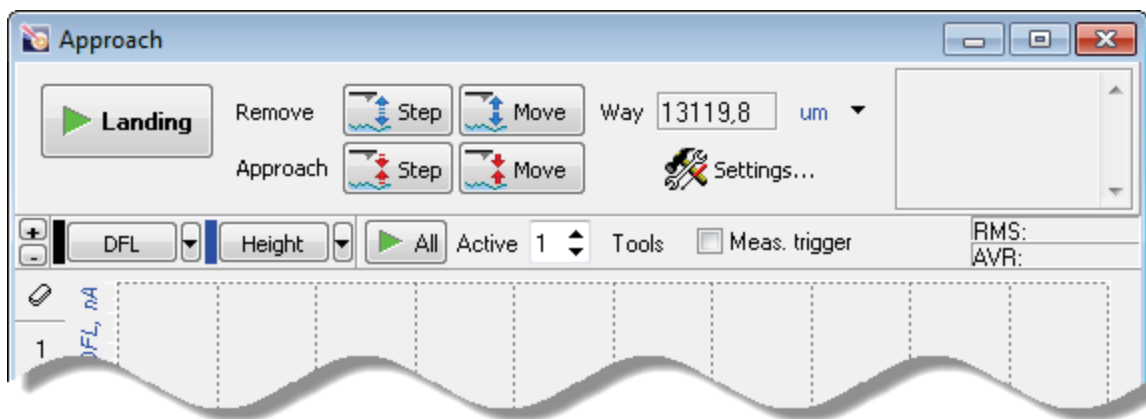
Fig. 6-28. **Approach** window

2. Open the **Approach Settings** window by clicking the [Settings...] button in the Control panel of the **Approach** window.
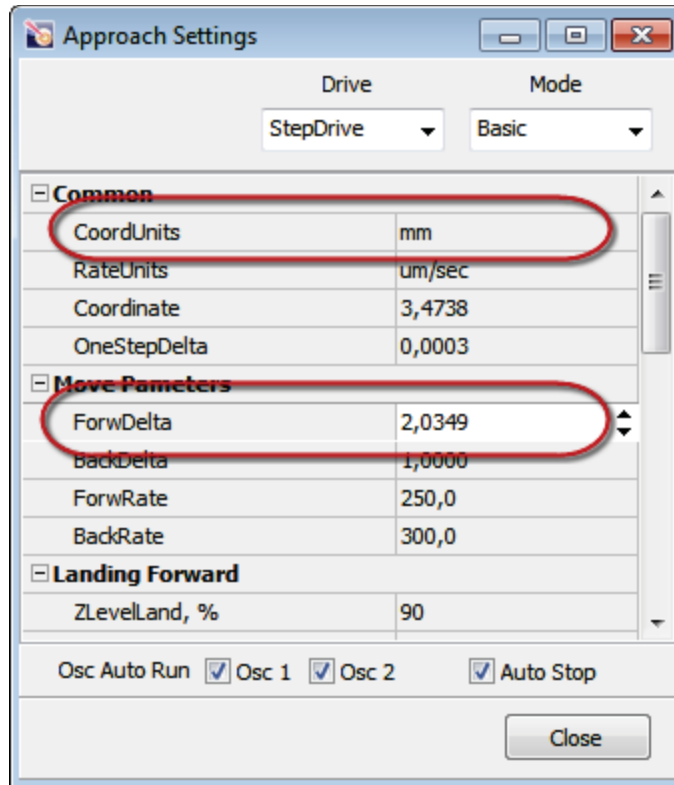
Fig. 6-29. Defining the sample-probe distance

3. Estimate distance between the probe tip and the sample surface. In the **ForwDelta** field of the **Move Parameters** group of the **Approach Settings** window, enter a value clearly less than the estimated distance. Unit of distance measurement is selected in the **CoordUnits** field of the **Common** group. Close the **Approach Settings** window.

   NOTE. During the initial approach keep the protective shield lifted to enable visual inspection of the probe-surface distance.

4. Launch approaching the probe by clicking ![Move] button. A message window (Fig. 6-30) will appear. To continue the approaching process, click the **Yes** button in this window. The sample will move up until the distance defined in the **Approach Settings** window is reached. Watch the approaching process on the image provided by the videomicroscope.
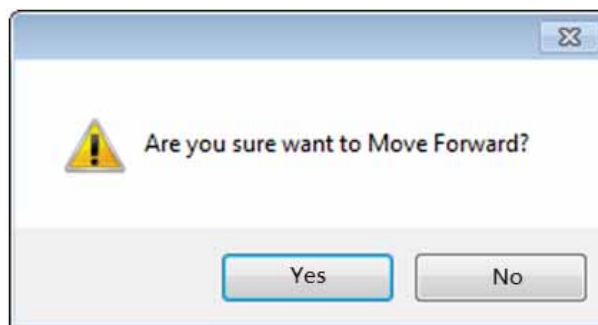
Fig. 6-30. Message window

5. On completion of approach, check the distance between the probe tip and the sample surface. If this distance is larger 2 mm, repeat steps 2-4.

Fig. 6-31. The distance between the probe tip and the sample surface is 1–2 mm

6. Move down the protective shield by clicking ![button] button.
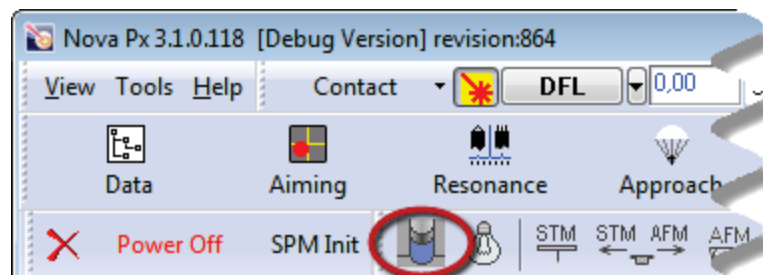
Fig. 6-32

6.5. Selecting Relevant Area

Selection of the relevant area is facilitated with the **Camera** window that provides a video image of the sample along with the probe tip.

For selecting the relevant area, perform the following steps:

1. Open the **Camera** window (Fig. 6-33) by clicking the ▣ Camera .
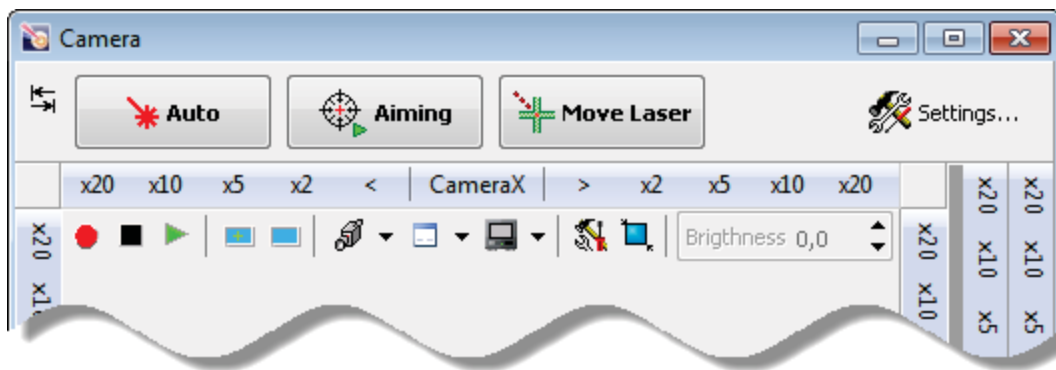
Fig. 6-33. Video image from the videomicroscope

To enable viewing image form the video camera in the **Camera** window, the video camera of the built-in videomicroscope of the SPM SOLVER NEXT should be selected as the active device at the start of the Control program. To this purpose, click the ▣ ▾ button and select DFx 31BF03 in the drop-down list (see Fig. 6-34).
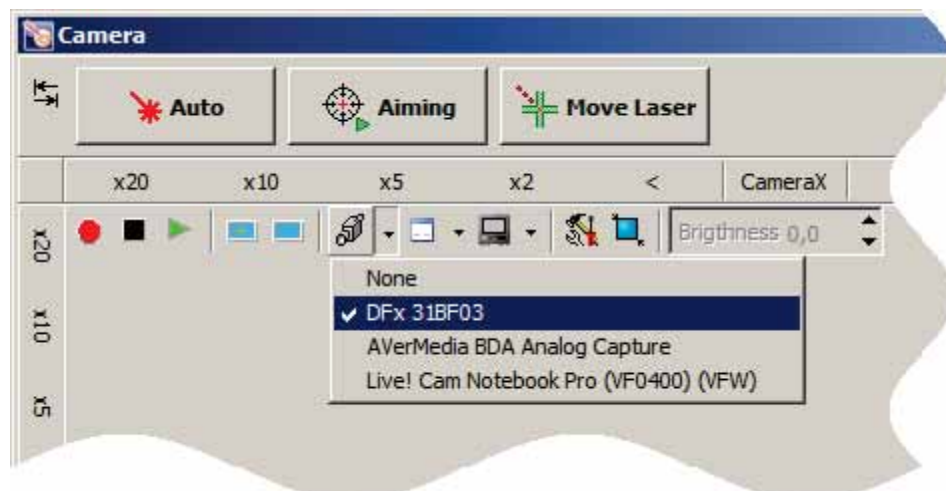
Fig. 6-34. Activating the video camera

2. Enable viewing by clicking the ▶ button. An image of the sample surface and of the probe will appear in the window (Fig. 6-35).
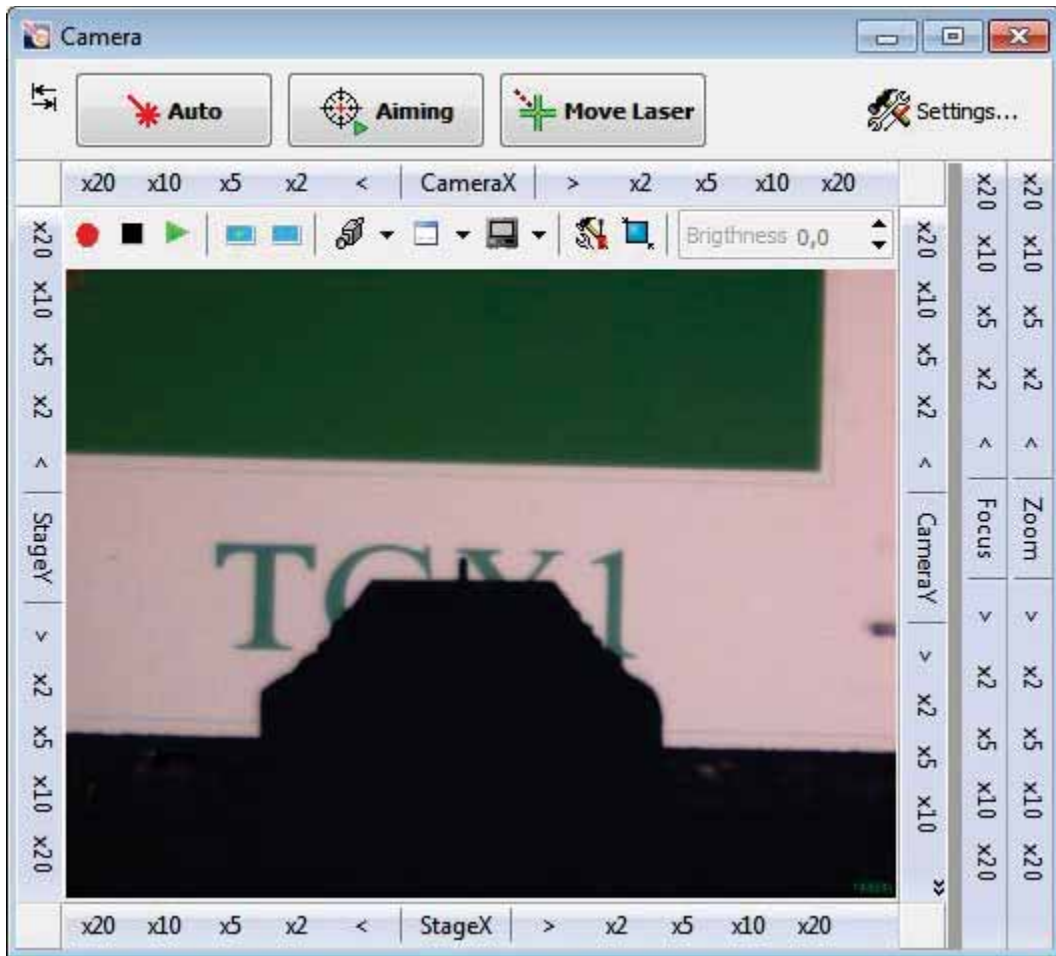
Fig. 6-35. Video image from the videomicroscope

3. Focus the videomicroscope on the sample with controls of the **Focus** panel if necessary.

4. Using the sample image (Fig. 6-35), find a relevant area for investigation. Move the sample so that the selected area is under the probe tip. The sample can be displaced in one of the following ways:

   ● using controls of the panels **StageX** and **StageY**;

   ● with the joystick[1];

   ● by catching a desired point in the video image as follows:

   a) press the <Shift> key and click at the image of the cantilever edge;

   b) press the <Ctrl> key and click on the desired point in the sample image. This will result in moving the sample so that the selected point comes under the probe tip.

___

[1] The joystick is not included in the standard delivery set of SPM SOLVER NEXT and can be purchased as an optional component.

6.6. Adjusting the Optical Cantilever Deflection Detection System

The procedure on adjusting the optical cantilever deflection detection system (hereafter, the detection system) is required only for the AFM head. If STM measurements are to perform, go to the procedure on preliminary approach (i. 6.4 «Initial Approach» on p. 30).

The procedure on adjusting the detection system consists of aiming the laser beam at the cantilever and positioning the photodiode so as the laser beam reflected by the cantilever comes to the center of the photodiode and illuminates uniformly four sections of the photodiode. The SPM SOLVER NEXT is equipped with the Adjusting Detection System in Automatic Mode (EXPERTFBA™ system).

NOTE. The laser of the detection system can be enabled only when the shield is in its lowest position. If the protective shield is above that position, the laser will be disabled even with the activation button being pressed in.
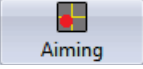
The Nova Px program provides two options for adjusting the detection system:

- by videoimage (see i. 6.6.1 on p. 37);
- using the **Laser-Diode Scanning** window (see i. 6.6.2 on p. 38).

The option with the videoimage is preferable in most cases because it is much easier and faster.

The option with the **Laser-Diode Scanning** window can be required, for example, when scanning is performed with a probe with two or more cantilevers or when higher accuracy of position of the laser spot on the cantilever is necessary.

The optimal position of the photodiode depends on the sample environment (air or liquid) due to refractive index of the environment. So, before starting the procedure on adjusting the detection system, always check which environment was previously selected. To this purpose, perform the following steps:

1. Open the **Aiming Movers** window by clicking the [Aiming] button in the Main Operations panel.
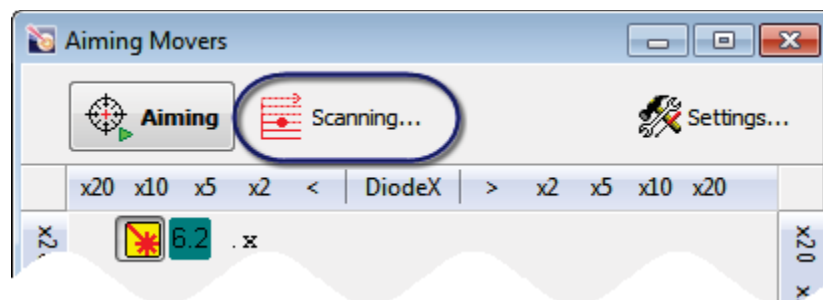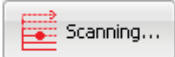
Fig. 6-36. **Aiming Movers** window

2. Open the window for adjusting the detection system (Fig. 6‑37) by clicking the [Scanning...] button in the Control panel of the **Aiming Movers** window.
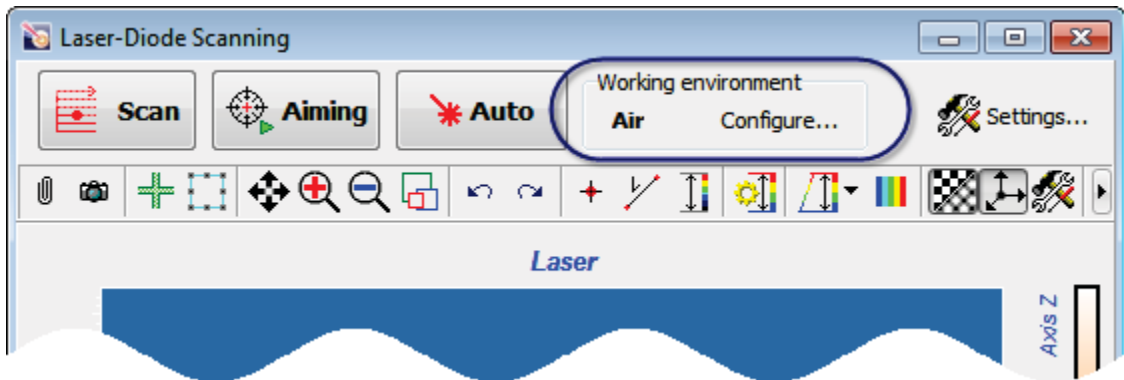
Fig. 6‑37. **Laser-Diode Scanning** window for adjusting the detection system

3. The selected operating environment (**Air** or **Liquid**) is displayed in the **Working Environment** panel. For changing the environment for adjusting the detection system, perform the following steps:

   a) Open the **Configure** window (Fig. 6‑38) by clicking the [Configure...] button.
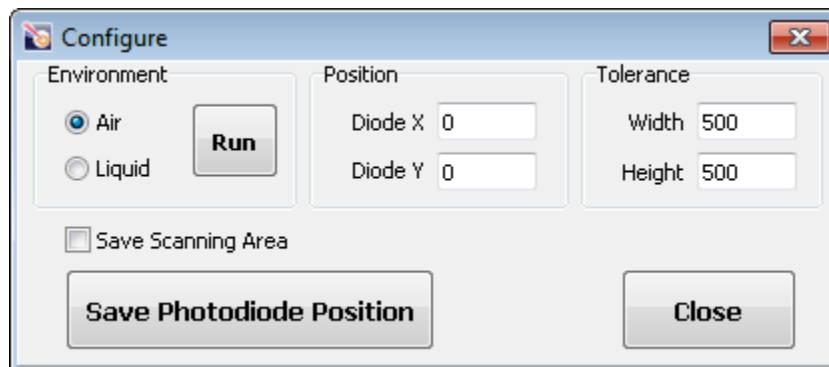
Fig. 6‑38. **Configure** window

   b) In the **Environment** panel of the newly opened window, select the desired option (**Air** in our example). Then click the **Run** button. This will automatically define limits for searching for the cantilever in the selected environment. The photodiode will be repositioned to the optimal position.

   c) Close the **Configure** window.

If the videoimage will be used for adjusting, the **Laser-Diode Scanning** window can be closed.

6.6.1. Adjusting Detection System by Videoimage

1. Verify that **Contact** or **Semicontact** methods are selected in the Main Parameters panel.
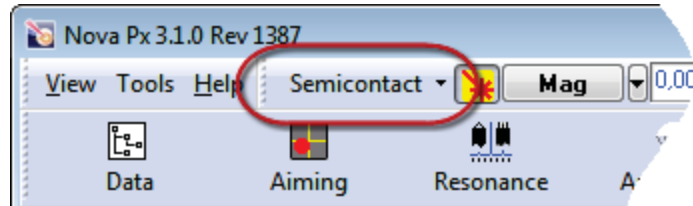
Fig. 6‑39

2. Open the window for viewing the videoimage by clicking .

3. Start displaying the videoimage by clicking button in the Control panel of the **Camera** window. This will result in viewing the image of the sample surface and of the probe in the Image displaying area (Fig. 6‑40).
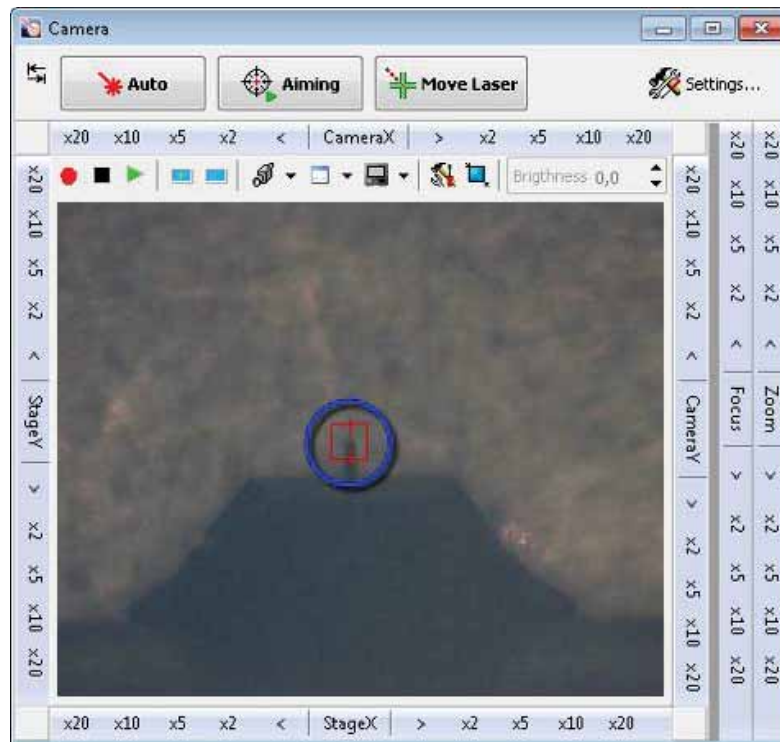
Fig. 6‑40. **Camera** window

4. Using controls of the panels **CameraX** and **CameraY**, move the videomicroscope so as the image of the cantilever comes to the center of the video frame.

5. If the image is unfocused, focus the video camera on the edge of the cantilever with controls of the **Focus** panel.

6. Mark position of the cantilever edge in the image by clicking on the desired point with left mouse button with the <Shift> key being pressed (Fig. 6‑40). This will display a red cross-hair at the selected point.

7. Start the procedure on adjusting the detection system by clicking the [✳ Auto] button. The procedure will align the laser for the laser beam to fall on the cantilever edge and reposition the photodiode so as the laser beam reflected by the cantilever comes to the center of the photodiode.

In the course of adjusting, watch the laser signal in the **Aiming Movers** window (opens by clicking the [Aiming] button). If the procedure fails to adjust the detection system, recalibration of stepper motors of the SOLVER NEXT is required.

8. The adjusting procedure completes with a message box (Fig. 6‑41). Close this box by clicking the **OK** button.
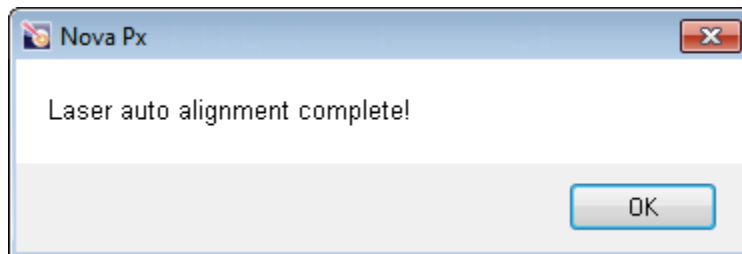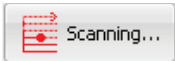
Fig. 6‑41. Message on completion of adjusting the detection system

When automatic adjusting the detection system completes, verify that the laser beam is reflected by the cantilever and not by the chip. Reflection by the cantilever produces the **Laser** signal in the range of 5–30 nA. If level of the **Laser** signal is about 50 nA, the laser beam is reflected by the chip. For better viewing of the laser spot, turn off illumination (by clicking the [💡] button).

6.6.2. Adjusting Detection System through Laser-Diode Scanning Window

The procedure on adjusting the detection system with the **Laser-Diode Scanning** window can be required, for example, when scanning is performed with a probe with two or more cantilevers or when higher accuracy of position of the laser spot on the cantilever is necessary.

The **Laser-Diode Scanning** window opens by clicking the [Scanning...] button in the Control panel of the **Aiming Movers** window.

The Control program keeps parameters of the latest area of searching for the cantilever. If measurements are to perform with the same probe, these parameters can be used without modification. If the probe was exchanged, scanning of the whole area is recommended to find position of the cantilever.

1. For defining parameters of the area for searching for the cantilever (hereafter, the search area), perform the following steps:

    a) Enable modifications of size and position of the search area by clicking the ⬚ button in the Control panel of the **Laser-Diode Scanning** window (Fig. 6‑42).
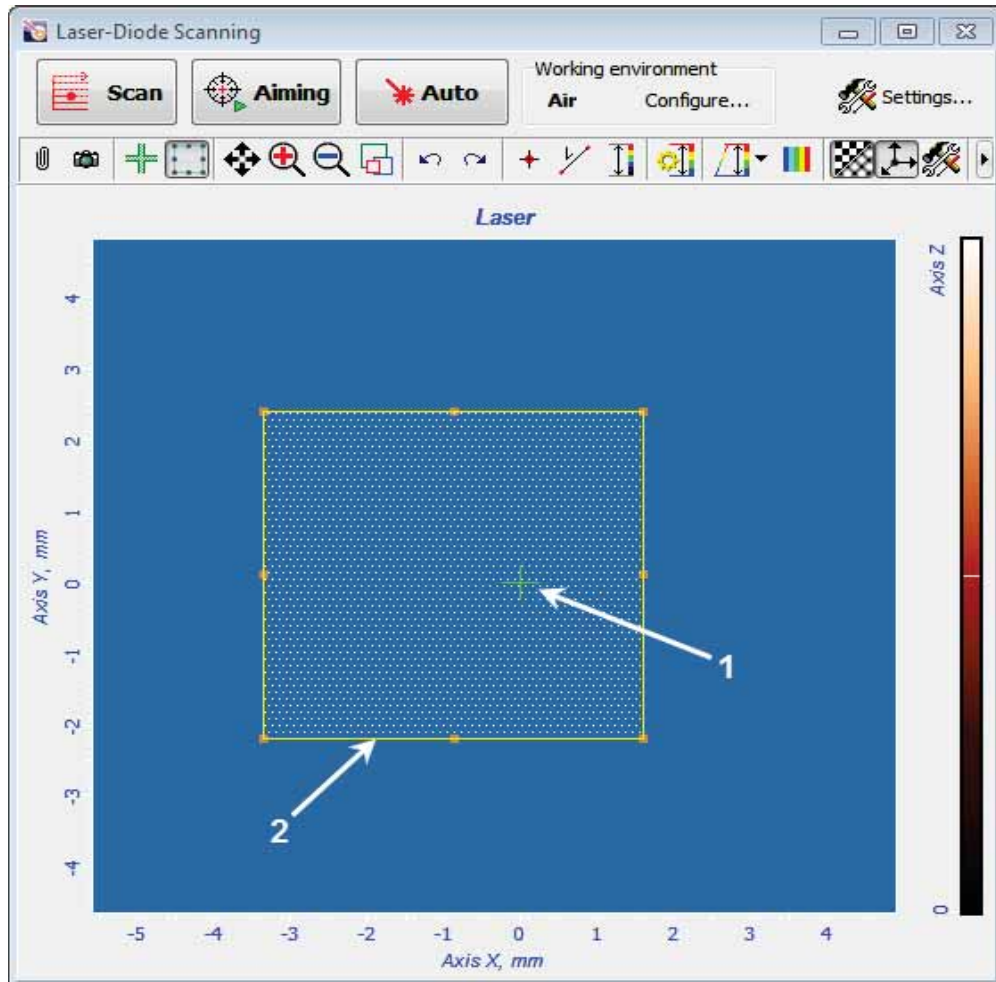
Fig. 6‑42. **Laser-Diode Scanning** window
1 – laser spot pointer; 2 – limits of the search area

    b) Change size and position of the scan area with the mouse (pos. 2 in Fig. 6‑42).

2. Start measurements of laser intensity by clicking the [Scan] button. This will initiate line-by-line sweep of the laser spot inside the predefined search area with parallel measurement of laser intensity by the photodiode. The 2D Data Viewer will collect the image line by line.

    The measurements will produce one of the two possible results:

    • The reflected laser beam comes to the photodiode (see Fig. 6‑43). Go to step 8 and perform fine search for position of the laser to maximize the laser signal.

    • The reflected laser beam misses the photodiode, the image is completely dark. This can be due to that the cantilever is outside the selected search area or the laser beam reflected by the cantilever is not pointed to the photodiode. In this case increase limits of the search area or reposition it (see ii. 4-7).
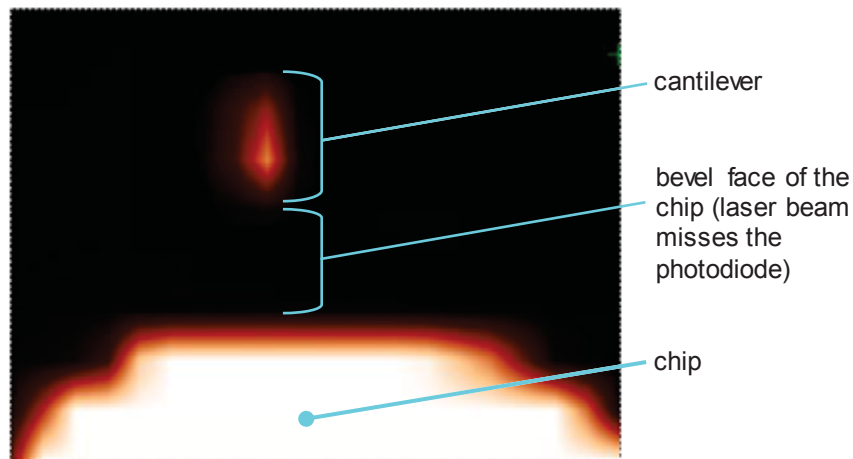
Fig. 6‑43. Laser intensity distribution in the search area

3. Make the search area narrower so as it covers only the laser spot on the cantilever.

4. Start the procedure on adjusting the detection system by clicking the ❋ Auto button. This will initiate line-by-line sweep and measurement of the laser intensity. After the measurements complete, a point of maximum laser intensity will be determined. The laser beam will be aimed at that point. The photodiode will be repositioned for the laser beam to fall to the center of the photodiode.

The procedure on adjusting the detection system completes with a message window «Scan process finished».

7. Contact AFM

7.1. Constant Force Mode

Operations performed in measurements of surface topography with the Constant Force Mode have much in common with those of other contact techniques, including the Lateral Force Mode, the Spreading Resistance Imaging Mode, the Force Modulation Mode, and the Contact Error Mode.

The measurement procedure with constant force technique will be explained with an example of scanning of a calibration grating TGQ1 with a probe CSG10.

Initial state

The following steps are assumed to be done:

- Launching the control program;

- Turning the SOLVER NEXT on;

- Installing the probe;

- Adjusting the cantilever deflection detection system;

- Mounting the sample;

- Moving the AFM head to its working position;

- Initial approach;

- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

Procedural Sequence

Measurement in the Constant Force mode is arranged in the following procedural sequence:

1. Switching the Instrument for Operating in the Contact Modes (see p. 42).

2. Testing Initial Level of the DFL (see p. 42).

3. Approaching the Sample to the Probe (see p. 44).

4. Adjusting Working Level of the Feedback Gain (see p. 46).

5. Adjusting Scanning Parameters (see p. 47).

6. Scanning (see p. 55).

7. Saving Measurement Data (see p. 59).

8. Operation Completing (see p. 60).

These procedures are explained below.

7.1.1. Switching the Instrument for Operating in the Contact Modes

Switch the SOLVER NEXT for operating in the Contact technique by selecting **Contact** in the controller configuration list (see Fig. 7-1).
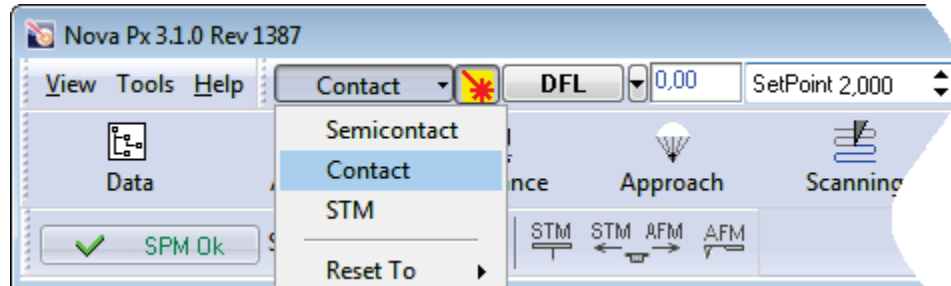
Fig. 7-1. Switching SPM for operating in the Contact modes

Once the configuration **Contact** is set, all switching sequences required for the instrument to operate in contact modes proceed automatically.

The following adjustments of the system will take place:

- Input of the feedback loop – **DFL**;
- Level of the **SetPoint** parameter (proportional to the force the probe presses the sample) – 2 nA;
- Gain of the feedback loop **Gain** – 10.

7.1.2. Testing Initial Level of the DFL

Initial level of the **DFL** signal that corresponds to the cantilever being free, is defined by positioning the photodide with respect to the laser beam reflected from the cantilever. Zero value of **DFL** implies that the laser spot is symmetrically distributed between the upper and the lower halves of the photodide.

This condition is optimal for measurements and so the system should be adjusted for the **DFL** signal to be 0. To this purpose, perform the following steps:

1. Open the **Aiming Movers** window by clicking the [Aiming button].

2. Verify, that the laser spot is at the center of the photodiode indicator and **DFL** is equal to zero (Fig. 7-2).
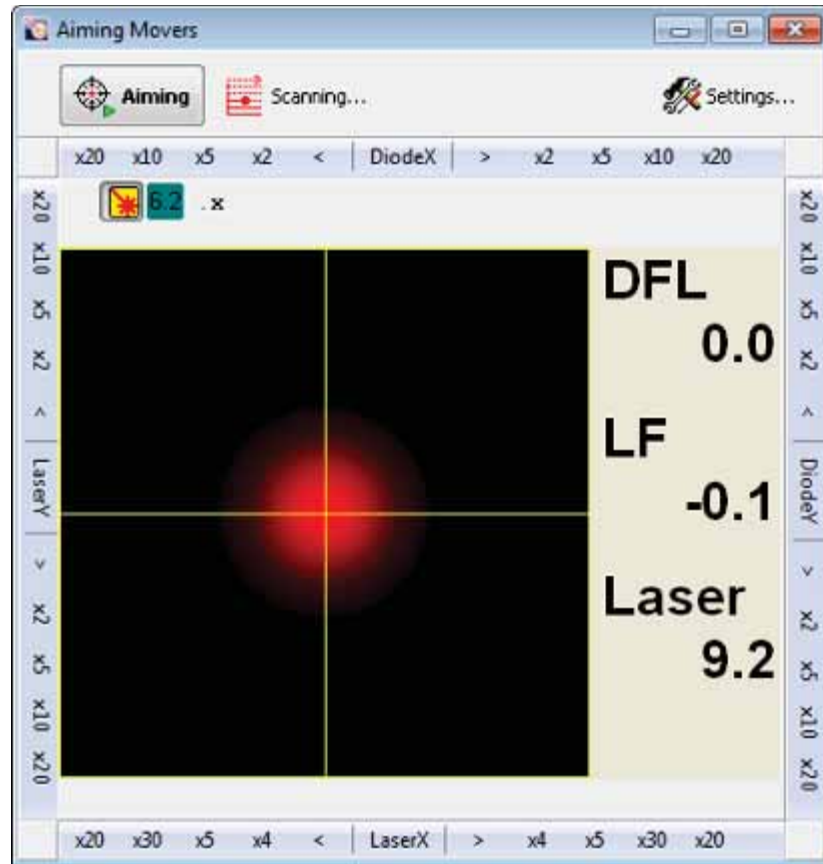
Fig. 7-2. Laser beam is directed to the center of the photodiode

3. If **DFL** deviates from zero, start the procedure on adjusting position of the photodiode by clicking the [⊕ Aiming] button. This will result in:

- the photodiode movement motors will start;
- the photodiode will be repositioned so that the laser spot will be distributed uniformly on the photodiode. The **DFL** value will be close to zero.

When the adjusting procedure completes, the [⊕ Aiming] button will be released.

7.1.3. Approaching the Sample to the Probe

To approach the sample to the probe, perform the following steps:

1. Click the [⚙ Off] button in the top bar to close the feedback loop (the button will change its appearance to [⚙ On]). This starts extension of the Z scanner to its maximum. Progress of the extension is displayed in the Scanner extension indicator (see Fig. 7-3) by degree of the colored filling.
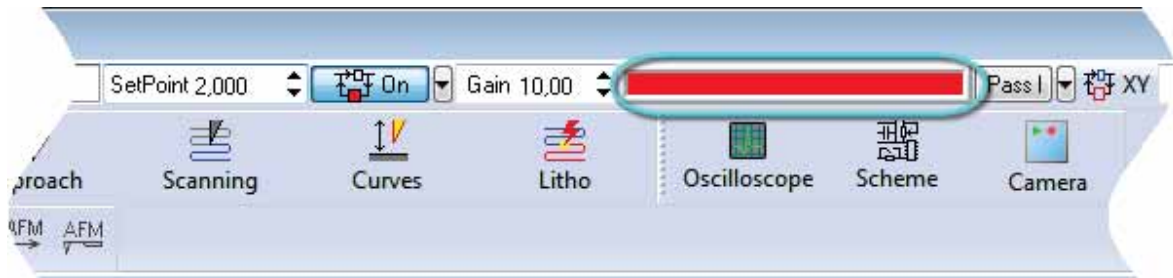
Fig. 7-3. Scanner reached its maximum extension

2. Open the **Approach** window by clicking the [Approach] button.

3. Start the approach procedure with the [▶ Landing] button (see Fig. 7-4).
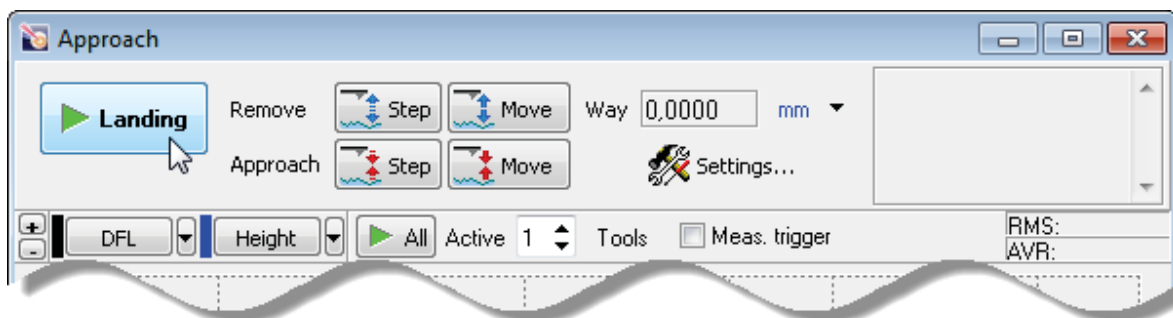
Fig. 7-4. Control panel of the **Approach** window

During the approach procedure, observe variations of the **DFL** signal in the software oscilloscope of the **Approach** window and state of the scanner extension indicator. Wait until the procedure completes. For viewing the profile of the **DFL** signal, the button with its name should be pressed in (Fig. 7-5).

Providing the approach parameters are set correctly, the approach procedure completes in about 10 to 30 seconds and the system goes to following state (see Fig. 7-5):
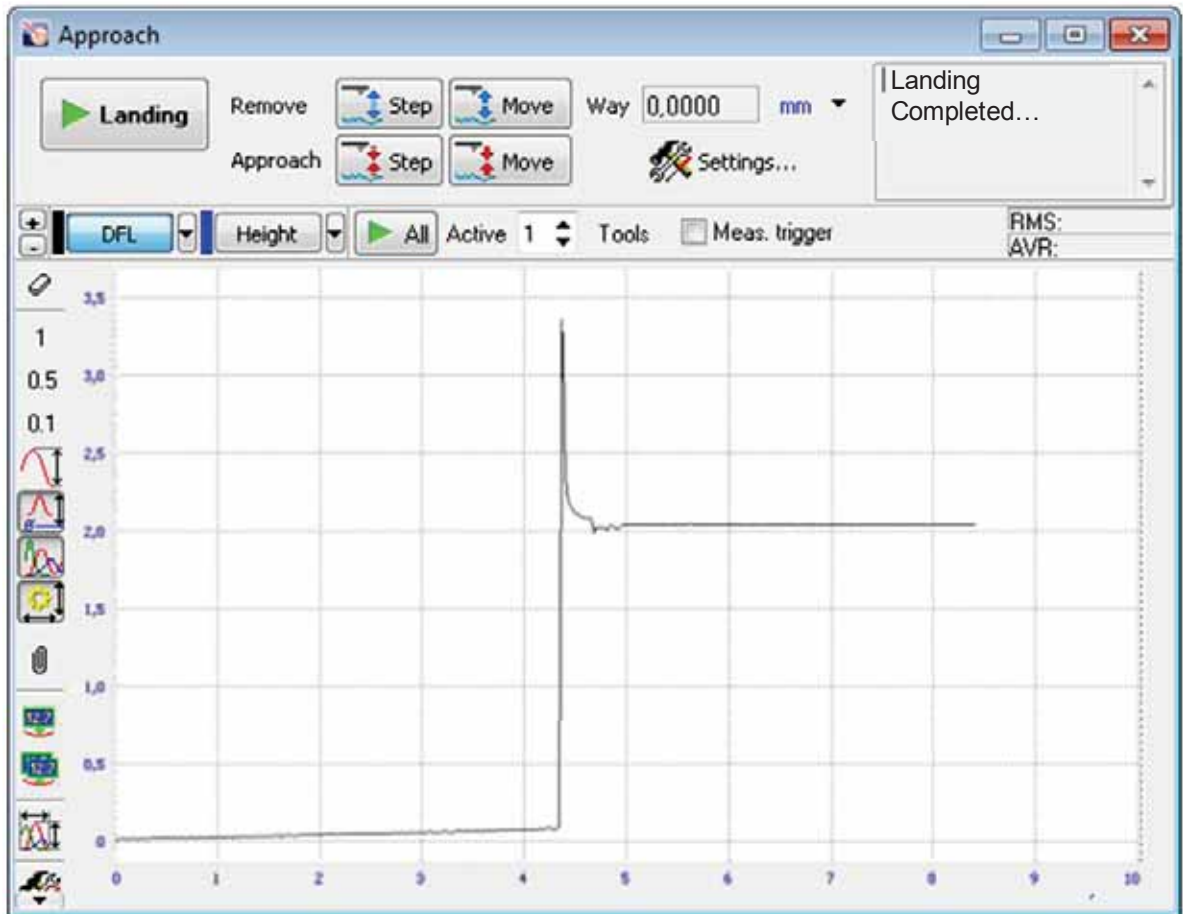
Fig. 7-5. Approach completed

- The signal **DFL** increases to the level of **Set Point** parameter, the feedback loop maintains the Z-scanner in the position where the **DFL** value is equal to **Set Point**. Note that this position of the scanner is approximately equal to half of the scanner extension range;

- The message "**Landing Completed**" appears at the right top of the Approach window.

- The stepper motor is disabled;

- Length of the indicator bar decreases down to some intermediate value (see Fig. 7-6).

Fig. 7-6. Scanner extension indicator after approaching

7.1.4.   Adjusting Working Level of the Feedback Gain

The greater is the level of the feedback gain (**Gain** parameter), the greater is data processing speed in the feedback loop. Nevertheless, at some high level of the feedback gain (let us call it threshold value), the mode of operation of the feedback loop becomes unstable and noise generation occurs. Under this condition, the **DFL** signal is supplied with a significant alternating component (see Fig. 7-7).
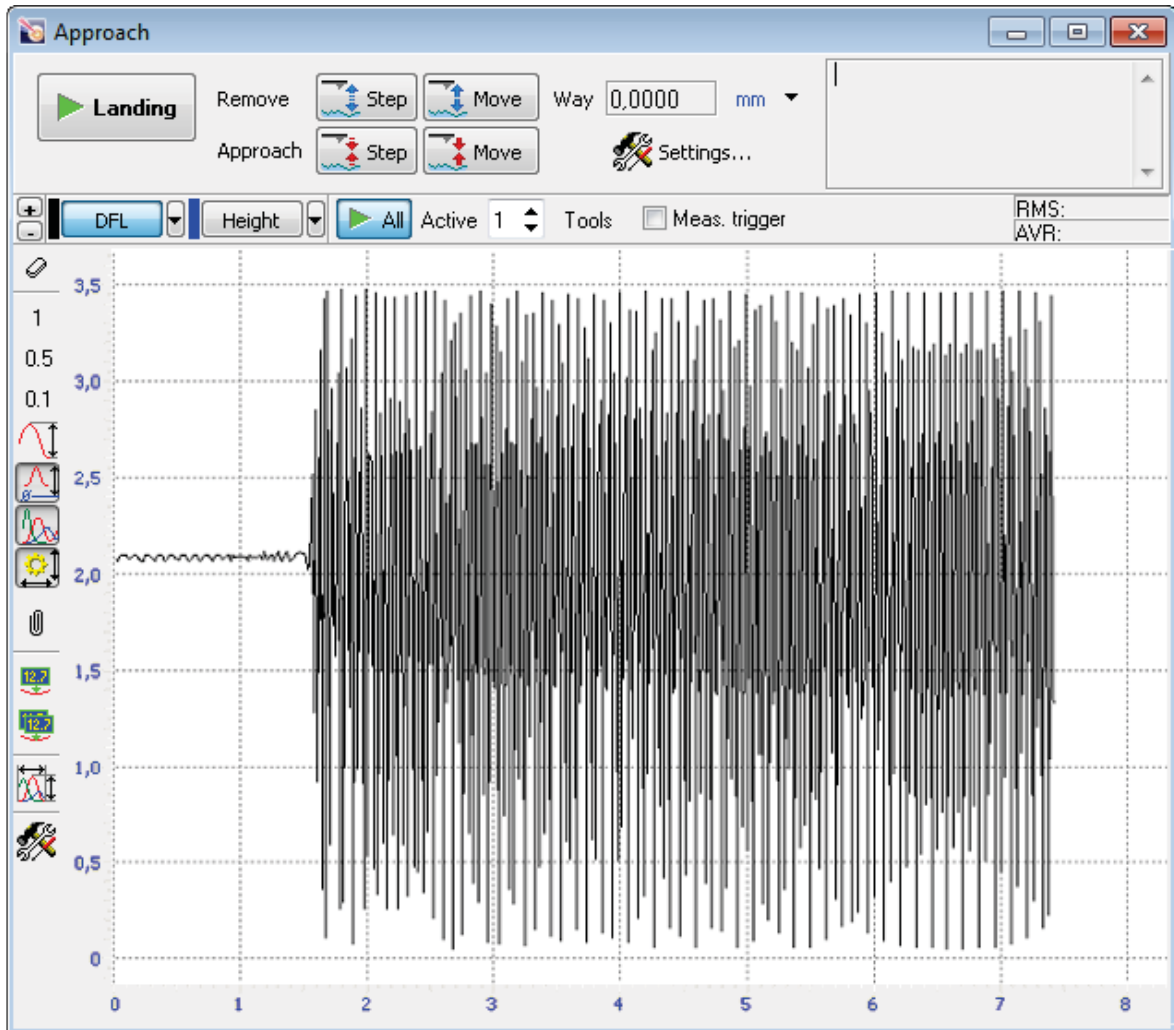
Fig. 7-7. Noise generation in the feedback loop

For stable operation, it is recommended to define the feedback gain to less 0.5÷0.7 of the threshold value where the noise generation occurs. The feedback gain is adjusted in the **Gain** input field.

To adjust the working level of the feedback gain, perform the following steps:

1. Double-click the **Gain** input field in the Main Parameters panel. The adjusting slider of the feedback gain will appear (see Fig. 7-8).
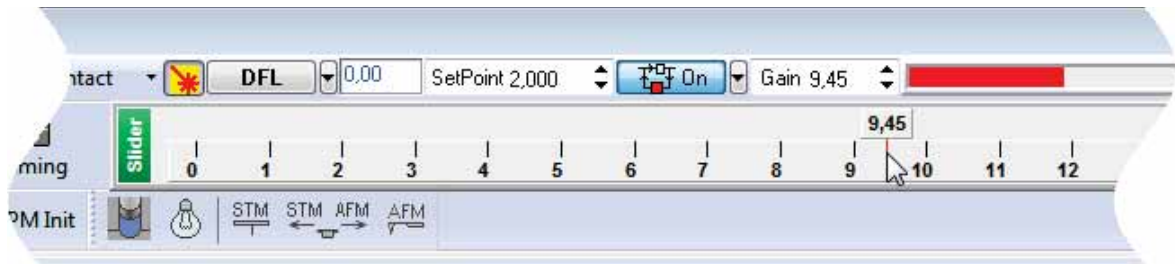
Fig. 7-8. Adjusting slider of the **Gain** parameter

2. Gradually decrease the **Gain** level and watch the **DFL** level in the software oscilloscope.

3. Determine the value of the **Gain** parameter at which noise generation occurs. The beginning of the generation is identified by appearance of a noticeable alternating component in the **DFL** signal (see Fig. 7-7).

4. Decrease the value of the **Gain** parameter to 0.5÷0.7 of the threshold. This value will be used in further operation.

7.1.5.   Adjusting Scanning Parameters

Open the **ScanForm** window by clicking the button in the Main Operations panel.

The top part of the **ScanForm** window contains a panel to control the scanning process (see Fig. 7-9). The bottom of the window contains a Viewer to display topography of the surface under investigation.
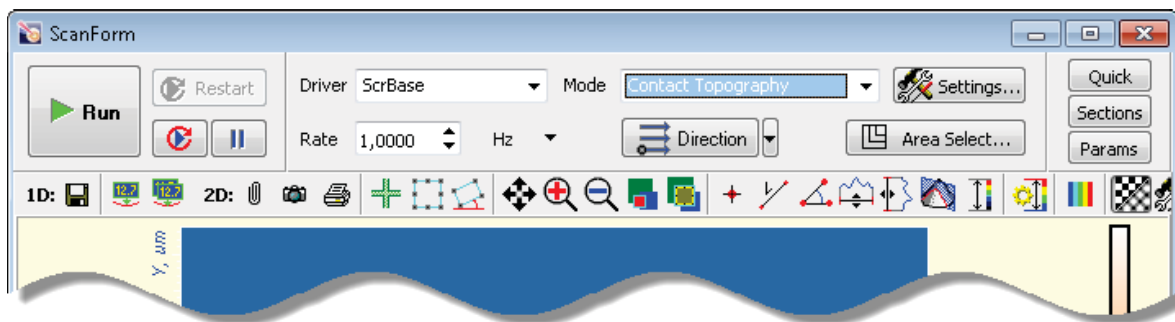
Fig. 7-9. Control panel of the **ScanForm** window

Selecting operating mode of scanner

The SCANSCALER™ system provides measurement in one of the two following modes:

- **"high voltage"** – the maximum scan area is at least 100×100 μm. Scanning will use the XY feedback loop (Close Loop,) (Close Loop, the button is pressed in);

- **"low voltage"** – the maximum scan area is approximately 2×2 μm. The low voltage mode is used for imaging of high resolution that can provide atomic scale details. In the low voltage mode, the XY feedback loop is open (the button is released).

ATTENTION! Before switching the operating mode of the scanner open the feedback loop (the button should be in the ⟦ ꘎ Off ⟧ state).

1. Open the **Calibrations** dialog box (see Fig. 7-10) by selecting the **Tools➔ Calibrations** menu command.
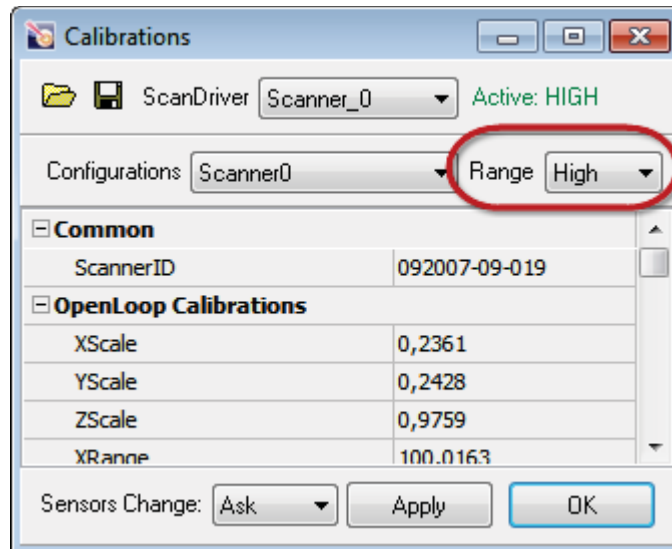
Fig. 7-10. Scanner operates in the high voltage mode

2. In the **Range** list, select:

- **High** for the high voltage mode;
- **Low** for the low voltage mode.

3. Click the **Apply** button. A Confirmation box for applying new values of the scanner parameters will appear (Fig. 7-11). Click the **Yes** button.

Fig. 7-11

4. For the **Low** mode, disable the feedback on XY position of the scanner (release the ꘎ XY button).

For the mode **High** mode, the ꘎ XY button should be pressed in for better accuracy of XY scanning.

Selecting AFM mode

Select the **Contact Topography** mode in the **Mode** list of the Control panel of the scanning window (see Fig. 7-12). The **Height** signal will be selected for detection.
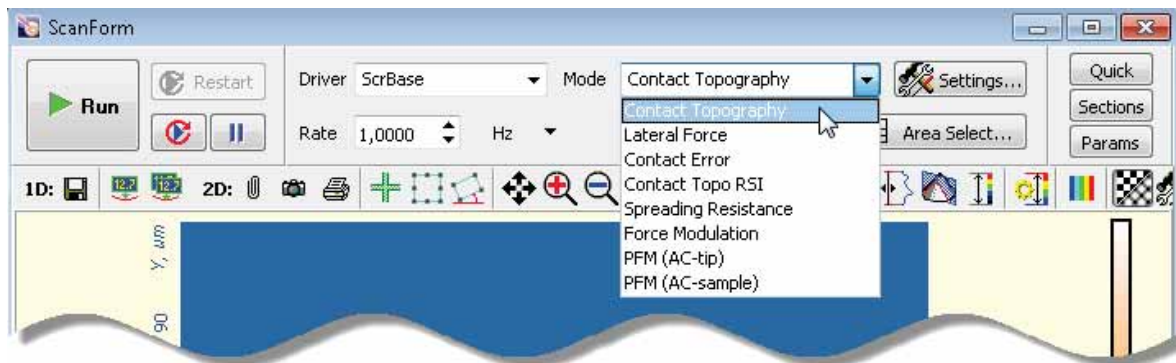
Fig. 7-12. Selecting the Constant Force Mode

Selecting the scan area

To choose preliminary dimensions of the scan area, regard for the following recommendations:

- If height difference in the surface profile is known to be within the scanner range, the maximum scan field is recommended;

- If height difference in the surface profile is unknown, a small sized scan area (about 10÷15 μm) should be preliminarily explored for finding proper levels of controlling parameters, including speed of scanning, **SetPoint**, **Gain**. Then the scan area can be enlarged.

Two options are provided for selecting the scan area:

- by defining the scan area parameters immediately (see p. 50) – number of scan points, rotation of the scan area in XY plane, and size of the scan area can be defined explicitly by the user;

- by defining the scan area in the 2D Data Viewer of the Scanning window (see p. 51) – only size and position of the scan area can be defined.

    These options are explained below in details.

Defining Scan Area Parameters

Size and position of the scan area can be defined in the **Scan Area Selection** window (Fig. 7‑13) accessible by clicking the [Area Select...] button.

Fig. 7‑13. Scan Area Selection window

The red rectangle in the left part of the window displays the scan area currently selected with respect to the maximum scan area.

The following rules of modification of the scan area (the specific one is selected by clicking on titles of the 2nd and 3rd columns) are available:

X, Y – X and Y dimensions of the scan rectangle are adjusted independently;

- **X~Y, Y~X** – when a particular dimension is changed, its counterpart will vary proportionally;

- **X=Y, Y=X** – the dimensions will be kept equal (square scan area).

- Position of the scan area can be redefined either with the mouse or with entering coordinates of the area center (in μm) in the **Center** fields. The origin of the coordinate system is at the left bottom corner of the maximum scan area.

Sizes of the scan area are defined by three parameters:

Size – size of the scan area in a particular direction (in μm). By default, dimensions of the scan area are 10×10 μm;

Step – distance between neighboring scan points in a particular direction (in μm);

Points – number of scan points in a particular direction. By default, number of points for every direction is 512.

To enable modifications in a particular cell, click on it with the mouse and the cell will become active. Value in the active cell can be edited in one of the following ways:

- with arrow keys <↑>, <↓>;

- with scroll keys ▲▼;

- with the slider (appears after double-click in the active cell).

The parameters **Size, Step,** and **Points** are interrelated through the formula:

$$Size = Step \times Points. \tag{1}$$

When a particular parameter varies, one of the two remaining follows these changes while the second remaining (fixed) keeps at the same level. The field of the fixed parameter is displayed on orange or purple background.

Purple background. Used for displaying the line being previously active.

Orange background. Appears after triple click on a parameter line. For changing the parameter to be fixed, click on the field of its name.

Rotation angle of the scan area in XY plane is defined in the green cell at the left top of the table.

NOTE. Scanning over a rotated scan area is not recommended if the XY displacement sensors are disabled.

Selecting Scan Area in Scanning Window

1. Enable the function of selecting the scan area by clicking the ⬚ button in the Toolbar.

   NOTE. If a rotated scan area is required, the function of selecting the scan area should be called by clicking the ⬚ button (the ⬚ button will be hidden in this case).

2. Change size and position of the scan area with the mouse.

3. Click the ✚ button. Verify that the probe tip can contact the sample surface everywhere inside the selected scan area without penetration. To this purpose, press and hold the left mouse button and move the mouse pointer inside the scan area. Motion of the mouse pointer will be followed by displacement of the probe tip over the sample surface. Monitor extension of the piezoscanner in the indicator in the top bar of the Main Program window.

Fig. 7‑14. 2D Data Viewer
Arrow indicates the boundary of the scan area

Selecting the scan direction

The scan direction is selected in the [Direction] drop-down list of the Control panel of the Scanning window (see Fig. 7-9).

Adjusting the scanning speed

Optimal value of the scanning speed depends on sample surface properties, scan area dimensions and external conditions. A smooth surface can be scanned at higher speed than that with uneven topography and large height difference.

To define the **Rate** parameter, perform the following steps:

1. In the Control panel of the **ScanForm** window, select unit of scan rate measurement (Fig. 7-15). For example, the **Hz** item is associated with the line scan frequency in Hz.
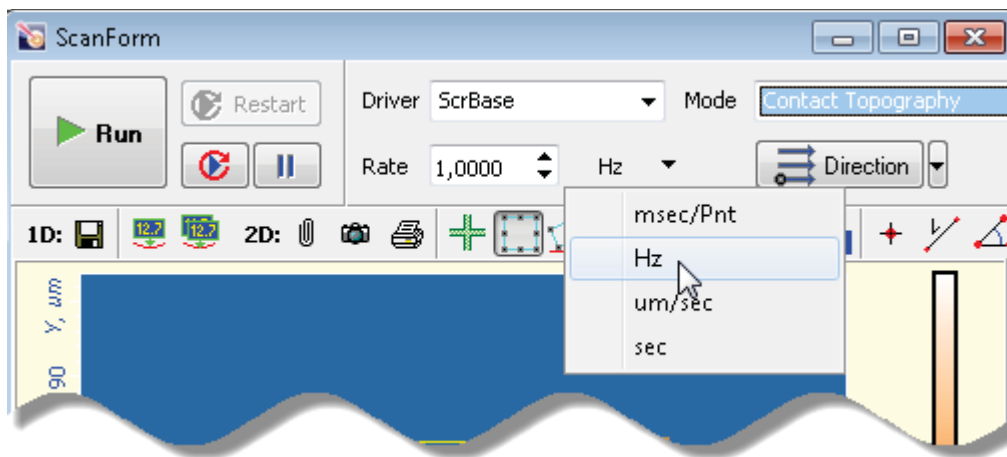
Fig. 7-15

2. In the **Rate** field, define level of the scan rate. For testing scans, values not exceeding 0.5 Hz are recommended.

Selecting detection signals

Every measurement technique is supplied with a default set of recorded signals. This set can be modified if required. The Control program uses the latest set of recorded signals.

For every recorded signal a separate 2D Data Viewer is reserved in the **ScanForm** window. The instrument is capable to register up to eight signals in parallel.

The set of recorded signals can be modified in the **ScanSettings** window (accessible through clicking the [Settings...] button in the Control panel of the **ScanForm** window).

Fig. 7-16. **ScanSettings** window

The **Scan Mode** field displays the SPM technique the set of recorded signals of which is viewed or edited at the moment.

The list of recorded signals is provided in the **Signals** column. Although data are measured both in forward (from beginning of the line to its end) and in backward (reverse) passes of the probe, the 2D image is generated only for one of those passes. That pass is selected in the **Direction** column as follows: **Forward** – the signal will be recorded for the forward pass, **Backward** – the signal will be recorded for the backward pass.

If displacement sensors are used, the **SensHeight** signal is recommended for recording because it is most sensitive to the surface landscape:

Height is a signal derived from voltage applied to the Z scanner.

SensHeight is a calibrated signal from the Z displacement sensor.

7.1.6.   Scanning

Hereafter, the scanning process will be explained with the example of a rectangular grating (standard grating TGQ-1, 3 µm resolution).

<u>**Starting measurements**</u>

Scanning of the sample surface should be preceded by necessary preparation procedures (the sample has approached to the probe, the operation point is adjusted, and all scanning parameters have been set).

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window (see Fig. 7-17).
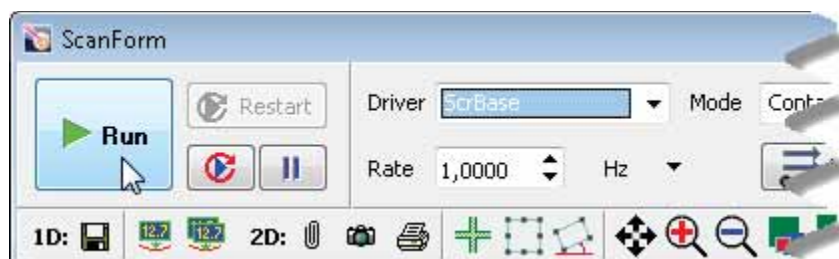
Fig. 7-17. Control panel of the **ScanForm** window

This results in the following activity of the system:

- Line-by-line scanning of the sample surface starts and the corresponding image of the scan area appears in the 2D Viewer of the scan data (see Fig. 7-18). In our example, this is an image of the rectangular grating;
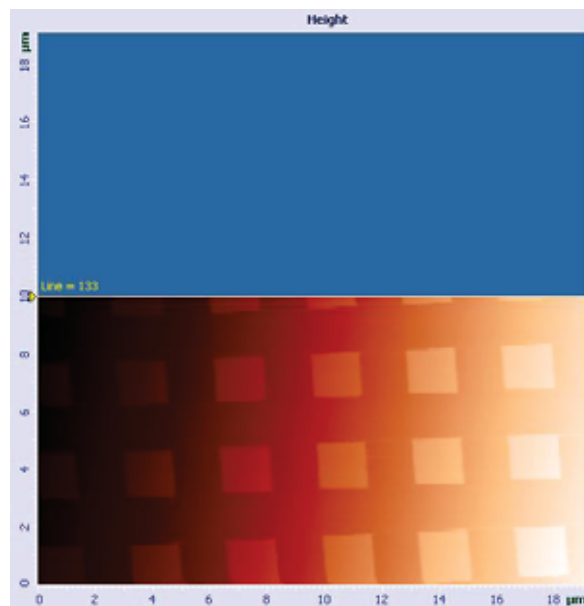
Fig. 7-18. Surface topography image

- If the **S** button in the left bottom of the 2D Viewer is pressed, the 1D Viewer will display the detection signal in real time (see Fig. 7-19);

Fig. 7-19. **Height** signal

- The **Restart** button becomes enabled and caption of the **Run** button changes to **Stop** (see Fig. 7-20).
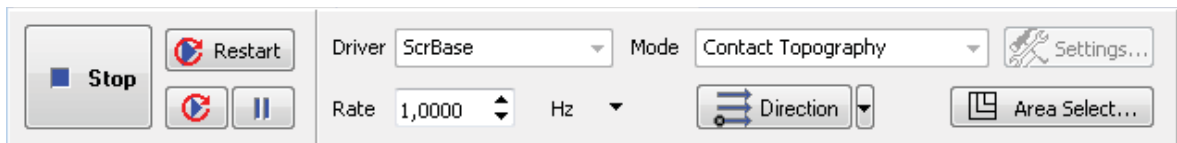
Fig. 7-20. Control panel of the **ScanForm** window in the course of scanning

Should the scanning procedure be interrupted, click the **Stop** button or use the <Esc> key.

Tilt subtraction

Data in the example above (see Fig. 7-18, Fig. 7-19) reveal that the sample is tilted to some degree in X direction.

Contribution of the tilt to the detected landscape can be eliminated immediately in the scan process with proper selection of the **Subtract** control at the right bottom of the **Scanning** window. By default, this button is in the **None** state (see Fig. 7-19).

To activate run-time subtraction, click this button and select the **Plane** option in the corresponding list (see Fig. 7-21). This entails subtraction of the plane tilt, and the original image (see Fig. 7-18) is transformed into the image shown in Fig. 7-22.

Fig. 7-21
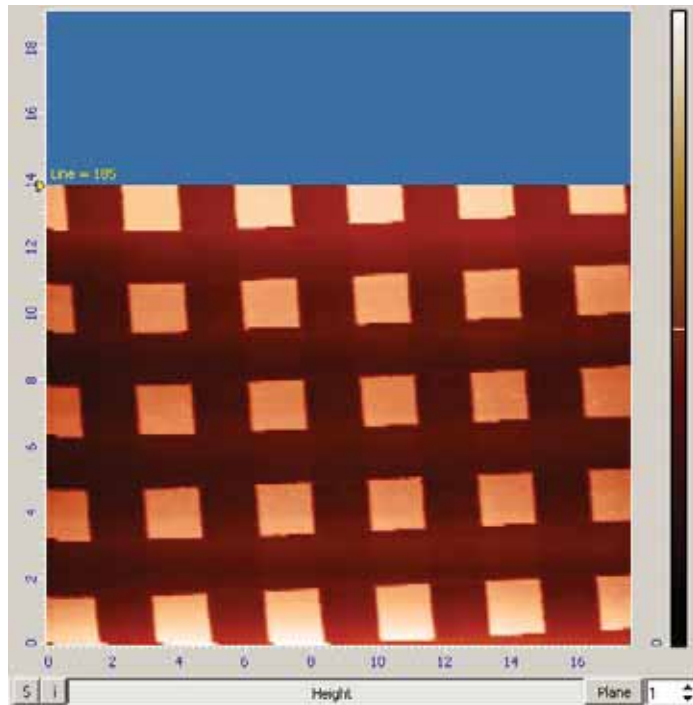
Fig. 7-22. Surface topography image

With the correction defined, the 1D Viewer of the scan data will display the detection signal corrected for the tilt contribution. The resulting signal is shown in Fig. 7-23 (compare with the signal in Fig. 7-19).

Fig. 7-23. **Height** signal

NOTE. Collected frames miss information on corrections of the scan data performed with the **Subtract** *option.*

Adjusting parameters in the course of scanning

Quality of the acquired surface image essentially depends on such parameters as scanning **Rate**, **Set Point**, and the feedback gain, **Gain**. Either of these parameters can be adjusted instantly in the course of scanning with visual inspection how modifications impact on the image being collected. We recommend testing for occurrence of oscillations in the measurement circuit using profile of the DFL signal because it deviates much less than the Height signal.

If the **Gain** parameter is decreased, the **Rate** parameter should be decreased as well since otherwise the feedback loop would fail to follow details of the surface landscape. This is explained by Fig. 7‑24 which demonstrates that at Rate = 2 Hz and Gain=1 the feedback loop is unable to trace sharp changes in the landscape height and so it smoothens vertical steps in the landscape profile. This effect can be eliminated either by increase of **Gain** or by decrease of the scanning rate.
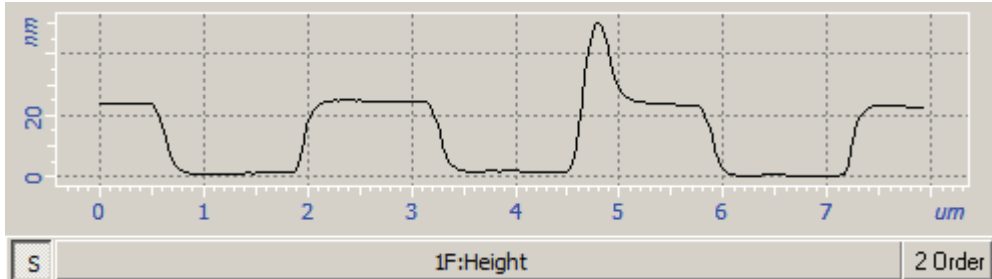
Fig. 7‑24. Landscape profile of test grating: Rate = 2 Hz, Gain = 1

Fig. 7‑25 shows an example of correct adjustment of the scanning parameters when initial values of the rate and of the gain were decreased and increased, respectively.
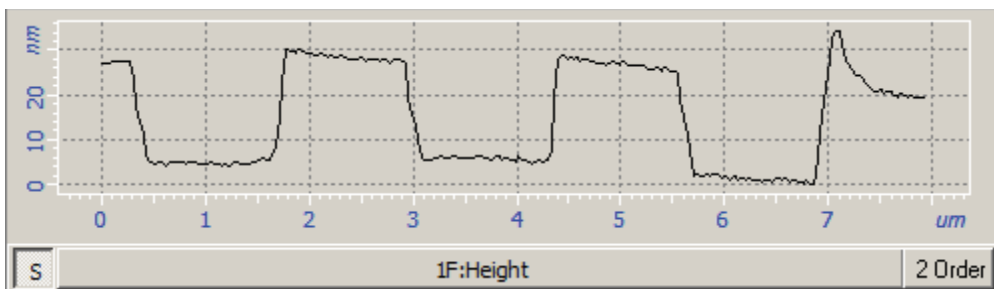
Fig. 7‑25. Landscape profile of test grating
taken with adjusted scanning parameters: Rate = 1 Hz, Gain = 9

NOTE. Scanning is recommended to restart (using the **Restart** *button) after any of scanning parameters is changed.*

Some recommendations on optimization of scanning parameters

The choice of the optimal value of scanning speed depends on properties of the sample, on dimensions of the scan area, and on external conditions.

Smooth surfaces can be scanned at a speed higher than those with sharp features and steep slopes.

It is recommended to start scanning at a lower scanning speed, increasing it gradually until distortions affect the landscape profile.

Scanning speed should be reduced when surface features in the scan direction are not detected.

In measurements of soft materials, the images may be affected by "dragging effects" caused by surface features in the scan direction. In the event of such effects, it is recommended to reduce the scanning speed as well as to increase the **Set Point** level in order to lower pressure on the sample.

When adjusting the **Set Point** parameter you should account for the following:

- difference between **Set Point** and the initial level of **DFL** defines intensity of interaction between the tip apex and the sample surface. The bigger is this difference, the larger is the cantilever deflection and so the larger is the force the tip presses the surface. Thus, adjusting the **Set Point** parameter relative to the initial level of **DFL** allows to change or to define the desired interaction intensity between the tip and the sample surface;

- if the difference between **Set Point** and the initial level of **DFL** is large (this corresponds to high pressure applied to the sample surface with the tip), the tip or the sample surface can be damaged in the course of scanning;

- if the difference between **Set Point** and the initial level of **DFL** is small (this corresponds to low pressure applied to the sample surface with the tip), control with the feedback system may become unstable;

- the **Set Point** level cannot be set lower than the initial level of **DFL** or higher than the Laser level.

7.1.7. Saving Measurement Data

To save measurement data on the hard drive, perform the following steps:

1. Open the **Image Analysis** window by clicking the button in the Main Operations panel.

2. In the Main menu of the **Image Analysis** window, select the **File → Save All Frames** command.

3. In the standard dialog, select a folder for saving the collected data.

4. Type in a filename and save it with the extension ***.mdt**.

7.1.8. Operation Completing

1. Open the feedback loop by clicking the [🔲 On] button in the Main Parameters panel (the button will change its appearance to [🔲 Off]).

2. Open the **Approach** window by clicking the [Approach] button. To prevent damage of the probe tip by the sample surface when removing the measuring head, retract the sample from the probe by clicking the [Move] button for several times (Fig. 7-26).
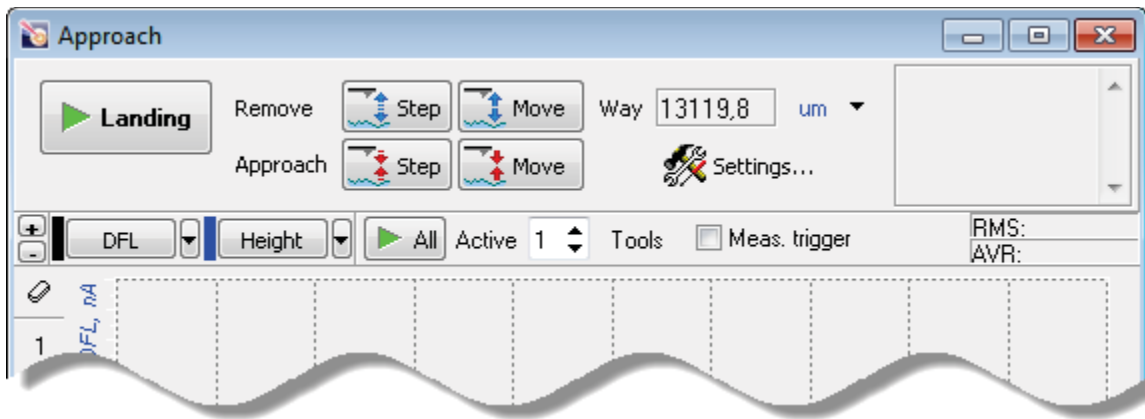
Fig. 7-26. **Approach** window

3. Move the heads to their inoperative positions with the [STM AFM] button

4. Close the Nova Px Control program.

5. Turn off the SPM controller.

6. Turn off the vibration isolation system.

7. Turn off the computer.

7.2. Lateral Force Microscopy

7.2.1. Brief Description

The Lateral Force Microscopy facilitates discrimination between areas with different coefficients of friction as well as reveals specific features of surface topography. This mode is useful in research of semiconductors, polymers, film coatings, memory media for physico-chemical properties of the surface (in particular, deterioration), tribology characteristics etc.

