# PART 3. Atomic Force Microscopy

## 1. PREPARATION FOR OPERATION .............................................................. 3-3

1.1. **Basic Procedure of the Instrument Preparation for the Operation** ............... 3-3
1.2. **Electromechanical Configuration** ................................................. 3-3
    1.2.1. "Scanning-by-sample" Configuration ......................................... 3-4
    1.2.2. "Scanning-by-probe" Configuration ........................................ 3-5
    1.2.3. Connection Used with Closed-Loop Scanner Equivalent .................. 3-6
    1.2.4. Deactivating Scanning Axes .................................................... 3-7
1.3. **Instrument Turning On** .................................................................. 3-7
1.4. **Loading Scanner Calibration Parameters** ...................................... 3-8
1.5. **Preparation of the Instrument for "Scanning-by-sample" Configuration** .... 3-10
    1.5.1. Installing the Probe ................................................................. 3-10
    1.5.2. Adjusting the System for Detecting the Cantilever Deflections .......... 3-13
        1.5.2.1. General information about the laser beam alignment onto the cantilever .. 3-13
        1.5.2.2. Laser Beam Alignment onto the Cantilever ............................ 3-15
        1.5.2.3. Accurate alignment of the detection system .......................... 3-18
    1.5.3. Centering the scanner .................................................................. 3-19
    1.5.4. Mounting the Sample ................................................................. 3-20
    1.5.5. Installing the Measuring Head .................................................... 3-26
    1.5.6. Initial Approach ...................................................................... 3-26
    1.5.7. Installation of a Protective Hood ............................................... 3-26
    1.6.1. Installing the Probe ................................................................. 3-28
    1.6.2. Adjusting the Optical System for Detecting the Cantilever Deflections 3-30
        1.6.2.1. General information about the laser beam alignment onto the cantilever .. 3-30
        1.6.2.2. Laser Beam Alignment onto the Cantilever ............................ 3-32
        1.6.2.3. Accurate alignment of the detection system .......................... 3-35
    1.6.3. Mounting the Sample ................................................................. 3-36
    1.6.4. Installing the Measuring Head .................................................... 3-40
    1.6.5. Initial Approach ...................................................................... 3-41
    1.6.6. Installation of a Protective Hood ............................................... 3-41

## 2. CONTACT ATOMIC FORCE MICROSCOPY ............................................. 3-43

2.1. **Constant Force Mode** .................................................................. 3-43
    2.1.1. Preparations for Measurements ................................................. 3-43
    2.1.2. Set the Electronic Configuration ............................................... 3-44
    2.1.3. Set initial Level for the DFL Signal ........................................... 3-44
    2.1.4. Approach the Sample to the Probe .......................................... 3-45
    2.1.5. Setting the Feedback Gain Factor Working Level ....................... 3-48
    2.1.6. Set parameters of Scanning ...................................................... 3-49
    2.1.7. Scanning ................................................................................... 3-53
    2.1.8. Saving of Measurement Data .................................................... 3-57
    2.1.9. Completion of Measurements ................................................... 3-57
2.2. **Lateral Force Microscopy** ....................................................... 3-58
    2.2.1. Brief Description of the Mode ................................................... 3-58
    2.2.2. Preparation for Measurements ................................................. 3-59
    2.2.3. Scanning ................................................................................... 3-60
2.3. **Constant Height Mode** .......................................................... 3-60
    2.3.1. Brief Description of the Mode ................................................... 3-60
    2.3.2. Preparation for Measurements ................................................. 3-62
    2.3.3. Setting of Parameters ............................................................... 3-62
    2.3.4. Scanning ................................................................................... 3-63
2.4. **Spreading Resistance Imaging** .................................................. 3-63
    2.4.1. Brief Description of the Mode ................................................... 3-63
    2.4.2. Preparation for Measurements ................................................. 3-64
    2.4.3. Setting of Parameters ............................................................... 3-65
    2.4.4. Scanning ................................................................................... 3-66
    2.4.5. Modes of Improving Image Quality ........................................... 3-67
Performing measurements