The Lateral Force Microscopy is based on the following physical ground. While scanning in this mode, a torsion deflection of the probe occurs in the direction perpendicular to the longitudinal axis of the cantilever additionally to the normal deflection (see Fig. 7-27). It is caused by moment of the force acting on the tip of the probe.
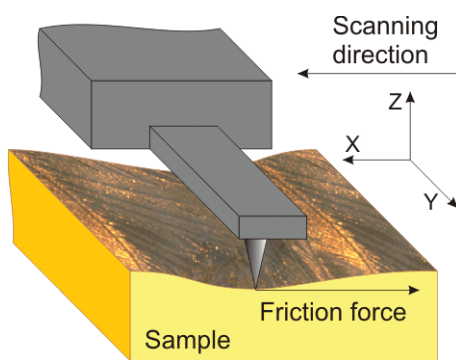
Fig. 7-27
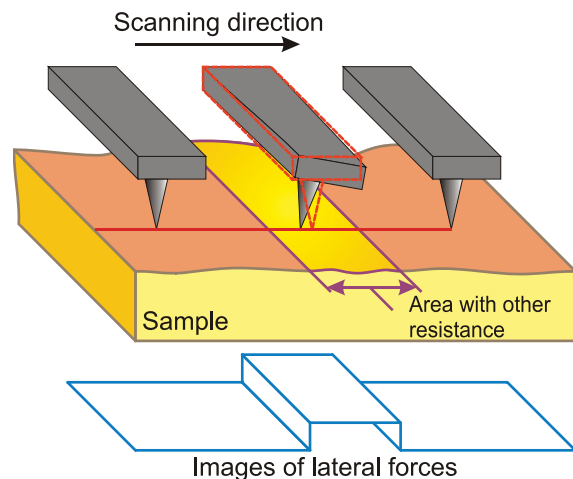
Fig. 7-28

At small deflections, the twist angle of the probe is proportional to the lateral force. The magnitude of the twist is measured with the optical detection system of the instrument. This detection system generates the **LF** signal whose variation is proportional to the value of the torsion deflection of the cantilever. This signal provides imaging of the local friction along the sample surface.

The twist angle of the probe varies when the probe moves along a flat surface with regions that differ in their friction coefficients (see Fig. 7-28). This variation provides information on distribution of local friction properties. Unfortunately, such interpretation becomes invalid if the landscape is not flat.

Nevertheless, this technique offers good imaging of surface features in small scales. For example, lateral forces data provided easy determination of the atomic lattice parameters of mica and other lamellar materials.

7.2.2.   Scanning

Initial state

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the scan area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

Procedures of the Lateral Force Mode are based on those of the Constant Force Mode that is described in detail in sect. 7.1 "Constant Force Mode" on p. 41.

The basic difference of operation in this mode from that of the Constant Force Mode is in the step of Selecting AFM mode (see p. 49) where the **Lateral Force** option in the **Mode** list should be selected instead of **Contact Topography** (see Fig. 7-29). The **Height** and **LF** signals will be selected for detection.
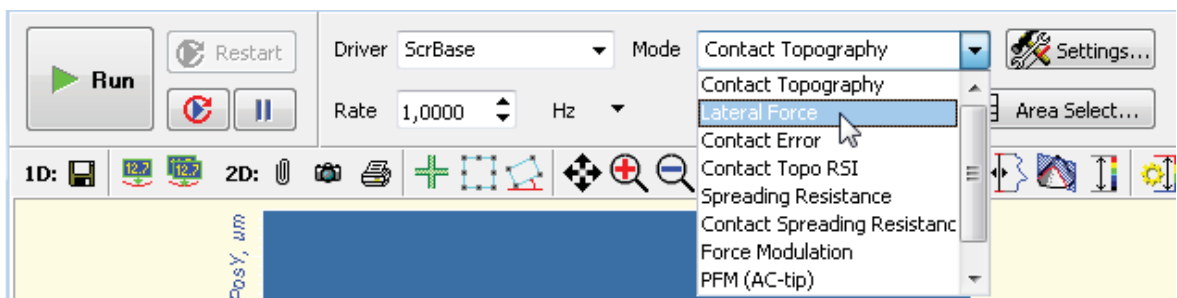
Fig. 7-29. Selecting the Lateral Force Microscopy

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window.

This launches line-by-line scanning of the sample surface and the 2D Viewer of the scan data will display three 2D data views. The first view shows the surface landscape while the other two display distributions of the lateral force (**LF** signal) (see Fig. 7-30) detected in forward and backward sweeps of the scan lines.
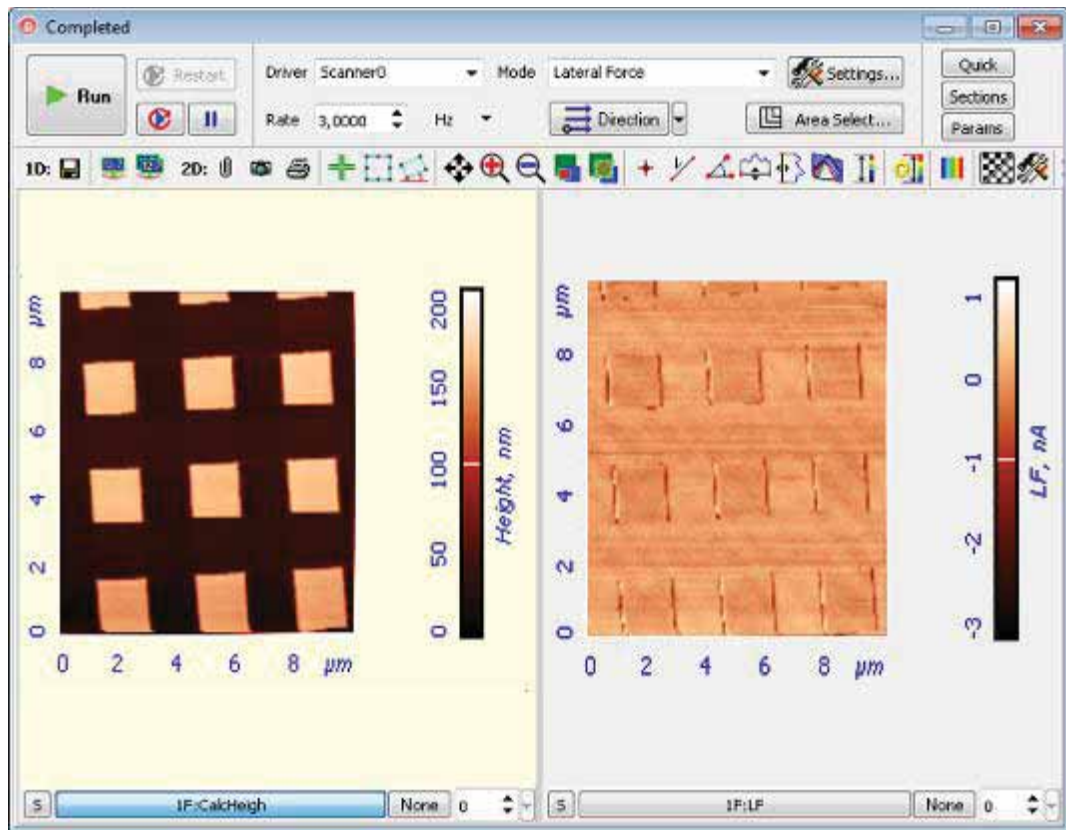
Fig. 7-30. Images of surface topography and distribution of lateral forces

7.3. Spreading Resistance Imaging

7.3.1. Brief Description

The **Spreading Resistance Imaging** mode is a very efficient AFM mode. It is employed in various fields, including defects in conductive and low conductive films, materials characterization in terms of local resistance etc. This mode uses a conductive probe that comes in contact with the sample surface. The probe is applied to a bias voltage. The resulting current through the sample is measured as a function of the probe position simultaneously with surface topography acquisition. The latter uses the Constant Force mode. It is easy to prove that, under the assumption of constancy of contact probe-surface resistance for a given bias voltage, the detected current is proportional to local resistance of the sample.

Among typical applications of the mode are finding actual geometrical dimensions of source-drain regions in MIS transistors, localization of p-n junctions, study of dopants distribution in semiconductors *etc*.

7.3.2. Preparing for Measurements

Remember to use the measuring head equipped with the conducting contact probe.

The sample should be installed so as it will be in electric contact with the scanner. For spreading resistance imaging, the sample is either mounted on a substrate with a spring contact or fixed with conductive glue (see i. «Mounting Sample» on p. 20).

<u>**Initial state**</u>

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the scan area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

7.3.3.   Adjusting Scanning Parameters

Procedures of the Spreading Resistance Imaging Mode are based on those of the Constant Force Mode that is described in detail in sect. 7.1 "Constant Force Mode" on p. 41.

Before starting the Spreading Resistance Imaging, make preparations for measurements and perform measurements of surface topography by the Constant Force Mode

After preliminary measurements of surface topography by the Constant Force Mode, adjust parameters for operating in the Spreading Resistance Imaging mode.

1. Open the Scanning window with the button in the Main Operations panel.

2. Select the **Spreading Resistance** mode in the **Mode** list on the Control panel of the scanning window (see Fig. 7-31). The **Height** and **IprAFM** signals will be selected for detection.
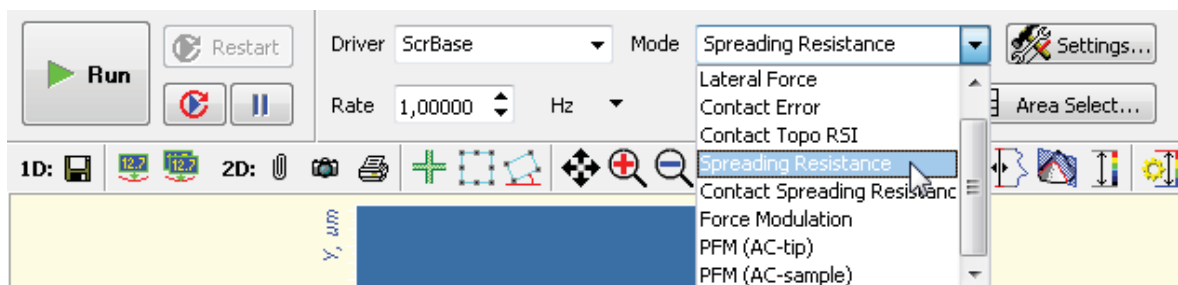
Fig. 7-31. Selecting the Spreading Resistance Imaging Mode

3. Open the software oscilloscope window with the button.

4. In the Oscilloscope window, select displaying of the **IprAFM** signal (see Fig. 7-32).
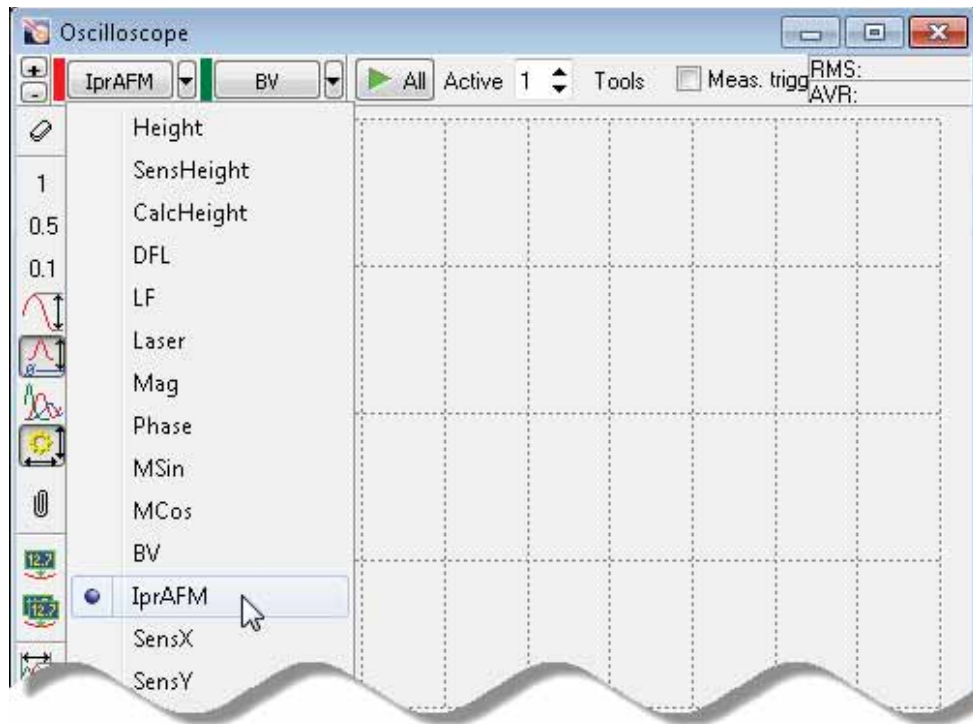
Fig. 7-32. Selecting the **IprAFM** signal in the **Oscilloscope** window

5. Enter the probe-sample bias voltage in the **BV** input field of the top bar of the Main program window. Adjust the **BV** bias voltage so that the **IprAFM** level is in the range 1÷10 nA.

If no current flows between the sample and the probe (the **IprAFM** signal is zero), this can indicate that the probe is on a non-conductive area of the sample surface. For selecting a relevant scan area, reposition the sample after retracting it form the probe tip distance of about 1 mm and disabling the feedback.

7.3.4. Scanning

Click the button **Run** in the Control panel of the **ScanForm** window to start the process of scanning.

This launches line-by-line scanning of the sample surface and the 2D Viewer of the scan data will display two 2D data views. The first view shows the surface landscape (**Height** signal) while the second one displays distribution of the spreading current (**IprAFM** signal).
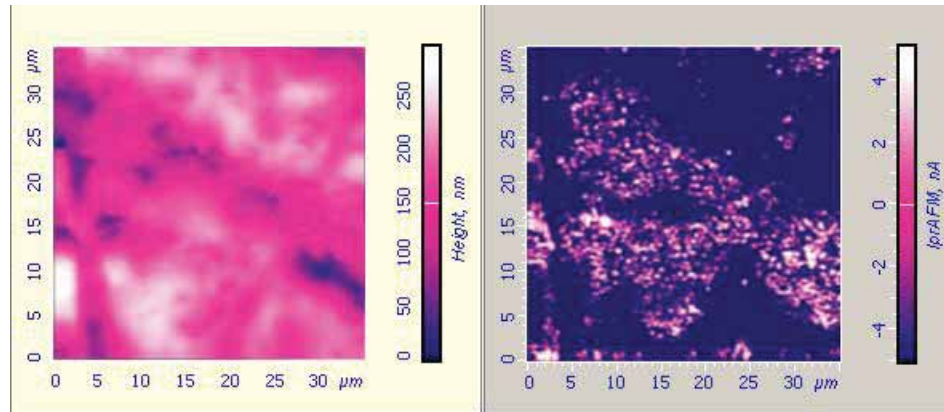
Fig. 7-33. Landscape image and distribution of spreading current

*NOTE. Brightness in the **IprAFM** signal image is proportional to conductivity.*

How to Improve Image Quality

⚠️ **ATTENTION! Too tight mechanical contact between the probe and the sample may cause damage to the conductive coating of the probe.**

If necessary, you can try the following recommendations:

● Adjust the **SetPoint** parameter (it controls pressure applied to the sample by the probe);

● Adjust the bias voltage applied between the probe and the sample (**BV** parameter). Note that excessive voltage may cause local oxidization of the surface;

● Replace the probe (with different elasticity or another type of conductive coating).

7.4. Contact Error Mode

7.4.1. Brief Description of the Mode

When scanning, the value of the cantilever deflection varies following the surface topography of the sample. The feedback loop tries to preserve the given level of the cantilever deflection (**Set Point**) by maintaining the **DFL** signal level, which is linked with the deflection. However the feedback loop cannot compensate for variations of the **DFL** signal instantaneously as it has some inertia (characterized by a time delay).

During scanning, the current value of the **DFL** signal (which is linked with the cantilever deflection) is the error signal of the feedback loop and it contains additional information on surface topography. This signal can be used for a more detailed reproduction of the topography. The mode that allows imaging of surface topography by the Constant Force Mode simultaneously with measurements of the error signal (the **DFL** signal, in our case) is called the **Contact Error Mode**.

The Contact Error Mode can be considered as an intermediate mode between the Constant Force Mode and the Constant Height Mode. This is possible providing that the speed of error signal data processing (the feedback gain factor, in our case) is optimized so that the feedback loop is able to trace smooth topography variations while not been able to recognize sharp features. Then, scanning of smooth surfaces, with smooth and extended features, is performed with the piezo-scanner of almost constant length. As a result, the final image will have higher contrast for sharp features and lower contrast for smooth and large ones. This can be useful for identification of small features on the background of large and relatively smooth variations of surface.

7.4.2. Scanning

Initial state

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
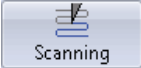- Initial approach;
- Selecting the scan area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

The Contact Error Mode is based on the Constant Force Mode, which is described in detail in i. 7.1 "Constant Force Mode" on page 41.

Before the Contact Error Mode measurements, prepare for the measurements and perform measurements of surface topography by the Constant Force Mode.

After the completion of preliminary measurements of surface topography by the Constant Force Mode, perform setting of parameters for operating by the Contact Error Mode.

To adjust the instrument for measurements by Contact error mode select the **Contact Error** mode in the **Mode** list on the Control panel of the **ScanForm** window (Fig. 7-34). The **Height** and **DFL** signals will be selected for detection.
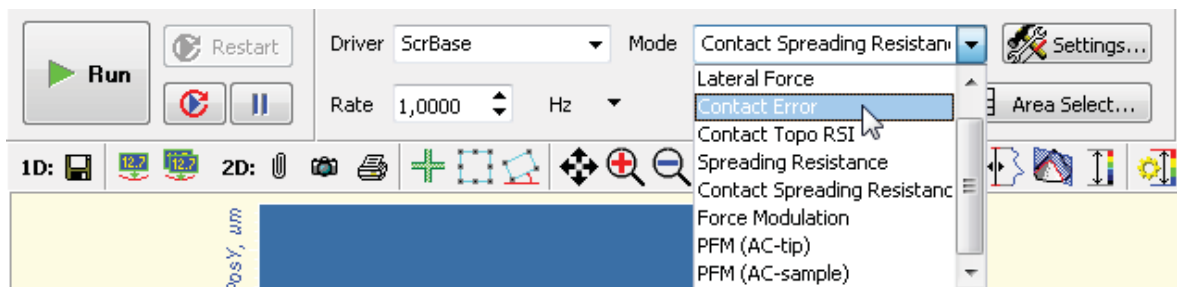
Fig. 7-34. Selecting the **Contact Error** Mode

Click the button **Run** to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts

- Two images appear in the field of 2D visualization of the scanning data. One of the images is an image of surface topography (signal **Height**), while the other one is the error signal (signal **DFL**) (Fig. 7-35);
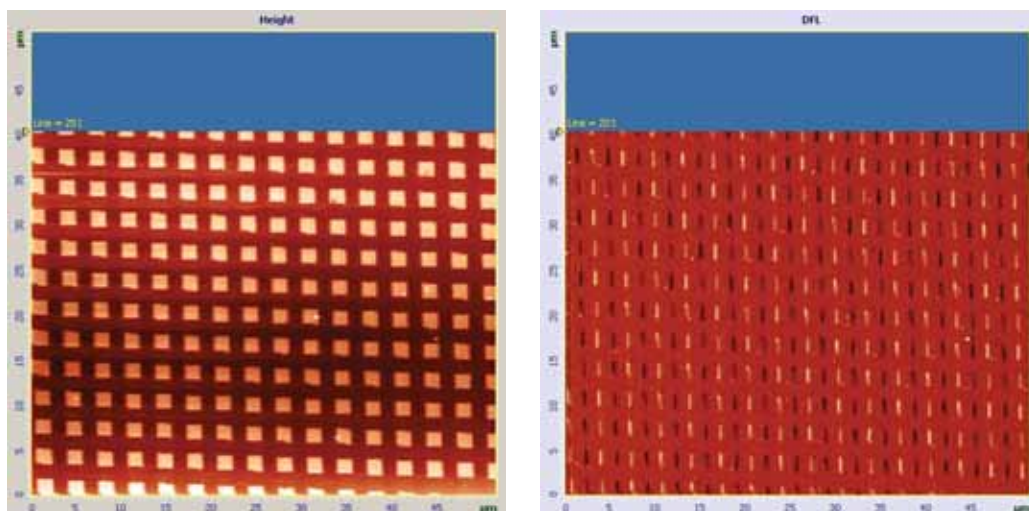
Fig. 7-35. Images of surface topography and the **Error** signal

- If the **S** button in the left bottom of the 2D Viewer is pressed, the 1D Viewer will display the detection signal of selecting image in real time (Fig. 7-36).
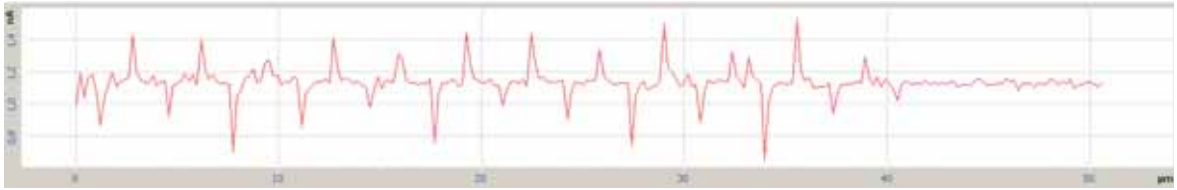
Fig. 7-36. Signal **DFL**

7.5. Piezoresponse Force Microscopy

7.5.1. Brief Description of the Mode

Piezoresponse Force Microscopy is usually used in material sciences in the study of ferroelectrics and it allows investigations of their domain structure. Especial interest lies in the field of applied research for the development of ferroelectrics data storage devices and modes of data reading/writing related to them.

The interaction between the local electric field and the surface of a piezo-electric sample (with subsequent analysis of resulting displacements of the surface) lies in the basis of this mode. This displacement results from the piezo-electric effect, which reveals itself in the form of a linear relationship between the sample geometry and the electric field applied to it. Variations of the sample geometry depend on the magnitude and mutual orientation of the electric field and the polarization vector of the sample.

In this mode, a bias voltage is applied to the conductive probe. This induces a local electric field under the probe. As ferroelectrics have the domain structure, the application of the same local electric field to different areas of the surface yields different results.

Fig. 7-37 illustrates displacements of the structure whose domains are oriented both vertically and horizontally. The case of the altering electric field is considered. Oscillations of the surface are shown both for the positive direction of the electric field, above, and for the negative, below.
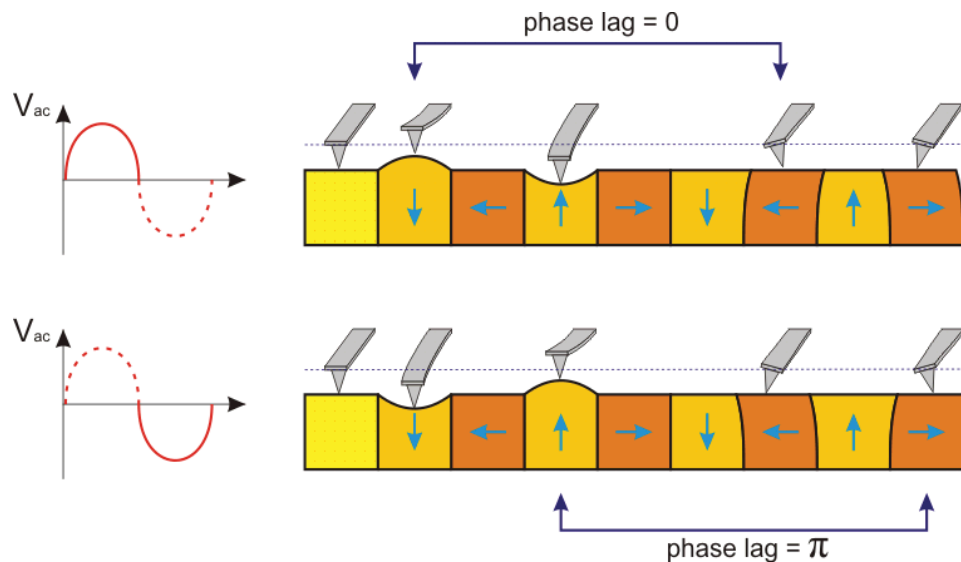
Fig. 7-37

Provided that domains have the same sizes, the magnitude of surface oscillations is the same. Correspondingly, the magnitude of the cantilever normal deflection is also the same. However the oscillation phase will be different for those domains with different orientation of the polarization vector.

In the case of horizontally oriented domains, surface displacements will be lateral with respect to the surface rather than normal. Respectively, the magnitude of normal oscillations of the cantilever is equal to zero. However, due to friction between the tip and the sample surface, some displacement of the tip will occur and, also, its orientation to the surface will be changing. At the same time, as in the case of normal oscillations, phase of lateral oscillations depends on the direction of the polarization vector of the domains oriented horizontally.

Therefore, Piezoresponse Force Microscopy yields both surface topography images and distributions of amplitudes of lateral and normal oscillations. In addition, the user obtains phase distributions of lateral and normal oscillations (Fig. 7-38). This information makes it possible to study the domain structure of the sample under investigation.
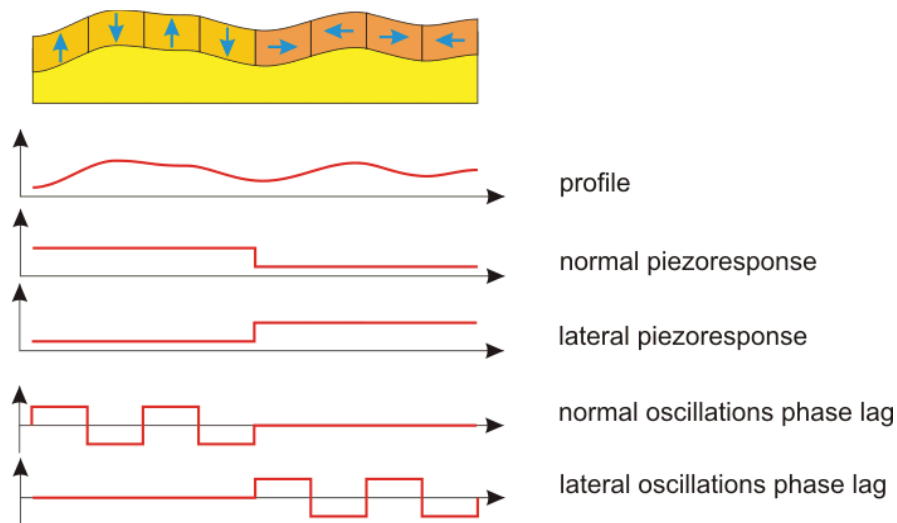
Fig. 7-38

However interpretation of the results obtained is not always simple. It is necessary to consider a number of factors which can affect or influence images obtained during measurements.

For better signal (oscillation amplitude), it is desirable to operate on a frequency that is close to the frequency of the system cantilever-probe-surface (so that oscillations of the cantilever pressed to the sample would be resonance). This approach is, however, good only for homogeneous materials. If the sample is inhomogeneous, containing some inclusions, for example, the resonance oscillation amplitude will reflect the distribution of surface elasticity in addition to the domain structure. In this case it is recommended to operate on a frequency that is afar from the resonance.

For inhomogeneous materials, under certain conditions, the oscillation amplitude can reflect the sample structure even afar from the resonance. This might be due to camber of the surface induced by electrostatic forces.

7.5.2.   Preparation for Measurements

To operate in Piezoresponse Force Microscopy a sample should be glued to the substrate with the conductive glue or mounted on the substrate with a spring contact and it should be used conductive probes for contact modes.

Initial state

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the scan area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

Before the Piezoresponse Force Microscopy measurements, prepare for the measurements and perform measurements of surface topography by the Constant Force Mode.

7.5.3.   Adjusting Scanning Parameters

After the completion of preliminary measurements of surface topography by the Constant Force Mode, perform setting of parameters for operating on Piezoresponse Force Microscopy.

1. Open the scanning window by clicking the [Scanning] button.

2. For Piezoresponse Force Microscopy, alternating voltage can be applied either to the probe or to the sample. Select the desired measurement technique in the **Mode** drop-down list (Fig. 7‑39):

   - **PFM (AC-tip)** – alternating voltage will be applied to the probe;
   - **PFM (AC-sample)** – alternating voltage will be applied to the sample.
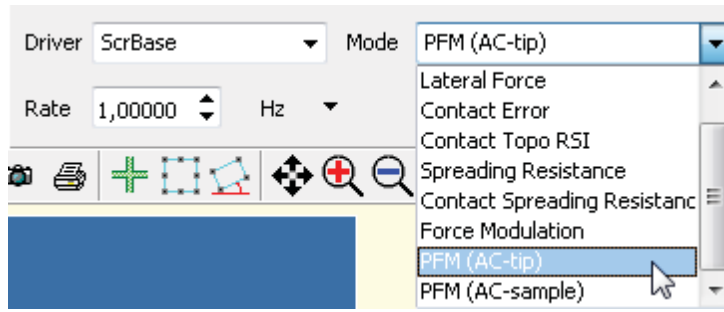
Fig. 7‑39. Selecting Piezoresponse Force Microscopy for measurements

This will result in the following (Fig. 7‑40):

- the **DFL** signal will be delivered to the input of the lock-in detector;

- gains of the lock-in detector (**Gain**) and of the preamplifier will be set to their maxima;

- the set of recorded signals will include signals **Height**, **Mag**, and **Phase** (this set can be modified in the **ScanSettings** window that is accessible through clicking Settings...);

- instrument components will be connected as follows:

    - **PFM (AC-tip)** – the sample is grounded, the probe is applied to alternating voltage from the generator;

    - **PFM (AC-sample)** – the probe is grounded, the sample is applied to alternating voltage from the generator.
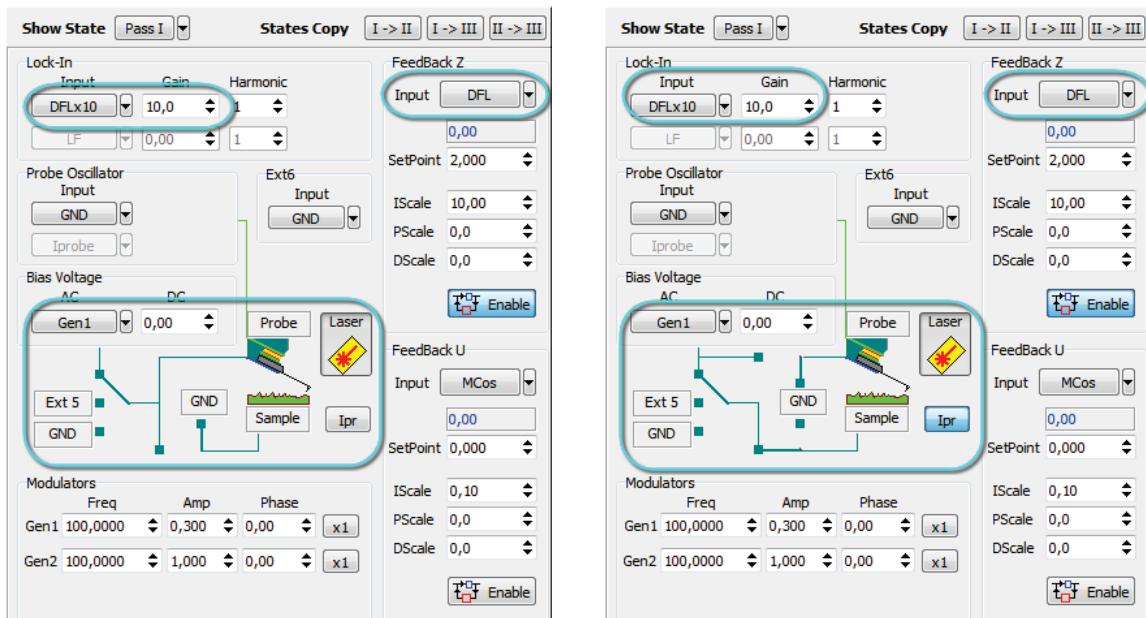
a) Alternating voltage
is applied to the probe

b) Alternating voltage
is applied to the sample

Fig. 7‑40. Block scheme of the instrument for Piezoresponse Force Microscopy

7.5.4. Scanning

Click the button **Run**, which is on the control panel of the **ScanForm** window, to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts

- Three images appear in the field of 2D visualization of the scanning data. One of them is an image of surface topography (signal **Height**), the second image is the amplitude of normal oscillations (signal **Mag**) and the third one is the distribution of normal phase (signal **Phase**).

- The panel of 1D images visualizes the signal measured line-by-line.

*NOTE. Normal oscillations of the cantilever (signal **DFL**) are registered for the default settings in the Piezoresponse Force Microscopy mode. For registration of lateral oscillations, connect the input of the lock-in amplifier to the **LF** (**LF×10**) signal.*
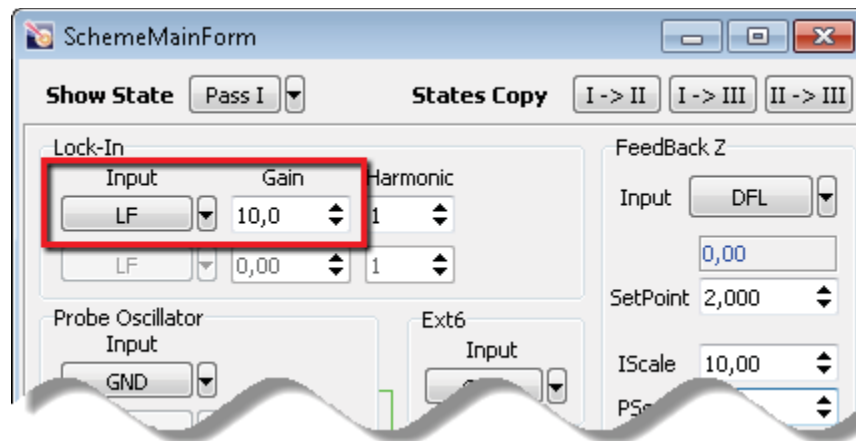
Fig. 7-41

After switching the inputs of the lock-in amplifier and the phase detector from **DFL** to **LF,** the second image of the scanning 2D data visualizes the distribution of the lateral oscillations amplitude, while the third image is the distribution of lateral phase (Fig. 7-42).
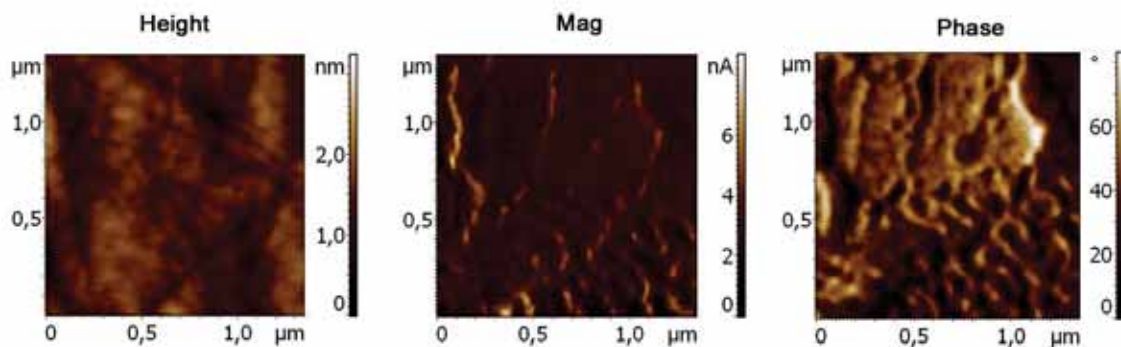
Fig. 7-42. Images of the surface under investigation

7.6. *Force Modulation Mode*

7.6.1. Brief Description of the Mode

Local elasticity can be measured using the **Force Modulation mode**. When scanning, an additional modulated voltage is applied to the Z-section of the scanner. This voltage provides vertical sinusoidal oscillations of the scanner. Following local elasticity of the sample surface, the values of camber of the sample and deflection of the cantilever vary. Harder areas of the sample surface are characterized with smaller camber of the surface, while deflection of the cantilever is greater. Softer areas of the sample surface are characterized with higher camber of the surface, while deflection of the cantilever becomes smaller (Fig. 7-43).
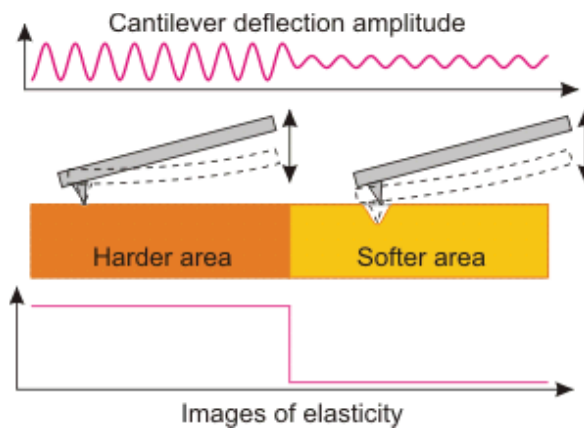
Fig. 7-43

Reading of surface topography of samples is conducted with medium deflection of the cantilever in the feedback loop.

The Force Modulation Mode is broadly used in the study of polymers, semiconductors and bio-objects, especially composite materials.

7.6.2.   Adjusting Scanning Parameters

__Initial state__

- Launching the control program;

- Turning the controller SPM on;

- Installing the probe;

- Adjusting the cantilever deflection detection system;

- Mounting the sample;

- Moving the measuring head to its working position;

- Initial approach;

- Selecting the scan area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

The Force Modulation Mode is based on the Constant Force Mode, which is described in detail in i. 7.1 "Constant Force Mode" on page 41.

Before the Force Modulation mode measurements, prepare for the measurements and perform measurements of surface topography by the Constant Force Mode.

Once a preliminary data on landscape were taken with the Constant mode technique, the instrument should be readjusted for the Force modulation method as follows:

1. Select **Force Modulation** in the **Mode** drop-down list of the Control panel of the **ScanForm** window (Fig. 7-44). The signals **Height**, **Mag** and **Phase** will be selected for measurements.
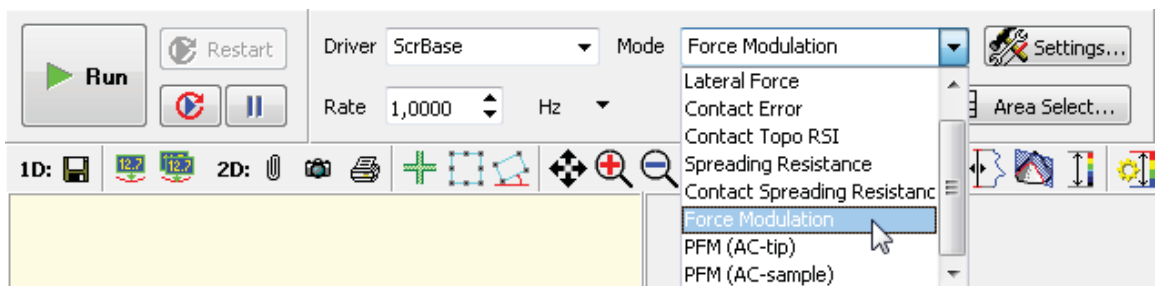
Fig. 7-44. Selecting the Force modulation method

2. Find the resonance frequency of the scanner. To this purpose, perform the following steps:

    a) Open the window **Resonance** by clicking the [Resonance] button in the Main Operations panel.

    b) In the **Configs** drop-down list select **GenZ-Lock1** (Fig. 7-45). This option applies AC voltage from a special oscillator to the Z-section of the scanner.

c) Open the Frequency response parameters panel by clicking the ⚒ button (Fig. 8-2).
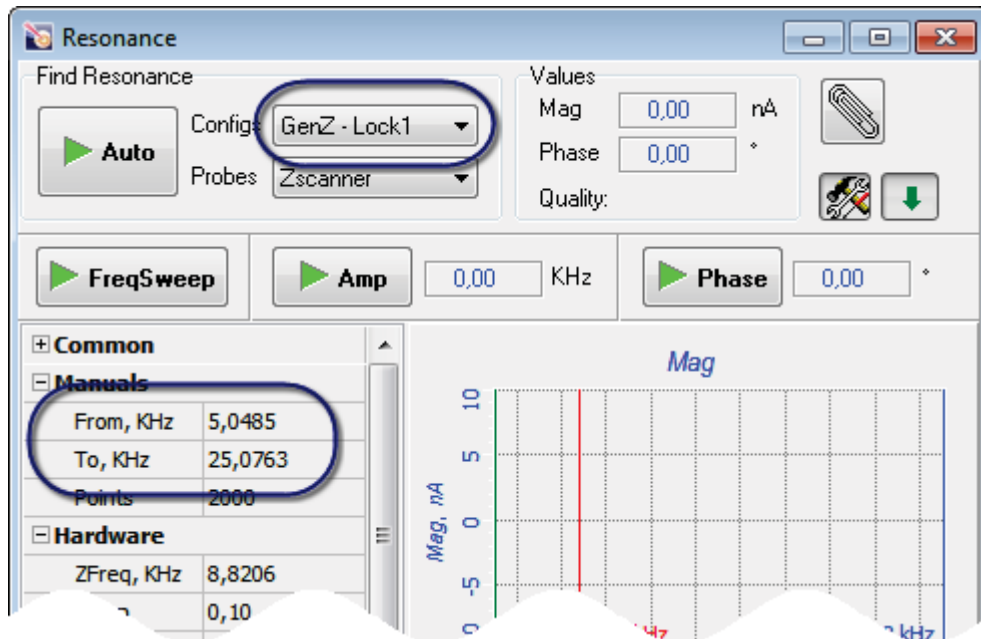
Fig. 7-45. **Resonance** window

d) In the fields **From** and **To** of the **Manual** group, defined a frequency range for searching for resonance. The recommended range is 5÷25 kHz.

e) Close the feedback loop (the feedback button is in the `On` state).

f) Start the procedure on searching for the resonance frequency by clicking the **FreqSweep** button.

When the procedure completes, the Frequency relations panel will display the frequency response of cantilever oscillation (**Mag** signal) (Fig. 7-46).
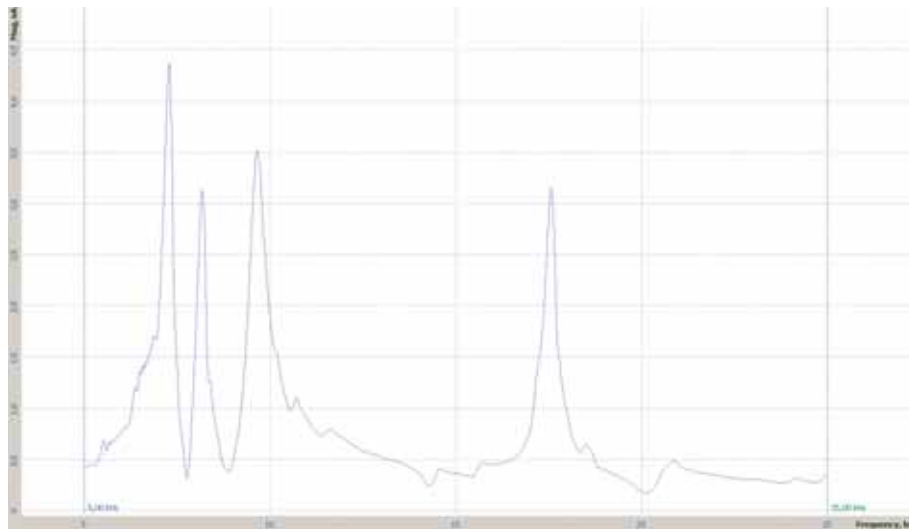
Fig. 7-46. Frequency relation of cantilever oscillation

3. Set the operating frequency of the generator at one of peaks in the frequency relation profile by moving the red marker to the desired position. Frequency corresponding to the current position of the marker is shown in the **ZFreq** field of the **Hardware** group (Fig. 7-45).

4. Adjust height of the desired peak to be in the range 2÷5 nA. This can be achieved by varying output voltage of the generator (**ZAmp** parameter) (see Fig. 7-45) or gain of the lock-in detector (**LockGain** parameter).

7.6.3.   Scanning

Click the button **Run**, which is on the control panel of the **ScanForm** window, to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts ;

- Two images appear in the field of 2D visualization of the scanning data. One of the images is an image of surface topography (signal **Height**), while the other one is a distribution of local elasticity (signal **Mag**);

- The panel of 1D images (opens when $\boxed{\text{S}}$ button is pressed) visualizes the signal measured line-by-line.

7.6.4.   Modes of Improving Image Quality

Image quality can be improved by the following:

- Alter parameter **Set Point** (pressure between the probe and the sample);

- Tune to another resonance frequency (another resonance peak);

- Alter the generator voltage (parameter **ZAmp**), gain coefficients of the lock-in amplifier (parameter **LockGain**);

- Replace the probe (with different elasticity and resonance frequency).

8. Semicontact AFM

8.1. Semicontact Mode

Operations performed in measurements of surface topography with the Semicontact Atomic Force Microscopy of different modes have much in common with those of other techniques based on resonant oscillations of the cantilever.

Initial state

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

Procedural Sequence

Measurement in a Semicontact mode is arranged in the following procedural sequence:

1. Switching the Instrument for Operating in the Semicontact Modes (see page 81)
2. Taking Resonance Curve and Adjusting Parameters of Generator Output (see page 81)
3. Approaching the Sample to the Probe (see page 88)
4. Adjusting Working Level of the Feedback Gain (see page 90)
5. Adjusting Scanning Parameters (see page 91)
6. Scanning (see page 97)
7. Saving Measurement Data (see page 100)
8. Operation Completing (see page 101)

These procedures are explained below.

8.1.1. Switching the Instrument for Operating in the Semicontact Modes

Switch the SOLVER NEXT for operating in the Semicontact mode by selecting **Semicontact** in the controller configuration list (see Fig. 8-1) in the Main Program window.
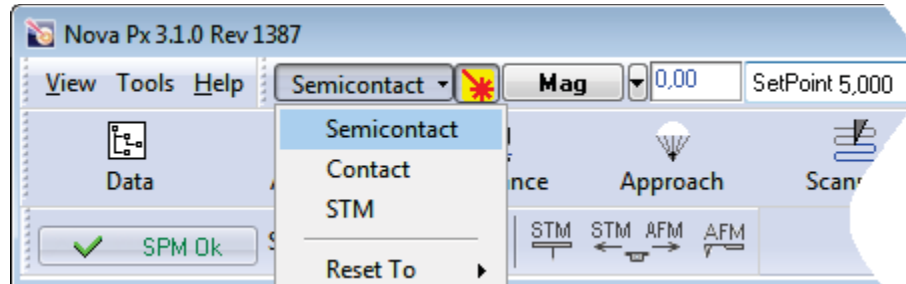
Fig. 8-1. Switching the instrument for operating in the Semicontact modes

Once the configuration **Semicontact** is set, all switching sequences required for the instrument to operate in semicontact modes proceed automatically.

The following adjustments of the system will take place:

- Input of the feedback loop – **Mag**;
- Level of the **SetPoint** parameter (proportional to the amplitude of the probe oscillation) – 5 nA;
- Gain of the feedback loop **Gain** – 1.

8.1.2. Taking Resonance Curve and Adjusting Parameters of Generator Output

Output of the generator feeds the piezodrive that provides oscillation of the cantilever. Characteristics of the cantilever oscillation during scanning process are adjusted with parameters of the generator output (frequency, magnitude, phase shift).

The Control program allows for two options of adjusting the generator output parameters, automatic and manual. These options are explained below.

Adjusting generator output parameters automatically

The automatic adjusting proceeds as follows:

1. The amplitude-frequency response (AFR) of cantilever oscillation is collected in the predefined frequency range.

2. Using this AFR, the resonance peak is located and its frequency is determined, then the AFR range is corrected. Further, the AFR is taken over the corrected range and refined resonance frequency is found.

3. The generator output amplitude and the gain are adjusted for the cantilever oscillations amplitude to be at its predefined level.

4. Phase of the generator output is tuned so as the phase shift between the detected signal and the reference signal is at its predefined level.

5. The AFR is collected with the adjusted parameters.

For automatic adjustment, perform the following steps:

1. Open the **Resonance** window by clicking the ![Resonance button] button in the Main Operations panel.

2. In the **Probes** drop-down list of the **Find Resonance** panel, select the desired type of the probe. This will preset parameters of automatic adjustment of the generator output parameters for the selected type of the probe.

    Parameters of the piezodrive operating frequency can be viewed and edited in the **Auto** group of the Frequency response parameters panel (opens by clicking the ![tools button] button) (Fig. 8-2).
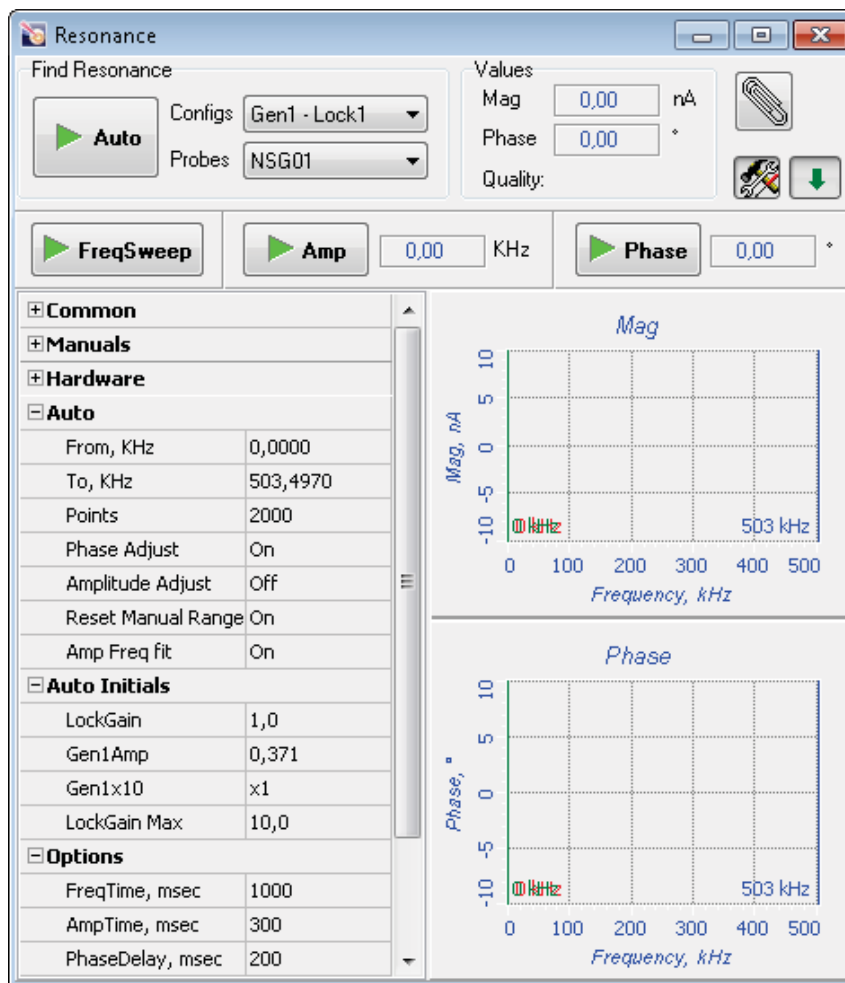
Fig. 8-2. Resonance window. Piezodrive adjustable parameters panel is opened

*NOTE. To display frequency responses of the signals **Mag** and **Phase**, select the corresponding options in the **Common** parameters group:*

Signal 1   **Mag**

Signal 2   **Phase**.
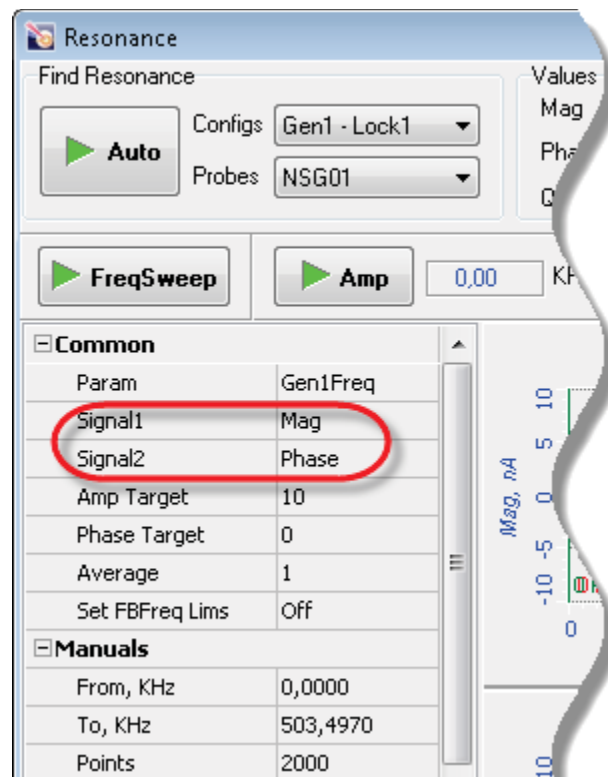
Fig. 8-3. Piezodrive adjustable parameters panel

3. To start automatic adjustment of the generator output parameters, click the button in the **Find Resonance** panel (see Fig. 8-2). This will provide:

- Acquiring of frequency responses of the magnitude (**Mag** signal) and of the phase (**Phase** signal) of the cantilever oscillation;

- Finding of the cantilever resonance frequency;

- Adjusting the working frequency of the generator output signal to the cantilever resonance frequency;

- Adjusting the gain (**Lock Gain** parameter) to the level at which magnitude of the cantilever oscillation at the resonance is equal to the prescribed one (**Amp Target** parameter);

- Adjusting the phase shift to the prescribed value (**Phase Target** parameter);

- Plotting frequency responses of the signals **Mag** and **Phase** in the range around the resonance frequency (see Fig. 8-4).
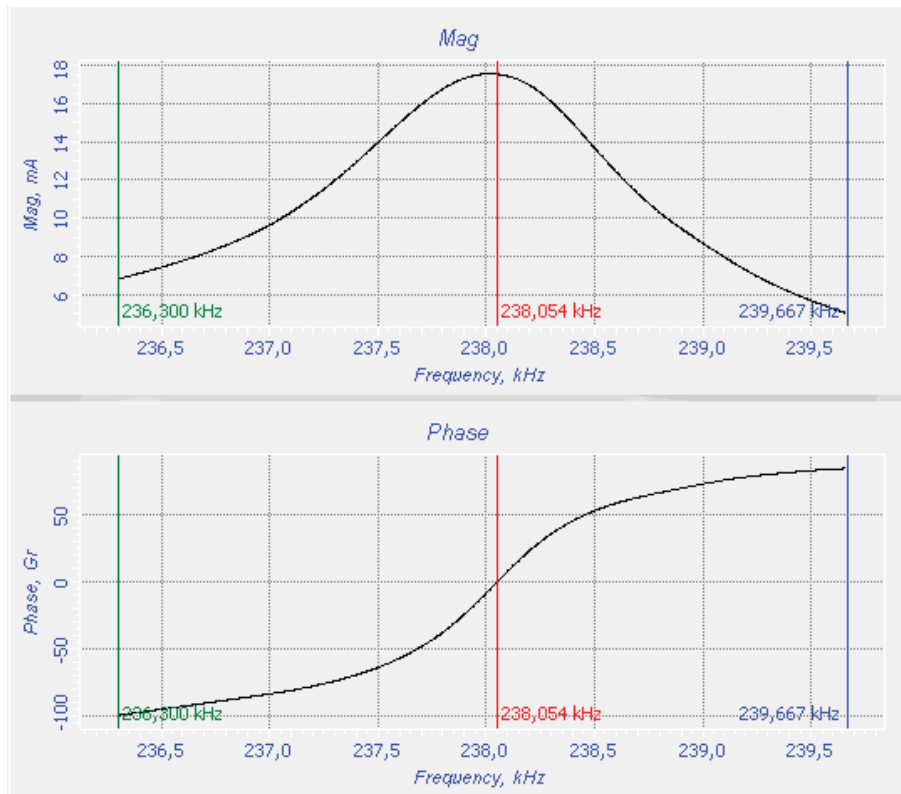
Fig. 8-4. Frequency dependencies of the signals **Mag** and **Phase**

Adjusting generator output parameters manually

1. Open the **Resonance** window by clicking the [Resonance] button in the Main Operations panel.

2. In the **Probes** drop-down list of the **Find Resonance** panel, select the desired type of the probe. This will preset parameters of automatic adjustment of the generator output parameters for the selected type of the probe.

3. Open the Frequency response parameters panel (see Fig. 8-2) by clicking the 🛠 button in the Resonance window Control.
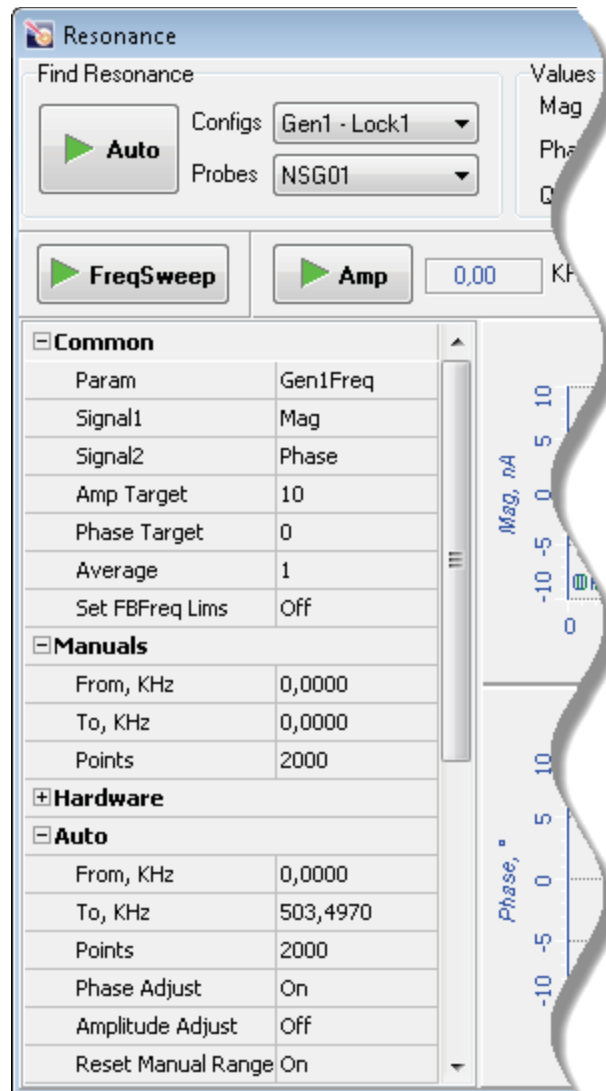
Fig. 8-5. Frequency response parameters panel

4. Defined the range of searching for the resonance frequency. This can be done in one of two ways:

   - with markers (by default, colors of the beginning and of the ending markers are green and blue, respectively);

   - by editing the fields **From** and **To** of the **Manuals** group.

5. Click the **▶ FreqSweep** button. This will acquire frequency responses of the magnitude and of the phase shift in the preselected frequency range.

6. Restrict the search range to cover only the desired resonance peak (Fig. 8-6).
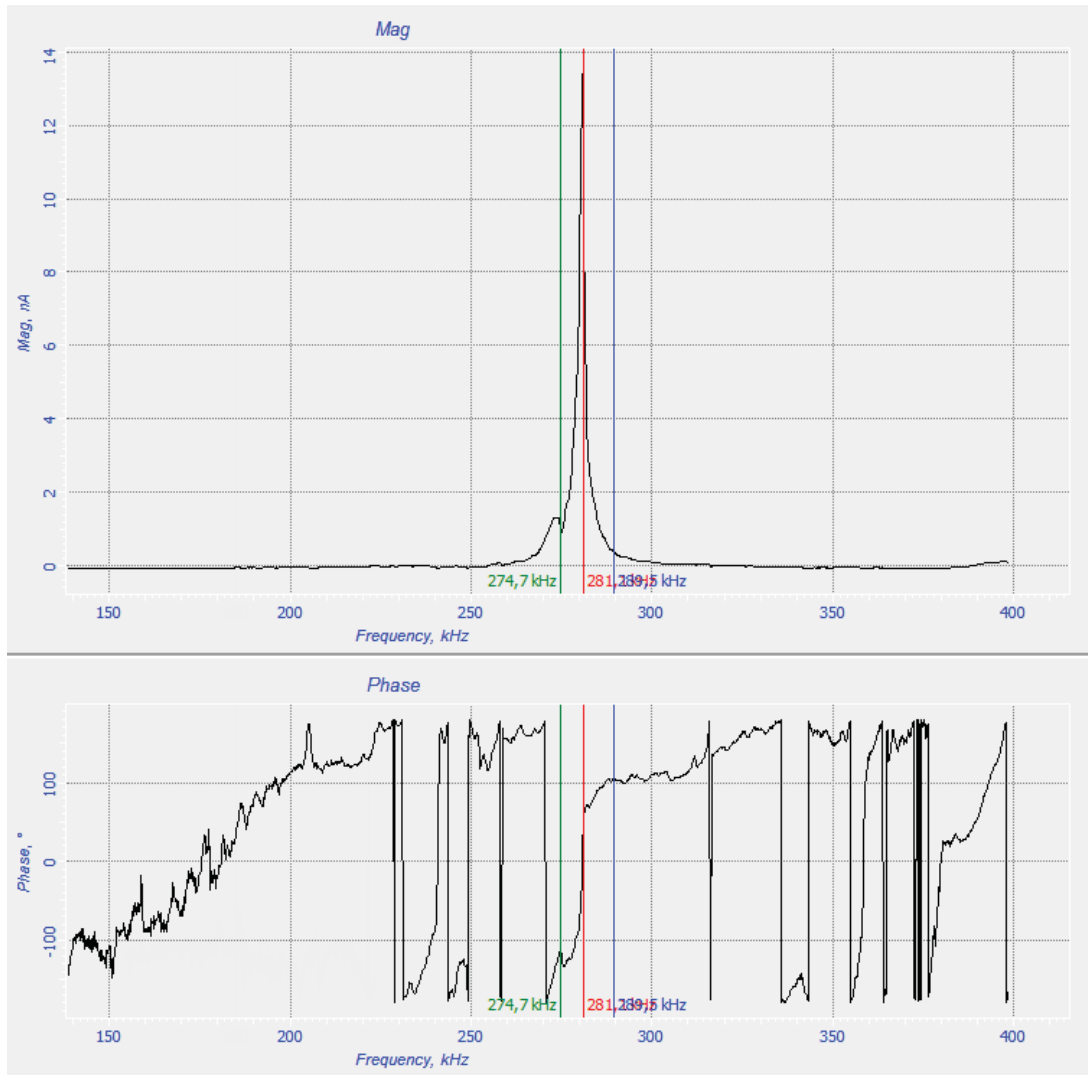
Fig. 8-6. Redefining range of taking frequency relations.
New limits are shown with the green and the blue markers

7. Repeat measurement of the frequency responses by clicking the ▶ **FreqSweep** button.

8. Adjust the generator working frequency at the maximun frequency of the resonance peak by moving the red marker to the position at which the **Mag** signal achieves its maximum in the vicinity of the selected peak (Fig. 8-7).
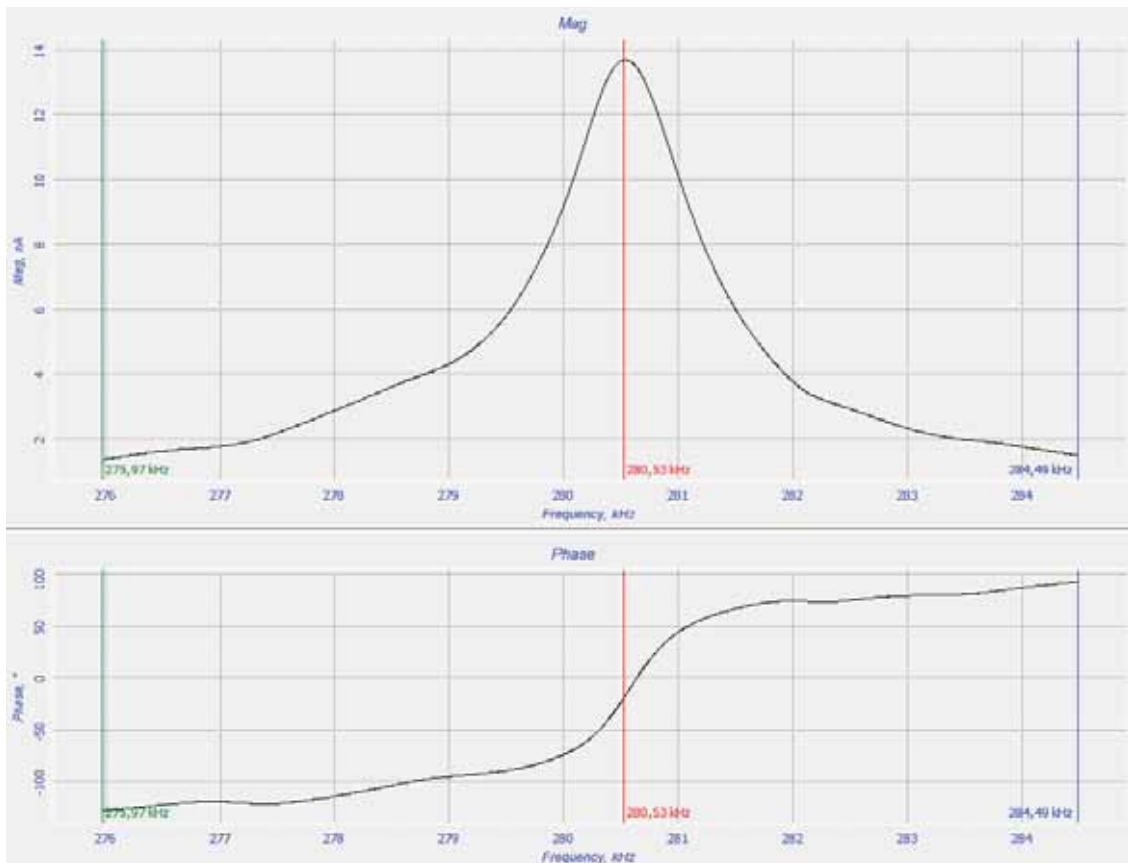
Fig. 8-7

9. Define the operating level of the **Mag** signal (cantilever oscillations amplitude).

    The **Mag** value can be set manually or automatically.

    In manual setting, the desired **Mag** level is adjusted through varying the parameters **Gen1Amp** (generator output amplitude) and **Lock Gain**. The Mag value can be monitored in the software oscilloscope or in the top bar of the Main Program window.

    For automatic setting the predefined level of **Mag**, click the [▶ Amp] button in the Additional adjustments panel (see Fig. 8-2, opens by clicking the [⬇] button). In automatic setting, the gain (**Lock Gain**) is adjusted first and then the generator output amplitude (**Gen1Amp**) is fitted. If the adjusted **Mag** level is not equal to the predefined one (possibly, due to discreteness of **Lock Gain** and **Gen1Amp**), repeat automatic adjustment by clicking the [▶ Amp] button.

    This will adjust the lock-in gain so as the **Mag** signal takes to the predefined level (**Amp Target**).

10. Set the desired phase by clicking the [▶ Phase] button. This will adjust the generator output phase so as the phase shift of the cantilever oscillations takes the predefined level (**Phase Target**).

11. Take the AFR and the phase frequency response again with the adjusted parameters by clicking the [▶ FreqSweep] button. Verify that the resonance peak is of a correct shape.

8.1.3. Approaching the Sample to the Probe

To approach the sample to the probe, perform the following steps:

1. Click the [⚄ Off] button in the top bar to close the feedback loop (the button will change its appearance to [⚄ On]). This starts extension of the Z scanner to its maximum. Progress of the extension is displayed in the Scanner extension indicator (see Fig. 8-8) by degree of the colored filling.
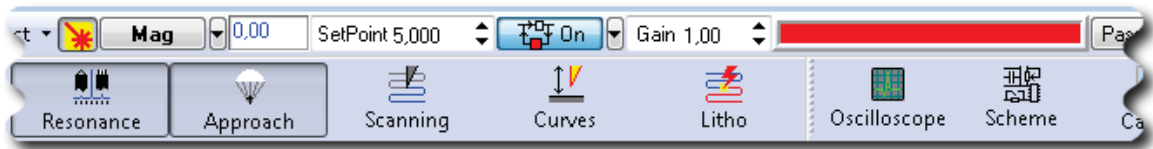
Fig. 8-8. The feedback loop is closed

2. Define the **SetPoint** parameter in the input field of the Main Parameters panel. Our recommendation is to take its initial value equal half of the **Mag** level (i.e. **SetPoint**=**Mag**/2).

3. Define the initial feedback gain in the **Gain** input field to be in the range of $0.5 \div 2$.

4. Open the **Approach** window by clicking the [Approach] button in the Main Operations panel.

5. Start the approach procedure with the [▶ Landing] button in the Control panel of the **Approach** window (see Fig. 8-9).

Fig. 8-9. Control panel of the **Approach** window

During the approach procedure, observe variations of the **Mag** signal in the software oscilloscope and state of the scanner extension indicator. Wait until the procedure completes.

Providing the approach parameters are set correctly, the approach procedure completes in about 10 to 30 seconds and the system goes to following state (see Fig. 8-10):
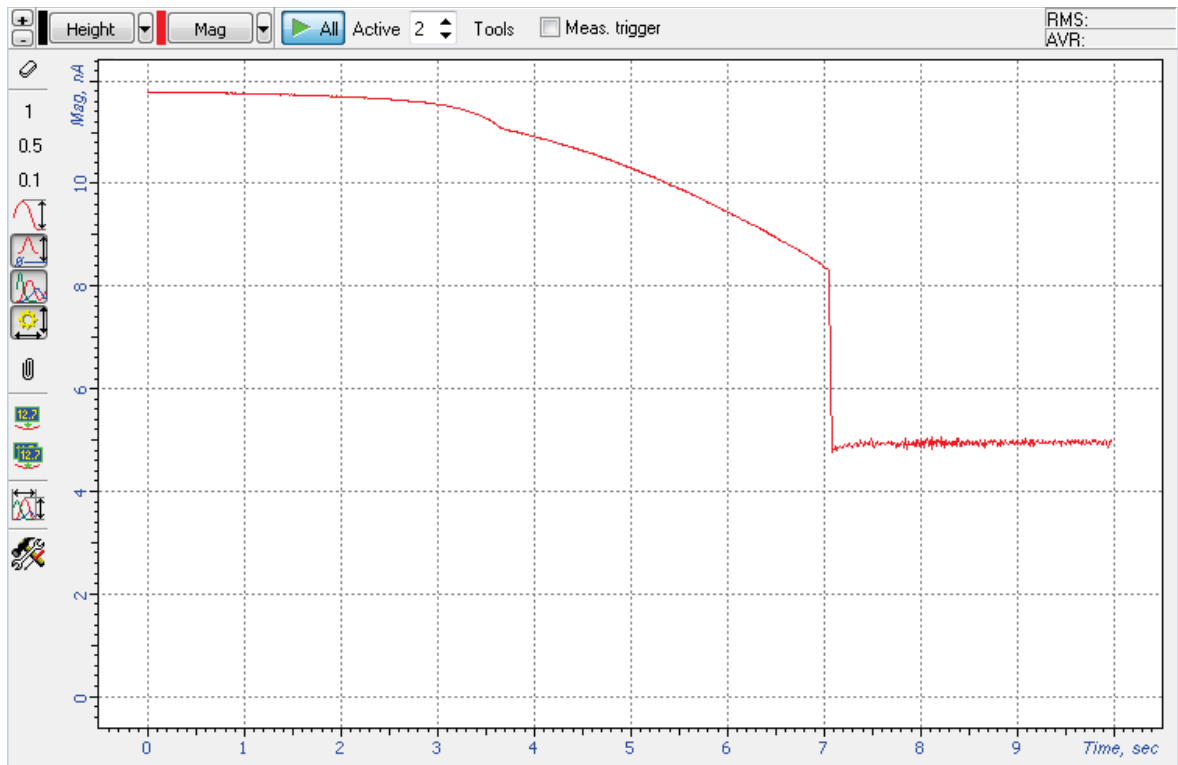
Fig. 8-10. Approach completed

- The **Mag** signal decreases to the level of parameter **Set Point**, the feedback loop maintains the Z-scanner in the position where the value of **Mag** is equal to **Set Point**. Note that this position of the scanner is approximately equal to half of the scanner extension range;

- The message "**Operation Completed**" appears at the right top of the Approach window.

- The stepper motor is disabled;

- Length of the indicator bar decreases down to some intermediate value (see Fig. 8-11).

Fig. 8-11. Scanner extension indicator after approaching

8.1.4.   Adjusting Working Level of the Feedback Gain

The greater is the level of the feedback gain (**Gain** parameter), the greater is data processing speed in the feedback loop. Nevertheless, at some high level of the feedback gain (let us call it threshold value), the mode of operation of the feedback loop becomes unstable and noise generation occurs. Under this condition, the **Mag** signal is supplied with a significant alternating component (see Fig. 8-12).
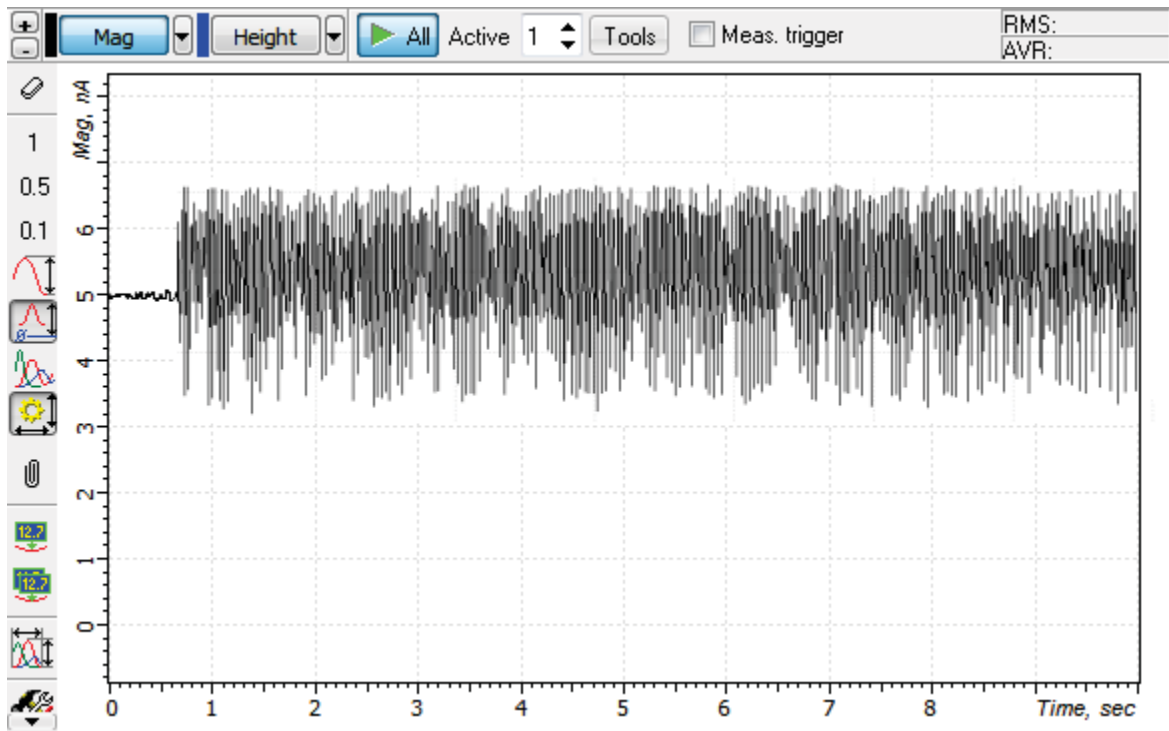
Fig. 8-12. Noise generation in the feedback loop

For stable operation, it is recommended to define the feedback gain to less 0.5÷0.7 of the threshold value where the noise generation occurs. The feedback gain is adjusted in the **Gain** input field.

To adjust the working level of the feedback gain, perform the following steps:

1. Double-click the **Gain** input field in the Main Parameters panel. The adjusting slider of the feedback gain will appear (see Fig. 8-13).
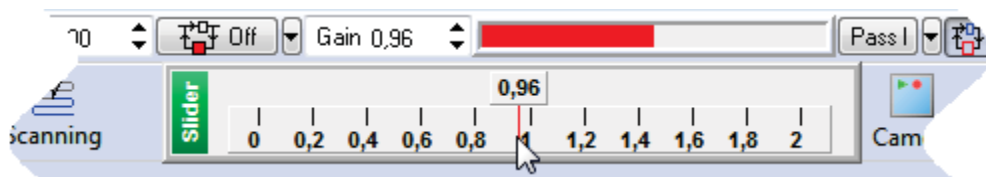
Fig. 8-13. Adjusting slider of the **Gain** parameter

90

2. Gradually decrease the **Gain** level and watch the **Mag** level in the software oscilloscope.

3. Determine the value of the **Gain** parameter at which noise generation occurs. The beginning of the generation is identified by appearance of a noticeable alternating component in the **Mag** signal (see Fig. 8-12).

4. Increase the value of the **Gain** parameter to 0.5÷0.7 of the threshold. This value will be used in further operation.

8.1.5. Adjusting Scanning Parameters

Open the **ScanForm** window by clicking the [Scanning] button.

The top part of the **ScanForm** window contains a panel to control the scanning process (see Fig. 8-14). The bottom of the window contains a Viewer to display topography of the surface under investigation.
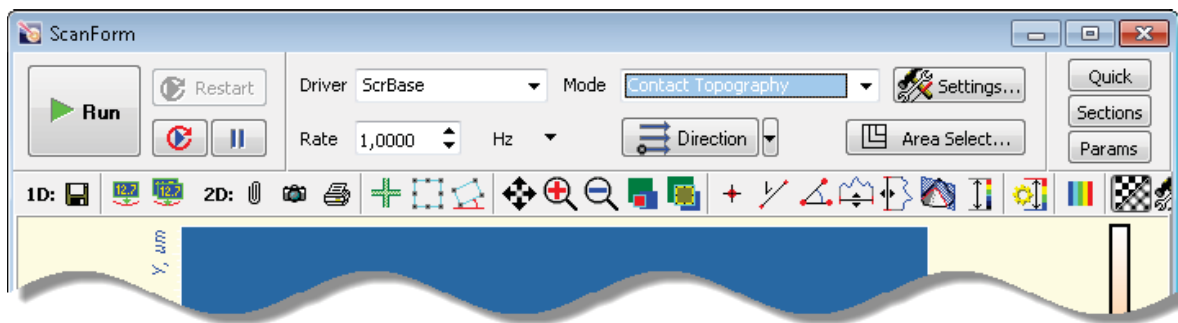
Fig. 8-14. Control panel of the **ScanForm** window

Selecting AFM mode

Select the **Semicontact Topo** mode in the **Mode** list of the Control panel (see Fig. 8-15). The **Height** signal will be selected for detection.
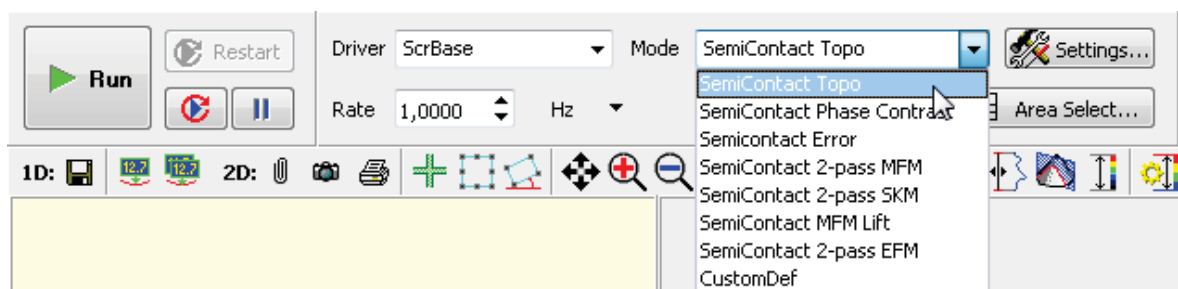
Fig. 8-15. Selecting the Semicontact Mode

<u>**Selecting the scan area**</u>

To choose preliminary dimensions of the scan area, regard for the following recommendations:

- If height difference in the surface profile is known to be within the scanner range, the maximum scan field is recommended;

- If height difference in the surface profile is unknown, a small sized scan area (about 10÷15 μm) should be preliminarily explored for finding proper levels of controlling parameters, including speed of scanning, **SetPoint**, **Gain**. Then the scan area can be enlarged.

Two options are provided for selecting the scan area:

- by defining the scan area parameters immediately (see p. 92) – number of scan points, rotation of the scan area in XY plane, and size of the scan area can be defined explicitly by the user;

- by defining the scan area in the 2D Data Viewer of the Scanning window (see p. 94) – only size and position of the scan area can be defined.

These options are explained below in details.

Defining Scan Area Parameters

Size and position of the scan area can be defined in the **Scan Area Selection** window (Fig. 8‑16) accessible by clicking the [⊞ Area Select...] button.
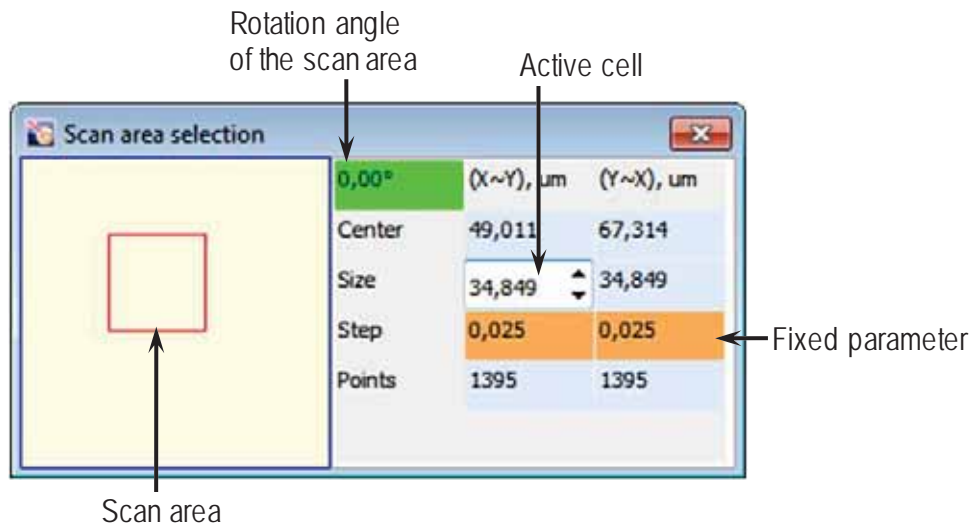
Fig. 8‑16. Scan area selection window

The red rectangle in the left part of the window displays the scan area currently selected with respect to the maximum scan area.

The following rules of modification of the scan area (the specific one is selected by clicking on titles of the 2nd and 3rd columns) are available:

- **X, Y** – X and Y dimensions of the scan rectangle are adjusted independently;
- **X~Y, Y~X** – when a particular dimension is changed, its counterpart will vary proportionally;
- **X=Y, Y=X** – the dimensions will be kept equal (square scan area).

   Position of the scan area can be redefined either with the mouse or with entering coordinates of the area center (in μm) in the **Center** fields. The origin of the coordinate system is at the left bottom corner of the maximum scan area.

Sizes of the scan area are defined by three parameters:

Size – size of the scan area in a particular direction (in μm). By default, dimensions of the scan area are 10×10 μm;

Step – distance between neighboring scan points in a particular direction (in μm);

Points – number of scan points in a particular direction. By default, number of points for every direction is 512.

To enable modifications in a particular cell, click on it with the mouse and the cell will become active. Value in the active cell can be edited in one of the following ways:

- with arrow keys <↑>, <↓>;
- with scroll keys ↕;
- with the slider (appears after double-click in the active cell).

The parameters **Size, Step, and Points** are interrelated through the formula:

$$Size = Step \times Points. \tag{1}$$

When a particular parameter varies, one of the two remaining follows these changes while the second remaining (fixed) keeps at the same level. The field of the fixed parameter is displayed on orange or purple background.

Purple background. Used for displaying the line being previously active.

Orange background. Appears after triple click on a parameter line. For changing the parameter to be fixed, click on the field of its name.

Rotation angle of the scan area in XY plane is defined in the green cell at the left top of the table.

NOTE. Scanning over a rotated scan area is not recommended if the XY displacement sensors are disabled.

Selecting Scan Area in Scanning Window

1. Enable the function of selecting the scan area by clicking the [⊡] button in the Toolbar.

   NOTE. If a rotated scan area is required, the function of selecting the scan area should be called by clicking the [▨] button (the [⊡] button will be hidden in this case).

2. Change size and position of the scan area with the mouse.

3. Click the [✛] button. Verify that the probe tip can contact the sample surface everywhere inside the selected scan area without penetration. To this purpose, press and hold the left mouse button and move the mouse pointer inside the scan area. Motion of the mouse pointer will be followed by displacement of the probe tip over the sample surface. Monitor extension of the piezoscanner in the indicator in the top bar of the Main Program window.

Fig. 8-17. 2D Data Viewer
Arrow indicates the boundary of the scan area

Selecting the scan direction

The scan direction is selected in the [⇄ Direction ▼] drop-down list of the Control panel of the Scanning window.

Adjusting the scanning speed

Optimal value of the scanning speed depends on sample surface properties, scan area dimensions and external conditions. A smooth surface can be scanned at higher speed than that with uneven topography and large height difference.

To define the **Rate** parameter, perform the following styeps:

1. In the Control panel of the **ScanForm** window, select unit of scan rate measurement (Fig. 8-18). For example, the **Hz** item is associated with the line scan frequency in Hz.
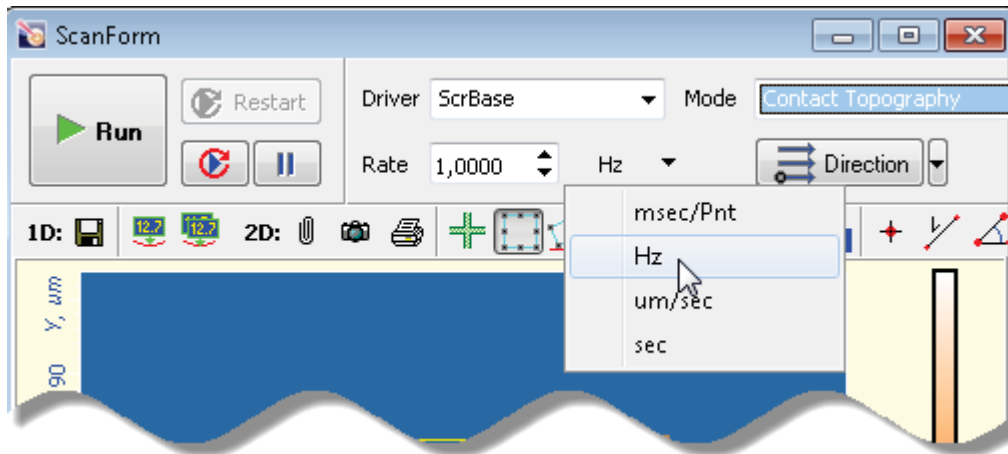
Fig. 8-18. Control panel of Scanning window

2. In the **Rate** field, define level of the scan rate. For testing scans, values not exceeding 0.5 Hz are recommended.

Selecting detection signals

Every measurement technique is supplied with a default set of recorded signals. This set can be modified if required. The Control program uses the latest set of recorded signals.

For every recorded signal a separate 2D Data Viewer is reserved in the **ScanForm** window. The instrument is capable to register up to eight signals in parallel.

The set of recorded signals can be modified in the **ScanSettings** window (accessible through clicking the ⚒ Settings... button in the Control panel of the **ScanForm** window).

Fig. 8-19. **ScanSettings** window

The **Scan Mode** field displays the SPM technique the set of recorded signals of which is viewed or edited at the moment.

The list of recorded signals is provided in the **Signals** column. Although data are measured both in forward (from beginning of the line to its end) and in backward (reverse) passes of the probe, the 2D image is generated only for one of those passes. That pass is selected in the **Direction** column as follows: **Forward** – the signal will be recorded for the forward pass, **Backward** – the signal will be recorded for the backward pass.

If displacement sensors are used, the **SensHeight** signal is recommended for recording because it is most sensitive to the surface landscape:

Height is a signal derived from voltage applied to the Z scanner.

SensHeight is a calibrated signal from the Z displacement sensor.

8.1.6.   Scanning

Hereafter, the scanning process will be explained with the example of a rectangular grating (standard grating TGQ-1, 3 µm resolution).

Starting measurements

Scanning of the sample surface should be preceded by necessary preparation procedures (the sample has approached to the probe, the operation point is adjusted, and all scanning parameters have been set).

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window (see Fig. 8-20).
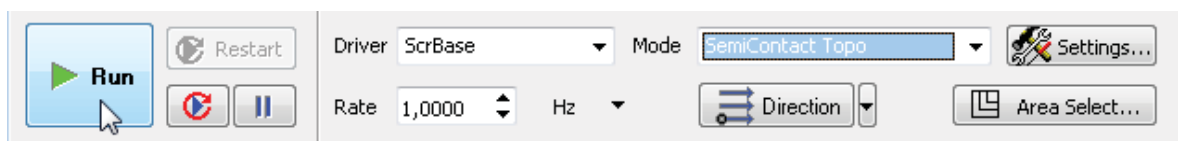
Fig. 8-20. Control panel of the **ScanForm** window

This results in the following activity of the system:

- Line-by-line scanning of the sample surface starts and the corresponding image of the scan area appears in the 2D Viewer of the scan data (see Fig. 8-21);
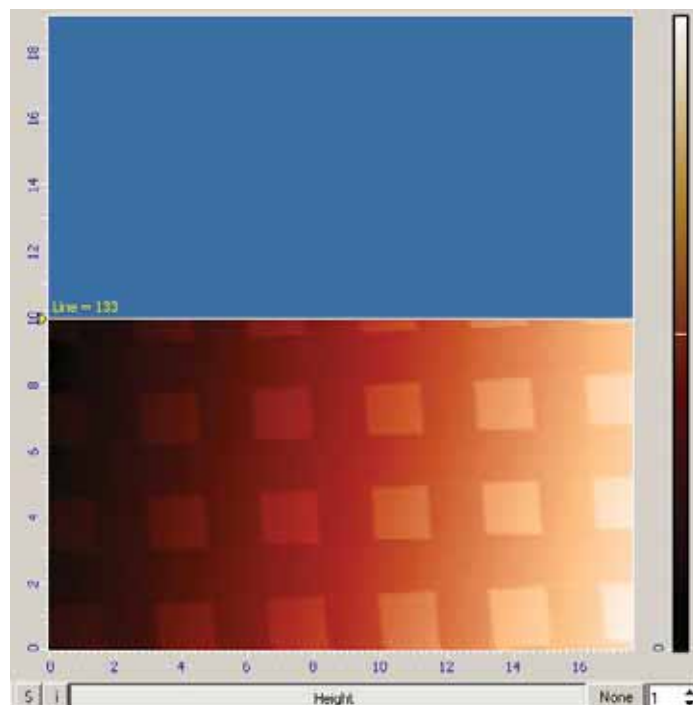
Fig. 8-21. Surface topography image

- If the **S** button in the left bottom of the 2D Viewer of the scan data is pressed, the 1D Viewer will display the detection signal in real time (see Fig. 8-22);
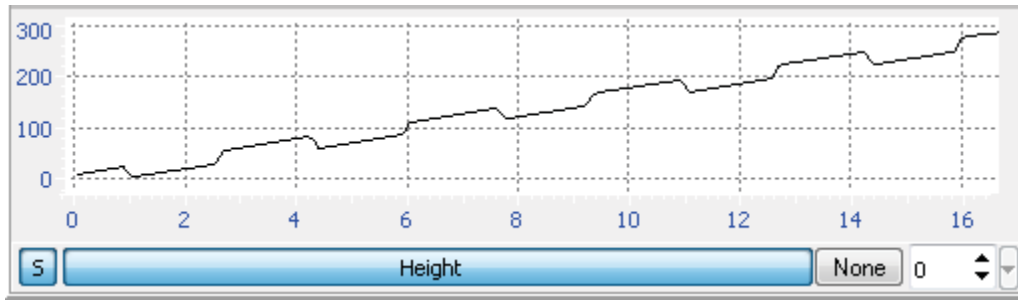
Fig. 8-22. **Height** signal

- The **Restart** button becomes enabled and caption of the **Run** button changes to **Stop** (see Fig. 8-23).
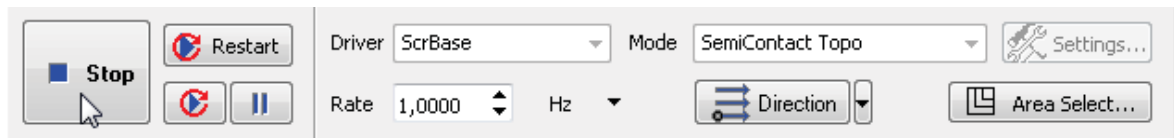
Fig. 8-23. Control panel of the **Scanning** window in the course of scanning

Should the scanning procedure be interrupted, click the **Stop** button or use the <Esc> key.

Tilt subtraction

Data in the example above (see Fig. 8-21, Fig. 8-22) reveal that the sample is tilted to some degree along the X direction.

Contribution of the tilt to the detected landscape can be eliminated immediately in the scan process with proper selection of the **Subtract** control at the right bottom of the **ScanForm** window. By default, this button is in the **None** state (see Fig. 8-22).

To activate run-time subtraction, click this button and select the **Plane** option in the corresponding list (see Fig. 8-24). This entails subtraction of the plane tilt, and the original image, illustrated in Fig. 8-21, is transformed into the image shown in Fig. 8-25.
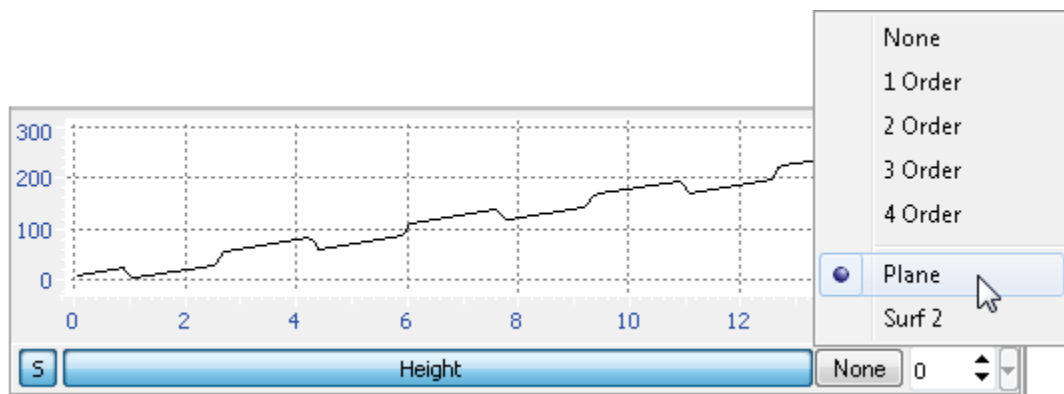
Fig. 8-24
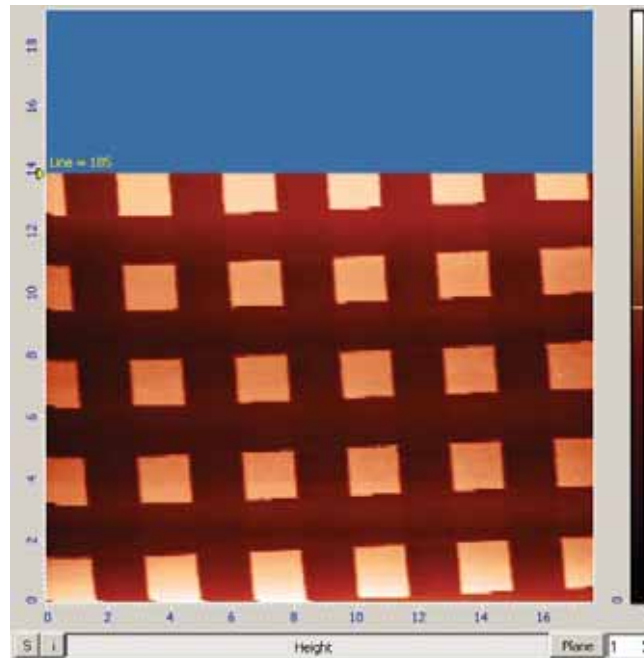
Fig. 8-25. Surface topography image

With the correction defined, the 1D Viewer of the scan data will display the detection signal corrected for the tilt contribution. The resulting signal is shown in Fig. 8-26 (compare with the signal in Fig. 8-22).
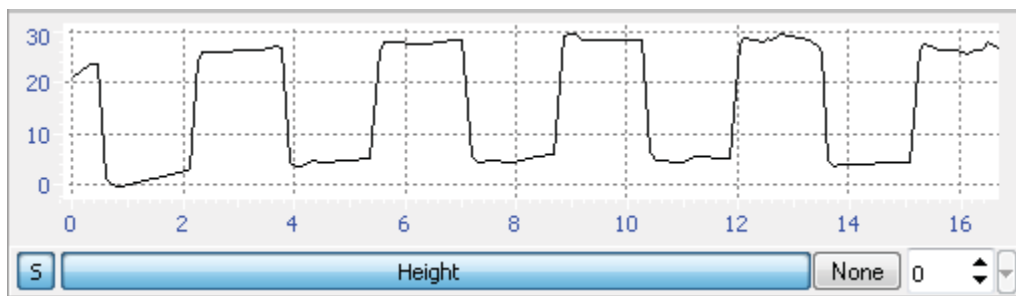
Fig. 8-26. **Height** signal

NOTE. Collected frames miss information on corrections of the scan data performed with the **Subtract** *option.*

Tuning parameters in the scan process

Quality of the acquired surface image essentially depends on such parameters as scanning frequency, **Rate** (see Fig. 8-27), the operating point, **Set Point** (see Fig. 8-28), and the feedback gain, **Gain**. Either of these parameters can be changed instantly in the course of scanning with visual inspection how modifications impact on the image being collected.
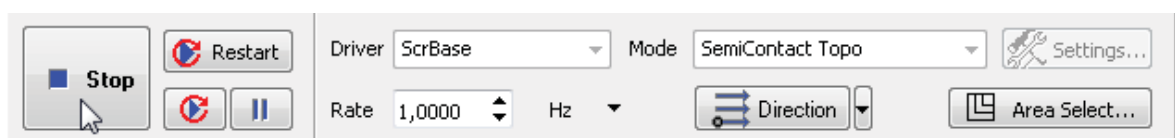
Fig. 8-27

Fig. 8-28

*NOTE. Scanning is recommended to restart (using the **Restart** button) after any of scanning parameters is changed.*

Some recommendations on optimization of scanning parameters

The choice of the optimal value of scanning speed depends on properties of the sample, on dimensions of the scan area, and on external conditions.

Smooth surfaces can be scanned at a speed higher than those with sharp features and steep slopes.

It is recommended to start scanning at a lower scanning speed, increasing it gradually until distortions affect the landscape profile.

Scanning speed should be reduced when surface features in the scan direction are not detected.

In measurements of soft materials, the images may be affected by "dragging effects" caused by surface features in the scan direction. In the event of such effects, it is recommended to reduce the scanning speed as well as to increase the **Set Point** level in order to lower pressure on the sample.

8.1.7. Saving Measurement Data

To save measurement data on the hard drive, perform the following steps:

1. Open the **Image Analysis** window by clicking the [Data] button in the Main Operations panel.

2. In the Main menu of the **Image Analysis** window, select the **File → Save All Frames** command.

3. In the standard dialog, select a folder for saving the collected data.

4. Type in a filename and save it with the extension ***.mdt**.

8.1.8. Operation Completing

1. Open the feedback loop by clicking the 〔🔲 On〕 button in the Main Parameters panel (the button will change its appearance to 〔🔲 Off〕).

2. Open the **Approach** window by clicking the 〔Approach〕 button. To prevent damage of the probe tip by the sample surface when removing the measuring head, retract the sample from the probe by clicking the 〔Move〕 button for several times (Fig. 8-29).
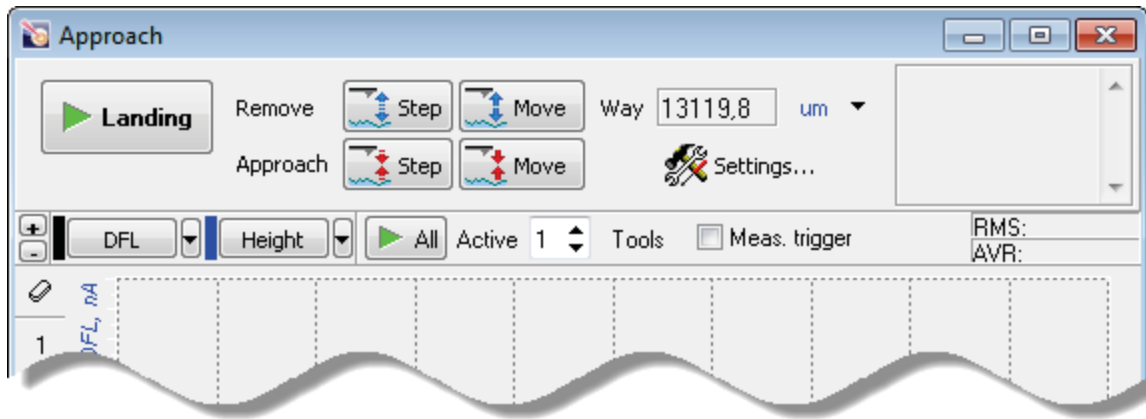
Fig. 8-29. **Approach** window

3. Move the heads to their inoperative positions with the 〔STM AFM〕 button

4. Close the Nova Px Control program.

5. Turn off the SPM controller.

6. Turn off the vibration isolation system.

7. Turn off the computer.

8.2. Semicontact Error Mode

8.2.1. Brief Description of the Mode

When scanning in the Semicontact mode, the value of the cantilever oscillation amplitude varies following the surface topography of the surface sample. The feedback loop tries to preserve the given level of the cantilever oscillation amplitude (**Set Point**) by maintaining the reference signal linked with the oscillation amplitude (in our case, this is the signal **Mag**). However the feedback loop cannot compensate for variations of the **Mag** signal instantaneously as it has some inertia (characterized by a time delay).

During scanning, the current value of the **Mag** signal (which is linked with the cantilever oscillation amplitude) is the error signal of the feedback loop and it contains additional information on surface topography. This signal can be used for a more detailed reproduction of the topography. The mode that allows imaging of surface topography by the Constant Force Mode simultaneously with measurements of the error signal (the **Mag** signal, in our case) is called the **Semicontact Error Mode**.

The Semicontact Error Mode, similarly to the Contact Error Mode, can be considered as an intermediate mode between the Constant Force Mode and the Constant Height Mode. This is possible providing that the speed of the error signal data processing (the feedback gain factor, in our case) is optimized so that the feedback loop is able to trace smooth topography variations while not been able to recognize sharp features. Then, scanning of smooth surfaces with smooth and extended features is performed with the piezo-scanner of almost constant length. As a result, the final image will have higher contrast for sharp features and lower contrast for smooth and large ones. This can be useful for identification of small features on the background of large and relatively smooth variations of the surface.

8.2.2. Scanning

<u>Initial state</u>

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

The Semicontact Error Mode is based on the Semicontact Mode, which is described in detail in i. 8.1 Semicontact Mode on page 80.

Before the Semicontact Error Mode measurements, prepare for the measurements and perform measurements of surface topography by the Semicontact Mode.

After the completion of preliminary measurements of surface topography by the Semicontact Mode, perform setting of parameters for operating by the Semicontact Error Mode. Select the **Semicontact Error** mode in the **Mode** list on the Control panel of the **ScanForm** window (Fig. 8-30). The **Height** and **Mag** signals will be selected for detection.

Fig. 8-30. Selecting the **Semicontact Error** Mode

Click the button **Run**, which is on the control panel of the **Scan** tab, to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts;
- Two images appear in the field of 2D visualization of the scanning data. One of the images is an image of surface topography (signal **Height**), while the other one is the error signal (signal **Mag**) (Fig. 8-31);

Fig. 8-31. Images of surface topography and the error signal (TGQ-1 grating)

- If the **S** button in the left bottom of the 2D Viewer is pressed, the 1D Viewer will display the detection signal line-by-line (Fig. 8-32).
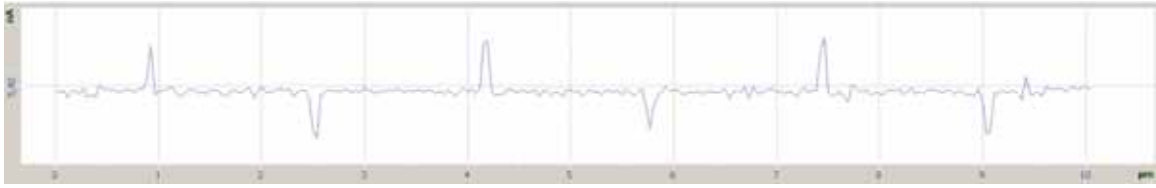
Fig. 8-32. Signal **Mag**

8.3. Phase Imaging Mode

8.3.1. Brief Description of the Mode

When scanning in the Phase Imaging mode, the tip of the oscillating probe periodically comes in touch with the sample surface. Its behavior is affected by the influence of various repulsive, adhesive, capillary and other forces. This can affect both the oscillation amplitude and phase. If the sample surface is inhomogeneous by its properties, this correspondingly results in some shift of the phase (Fig. 8-33).
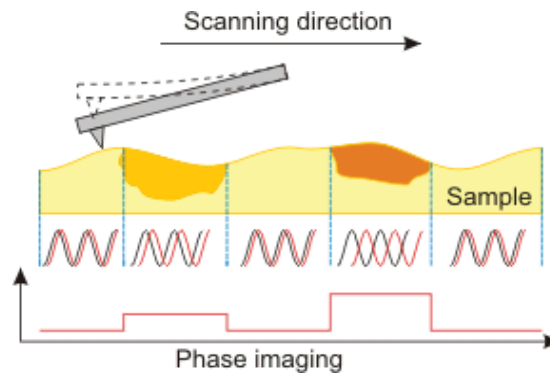
Fig. 8-33

The phase shift distribution over the sample surface visualizes distributions of characteristics of the sample substance.

The **Phase Imaging Mode** yields valuable information for a broad area of applications. In some cases it can uncover hidden contrasts in materials properties. This modes employed, for example, in the study of biological objects, samples with electrical and magnetic properties and a number of other areas.

8.3.2. Scanning

<u>Initial state</u>

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the AFM head to its working position;
- The sample approached to the tip to distance of 1÷2 mm;
- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

Before the Phase Imaging measurements, prepare for the measurements and perform measurements of surface topography by the Semicontact mode (see i. 8.1 on page 80).

After the completion of preliminary measurements of surface topography by the Semicontact Mode, perform setting of parameters for operating by the Phase Imaging Mode. Select the mode **Semicontact Phase Contrast** from the list **Mode** (scan mode list) of the control panel of the **ScanForm** window (Fig. 8-34). The **Height** and **Phase** signal will be selected for detection.
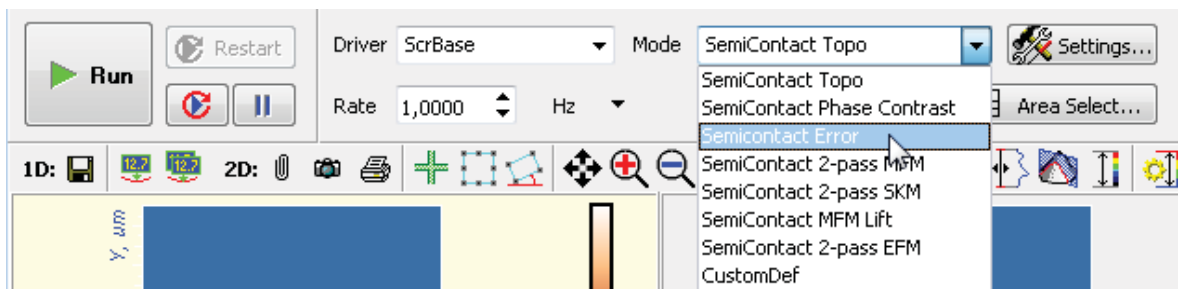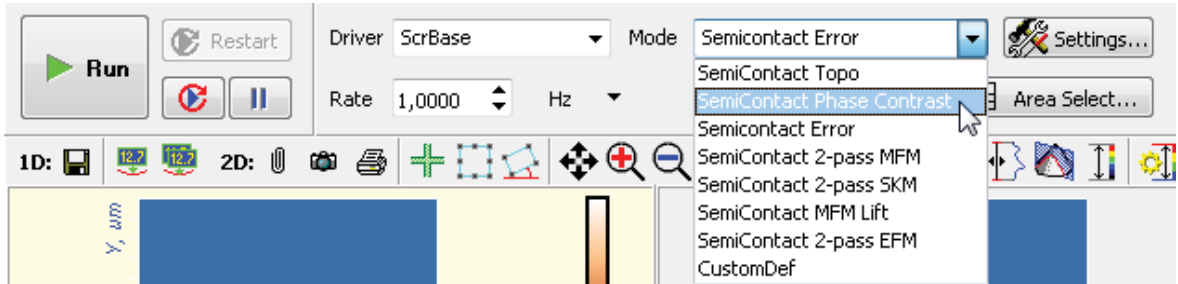

Fig. 8-34. Selection of the Phase Imaging Mode

Click the button **Run**, which is on the control panel of the **ScanForm** window, to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts;
- Two images appear in the field of 2D visualization of the scanning data. One of the images is an image of surface topography (signal **Height**), while the other one is the phase distribution (signal **Phase**) (Fig. 8-35);
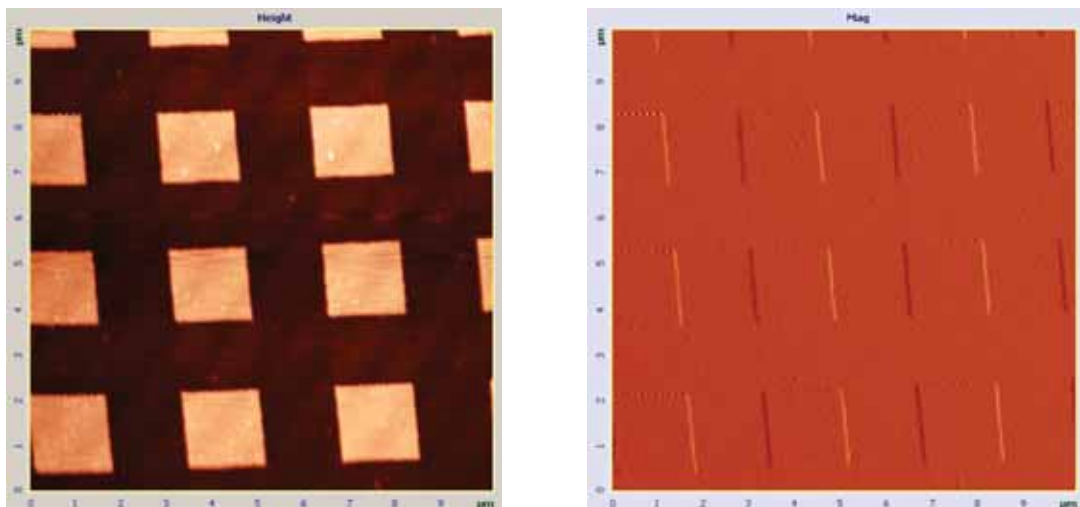- If the **S** button is pressed, the 1D Viewer will display the detection signal of selecting image in real time.
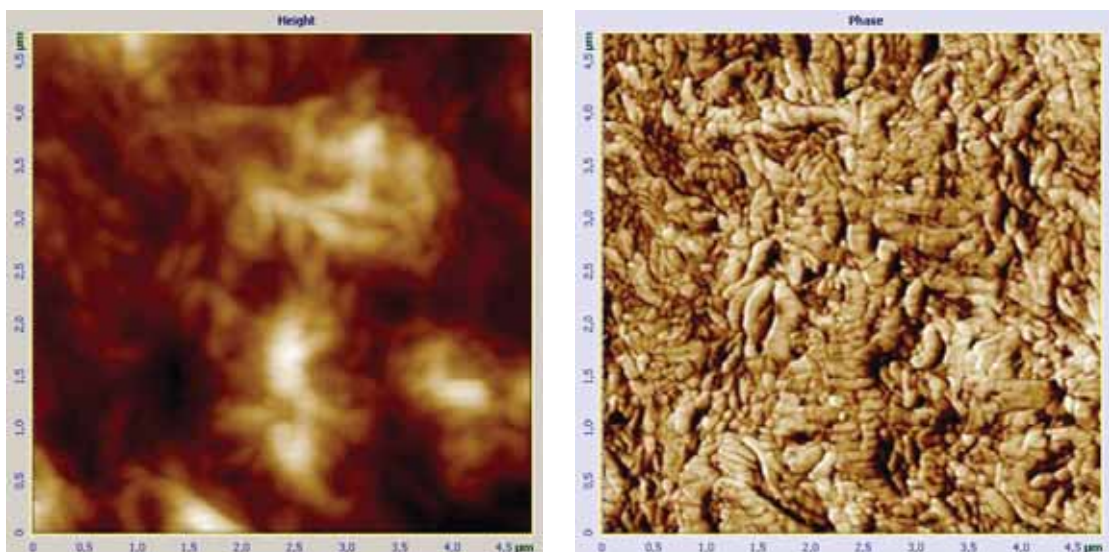
Fig. 8-35. Images of surface topography and phase distribution of polyethylene

8.3.3.   Modes of Improving Image Quality

Image quality can be improved by the following:

- Alter parameter **Set Point** (pressure between the probe and the sample);
- Alter initial phase of the generator (parameter **Gen1Phase**) (Fig. 8-36);
- Alter amplitude of cantilever oscillations (parameter **Gen1Amp**) (Fig. 8-36);
- Replace the probe (with different elasticity and resonance frequency).
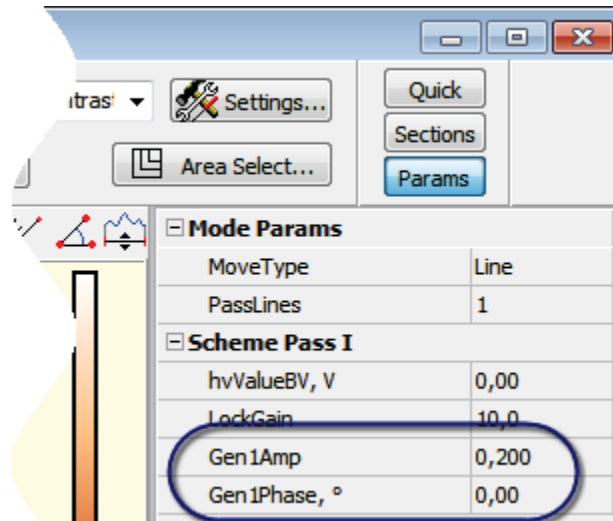
Fig. 8-36. Additional parameters panel

9. Many-Pass AFM Techniques

This chapter assumes the reader is experienced in performing measurements with NT-MDT instruments using techniques of contact and semicontact atomic force microscopy.

In general many-pass techniques are used in tasks where other than topography data are to be obtained at that topography unwanted side effects must be eliminated. The line scanning proceeds as follows. The first pass along the scanning line is performed in a contact or semicontact mode to collect local topography characteristics. During the second pass, acquiring of electrical forces (or potentials), magnetic fields, dissipations, capacitance distributions *etc.* is performed. To avoid any influence of surface features on data collected during the second pass, the probe is raised above the surface at a distance dZ and then moves following the surface topography contour. The distance dZ is defined so that the clearance between the oscillating probe and the sample would be big enough to avoid the influence of surface topography on measurement results. However, this distance should not be too big so that the detection signal is strong enough and the lateral resolution is not lost. Some techniques needs the third pass to minimize the influence of surface electrical fields (in addition to the influence of surface topography features).

9.1. Magnetic Force Microscopy

9.1.1. Brief Description

Magnetic Force Microscopy (MFM) is a technique for imaging of spatial distributions of magnetic fields on the sample surface. MFM is used to study properties of magnetic data storage devices, magnetic structure of magnetics with sub-micron resolution, magnetic fields of busbars *etc.* For this technique, elimination of the influence of surface topography features on the image of the magnetic forces distribution is the most critical problem. To solve this problem, magnetic measurements are performed using the two-pass measurement scheme.

Scanning proceeds as follows. In the first pass, surface topography of the scan line is measured in the Semicontact Mode (see Fig. 9-1).
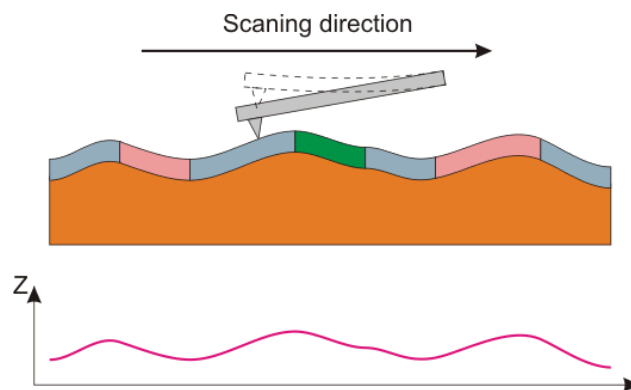
Fig. 9-1. First pass
Surface topography imaging

Then, the probe is lifted above the surface to the height dZ. In the second pass, the probe moves over the surface along the same line following the surface topography contour (see Fig. 9-2) maintaining the surface-probe distance constant. This distance should be big enough to eliminate the surface topography influence. The probe is only affected by long-range forces whose most prominent yield is due to magnetic forces of the sample. On the other hand, the value of dZ should not be too great as that would reduce the measured signal and impair the lateral resolution.
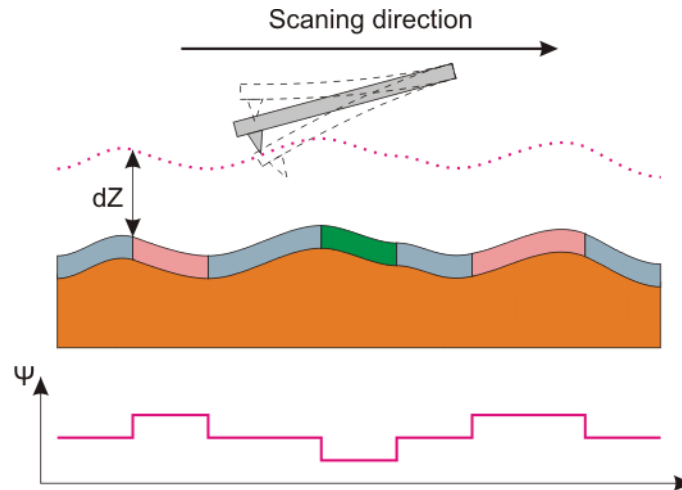
Fig. 9-2. Second pass
Ψ – phase shift

In the second pass, the piezodrive excites oscillation of the probe at its resonance frequency. Imaging of the Z-component of the magnetic forces gradient on the sample surface is performed through data on variations of the probe oscillation phase.

9.1.2.   Preparations for Measurements

MFM uses magnetic probes which are typically made of silicon coated with a thin magnetic film.

NOTE. The magnetic probes may become demagnetized after long-term storage. Before using a probe, magnetize it with a permanent magnet.

Hereafter, the scanning process will be explained with the example of a hard magnetic disk.

<u>Initial state</u>

The following steps are assumed to be done:

- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;

- Adjusting the cantilever deflection detection system;

- Mounting the sample;

- Moving the measuring head to its working position;

- The sample approached to the tip to distance of 1÷2 mm;

- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

Measuring in the MFM Mode are generally similar to those of the Semicontact Mode, which are described in detail in item. 8.1 "Semicontact Mode" on p. 80 . A sequence of basic procedures, along with a detailed description of instrument adjustment and specifics of MFM measurements, is provided below.

Procedural Sequence

Measurement in a MFM mode is arranged in the following procedural sequence:

1. Switching the Instrument for Operating in the Semicontact Modes.

2. Taking Resonance Curve and Adjusting Parameters of Generator Output.

3. Approaching the Sample to the Probe.

4. Adjusting Working Level of the Feedback Gain.

5. Adjusting Scanning Parameters.

6. Scanning.

Besides, you have to perform a preliminary scan of the sample in the Semicontact mode (see "Semicontact Mode" on p. 80) and then to select the scan area for MFM measurements.

9.1.3. Scanning

After the completion of preliminary measurements of surface topography by the Semicontact Mode, switch the instrument for operating in the MFM mode by selecting **Semicontact 2-pass MFM** in the list (see Fig. 9-3) of the Control panel of the **ScanForm** window. The **Height** signal will be selected for detection on the first pass and the **Phase** signal – on the second pass.
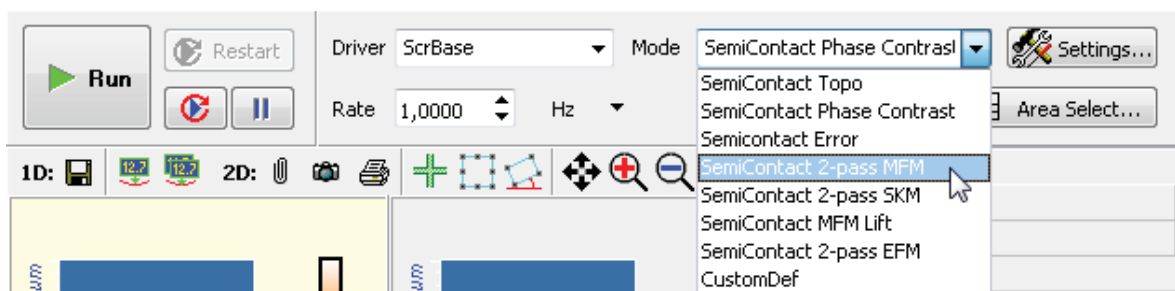
Fig. 9-3. Selecting the Magnetic Force Microscopy mode

Starting measurements

Scanning of the sample surface should be preceded by necessary preparation procedures (the sample has approached to the probe, the Set Point is adjusted, and all scanning parameters have been set).

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window.

This results in the following activity of the system:

- Line-by-line scanning of the sample surface starts;

- Two images of the scan area appear in the 2D Viewer of the scan data. One of these images reveals the landscape taken with the **Height** signal and the second image displays distribution of the z-component of the gradient of the magnetic force over the sample surface (**Phase** signal) (see Fig. 8-27);
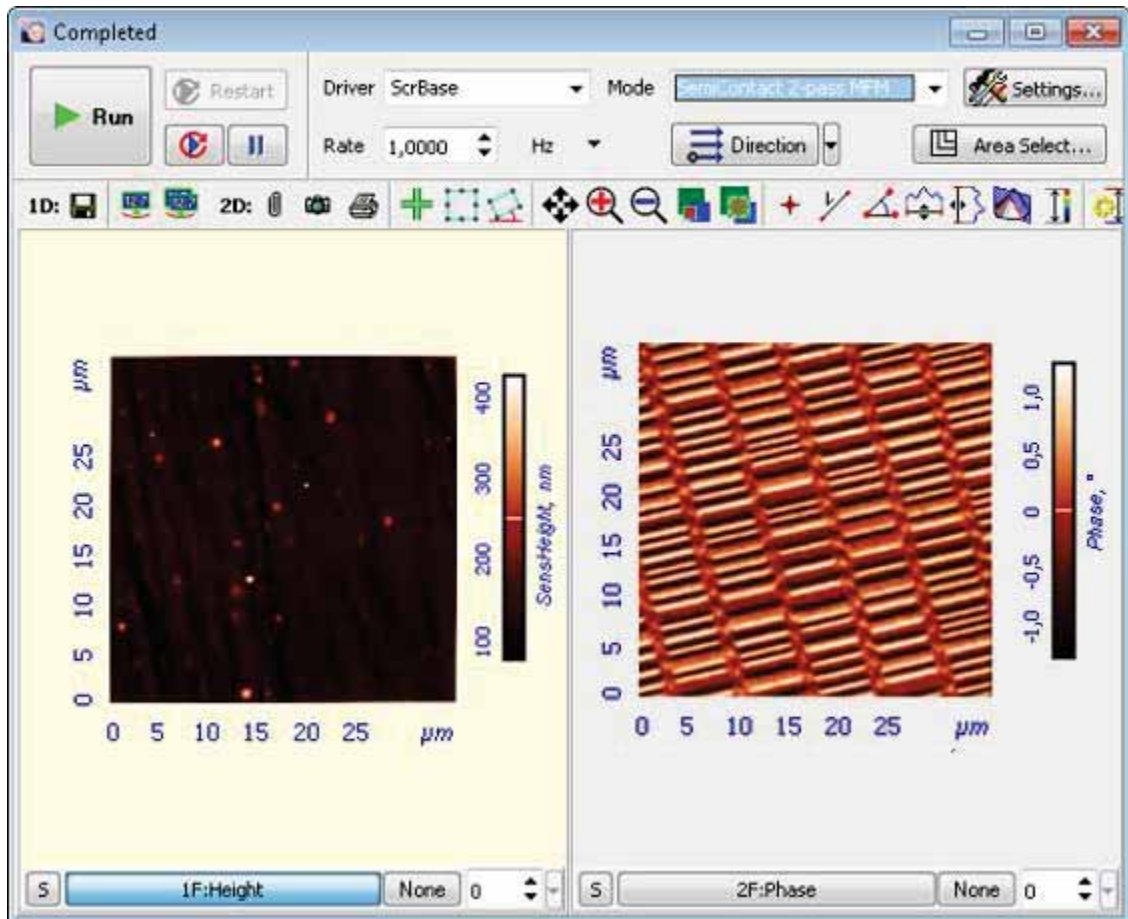
Fig. 9-4. Images of surface topography (left) and spatial distribution
of the Z-component of the magnetic forces gradient
on the surface of a magnetic hard disc (right)

*NOTE. During the second pass, input of the feedback is taken from the profile of the **SensZ** signal collected at the first pass.*

How to improve the image

When the MFM mode is selected, the parameters of the second pass are set to their defaults. The acquired image can be improved with run-time adjustment of the following parameters:

DZ probe-surface distance (offset) kept during the second pass. The larger **DZ**, the smaller is effect of the landscape variation on the detection signal of the second pass. However, increase of the offset reduces sensitivity of the technique.

Gen1Ampamplitude of the generator output. Reducing the generator output (and thus the amplitude of the probe oscillation) improves sensitivity of the technique but worsens the signal/noise ratio.

To correct the parameters of the second pass, perform the following steps:

1. Open the Additional Scanning Parameters panel by clicking the **Params** button in the **ScanForm** window (Fig. 9-5).

Fig. 9-5

2. Observe quality of the acquired image and vary the sample-probe distance. This needs modification of the **DZ** parameter in the **Mode Params** group (Fig. 9-6).
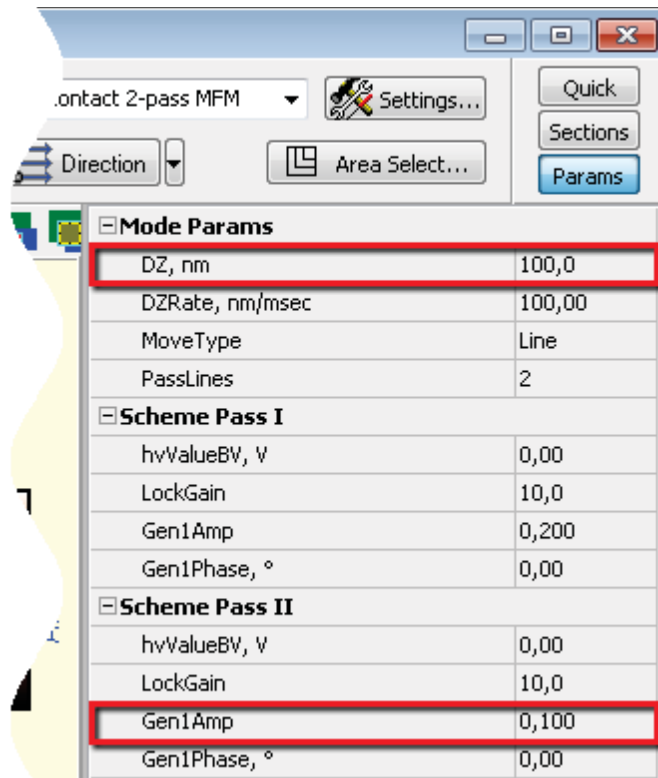
Fig. 9-6. Additional Parameters panel

3. Observe quality of the acquired image and vary the generator output amplitude. This needs modification of the **Gen1Amp** parameter in the **Pass II** group (Fig. 9-6).

The rest of adjustments and modifications available in the course of scanning are explained in i. 8.1.6 "Scanning" on p. 97).

9.2. Kelvin Probe Microscopy

9.2.1. Brief Description

Kelvin Probe Microscopy (SKM) is a technique to study distribution of surface electric potentials. This technique uses the effect that amplitude of the probe oscillation (excited by electric fields at the resonance frequency) in the electric field of the surface is zero when a proper dc bias U_0 is applied to the probe.

The value of the ac component, $U_1 \cdot \sin(\omega t)$, of the bias voltage should be big enough to induce oscillations of the probe. Frequency of the ac electric field is selected to be equal to the resonance frequency of the probe.

Once amplitude of the oscillation becomes zero at some value of the dc bias, this voltage U_0 is equal to the local surface potential. Influence of surface topography features on the measurement data is eliminated with using the two-pass measurement scheme.

Scanning proceeds as follows. In the first pass, surface topography of the scan line is measured in the Semicontact Mode (see Fig. 9-7).
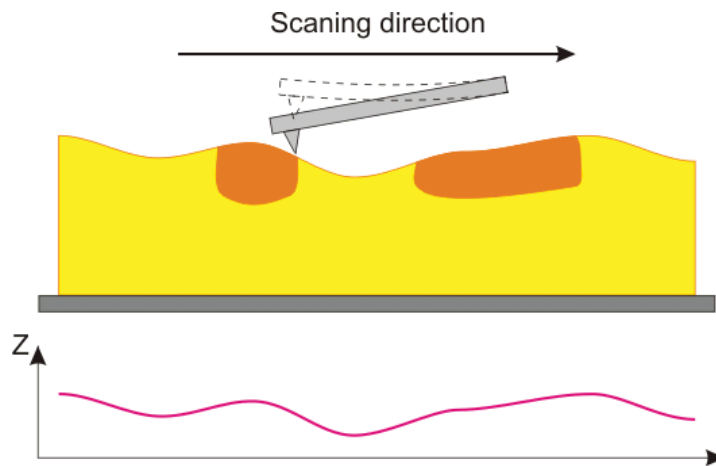
Fig. 9-7. First pass
Surface topography imaging

Then, the probe is lifted above the surface to the height dZ. An ac voltage $U_1 \cdot \sin(\omega t)$ is applied to the probe. This voltage induces oscillations of the probe at its resonance frequency ω. The probe oscillation amplitude is maintained equal to zero through the feedback loop by altering the dc component of the bias voltage $U_0(x,y)$. The probe moves over the surface following the surface topography contour (see Fig. 9-8).
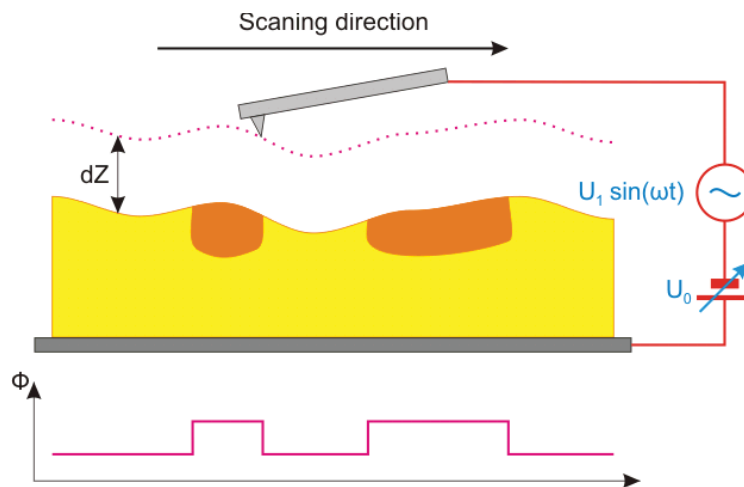
Fig. 9-8. Second pass
Φ – Surface potential

During the second pass the distance between the scanned surface and the probe is kept constant. This distance should be big enough to eliminate the surface topography influence. In this case, long-range forces only affect the probe. On the other hand the value of dZ should not be too great as that would reduce the measured signal and impair the lateral resolution.

9.2.2.   Preparation for Measurements

The Kelvin Probe Microscopy uses substrates with the spring for mounting a sample (see item 6.2 "Mounting Sample" on p. 20) and conductive probes for semicontact modes.

To provide an example of operations in the Kelvin Mode, we will use a bicomponent LB film.

Initial state

The following steps are assumed to be done:

- Launching the control program;

- Turning the controller SPM on;

- Installing the probe;

- Adjusting the cantilever deflection detection system;

- Mounting the sample;

- Moving the measuring head to its working position;

- Initial approach;

- Selecting the investigation area.

For details on these operations see Ch. 6 "Preparing for Measurements" on p. 19.

Besides, you have to perform a preliminary scan of the sample in the Semicontact mode (see sect. 8.1 "Semicontact Mode" on p. 80) and then to select the scan area for SKM measurements.

9.2.3.   Scanning

1. Switch the instrument for operating in the SKM mode by selecting **Semicontact 2-pass SKM** of the Control panel of the **ScanForm** window (seeFig. 9-9). The **Height** signal will be selected for detection on the first pass and the **SurfPot** signal – on the second pass.
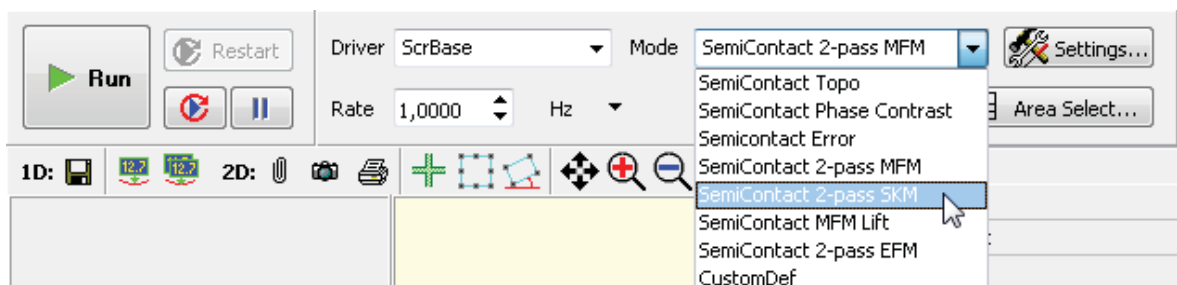
Fig. 9-9. Selecting the Kelvin Probe Microscopy

2. Once the **Semicontact 2-pass SKM** method is selected, the **Input** dialog box will appear to define the cutoff frequency of the low-pass filter for the second pass. Enter 2000 in the input field. Click the **OK** button.
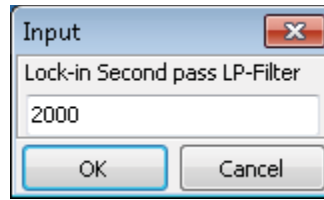
Fig. 9-10. **Input** dialog box

3. Select the scan area and define the scanning rate.

4. To start scanning, click the **Run** button (see Fig. 9-11).
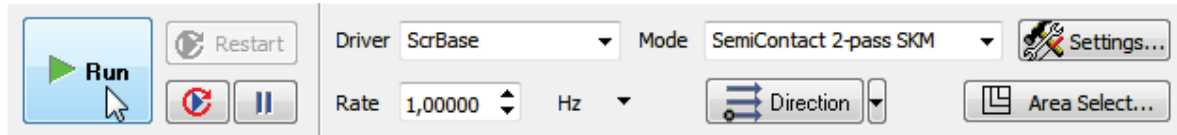
Fig. 9-11

This results in the following activity of the system:

● Line-by-line scanning of the sample surface starts;

● Two images of the scan area appear in the 2D Viewer of the scan data. One of these images reveals the landscape taken with the **Height** signal and the second image displays distribution of the surface potential (**SurfPot** signal) (see Fig. 9-12);
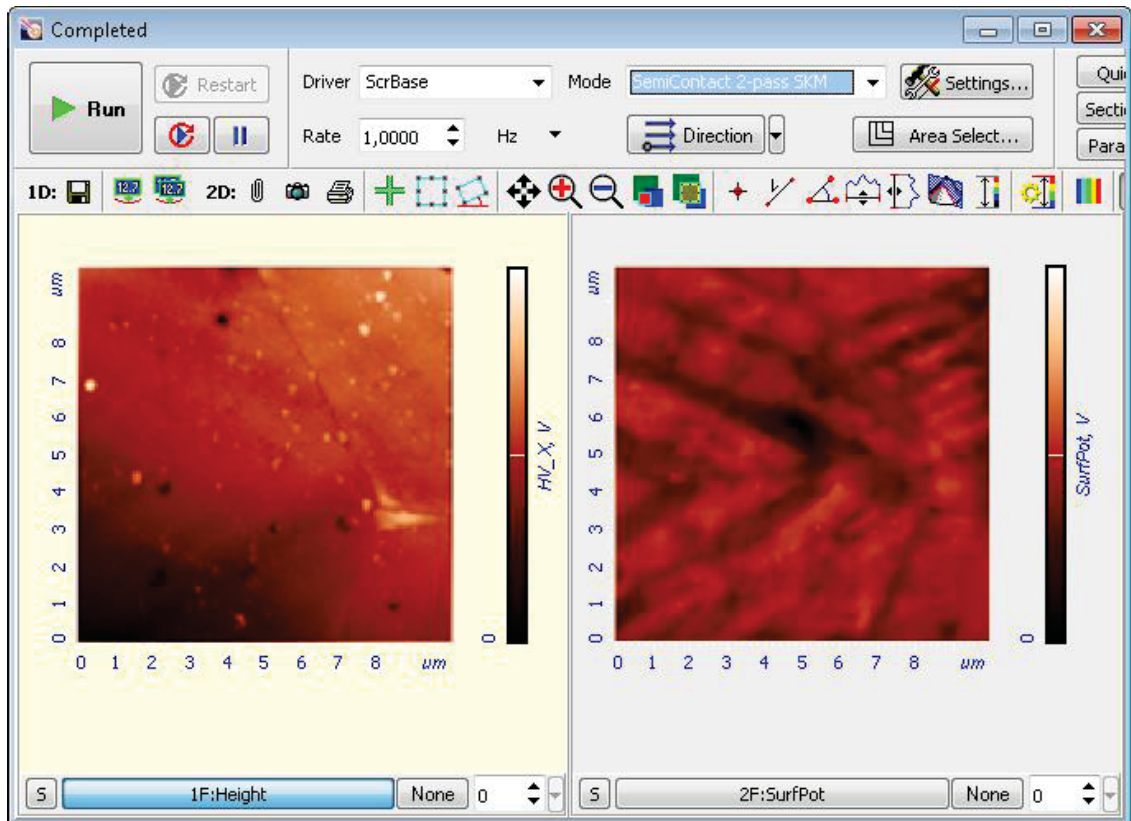
Fig. 9-12. Images of surface topography (left) and spatial distribution of the surface potential (right) over the surface of a LB bicomponent film

*NOTE. During the second pass, input of the feedback is taken from the profile of the **SensZ** signal collected at the first pass.*

How to improve the image

When the SKM mode is selected, the parameters of the second pass are set to their defaults. The acquired image can be improved with run-time adjustment of the following parameters:

DZ   probe-surface distance (offset) kept during the second pass. The larger **DZ**, the smaller is effect of the landscape variation on the detection signal of the second pass. However, increase of the offset reduces sensitivity of the technique.

Gen1Amp   amplitude of the generator output. Reducing the generator output (and thus the amplitude of the probe oscillation) improves sensitivity of the technique but worsens the signal/noise ratio.

To correct the parameters of the second pass, perform the following steps:

1. Open the Additional Scanning Parameters panel by clicking the **Params** button in the **ScanForm** window (Fig. 9-3).

Fig. 9-13

2. Observe quality of the acquired image and vary the sample-probe distance. This needs modification of the **DZ** parameter in the **Mode Params** group (Fig. 9-14).

Fig. 9-14. Additional Parameters panel

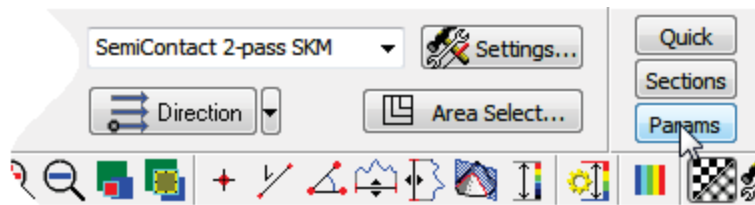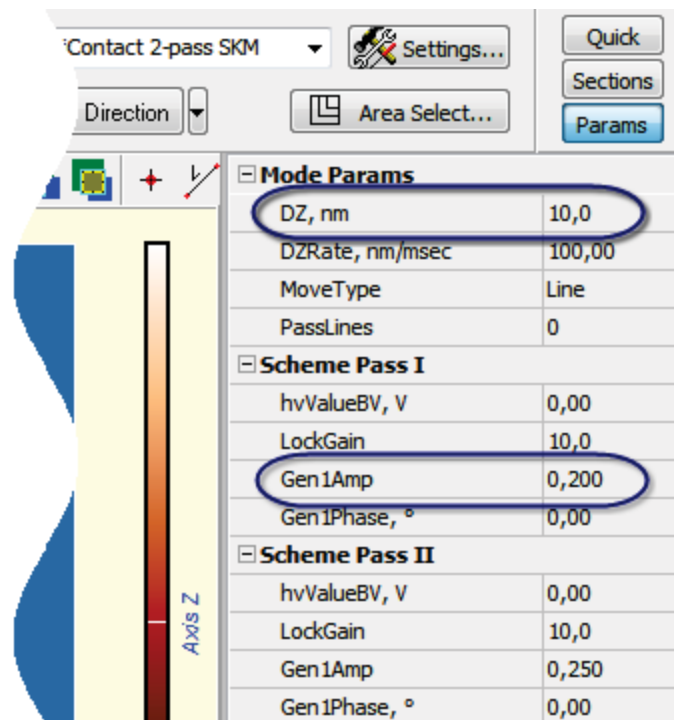3. Observe quality of the acquired image and vary the generator output amplitude. This needs modification of the **Gen1Amp** parameter in the **Pass II** group (Fig. 9-14).

Other run-time operations on adjusting the parameters are explained in i. 8.1.6 "Scanning" on p. 97.

9.3. Electric Force Microscopy

9.3.1.   Brief Description of the Technique

Electric Force Microscopy (EFM) is an efficient technique for imaging of electric fields and electric charges distributions on the sample surface with sub-micron resolution. Images obtained by means of this technique are interpreted as spatial distributions of the Z-component of the electric field gradient on the sample surface. Minimization of the influence of surface topography features on the measurement results is achieved by means of the two-pass measurement scheme.

The following procedure is performed during the process of scanning. Surface topography of the scanning line is determined in the first pass in the Semicontact Mode of operation (Fig. 9-15).
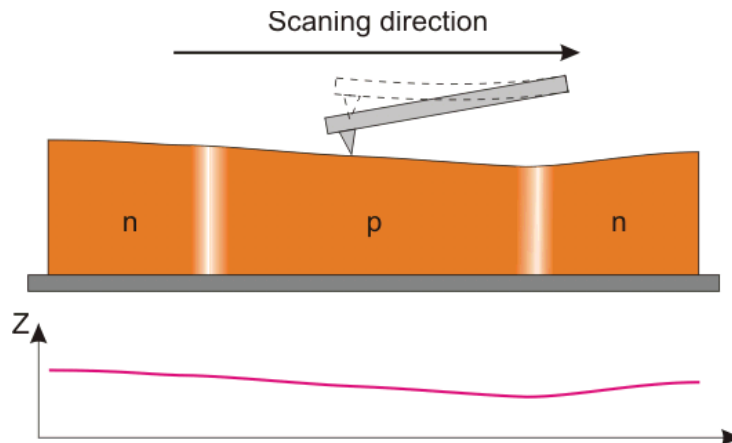
Fig. 9-15. First pass
Surface topography imaging

Then, in the second pass, the probe is lifted above the surface at the height dZ. The piezodriver induces oscillations of the probe at its resonance frequency and a dc bias voltage U_0 is set between the probe and surface. During the second pass the probe is moved over the surface following the surface topography contour (Fig. 9-16). Imaging of the Z-component of the electric field gradient on the sample surface is performed by means of reading variations of the probe oscillation phase.
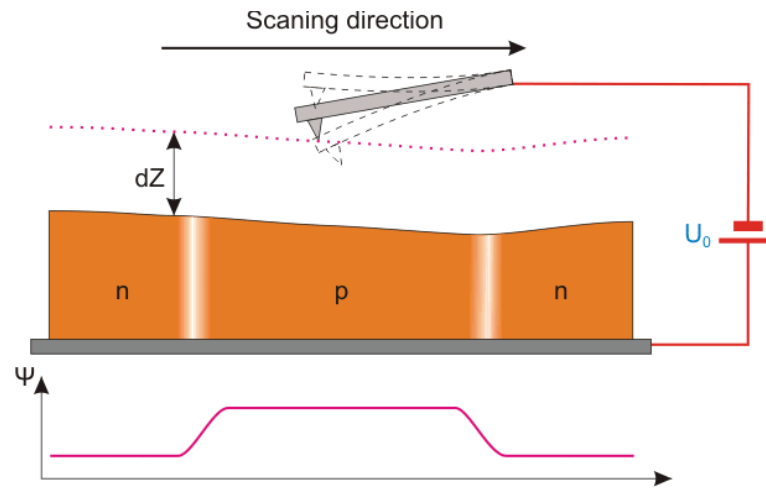
Fig. 9-16. Second pass
Ψ – phase shift

During the second pass the distance between the scanned surface and the probe is kept constant. This distance should be big enough to eliminate the surface topography influence. In this case, the probe is only affected by long-range forces whose most prominent yield is due to electric properties of the sample. On the other hand the value of dZ should not be too great as that would reduce the measured signal and impair the lateral resolution.