2.5. FORCE MODULATION MODE ................................................................. 3-67
  2.5.1. Brief Description of the Mode ................................................... 3-67
  2.5.2. Preparation for Measurements .................................................. 3-68
  2.5.3. Setting of Parameters ............................................................. 3-68
  2.5.4. Scanning ............................................................................. 3-70
  2.5.5. Modes of Improving Image Quality ........................................ 3-71

2.6. CONTACT ERROR MODE ................................................................. 3-72
  2.6.1. Brief Description of the Mode ................................................... 3-72
  2.6.2. Preparation for Measurements .................................................. 3-72
  2.6.3. Setting of Parameters ............................................................. 3-73
  2.6.4. Scanning ............................................................................. 3-73

2.7. PIEZORESPONSE FORCE MICROSCOPY ........................................... 3-74
  2.7.1. Brief Description of the Mode ................................................... 3-74
  2.7.2. Preparation for Measurements .................................................. 3-76
  2.7.3. Setting of Parameters ............................................................. 3-77
  2.7.4. Scanning ............................................................................. 3-80

3. SEMICONTACT ATOMIC FORCE MICROSCOPY ..................................... 3-82
  3.1. SEMICONTACT MODE ................................................................. 3-82
   3.1.1. Preparation for Measurements ............................................... 3-82
   3.1.2. Set the Electronic Configuration ............................................ 3-83
   3.1.3. Set the Piezodriver Operating Frequency ................................ 3-83
   3.1.4. Set Initial Level for the Signal Mag ....................................... 3-87
   3.1.5. Approach the Sample to the Probe ........................................ 3-88
   3.1.6. Set the Feedback Gain Factor Working Level ......................... 3-90
   3.1.7. Set parameters of Scanning .................................................. 3-92
   3.1.8. Scanning ............................................................................ 3-95
   3.1.9. Saving of Measurement Data .................................................. 3-99
   3.1.10. Completion of Measurements ............................................... 3-99

  3.2. SEMICONTACT ERROR MODE ...................................................... 3-100
   3.2.1. Brief Description of the Mode ................................................ 3-100
   3.2.2. Preparation for Measurements ............................................... 3-101
   3.2.3. Setting of Parameters .......................................................... 3-102
   3.2.4. Scanning ............................................................................ 3-102

  3.3. PHASE IMAGING MODE .............................................................. 3-103
   3.3.1. Brief Description of the Mode ................................................ 3-103
   3.3.2. Preparation for Measurements ............................................... 3-104
   3.3.3. Setting of Parameters .......................................................... 3-105
   3.3.4. Scanning ............................................................................ 3-106
   3.3.5. Modes of Improving Image Quality ........................................ 3-107

4. AFM SPECTROSCOPIES ........................................................................ 3-108
  4.1. INTRODUCTION ........................................................................... 3-108
  4.2. FORCE-DISTANCE SPECTROSCOPY DFL(HEIGHT) ....................... 3-108
   4.2.1. Basic Operations ................................................................... 3-108
   4.2.2. Switching to Spectroscopy Tab .............................................. 3-109
   4.2.3. Selecting the Function to be Measured ................................... 3-109
   4.2.4. Selecting Spectroscopy Points .............................................. 3-111
   4.2.5. Starting the Measurements .................................................... 3-113
   4.2.6. Viewing Spectroscopy Data ................................................... 3-114
   4.2.7. Calculating Adhesion Force .................................................. 3-116

  4.3. CURRENT SPECTROSCOPY EPR-LOW(BIAS VOLTAGE) ............... 3-118
   4.3.1. Configuring and Making Measurements ................................. 3-118
   4.3.2. Viewing Spectroscopy Data ................................................... 3-120