9.3.2. Preparation for Measurements

To operate in the Electric Force Microscopy Mode a sample should be glued to the substrate with the conductive glue or mounted on the substrate with a spring contact. And it should be used conductive probes for semicontact modes.

To provide an example of operations in the EFM Mode, we will use a GaAs sample.

Initial state

The following steps are assumed to be done:
- Launching the control program;
- Turning the controller SPM on;
- Installing the probe;
- Adjusting the cantilever deflection detection system;
- Mounting the sample;
- Moving the measuring head to its working position;
- Initial approach;
- Selecting the investigation area.

For details on these operations see Ch. 6 Preparing for Measurements on p. 19.

Preparations of the instrument for operation and measurements in the EFM Mode are generally similar to those performed in the Semicontact Mode of operations, which are described in detail in (see i. 8.1 on page 80). A sequence of basic procedures, along with a detailed description of instrument settings and specifics of EFM measurements, is provided below.

Procedural Sequence

1. Switching the Instrument for Operating in the Semicontact Modes.

2. Taking Resonance Curve and Adjusting Parameters of Generator Output.

3. Approaching the Sample to the Probe.

4. Adjusting Working Level of the Feedback Gain.

5. Adjusting Scanning Parameters.

6. Scanning.

Perform preliminary scanning of the sample in the Semicontact Mode (see i. 8.1 on page 80) and select an operating area for EFM measurements.

9.3.3.   Scanning

Select the mode **Semicontact 2-pass EFM** (Fig. 9-17) mode in the **Mode** list of the Control panel of the **ScanForm** window.
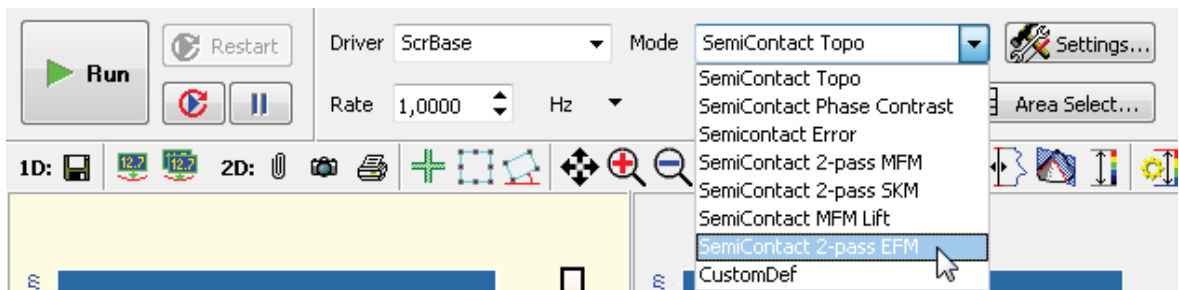

Fig. 9-17. Selection of Electric Force Microscopy

Define the voltage to apply to the probe in the **BV** field in the top bar of the Main Program window.
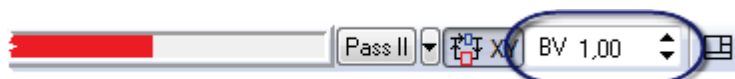

Fig. 9-18

Select the scan area and define the scanning rate.

Starting measurements

Scanning of the sample surface is started after the completion of all necessary preparation procedures: the sample is approached to the probe, setting of the operation point is done, and all scanning parameters have been set.

To trigger scanning, click the button **Run** available on the control panel (Fig. 9-19).
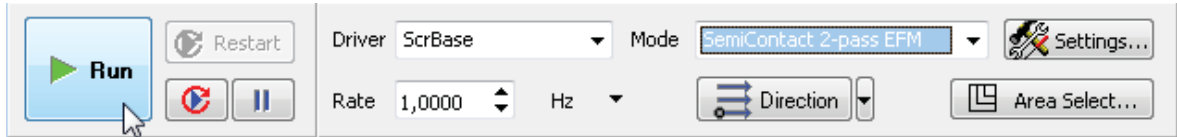
Fig. 9-19

The following actions take place during scanning:

- Line-by-line scanning of the sample surface starts;

- Two images appear in the 2D Scanning Data View panel. One of the images is an image of surface topography (signal **Height**), while the other one is the distribution of the Z-component of the electric field gradient on the sample surface (signal **Phase**) (Fig. 9-20).

Fig. 9-20. Images of surface topography (left) and spatial distribution of the Z-component of the electric potential gradient on the surface of a GaAs (right)

How to improve the image

When selecting the EFM mode, all parameters required for the second pass are set automatically.

To achieve better quality of the images obtained, the values of the following parameters can be varied during scanning:

DZ
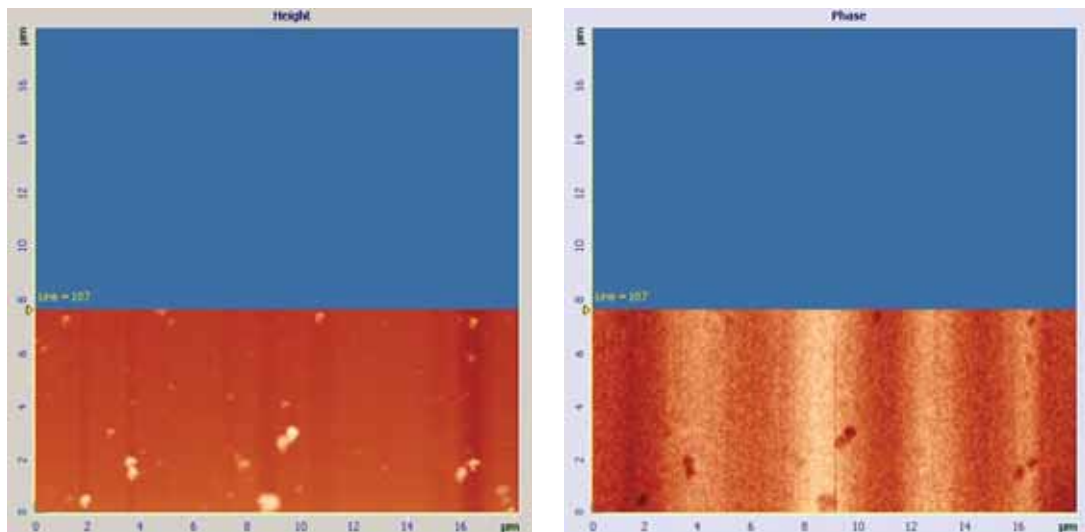: separation between the probe and the surface during scanning in the second pass. The greater DZ, the smaller influence of surface topography on measurement results during the second pass. However sensitivity of this technique decreases with higher separation.

Gen1Amp
: magnitude of the generator output signal. Decreasing the generator output value and, respectively the probe oscillation amplitude, results in better resolution for this technique. However, on the other hand, it reduces the signal-to-noise ratio. To alter parameter Amp during scanning, open the Block Scheme window. Set the desirable value of amplitude in the text box Amp for MOD generator.

hvValueBV
: alteration of the bias voltage sign and value can influence the behavior of surface potentials in the second pass.

To correct the parameters of the second pass, perform the following steps:

1. Open the Additional Scanning Parameters panel by clicking the Params button in the ScanForm window (Fig. 9-21).

Fig. 9-21

2. Observe quality of the acquired image and vary the sample-probe distance. This needs modification of the DZ parameter in the Mode Params group (Fig. 9-22).

Fig. 9-22. Additional Parameters panel

3. Observe quality of the acquired image and vary the generator output amplitude. This needs modification of the **Gen1Amp** parameter in the **Pass II** group (Fig. 9-22).
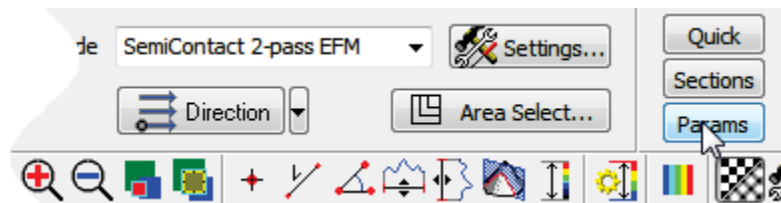
4. Observe quality of the acquired image and vary the bias voltage. This needs modification of the **hvValueBV** parameter in the **Pass II** group (Fig. 9-22).

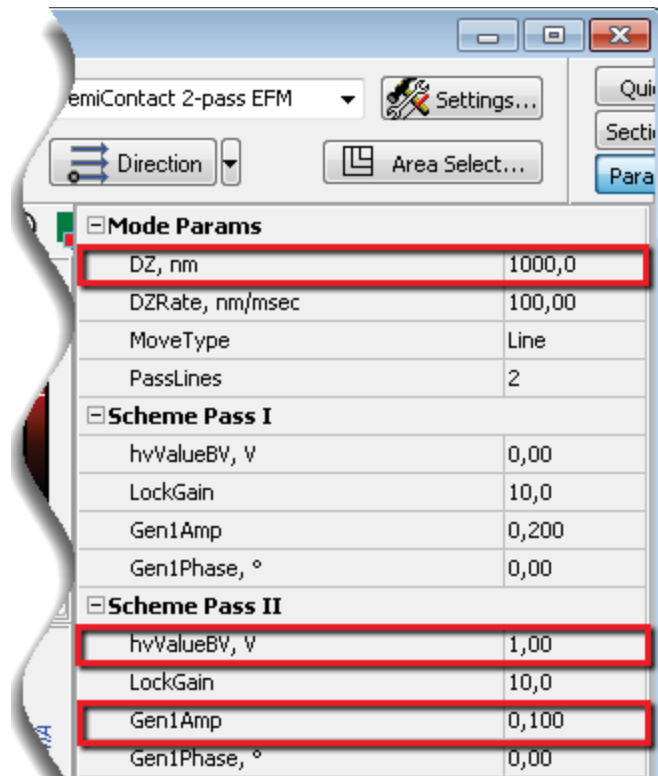All other settings and parameters alteration procedures during scanning are described in detail in the Semicontact Mode of operations (i. "Scanning" on page 97).

10. Scanning Tunneling Microscopy

The Scanning Tunneling Microscopy is capable to study surfaces properties of conductive materials with resolution down to atomic scale. The tunnel current recorded during scanning is small enough (0.5 pA ÷ 50 nA) to enable investigating samples with low conductance, biological objects in particular.

Special modes facilitate acquisition of spatial distributions of various electrical characteristics, such as work function, local density of electron states, etc.

Interpretation of the collected STM data is rather involved because details of the STM image are determined not only by the surface topography, but also by its local electrical characteristics.

10.1. Constant Current Mode

Initial state

The following steps are assumed to be done (see Ch. 6 "Preparing for Measurements" on p. 19):

- Launching the control program;
- Turning the controller SPM on;
- Mounting the sample;
- Moving the STM head to its working position;
- The sample approached to the tip to distance of 1÷2 mm;
- Selecting the investigation area.

Procedural Sequence

Measurement in the Constant Current mode is arranged in the following procedural sequence:

1. Switching the Instrument for Operating in the Tunneling Microscopy Modes (see page 125).
2. Approaching the Sample to the Probe (see page 125).
3. Adjusting Working Level of the Feedback Gain (see page 127).
4. Adjusting Scanning Parameters (see page 128).
5. Scanning (see page 135).
6. Saving Measurement Data (see page 138).
7. Operation Completing (see page 138).

These procedures are explained below.

10.1.1. Switching the Instrument for Operating in the Tunneling Microscopy Modes

Switch the instrument for operating in the Tunneling Microscopy modes by selecting **STM** in the controller configuration list (see Fig. 10-1) at the top bar of the Main Program window.
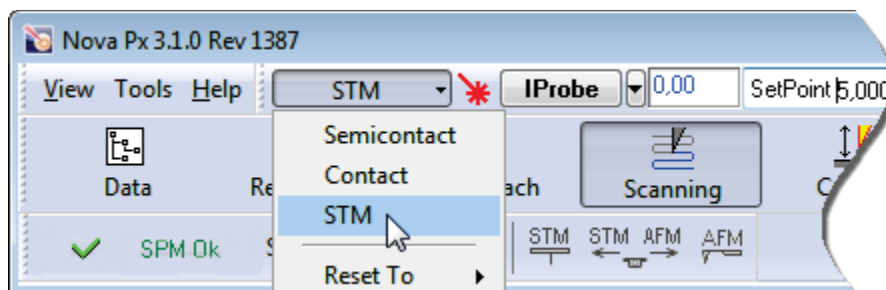

Fig. 10-1. Switch the instrument for operating in the STM

Once the configuration **STM** is set, all switching sequences required for the instrument to operate in contact modes proceed automatically.

The following adjustments of the system will take place:

- Input of the feedback loop – **IProbe**;
- The probe is grounded;
- Gain of the feedback loop **Gain** – 0.3.

10.1.2. Approaching the Sample to the Probe

The feedback uses one of two signals, **IProbe** or **IprLog**, as input. The **IProbe** signal serves for feedback at the stage of approaching while the **IprLog** signal is involved during scanning.

To approach the sample to the probe, perform the following steps:

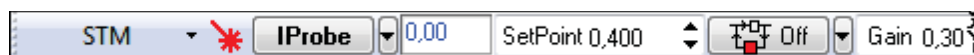1. Select **IProbe** as the feedback input (see Fig. 10-2).

Fig. 10-2. Selecting the feedback input

2. Click the ![Off] button in the top bar to close the feedback loop (the button will change its appearance to ![On]). This starts extension of the Z scanner to its maximum. Progress of the extension is displayed in the Scanner extension indicator (see Fig. 10-3) by degree of the colored filling.
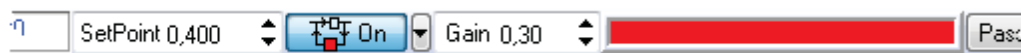

Fig. 10-3. Scanner is maximally extended

3. Enter the probe-sample bias voltage in the **BV** input field to be in the range 0.1÷0.2 V.

4. Define the **SetPoint** parameter in the input field at the top bar. We recommend to enter value in the range 0.4 nA as the initial level of the parameter.

5. Define the initial feedback gain in the **Gain** input field to be in the range of 1÷5.

6. Open the **Approach** window with the [Approach] button of the Main Operations panel.

7. Start the approaching procedure with the [▶ Landing] button in the Control panel of the **Approach** window (see Fig. 10-4).
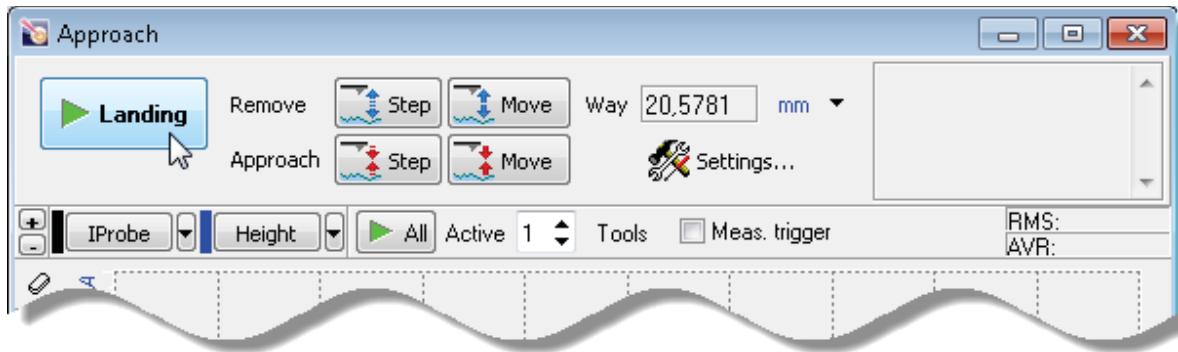

Fig. 10-4. Control panel of the **Approach** window

During the approach procedure, observe variations of the **IProbe** signal in the software oscilloscope and state of the scanner extension indicator. Wait until the procedure completes.

Providing the approach parameters are set correctly, the approach procedure completes after a while and the system goes to following state (Fig. 10-5):

- The **IProbe** signal increases to the level of parameter **Set Point**, the feedback loop maintains the Z-scanner in the position where the value of **IProbe** is equal to **Set Point**. Note that this position of the scanner is approximately equal to half of the scanner extension range;

- The message "**Landing Completed**" appears at the right top of the Approach window.

- The stepper motor is disabled;

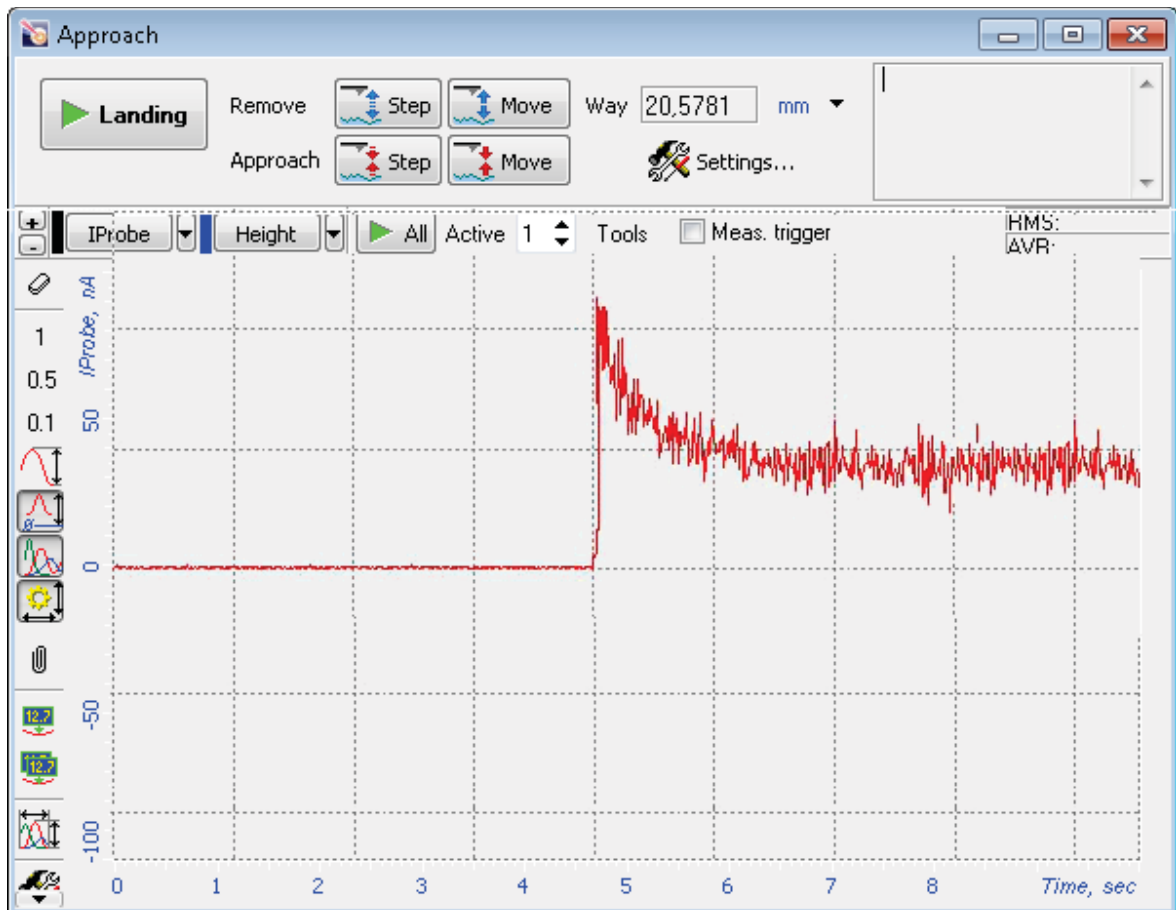- Length of the indicator bar decreases down to some intermediate value.

Fig. 10-5. Approach completed

10.1.3. Adjusting Working Level of the Feedback Gain

The greater is the level of the feedback gain (**Gain** parameter), the greater is data processing speed in the feedback loop. Nevertheless, at some high level of the feedback gain (let us call it threshold value), the mode of operation of the feedback loop becomes unstable and noise generation occurs. Under this condition, the **IProbe** signal is supplied with a significant alternating component.

For stable operation, it is recommended to define the feedback gain to less 0.5÷0.7 of the threshold value where the noise generation occurs. The feedback gain is adjusted in the **Gain** input field.

To adjust the working level of the feedback gain, perform the following steps:

1. Double-click the **Gain** input field in the Main Parameters panel. The adjusting slider of the feedback gain will appear.

2. Gradually increase the **Gain** level and watch the **IProbe** level in the software oscilloscope.

3. Determine the value of the **Gain** parameter at which noise generation occurs. The beginning of the generation is identified by appearance of a noticeable alternating component in the **IProbe** signal.

4. Decrease the value of the **Gain** parameter to 0.5÷0.7 of the threshold. This value will be used in further operation.

10.1.4. Adjusting Scanning Parameters

Adjusting the feedback input

1. Click the [⚙ On] button in the top bar to open the feedback loop (the button will change its appearance to [⚙ Off]).

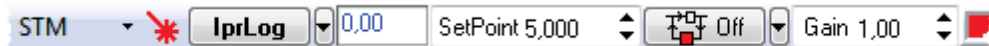2. Select the **IprLog** signal as the feedback input (see Fig. 10-6).

Fig. 10-6. Selecting the feedback input

3. Click the [⚙ Off] button in the top bar to close the feedback loop (the button will change its appearance to [⚙ On]).

4. Observe the **IprLog** signal with the software oscilloscope and adjust the **SetPoint** parameter so that the **IprLog** level is 0.1 nA. Usually, with this level of the **IprLog** current the **SetPoint** parameter is about 20.

5. Increase the **Gain** value and watch the **IprLog** value in the software oscilloscope.

6. Determine the value of the **Gain** parameter at which noise generation occurs. The beginning of the generation is identified by appearance of a noticeable alternating component in the **IprLog** signal.

7. Decrease the value of the **Gain** parameter to 0.5÷0.7 of the threshold. This value will be used in further operation.

Selecting STM mode

Open the **ScanForm** window by clicking the [Scanning] button in the Main Operations panel.

Fig. 10-7. **ScanForm** window Control panel

Select the **Constant Current** mode in the **Mode** list of the Control panel of the **ScanForm** window (see Fig. 10-8). The **Height** signal will be selected for detection.
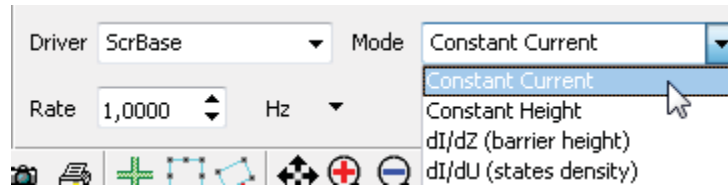
Fig. 10-8. Selecting the Scanning Tunneling Microscopy Mode
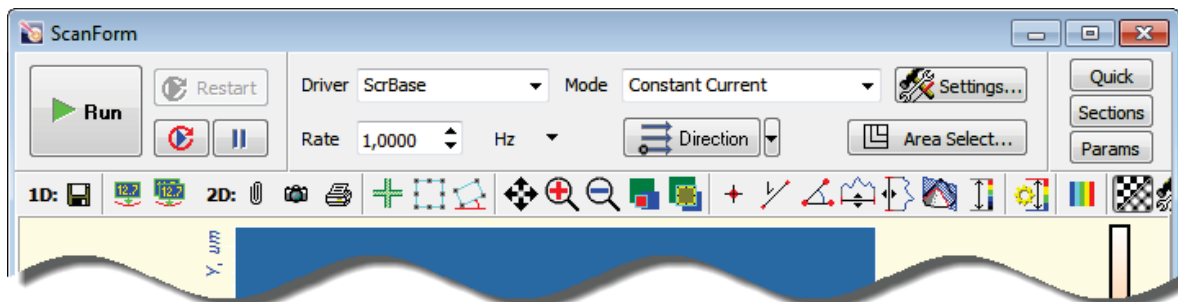
Selecting the scan area

To choose preliminary dimensions of the scan area, regard for the following recommendations:

- If height difference in the surface profile is known to be within the scanner range, the maximum scan field is recommended;

- If height difference in the surface profile is unknown, a small sized scan area (about 10÷15 μm) should be preliminarily explored for finding proper levels of controlling parameters, including speed of scanning, **SetPoint**, **Gain**. Then the scan area can be enlarged.

Two options are provided for selecting the scan area:

- by defining the scan area parameters immediately (see p. 130) – number of scan points, rotation of the scan area in XY plane, and size of the scan area can be defined explicitly by the user;

- by defining the scan area in the 2D Data Viewer of the Scanning window (see p. 131) – only size and position of the scan area can be defined.

  These options are explained below in details.

Defining Scan Area Parameters

Size and position of the scan area can be defined in the **Scan Area Selection** window (Fig. 10‑9) accessible by clicking the [Area Select...] button.
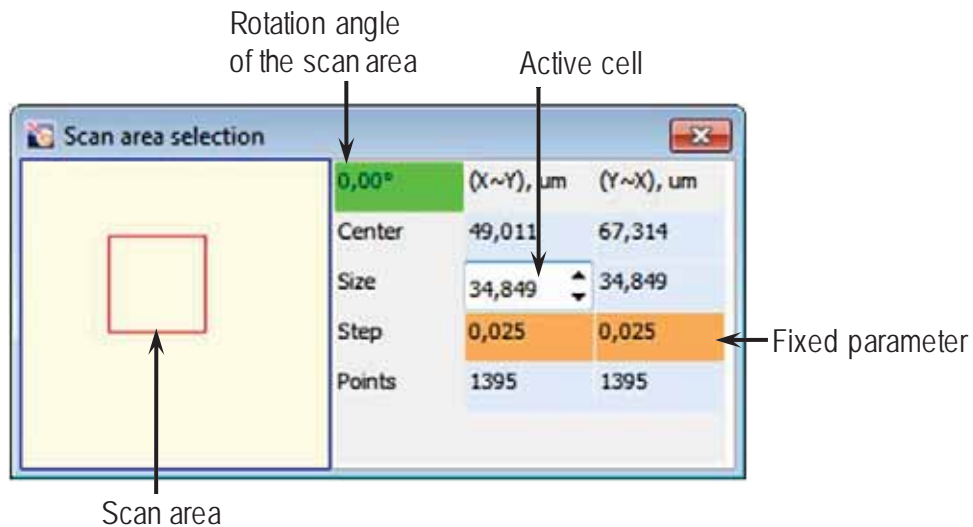

Fig. 10‑9. Scan Area Selection window

The red rectangle in the left part of the window displays the scan area currently selected with respect to the maximum scan area.

The following rules of modification of the scan area (the specific one is selected by clicking on titles of the 2nd and 3rd columns) are available:

- **X, Y** – X and Y dimensions of the scan rectangle are adjusted independently;

- **X~Y, Y~X** – when a particular dimension is changed, its counterpart will vary proportionally;

- **X=Y, Y=X** – the dimensions will be kept equal (square scan area).

  Position of the scan area can be redefined either with the mouse or with entering coordinates of the area center (in μm) in the **Center** fields. The origin of the coordinate system is at the left bottom corner of the maximum scan area.

Sizes of the scan area are defined by three parameters:

Size – size of the scan area in a particular direction (in μm). By default, dimensions of the scan area are 10×10 μm;

Step – distance between neighboring scan points in a particular direction (in μm);

Points – number of scan points in a particular direction. By default, number of points for every direction is 512.

To enable modifications in a particular cell, click on it with the mouse and the cell will become active. Value in the active cell can be edited in one of the following ways:

- with arrow keys <↑>, <↓>;
- with scroll keys ↕;
- with the slider (appears after double-click in the active cell).

The parameters **Size, Step, and Points** are interrelated through the formula:

$$Size = Step \times Points. \tag{1}$$

When a particular parameter varies, one of the two remaining follows these changes while the second remaining (fixed) keeps at the same level. The field of the fixed parameter is displayed on orange or purple background.

Purple background. Used for displaying the line being previously active.

Orange background. Appears after triple click on a parameter line. For changing the parameter to be fixed, click on the field of its name.

Rotation angle of the scan area in XY plane is defined in the green cell at the left top of the table.

NOTE. Scanning over a rotated scan area is not recommended if the XY displacement sensors are disabled.

Selecting Scan Area in Scanning Window

1. Enable the function of selecting the scan area by clicking the ▦ button in the Toolbar.

   NOTE. If a rotated scan area is required, the function of selecting the scan area should be called by clicking the ◹ button (the ▦ button will be hidden in this case).

2. Change size and position of the scan area with the mouse.

3. Click the ✚ button. Verify that the probe tip can contact the sample surface everywhere inside the selected scan area without penetration. To this purpose, press and hold the left mouse button and move the mouse pointer inside the scan area. Motion of the mouse pointer will be followed by displacement of the probe tip over the sample surface. Monitor extension of the piezoscanner in the indicator in the top bar of the Main Program window.

Fig. 10‑10. 2D Data Viewer
Arrow indicates the boundary of the scan area

Selecting the scan direction

The scan direction is selected in the [Direction] drop-down list of the Control panel of the Scanning window.

Adjusting the scanning speed

Optimal value of the scanning speed depends on sample surface properties, scan area dimensions and external conditions. A smooth surface can be scanned at higher speed than that with uneven topography and large height difference.

To define the **Rate** parameter, perform the following styeps:

1. In the Control panel of the **ScanForm** window, select unit of scan rate measurement (Fig. 10-11). For example, the **Hz** item is associated with the line scan frequency in Hz.

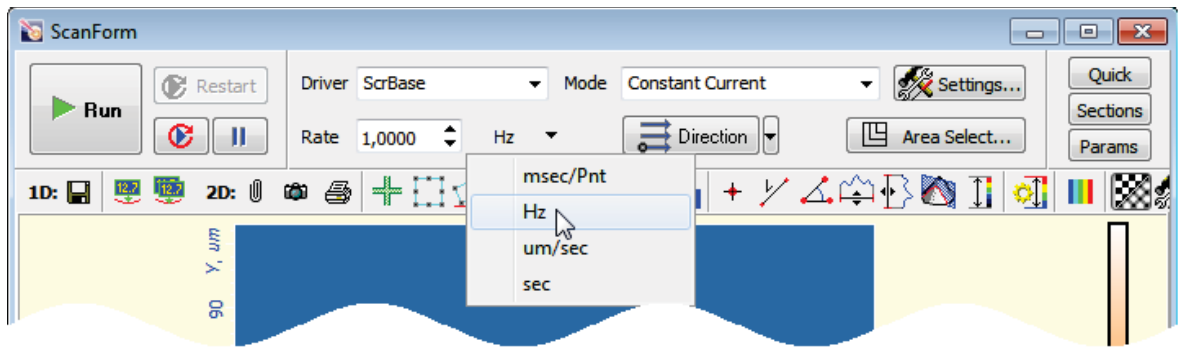Fig. 10-11. Control panel of Scanning window

2. In the **Rate** field, define level of the scan rate. For testing scans, values not exceeding 0.5 Hz are recommended.

Switching the detection signals

Every measurement technique is supplied with a default set of recorded signals. This set can be modified if required. The Control program uses the latest set of recorded signals.

For every recorded signal a separate 2D Data Viewer is reserved in the **ScanForm** window. The instrument is capable to register up to eight signals in parallel.

The set of recorded signals can be modified in the **ScanSettings** window (accessible through clicking the ![Settings...] button in the Control panel of the **ScanForm** window).

Fig. 10‑12. **ScanSettings** window

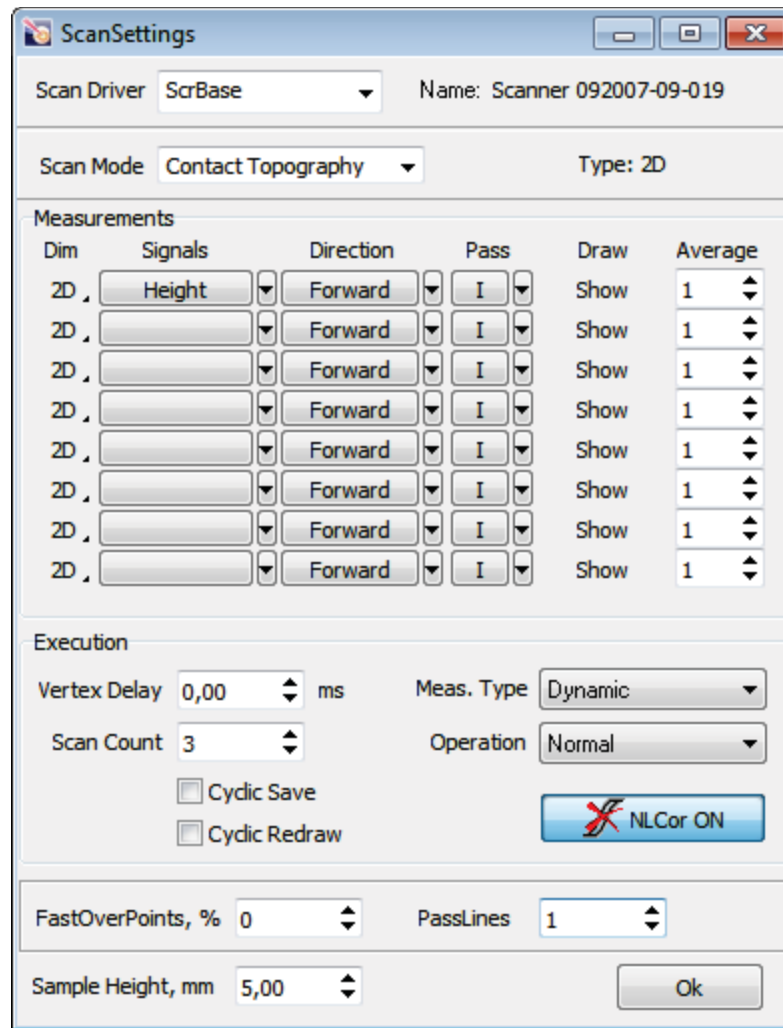The **Scan Mode** field displays the SPM technique the set of recorded signals of which is viewed or edited at the moment.

The list of recorded signals is provided in the **Signals** column. Although data are measured both in forward (from beginning of the line to its end) and in backward (reverse) passes of the probe, the 2D image is generated only for one of those passes. That pass is selected in the **Direction** column as follows: **Forward** – the signal will be recorded for the forward pass, **Backward** – the signal will be recorded for the backward pass.

If displacement sensors are used, the **SensHeight** signal is recommended for recording because it is most sensitive to the surface landscape:

Height is a signal derived from voltage applied to the Z scanner.

SensHeight is a calibrated signal from the Z displacement sensor.

10.1.5. Scanning

Hereafter, the scanning process will be explained with the example of highly oriented pyrolytic graphite (HOPG).

<u>Starting measurements</u>

Scanning of the sample surface should be preceded by necessary preparation procedures (the sample has approached to the probe, the operation point is adjusted, and all scanning parameters have been set).

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window (see Fig. 10-13).
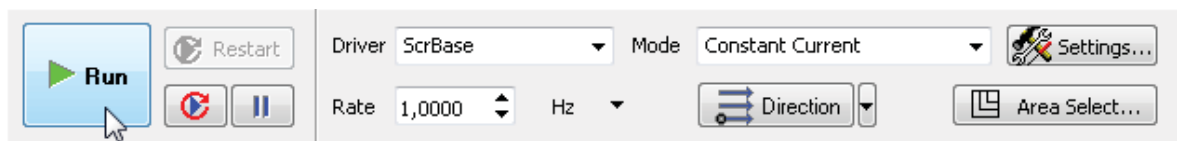

Fig. 10-13. Control panel of the **ScanForm** window

This results in the following activity of the system:

- Line-by-line scanning of the sample surface starts and the corresponding image of the scan area appears in the 2D Viewer of the scan data (see Fig. 10-14). In the example under consideration, this is an image of the graphite;
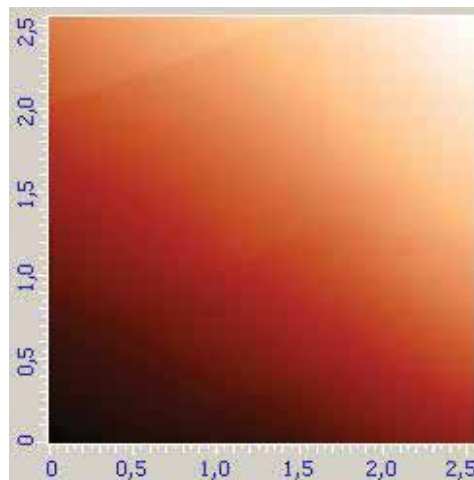

Fig. 10-14. Surface topography image

- If the **S** button in the left bottom of the 2D Viewer of the scan data is pressed, the 1D Viewer will display the detection signal in real time (see Fig. 10-15);
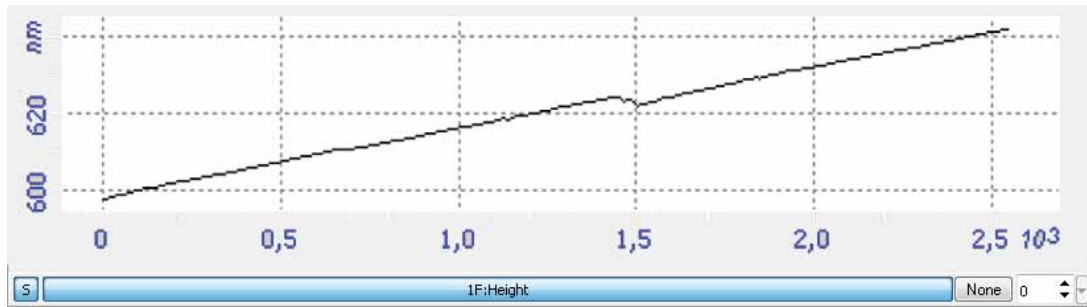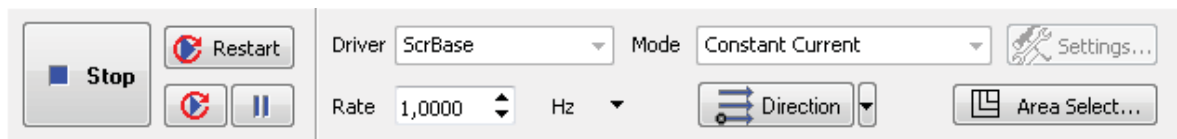
Fig. 10-15. **Height** signal

● The **Restart** button becomes enabled and caption of the **Run** button changes to **Stop** (see Fig. 10-16).

Fig. 10-16. Control panel of the **ScanForm** window in the course of scanning

Should the scanning procedure be interrupted, click the **Stop** button or use the <Esc> key.

Tilt subtraction

Data in the example above (see Fig. 10-14, Fig. 10-15) reveal that the sample is tilted to some degree in X direction.

Contribution of the tilt to the detected landscape can be eliminated immediately in the scan process with proper selection of the **Subtract** control at the right bottom of the **Scanning** window. By default, this button is in the **None** state (see Fig. 10-15).

To activate run-time subtraction, click this button and select the **Plane** option in the corresponding list. This entails subtraction of the plane tilt, and the original image, illustrated in Fig. 10-14, is transformed into the image shown in Fig. 10-17.
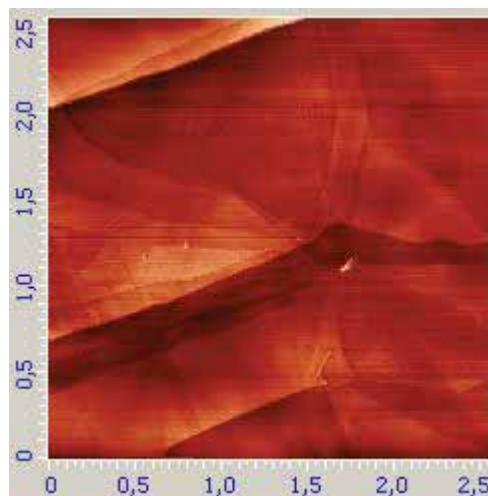

Fig. 10-17. Surface topography image

With the correction defined, the 1D Viewer of the scan data will display the detection signal corrected for the tilt contribution. The resulting signal is shown in Fig. 10-18 (compare with the signal in Fig. 10-15).
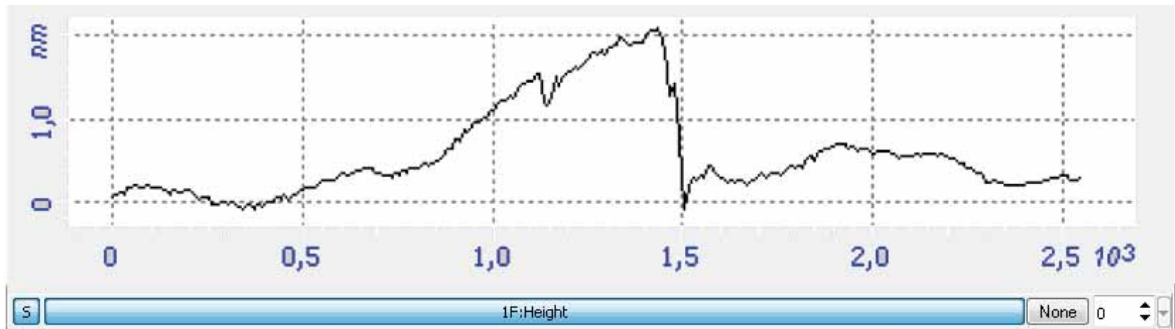

Fig. 10-18. **Height** signal

*NOTE. Collected frames miss information on corrections of the scan data performed with the **Subtract** option.*

Tuning parameters in the scan process

Quality of the acquired surface image essentially depends on such parameters as scanning frequency, **Rate**, the operating point, **Set Point**, the feedback gain, **Gain**, and the constant bias voltage, **BV**. Either of these parameters can be changed instantly in the course of scanning with visual inspection how modifications impact on the image being collected.

Along with optimization of the scanning parameters, you should account for the tip's sharpness and for secure fixing of the probe in the fastener as the main image quality factors.

*NOTE. Scanning is recommended to restart (using the **Restart** button) after any of scanning parameters is changed.*

Some recommendations on optimization of scanning parameters

The choice of the optimal value of scanning speed depends on properties of the sample, on dimensions of the scan area, and on external conditions.
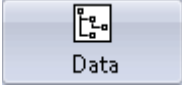
Smooth surfaces can be scanned at a speed higher than those with sharp features and steep slopes.

It is recommended to start scanning at a lower scanning speed, increasing it gradually until distortions affect the landscape profile.

Scanning speed should be reduced when surface features in the scan direction are not detected.

10.1.6. Saving Measurement Data

To save measurement data on the hard drive, perform the following steps:

1. Open the **Image Analysis** window by clicking the [Data] button in the Main Operations panel.

2. In the Main menu of the **Image Analysis** window, select the **File → Save All Frames** command.

3. In the standard dialog, select a folder for saving the collected data.

4. Type in a filename and save it with the extension ***.mdt**.

10.1.7. Operation Completing

1. Open the feedback loop by clicking the [On] button in the Main Parameters panel (the button will change its appearance to [Off]).

2. Open the **Approach** window by clicking the [Approach] button. To prevent damage of the probe tip by the sample surface when removing the measuring head, retract the sample from the probe by clicking the [Move] button for several times (Fig. 10-19).
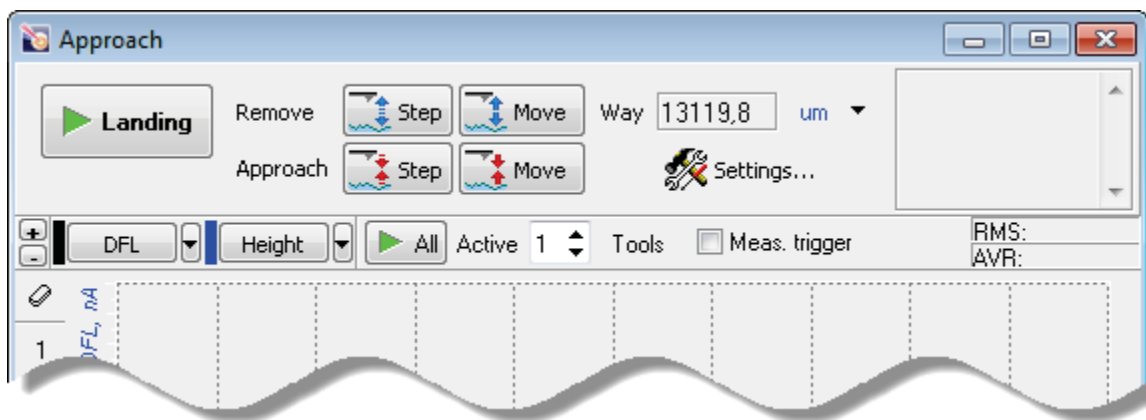

Fig. 10-19. **Approach** window

3. Move the heads to their inoperative positions with the [STM AFM] button

4. Close the Nova Px Control program.

5. Turn off the SPM controller.

6. Turn off the vibration isolation system.

7. Turn off the computer.

10.2. Constant Height Mode. Atomic Resolution on Graphite

In measurements with the Constant height technique, the probe tip moves only in plane and so variation the tip-sample current reflects the surface landscape.

> **ATTENTION! When using the Constant Height Mode ("the flying apex") it is possible to scan only very smooth surfaces without any relief, since the feedback during scanning is practically cut-out. Therefore the tip can "hit" the sample, when the roughness is not small enough.**

10.2.1. Preparation for Measurements

__Initial state__

- Launching the control program;
- Turning the controller SPM on;
- Mounting the sample;
- Moving the STM measuring head to its working position;
- Initial approach;

An example of use of Constant Height Mode to acquire the topography of a HOPG sample will be described hereafter.

Perform preliminary scanning of the sample in the Constant Current Mode.

10.2.2. Adjusting Scanning Parameters

1. Open the **ScanForm** window (the [Scanning] button on the Main operations panel).

2. Select the **Constant Height** mode in a drop-down **Mode** menu (Fig. 10-20).

Driver ScrBase ▼ Mode Constant Current ▼
Constant Current
Constant Height
Rate 1,0000 ▲▼ Hz ▼ dI/dZ (barrier height)
dI/dU (states density)

Fig. 10-20

The device will be automatically configured to implement the selected mode:

- The feedback gain factor will be decreased to 0.01;
- The **IProbe** signal will be selected as the signal displayed during scanning.

3. Disable the feedback on XY position of the scanner (the ⊞ XY button is released).
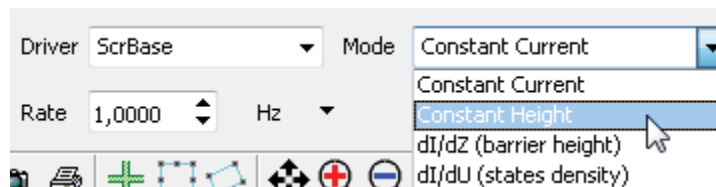
4. Verify that the feedback is disabled (⊞ Off).

5. Switch the scanner to the «low voltage» mode (see Fig. 10-21):

a) Open the **Calibrations** dialog by selecting the **Tools → Calibrations** menu command.

b) In the **Range** list, select **Low**.

c) Click the **Apply** button.
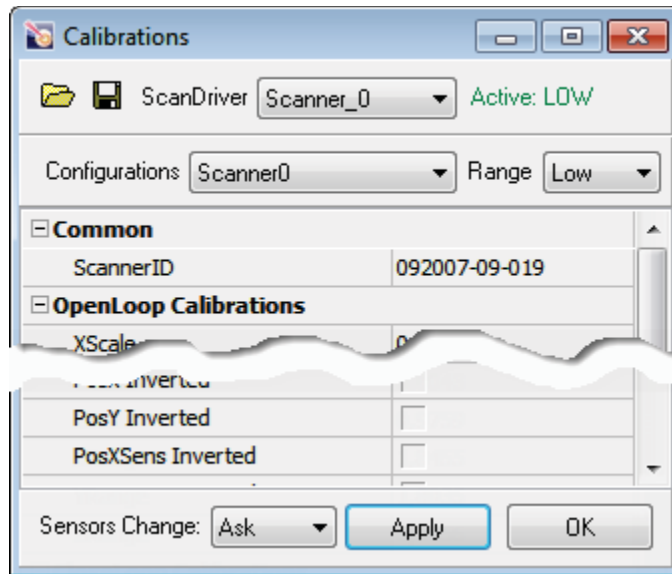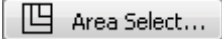
Fig. 10-21. Scanner switched to the «low voltage» mode

6. Enable the feedback (⊞ On).

7. In the **Scan Area selection** window (opens by clicking the [Area Select...] button), define the following scanning parameters:

- size of the scan area (**Size**) – no more than several nanometers;

- number of points (**Points**) – 128.

8. Select the scan area. To this purpose, perform the following steps:

a) Enable the scan area definition tool by clicking the ⊡ button in the Toolbar of the 2D Data Viewer.

b) Select the scan area by moving the selection frame with the mouse in the latest scan (Fig. 10-22). It is recommended that the scan area is located close to the center of the scan and the sample landscape is smooth inside this area.

NOTE. If the scan area selection frame is not viewed in the scan (due to its locating outside the scan area), click the ▣ button in the Toolbar to display the maximum scan area.
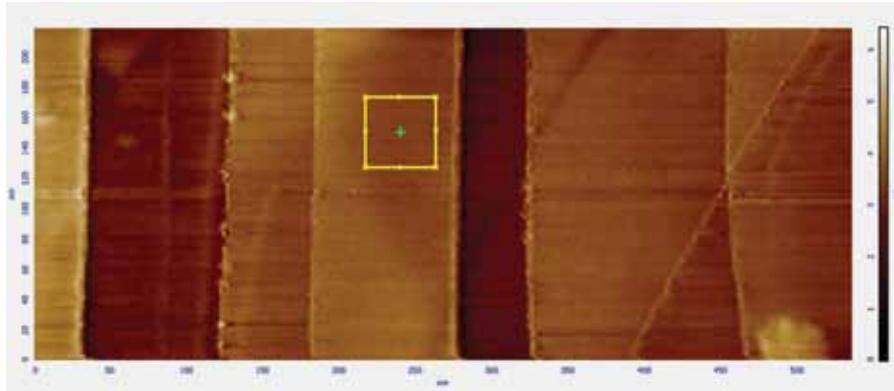
Fig. 10-22

9. Set the scan rate to its maximum in the **Rate** field.

10. For subtracting a geometric profile, select **1 Order** (subtraction of a curve of the first order).

10.3. Scanning

To start scanning, click the **Run** button in the Control panel of the **ScanForm** window (Fig. 10-23)
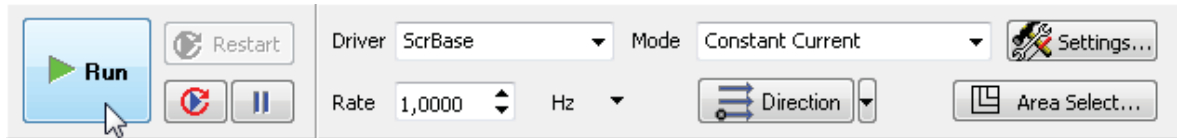
Fig. 10-23. Control panel of the **ScanForm** window

Fig. 10-24 shows an example of a HOPG surface image with atomic resolution, obtained using the STM.
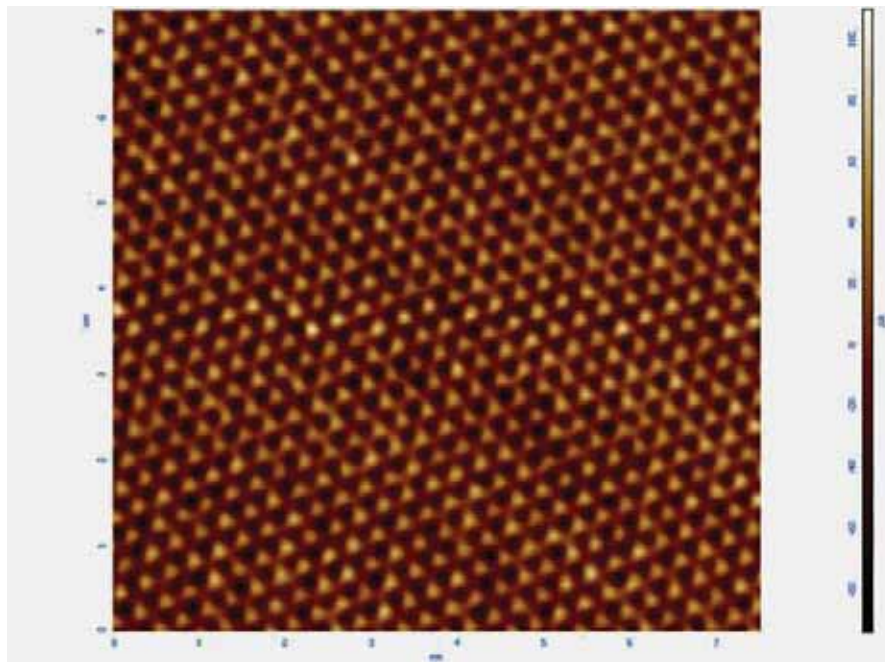
Fig. 10-24. HOPG surface image

If necessary, you can try the following recommendations:

- Adjust the **SetPoint** parameter;
- Alter the Points, Step, Rate;
- Alter the level of the feedback gain (**Gain** parameter).

11. AFM Spectroscopies

This section discusses the spectroscopy curves measurement procedures.

Prior to conducting spectroscopy measurements it is recommended to perform a preliminary scan.

The measurement procedures for various types of spectroscopy are similar. As an example, this section considers one of the techniques – Force Spectroscopy **DFL(Z)**. In the sections concerning other spectroscopy techniques a general description of the procedure is given together with comments on the differences in completing certain operations.

11.1. Force-distance Spectroscopy

In Force-distance spectroscopy cantilever deflection is measured as a function of the scanner extension, i.e. the **DFL(Z)** curve is obtained.

After the probe has touched the surface any variation in the voltage applied to the z-contact of the scanner (the **Z** signal) results in a proportional change in the **DFL** signal. Using the **DFL(Z)** function and assuming that the cantilever stiffness is known one can calculate the forces acting on the probe in the measurement point including adhesive force.

Contact probes shall be used for force-distance spectroscopy. However, using too "soft" cantilever at high humidity may cause "sticking".

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the contact mode (see i. 7 "Contact AFM" on p. 41).

<u>**Basic operations:**</u>

The general procedure on force-distance spectroscopy is represented by the following sequence of basic operations:

1. Selecting the Function to be Measured, i.e. selecting a function and its argument (see i. 11.1.1 on p. 144);

2. Selecting Points for Spectroscopy (see i. 11.1.2 on p. 146);

3. Starting the Measurements (see i. 11.1.3 on p. 148);

4. Viewing Spectroscopy Data and processing them (see i. 11.1.4 on p. 148);

5. Calculating Adhesion Force (see i. 11.1.5 on p. 151);

6. Saving Measurement Data (see i. 11.1.6 on p. 153);

A detailed description of these basic operations is given below.

11.1.1. Selecting the Function to be Measured

To select the function to be measured, perform the following steps:

1. Switch the instrument to operate in contact modes by selecting **Contact** in the drop-down list of controller configuration (Fig. 11-1).
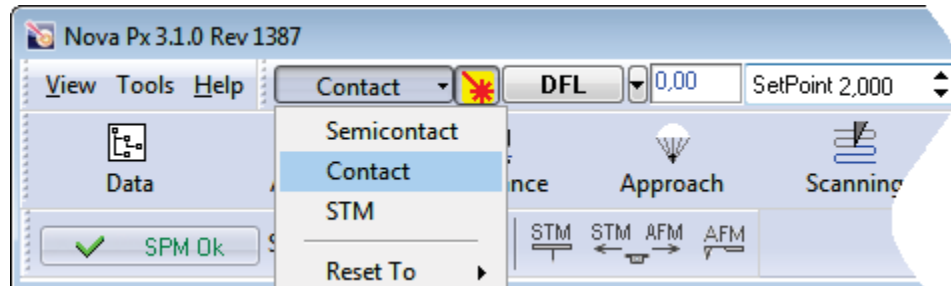
Fig. 11-1. Selecting the controller configuration

2. Open the **Spectroscopy** window by clicking the [Curves] button (Fig. 11-2).
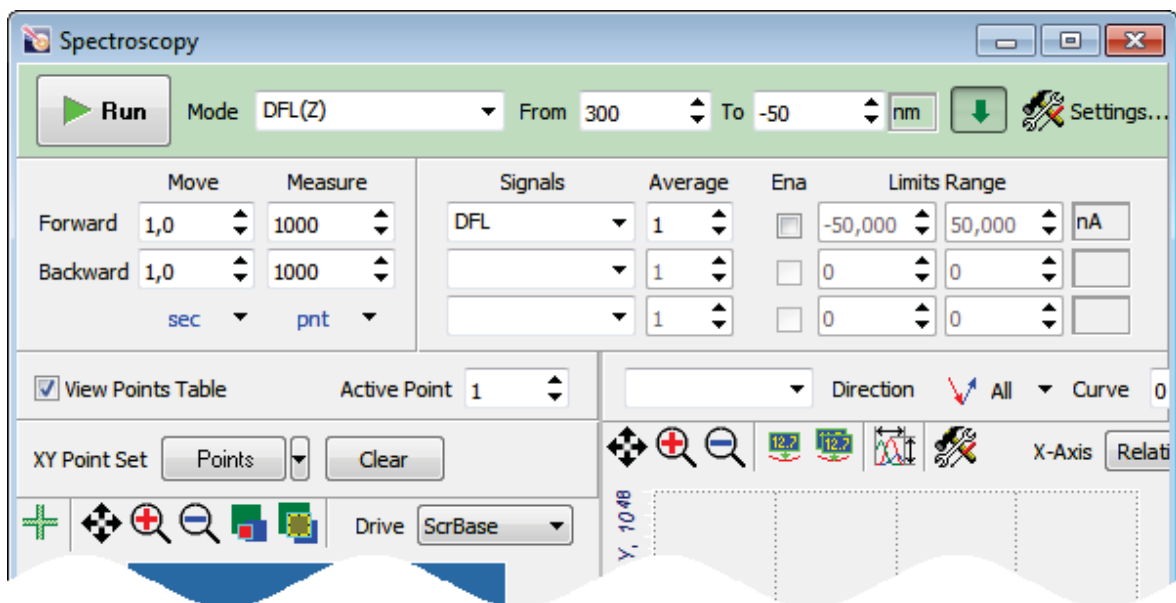
Fig. 11-2. **Spectroscopy** window

3. In the **Mode** drop-down list, select **DFL(Z)** (see Fig. 11-2). The **Signals** list of the Spectroscopy parameters panel is appended with signals standard for the selected measurement mode (for the discussed mode, the standard signal is **DFL**).

   *NOTE. The Spectroscopy parameters panel is displayed in the **Spectroscopy** window if the [↓] button is pressed in (see Fig. 11-2).*

   If desired, redefine the set of recorded signals.

4. In the fields **From** and **To**, define limits of the argument of the measured relation (for the particular case, range of sample displacement along Z).

The limits should be given in relative units with the coordinate origin located at that **Height** where the tip presses the sample with the predefined force, i.e. where **DFL** = **Set Point**.

From provides the total decrease of the scanner length when the tip is retracted from the sample. This value should be positive.

To provides the total increase of the scanner length when the tip is in contact with the sample. This value should be negative.

*NOTE. For exploratory measurements, recommended values for **From** and **To** are in the ranges from 200 to 500 nm and from –100 to –10 nm, respectively.*

In the course of measurement, the Height value will behave as shown in Fig. 11-3:

1) Part 1 – the scanner brings the sample along the z axis to the point at which the measurements begin. The **Height** level increases stepwise by the **From** value.

2) Part 2 – forward scan measurements. The **Height** value linearly decreases to its original level and then to the step defined by **To** value.

3) Part 3 – backward scan measurements. The **Height** value linearly increases.

4) Part 4 – the sample returns to the original position. The **Height** value returns to its original level.
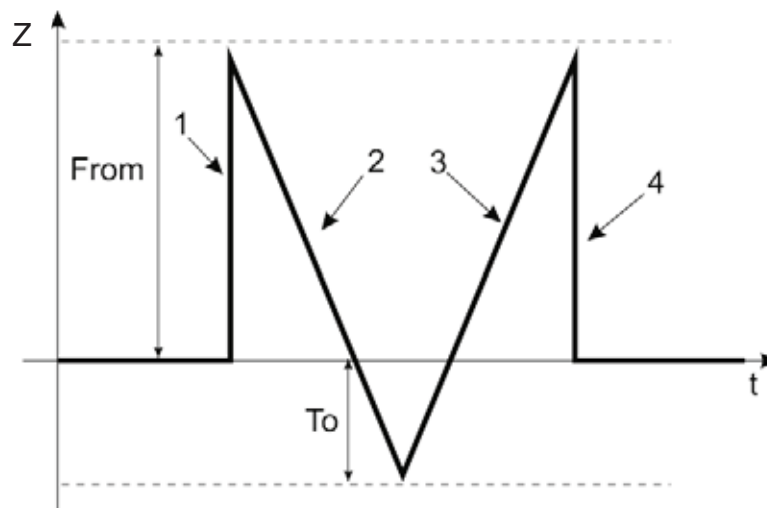
Fig. 11-3. **Height** behavior during measurements at a single point

5. If the **Ena** option of the signal in the Recorded relations parameters panel is unselected, measurements will be performed throughout the whole range of the argument.

If necessary, define limiting levels of the recorded signal. To this purpose, for the desired signal select the **Ena** option and in the range **Limits Range** fields define the minimum and the maximum of the signal.

*NOTE. For the force spectroscopy, proper upper limit of **DFL** is important.*

6. Define measurement parameters at every point of spectroscopy:

- in the **Move field** – total time of acquisition (in sec) or rate of change of Z under measurements (in um/sec),

- in the **Measure field** – number of points in the range of measurements,

- in the **Average field** – number of measurements at every point. The collected data will result from averaging over this number of measurements.

11.1.2. Selecting Points for Spectroscopy

Taking a scan of the sample surface is recommended before collecting spectroscopy. This scan facilitates selection of points for spectroscopy.

The Spectroscopy window will display the latest scan. If desired, another frame of the current session can be selected through clicking the **Select Frame** button (Fig. 11-4) and selecting a desired one in the newly opened list.
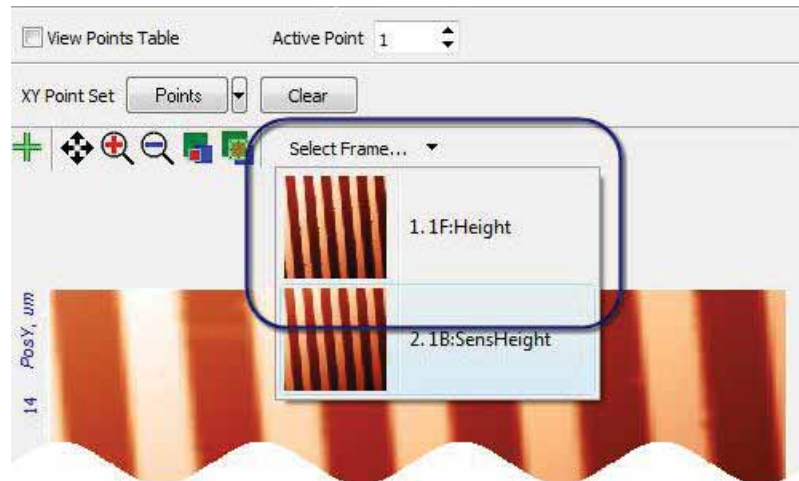

Fig. 11-4. Selecting the desired image of the sample surface

Locations for taking spectroscopy are defined in the 2D Data Viewer of the Spectroscopy window. Available constraints on the point set are given in the **XY Point Set** drop-down list (Fig. 11-5).
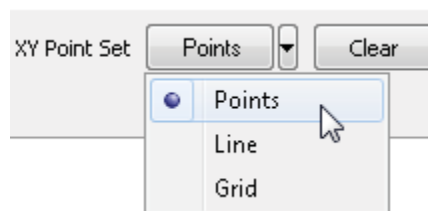

Fig. 11-5. Selecting a mode of indenting

The following modes of selecting points for spectroscopy are available:

Point – points are independent. For selecting a point, click at a corresponding location in the image of the sample surface with the <Ctrl> key pressed.

Line – spectroscopy will be taken at points lying on a predefined straight segment. The segment is defined with the mouse in the 2D Data Viewer. Number of measurement points is defined in the field to the right from the drop-down list (Fig. 11-6). The points will be scattered equidistantly along the segment.

Fig. 11-6

Grid – spectroscopy will be taken at vertices of a predefined grid. The desired rectangular area is defined with the mouse. X and Y grid dimensions are defined in corresponding input fields to the right from the drop-down list (Fig. 11-7).

Fig. 11-7

The predefined points are numbered with their ordinals. Measurements are performed in the order given by the sequence of the point ordinals.

Table of measurement parameters

The Table of measurement parameters serves for independent modification of parameters for particular points.

The Table of measurement parameters (Fig. 11-8) is displayed in the **Spectroscopy** window if the **View Points Table** option is selected.

Every predefined point of spectroscopy is supplied with a record in the Table where the point name along with associated measurement parameters are presented (Fig. 11-8).

By default, all parameters are set to values defined in the Control panel of the **Spectroscopy** window. For individual modification, enter desired values in corresponding cells of the Table.

N	X	Y		From	To	ForwRate	BackRate	LimMin1	LimMax1	▲
1	38,24	17,63	▲▼	317	-50	1,0	1,0	-50,000	50,000	
2	36,77	45,55		317	-50	1,0	1,0	-50,000	50,000	
3	33,83	86,69		317	-50	1,0	1,0	-50,000	50,000	
4	82,37	41,14		317	-50	1,0	1,0	-50,000	50,000	▼

Fill Tools

		Count	Collumn	Fill Type	From	To	
Fill by Active	Fast	5 ▲▼	None ▼	Line ▼	1,0000 ▲▼	1,0000 ▲▼	Fill
	Slow	5 ▲▼	None ▼	Const ▼	1,0000 ▲▼	1,0000 ▲▼	

Fig. 11-8. Table of measurement parameters

11.1.3. Starting the Measurements

To start measurements, click the [▶ Run] button in the Control panel of the **Spectroscopy** window. The **DFL(Z)** relation will be collected at the predefined points for spectroscopy. The point where the probe tip is currently located is marked with a white cross. The measurement process will stop when spectroscopy is taken for all selected measurement points.

11.1.4. Viewing Spectroscopy Data

In the course of measurements, the viewing area of the Spectroscopy window displays spectroscopy data acquired at a point where the tip is located. When the measurements complete, the Viewing Area displays a plot for the last measurement point.

In the course of measurements, the viewing area of the Spectroscopy window displays spectroscopy data acquired at a point where the tip is located. When the measurements complete, the Viewing Area displays a plot for the last measurement point.

The Viewing Area provides displaying either of one of the measured curve or of both of them. A particular appearance of the plot can be adjusted with the [⋁ All ▼] button in the Toolbar.

If spectroscopy was taken at several points and a number of measurements was collected at those points, the curve to be displayed is selected in the following way (see Fig. 11-9):

1. This point can be indicated in the image or selected by its number in the **Active Point** field. The selected point is outlined with a yellow frame in the surface mage.

2. In the list below the Viewing Area, the signal to be displayed is selected.

3. In the **Curve** list, the number of the curve taken at the active point is selected.
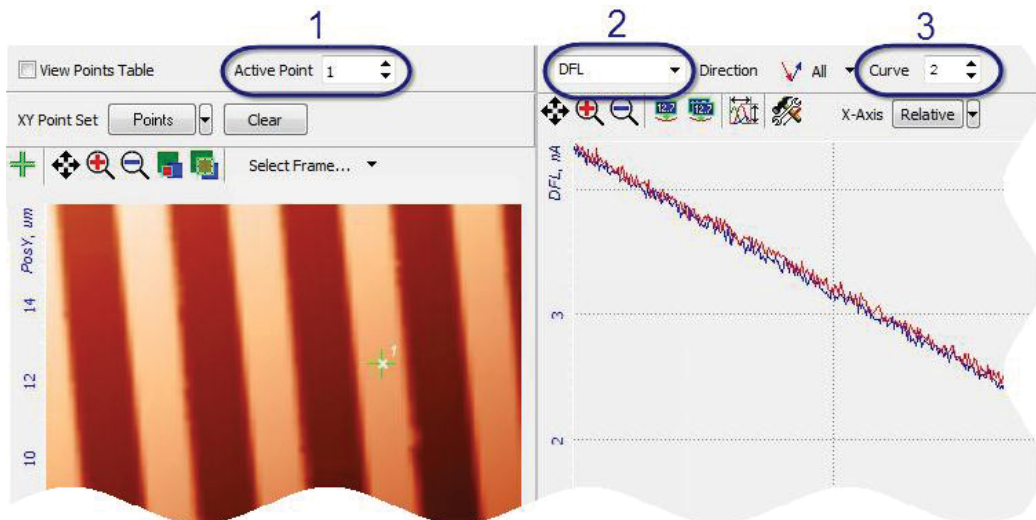
Fig. 11-9

A typical plot of the **DFL(Z)** relation at a point is shown in Fig. 11-10.
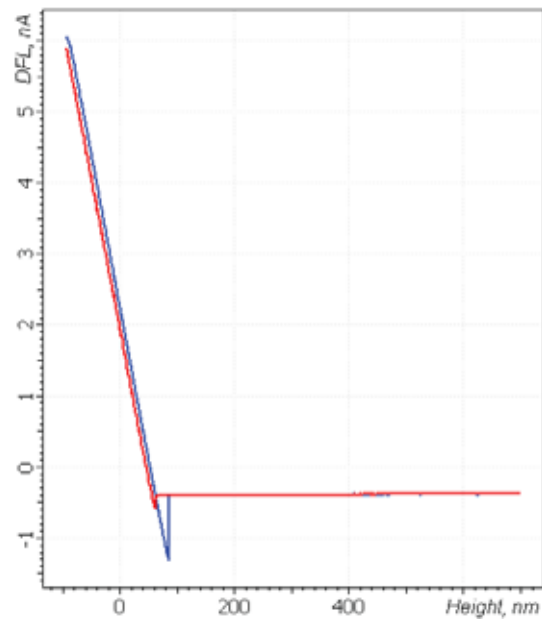

Fig. 11-10. DFL(Z) curve in a point

Using a too "soft" cantilever at high humidity may cause "sticking". If this is the case the **DFL(Z)** curve will look as shown in Fig. 11-11.
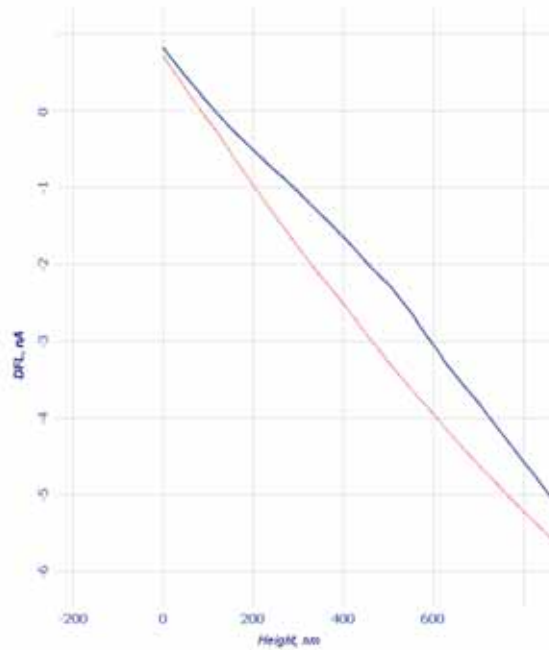
Fig. 11-11. Spectroscopy curve when the cantilever is "stuck"

Curves of Fig. 11-10, Fig. 11-11 display relations acquired with the argument varying in the range from the **From** value to the **To** value in forward (red curve) and in backward (blue curve) directions.

*NOTE. When the argument is **Height**, the curves are called approach (red) and retract (blue) curves.*

11.1.5. Calculating Adhesion Force

In contact modes adhesion forces have a significant effect on the cantilever during the probe withdrawal from the sample. These forces result in the deflection of the cantilever before it breaks contact with the surface. With the z-scanner length being reduced, the **DFL** signal first falls below its value observed well away from the surface, and then abruptly reaches the free-state value, thus forming a specific dip (Fig. 11-12).
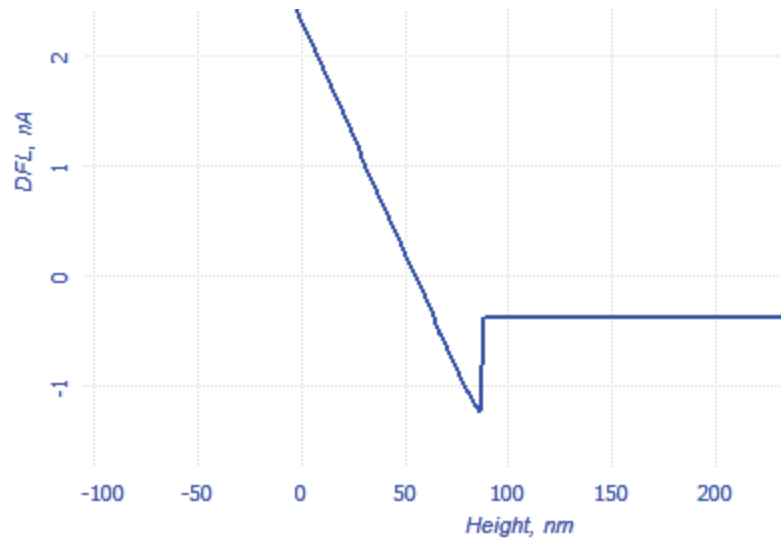

Fig. 11-12

The adhesion force can be calculated assuming the force is a linear function of the probe displacement relative to the sample surface along the Z-axis.

By Hooke's law:

$$F = k \times \varDelta Z, \tag{1}$$

where k – is the cantilever stiffness (see the calibration chart).

For calculating the adhesion force, take the $\varDelta Z$ value. To this purpose, press the button (**Pair Markers**) in the 1D Data Viewer and place the two markers on the inclined portion of the curve as shown in Fig. 11-13.

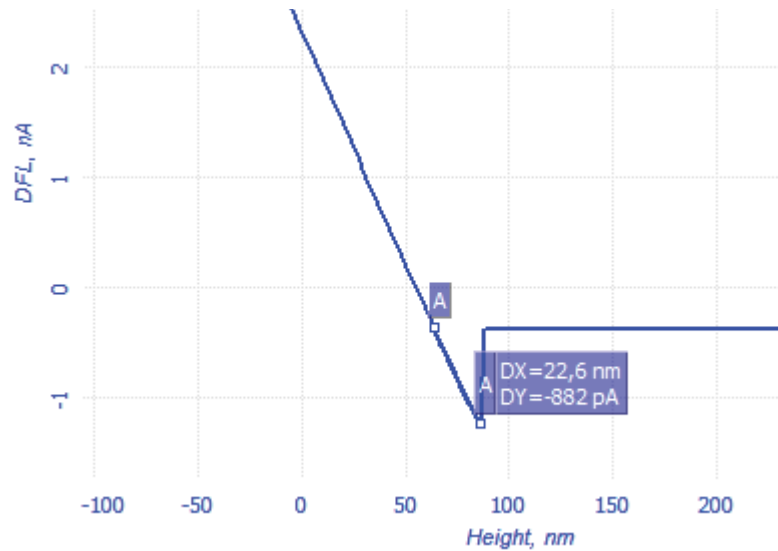*NOTE. For precise measurement of the adhesion force, take the $\varDelta Height$ value for a point where the sample does not bend.*

Fig. 11-13

The values of **DX** and **DY** measured with the markers are Δ **Z** and Δ **DFL**, respectively. In the example Δ **Height** = 22.6 nm.

In our case the cantilever stiffness was ~0.03 N/m. Thus the adhesion force in the spectroscopy point is:

F = 0.03 N/m × 22.6 nm ≈ 0.7 nN.   (2)

In a similar manner, the force acting on the probe (and the sample) during scan can be calculated.

As an example, we can calculate the force acting on the probe during scanning using Constant Force Mode with the parameter **Set Point** = 2 nA. This force results in the Δ **Z** value measured as shown in Fig. 11-14. The position of the first marker corresponds to the **Set Point** value, while that for the second marker reflects the level of the **DFL** signal immediately before the cantilever detached from the sample surface.
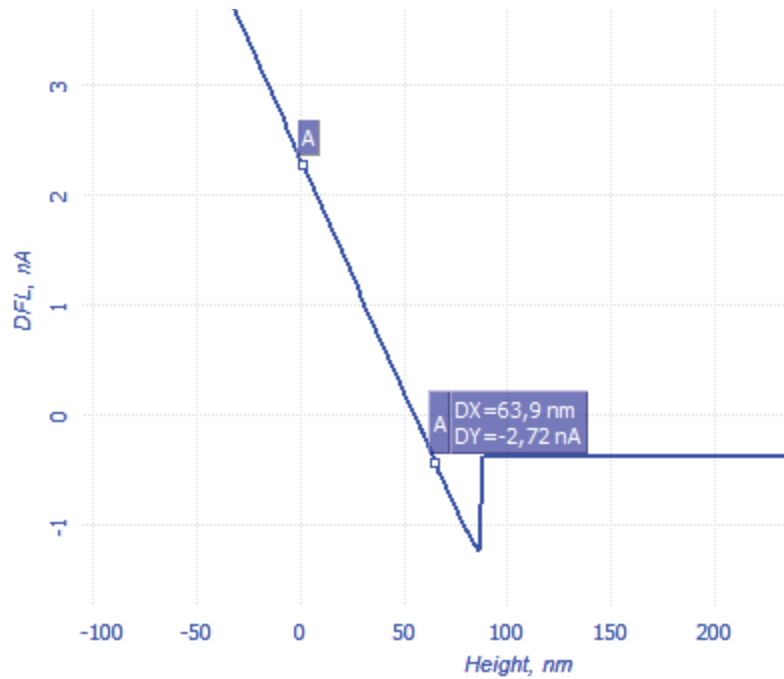
Fig. 11-14

In our example of measurements in the Constant Force mode with **Set Point** = 2 nA, the pressing force is maintained equal to:

$$F = 0.03 \; N/m \times 63.9 \; \text{nm} \approx 1.9 \; nN. \tag{3}$$

11.1.6. Saving Measurement Data

The frame of spectroscopy data is automatically attached to the data file. To save the spectroscopy data in a separate text file, click the 💾 button in the Toolbar of the Viewing Area

11.2. Amplitude Spectroscopy Mag(Z)

In amplitude spectroscopy the amplitude of the cantilever oscillations is measured as a function of the scanner z-tube extension, i.e. the **Mag(Z)** curve is obtained, where **Mag** – is the signal proportional to the cantilever oscillation amplitude and **Z** – is the parameter describing the variation of the scanner z-tube length.

Using the dependence of the amplitude from the piezotube extension it is possible to calibrate the amplitude of the cantilever oscillations.

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the semicontact mode (see i. 8.1 "Semicontact Mode" on p. 80).

11.2.1. Configuring and Making Measurements

The preparatory and measurement procedures are similar to those discussed in i. 11.1 "Force-distance Spectroscopy" on p. 143. Hence, just a brief algorithm is given below. Only the stages that differ from the standard procedure are considered in detail.

For making, the measurements complete the following procedures:

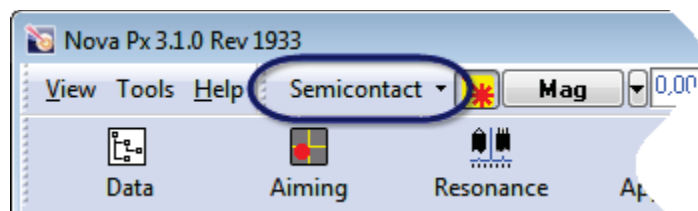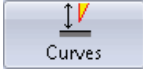1. Check that **Semicontact** is chosen in the controller configuration list (Fig. 11-15).

Fig. 11-15. Selecting the controller configuration

2. Open the spectroscopy window by clicking the [Curves] button.

3. In the **Mode** drop-down list, select **Mag(Z)**. The **Signals** list of the detected relations panel is appended with the **Mag** and **Phase** signals.

   Redefine the set of measured signals, if necessary.

4. In the fields **From** and **To**, define the variation range of *Z*. The range is defined in relative units with the origin at the **Z** level at which **Mag** = **Set Point**.

5. Adjust limits of detecting the **Mag** signal.
   *NOTE. When completing amplitude spectroscopy it is critical to limit the minimum value of **Mag** to avoid excessive impact on the probe and the sample. The minimum **Mag** value should be set about 10 % of the corresponding **Mag** value for a probe fully withdrawn from the surface. With the limits thus set, any range for the **Z** parameter could be specified without danger of damaging the sample, the cantilever or the scanner.*

6. Define measurement parameters at every point of spectroscopy:

   - in the **Move** field – total time of acquisition (in sec) or rate of change of Z under measurements (in um/sec),

   - in the **Measure** field – number of points in the range of measurements,

   - in the **Average** field – number of measurements at every point. The collected data will result from averaging over this number of measurements.

7. Define points for taking spectroscopy. If desired, open the parameter table and redefine measurement parameters of selected points.

8. Launch measurements by clicking the button ▶ **Run** .

When the measurements complete, the Viewing Area displays a plot for the last measurement point. A typical plot of the **Mag(Z)** relation at a point is shown in Fig. 11-16.
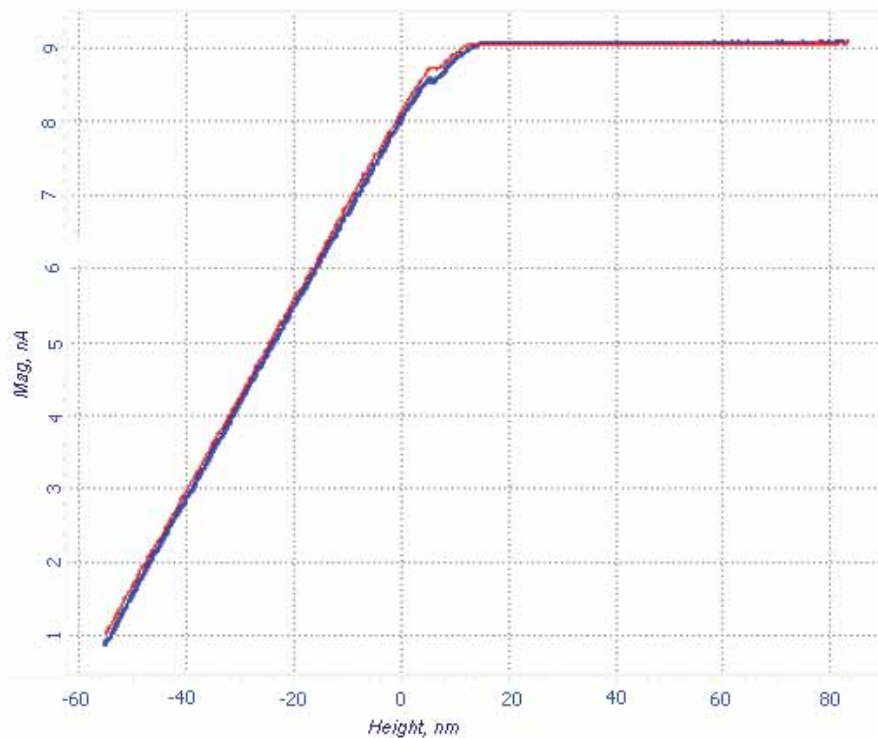

Fig. 11-16. Spectroscopy results

Curves of display relations acquired with the argument varying in the range from the **From** value to the **To** value in forward (red curve) and in backward (blue curve) directions.

11.2.2. Calibration of Cantilever Oscillations Amplitude

Using the measured **Mag(Z)** curve one can calibrate the cantilever oscillations amplitude.

The possibility of such calibration is based on the following assumption. In semicontact mode when the probe starts "tapping" the sample surface any further extension of on the scanner z-tube results in limitation of the cantilever oscillations amplitude. The amplitude decrease is considered equal to the scanner extension (Δ **Height**):

$$\Delta \, Amplitude = \Delta \, Z. \tag{1}$$

This assumption holds if the Q-factor of the system is sufficiently high and the sample is absolutely rigid. In case of standard probes intended for the semicontact modes with rigid samples this assumption is fairly realistic. Therefore, the Δ **Height** range corresponding to the variation of the **Mag** signal from its initial level (equal to **Set Point**) to zero will be equal to the cantilever oscillations amplitude.

As seen from the spectroscopy data, the sloped part of the curve has a region with linear variation of **Mag** against **Height**:

$$\Delta \, Z = K \times \Delta \, Mag, \tag{2}$$

where K – is proportionality constant.

To calculate the calibration coefficient measure the values of Δ **Mag** and Δ **Z**. To this purpose, press the [12.7] button (**Pair Markers**) in the 1D Data Viewer and place the two markers on the inclined portion of the curve (Fig. 11-17). The values of **DX** and **DY** measured using the markers are Δ **Z** and Δ **Mag**, respectively.
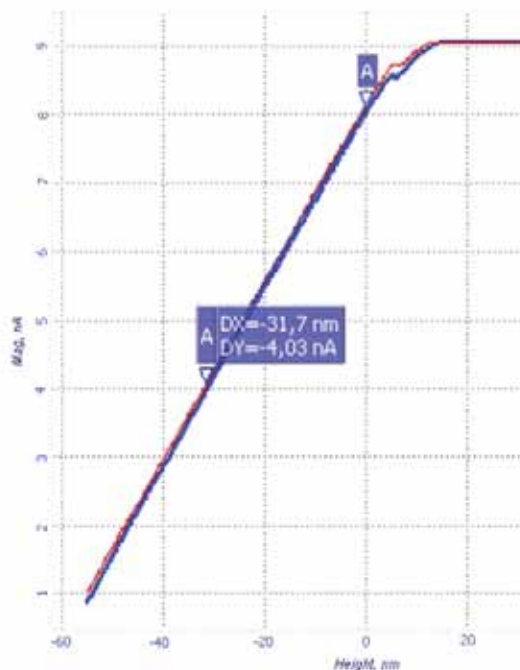

Fig. 11-17

Thus, following equation (2) the proportionality constant connecting the cantilever oscillations amplitude with the value of the **Mag** signal is given by:

$$K = \Delta Z / \Delta Mag = DX / DY = 31.7 / 4 = 7.93 \text{ [nm/nA]}. \tag{3}$$

The actual amplitude of the cantilever oscillations can be calculated from the following equation:

$$Amplitude = K \times Mag. \tag{4}$$

In the example, when performing a scan with the parameter **Set Point** = 8 nA, the amplitude of oscillations is 64 nm.

*NOTE. It should be mentioned that the actual probe oscillations amplitude is independent from the **Gain** parameter specified in the **Lock-In** set of parameters on the instrument block scheme (opens by clicking [Scheme icon]). At the same time, a modification of the **Lock-In** circuit parameters also changes the calculation coefficient. Therefore, the amplitude signal will have to be recalibrated.*

11.3. Current Spectroscopy I_AFM(BV)

In current spectroscopy the current through the probe (the **IprAFM** signal) is measured as a function of the voltage applied between the probe and the sample (the parameter **BV**). For obvious reasons, current measurements are only applicable to conducting samples.

The **IprAFM(V)** plot provides information on local conductivity of the sample under study.

The sample is mounted on the substrate with a spring contact and fixed with double-sided adhesive tape or with glue. Measurements are performed with a probe with conductive coating.

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the contact mode (see i. 7 "Contact AFM" on p. 41).

The preparatory and measurement procedures are similar to those discussed in i. 11.1 "Force-distance Spectroscopy" on p. 143. Hence, just a brief algorithm is given below. Only the stages that differ from the standard procedure are considered in detail.

For making, the measurements complete the following procedures:

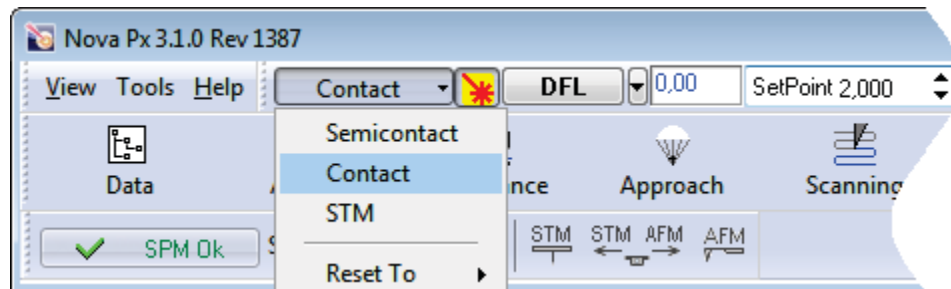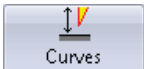1. Check that **Contact** is chosen in the controller configuration list (Fig. 11-15).

Fig. 11-18. Selecting the controller configuration

2. Open the **Spectroscopy** window by clicking the [Curves] button.

3. In the **Mode** drop-down list, select **IprobeAFM(BV)**. The **Signals** list of the detected relations panel is appended with the **IprAFM** signal.

   Redefine the set of measured signals, if necessary.

4. In the fields **From** and **To**, define the variation range of voltage.

5. Define measurement parameters at every point of spectroscopy:

   - in the **Move** field – total time of acquisition (in sec) or rate of change of Z under measurements (in um/sec),

   - in the **Measure** field – number of points in the range of measurements,

   - in the **Average** field – number of measurements at every point. The collected data will result from averaging over this number of measurements.

6. Define points for taking spectroscopy. If desired, open the parameter table and redefine measurement parameters of selected points.

7. Launch measurements by clicking the button [▶ Run] .

When the measurements complete, the Viewing Area displays a plot for the last measurement point. A typical plot of the **IprAFM(BV)** relation is shown in Fig. 11-19.
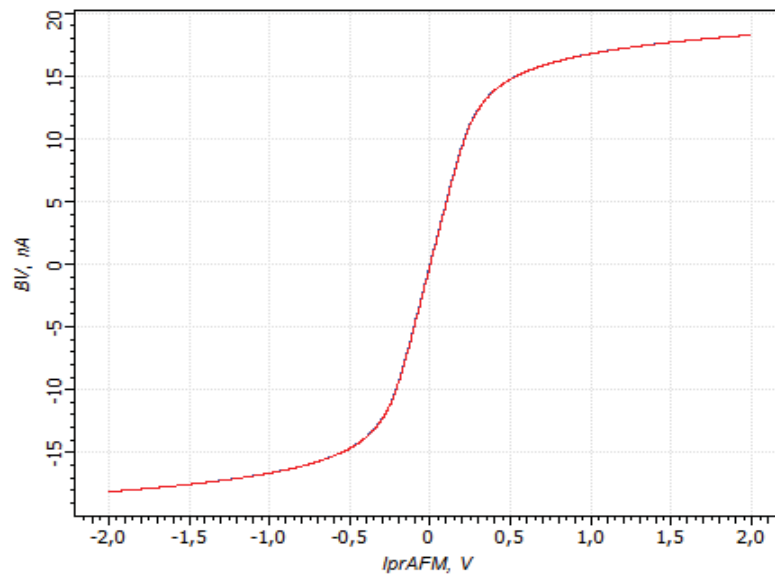

Fig. 11-19

Curves of display relations acquired with the argument varying in the range from the **From** value to the **To** value in forward (red curve) and in backward (blue curve) directions.

12. STM Spectroscopies

Before performing the STM spectroscopy it is necessary to perform preliminary STM measurements of the investigated area topography (see i. 10 "Scanning Tunneling Microscopy" on p. 124).

The preparatory and measurement procedures are similar to those discussed in i. 11.1 "Force-distance Spectroscopy" on p. 143. Hence, just a brief algorithm is given below. Only the stages that differ from the standard procedure are considered in detail.

12.1. *Current Imaging Tunneling Spectroscopy I(BV)*

The procedure of Current Imaging Tunneling Spectroscopy (CITS) consists in acquisition of volt-ampere characteristics (VAC) of a tip – sample tunneling junction. VACs provide information on local conductivity of the sample. Besides, this experimental technique is useful in studying heterogeneities of electrical properties in samples of complex composition.

To making measurements, perform the following steps:

1. Verify that the controller is configured for STM measurements (Fig. 11-15).
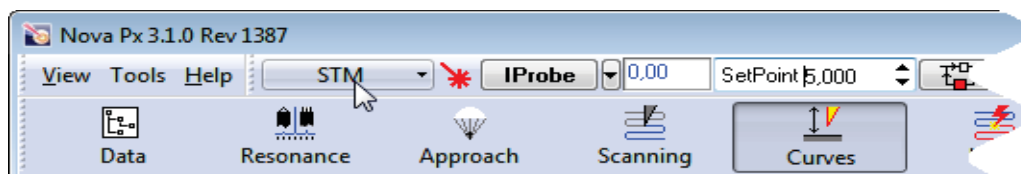

Fig. 12-1. Selecting the controller configuration

2. Open the **Spectroscopy** window by clicking the [Curves] button.
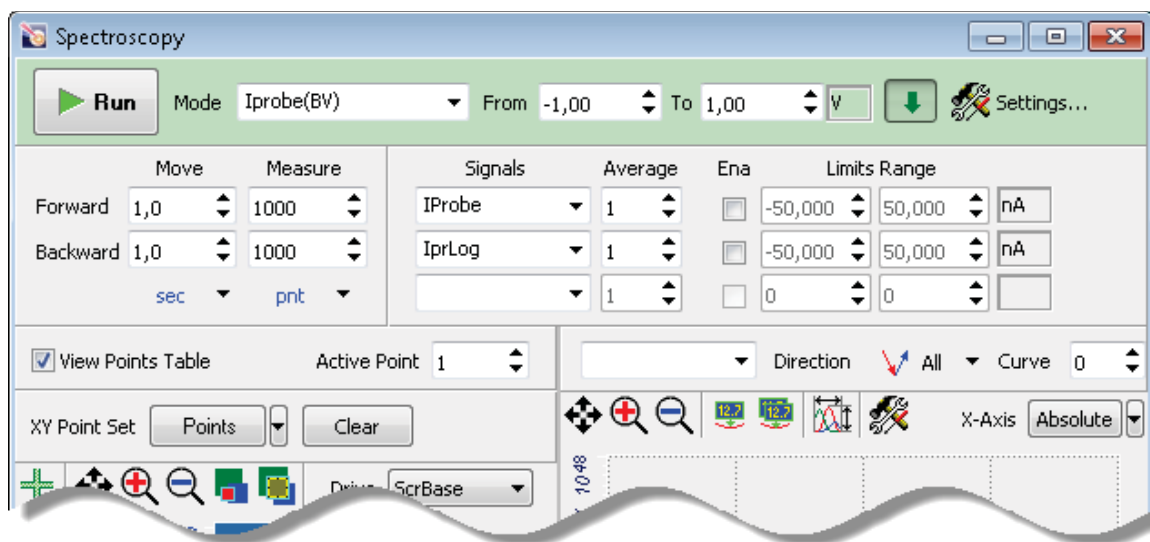

Fig. 12-2. **Spectroscopy** window

3. In the **Mode** drop-down list, select **Iprobe(BV)**. The **Signals** list of the detected relations panel is appended with the **IProbe**, **IprLog** signals.

    Redefine the set of measured signals, if necessary.

4. In the fields **From** and **To**, define the variation range of the voltage.

    NOTE. For variation of the **BV** *parameter, the range from -1 V to +1 V in absolute units is recommended to prevent strong loads on the tip.*

5. Define the desired limits for a signal if necessary.

6. Define measurement parameters at every point of spectroscopy:

    - in the **Move** field – total time of acquisition (in sec) or rate of change of Z under measurements (in um/sec),

    - in the **Measure** field – number of points in the range of measurements,

    - in the **Average** field – number of measurements at every point. The collected data will result from averaging over this number of measurements.

7. Define points for taking spectroscopy. If desired, open the parameter table and redefine measurement parameters of selected points.

8. Launch measurements by clicking the button [▶ Run].

When the measurements complete, the Viewing Area displays a plot for the last measurement point. An example of the current-voltage characteristic of the tip-sample tunnel junction is shown in Fig. 12-3.
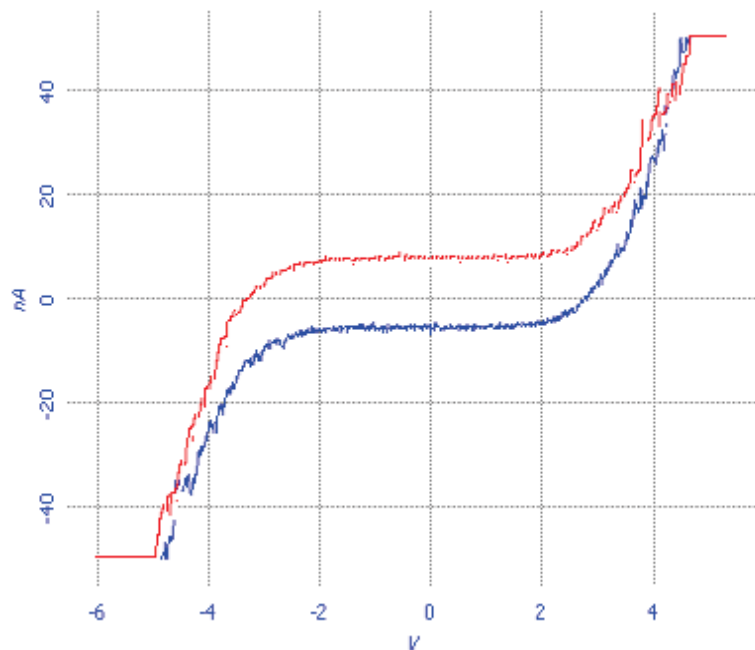

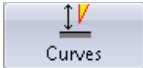Fig. 12-3. CVC of tip-sample tunnel junction

Curves of display relations acquired with the argument varying in the range from the **From** value to the **To** value in forward (red curve) and in backward (blue curve) directions.

12.2. I(Z) Spectroscopy

I(Z)–spectroscopy uses the relation between the tunneling current (IProbe signal) and the distance from the probe to the sample (parameter Z).

With the relation between the tunneling current and the distance from the probe to the sample, sharpness of the tip can be evaluated.

To making measurements, perform the following:

1. Verify that the controller is configured for STM measurements (Fig. 11-15).

2. Open the spectroscopy window by clicking the [Curves] button.

3. In the **Mode** drop-down list, select I(Z). The **Signals** list of the detected relations panel is appended with the **IProbe** signal.

   Redefine the set of measured signals, if necessary.

4. In the fields **From** and **To**, define the variation range of the distance from the probe to the sample.

5. Define the desired limits for a signal if necessary.

6. Define measurement parameters at every point of spectroscopy:

   - in the **Move** field – total time of acquisition (in sec) or rate of change of Z under measurements (in um/sec),

   - in the **Measure** field – number of points in the range of measurements,

   - in the **Average** field – number of measurements at every point. The collected data will result from averaging over this number of measurements.

7. Define points for taking spectroscopy. If desired, open the parameter table and redefine measurement parameters of selected points.

8. Launch measurements by clicking the button [▶ Run].

When the measurements complete, the Viewing Area displays a plot for the last measurement point. An example of the I(Z) Fig. 12-4.
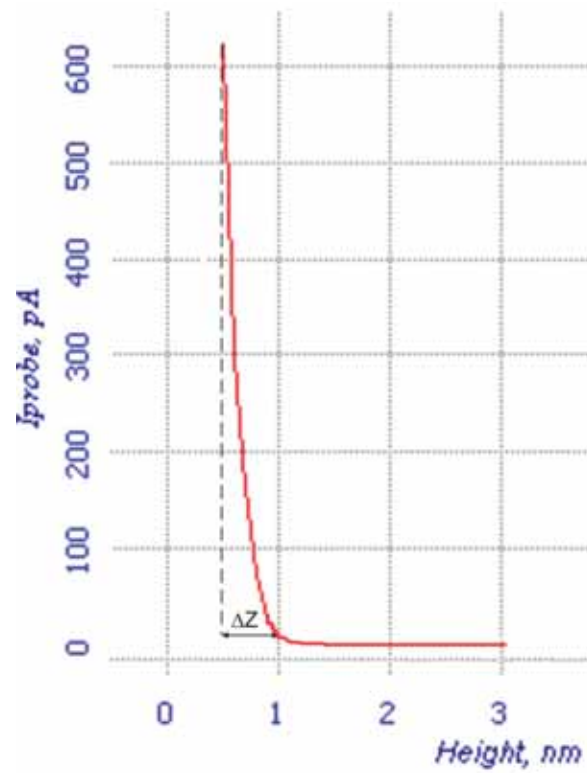
Fig. 12-4. I(Z)

For high resolution of STM images, the condition $\Delta Z < 1$ nm should be met (sharp drop of current in Fig. 12-4). Violation of this condition implies the tip become blunt and it should be exchanged for a sharp one.

13. Lithography

13.1. Overview

This manual is intended for a user experienced in conducting measurements with NT-MDT instruments using contact and semicontact atomic force microscopy and spectroscopy.

The Nova Px program provides three lithography technologies:

- **Force lithography**. During the lithography process, the force applied to the sample by the sharp probe tip is maintained constant (see i. 13.2 on p. 169);

- **Charge lithography**. During the lithography process, the electrical sample-probe bias is maintained constant (see i. 13.3 on p. 186);

- **Current lithography**. During the lithography process, the electrical sample-probe bias is controlled to maintain the current constant (see i. 13.3 on p. 186).

Every of those technologies can operate either in the vector (see i. 13.1.1 on p. 164) or in the raster mode (see i. 13.1.2 on p. 168).

13.1.1. Vector Lithography

Vector lithography is the process of influence on the sample surface during the probe movement along the predefined vector template.

The vector lithography procedure needs a lithography area and a lithography template (a series of lithography objects) to be defined before starting the procedure.

Impact produced by the probe on the sample depends on the kind of displacement of the probe (along the template elements or between the elements). When the tip moves to the beginning of the template or between separated elements of the template, it impacts the sample surface with the force defined by the $A0$ parameter (Fig. 13-1) viewed to the right from the list of lithography methods. When the tips moves along a template element, the force can be constant (defined by the $A1$ parameter in the **Action** group) or varying linearly inside a predefined range (from $A1$ to $A2$). Besides, the pulse shape option is available for the pulse lithography.
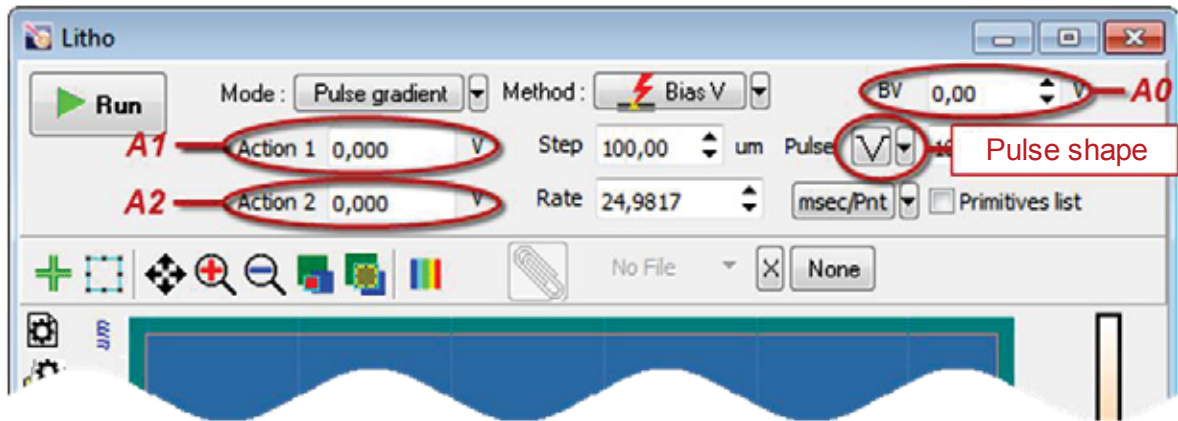
Fig. 13-1. Main lithography parameters in the Control panel
of Pulse-gradient charge lithography

The Nova Px program provides four options to perform the vector lithography:

- Simple vector lithography (see p. 165);
- Gradient lithography (see p. 166);
- Pulse lithography (see p. 166);
- Pulse-gradient lithography (see p. 168).

These options are explained in details below.

Simple vector lithography

The Figure below demonstrates a time diagram of the force applied to the sample in the course of the Simple vector lithography (Fig. 13-2).
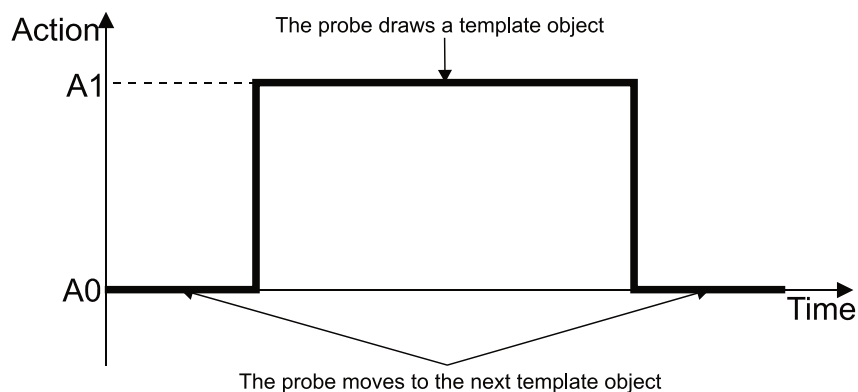
Fig. 13-2. Force applied to the sample in the course of the Simple vector lithography

When the probe draws an object of the lithography template, the sample is applied to the constant force *A1*. Level of the *A1* parameter is defined in the **Action** field of the Control panel of the **Litho** window.

Gradient lithography

The Figure below demonstrates a time diagram of the force applied to the sample in the course of the Gradient lithography (Fig. 13-3).
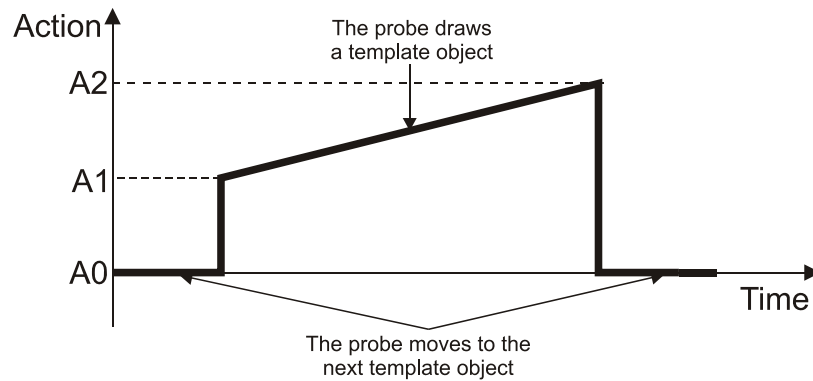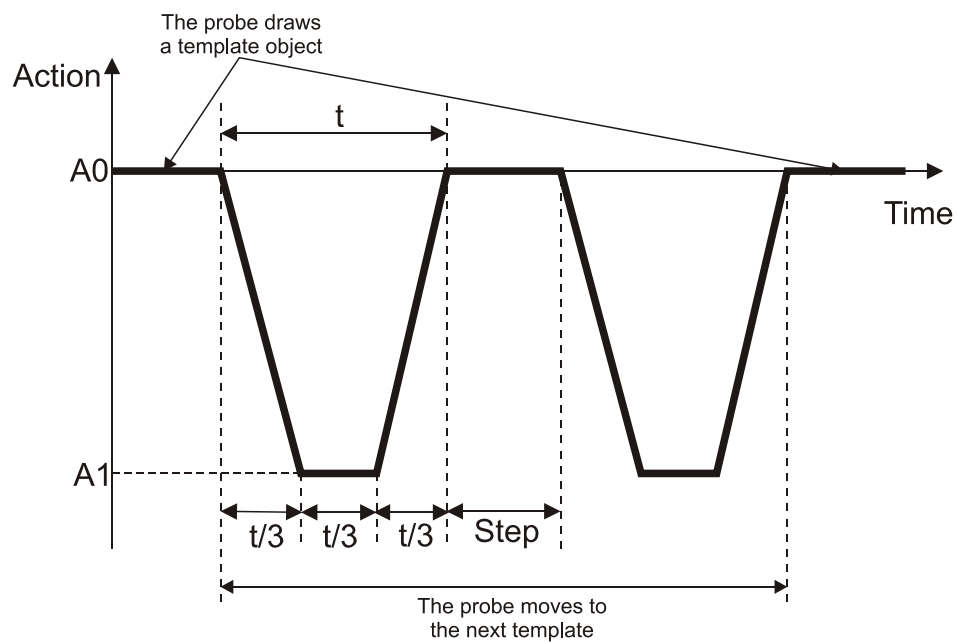

Fig. 13-3. Force applied to the sample in the course of the Gradient lithography

When the probe draws an object of the lithography template, the sample is applied to the force is ramping linearly from the *A1* level to the *A2* level. The lowest (*A1*) and the highest (*A2*) levels are defined in the fields **Action 1** and **Action 2** of the Control panel of the **Litho** window, respectively.

Pulse lithography

The Figure below demonstrates a time diagram of the force applied to the sample in the course of the Pulse lithography (Fig. 13-4).

Fig. 13-4. Force applied to the sample in the course of the Pulse lithography.
Example of the ⏝ pulse shape is shown

When the probe draws an object of the lithography template, the sample is applied to pressure pulses of predefined amplitude *A1*. The *A1* level is defined in the **Action** field of the Control panel of the **Litho** window. The pulse shape is selectable and is adjusted according to application.

Time step is defined in the **Step** field of the Control panel of the **Litho** window.

Available pulse shapes are given in the **Pulse** drop-down list of the Control panel of the **Litho** window. The field to the right of the **Pulse** list serves for adjusting the pusle duration t in ms (Fig. 13-5).

Fig. 13-5. List of available pulse shapes

Depending on the selected pulse shape, the time interval of a pulse is subdivided as follows:

1) Pulse shape . The time interval *t* is divided by two parts. Initial level of the applied force is *A0*. During the first part (of duration *t/2*) the force ramps to the level *A1* and then in the second part (of the same duration *t/2*) the force returns to its initial level (*A0*).

2) Pulse shape . The applied force gets its maximum level *A1* instantly and then the force remains constant for the time *t*. After the time t expires the force returns to the level *A0*.

3) Pulse shape . The time interval *t* is divided by three equal parts (see Fig. 13-4). Initial level of the applied force is *A0*. The applied force ramps linearly during the first part (of duration *t/3*), to the level *A1* and remains constant for the next subinterval *t/3*. Finally, during the last subinterval *t/3* the force returns to its initial level *A0*.

4) Pulse shape . The applied force ramps linearly to the level *A1* during the time *t*. After the time *t* expires the force returns to the level *A0*.

5) Pulse shape . The applied force gets the level *A1* instantly. Then, during the time *t* the force returns linearly to its initial level *A0*.

6) Pulse shape . The time interval *t* is divided by two equal parts. The applied force ramps linearly to level *A1* during time *t/2* and then remains constant during the second subinterval *t/2*. After the time *t/2* expires the force returns to the level *A0*.

7) Pulse shape . The time interval *t* is divided by two equal parts. The applied force gets its maximum level *A1* instantly and then the force remains constant for the time *t/2*. Finally, during the subinterval *t/2* the force returns to its initial level *A0*.

Pulse-gradient lithography

Pulse-gradient lithography is a combination of two modifications of lithography, the pulse and the gradient ones. The Figure below demonstrates a time diagram of the force applied to the sample in the course of the Pulse-gradient lithography(Fig. 13-6).
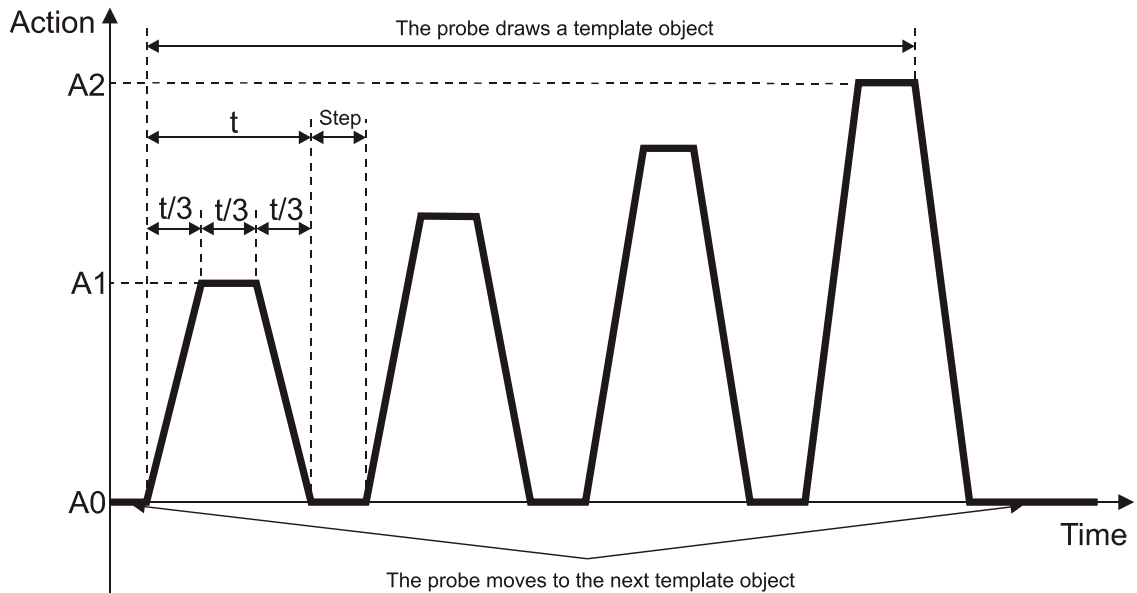

Fig. 13-6. Force applied to the sample in the course of the Pulse-gradient lithography. Example of the pulse shape is shown

When the probe draws an object of the lithography template, the sample is applied to pressure pulses of varied amplitude. The amplitude varies in a predefined range from *A1* to *A2*. Initial and final levels of the amplitude (*A1* and *A2*) are defined in the fields **Action 1** and **Action 2** of the Control panel of the **Litho** window, respectively.

The Control panel of the **Litho** window contains a drop-down list for adjusting the pulse shape and a field for adjusting the pulse duration t in ms (Fig. 13-5).

Time step is defined in the **Step** field of the Control panel of the **Litho** window.

13.1.2. Raster lithography

The template is taken from a graphics file of raster format.

In the course of raster lithography, the probe tip moves along lines of the lithography template and the tip impacts the sample at points of the raster. Step between the template lines at which the surface will be impacted is defined in the **Step** input field.

Levels of the applied force are proportional to brightness of pixels in the template. The impact range is defined in the input fields **Action 1** and **Action 2**. The **Action 1** is associated with the lower limit (black) of the color scale while the **Action 2** is associated with the upper level (white).

Simple raster lithography

Time the probe tip impacts the sample surface depends on the rate of performing lithography and on the number of points in lines of the template.

Pulse-raster lithography

Pulse-raster lithography combines features of two lithography techniques, the pulse and the raster ones.

In the course of pulse-raster lithography, the probe tip moves along lines of a raster template the tip impacts the sample at points of the raster by pulses of a predefined shape. The impact range is defined in the input fields **Action 1** и **Action 2**. The **Action 1** is associated with the lower limit (black) of the color scale while the **Action 2** is associated with the upper level (white). Impact during motion between pulses is defined in an input field to the right from the list of lithography techniques.

Total time of pulse-raster lithography depends on the speed at which the tip moves between the raster points (**Rate** parameter) and time of a pulse (**Pulse** parameter).

13.2. Force lithography

Force lithography is based on direct mechanical impact produced by a sharp probe on the sample surface. The probe tip pressure on the surface is sufficient to cause plastic deformation (modification) of the substrate surface. This type of modification has a range of applications in nanoelectronics, nanotechnology, material science, etc. It enables the fabrication of electronic components with active areas of nanometer scale, super dense information recording and study of the mechanical properties of materials.

Requirements on the sample and on the probe

For force lithography the hardness of the probe material shall exceed that of the sample. Also, the cantilever sticking and the material pickup shall be avoided. Small sample roughness is required (typically below 1÷10 nm). Moreover, the sample surface has to be clean. Force lithography is best performed on certain polymers including polycarbonate and polyethylene and LB-films.

Semicontact probes with the stiffness up to 5.5 N/m are typically used in force lithography.

Depending on the selected sample there are three options of performing lithography and viewing the results:

- carrying out lithography using contact mode and scanning by semicontact mode to view the results;

- performing lithography in semicontact mode (with the value of **Level2** within 5÷10 % from the **Mag** signal for free cantilever oscillations) and scanning also using the semicontact mode (with **SetPoint** around 80 % from the **Mag** signal for free cantilever oscillations);

- using contact mode to conduct lithography and scanning also by the contact mode (since the pressure has little variations depending on the **SetPoint** value viewing lithography results in this mode might be difficult).

Force lithography can be performed in the vector or in the raster mode. Whatever mode is used, preparation operations are the same.

This Chapter deals with:

1. Preparation for operation (see i. 13.2.1 on p. 170);

2. Performing vector force lithography (see i. 13.2.2 on p. 174);

3. Performing raster force lithography (see i. 13.2.3 on p. 180).

The lithography procedures will be demonstrated using a fragment of CD as the sample and the probe of NSG30 model. In this case, it is appropriate to perform lithography by a contact technique while to use a semi-contact technique for scanning.

13.2.1. Preparation for Operation

Basic preparatory operations are:

1. Estimation of the Probe Pressure on the Sample (see i. 13.2.1.1 on p. 170);

2. Preliminary Scanning and Selecting Lithography Region (see i. 13.2.1.1 on p. 170).

These operations are explained in details below.

13.2.1.1. Estimation of the Probe Pressure on the Sample

In lithographic experiments, it is often critical to know the pressure which the probe applies to the sample. To estimate the pressure corresponding to a specified level of the probe impact, the force created by the probe has to be determined first. It can be calculated from force spectroscopy data. The only requirement here is that the sample has a rigid surface to avoid punching.

Acquire the force spectroscopy data at a point (see i. 11.1 on page 143 in details). To this purpose, perform the following steps:

1. In the controller configuration drop-down list of the Nova PX program, select **Contact** (Fig. 13-7).
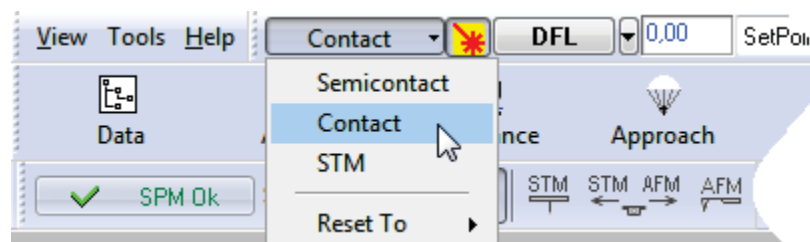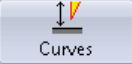
Fig. 13-7. Selecting the controller configuration

2. Close the feedback loop (the ⟨ Off ⟩ button will change its appearance to ⟨ On ⟩).

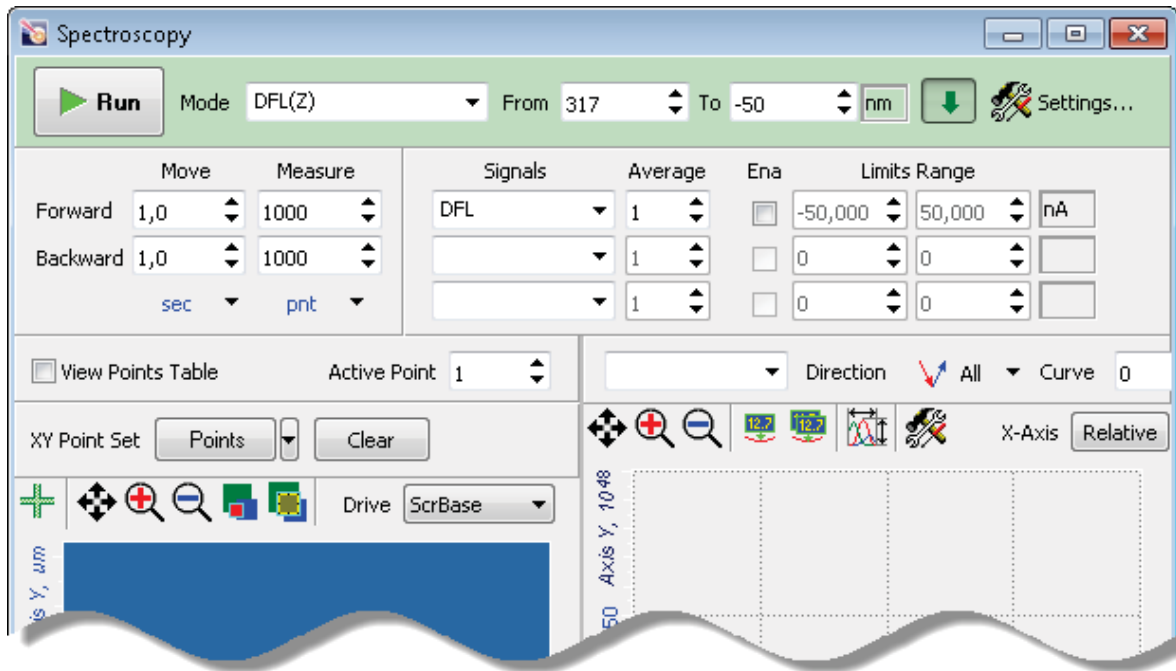3. Open the **Spectroscopy** window by clicking the button in the Main Operations panel (Fig. 13-8).

Fig. 13-8. **Spectroscopy** window

4. In the **Mode** drop-down list, select **DFL(Z)**. This will append the list of detected signals with the **DFL** signal.

5. In the fields **From** and **To** define the range of the scanner height. The range is relative to the current scanner position. We recommend to use the range from 0.1 to 0.5 μm.

6. Select a point for spectroscopy.

7. Launch the acquisition procedure by clicking the button. Spectroscopy acquisition will provide an approach curve (Fig. 13-9).
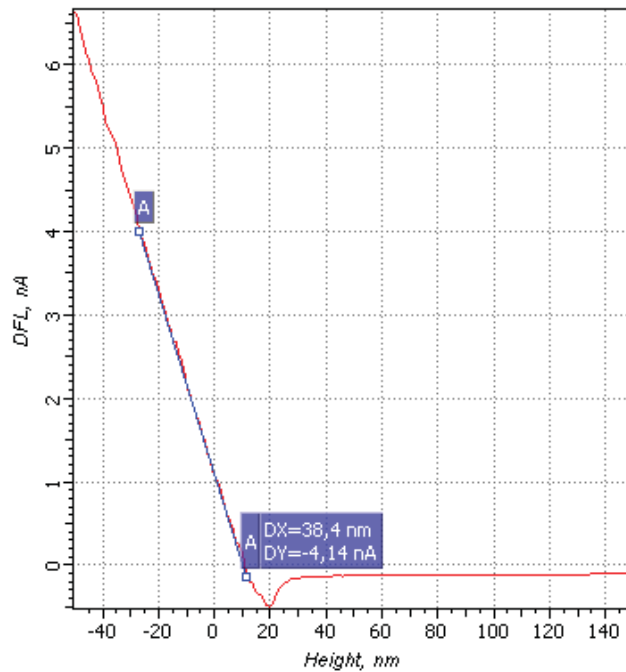
Fig. 13-9. Approach curve

Calculating the force applied to the probe

With the spectroscopy data, calculate the force F_Z applied to the probe as:

$$F_Z = \Delta Z \cdot k,$$

where ΔZ is deflection of the cantilever from its equilibrium, and k is rigidity of the cantilever (see specification).

As an example, let's consider calculation of the force applied to the probe under scanning in the Constant Force mode at **Set Point** = 4 nA with k = 10 N/m.

First, place the pair marker on the acquired approach curve (see Fig. 13-9). The first marker of the pair should be moved to the **Set Point** level while the second one is positioned at the **DFL** level immediately before the cantilever detaches the sample surface. . ΔZ corresponds to **DX**.

Value of the $\Delta Height$ parameter is given by **DX** measured with the approach curve as shown in Fig. 13-9.

In our example, with the parameter **Set Point** = 4 nA, constant pressing force is maintained:

$$F_Z = 38.4nm \cdot 10N/m = 384\,nN$$
.

Now, find the **SetPoint** level corresponding to the desired interaction force.

To find the probe pressure on the sample, the surface of the probe-sample interaction S is to be estimated. You can use the following equation:

$$S = \pi \cdot R^2 \,,$$

where R ≈ 10÷50 nm – is the tip apex radius.

Thus, the probe pressure on the sample can be found as:

$$P = F_z / S = 384 nN / 7850 N^2 m^2 \approx 40 MPa \,.$$

In the example above **SetPoint** = 4 mA corresponds to the pressure of 40 MPa.

NOTE. The errors of these estimations are combined from the uncertainty of parameters including tip curvature radius R and stiffness coefficient k, the dependence between the contact area and the applied force, and also from nonlinear behavior of force Fz as a function of the scanner displacement.

13.2.1.2. Preliminary Scanning and Selecting Lithography Region

Before performing the force lithography, take a testing scan in a semicontact mode over the maximum scanning area. For details on measurements with semi-contact AFM techniques, refer to i. 8 on p. 80.

NOTE. For lithography, plain regions are preferable to minimize effects of landscape features on results of lithography.

In the acquired scan, select a region for lithography. Take a scan of the selected region.

This completes preparing for force lithography. An example of the surface image in the selected region is shown in Fig. 13-10. Scanning parameters are: scanning mode – semicontact; **Mag**=10; **SetPoint**=5; **BV**=0.4 V.
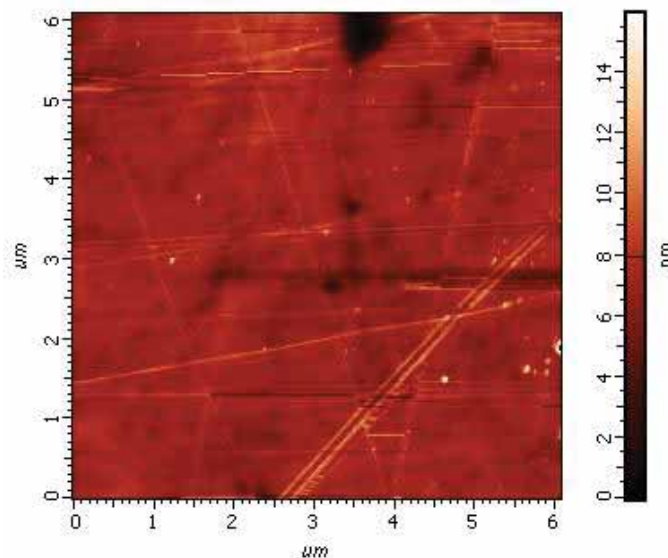
Fig. 13-10. Selected region for lithography

13.2.2. Vector Force Lithography

⚠️ **ATTENTION!** Before performing vector lithography, lern i. 13.1 "Overview" on p. 164.

Initial state

The following preparatory operations are assumed to be done:

- pressure of the probe onto the sample surface is estimated;
- the surface region for lithography is selected and scanned.

For details on these operations, refer to i. 13.2.1 "Preparation for Operation" on p. 170.

Basic operation

Basic operations of force vector lithography are:

1. Selecting the lithography method (see i. 13.2.2.1 on p. 174);
2. Creating the Lithography Template (see i. 13.2.2.2 on p. 176);
3. Adjusting Vector Lithography Parameters (see i 13.2.2.3 on p. 178);
4. Performing Lithography (see i. 13.2.2.4 on p. 179).

These operations are explained in details below.

13.2.2.1. Selecting the lithography method

To select the lithography mode, perform the following steps:

1. In the controller configuration drop-down list of the Nova Px program, select **Contact** (Fig. 13-11).
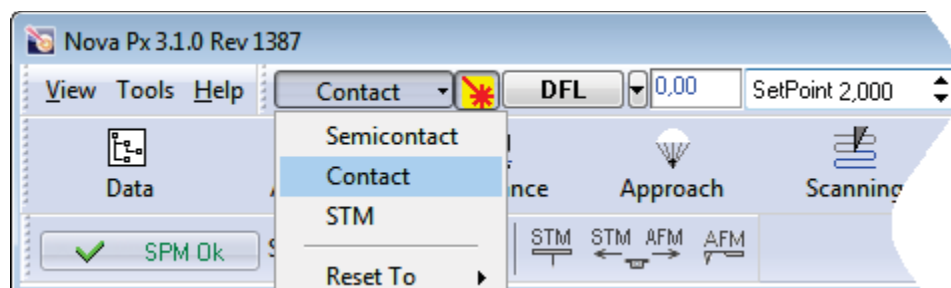
Fig. 13-11. Selecting the controller configuration

2. Adjust the **SetPoint** value for the contact mode as follows:

   a. Open the feedback loop (the **On** button will change its appearance to ⎡🔲 Off⎤);

   b. Define the **SetPoint** to be two times less than **DFL**;

   c. Close the feedback loop (the ⎡🔲 Off⎤ button will change its appearance to **On**);

   d. Gradually increase **SetPoint** and monitor Z-scanner extension in the progress bar located in the Main Operations panel (Fig. 13-12). For the **SetPoint**, enter a value at which extension of the Z-scanner stops.

Fig. 13-12. Scanner extension progress bar

3. Define operating value of the feedback gain (**Gain** parameter) as follows:

   a. Double-click on the **Gain** field. This will open the adjusting slider of the feedback gain (Fig. 13-13).
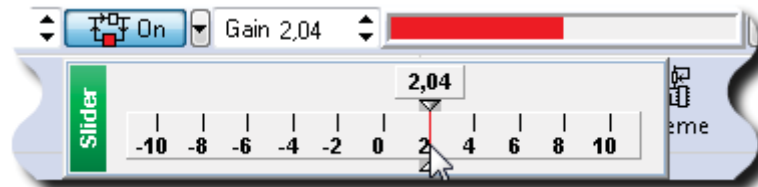
Fig. 13-13. Adjusting slider of the **Gain**

   b. Gradually decrease the **Gain** value and monitor level of the feedback input in the software oscilloscope.

   c. Find the **Gain** value at which generation starts. Generation originates when the fluctuating component of the input becomes noticeable.

   d. Define the working level of the **Gain** to be 0.5÷0.7 of the gain when oscillations start.

4. Open the **Litho** window (Fig. 13-14) by clicking the ⎡🔺 Litho⎤ button in the Main Operations panel.
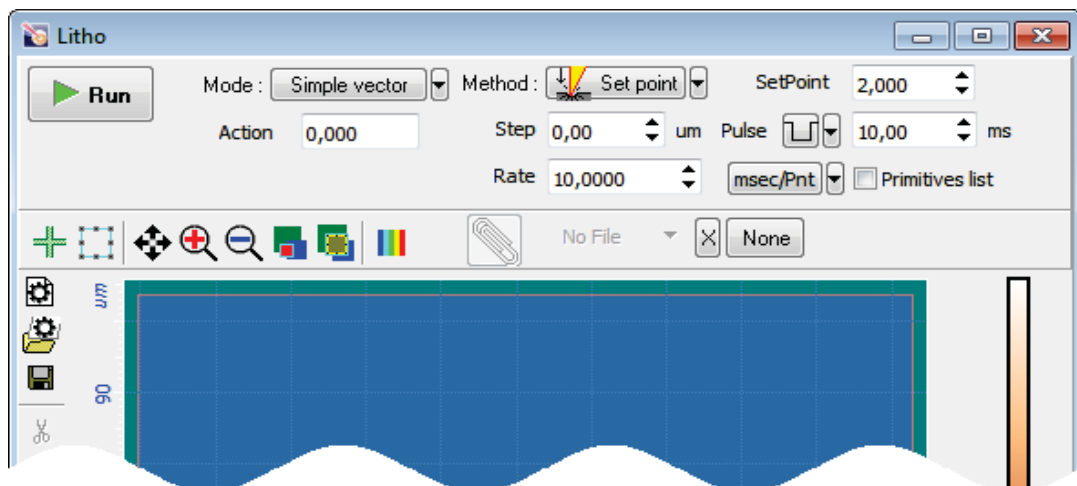
Fig. 13-14. **Litho** window

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5. For Force lithography, select **SetPoint** in the **Method** drop-down list (Fig. 13-15).
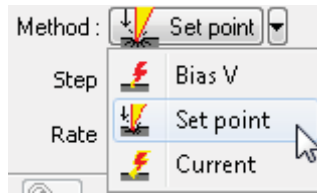
Fig. 13-15. Selecting the lithography method

NOTE. Appearance of the Control panel of the Lithography window depends on the selected mode of vector lithography.

6. In the **Mode** drop-down list, select a desired option of vector lithography (Fig. 13-16). Four options of vector lithography are available:

- **Simple vector** – simple vector lithography. Impact level is maintained constant through the whole lithography cycle;

- **Gradient** – gradient lithography. Impact level varies linearly in a predefined range;

- **Pulse** – pulse lithography. The sample is exposed to pulses of constant level;

- **Pulse gradient** – pulse-gradient lithography. Combines pulse and gradient lithography. The sample is exposed to pulses with amplitude linearly varying in a predefined range.

  NOTE. For details on options of vector lithography refer to i. 13.1.1 on p. 164.
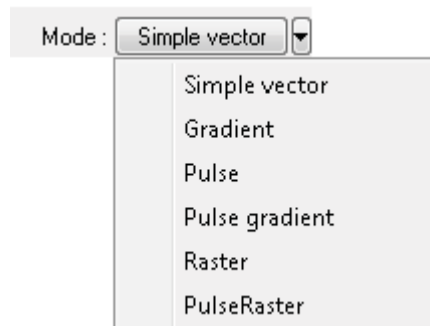
Fig. 13-16. Selecting option of vector lithography

13.2.2.2. Creating the Lithography Template

1. Create a new lithography template. To this purpose, click the ⊞ button in the Toolbar of the Lithography template (Fig. 13-17). This will remove all objects created before. This step is required to prevent any object outside the selected region to appear in the final template.

2. In the Lithography template viewing area, define positions and sizes of the desired objects with the tools of the Toolbar.
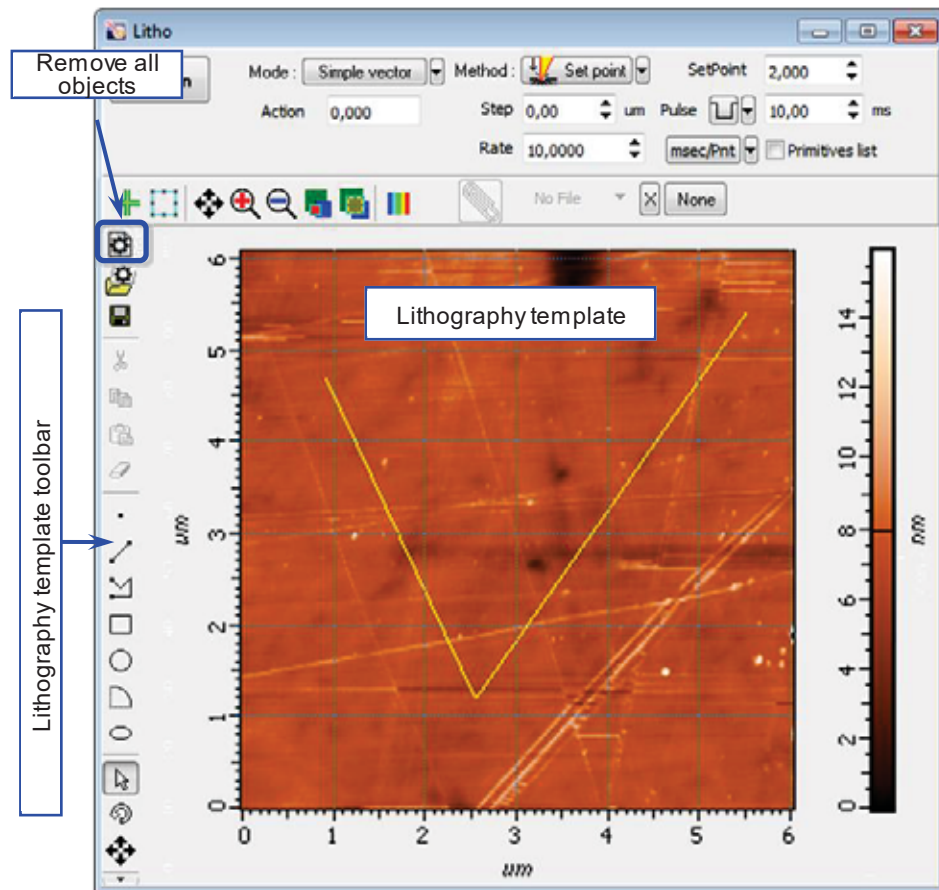
Fig. 13-17. An example of the template of vector lithography

Parameter table of the lithography template

By default, all lithography elements are attributed with parameters defined in the Control panel of the **Litho** window. If desired, the parameters can be adjusted individually for particular points. Their values can be edited in the Parameter table of the lithography template (Fig. 13-18) opened with the **Primitives list** option selected.

	Action 1	Action 2	Rate	Step	Pulse
line 5	6,985		10,000	0,000	10,000
line 14	6,985		10,000	0,000	10,000
rect 20	6,985		10,000	0,000	10,000
circle 59	6,985		10,000	0,000	10,000
arc 67	6,985		10,000	0,000	10,000

Save   Load   Reset to defaults

Measure None   Select Scripts

Fig. 13-18. Parameter table of the lithography template

Adding a new element to the lithography template extends the table by a row в containing the name of the element and its lithography parameters. Desired values of the parameters can be entered in corresponding cells of the table. For synchronized modification of parameters of several elements, mark their names with the mouse with the <Ctrl> key pressed. Then changes of an element in the marked group will spread to all members of the group.

13.2.2.3. Adjusting Vector Lithography Parameters

Composition of the set of adjustable parameters depends on the selected mode of vector lithography (for details on the vector lithography modes, refer to i. 13.1.1 on page 164).

1. Define the level the probe tip impacts the sample surface during motion along the elements of the lithography template:

   - For simple and pulse lithography – enter the impact level in the **Action** field;

   - For gradient and pulse-gradient lithography – enter the minimum and the maximum of the impact level in the fields **Action 1** and **Action 2**, respectively.

   Relation between pressure on the sample and the **Action** value is given in i. 13.2.1.1 "Estimation of the Probe Pressure on the Sample" on p. 170.
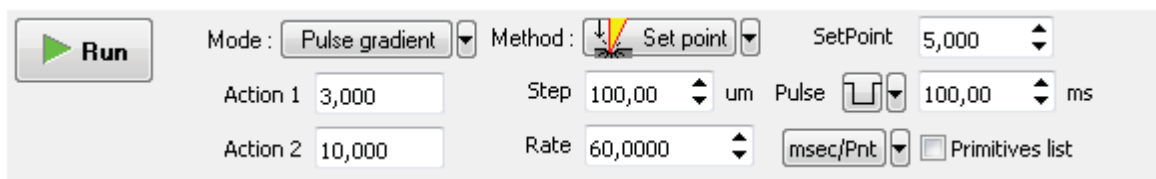
Fig. 13-19. Control panel for pulse gradient lithography

2. In the **SetPoint** field, define the force applied to the sample when the probe moves to starting points of the template objects after finishing drawing or at the start of lithography.

3. In the **Rate** field, define the lithography rate (the speed of moving the probe relative to the sample surface).

   The lithography rate influences on results of the sample exposure to lithography. The lower is the rate, the deeper is the exposure and the better is the correspondence between the template and the resulting surface.

   NOTE. The parameters **Step** *and* **Pulse** *are irrelevant for the simple vector lithography and gradient vector lithography.*

4. For pulse and pulse-raster lithography, define parameters of pulses:

   - In the **Step** field, define the time step between the pulses.

   - In the drop-down list **Pulse**, select the desired pulse shape.

   - In the **Pulse** field define duration of a pulse.
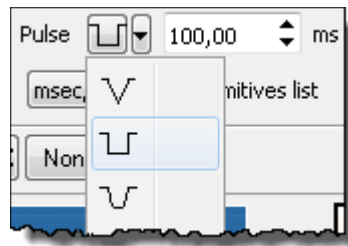
Fig. 13-20. Pulse shape

13.2.2.4. Performing Lithography

When the lithography template is prepared and the lithography parameters are adjusted, the operator can start the lithography process.

Operations of the lithography procedures are the same for all modes.

To start lithography, click the ▶ Run button in the Control panel of the **Litho** window. This will change appearance of the **Run** button to ■ Stop . Below the **Stop** button, the progress bar will appear to display percentage of the procedure progress in blue.

When the procedure completes, the **Stop** button returns to its initial appearance.

The lithography process can be stopped by clicking the **Stop** button or by pressing the <Esc> key. This will stop the process immediately. Next clicking the **Run** button would start the lithography procedure from the very beginning, i.e. from the first template point.

Lithography Results

Take a scan of the sample to acquire an image of the surface region exposed to lithography.

Fig. 13-21 shows the image for the Simple vector lithography applied to the CD sample with **SetPoint** = 0.5 nA, **Action** = 4 nA by the NSG30 probe.
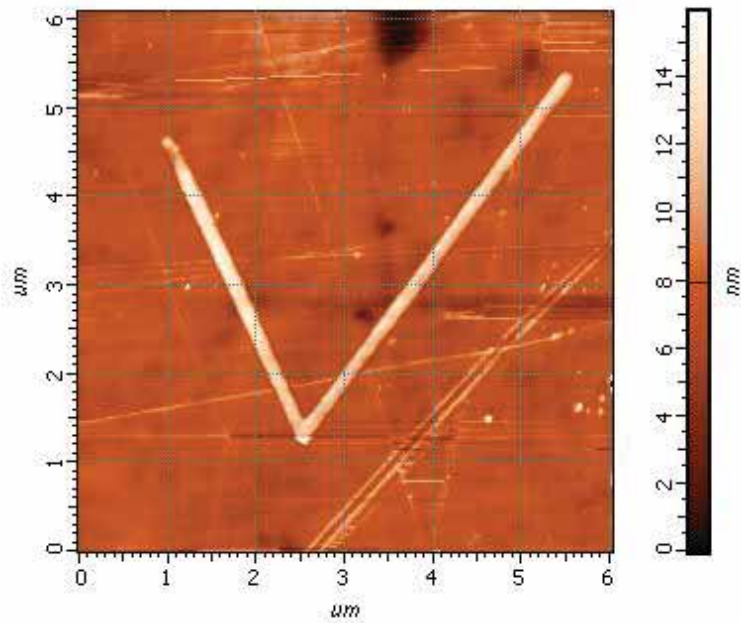
Fig. 13-21. Results of the Simple vector lithography

13.2.3. Raster Force Lithography

⚠️ **ATTENTION!** **Before performing raster lithography, learn i. 13.1 «Overview » on p. 164.**

Initial state:

The following preparatory operations are assumed to be done:

- pressure of the probe onto the sample surface is estimated;
- the surface region for lithography is selected and scanned.

For details on these operations, refer to i. 13.2.1 «Preparation for Operation» on p. 170.

Basic operation

Basic operations of force raster lithography are:

1. Selecting the lithography method (see i. 13.2.3.1 on p. 181);
2. Loading Template (see i. 13.2.3.2 on p. 183);
3. Adjusting Lithography Parameters (see i. 13.2.3.3 on p. 183);
4. Performing Lithography (see i. 13.2.3.4 on p. 185).

These operations are explained in details below.

13.2.3.1. Selecting the lithography method

To select the lithography method, perform the following steps:

1. Select **Contact** in the drop-down list of controller configurations of the Nova Px program (Fig. 13-22).
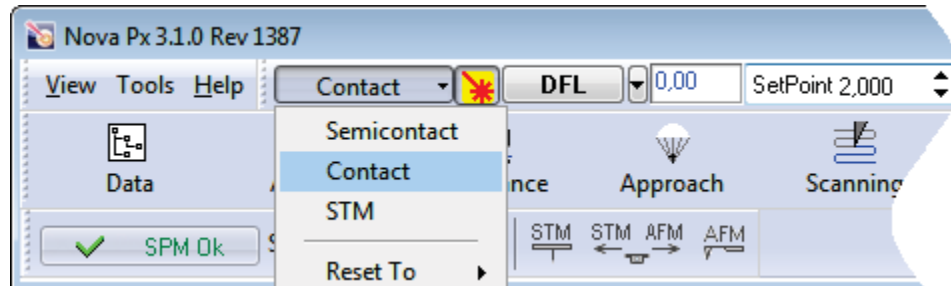
Fig. 13-22. Selecting controller configuration

2. Adjust the **SetPoint** value for the contact mode as follows:

    a. Open the feedback loop (the [On] button will change its appearance to **Off**);

    b. Define the **SetPoint** to be less than **DFL** by 0.5;

    c. Close the feedback loop (the [Off] button will change its appearance to **On**);

    d. Gradually increase **SetPoint** and monitor Z-scanner extension in the indicator located in the Main Operations panel (Fig. 13-23). For the **SetPoint**, enter a value at which extension of the Z-scanner stops.

Fig. 13-23. Scanner extension indicator

3. Define operating value of the feedback gain (**Gain** parameter) as follows:

    a. Double-click on the **Gain** field (Fig. 13-24).
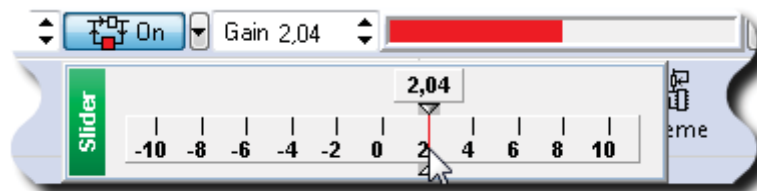
Fig. 13-24. Adjusting slider of the **Gain**

    b. Gradually decrease the **Gain** value and monitor level of the feedback input in the software oscilloscope.

    c. Find the **Gain** value at which generation starts. Generation originates when the fluctuating component of the input becomes noticeable.

    d. Define the working level of the **Gain** to be 0.5÷0.7 of the gain when oscillations start.