  4.4. AMPLITUDE SPECTROSCOPY MAG(HEIGHT) ................................ 3-122
   4.4.1. Configuring and Making Measurements ................................. 3-122
   4.4.2. Viewing Spectroscopy Data ................................................... 3-123
   4.4.3. Calibration of Cantilever Oscillations Amplitude ...................... 3-125

  4.5. PHASE SPECTROSCOPY PHASE(HEIGHT) ..................................... 3-126
   4.5.1. Configuring and Making Measurements ................................... 3-126
   4.5.2. Viewing and Processing Spectroscopy Data ............................. 3-128
PART 3. Atomic Force Microscopy

1. Preparation for Operation

This section describes general preparation procedures with the instrument to perform AFM measurements. These procedures are mandatory during the instrument preparation for operation using any AFM technique.

1.1. Basic Procedure of the Instrument Preparation for the Operation

Preparation of the instrument for operation using AFM modes generally can be divided into the following basic operations:

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1.</td>
<td>Electromechanical Configuration (see page 3-3).</td>
</tr>
<tr>
<td>Step 2.</td>
<td>Instrument Turning on (see page 3-7).</td>
</tr>
<tr>
<td>Step 3.</td>
<td>If Equivalent Scanner is used, follow the instruction in the Appendix to prepare it (see i. “Preparing the Scanner Equivalent for Operation”). In this case the 4-th step should be omitted.</td>
</tr>
<tr>
<td>Step 4.</td>
<td>Loading Scanner Calibration Parameters (see page 3-8).</td>
</tr>
</tbody>
</table>
| Step 5. | Preparation the instrument:  
- Preparation of the Instrument for "Scanning-by-sample" Configuration (see page 3-10);  

1.2. Electromechanical Configuration

⚠️ ATTENTION! All commutations during the setting of a configuration are performed with the electrical power switched off.

The instrument completed with all components, parts and accessories allows the user to choose one of three configurations:

1. **Scanning-by-sample** configuration, including a “universal measuring head”, the base unit and a scanner.
2. **Scanning-by-probe** configuration, including a scanning head and the base unit with a sample holder.

3. **DualScan** configuration, including a scanning head, the base unit and a scanner.

### 1.2.1. “Scanning-by-sample” Configuration

In this configuration:

1. The “universal measuring head” is installed onto the auxiliary table. The head cable is connected to any of the two **HEAD** sockets located on the instrument base unit (Fig. 1-1).

![Fig. 1-1](image)

2. The chosen scanner is inserted into the positioner on the exchangeable mount (Fig. 1-2, Fig. 1-3).
3. If a scanner without sensors is used, the scanner connector is plugged into the **SCANNER** socket or into the **SCAN+SENSOR** socket, located on the base unit. If a scanner with sensors is used, the scanner connector is plugged into the **SCAN+SENSOR** socket.

### 1.2.2. “Scanning-by-probe” Configuration

In this configuration:

1. The scanning head is installed onto the auxiliary table (Fig. 1-4). The **HEAD** cable is connected to any of the two **HEAD** sockets located on the instrument base unit (Fig. 1-4).

![Figure 1-4](image)

2. If a scanner without sensors is installed in the scanning head, then the cable of the measuring head with **SCANNER** connector is plugged into the **SCANNER** socket (Fig. 1-4, Fig. 1-7) or to the **SCAN+SENSOR** (Fig. 1-7) socket. If a scanner with sensors is used, the scanner connector is plugged into the **SCAN+SENSOR** socket.

3. A carriage with the sample holder is installed into the positioning device (Fig. 1-5, Fig. 1-6).
1.2.3. Connection Used with Closed-Loop Scanner Equivalent

If the scanner is used without sensors, then in order to improve the measurements quality, a Closed-Loop Scanner Equivalent can be installed additionally:

1. The Closed-Loop Scanner Equivalent (CLE) is placed near to the base unit. The feedthrough connector of the CLE is connected to CONTROLLER 2 socket located on the instrument base unit (Fig. 1-7).