4. Open the **Litho** window (Fig. 13-25), by clicking the [Litho] button in the Main Operations panel.
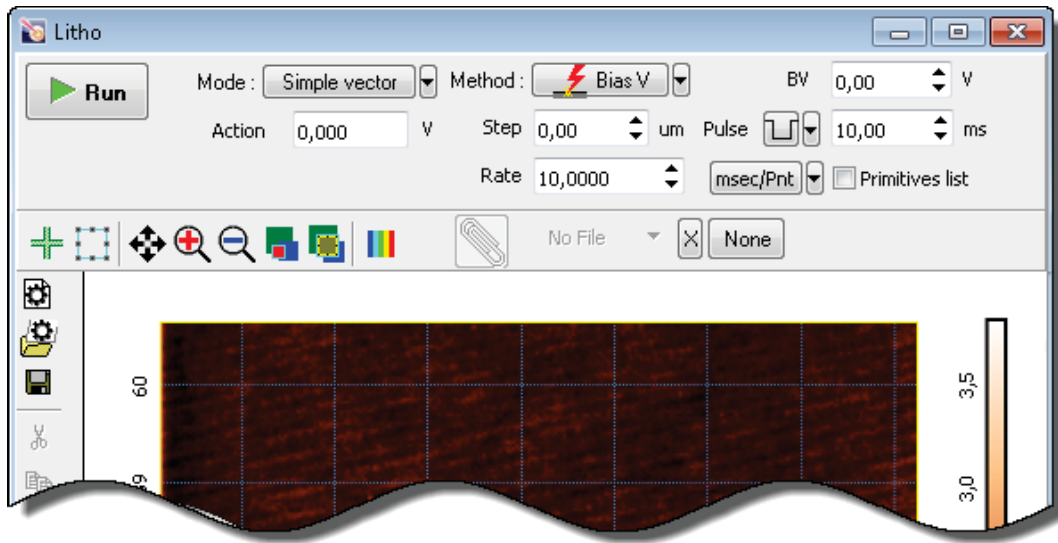
Fig. 13-25. **Litho** window

5. For Force lithography, select **SetPoint** in the **Method** drop-down list (Fig. 13-26).
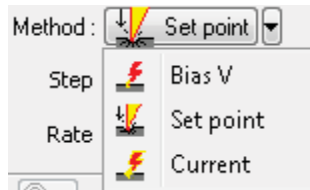
Fig. 13-26. Selecting the lithography method

6. In the **Mode** drop-down list, select a desired option of raster lithography (Fig. 13-27). Two options of raster lithography are available:

- **Raster** – simple raster lithography;
- **PulseRaster** – pulse-raster lithography.
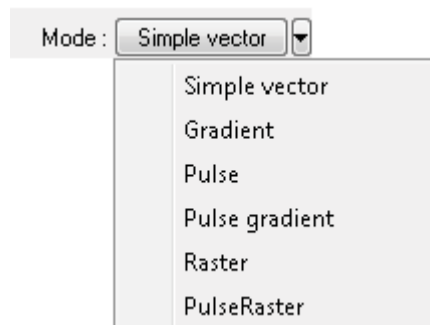
Fig. 13-27. Selecting the lithography mode

13.2.3.2. Loading Template

The template can be loaded from an external file of raster format. We recommend to use images of maximum contrast.

To load a lithography template, click the [...] button in the Control panel of the **Litho** button (see Fig. 13-25). This will open the standard **Open** dialog. In this window, select the desired file and click the **Open** button. The template image will appear in the **Litho** window (Fig. 13-28).
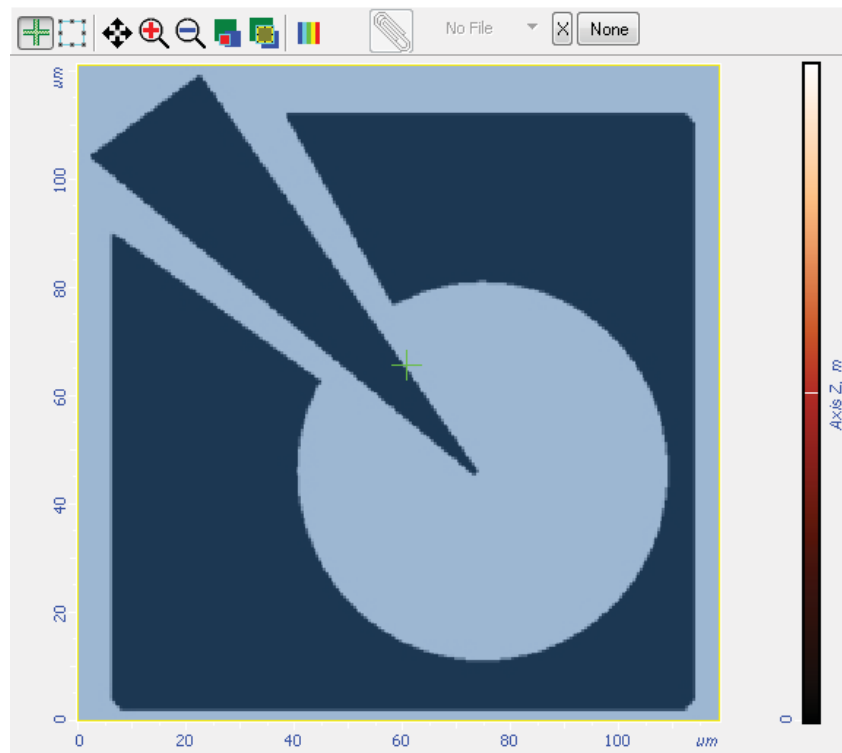
Fig. 13-28. Template for raster lithography is loaded

13.2.3.3. Adjusting Lithography Parameters

Number of parameters to define depends on the type of raster lithography (for details on raster lithography refer to i. 13.1.2 "Raster lithography" on page 168).

1. In the **Action 1** and **Action 2** of the Control panel of Litho window, define the range within which the impact of the tip on the sample will vary. Enter the lower and the upper limits of the range in the fields **Action 1** and **Action 2**, respectively (Fig. 13-29).

   The **Action 1** is associated with the lower limit (black) of the color scale while the **Action 2** is associated with the upper level (white).

   Range of pressures corresponding to the range of the **Action** parameter can be estimated with the algorithm explained in i. 13.2.1.1 "Estimation of the Probe Pressure on the Sample" on p. 170.

Fig. 13-29. Simple raster lithography Control panel
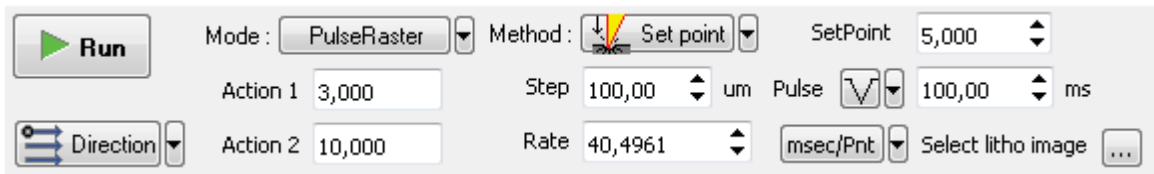
Fig. 13-30. Pulse raster lithography Control panel

2. In the **Rate** field, define rate of drawing lithography features (speed at which the tip travels along the sample surface).

    Lithography rate influences both on impact on the sample and on accuracy at which the template will be copied. The lower is the rate, the more intense will be impact and more accurate will be copying.

3. In the **Direction** drop-down list, select direction of performing lithography and the starting point of lithography process.
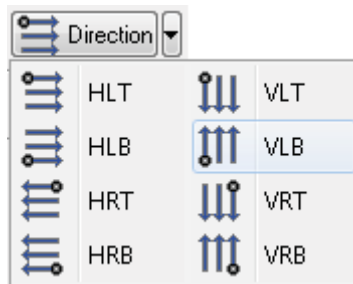
Fig. 13-31. Selecting direction of performing lithography

4. In the **Step** field, define the distance between points of lithography (step value between pulses at pulse-raster lithography).

5. For pulse-raster lithography, define also the following parameters:

    *   In the **SetPoint** field (Fig. 13-29) – the impact level the probe tip presses the sample during motion to the beginning of the sample and between pulses.

    *   In the drop-down list **Pulse**, select the desired pulse shape (Fig. 13-32).

    *   In the **Pulse** field define duration of a pulse.
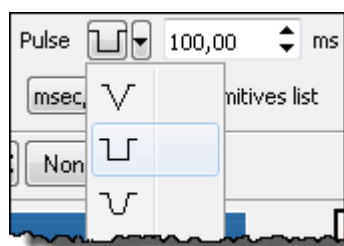
Fig. 13-32. Pulse shape

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13.2.3.4. Performing Lithography

When the lithography template is prepared and the lithography parameters are adjusted, the operator can start the lithography process.

To start lithography, click the [▶ Run] button in the Control panel of the **Litho** window.

This will change appearance of the **Run** button to [■ Stop]. Below the **Stop** button, the progress bar will appear to display percentage of the procedure progress in blue.

When the procedure completes, the **Stop** button returns to its initial appearance.

The lithography process can be stopped by clicking the **Stop** button or by pressing the <Esc> key. This will stop the process immediately. Next clicking the **Run** button would start the lithography procedure from the very beginning, i.e. from the first template point.

Lithography Results

Take a scan of the sample to acquire an image of the surface region exposed to lithography.

Fig. 13-33 shows results of raster lithography. A CD fragment was used as the sample. Lithography parameters were **Action 1** = 4 nA, **Action 2** = 0.8 nA; the probe was of NSG30 model.
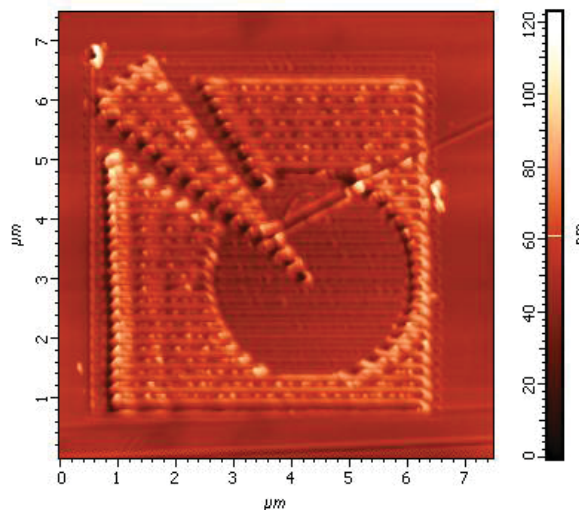
Fig. 13-33. Image of the sample exposed to lithography

13.3. Electrical Lithography

There are two sorts of electrical lithography:

- **Charge lithography**. The probe-sample voltage is maintained constant during the lithography process;

- **Current lithography**. The current through the sample is maintained constant during the lithography process.

Charge lithography

Charge lithography is based on effects of electrical field at the contact between the probe tip and the sample surface. When a sufficiently large voltage is applied between the tip and the sample, exposure of electrical field can locally modify the surface. A number of modification mechanisms are possible: local heating due to high current density; in a gas environment, deposition of decomposition products that results from decomposing the gas in the region of strong field close to the probe tip; electric-field evaporation of the sample material or deposition of the tip material evaporated under strong electric field.

Current lithography

Current lithography operates with the sample-probe current maintained at a constant level. During the lithography process, the feedback system maintains the predefined current by controlling the applied electrical bias.

This technique uses the fact that electrochemical processes resulting to surface modification of the sample depends, eventually, not on the applied voltage but on the probe-sample current. Thus, controlling the current will provide much more stable results because it is less sensitive to local properties of the sample and to quality of the sample-probe contact.

When a sufficiently large voltage is applied between the tip and the sample, exposure of electrical field can locally modify the surface. A number of modification mechanisms are possible: local heating due to high current density; in a gas environment, deposition of decomposition products that results from decomposing the gas in the region of strong field close to the probe tip; electric-field evaporation of the sample material or deposition of the tip material evaporated under strong electric field.

Requirements on the sample and on the probe

The sample must be a conductor or semiconductor. A substrate with the conducting contact shall be used to mount the sample in order to enable the application of voltage between the probe and the sample.

For electrical lithography probes with conducting coating shall be used. Typically, semicontact probes with the coating of TiN, W_2C and conducting diamond-like coating (DCP) are used.

When carrying out electrical lithography, there are two major options of sample surface modification:

- changing sample topography;

- modifying physical properties of the sample surface without alternating the topography.

Electrical lithography can be performed in the vector or in the raster mode.

This Chapter deals with:

1. Performing vector electrical lithography (see i. 13.3.1 on p. 187);

2. Performing raster electrical lithography (see i. 13.3.2 on p. 195).

13.3.1. Electrical Vector Lithography

ATTENTION! Before performing raster lithography, learn i. 13.1.1 "Vector Lithography" on p. 164.

Basic operations of electrical vector lithography are:

1. Preliminary Scanning and Selecting Lithography Region (see page 187);

2. Selecting the lithography method (see page 189);

3. Creating Lithography Template (see page 191);

4. Adjusting Lithography Parameters (see page 192).

These operations are explained in details below.

13.3.1.1. *Preliminary Scanning and Selecting Lithography Region*

Before performing the electrical lithography, take a testing scan in a semicontact mode over the maximum scanning area.

NOTE. The preliminary scan can be taken with a contact technique as well, but this can result in noticeable degradation of the conductive layer of the probe tip. This will lower exposure level to the sample and expand the area under the tip undergoing exposure.

In the acquired scan, select a region for lithography. Take a scan of the selected region by Kelvin Probe Microscopy (see i. 9.2 on p. 113).

Selecting the lithography region for charge lithography

Charge lithography will be explained with a GaAs sample and a probe of NSG01/TiN model. Charge lithography will cause accumulation of electrical charge in the subsurface layer under the exposing tip.

Preliminary scanning provides the surface topography and the surface potential distribution of the region selected for topography (Fig. 13-34).
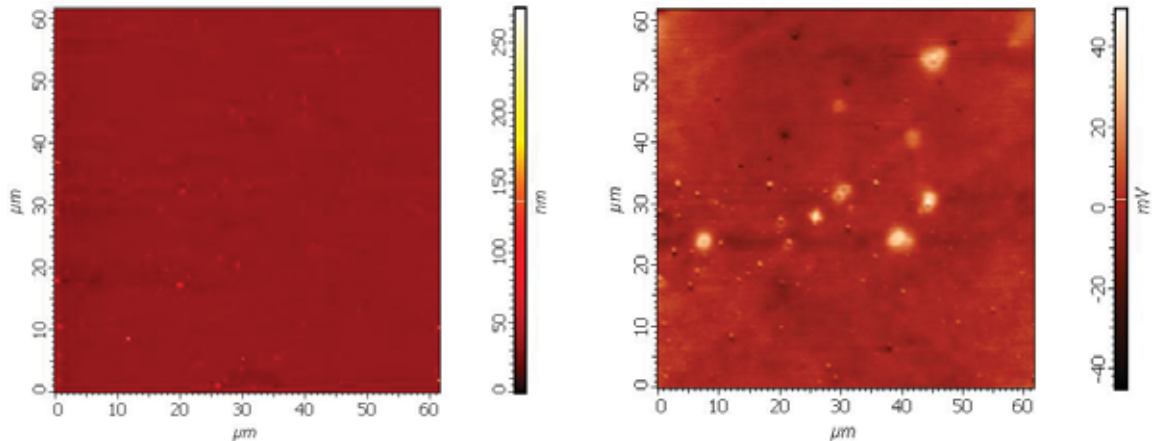
Fig. 13-34. Sample image before lithography
left – topography; right – surface potential distribution

Selecting the lithography region for current lithography

Current lithography will be explained with a octadecyltriclorsylanum sample and a probe of DCP11 model. Current lithography will result in changing surface properties of the sample monolayer. The surface portions exposed to voltage will transform from hydrophobic to hydrophilic, thus providing modulation of friction and contrast for Lateral Force Microscopy.

Preliminary scanning provides the lateral force distribution (**LF** signal) of the region selected for topography.
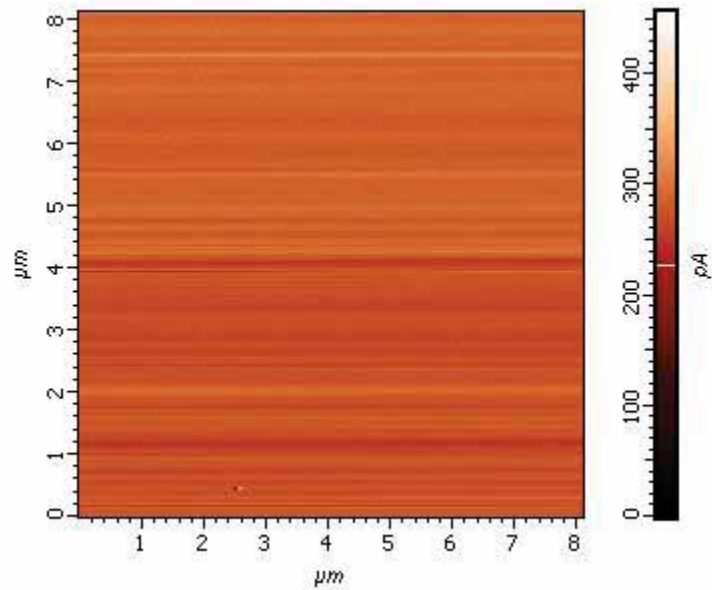
Fig. 13-35. Image of the lateral force distribution

13.3.1.2. Selecting the lithography method

To select the lithography method, perform the following steps:

1. In the controller configuration drop-down list of the Nova PX program, select **Semicontact** (Fig. 13-36).
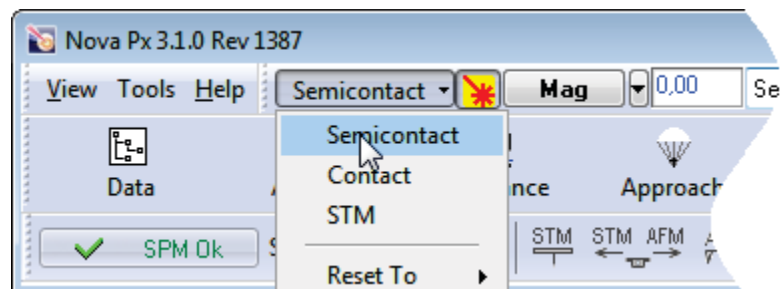
Fig. 13-36. Selecting the controller configuration

2. Open the **Litho** window (Fig. 13-37) by clicking the button.
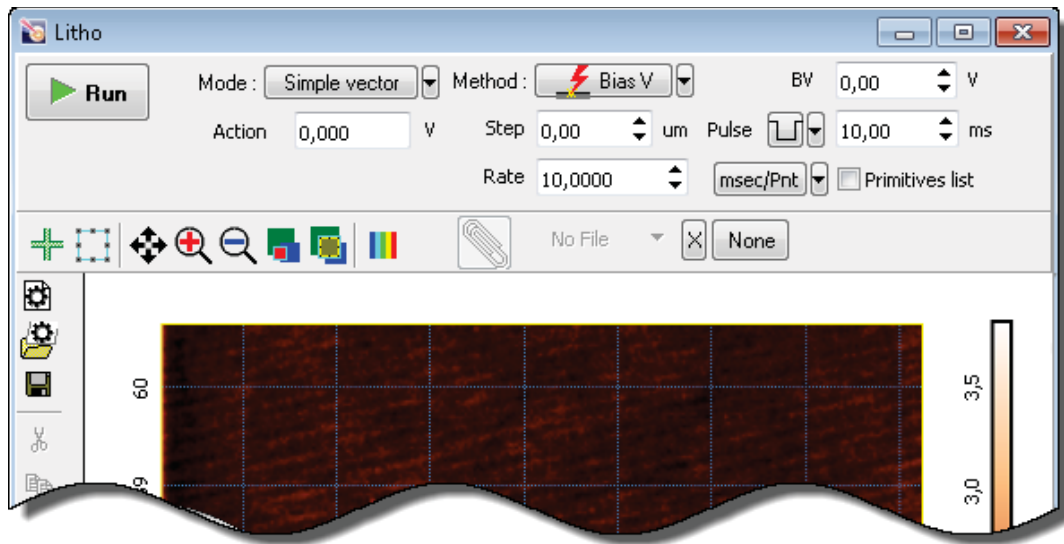
Fig. 13-37. **Litho** window

3. In the drop-down list, select the desired mode of lithography (Fig. 13-38):
   - For charge lithography, select **Bias V**;
   - For current lithography, select **Current**.
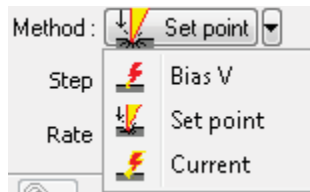
Fig. 13-38. Selecting the lithography method

NOTE. Appearance of the Control panel of the Lithography window depends on the selected mode of vector lithography.

4. In the **Mode** drop-down list, select the desired mode of vector lithography (Fig. 13-39):
   - **Simple vector** – simple vector lithography. The force applied to the sample is constant during drawing objects;
   - **Gradient** – gradient lithography. The force applied to the sample ramps linearly during drawing objects;
   - **Pulse** – pulse lithography. The sample is exposed to a series of force pulses of the same amplitude during drawing objects;
   - **Pulse gradient** – pulse-gradient lithography. Combines two lithography modes, the pulse and the gradient ones. The sample is exposed to a series of force pulses of amplitudes varying in a predefined range during drawing objects.

NOTE. For details on vector lithography modes, refer to i. 13.1.1 "Vector Lithography".
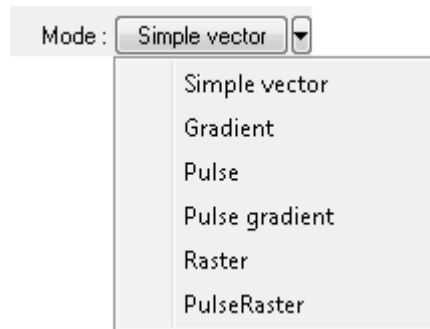
Fig. 13-39. Selecting the lithography mode

13.3.1.3. Creating Lithography Template

1. Create a new lithography template. To this purpose, click the ⚙ button in the Toolbar of the Lithography template (Fig. 13-40). This will remove all objects created before. This step is required to prevent any object outside the selected region to appear in the final template.

2. In the Lithography template viewing area, define positions and sizes of the desired objects with the tools of the Toolbar.
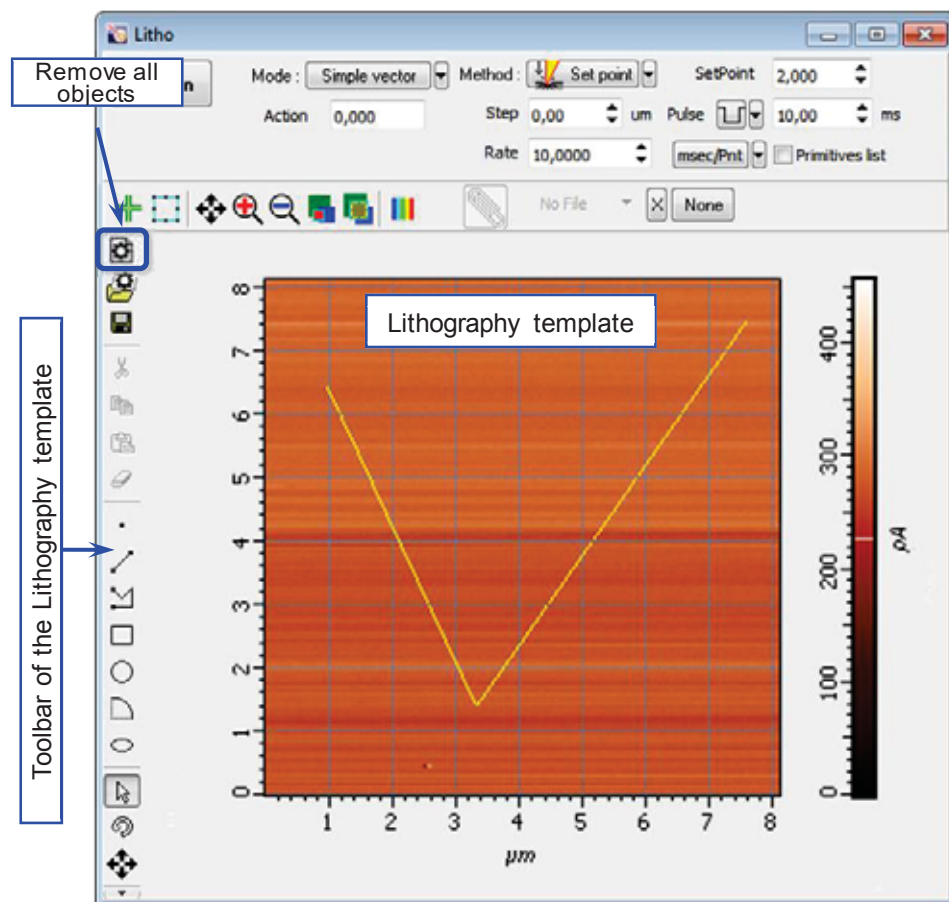
Fig. 13-40. Example of the lithography template

Parameter table of the lithography template

By default, all lithography elements are attributed with parameters defined in the Control panel of the **Litho** window (Fig. 13-19). If desired, the parameters can be adjusted individually for particular points. Their values can be edited in the Parameter table of the lithography template (Fig. 13-41) opened with the **Primitives list** option selected.

	Action 1	Action 2	Rate	Step	Pulse
line 5	6,985		10,000	0,000	10,000
line 14	6,985		10,000	0,000	10,000
rect 20	6,985		10,000	0,000	10,000
circle 59	6,985		10,000	0,000	10,000
arc 67	6,985		10,000	0,000	10,000

Save Load   Reset to defaults

Measure None   Select Scripts

Fig. 13-41. Parameter table of the lithography template

Adding a new element to the lithography template extends the table by a row в containing the name of the element and its lithography parameters. Desired values of the parameters can be entered in corresponding cells of the table. For synchronized modification of parameters of several elements, mark their names with the mouse with the <Ctrl> key pressed. Then changes of an element in the marked group will spread to all members of the group.

13.3.1.4. Adjusting Lithography Parameters

Composition of the set of adjustable parameters depends on the selected mode of vector lithography (for details on the vector lithography modes, refer to i. 13.1.1 "Vector Lithography").

1. Decrease the level **SetPoint** window by a factor of 2÷10 relatively to the **SetPoint** level used for scanning.

2. Define the exposure level to be applied to the sample during drawing the template objects:

   - **For simple lithography and pulse lithography.** In the **Action** field of the lithography Control panel, define the exposure level to be applied to the sample during drawing the template objects.

   - **For gradient lithography and pulse gradient lithography.** Define the lower boundary of the range in the **Action 1** field and the higher boundary in the **Action 2** field.

Sign of the applied voltage influences significantly results of lithography. For example, at large negative $(< - 8\text{ V})$ voltage applied to the probe tip (**Action** parameter), lithography can result not only in surface charging but also in local oxidation.
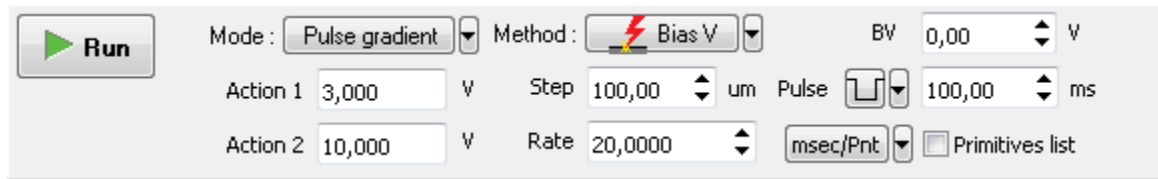
Fig. 13-42. Lithography Control panel. Select the pulse gradient charge lithography

3. Define the probe-sample bias during motion of the sample between the elements of the lithography template (**BV** – for charge lithography, **BV_SP** – for current lithography). Usually this parameter is set to 0.

4. In the **Rate** field, define the lithography rate (the speed of moving the probe relative to the sample surface).

   The lithography rate influences on results of the sample exposure to lithography. The lower is the rate, the deeper is the exposure and the better is the correspondence between the template and the resulting surface.

   NOTE. The parameters Step and Pulse are irrelevant for the simple vector lithography and gradient vector lithography.

For pulse and pulse-raster lithography, define parameters of pulses:

1. In the **Step** field, define the time step between the pulses.

2. In the **Pulse** drop-down list, select the desired pulse shape.

3. In the **Pulse** field, define duration of a pulse.
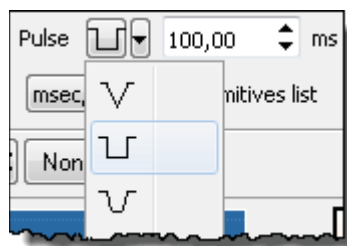
Fig. 13-43. Pulse shape

13.3.1.5. Performing Lithography

When the lithography template is prepared and the lithography parameters are adjusted, the operator can start the lithography process.

Operations of the electrical vector lithography procedures are the same for all modes.

To start lithography, click the [▶ Run] button in the Control panel of the **Litho** window.

This will change appearance of the **Run** button to [■ Stop]. Below the **Stop** button, the progress bar will appear to display percentage of the procedure progress in blue.

When the procedure completes, the **Stop** button returns to its initial appearance.

The lithography process can be stopped by clicking the **Stop** button or by pressing the <Esc> key. This will stop the prcess immediately. Next clicking the **Run** button would start the lithography procedure from the very beginning, i.e. from the first template point.

Lithography Results

After the lithography finish take a scan of the sample by Kelvin Probe Microscopy. Results of electrical lithography can be assessed with the surface potential distribution. For details on scanning, refer to i. 9.2 "Kelvin Probe Microscopy" on p. 113.

Results of charge lithography

Fig. 13-44 shows results of the Simple vector lithography applied to the GaAs sample with **BV** = 0 V, **Action** = 10 V by the NSG01/TiN probe.
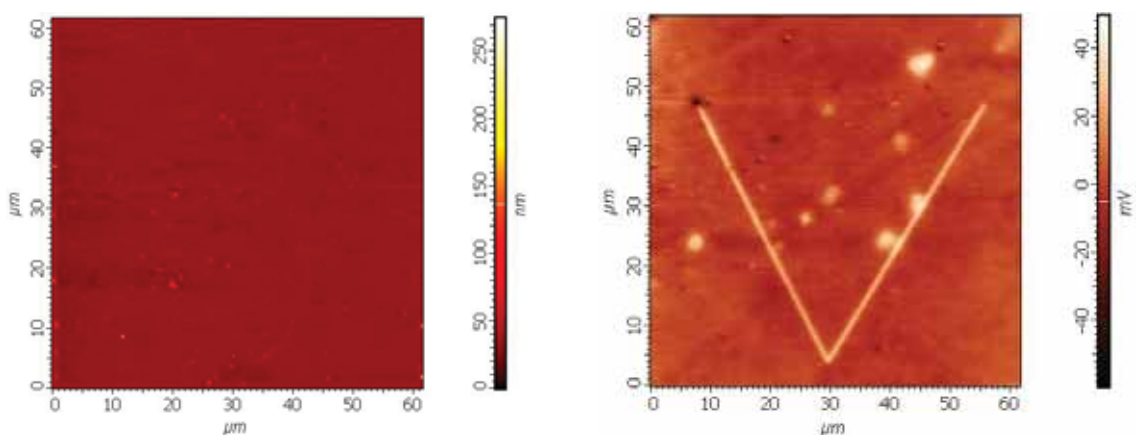
Fig. 13-44. Sample surface after lithography
left – topography; right – surface potential distribution

Results of current lithography

Fig. 13-45 shows results of the Simple vector lithography applied to the octadecyltrichliorsylanum with **Action** = 0.3 nA by the DCP 11 probe.
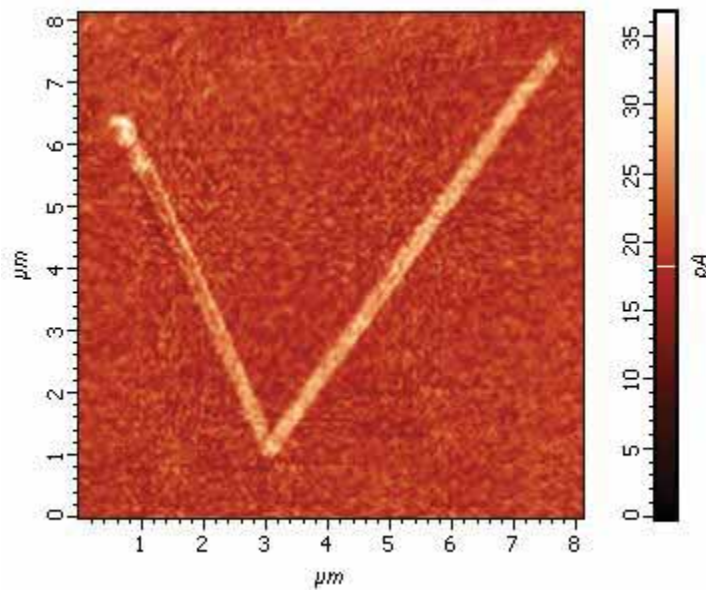
Fig. 13-45. Surface potential distribution after lithography

13.3.2. Electrical Raster lithography

⚠ **ATTENTION! Before performing raster lithography, learn i. 13.1 "Overview" on p. 164.**

Basic operations of force raster lithography are:

1. Preliminary Scanning and Selecting Lithography Region (see i. 13.3.2.1 on p. 196);

2. Selecting the lithography method (see i. 13.3.2.2 on p. 197);

3. Loading Template (see i. 13.3.2.3 on p. 199);

4. Adjusting Lithography Parameters (see i. 13.3.2.4 on p. 199);

5. Performing Lithography (see i. 13.3.2.5 on p. 201).

These operations are explained in details below.

13.3.2.1. Preliminary Scanning and Selecting Lithography Region

Before performing the electrical lithography, take a testing scan in a semicontact mode over the maximum scanning area.

NOTE. The preliminary scan can be taken with a contact technique as well, but this can result in noticeable degradation of the conductive layer of the probe tip. This will lower exposure level to the sample and expand the area under the tip undergoing exposure.

In the acquired scan, select a region for lithography. Take a scan of the selected region by Kelvin Probe Microscopy (see i. 9.2 on p. 113).

Selecting the lithography regions for charge lithography

Charge lithography will be explained with a GaAs sample and a probe of NSG01/TiN model. Charge lithography will cause accumulation of electrical charge in the subsurface layer under the exposing tip.

Preliminary scanning provides the surface topography and the surface potential distribution of the region selected for topography (Fig. 13-46).
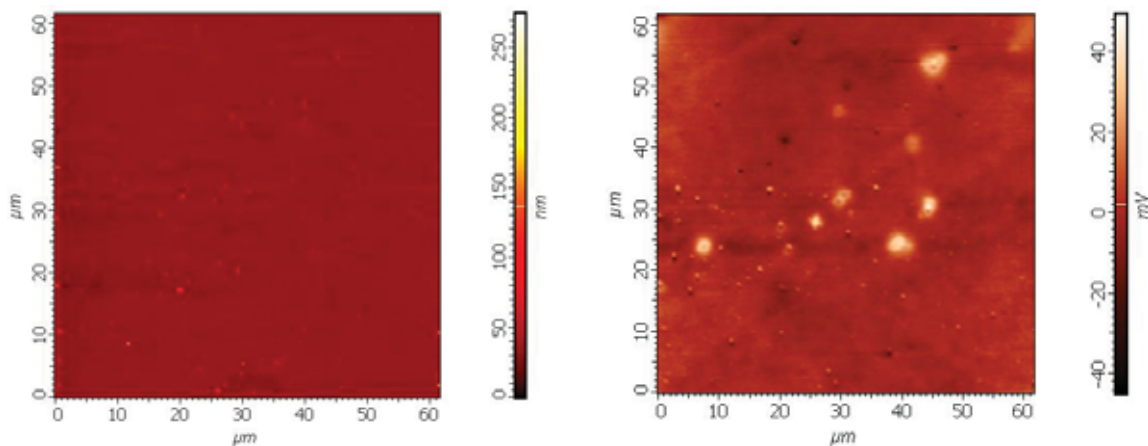
Fig. 13-46. Sample surface of GaAs before lithography
left – topography; right – surface potential distribution

Selecting the lithography region for current lithography

Current lithography will be explained with a octadecyltriclorsylanum sample and a probe of DCP11 model. Current lithography will result in changing surface properties of the sample monolayer. The surface portions exposed to voltage will transform from hydrophobic to hydrophilic, thus providing modulation of friction and contrast for Lateral Force Microscopy.

Preliminary scanning provides the lateral force distribution (**LF** signal) of the selected region.

Fig. 13-47. Image of the lateral force distribution

13.3.2.2. *Selecting the lithography method*

1. In the controller configuration drop-down list, select **Semicontact** (Fig. 13-48).
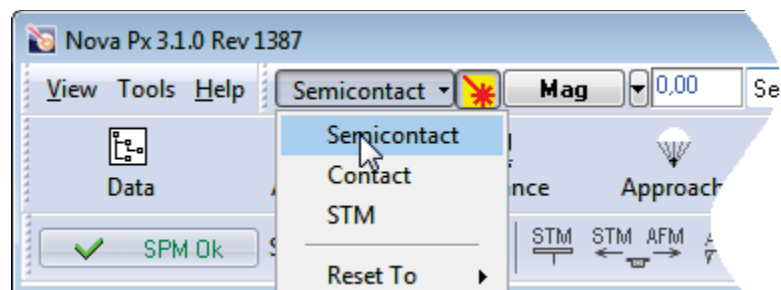
Fig. 13-48. Selecting controller configuration

2. Open the **Litho** window by clicking the [Litho] button (Fig. 13-49).
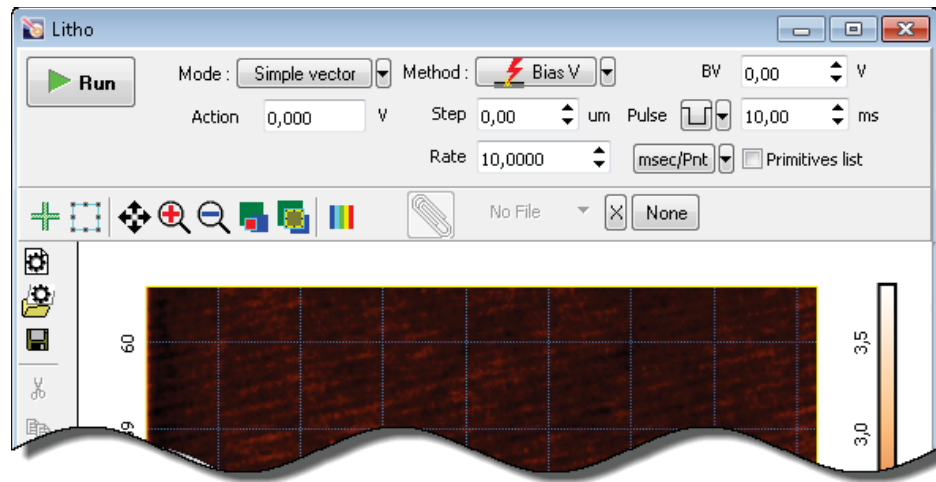
Fig. 13-49. **Litho** window

3. In the drop-down list **Method,** select the desired mode of lithography (Fig. 13-50):
   - For charge lithography, select – **Bias V**;
   - For current lithography, select – **Current**.
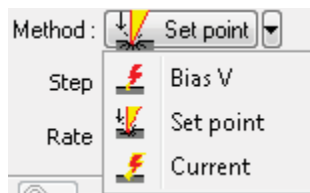
Fig. 13-50. Selecting the lithography method

4. In the **Mode** drop-down list select the mode of raster lithography (Fig. 13-51):
   - **Raster** – for simple raster lithography;
   - **PulseRaster** – for pulse raster lithography.
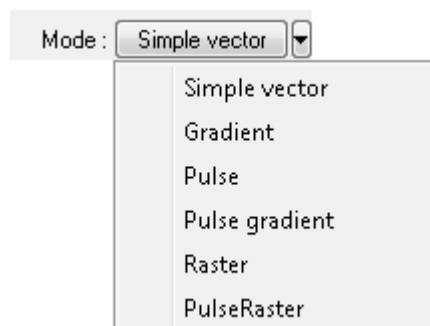
Fig. 13-51. Selecting the lithography mode

13.3.2.3. Loading Template

The template can be loaded from an external file of raster format. We recommend to use images of maximum contrast.

To load a lithography template, click the **Select Litho Image...** button in the Control panel of the **Litho** button (see Fig. 13-49). This will open the standard **Open** dialog. In this window, select the desired file and click the **Open** button. The template image will appear in the **Litho** window (Fig. 13-52).
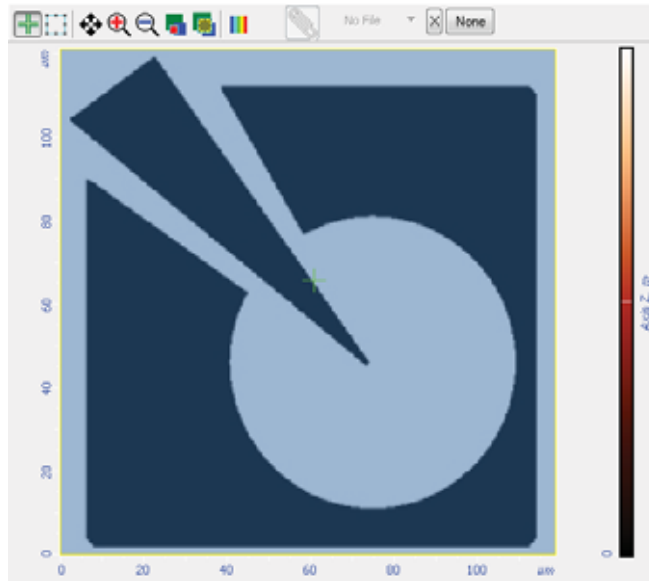
Fig. 13-52. Template for raster lithography is loaded

13.3.2.4. Adjusting Lithography Parameters

Number of parameters to define depends on the type of raster lithography (for details on raster lithography refer to i. 13.1.2 on page 168).

1. Decrease the level **SetPoint** window by a factor of 2÷10 relatively to the **SetPoint** level used for scanning.

2. In the Lithography Control panel, define the range of varying the force applied to the sample. Define the lower boundary of the range in the **Action 1** field and the higher boundary in the **Action 2** field.

   Value of Action 1 corresponds to the bottom of the color scale (black), while value of Action 2 is linked to the top of the color scale (white).

   Sign of the applied voltage influences significantly results of lithography. For example, at large negative (< -8 V) voltage applied to the probe tip (**Action** parameter), lithography can result not only in surface charging but also in local oxidation.
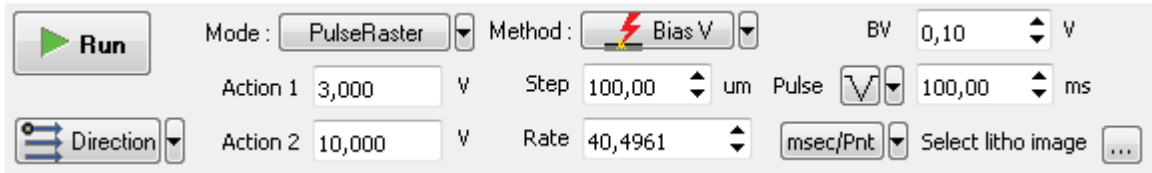
Fig. 13-53. Control panel for Charge pulse raster lithography

3. In the **Rate** field, define the lithography rate (the speed of moving the probe relative to the sample surface).

   The lithography rate influences on results of the sample exposure to lithography. The lower is the rate, the deeper is the exposure and the better is the correspondence between the template and the resulting surface.

4. In the **Step** input field, define:

   - for simple raster lithography – distance between points of the raster;

   - for pulse-raster lithography – step between pulses.

5. In the **Direction** drop-down list, select the direction for lithography and position of the starting point.
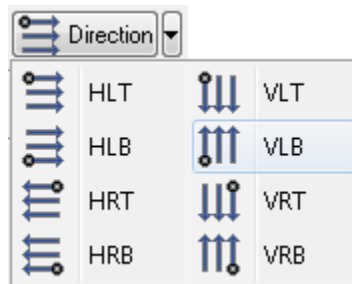
Fig. 13-54. Direction for lithography

6. For pulse-raster lithography, define also the following parameters:

   - Define the probe-sample bias during motion of the sample between the elements of the lithography template (**BV** – for charge lithography, **BV_SP** – for current lithography). Usually this parameter is set to 0.
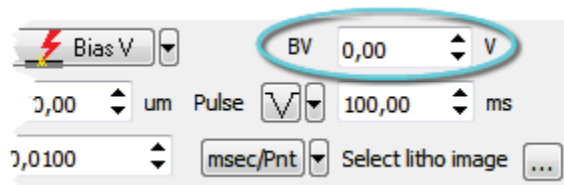
Fig. 13-55

   - In the **Pulse** drop-down list, select the desired pulse shape.

   - In the **Pulse** field, define duration of a pulse.
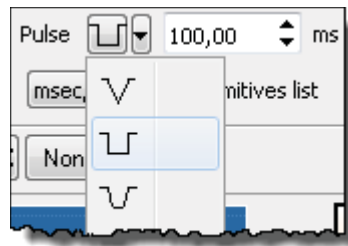
Fig. 13-56. Pulse shape

13.3.2.5. Performing Lithography

When the lithography template is prepared and the lithography parameters are adjusted, the operator can start the lithography process.

Operations of the electrical vector lithography procedures are the same for all modes.

To start lithography, click the [▶ Run] button in the Control panel of the **Litho** window.

This will change appearance of the **Run** button to [■ Stop]. Below the **Stop** button, the progress bar will appear to display percentage of the procedure progress in blue.

When the procedure completes, the **Stop** button returns to its initial appearance.

The lithography process can be stopped by clicking the **Stop** button or by pressing the <Esc> key. This will stop the process immediately. Next clicking the **Run** button would start the lithography procedure from the very beginning, i.e. from the first template point.

Lithography Results

After the lithography finish take a scan of the sample by Kelvin Probe Microscopy. Results of electrical lithography can be assessed with the surface potential distribution.

Fig. 13-57 shows results of the current raster lithography applied to the octadecyltrichliorsylanum sample with **Action 1** = 0.3 nA, **Action 2** = 0 nA, by the DCP11 probe.
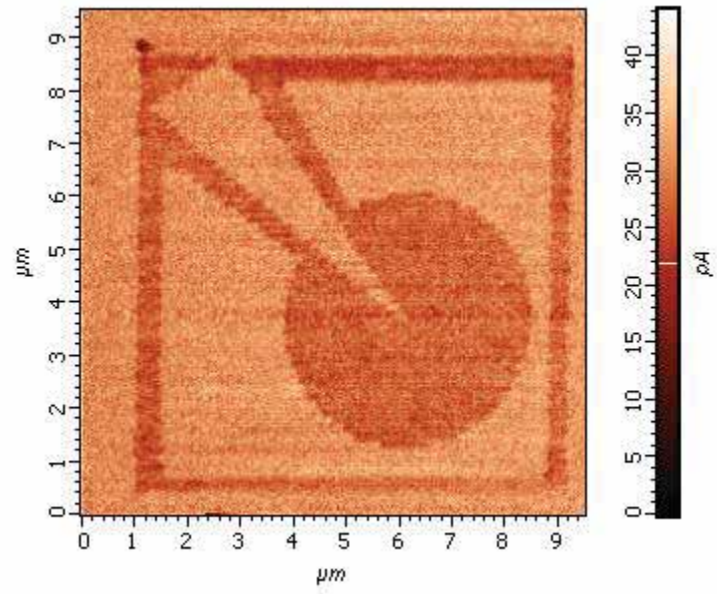
Fig. 13-57. Distribution of the surface potential after current lithography

Appendices

1. Scanners Calibration and Maintenance

A scanner is the next critical part of the Scanning Probe Microscope after the probe. It is usually manufactured from piezoceramic tubes and contains X-, Y- and Z-sections that, with voltage applied, provide probe and/or sample displacement along the corresponding axes (directions). Among the most important parameters of the scanner is its accuracy. A current value of the voltage applied to a section of the scanner, to some extent, can be used as an indicator of the scanner displacement along the respective axis. However there is no direct correspondence between the voltage and the displacement since piezoceramic materials feature large creep and hysteresis.

There are software procedures to correct for the spurious effects resulting from these properties of piezoceramics, but usually it is not much one can gain from using them. Any nonperiodic displacement of the probe in the XY-plane (e.g. while performing vector lithography) cannot be accomplished without noticeable errors. Probe displacement along the Z-axis is performed using probe-sample interaction feedback to track sample topography, but it cannot be measured without significant distortions. Acceptable accuracy can only be achieved when measuring lateral dimensions of objects during a simple sample surface scan.

The metrological performance of the Probe Microscope can be greatly improved by using scanner displacement sensors. The accuracy of measurement in many important cases is increased by an order of magnitude. Nonlinearity of the scanner displacements and drift in the relative position of the probe and the sample decrease significantly. Precise operations like vector lithography and nano scale manipulations become possible. Sample topography is displayed without noticeable distortions on the Z-axis (it has to be admitted though, that during scans of large sample surfaces some bow can be observed in the scan image.) The ability to control the scanner displacement not only along the XY-plane but also Z-axis allows the measurement results to be significantly improved when using two-pass techniques and conducting spectroscopy measurements (F(z)).

In our instruments capacitance displacement sensors are used. One plate of the sensor plane capacitor is secured on the scanner free end while the position of the second plate is fixed. As the scanner moves the gap between the capacitor plates changes, with the output of electronic circuit connected to the capacitor generating voltage inversely proportional to the value of capacitor. Thus, neglecting the edge effect and nonparallelism of the capacitor plates, the output voltage can be considered proportional to the gap, i.e. to the scanner displacement along the corresponding axis.

Capacitance displacement sensor by itself is a fairly precise instrument for measuring displacements. Sensor manufacturers claim typical linearity error (with some constraints on the conditions of use) of 0.01 % from nominal range of measurements. However when integrating sensors in a specific positioning system a problem inevitably arises due to imperfect bond between the probe location point and the position of the movable sensor plate. Spurious sensor plate rotations or additional small displacements of the probe or the sample (e.g. caused be thermal drift) can arise, which are not registered by the sensor. All

this often leads to noticeable degradation of the system performance compared to one that could have been expected from using the sensors. Therefore the values of scanner linearity errors stated by SPM manufacturers are typically not less than 0.1%.

When using probes with sensors one should recall that apart from feedback on Z - axis (**FB**) controlling the interacting force between the probe and the sample there is also X- and Y- axes feedback (**XY Closed-Loop**). When scanning by probe the parameters of this feedback (similar to **FB** Z) usually need not be adjusted. However, when scanning by sample the mass of the latter together with the holder can vary in a large range. With the gain of the feedback loop set, the system becomes more unstable as the sample mass increases, finally resulting in excitation of the feedback system. A procedure for adjusting the feedback gain is described below.

1.1. Scanner with Displacement Sensors: Principle of Operation

The use of capacitance sensors allows the scanner displacement nonlinearity to be compensated for. Capacitance sensors are used both in measuring heads and in exchangeable scanners.

Fig. 1-1 shows a functional diagram of a scanner with X- and Y-sensors. The sensor signal is compared with a value defined in the program during a scan, and if there is a mismatch the differential signal is amplified and applied to the corresponding section of the scanner producing the displacement which corresponds to the specified value. In this way the scanner moves in the XY-plane, the effects of creep and hysteresis being compensated for.
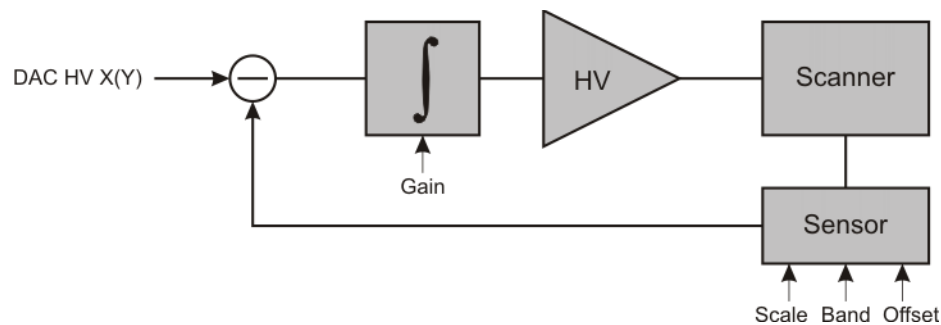
Fig. 1-1. Functional diagram of a scanner
with capacitance sensors (for X- and Y-sensors)

The Z-sensor signal is used for measuring topography and when applying techniques that require controlled scanner displacement on Z (e.g. spectroscopy, two-pass techniques etc.).

It should be noted that the use of displacement sensors can somewhat degrade the instrument resolution. This usually happens with the resolution on the XY-axes. The sensor has electronic self-generated noise, which is applied to the high-voltage amplifier through the feedback circuits, see Fig. 1-1, causing greater (compared to a scanner without sensors) scanner "jitter" along the given axis. The Z-sensor usually generates less noise because the scanner range on Z is several times smaller than that on XY, enabling the gap between the sensor plates to be decreased (the noise level is nearly a quadratic function of the gap when the plates area is constant).

1.2. Verification and Removing Generation

If a sample with a weight exceeding 1 g, a heavy sample holder or a liquid cell is installed on the scanner with sensors then the absence of generation in the feedback loop controlling the scanner displacement in the XY-plane shall be verified. Furthermore, the absence of generation shall be checked if the quality of AFM-image during operation using the sensors is lower than that without sensors.

In order to check and remove generation complete the following procedures:

1. Open the displacement sensors adjustment window by selecting the **Tools → Sensors** menu command.
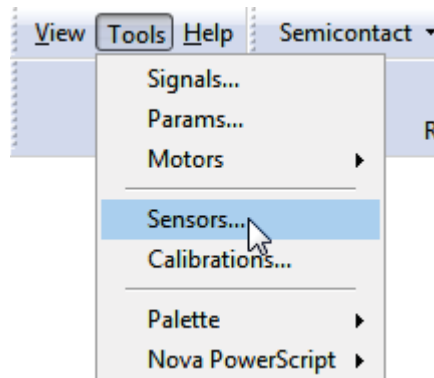
Fig. 1-2
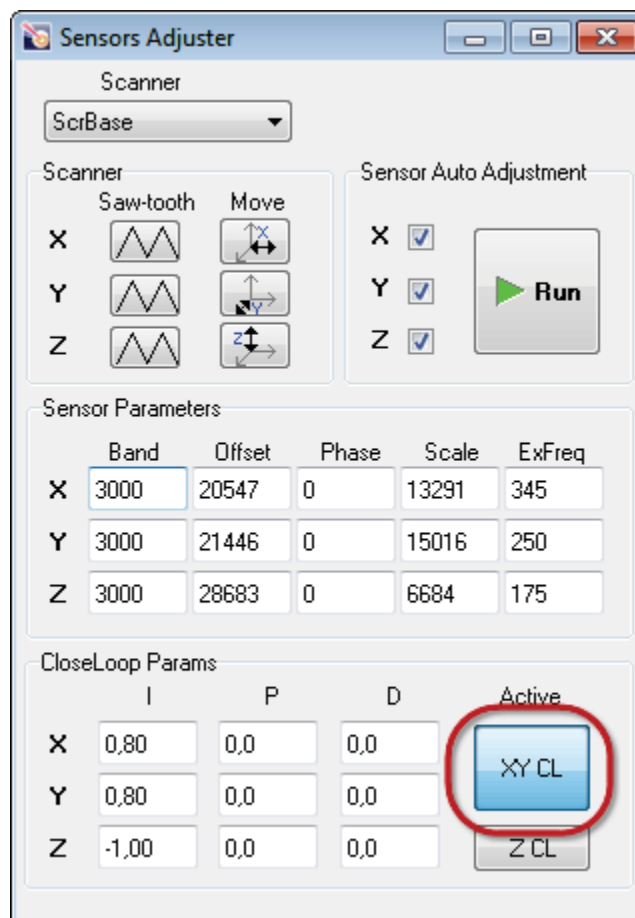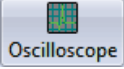
Fig. 1-3. Displacement sensors adjustment window

2. Close XY-feedback loop by clicking the **XY CL** button.

3. Position the probe in the lower left corner of the scan region. To do this:

    - click the [button] button and set the slider to zero;

    - click the [button] button and set the slider to zero.

4. In the oscilloscope window ([Oscilloscope] button) select first **Sens X** (Fig. 1-4), and then **Sens Y** for the signal to be recorded. Check the RMS values of the **Sens X** and the **Sens Y** signals. The RMS values shall be about 0.0002 V.
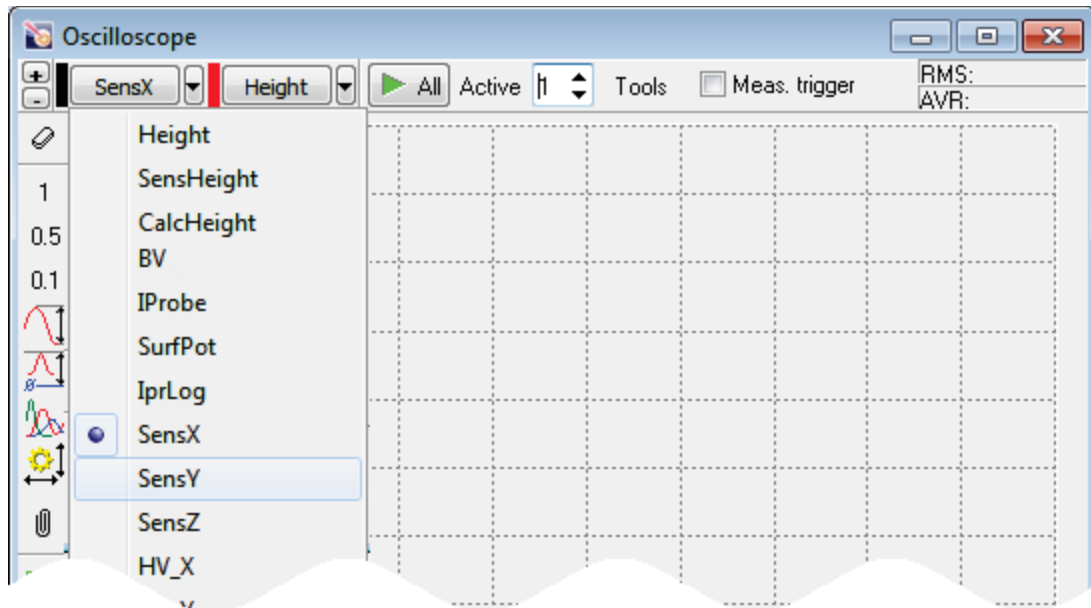
Fig. 1-4

5. If the RMS values are considerably greater than 0.0002 V this indicates the presence of generation in the XY-feedback loop. Verify values of the parameters **Band X** and **Band Y**. Typical values of **Band X** and **Band Y** at which generation is absent are 3000÷4000.

6. Correct values of **Band X** and **Band Y**, if necessary, and then check the instrument for generation. If generation occurs, decrease the integral loop gains **X** and **Y** (Fig. 1-5).

    *NOTE. The **X** and **Y** gains should be decreased by the same value. Test the instrument for generation when both gains are adjusted.*
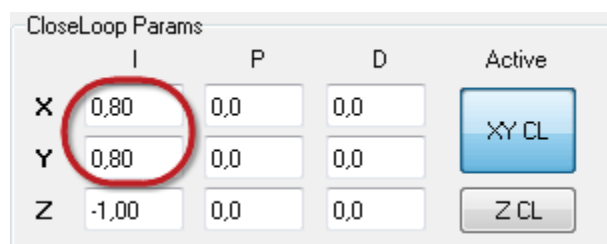
Fig. 1-5
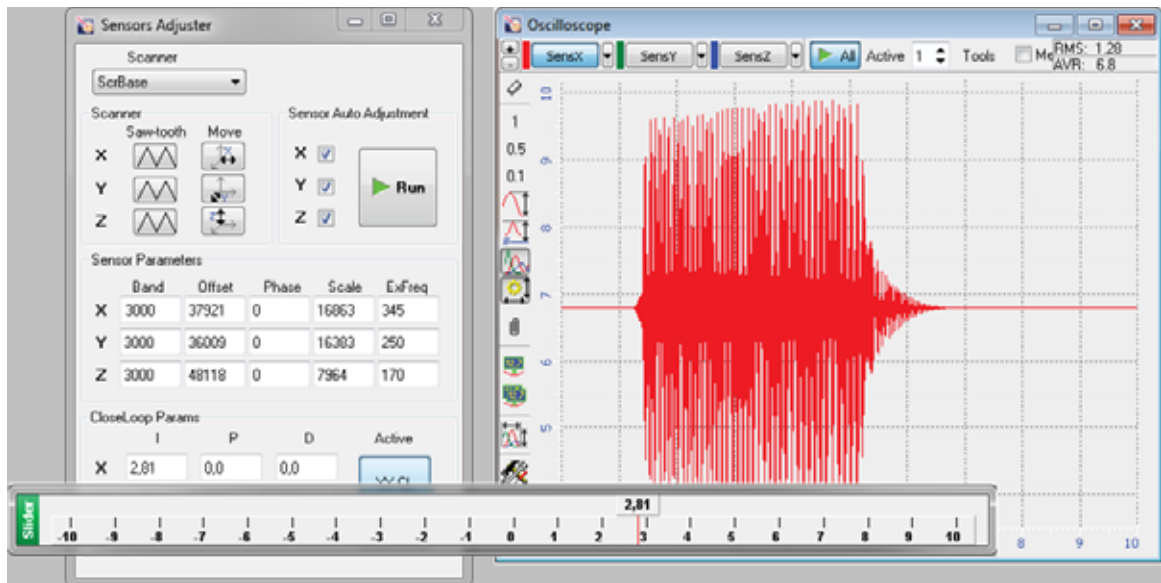
Fig. 1-6. Removing generation

7. Defined the operation levels of the **X** and **Y** gains at levels of 0.7 of values at which oscillation of the signals **SensX** and **SensY**, respectively, starts.

1.3. Maintenance

For scanners with displacement sensors, monthly verification of sensors adjustment is recommended. If any of the sensors appears is out of proper adjustment, perform the following steps:

- perform the procedure on adjusting this sensor;
- recalibrate the scanner on directions that ere readjusted.

⚠️ **ATTENTION! Before verifying the settings and adjusting the sensors the scanner shall be allowed for 6-8 hours in inoperative state, i.e. when no voltage is applied to the scanner piezotubes.**

Besides, the scanner with sensors should be recalibrated every six months similarly to the scanner without sensors.

1.3.1. Verification of the Displacement Sensors Adjustments

ATTENTION! The sensor verification procedure shall only be performed with the probe withdrawn from the sample surface.

Complete the following procedures to verify the adjustment of the sensors:

1. Start the control program. Power on the SPM controller.

2. Open the feedback loop (⊞ Off).

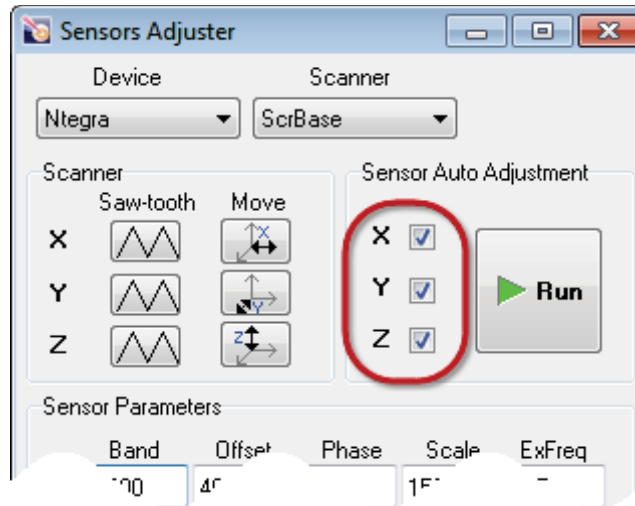3. Open the displacement sensors adjustment window by selecting the **Tools → Sensors** menu command.
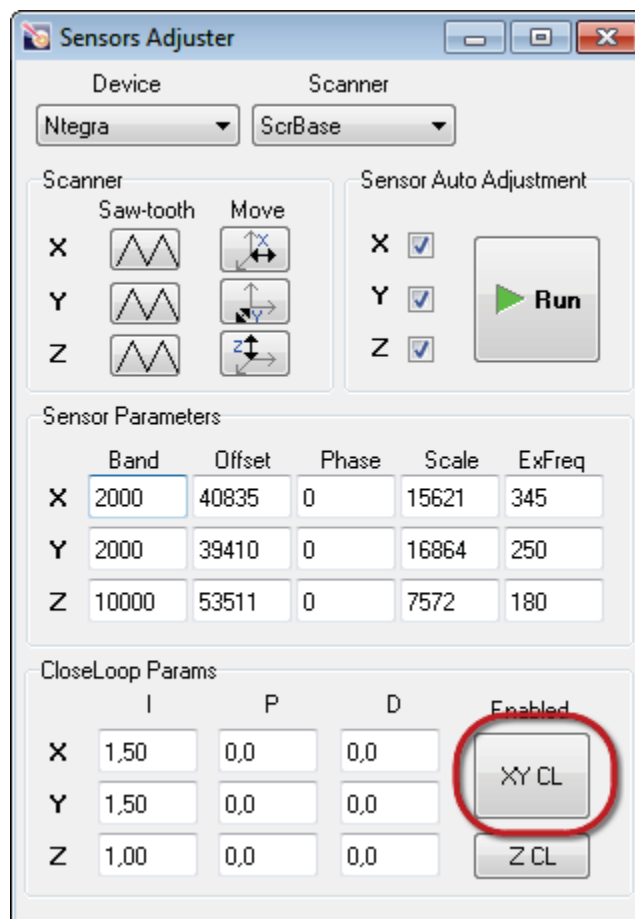
Fig. 1-7. Displacement sensors adjustment window

4. Open the XY-feedback loop (the **XY CL** button should be released).

5. Set the voltages applied to the X-, Y- and Z-sections of the piezoscanner to the middle of their range. This will cause the scanner to move approximately to the center of the scan range for the corresponding axes. To do this:

- click the **Move X** ⟨button icon⟩ button and using the slider which appears set the scanner position on the X-axis in the middle of the scan range (Fig. 1-8);
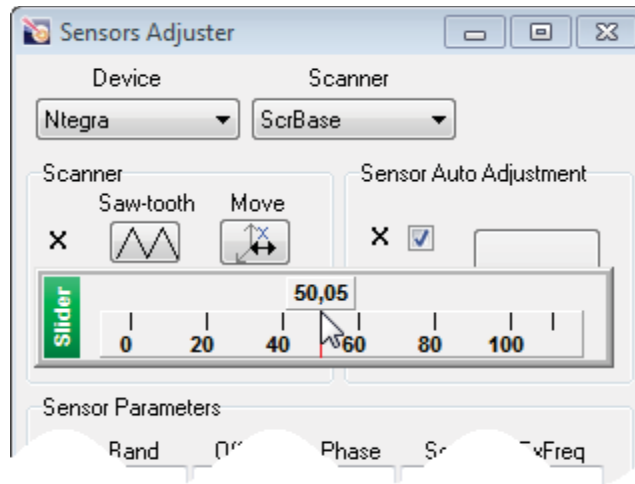
Fig. 1-8

- click the **Move Y** button ⟨icon⟩ and using the slider which appears set the scanner position on the Y-axis in the middle of the scan range;

- click the Move Z button ⟨icon⟩ and using the slider which appears set the scanner position on the Z-axis in the middle of the scan range.

6. Verify the adjustment of the X-sensor. To do so, complete the following operations:

   a. Click **Saw-tooth** ⟨icon⟩ button. At this triangular waveform voltage with the peak-to-peak amplitude of $\pm\,300$ V is applied to the X-section of the scanner (Fig. 1-9). The scanner displacement resulting from the application of this voltage will be registered by the sensors.
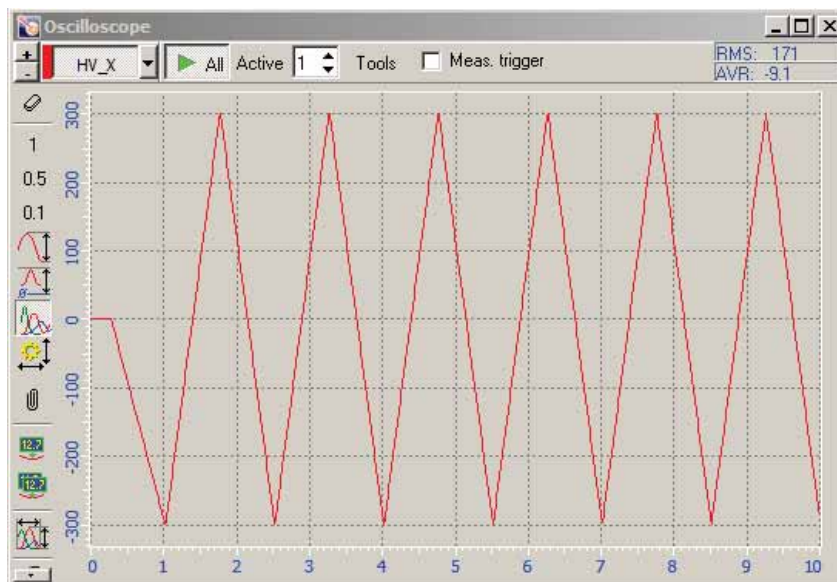
Fig. 1-9

   b. From the software oscilloscope select the **SensX** signal to be displayed.

○ If the signal peak-to-peak amplitude is not less 13 V and the signal average is in the range ±1 V (Fig. 1-10), adjustment of the X-sensor is not needed. The average value of the signal is shown in the **AVR** box of the panel for selecting the displayed signals.
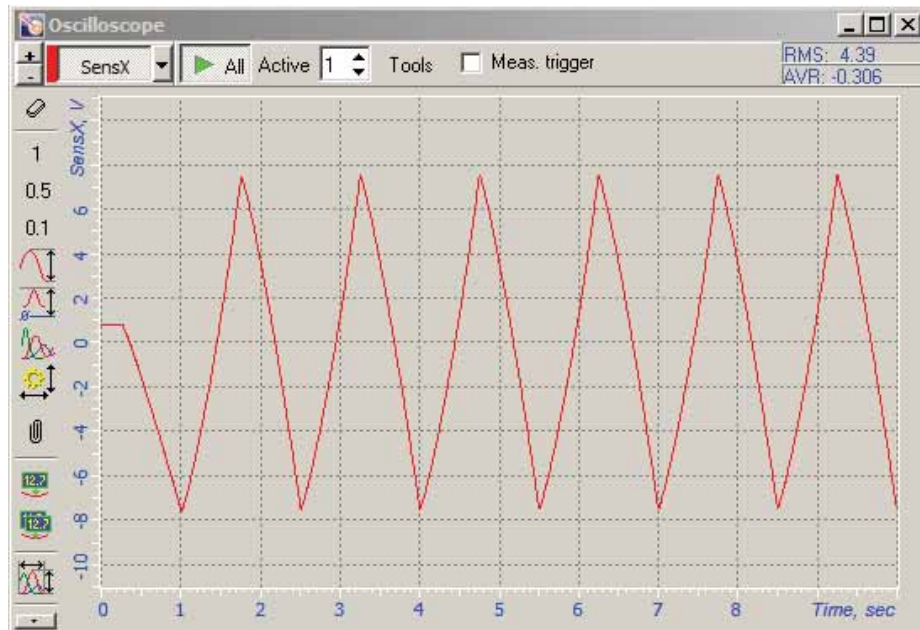
Fig. 1-10. Verification of the X-sensor adjustment.
The sensor does not require adjustment

○ If the signal peak-to-peak amplitude is not less 13 V and the signal average is outside the range ±1 V (Fig. 1-11), adjustment of the X-sensor is to be done (see i. 1.3.2 on page 211).
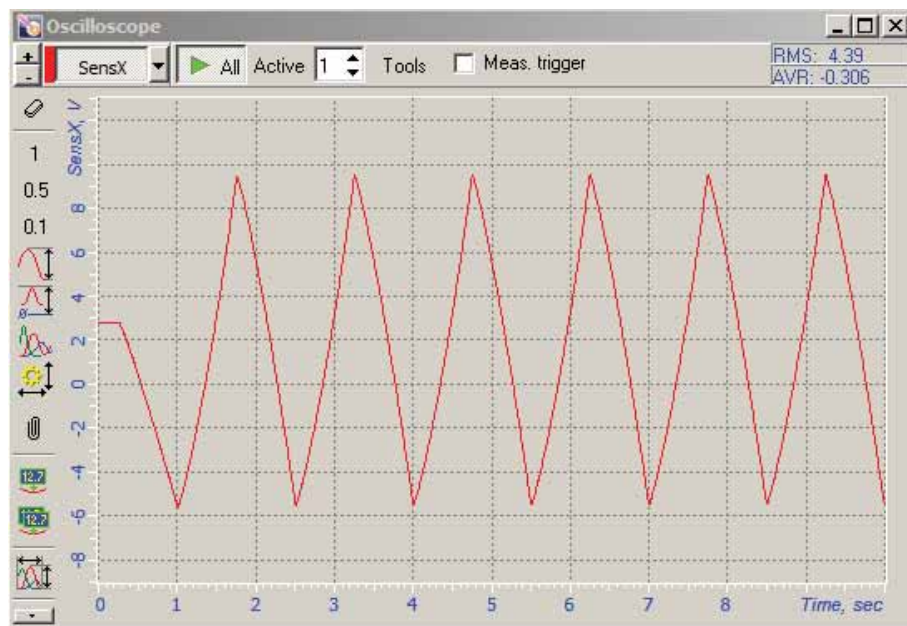
Fig. 1-11. Verification of the X-sensor adjustment.
The sensor requires adjustment

o If the signal peak-to-peak amplitude is less 13 V (Fig. 1-12), the procedures on adjustment of the X-sensor and calibration of X-scanner.
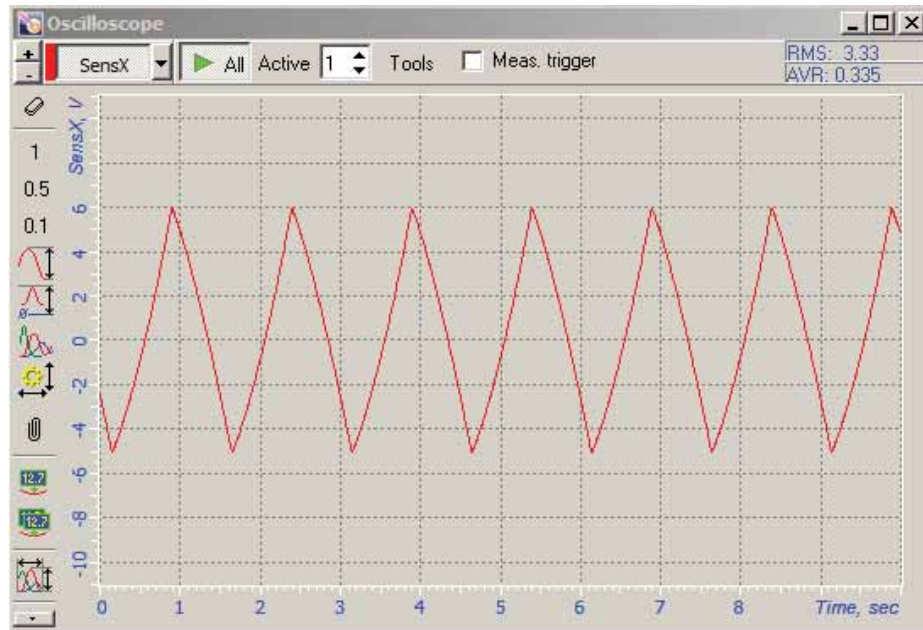
Fig. 1-12. Verification of the X-sensor adjustment.
The sensor adjustment and scanner calibration are required

7. In a similar way verify the adjustment of the Y-sensor and Z-sensor.

1.3.2. Adjusting the Displacement Sensors

The sensor adjustment procedure includes choosing **Offset** and **Scale** coefficients for each sensor (X-, Y-, Z-) so that the range of the recorded signal is: ±8 V. The coefficients are taken automatically after the adjustment procedure starts.

ATTENTION! The sensors adjustment should only be performed with the probe withdrawn from the sample surface.

To adjust the capacitance sensors, complete the following procedures:

1. Start the control program. Switch on the SPM controller.

2. Open the Z feedback loop ( ). Open the XY feedback loop (by releasing the XY button).

3. Open the displacement sensors adjustment window by selecting the **Tools → Sensors** menu command.

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Fig. 1-13. Displacement sensors adjustment window

4. In the **Sensor Auto Adjustment** panel (Fig. 1-13) mark the check boxes for the sensors to be adjusted.

5. Click the **Run** button.

   In the outcome of this:

   - instead of the **Run** button the **Stop** button appears;

   - saw-tooth voltage will be sequentially applied to the selected piezotubes of the scanner equivalent;

   - the scanner displacement in each direction produced by the application of the voltage is registered by the corresponding sensor;

   - the **Scale** and **Offset** coefficients will be adjusted for each sensor so that the output signal of each sensor varies in the range of ±8 V.

   After the adjustments are complete, the **Run** button will change to its original state.

   The sensors adjustment procedure takes no more than 1÷2 minutes.

6. Close the dialog by clicking ⬚. This will save the calibration parameters of the tested scanner in a corresponding ini-file.

1.3.3.   Calibration of Scanners

The scanners are calibrated at the factory before delivering the instrument. However, those calibrations may become invalid in the course of exploitation or transportation due to physical properties of piezo ceramics that forms working bodies of the scanners. That is why recalibration of the scanners is recommended one time per 6 months. With results of the recalibration procedure, the scanner calibration parameters should be modified if necessary.

When a scanner with sensors is used, both calibration and measurements with the sensors being involved in the feedback loop are recommended. If measurements will not use the sensors, calibration of the scanner with open sensors feedback loop should be performed. Other details of the calibration procedure are the same for scanners with and without sensors.

1.3.3.1. Calibration of the scanner along the XY-axes

For XY calibration of 100-µm scanners, the following test gratings can be used: TGQ01, one of TGZ series, or TDG01 with period (278±1) nm.

The TGQ01 grating provides calibration along both X and Y axes simultaneously.

If the scanner is to operate in the low-voltage mode, it should be calibrated in this mode using a test grating of TDG01 model.

Setting the sample height

When calibrating the scanner in X and Y directions, the collected image is affected by the height of the measured landscape above the scanner sample stage. This height includes thicknesses of the sample and the substrate.

At the same displacement of the scanner piezotube, the distance the sample moves in the XY plane relative to the probe tip increases with the sample height (Fig. 1-14). This results in magnification of the image size.

To avoid this type of image distortion, thickness of the sample should be regarded in the scanner calibration parameters.
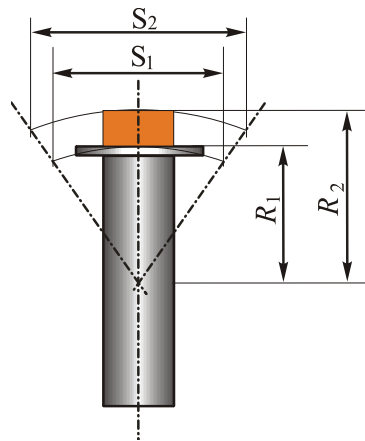
Fig. 1-14. Magnification of the image size with increasing thickness of the sample

For proper operation of the scanner, the sample height should be defined as height of the sample surface above the surface of the scanner sample stage, that is, the height should include thickness of the substrate (serving as the sample holder).

In order to set the sample height proceeds as follows:

a. Open the **ScanSettings** window by clicking the [Settings...] button (Fig. 1-15) of the scanning window.
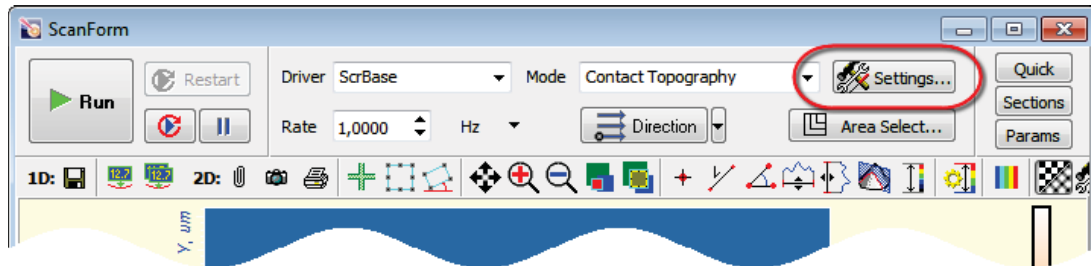
Fig. 1-15. **Settings** button

b. In the **Sample Height** entry field enter the corresponding value. Thickness is taken from the upper plane of the scanner.
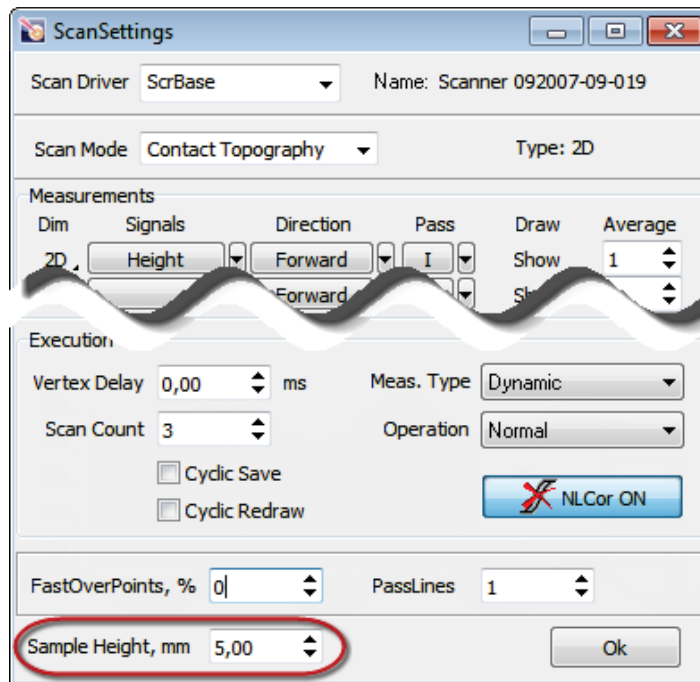
Fig. 1-16. Setting of the sample height

3. Click the **OK** button to apply the changes and closed the **ScanSettings** window.

Check of Scanner Calibration

Test gratings of TGQ01 model provide calibration along both X and Y axes simultaneously. With TGZ gratings, calibration along one of the directions (e.g., X) is performed first, then the grating is rotated by 90º and calibration along the other direction is performed.

The calibration procedure will be explained below using a TGQ01 test grating.

1. Take a scan of the test grating. The following conditions should be met:

   - the periodicity direction of the test grating should be oriented along the calibration direction with accuracy not worse several degrees.

   - for the scanner with sensors, the XY feedback loop should be closed (the 🔲 XY button is pressed in);

   - scanning goes over the maximum scan area.

2. Open the Image Analysis window by clicking 🔲 **Data** In the Frames Tree, select a frame to be used for verification.

3. Open the **Grating Calibration** window by double-clicking the **Calibration → Grating Calibration** item.
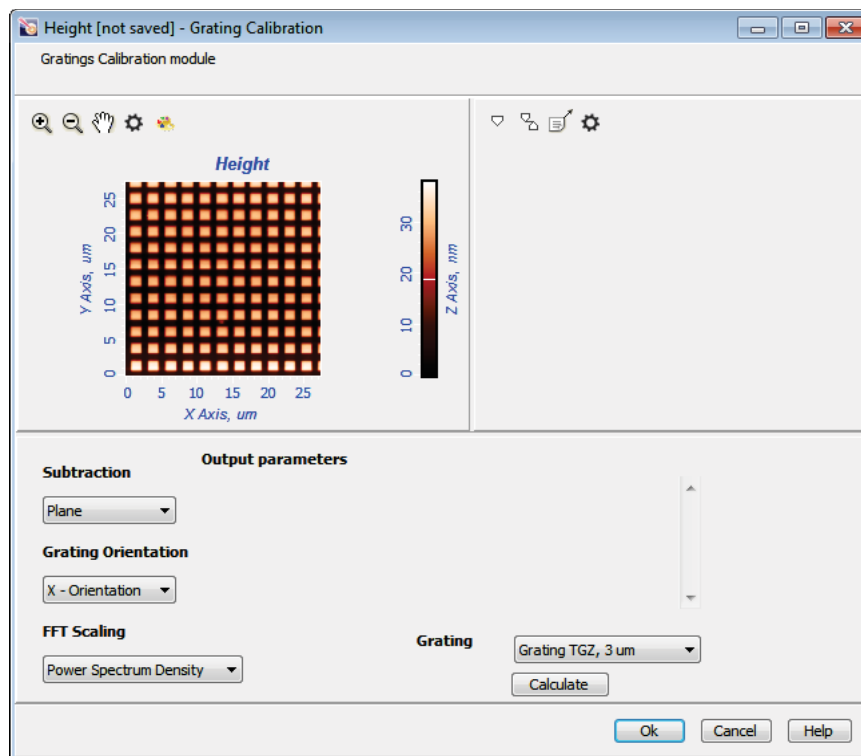
Fig. 1-17

NOTE. If size of the frame is less than 256×256 pixels, parameters of the grating will be calculated immediately.

4. Define calculation parameters for calibration:

   - in the **Subtraction** field – subtracting procedure;

   - in the **Grating Orientation** field, direction of calibration. For instance, determination of X period needs the **X-Orientation** option to be selected;

   - in the **FFT Scaling** field – **Power Spectrum Density**;

   - in the **Grating** field define the nominal period of the calibration grating. For the TGQ grating, the proper selection is **Grating TGZ, 3 um**.

5. Click the **Calculate** button. This will display a plot of power spectral density in X direction and calculate parameters of the grating for that direction which will be shown in the **Output parameters** panel.

Power Spectrum Density um*nm^2

Output parameters

Dominant peak position by FFT spectrum	2.478 um
Position Error	0.206 um
Dominant peak position by smoothed spectrum	2.406 um
Input x.scale	0.106 um
New x.scale	0.132 um
Scale ratio	1.246
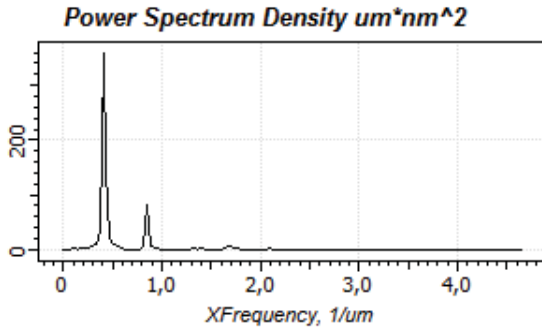
Fig. 1-18. Plot of power spectral density   Fig. 1-19. Calculated parameters of the grating

The **Input x.scale** line displays the current X calibration coefficient.

The **New x.scale** line provides the calibration coefficient calculated using the data of the scan.

6. Calculate Y calibration coefficients.
   For the TGQ01 grating, this needs to define **Y Orientation** in the **Grating Orientation** field and to start calculations by clicking the **Calculate** button.
   For gratings of TGZ and TDG01 models, before calculation another scan should be taken from the test grating rotated by 90º.

If the measured value of the grating period differs from the reference value by more than 3–5 %, the scanner calibration parameters are recommended to change.

In our example, the measured period is 2.406 μm against 3 μm by certificate. So, difference between the measured and the reference values is 25 %. This results from incorrect definitions of the calibration parameters that should be changed. Record the measured value of the grating period. The calibration parameters are edited in the **Scanner Calibrations Setup** window. The editing procedure is explained below.

Editing Scanner Calibration Parameters

The scanner calibration parameters are recommended to change if difference between the measured and reference values of the grating period exceeds 3–5 %. To change the scanner calibration parameters, perform the following steps:

1. In the Main menu select consecutively the following items: **Tools → Calibrations**. The **Calibrations** dialog box will appear on the screen (Fig. 1-20).

2. Verify that the scanner mode in the **Range** field is the same as that is used for calibration.
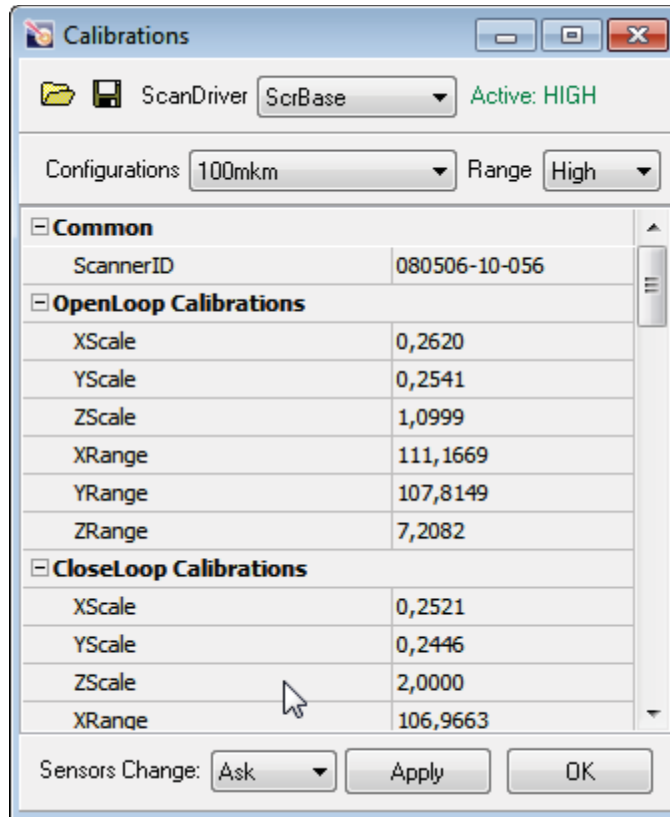
Fig. 1-20. **Calibrations** dialog box

3. Change values of the calibration parameters **XScale** and **YScale** by values given by the **Grating Calibration** method (parameters **New x.scale** and **New y.scale**).
For a scanner **with sensors**, coefficients in the **CloseLoop Calibration** group should be redefined.
For a scanner **without sensors**, coefficients in the **OpenLoop Calibration** group should be redefined.

4. Click the **OK** button to save your modifications and close the dialog. This will open a confirmation box with a question on modification of the calibration parameters of the displacement sensors. Select **No** to save calibration parameters of the sensors defined in the current session of the program.
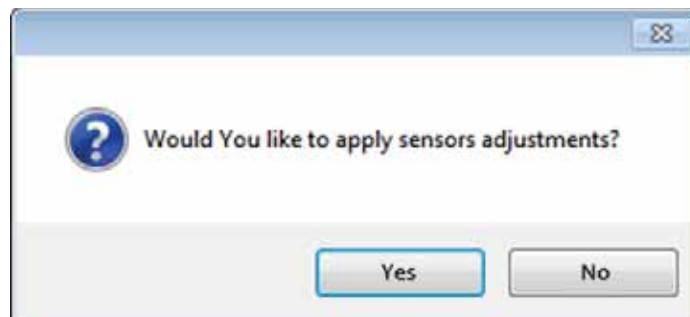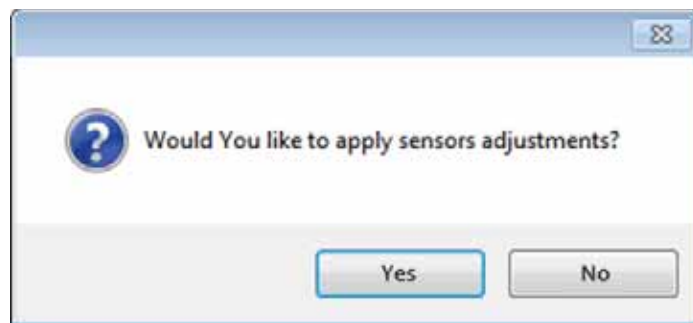

Fig. 1-21. Request on redefinition of the calibration parameters of the displacement sensors

1.3.3.2. Calibration of the scanner along the Z – axis

For Z calibration of 10-μm and 100-μm scanners, test gratings TGZ are recommended.

1- and 3-μm scanners, as well as 100-μm scanners in the low-voltage mode, do not need Z calibration.

Selection of a model of the TGZ grating should regard on roughness of the sample.

For highly smooth sample surfaces (roughness less 10 nm), the TGZ1 model is recommended for calibration. For surfaces of roughness higher 200 nm, the TGZ3 model is preferable for calibration.

Check of Scanner Calibration

Scanner calibration is verified with the **Z Calibration** module of the Image Analysis program. This module is described in detail in *Image Analysis. Reference Manual*.

Verification of Z scanner calibration will be explained for a TGZ test grating (period 3 μm, height 540 nm).

1. Take a scan of the test grating. The following conditions should be met:

   - Direction of fast scanning should be close to the periodicity direction of the grating;

   - The scan area should cover at least several periods of the grating. For correct processing of acquired data, numbers of scan points along each direction should be multiple of 2^n. Recommended size of the scan is at least 512×512;

   - for the scanner without sensors, **Height** should be selected as the recorded signal;

   - for the scanner with sensors, **Height** and **SensHeight** should be selected as recorded signals.

2. Open the Image Analysis window by clicking [Data].

3. Select a frame to be used for calibration (for example, **Height**).

4. Open the **Z Calibration** window by double-clicking the **Calibration → Z Calibration** item.
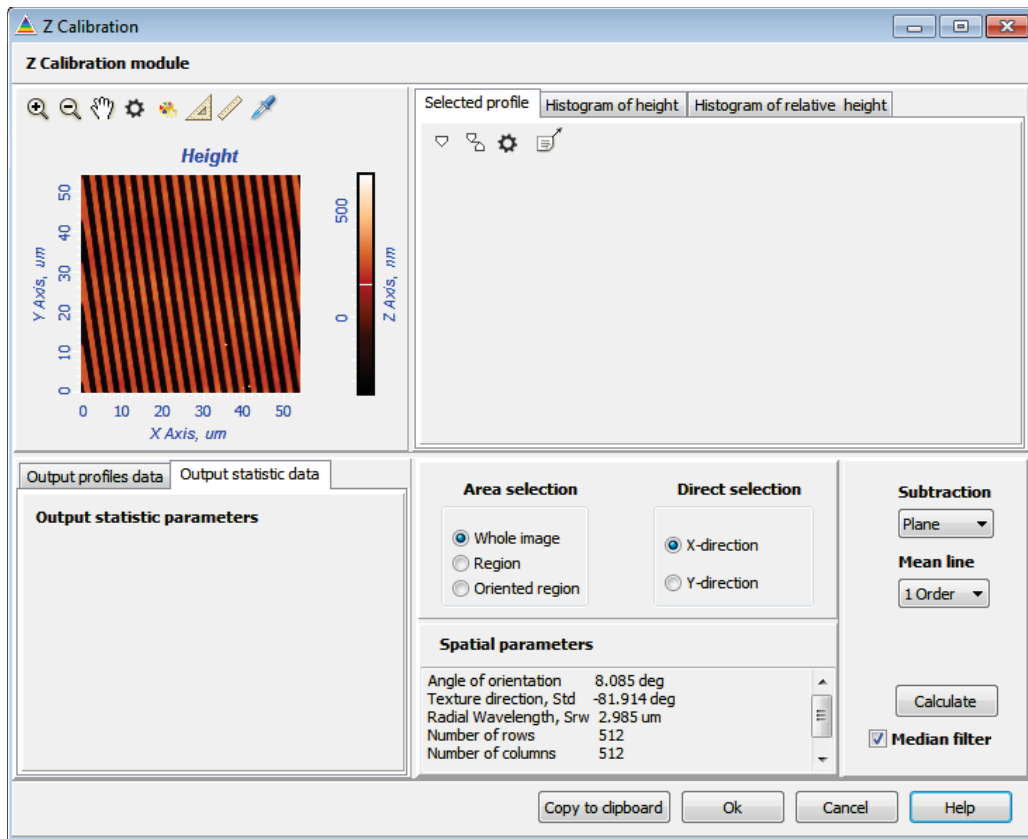
Fig. 1-22. Z Calibration window

5. Select an area to process by selecting a proper choice with the **Area selection** switch. If the grating is misoriented with respect to the XY basis (as in example shown in Fig. 1-22), the **Oriented Region** item should be selected. In this case, a square contour with sides oriented along the periodicity direction of the grating will appear.

6. Select a direction to take a profile for analysis by setting the **Direct selection** switch in proper position. For our case, **X-direction** is required.

7. In the **Subtraction** list, select a type of the surface to be subtracted from the original data before calculation.

8. Click the **Calculate** button. This will display plots of sectioning profiles and the histogram of profile heights and will calculate statistical parameters of the grating. When calculation completes, the **Calculate** button will become inactive.
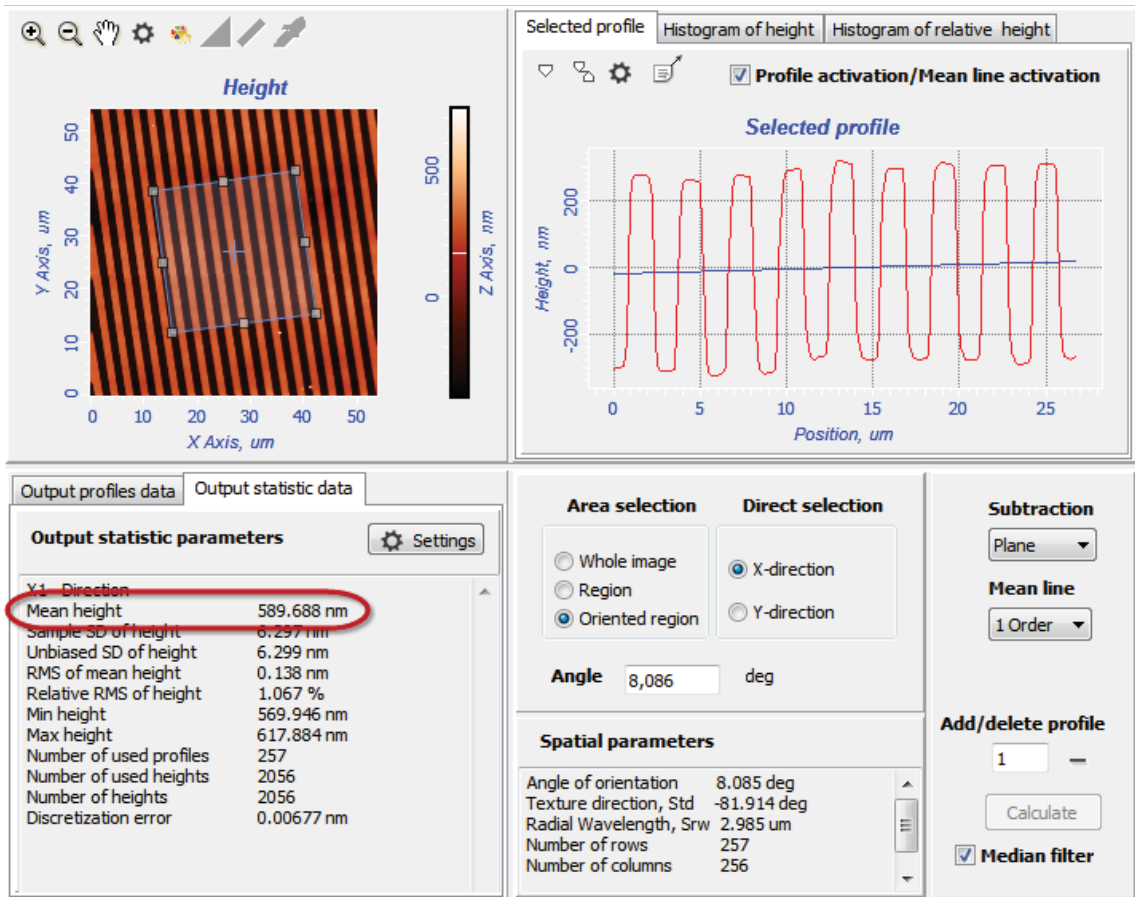
Fig. 1-23. Z Calibration window with results of calculation

9. Height of the grating periodic profile (**Mean Height**) is displayed in the **Output statistic data** tab (see Fig. 1-23).

Compare the **Mean Height** value with the reference one. If they differ by more than 3–5 %, redefinition of the scanner calibration parameters is recommended.

In our example, the calculated profile height (**Mean Height**) is 589.688 nm. It differs from the reference by 9 %. So, values of the calibration parameters are incorrect and should be redefined. Modification of the calibration parameters uses the **Scanner Calibrations Setup** window and will be explained below.

10. In a similar way, estimate the profile height using data given by the SensHeight signal.

Editing Scanner Calibration Parameters

The scanner calibration parameters are recommended to change if difference between the measured and reference values of the grating period exceeds 3-5 %. To change the scanner calibration parameters, perform the following steps:

1. Select the **Tools** → **Calibrations** menu command. This will open the **Calibrations** dialog (Fig. 1-24).
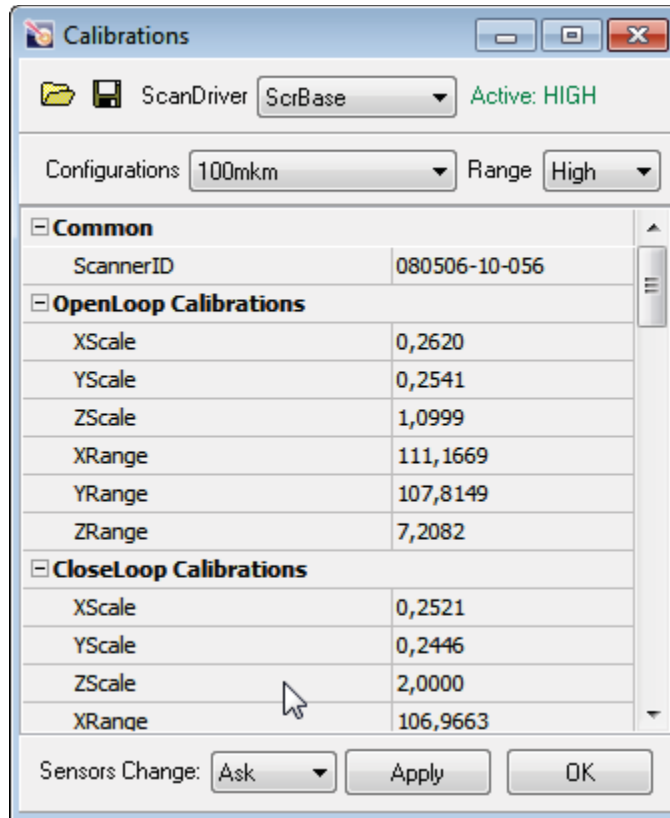
Fig. 1-24. Calibrations dialog

2. Verify that the scanner mode in the Range field is the same as that is used for calibration.

3. Calculate value of the calibration parameter by the following relation:

$ZScale_{new} = ZScale_{old} \times r' / r,$

where:

$ZScale_{new}$ is a corrected value of the calibration parameter;

$ZScale_{old}$ is the previous value of the calibration parameter (**ZScale**);

r' is the nominal height of the grating;

r is the measured value of the grating (**Mean Height**).

In our case, the corrected value of the **ZScale** parameter in the **OpenLoop** group is:

$ZScale_{new} = 1.0999 \times (540/589.688) = 1.0072.$

4. Redefine the **ZScale** parameter by its corrected value.

   For calibration of the **Height** signal, a corresponding coefficient in the **OpenLoop Calibration** group is to change.

   For calibration of the **SensHeight** signal, a corresponding coefficient in the **CloseLoop Calibration** group is to change.

5. Click the **OK** button to save your modifications and close the dialog. This will open a confirmation box with a question on modification of the calibration parameters of the displacement sensors (Fig. 1-25). Select **No** to save calibration parameters of the sensors defined in the current session of the program.

Fig. 1-25. Request on redefinition of the calibration parameters
of the displacement sensors

2. Installing Transport Brackets

⚠️ **ATTENTION! Please, ensure that all leading screws of carriages of the scanner and of the videomicroscope are fully retracted and the scanner is at its lowest position.**

Preparing SOLVER NEXT for installing brackets

For retracting the leading screws of carriages of the scanner and of the videomicroscope, perform the following steps with the Nova Px program:

1. Open the **Camera** window (Fig. 2-1).
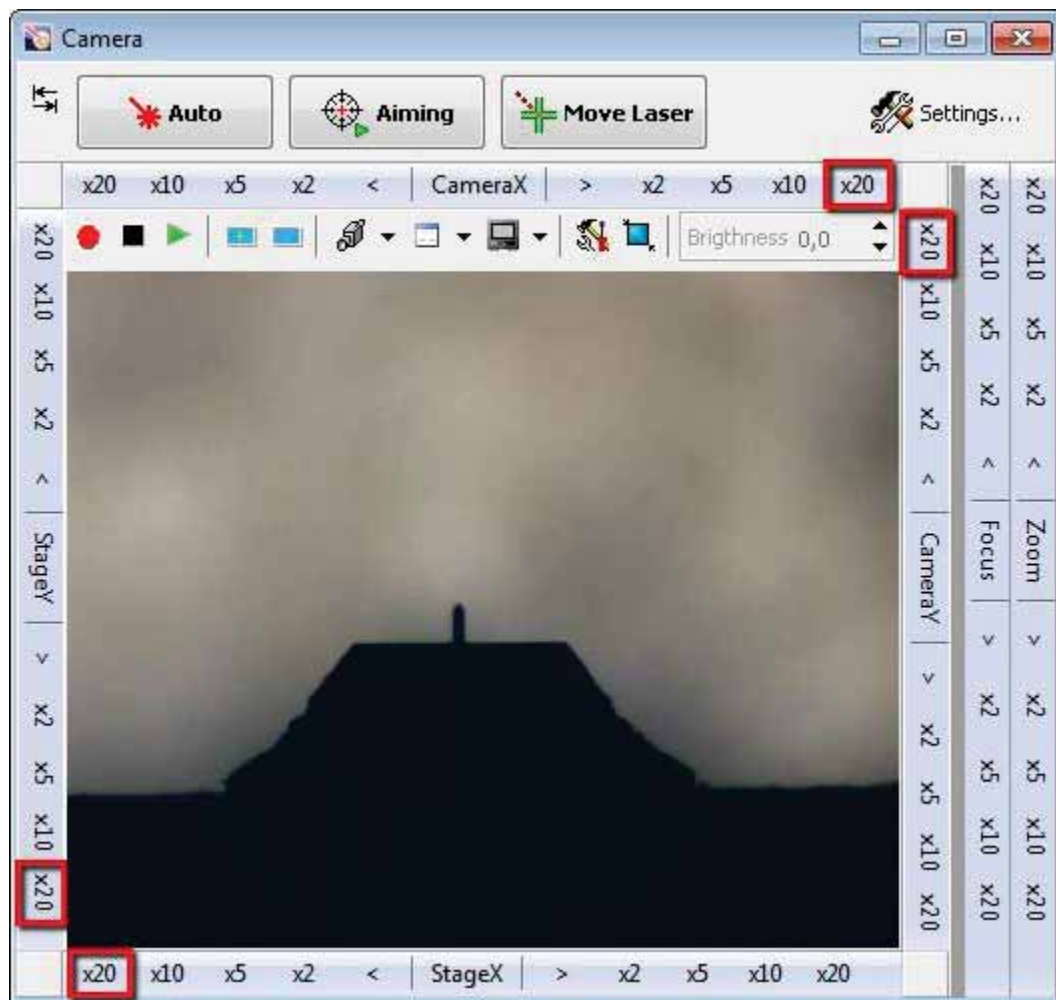
Fig. 2-1. **Camera**

2. Move the scanner carriage to the farthest right corner using controls `x20` in the panels **StageY** and **StageX** (see Fig. 2-1).

3. Move the videomicroscope carriage to the farthest right corner using controls `x20` in the panels **CameraY** and **CameraX** (see Fig. 2-1).

4. Close the **Camera** window.

For moving the scanner down, perform the following steps:

1. Open the **Approach** window by clicking the ⬜ **Approach** button.

2. Click the ⬜ **Move** button several times. This will make the scanner to move down. When the limit stop is reached, the log of the **Approach** window will be appended with the message «Backward Limiter Achieved». Click the **OK** button.

3. Close the **Approach** window.

Move up the protective shield.

Installing bracket of scanner carriage
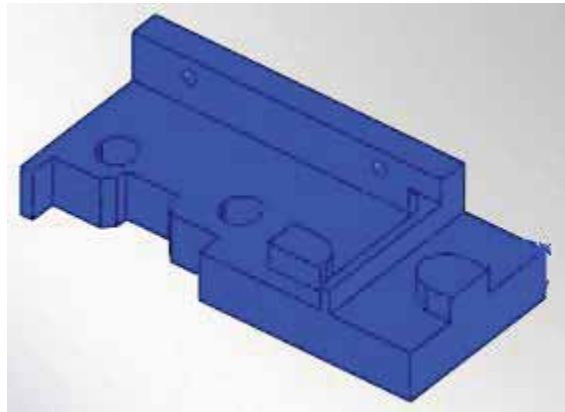
Fig. 2-2. Bracket of scanner carriage

1. Place the measuring unit on a smooth surface covered with clean tissue to prevent scratching the unit. Remove bottom panels of the measuring unit of SOLVER NEXT (Fig. 2-3).

⚠ **ATTENTION! Never remove the rear panel of the measuring unit (Fig. 2-3) because it can be assembled only at the factory.**
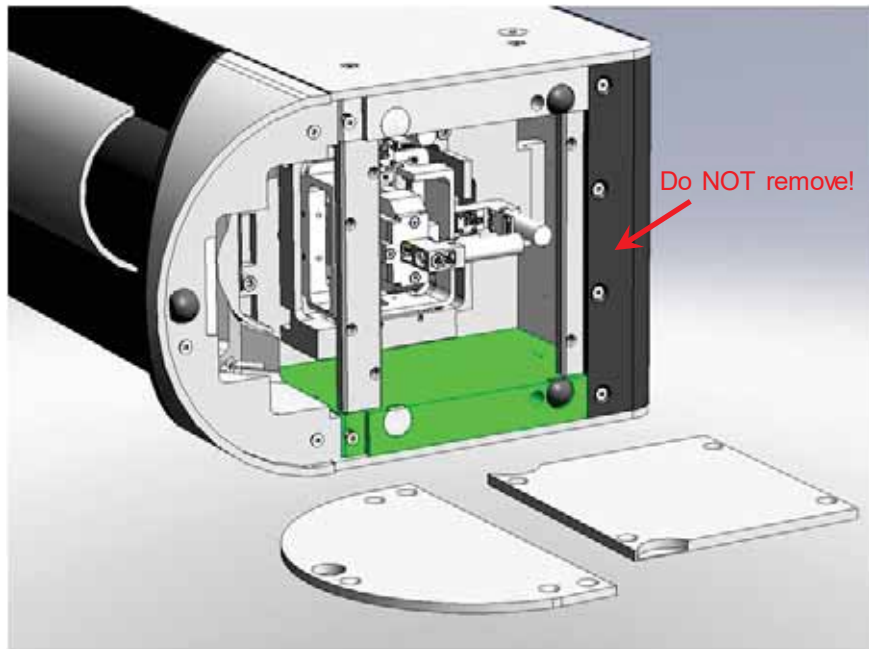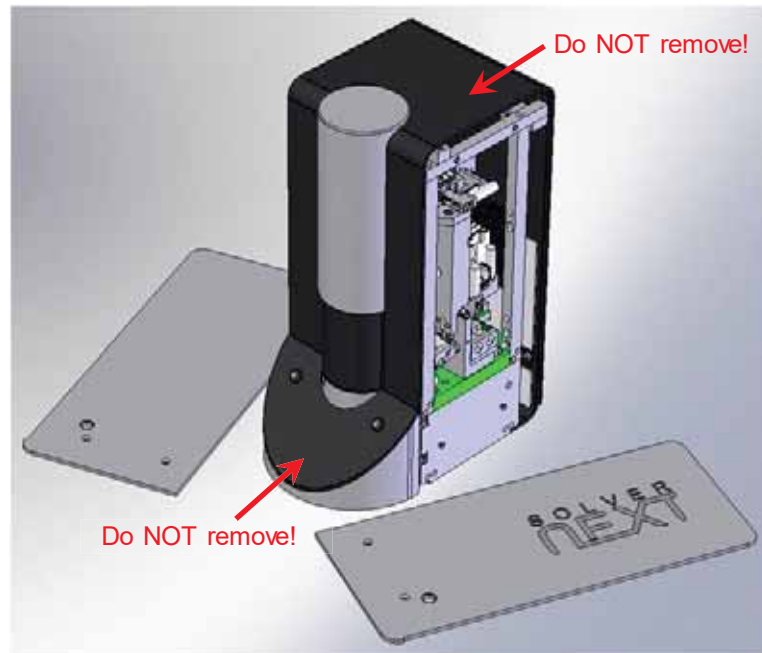
Fig. 2-3. Bottom panels of the measuring unit removed

2. Place the bracket of the scanner carriage so that outdents of the bracket marked in red fit cuts in the carriage ((see Fig. 2-4). For better match, move the frame of the carriage.
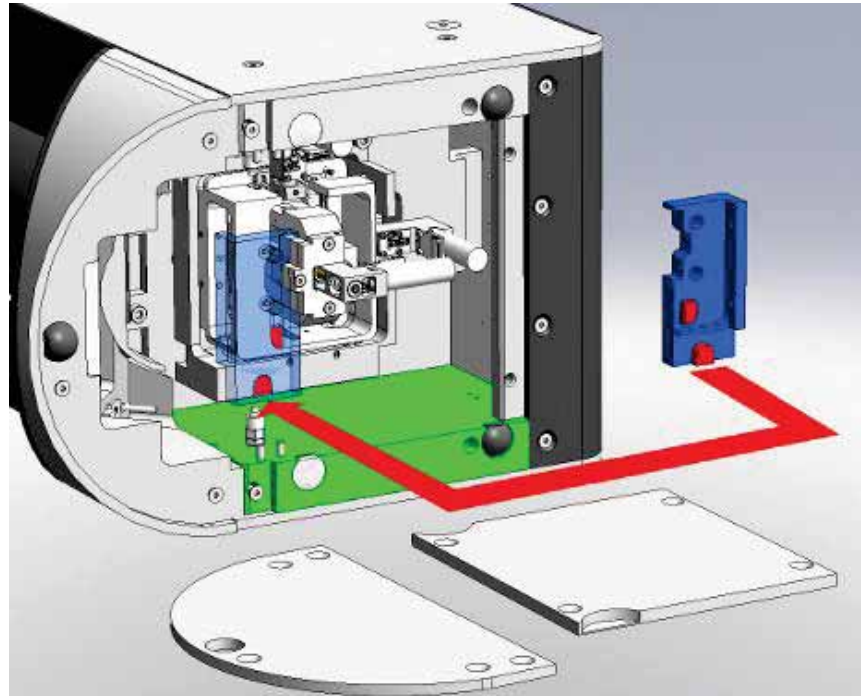
Fig. 2-4. Installing bracket

3. Secure the bracket with two screws M3 (Fig. 2-5).
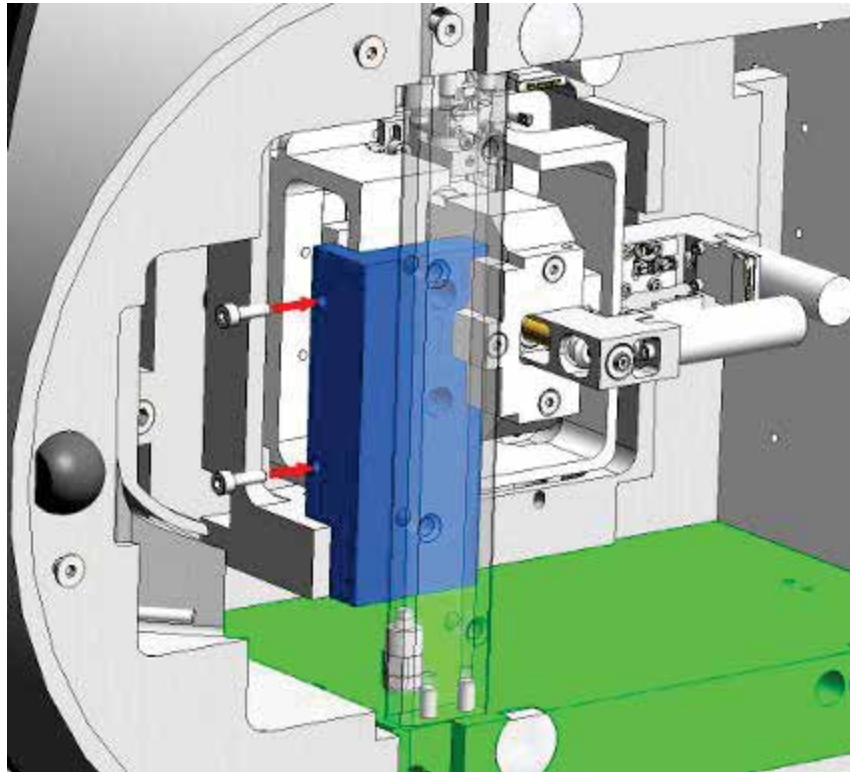
Fig. 2-5. Securing bracket

4. Emplace the bottom panels.

Installing scanner bracket
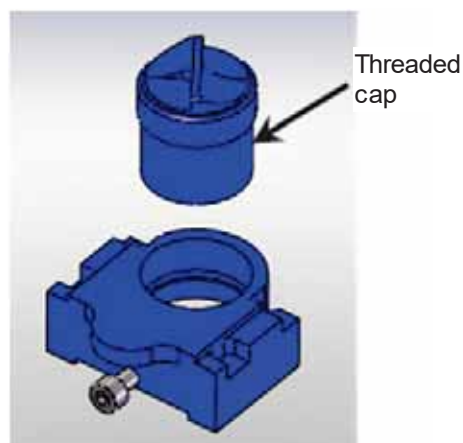
Fig. 2-6. Scanner bracket with threaded cap

1. Prepare the bracket. The threaded cap should be unscrewed to its maximum (Fig. 2-7).
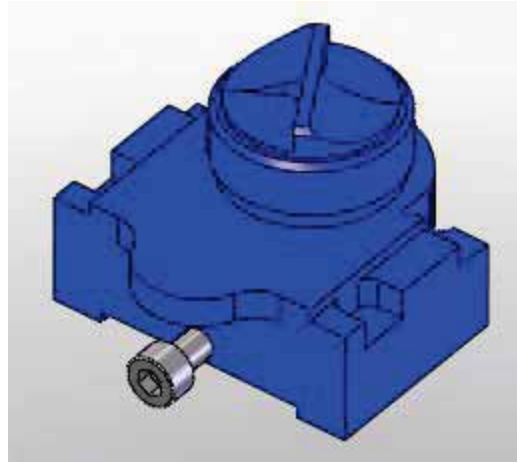
Fig. 2-7. Threaded cap inserted in scanner bracket

2. Insert the bracket from the front of the microscope, then lift the bracket so that its cuts are under guide pins of the left and the right heads (see Fig. 2-8).
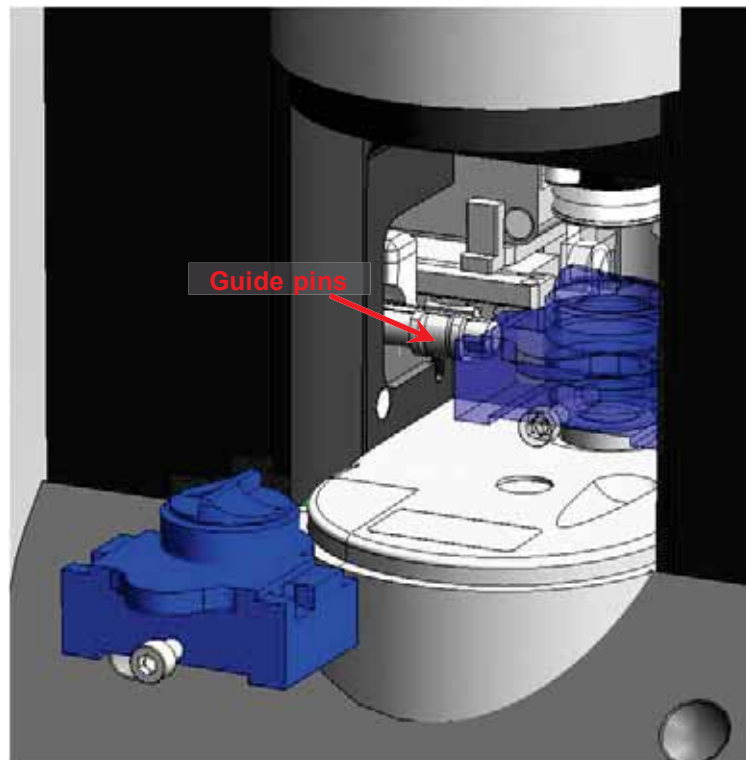
Fig. 2-8. Installing bracket

3. Screw the threaded cap until it touches the scanner. Secure the bracket on the bottom of the scanner with a screw M5 (Fig. 2-9).
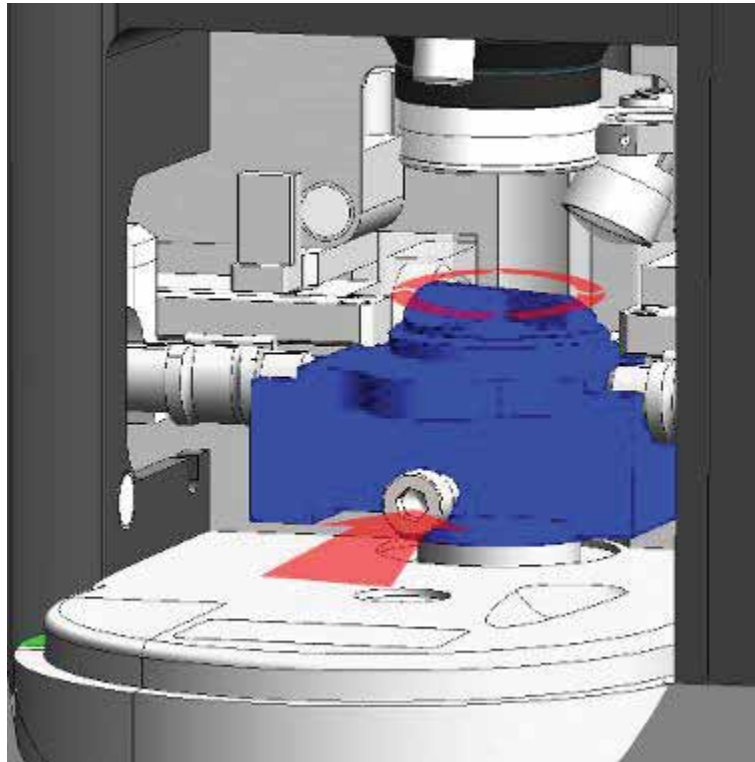
Fig. 2-9

4. Move down the protective shield by clicking ▣ button.

Installing bracket of videomicroscope carriage
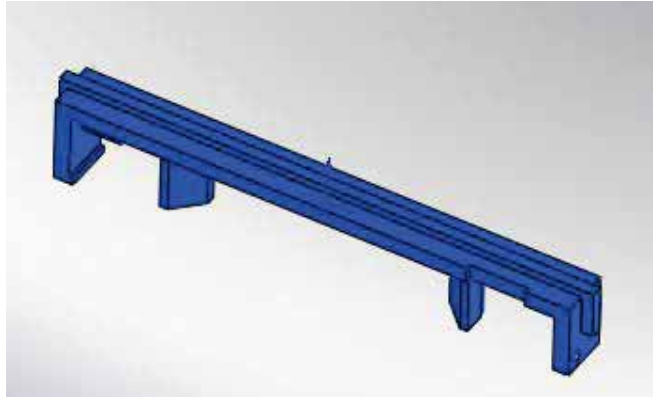
Fig. 2-10. Bracket of videomicroscope carriage

1. Remove the side panels of the measuring unit of SOLVER NEXT by unscrewing two screws at each side of the instrument (Fig. 2-11).

⚠️ **ATTENTION! Never remove panels of the measuring unit indicated with arrows in Fig. 2-11 because they can be assembled only at the factory.**
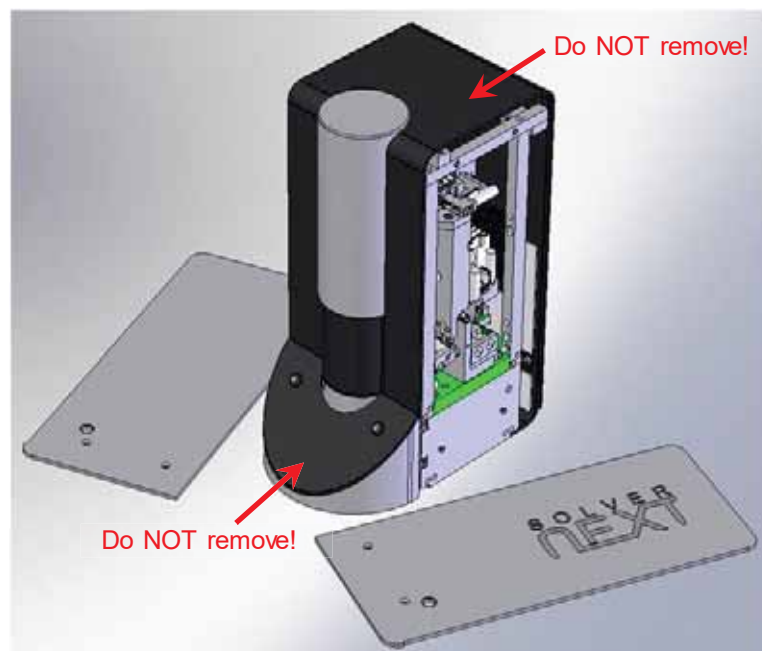
Fig. 2-11. Side panels of the measuring unit removed

2. Insert the bracket of the carriage behind the videomicroscope (see Fig. 2-12). Readjust the carriages, if necessary.
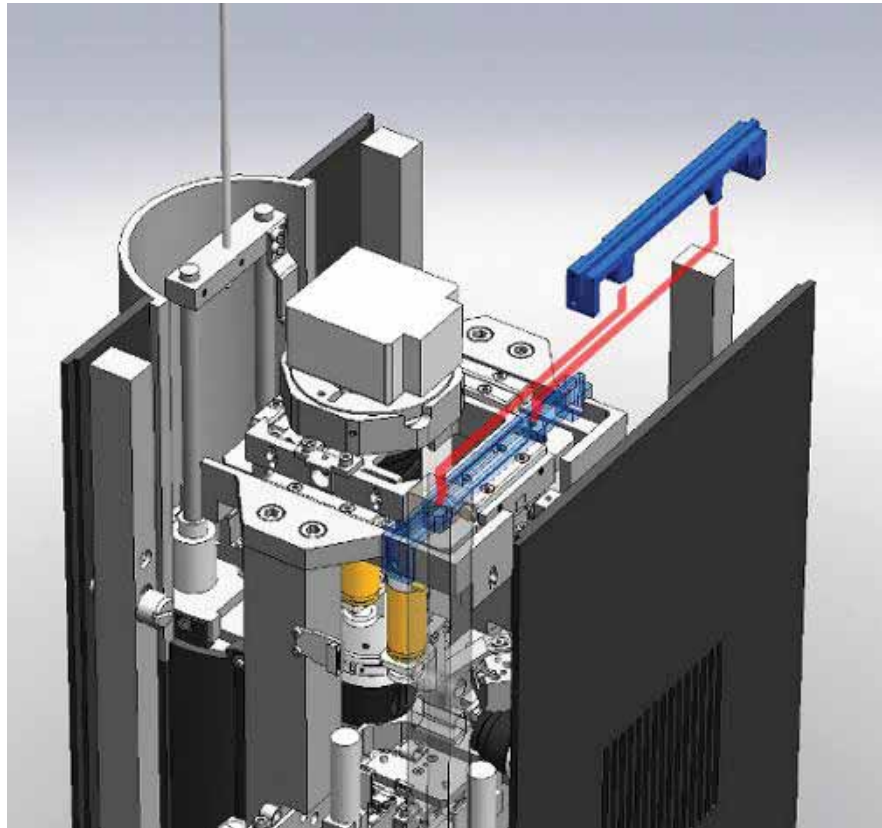
Fig. 2-12. Installing bracket

3. Secure the bracket with a screw M3 (Fig. 2-13).
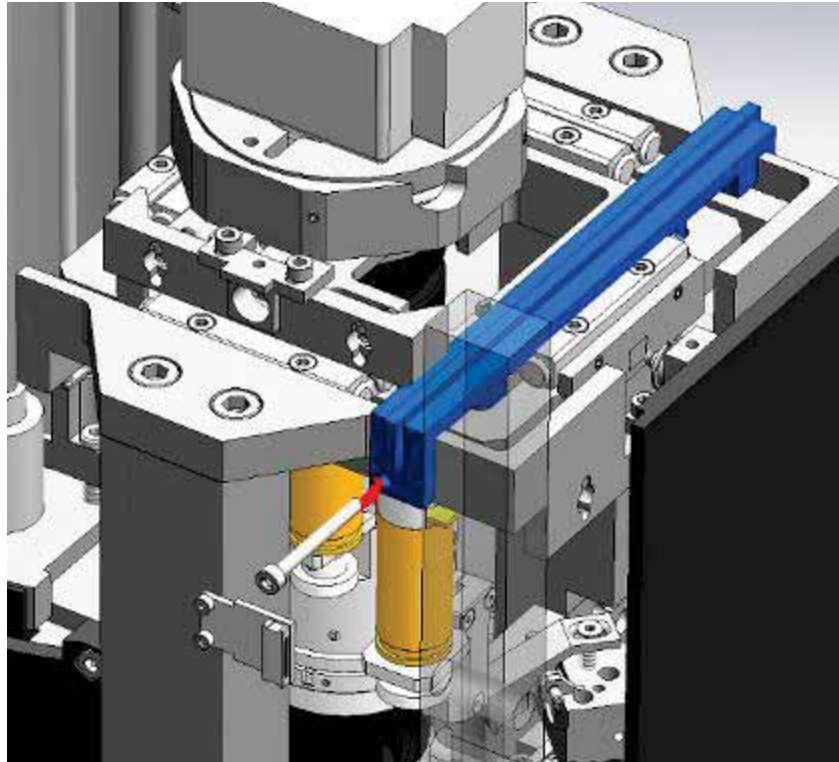
Fig. 2-13. Arrow indicates the hole for the screw

Installing bracket of videomicroscope

Фиксирующая скоба

Fig. 2-14. Bracket of videomicroscope.
The fixing bracket is unlocked

1. Prepare the bracket. It should be unlocked as shown in Fig. 2-14.

2. Insert the bracket between columns supporting the frame and the carriage of the videomicroscope, then rotate it 90 degrees so that the microscope and columns of the videomicroscope carriage are in cuts of the bracket (see Fig. 2-15).
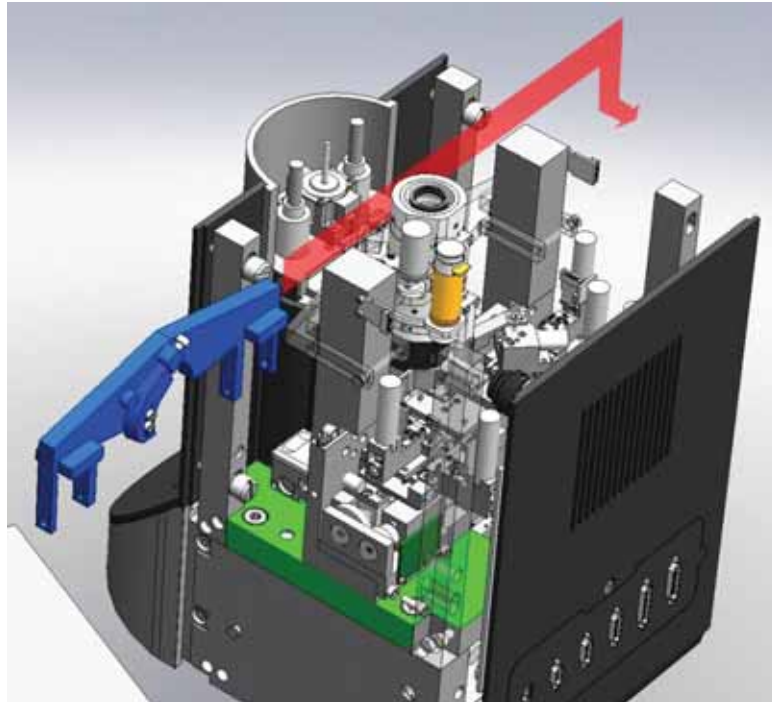
Fig. 2-15. Installing bracket

3. Secure the bracket on columns of the bracket of the videomicroscope carriage using two screws M3 (Fig. 2-16). Ensure that the screws are tightened enough.
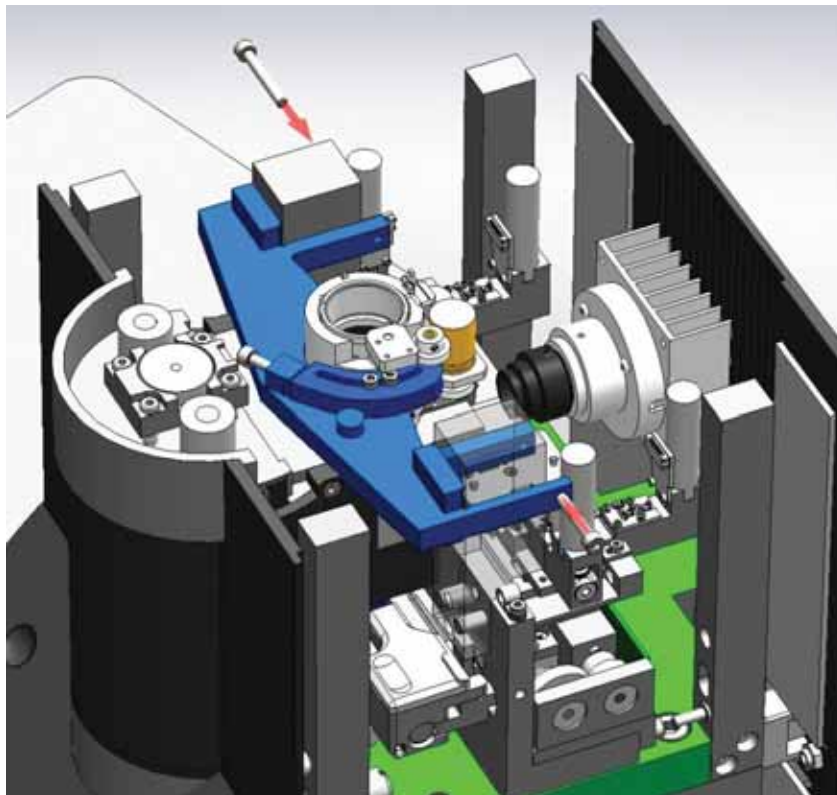
Fig. 2-16. Securing bracket

4. Lock the bracket by securing it with the screw (Fig. 2-17). Ensure that the videomicroscope is secured too.
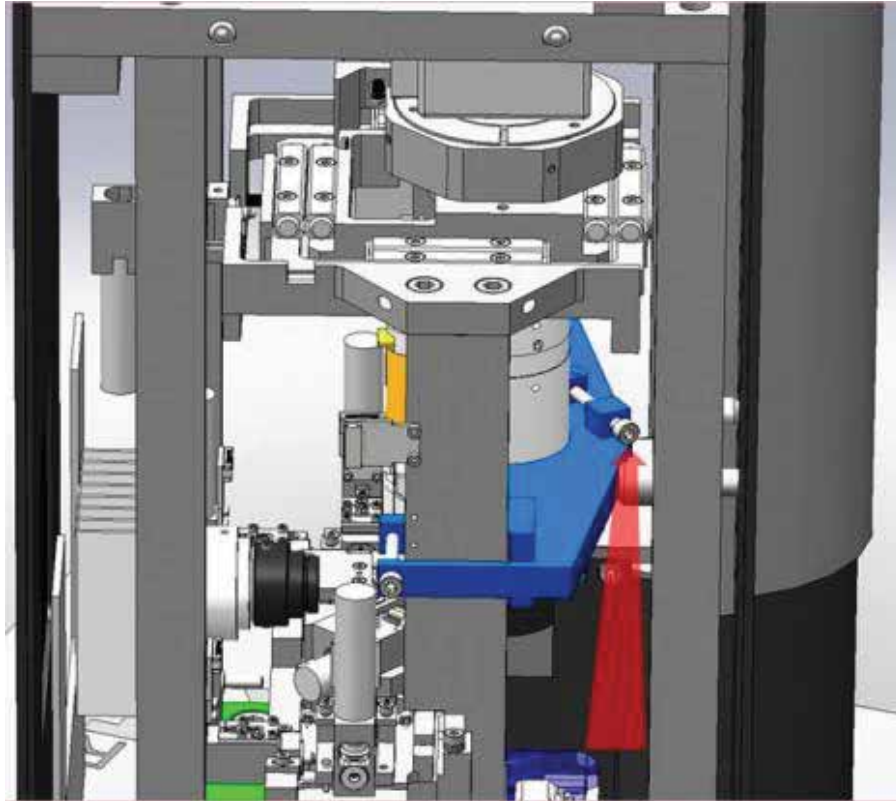
Fig. 2-17. Bracket installed and locked

5. Emplace the side panels.

This completes installing the transport brackets.

3. Removing Transport Brackets

Unpacking the measuring unit needs removal of transport brackets according to the procedures explained below.

Removing bracket of videomicroscope

1. Remove side panels of the measuring unit (see Fig. 3-1).

⚠️ **ATTENTION! Never remove the top and the front panels of the measuring unit (see Fig. 3-1) because they can be assembled only at the factory.**

Fig. 3-1. Side panels of the measuring unit removed

2. Unlock the bracket by unscrewing the screw M3 (Fig. 3-2).
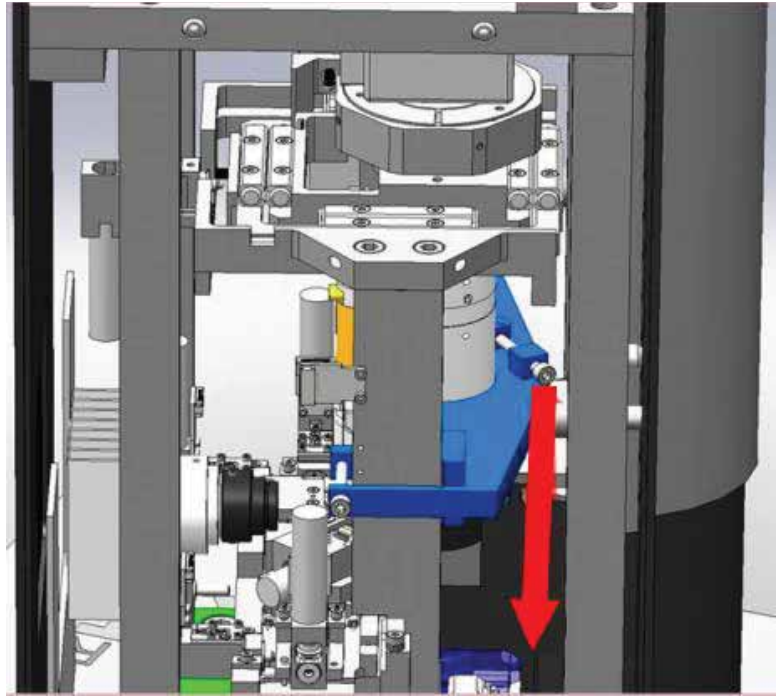
Fig. 3-2

3. Unscrew two screws M3 that fix the bracket on pillars of the videomicroscope carriage (Fig. 3-3).
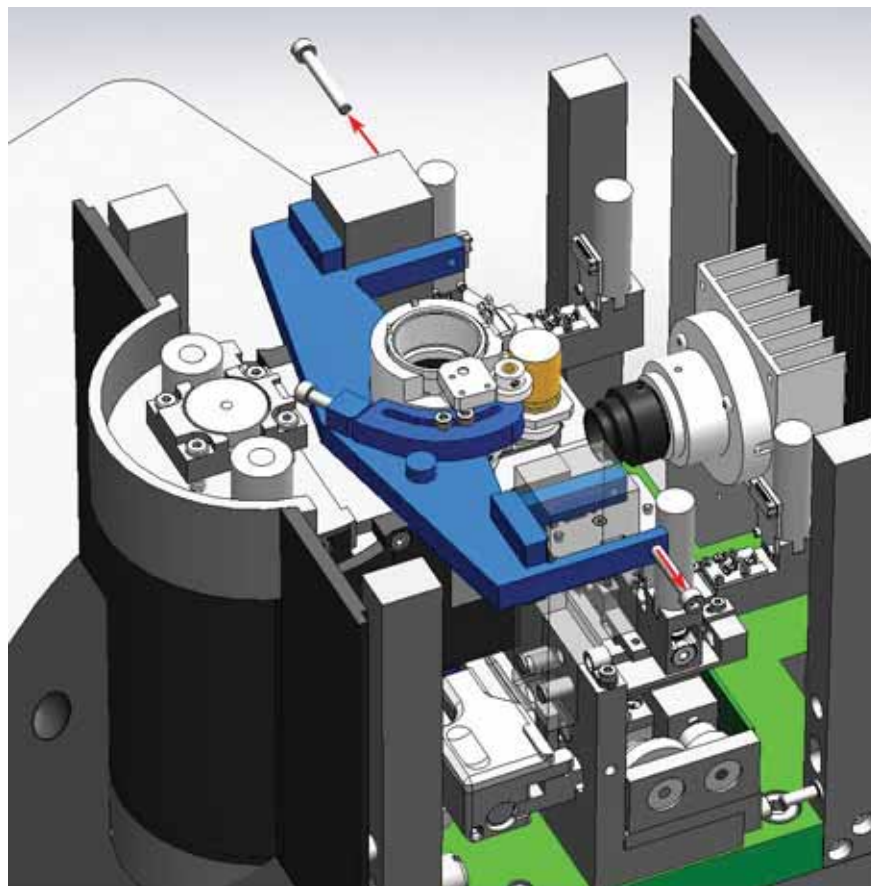
Fig. 3-3

4. Rotate the bracket 90 degrees. Pull out the bracket carefully (Fig. 3-4).
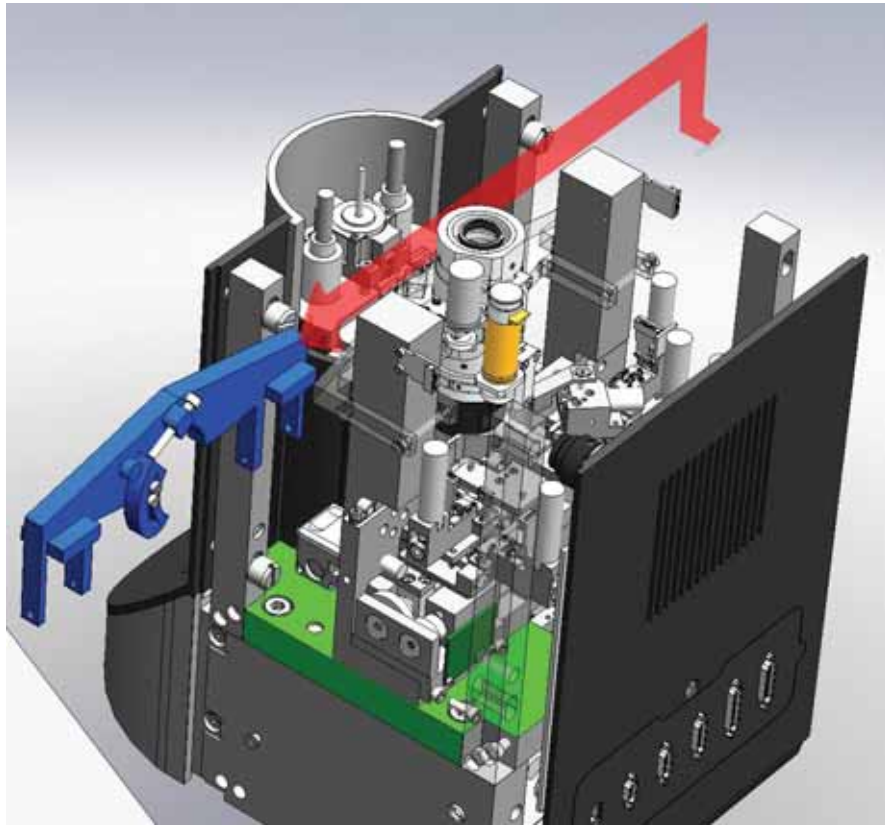
Fig. 3-4. Removing bracket

Removing bracket of videomicroscope

1. Remove the fastening screw M3 of the bracket (Fig. 3-5).
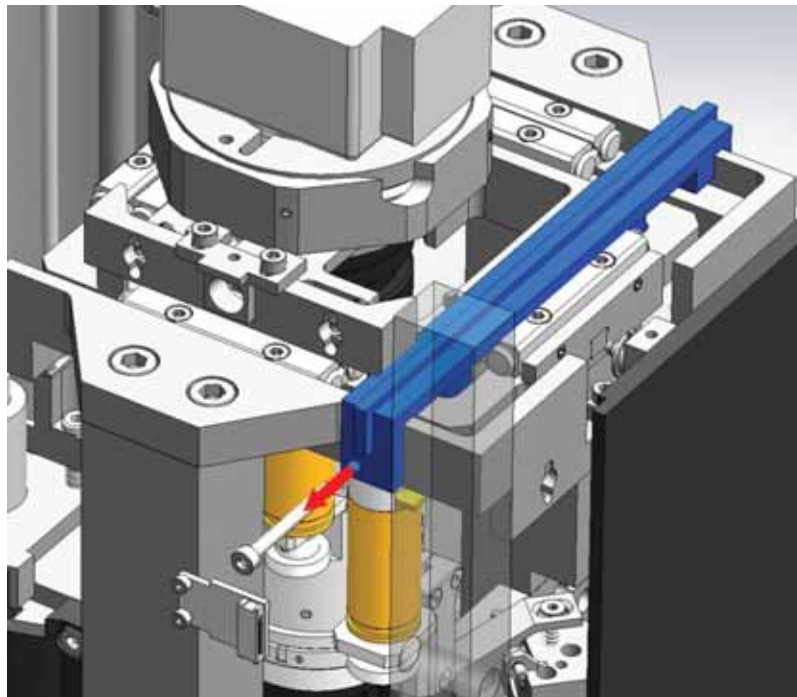
Fig. 3-5

2. Pull out the bracket of the videomicroscope carriage.
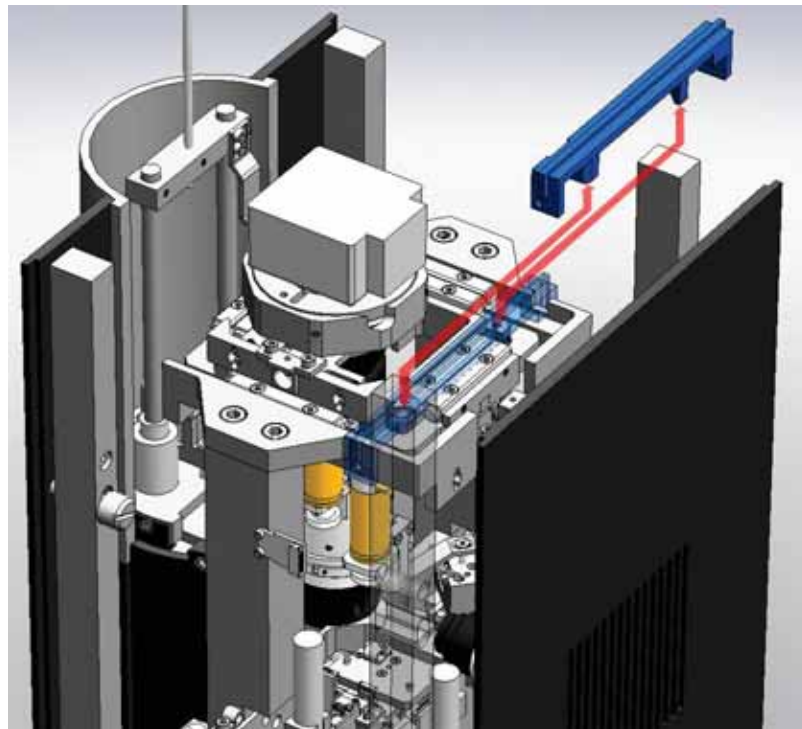
Fig. 3-6. Removing bracket

3. Emplace the side panels.

Removing Scanner Bracket

1. Unscrew the screw M5 on the body of the bracket. Loosen the scanner cap by several revolutions (Fig. 3-7).

Threaded cap
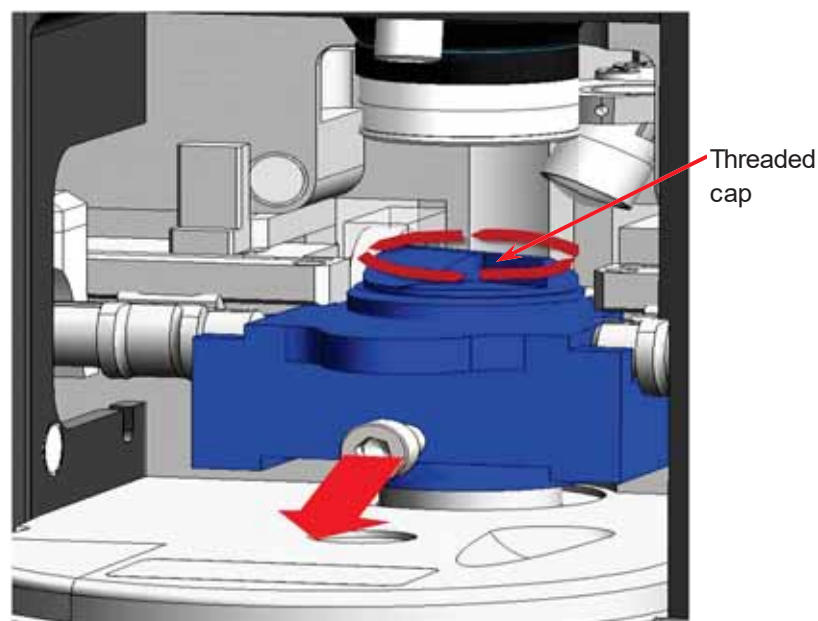
Fig. 3-7

2. Remove the scanner bracket (Fig. 3-8).
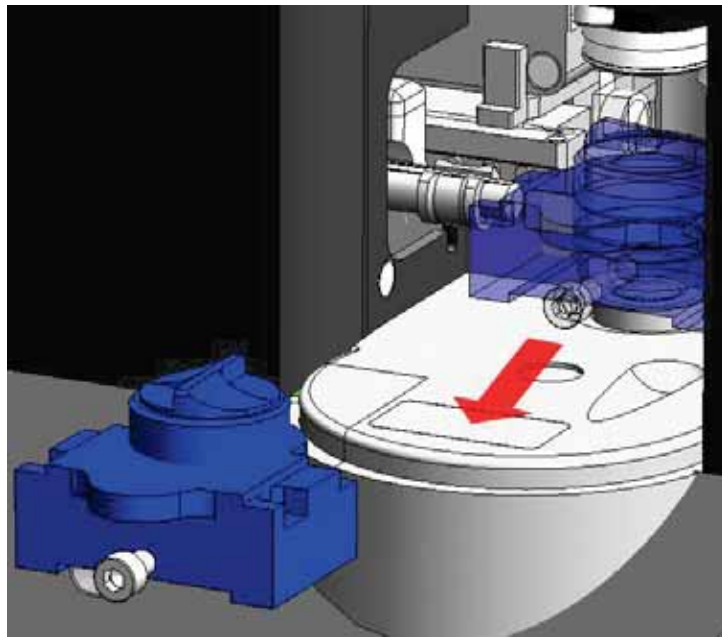
Fig. 3-8. Removing bracket

Removing bracket of scanner carriage

1. Remove bottom panels of the measuring unit.

⚠ **ATTENTION! Never remove the rear panel of the measuring unit (Fig. 3-9) because it can be assembled only at the factory.**

Do NOT remove!
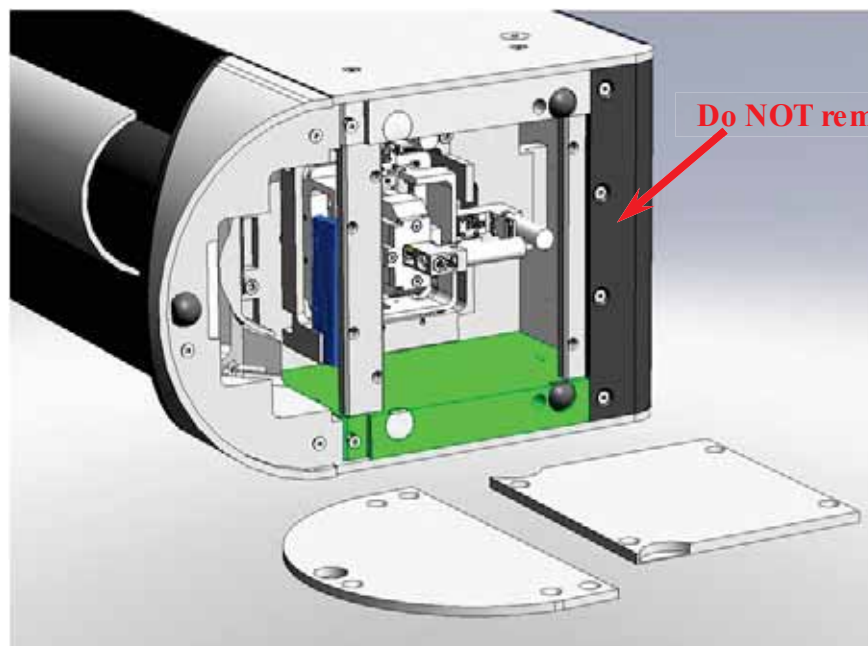
Fig. 3-9. Bottom panels of the measuring unit removed

2. Loosen two screws M3 by several revolutions.
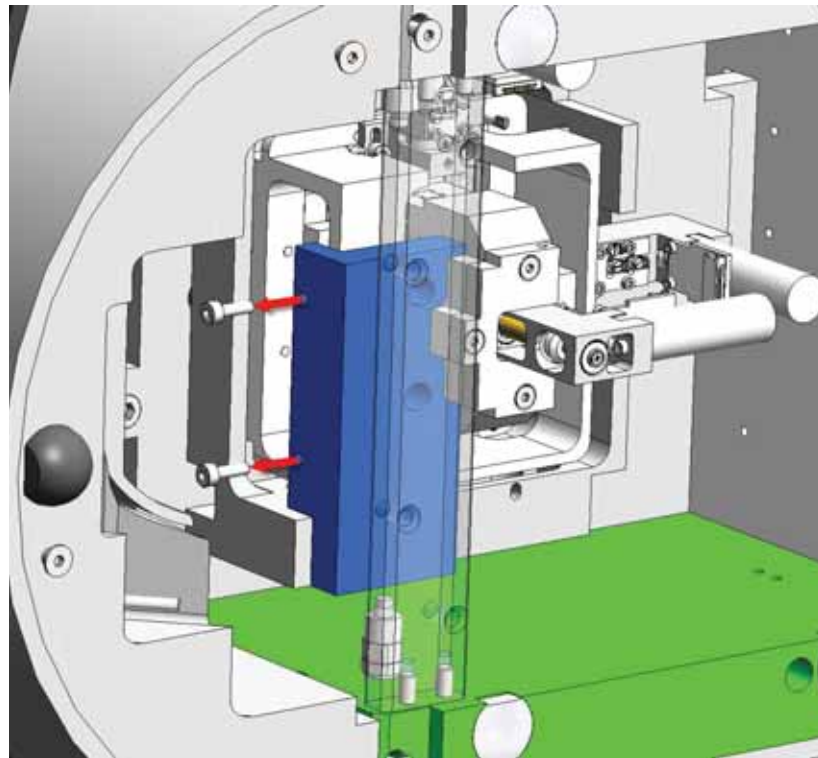
Fig. 3-10. Removing bracket

3. Hold the carriage and pull out the bracket carefully.
4. Emplace the bottom panels.

This completes removing the transport brackets.