2. The Closed-Loop Scanner Equivalent controller cable is connected from above to the CLE socket (Fig. 1-8).
1.2.4. **Deactivating Scanning Axes**

When using **Double scanning** configuration, scanning can be performed in three different ways:

- simultaneous scanning with both scanners along all three axes;
- deactivate scanning along axis $Z$ of one of the scanners;
- deactivate scanning along axes $X, Y$ of one of the scanners.

Scanning deactivation is made using special adapters $Z$-OFF or $X, Y$-OFF (Fig. 1-9).

![Fig. 1-9. Adapters to deactivate scanning along axes XY and Z](image)

To deactivate scanning along specific axes the scanner is connected through the corresponding adapter.

### 1.3. **Instrument Turning on**

1. The program is launched by double-clicking on the program icon (shortcut) on the desktop. (The program can be started also launching the NOVA.exe file, located in the program directory).

   The program window will be displayed in the PC monitor.

2. Turn on the SPM controller with the power switch located on the front panel.

   If turning on is successful, a green "tick" appears in the monitor screen bottom left corner.

3. Turn on the vibration isolation system.
1.4. **Loading Scanner Calibration Parameters**

Upon starting the program the **Default.par** file which contains calibration parameter for a specific scanner is loaded by default. If only one scanner is supplied with the instrument then the **Default.par** file contains the parameters for this particular scanner.

If several scanners are included in the package then **Default.par** stores parameters corresponding to one of the scanners.

After changing the scanner the related parameter file (par-file) shall be loaded.

**To load a par-file for the scanner complete the following operations:**

1. In the Main menu select successively the following commands **Settings → Calibrations → Load Calibrations** (Fig. 1-10).

![Fig. 1-10](image)

This will open a dialog box with a list of par-files contained in the **PARFiles** folder as in the example shown in Fig. 1-11.

**The names of the par-files have the following pattern:**

- **SmXXXcl.par** – for scanners installed in measuring heads;
- **zXXXcl.par** – for exchangeable scanners;
- **where XXX** – is the scanner number;
- **cl** – denotes the presence of capacitance displacement sensors.

![Fig. 1-11](image)
PART 3. Atomic Force Microscopy

2. Choose the par-file corresponding to the installed scanner.

3. Click the **Open** button to load the par-file. As a result the scanner calibration parameters will be loaded.

When a par-file for the scanner with sensors is loaded, it results in the following:
- The feedback controlling the scanner displacements in the XY-plane will be on;
- The software nonlinearity correction will switch off.

If you prefer the current scanner parameters to load by default at the program start-up save the file as **Default.par**. Proceed as follows:

1. In the Main menu select the commands: **Settings → Calibrations → Save Calibrations** (Fig. 1-12).

2. The **Save As** dialog box opens (Fig. 1-13). Save the file as **Default.par**.

**NOTE.** 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. The detailed description of this procedure is given in Appendix (see i. "Removing Generation").
Performing measurements

1.5. Preparation of the Instrument for "Scanning-by-sample" Configuration

The general procedure consists of the following sequence of basic operations:

1. Installing the Probe (see i. 1.5.1 on page 3-10).
2. Adjusting the System for Detecting the Cantilever Deflections (see i. 1.5.2 on page 3-13).
3. Centering the scanner (see i. 1.5.3 on page 3-19).
4. Mounting the Sample (see i. 1.5.4 on page 3-20).
5. Installing the Measuring Head (see i. 1.5.5 on page 3-26).
6. Initial Approach (see i. 1.5.6 on page 3-26).
7. Installation of a Protective Hood (see i. 1.5.7 on page 3-26).

More detailed description of the basic operations and procedures listed above is presented below.

1.5.1. Installing the Probe

It is assumed that the universal measuring head is installed onto the auxiliary table and is connected to the base unit (Fig. 1-14).

To install or replace the probe, the measuring insert should be removed and put near the head without disconnecting the cable as shown in Fig. 1-15. To remove the measuring insert, release the fastening clip by turning its knob clockwise (Fig. 1-14).