4. Cable Connection

⚠ **ATTENTION! Before connecting or disconnecting any unit, switch off the controller. Disconnecting or connecting cables while the device are operating may cause damage to their electronic circuits.**

⚠ **ATTENTION! Use only the power cords conforming to your local standards when connecting the equipment to mains supply.**

1. Place the computer on its stationary location.

2. Perform cable connections according to the diagram of Fig. 4-1.
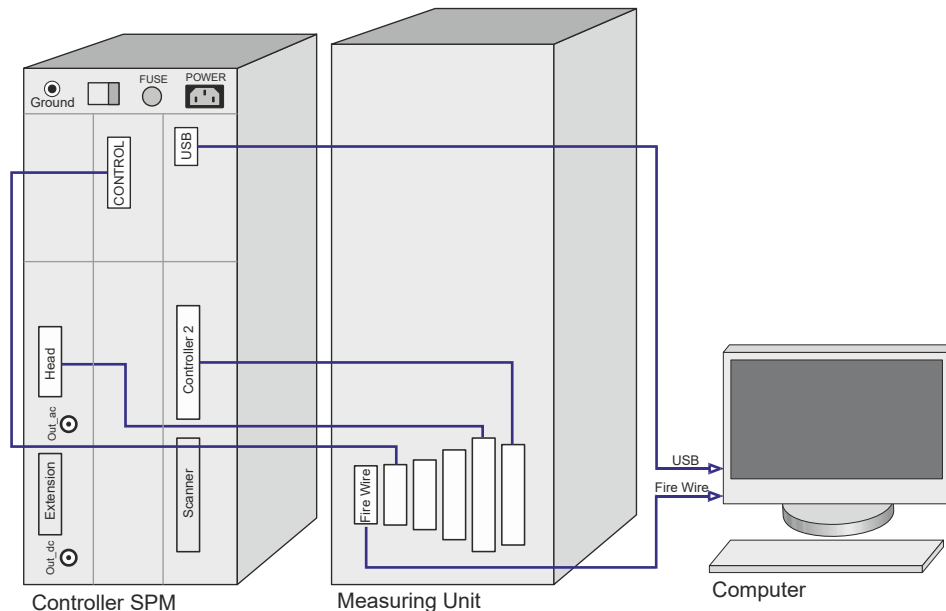
Fig. 4-1. Cable connections

3. Turn the voltage switch of the SPM controller to the position corresponding to the actual voltage of the mains supply. **The controller must be off at the moment of switching!**

⚠ **ATTENTION! The SPM controller can be connected to an electrical power supply line of 110/220 V (60/50 Hz) after setting the voltage selection switch to the position corresponding to this power supply line. Observe this instruction to prevent damage of the electronic components.**

4. Connect the SPM controller to the mains supply.

5. Installing Software

Nova PX program doesn't need special installation procedure. Just insert the software CD/DVD-ROM in the computer CD-drive and copy the content of Nova Px folder to the desired location of your hard drive, for example C:\NTMDT\. With the program installed, install drivers of the scanning probe microscope controller and video camera.

Installing Drivers of Controller SPM

1. Connect the SPM controller to the computer. This will make the operating system to detect a new hardware **USB <-> Serial** and to open the **Found New Hardware Wizard** dialog.
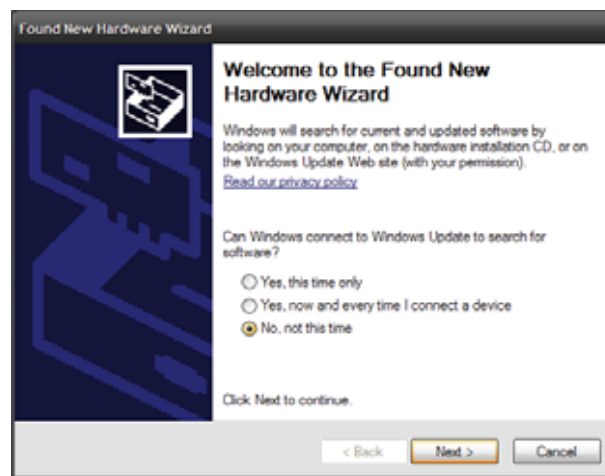
Fig. 5-1. **Found New Hardware Wizard** window

To install the software driver for the **USB <-> Serial** hardware, perform the following steps:

a) Skip the **Windows Update** option in the **Found New Hardware Wizard** dialog by selecting the **No, not this time** option and click the **Next** button.

b) The installation wizard will ask how to find location of drivers on your computer. Select the **Install from a list or specific location** option that allows for manual defining the path of drivers location. Click the **Next** button.
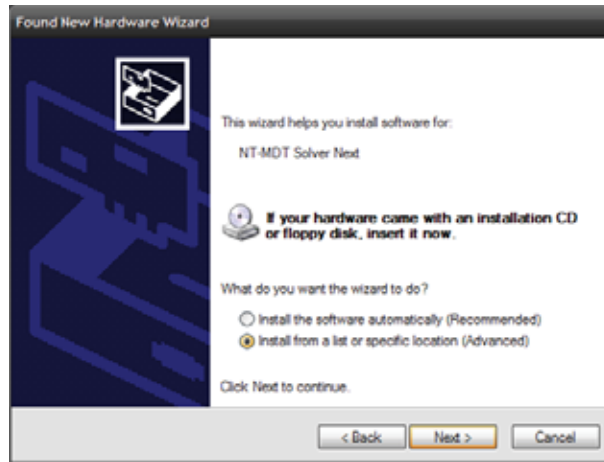
Fig. 5-2

c) For defining the desired path, select the options Search for the best driver in these location and Include this location in the search and then click the Browse button.
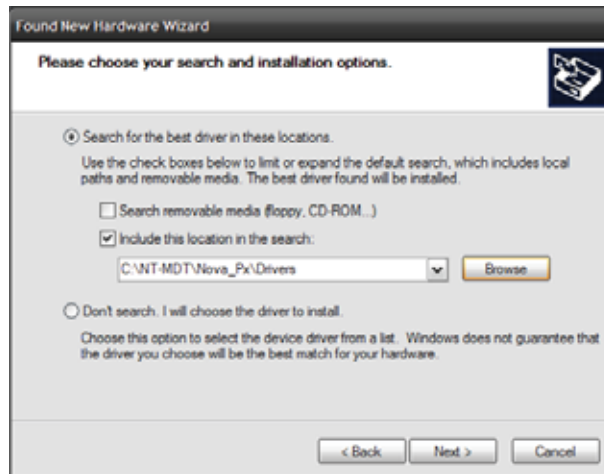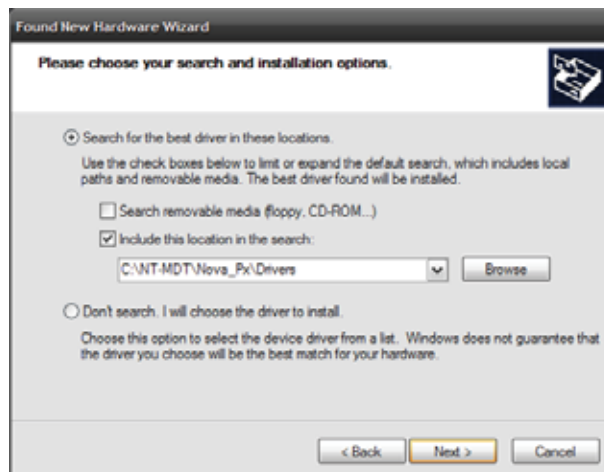

Fig. 5-3

d) The **Browse For Folder** dialog will open. Select the **Drivers** subfolder in the Nova Px program folder and click the **OK** button. This will start installing the controller's driver.
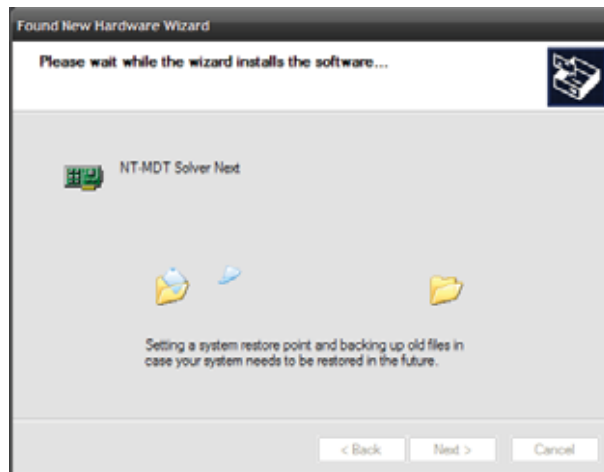
Fig. 5-4. Installing the controller's driver

e) When installing the drivers of the **USB <-> Serial** hardware completes, click the **Finish** button. The installation wizard will close.
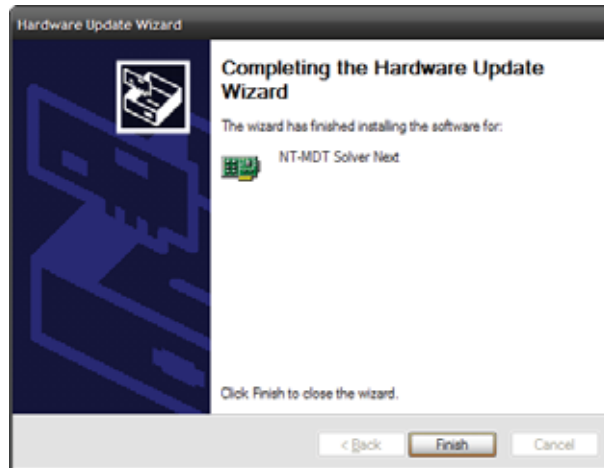
Fig. 5-5

2. With the drivers of the **USB <-> Serial** hardware installed, the operating system will find another hardware, **USB Serial Port**, and open the **Found New Hardware Wizard** dialog again.

Fig. 5-6. **Found New Hardware Wizard** window

To install the software driver of the **USB Serial Port** hardware, perform the following steps:

a) Skip the **Windows Update** option in the **Found New Hardware Wizard** dialog by selecting the **No, not this time** option and click the **Next** button.

b) The installation wizard will ask how to find location of drivers on your computer. Select the **Install from a list or specific location** option that allows for manual defining the path of drivers location. Click the **Next** button.
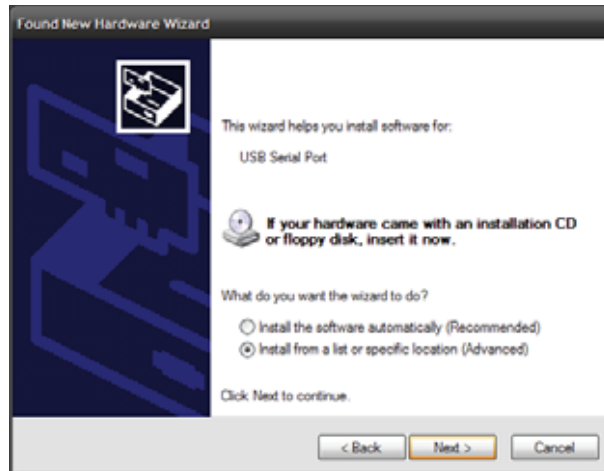
Fig. 5-7

c) For defining the desired path, select the options Search for the best driver in these location and Include this location in the search and then click the Browse button.

Fig. 5-8

d) The **Browse For Folder** dialog will open. Select the **Drivers** subfolder in the Nova PX program folder and click the **OK** button. This will start installing the controller's driver.

e) The **Hardware Installation** window will open to inform that the tested hardware is incompatible with the operating system. Click the **Continue Anyway** button. This will start installing the driver of the controller's USB port.
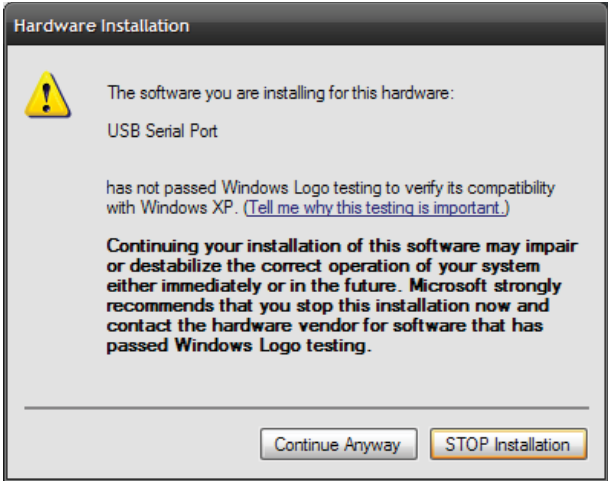
Fig. 5-9. **Hardware Installation** window
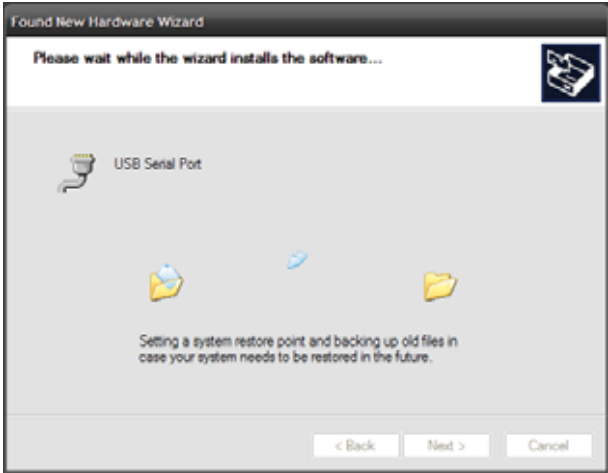
Fig. 5-10. Instaiing the driver of the controller's USB port

f) When installing the drivers of the **USB Serial Port** hardware completes, click the **Finish** button. The installation wizard will close.
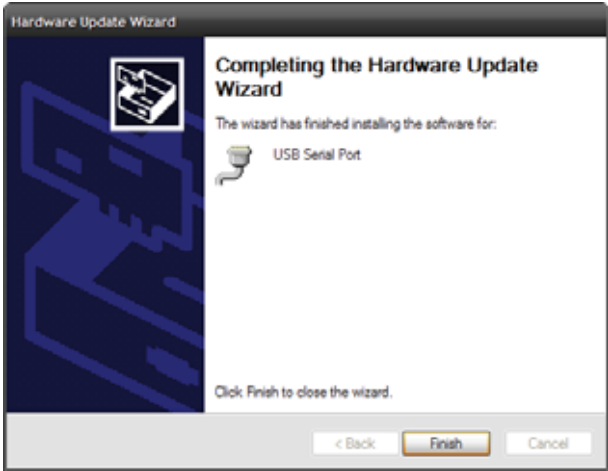
Fig. 5-11

Installing Drivers of Video camera

Drivers of the video camera are supplied on a separate CD-ROM named IMAGINGCONTROL.

1. Insert the CD-ROM into a CD/DVD drive of your computer.

2. The software installer will start automatically and its window will appear on the screen. In this window, select **Software Installation** (Fig. 5-12).

Fig. 5-12

3. A menu of software available for installation will be displayed. For drivers of the video camera, select **Driver** (Fig. 5-13).

Fig. 5-13

4. In the newly opened list of devices, select **FireWire(1394) Cameras** (Fig. 5-14).

Fig. 5-14

5. In the list of manufacturers, select **The Imaging Source** (Fig. 5-15).

Fig. 5-15

6. In the list of device models, select the standard one (Fig. 5-16).

Fig. 5-16

7. A request on connecting the video camera to the computer will appear (Fig. 5-17). Connect the camera to the FireWire 1394 port with a cable supplied with the delivery set of the video camera.

Fig. 5-17

8. When the camera has been connected, a message informing that new equipment was found and successfully connected is displayed at the right bottom of the screen. Then the Driver Installation Wizard window will appear (Fig. 5-18).
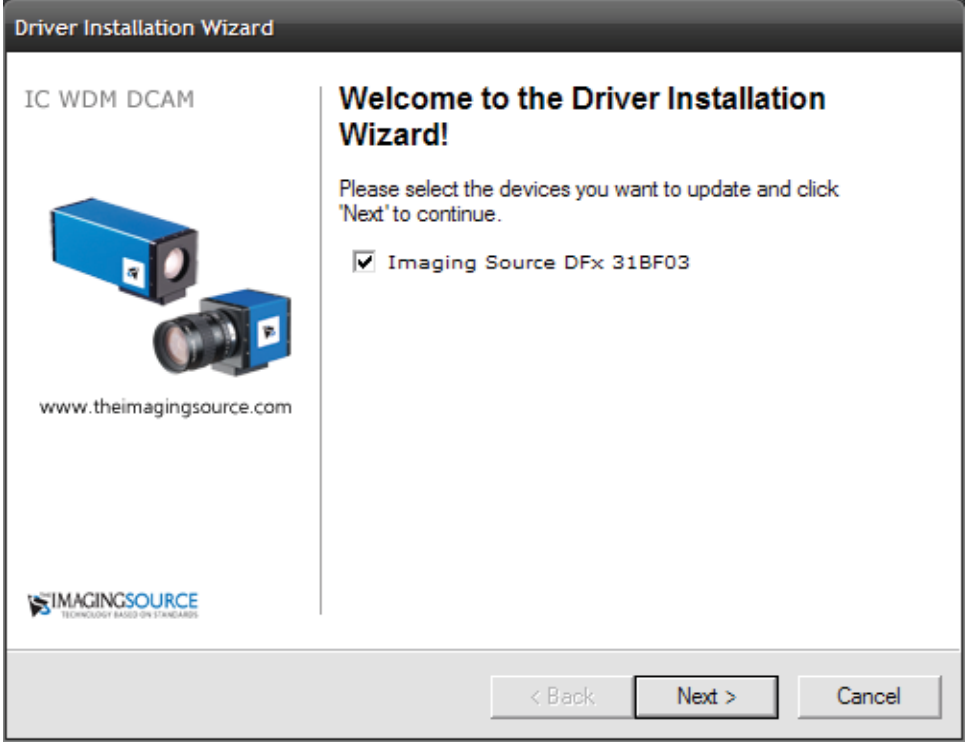
Fig. 5-18

9. Click the **NEXT** button to install the driver of the video camera. In the **Hardware Installation** window, click the **Continue Anyway** button (Fig. 5-19).
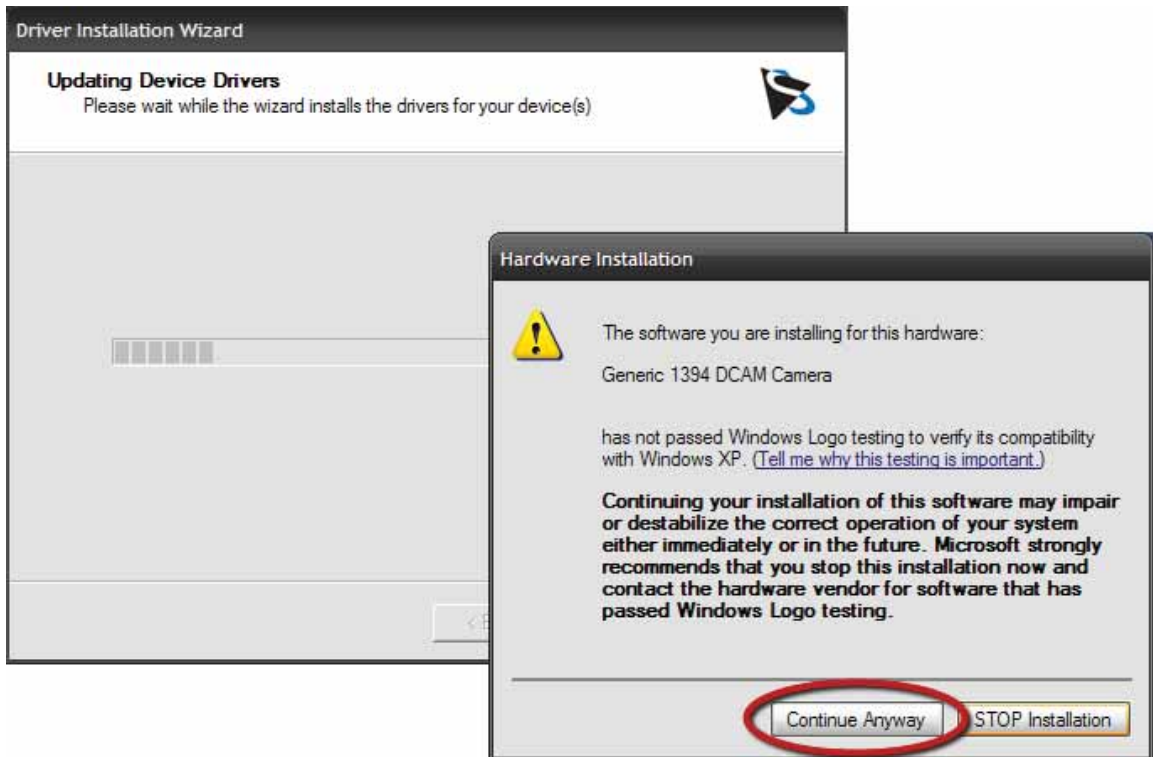
Fig. 5-19

This will start installing drivers of the video camera (Fig. 5-20).
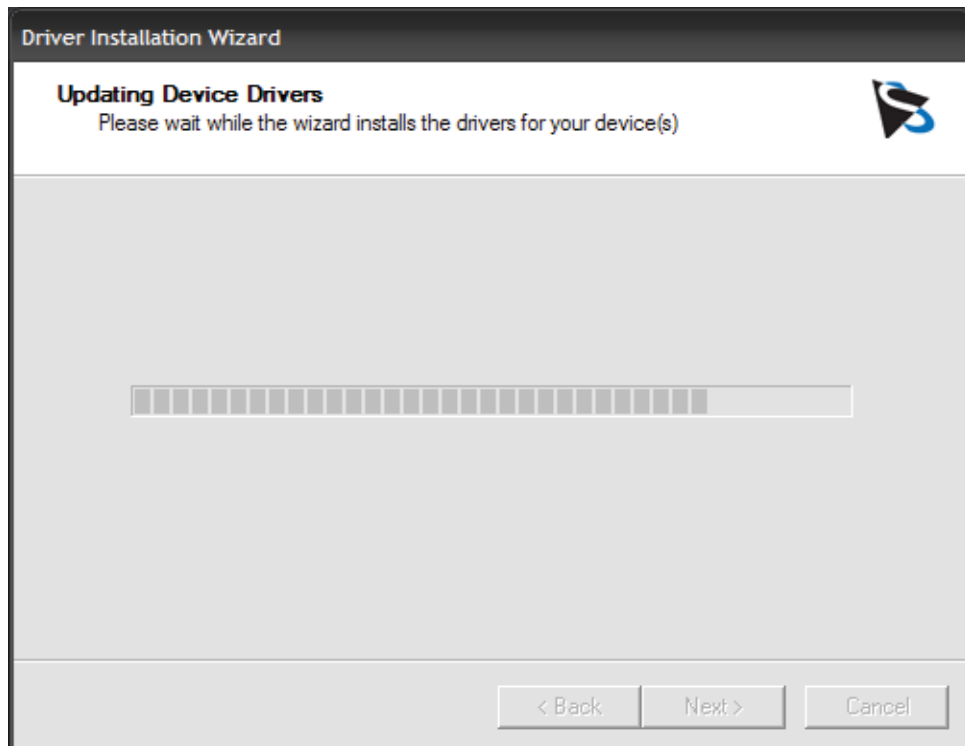
Fig. 5-20

10. When installation completes, a message window informing on success of software installation will appear (Fig. 5-21). Click the **Finish** button.
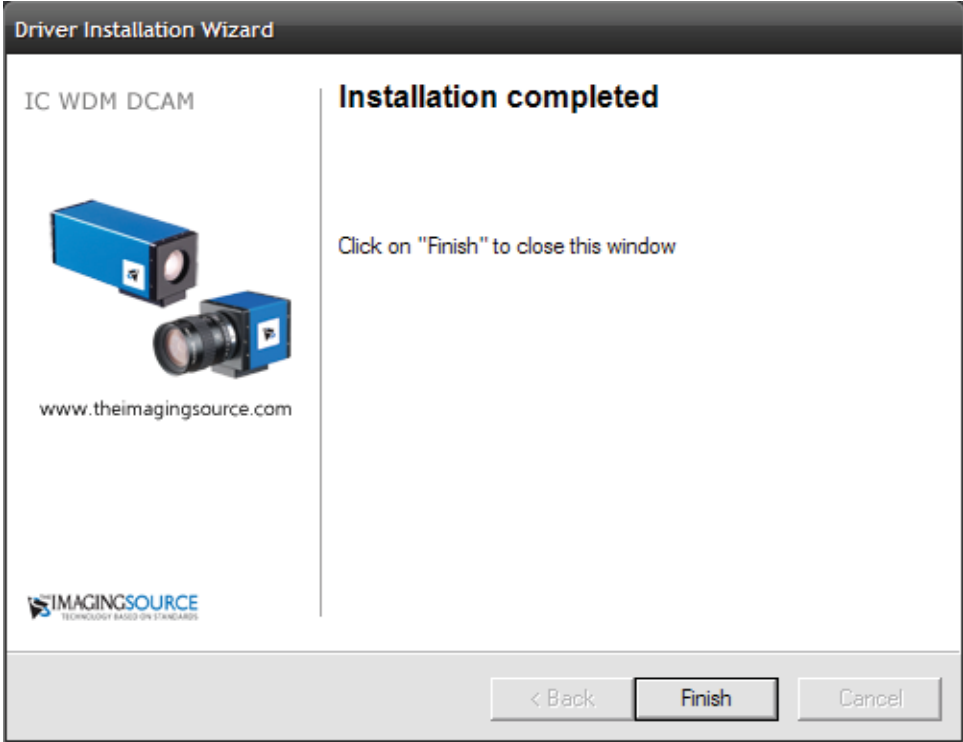
Fig. 5-21

This completes installing drivers of video camera.

For viewing the image from the video camera in the **Camera** window of the program, this device should be activated. To this purpose, perform the following steps:

1. Launch the Nova Px program.

2. Open the **Camera** window by clicking the [Camera] button.

3. Click the button. In the newly opened list, select DFx 31BF03 (see Fig. 5-22).
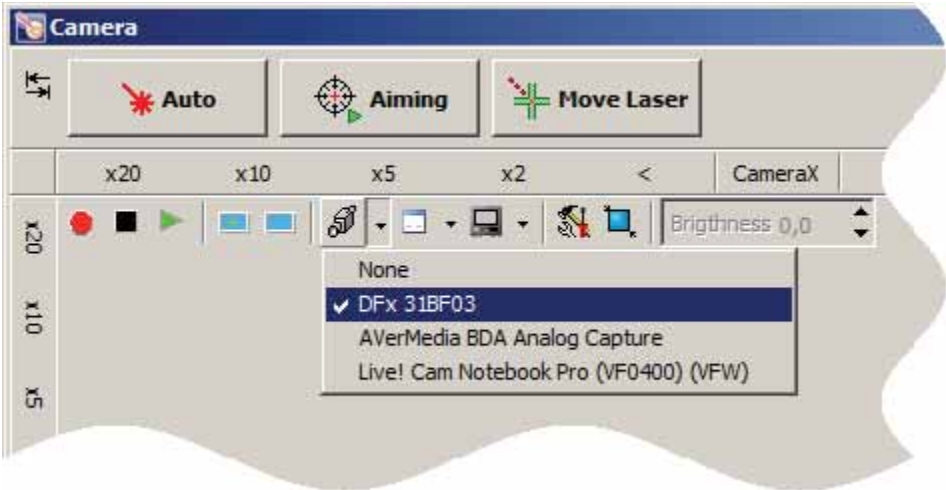
Fig. 5-22. Activating the video camera

4. Adjust parameters of viewing as necessary. The adjustment options are accessible through clicking the ⬜▾ button and selecting **Video Stream Format** in the list that then opens.

6. Calibrating Stepper Motors

Stepper motors of SPM SOLVER NEXT should be recalibrated every time when the Nova Px program is (re)installed or updated as well as when the instrument has been affected by a failure (for example, power failure during operation). For proper operation of SPM SOLVER NEXT, all of its stepper motor motors should be recalibrated.

ATTENTION! Before starting the stepper motors calibration procedure, remove the sample and the metal substrate form the sample holder! Otherwise, stepper motors of the measuring head can touch and damage the sample.

1. Select the **Tools → Motors → Motor Calibration** Main Menu command (Fig. 6-1). This will open the **Motor Calibration** window (Fig. 6-2) to control stepper motors for calibration.
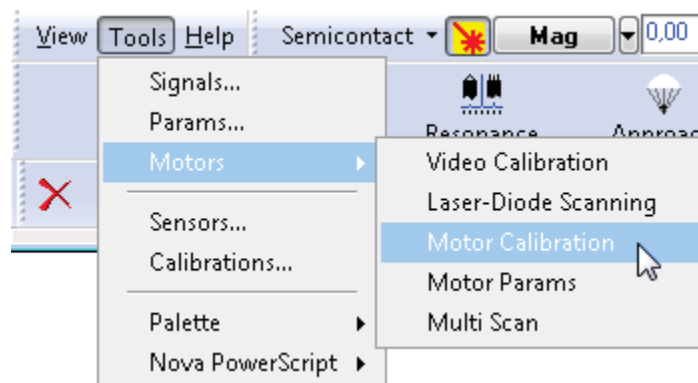
Fig. 6-1. Main Menu

2. In the left part of the **Motor Calibration** window, mark stepper motors to be calibrated (Fig. 6-2).

   *NOTE. If the **Check ALL** option (located below the list of stepper motors) is selected, all stepper motors of the instrument will be calibrated.*
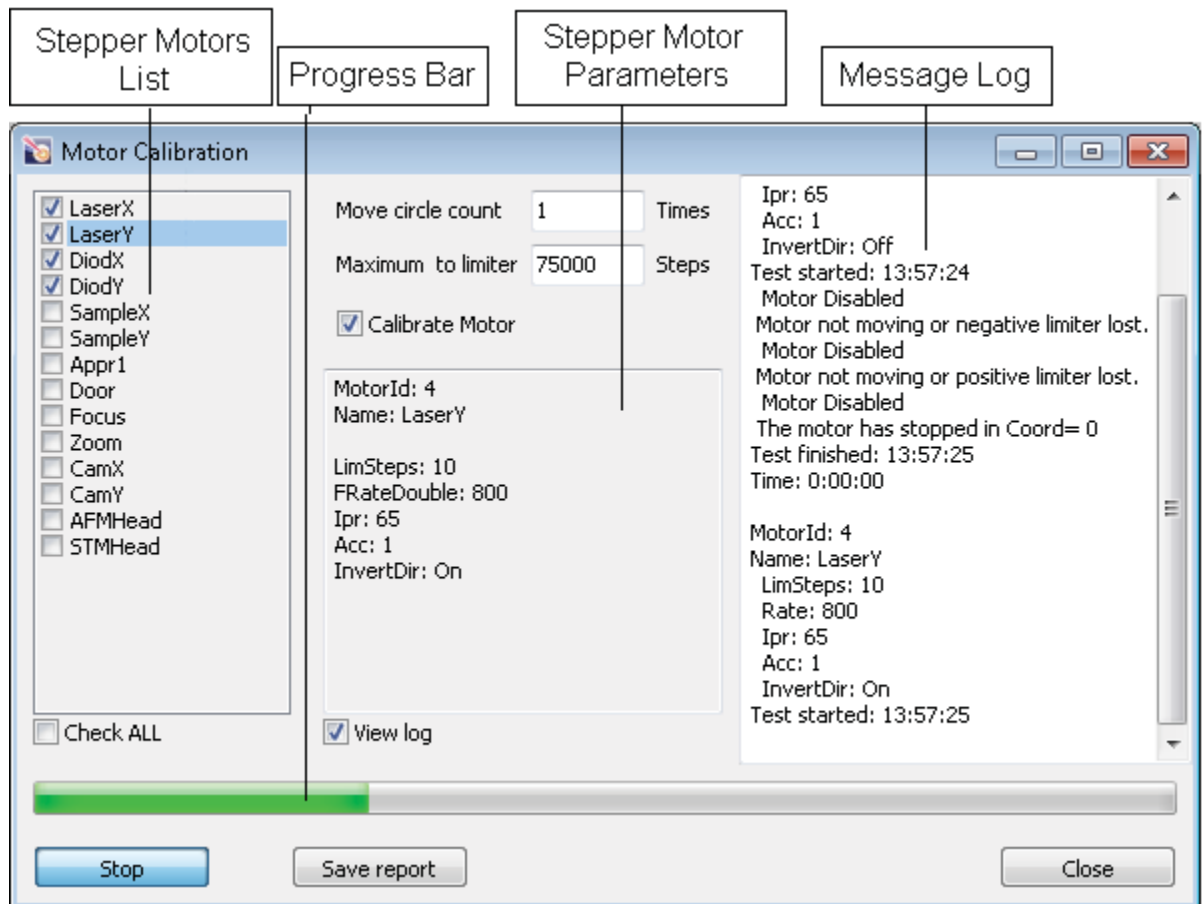
Fig. 6-2. **Motor Calibration window**

3. Select the **Calibrate Motor** option and click the **Run** button. This will start the procedure on calibrating the stepper Motors, and the **Run** button will change its caption to **Stop**. The Message log of the **Motors Calibration** window will provide the following information on the motor under calibration: motor's ID, time of starting calibration, time of finishing calibration, and the total time of calibration. The indicator at the bottom of the **Motors Calibration** window will display percentage of progress of the calibrating procedure (Fig. 6-2). Calibration of all stepper motors takes about 20 minutes in total.

4. When the calibration completes, a Message box will appear to inform on that (Fig. 6-3). Click the button **OK**.
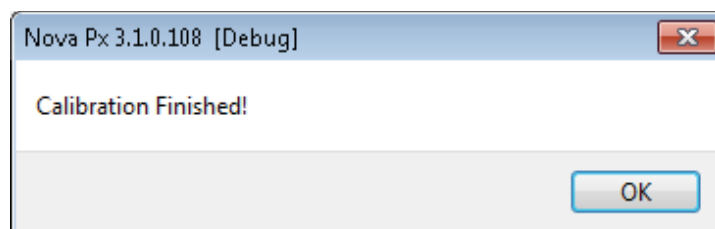
Fig. 6-3. Message box

5. Close the **Motor Calibration** window by clicking the **Close** button.

This completes calibrating the stepper motors.

7. Adjusting Videosystem

The videosystem should be properly adjusted for correct operation of the instrument in automatic positioning the sample by the videoimage and automatic adjusting the cantilever deflection detection system.

The videosystem should be recalibrated after any installation or updating the Nova Px program.

The procedure on adjusting the videosystem proceeds through three stages:

1. Calibrating position of the lens optical axis of the videomicroscope. Performed by the manufacturer. These calibration parameters are stored in the file **AimingParams.ini** located in the subfolder …\Devices\Next\Storage. Before reinstallation/updating of the Control program, this file should be saved for restoring in the program folder after modification of the program.

2. Calibrating stepper motors of aiming the laser beam at the cantilever with the videoimage.

3. Calibrating stepper motors of the sample positioning system with the videoimage.

The videosystem is adjusted through the **Video Calibration** window that opens by the **Tools → Motors → Video Calibration** menu command (Fig. 7-7). The **Calibration** tab provides three panels, which are related to successive step of the adjustment procedure.
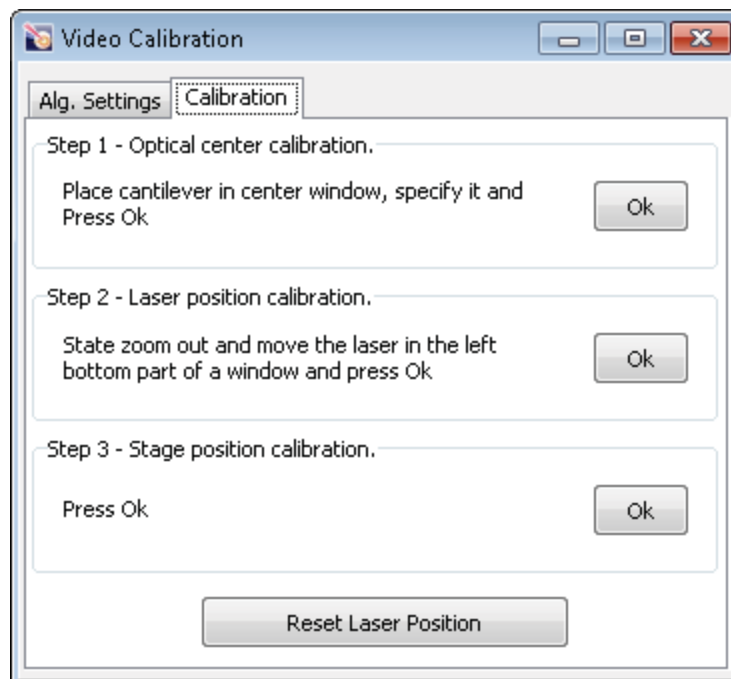
Fig. 7-1. **Calibration**

Preparing instrument for adjusting the videosystem

The adjusting procedure requires the AFM probe. For installing the probe, refer to the section 6.3.1 "Installing the AFM Probe" on p. 25. The sample and its supporting substrates are recommended to remove before calibrating the videomicroscope. Besides, for calibration purposes we recommend to place the cantilever (dark in image) on a uniform light colored background.

1. Move down the protective shield by clicking the [button] button. Enable the laser by clicking the [button] button.

2. Move the AFM head to its working position by clicking the [AFM] button.

3. Open the videoimage window by clicking the [Camera] button. Activate viewing by clicking the [button] button. The window will display an image of the probe (Fig. 7-2).
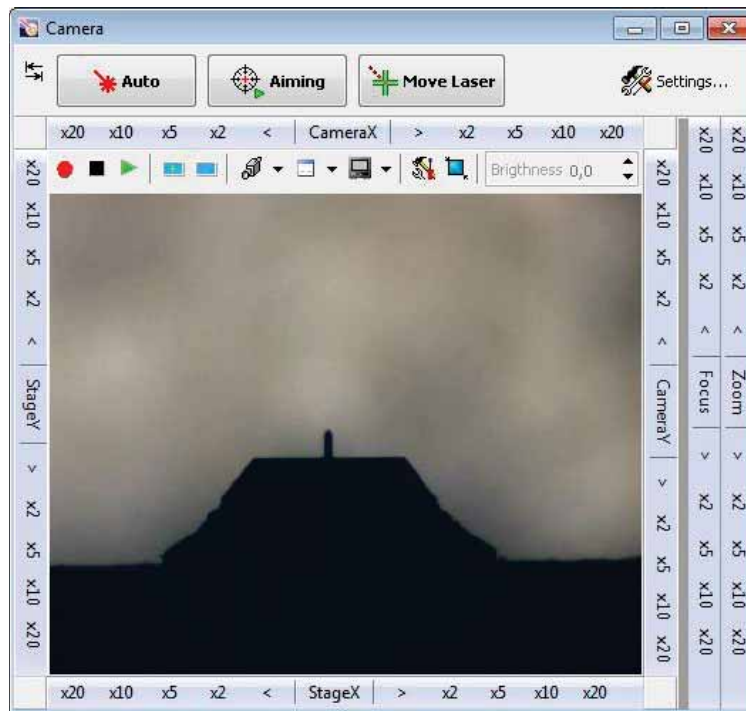
Fig. 7-2. **Camera**

Before starting the procedure on adjusting the detection system, always check which environment was previously selected (air or liquid). The videosystem should be adjusted in air.

1. Open the **Aiming Movers** window by clicking the [Aiming] button in the Main Operations panel.
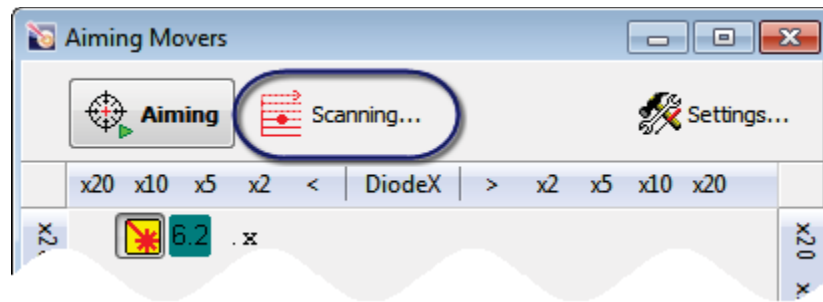
Fig. 7-3. **Aiming Movers**

2. Open the window for adjusting the detection system (Fig. 7-4) by clicking the **Scanning...** button in the Control panel of the **Aiming Movers** window.
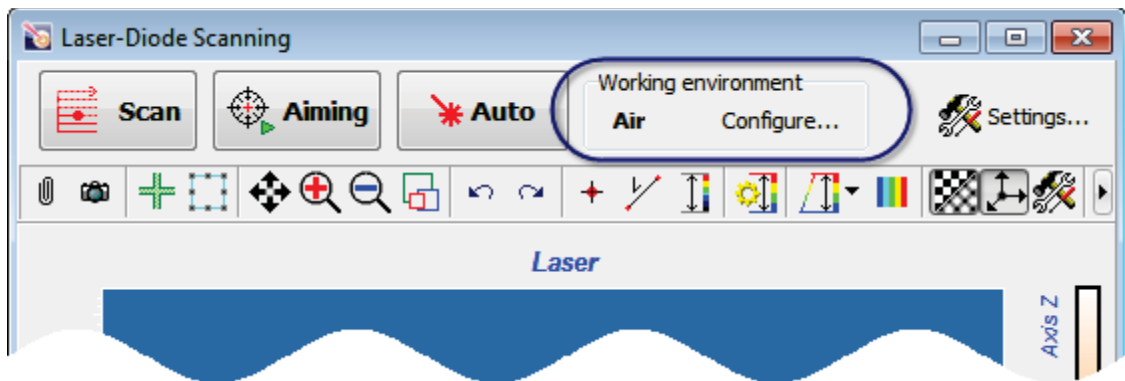

Fig. 7-4. **Laser-Diode Scanning**

3. Verify, that in the **Working Environment** panel the **Air** option is selected. If the (**Liquid**) environment is selected, switch SPM for operation in air as follows:

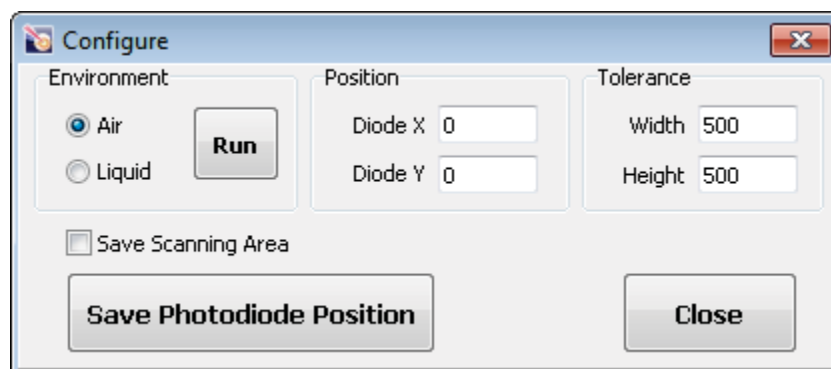a) Open the **Configure** window (Fig. 7-5) by clicking the **Configure...** button.

Fig. 7-5. **Configure**

b) In the **Environment** panel of the newly opened window, select the desired option (**Air** in our example). Then click the **Run** button. The photodiode will be repositioned to the optimal position.

c) Close the **Configure** window.

Step 1. Calibrating position of the optical axis of the videomicroscope

Normally, the optical axis is aligned by the manufacturer. If the file of calibration parameters was saved before modification of the Control program, this step of calibration can be skipped.

This step provides coordinates of the point in the videoimage that corresponds to the videomicroscope optical axis (this point should be invariant under changes of magnification).

1. Using controls of the panels **CameraX** and **CameraY**, reposition the videomicroscope so that the cantilever edge is viewed at the center of the video frame (Fig. 7-2).

2. Reduce the image to its minimum size with controls of the **Zoom** panel.

3. Focus the videomicroscope on the cantilever edge with controls of the **Focus** panel.

4. Click the left mouse button on the image of the edge of the cantilever with the <Shift > key being pressed. This will display a cross-hair marker at the selected point (Fig. 7-6).
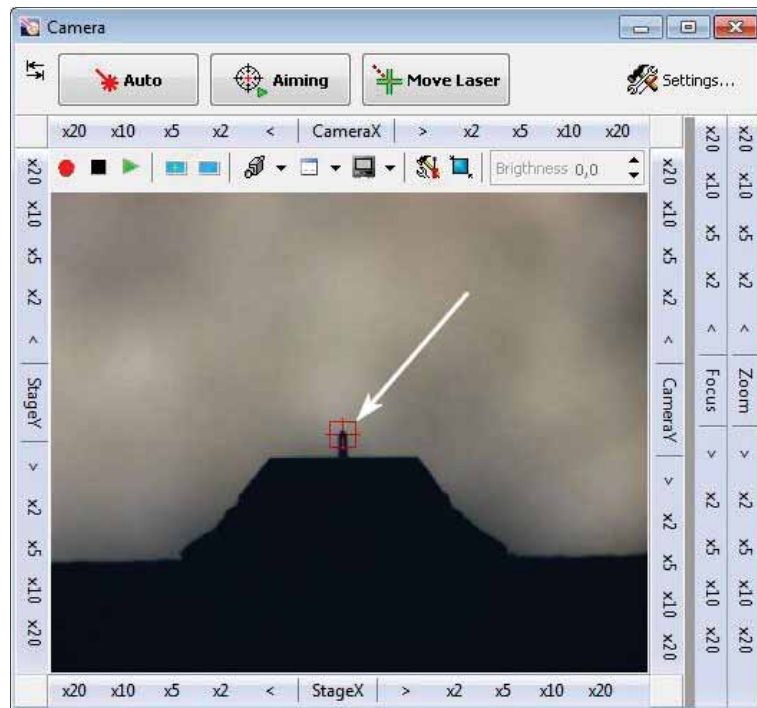
Fig. 7-6. **Camera** window.
Arrow indicates the cross-hair

5. Open the **Video Calibration window** by selecting the **Tools → Motors → Video Calibration** Main Menu command. Go to the **Calibration** tab (Fig. 7-7).
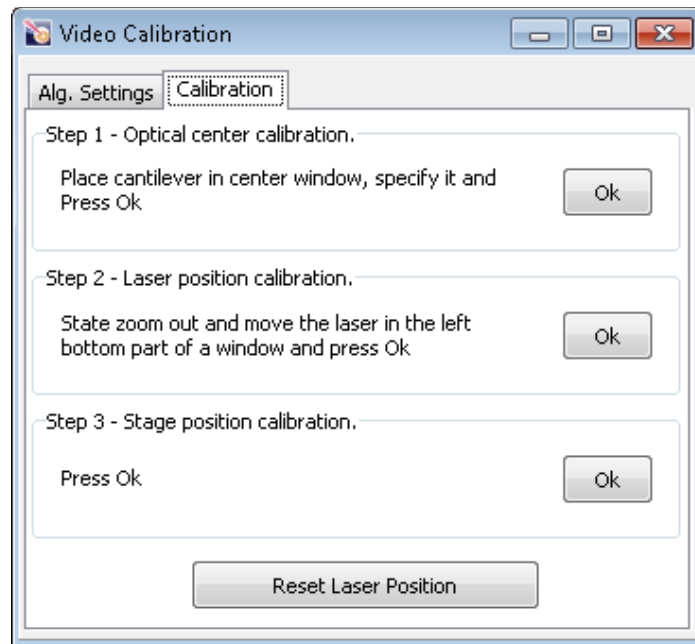
Fig. 7-7. **Calibration**

6. Click the [Ok] button in the **Step 1** panel. This will start searching for the location of the videomicroscope optical axis in the video image. The search implements the following algorithm:

a) The image is zoomed by 2.

b) The distance between the candidate center and the cantilever edge is taken. This value is compared with the allowed discrepancy (defined in the **Precision Immovable** field of the **Alg. Settings** tab). If the distance is lower the prescribed limit, the calibration step completes.

c) If the distance is larger than the allowed limit, the videomicroscope is repositioned so as the cantilever edge approaches to the videomicroscope optical axis.

d) The image is reduced to minimum size and zoomed by 2 again, displacement of the cantilever edge from the center is measured, and the videomicroscope is repositioned if necessary. Iterations continue until this displacement is greater than the prescribed limit.

If this adjustment fails to converge in 5–7 minutes, stop the procedure by clicking the **Break** button and increase the **Precision Immovable** parameter (it defines the allowed limit of displacement (in pixels) of the optical axis under zooming or unzooming the image).

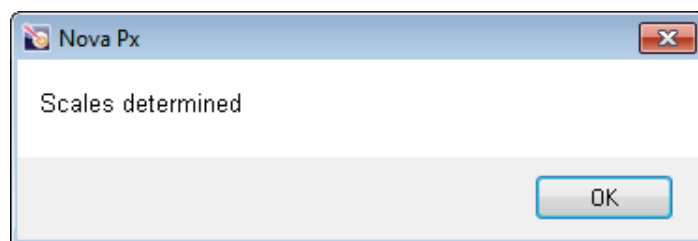When the optical axis is aligned, a Message box appears (see Fig. 7-8). Close it.

Fig. 7-8. Message box

Step 2. Calibrating stepper motors of aiming the laser beam at the cantilever

This calibration provides a relation between displacements of the laser and of position of the laser spot in the working plane (this is the plane where the cantilever edge is located).

1. Using buttons of the **Zoom** panel, make the image as large as possible.

2. Using buttons of the **Focus** panel, focus the videomicroscope at the edge of the cantilever.

3. Move the scanner to its lowest position. To this purpose, click the [Move] button in the Control panel of the **Approach** window. This will make the scanner to move down. When the lowest position is reached, the scanner will stop and a Message box «Backward Limiter Achieved» will appear. Click the **OK** button.

4. Move the AFM head to its inoperative position by clicking the [STM AFM] button in the Measurement Heads Control panel.

5. Place two metal substrates in stack on the sample holder (Fig. 7-9).

Fig. 7-9. Two metal substrates placed in stack

6. Move the scanner upwards so as the substrate surface comes to the focus of the videomicroscope (see Fig. 7-10). To this purpose, click the [Move] button. As the sample reaches the focus of the videomicroscope, stop movement by clicking the button again [Move].

ATTENTION! Focusing of the videocamera on the cantilever edge should be as sharp as possible. If the focus plane is shifted, calibration parameters of the aiming system can take incorrect values.
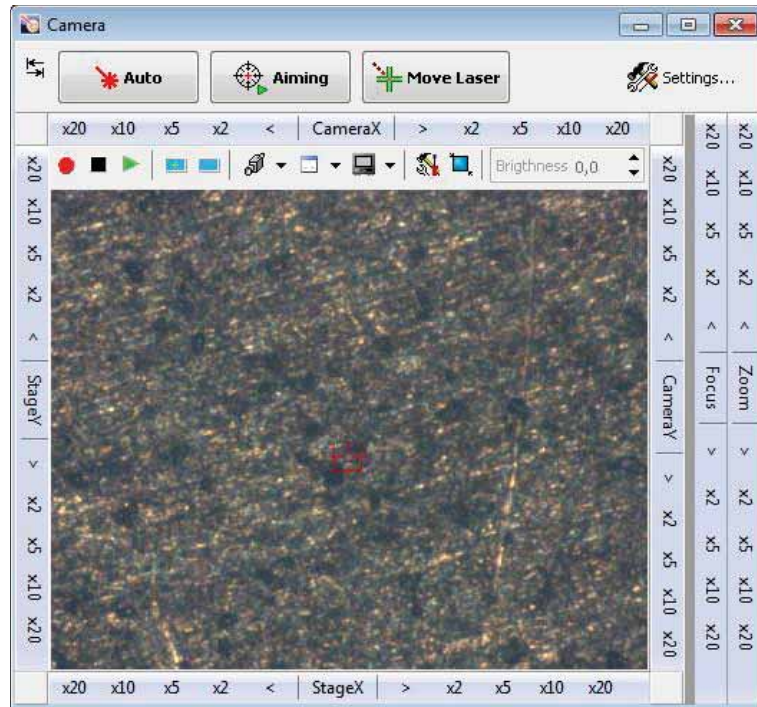
Fig. 7-10 Surface of the metal substrate in focus of the videomicroscope

7. Using controls of the **Zoom** panel, reduce zoom of the videocamera t its minimum.

8. If the laser is off, activate it by clicking the ![button] button.

   NOTE. If the protective shield is raised, the laser will be disabled even with the activation button being pressed in ![button].

9. Open the **Aiming Movers** window by clicking the button ![Aiming] . Using controls of the panels **Laser X** and **Laser Y**, move the laser so as the laser spot comes to the left bottom of the videoimage (Fig. 7-11).
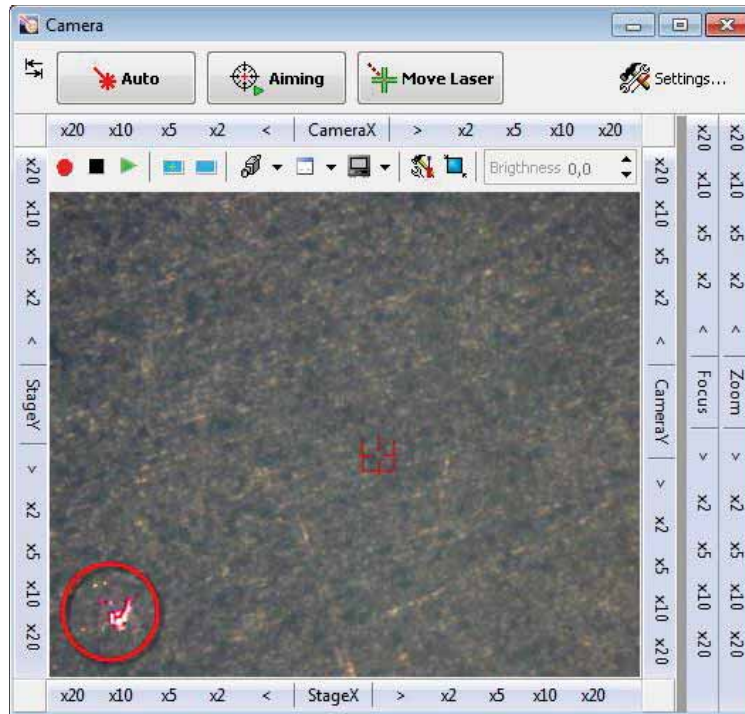
Fig. 7-11. Laser spot at the left bottom corner of the videoimage

10. In the **Step 2** panel of the **Video Calibration** window (Fig. 7-7) click the **OK** button. A request for pointing to the laser spot in the videoimage will appear (Fig. 7-12), close this box by clicking the **OK** button.
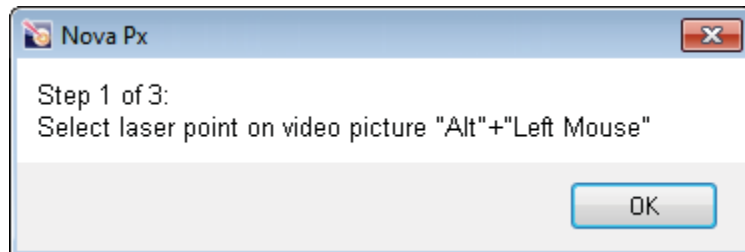

Fig. 7-12

11. Define position of the laser spot in the videoimage by clicking on a relevant location with the <Alt> key being pressed. This will display a cross-hair at the position of the laser spot, stepper motor of the laser carriage will start, and the laser will move to reposition the laser spot somewhere close to the center of the videoimage (Fig. 7-13).
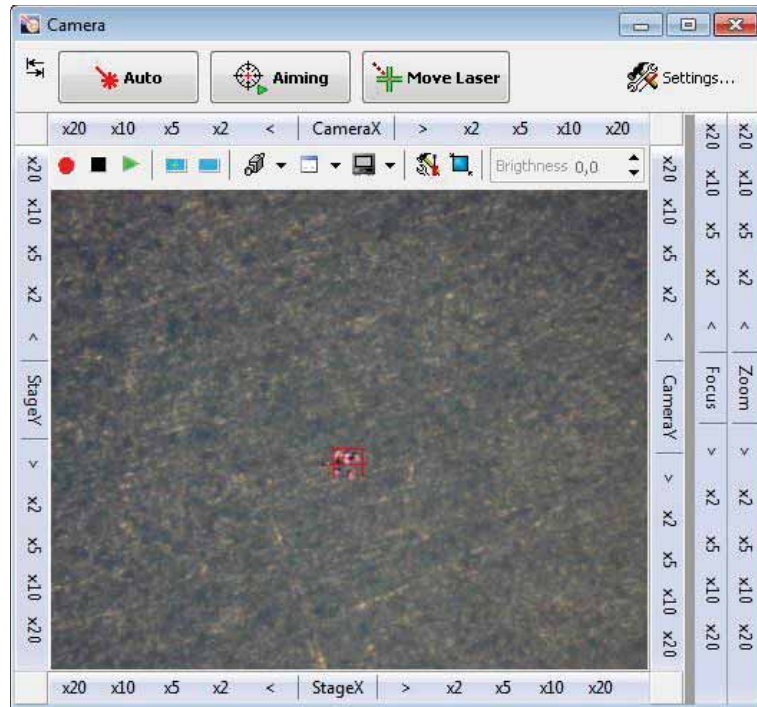
Fig. 7-13. Laser spot close to the center of the videoimage

As the laser carriage stops, the request on pointing to the laser spot in the videoimage will appear again (Fig. 7-14); close this box by clicking the **OK** button.
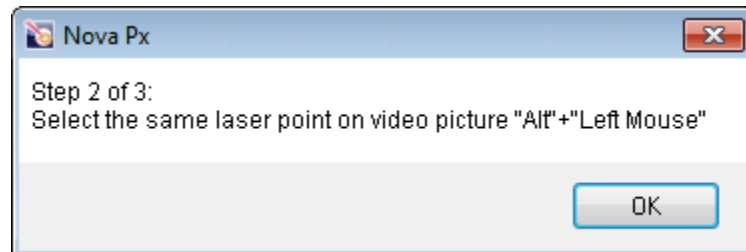

Fig. 7-14

12. Define position of the laser spot in the videoimage again by clicking on a relevant location with the <Alt> key being pressed. A cross-hair marker will appear at the center of the videoimage. As well, a request on moving the laser for centering the laser spot will appear (Fig. 7-16), close this box by clicking the **OK** button.
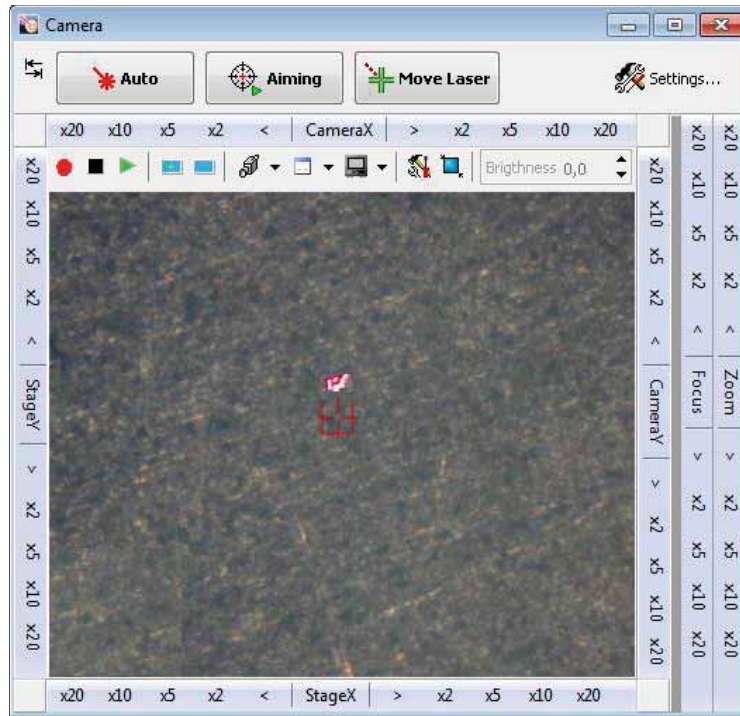
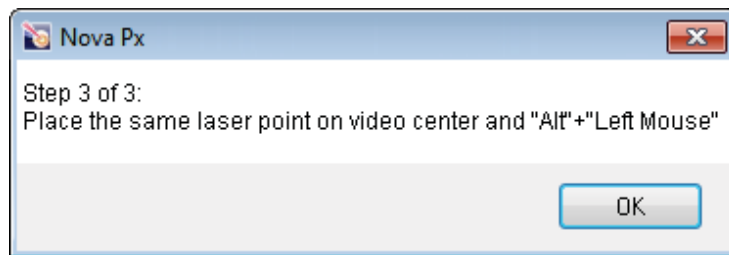Fig. 7-15. Cross-hair at the center of the videoimage

Fig. 7-16

13. Using controls in the panels **LaserX** and **LaserY** of the **Aiming Movers** window, move the laser so as the laser spot comes to the center of the cross-hair.

14. Define position of the laser spot in the videoimage by clicking on a relevant location with the <Alt> key being pressed. This will open a Message box (Fig. 7-17), close this box by clicking the **OK** button.
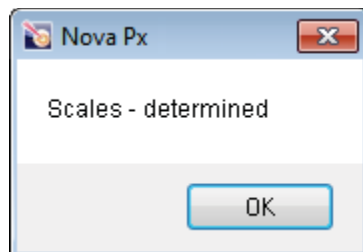

Fig. 7-17

This completes calibrating stepper motors for aiming the laser beam at the cantilever.

Step 3. Calibrating Stepper Motors of Sample Positioning System

1. Using controls of the **Zoom** panel, reduce zoom of the videomicroscope to its minimum.

2. In the **Step 3** panel of the **Calibration** tab (Fig. 7-7), click the [Ok] button. A request on pointing to a relevant point in the videoimage will appear (Fig. 7-18), close this box by clicking the **OK** button.
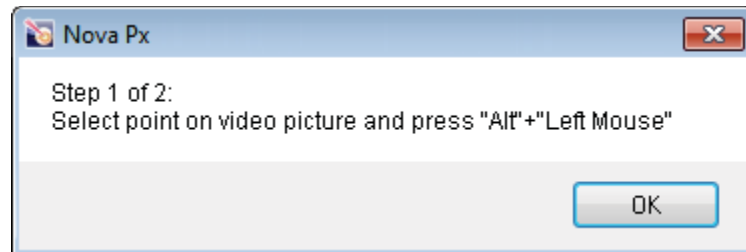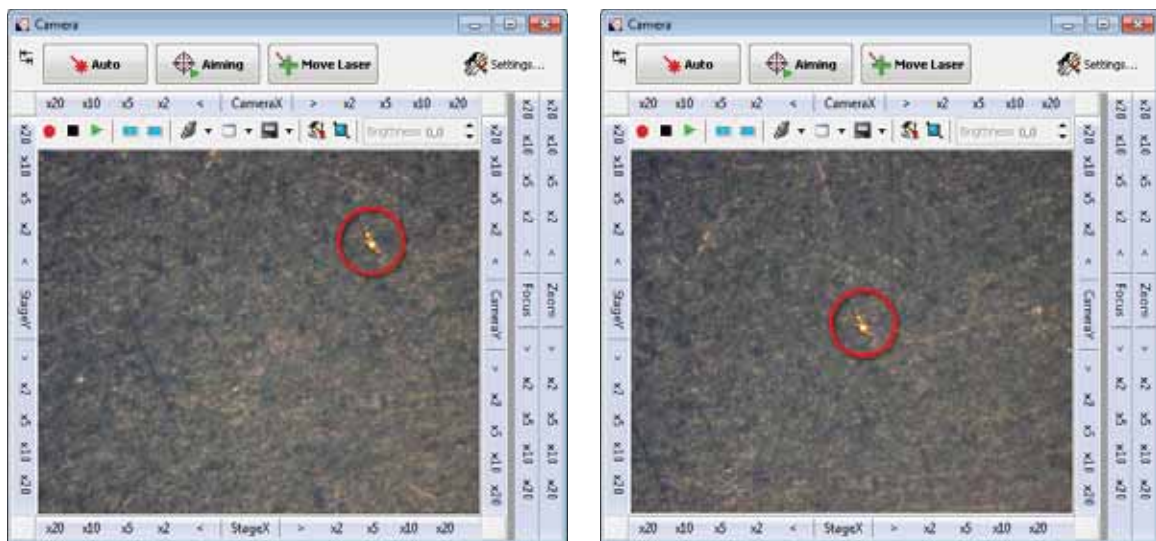

Fig. 7-18

3. Select a well resolved feature at the right top of the sample surface image. Click on a relevant point with the <Alt> key being pressed. This will start stepper motors of the XY sample carriage and the carriage will move so as the selected point is at the center of the videoimage (Fig. 7-19, b).

a) before repositioning   b) after repositioning

Fig. 7-19. Selecting feature on the sample surface

4. When the sample carriage stops, a request on pointing to a desired point will appear again (Fig. 7-20). Close this box by clicking the **OK** button.
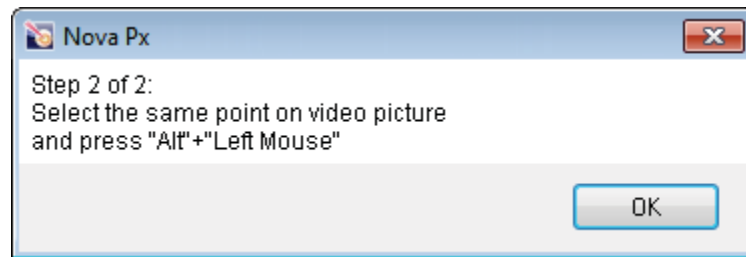

Fig. 7-20

5. Define position of the selected point in the videoimage again by clicking on a relevant location with the <Alt> key being pressed.

6. This will open a Message box (Fig. 7-21), close this box by clicking the **OK** button.
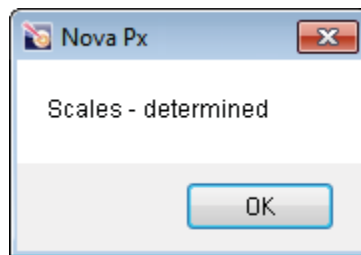

Fig. 7-21. Message box

This completes calibration of the sample positioning system.