If the measuring insert is not installed on the measuring head:

1. Choose the measuring insert needed for your measurements.
2. Turn the measuring insert over and place it near the measuring head (Fig. 1-15).
3. Connect the measuring insert cable to the measuring head socket (remove the socket cap beforehand).
To install or replace the probe, do the following:

1. Release the probe clip spring. To do this, turn downward the trapezoidal lever, located on the outer side of the holder body, with a sharp tweezers (Fig. 1-16).

![Fig. 1-16](image1)

![Fig. 1-17](image2)

2. Take the probe from the box with the tweezers (Fig. 1-17) making sure that the working side of the chip with cantilevers is directed to you during the installation. Do not turn the chip over because probes in the box are placed with their tips pointing upwards.

3. Place the probe on the sapphire pedestal to the left of the working position (Fig. 1-18, Fig. 1-19) which is in the right corner under the clip.

![Fig. 1-18](image3)

![Fig. 1-19](image4)

4. Move the probe with the tweezers to the working position (Fig. 1-20) as shown schematically in Fig. 1-19. The probe working position is shown in Fig. 1-21.
Performing measurements

5. Once the probe is in working position, clamp it with the clip. To do this, turn the lever to the holding position using the tweezers (Fig. 1-22).

6. Install the measuring insert into the measuring head (Fig. 1-23). To do this:
   a. Hold-down knob 3 (Fig. 1-21) should be loosen by turning its handle clockwise;
   b. Install the measuring insert so that the supporting balls (Fig. 1-24) rest on the sapphire platforms of the measuring head (Fig. 1-23); adjustment screws must rest upon insert respective seats.
   c. Clip the measuring insert to the adjustment screws by turning the down-hold knob counterclockwise.
1.5.2. Adjusting the System for Detecting the Cantilever Deflections

1.5.2.1. General information about the laser beam alignment onto the cantilever

The purpose of the laser beam aligning is to steer the laser spot onto the cantilever's end as shown schematically in Fig. 1-26.

The design of the universal measuring head allows to move the probe, mounted on the measuring insert, with respect to the laser beam using the screws 1 and 2 (Fig. 1-25, Fig. 1-26). Thus, the laser beam alignment means moving the cantilever with respect to the fixed beam spot.

For convenience, hereinafter, "the laser beam move relative to the cantilever" or "the laser spot steers onto the cantilever" means that the laser beam is actually fixed and the cantilever is moved.
Performing measurements

Setting-up of the detection system is performed by means of the control program.

Laser is switched on and off by the button which is located to the right in the main parameters panel (Fig. 1-27). The laser is automatically switched on upon starting the control program.

ATTENTION! Do not stare into the laser beam! (The laser safety class of this laser warrant a safe contact between the beam and human eye during the period of time corresponding to a normal human reaction to irritation (about 0.25 s in this case). This time is enough for a person to blink and turn away. Extended exposure can lead to short-term eyesight disorder).

Use the tab Aiming for setting the detection system up (Fig. 1-28) (this tab is enabled by clicking the button on the Main Operations Panel).

The panel of the detection system adjustment contains an indicator of the laser spot position relative to the photodiode segments (left side) and a panel for displaying the photodiode signals current values.
The displayed signals have the following meaning:

**DFL**  the difference signal between top and bottom halves of the photodiode;

**LF**  the difference signal between left and right halves of the photodiode;

**Laser**  the total signal that comes from all four segments of the photodiode that is proportional to the intensity of the laser radiation reflected from the cantilever.

### 1.5.2.2. Laser Beam Alignment onto the Cantilever

1. Trace the laser beam path with respect to the cantilever. View the image of the laser beam passing through the scanning head. For that, lift the scanning head approx. 10-15 cm above a sheet of white paper serving as a screen (Fig. 1-29).

![Fig. 1-29](image)

Three cases are possible:

a. **Undistorted** image (circular spot) on paper. (Fig. 1-30) This means that the laser beam is off the cantilever or the cantilever chip.

b. **Distorted** laser image on paper (see examples in Fig. 1-31). This means that the laser beam hits the cantilever or the edge of some other element. Note that image shape can be different from the one shown in Fig. 1-31.

c. No image at all. This means that the laser beam falls onto the chip or the probe holder (Fig. 1-32) and cannot pass through the measuring head.
Performing measurements

The design of the measuring head allows to visually observe the cantilever. Thus, it is possible to determine approximately position of the laser beam with respect to the cantilever.

2. Rotating the screw 2 (Fig. 1-33) make the laser spot appear undistorted. In general the laser spot will appear in position 1 (see Fig. 1-34).
3. Rotating the screw 2, move the beam perpendicularly to the front edge of the chip (1→2 in Fig. 1-34) until the laser spot becomes distorted.

![Diagram](image)

Fig. 1-34. Moving the laser spot (general case)

4. Rotating the screw 1, move the beam parallel to the front edge of the chip. Two variants are possible:
   a. The laser spot moves along the edge of the holder (2→3): in this case the spot will disappear when it hits the chip;
   b. The laser spot moves along the edge of the chip (4→5): in this case when it hits the cantilever, an interference pattern will appear (Fig. 1-35). The laser beam is located at the cantilever base.

5. When the spot disappears, rotate the screw 2, moving the laser beam towards the end face of the chip (3→4), until the laser spot appears. Now the laser beam is at the edge of the chip (position 4).

6. Rotating the screw 1, move the beam along the front edge of the chip (4→5) until an interference pattern appears (Fig. 1-35). The laser beam is at the cantilever base.

7. Move the laser beam, rotating screw 1, 2, in the direction of the cantilever free end.

![Images](image)

a) a rectangular cantilever  
b) a triangular cantilever (laser is on one of the cantilever ribs)  
c) a triangular cantilever, (laser is on the the vertex of triangle)

Fig. 1-35. The spot image at the laser beam hitting the cantilever
1.5.2.3. **Accurate alignment of the detection system**

When the laser beam is pointed onto the cantilever, some values different from zero appear on the photodiode indicator (Fig. 1-36).

During the adjustment it is necessary that the laser beam, being reflected from the cantilever, hits the central part of the photodiode, equally illuminating all four segments of the photodiode.

![Fig. 1-36](image)

**Fine adjustment of the detection system is performed as follows:**

1. Rotate photodiode adjustment screws 3 and 4 (Fig. 1-33) to center the laser spot on the photodiode indicator (Fig. 1-37). The values of DFL and LF signals shall be close to zero while the magnitude of the total Laser signal shall remain rather large.

![Fig. 1-37](image)

When adjusting the photodiode position it is important to make sure that turning the photodiode vertical displacement screw causes the vertical motion of the laser beam and similarly for the horizontal screw. If the laser beam moves arbitrarily and the Laser signal decreases to zero when turning any of the photodiode adjustment screws then it means that the laser beam hits the photodiode edge. In this event the corresponding screw shall be turned in the opposite direction.
2. Verify that the laser beam really hits the cantilever. A characteristic signature of laser beam hitting s the cantilever is a Laser signal decreasing during rotation of the screws 1 and 2 first in one direction and then in opposite direction.

3. Perform precise pointing of the laser beam onto the cantilever using the measured values of the photodiode signals. For that, turning the measuring insert X, Y screws 1 and 2, slightly move the beam with respect to the cantilever, adjusting to the maximal value of the photodiode signal (Laser). The Laser signal value should be within 20-50 nA range.

4. Upon laser readjustment repeat the photodiode alignment procedure with photodiode adjustments 3, 4 because of the beam possible shifting from the center.

1.5.3. Centering the scanner

After the measuring insert has been adjusted and the photodiode has been aligned, but prior to sample mounting, it is recommended to align the scanner with the measuring head in such a way that the probe is positioned on the scanner axis. This would reduce any undesired tilting of the surface that takes may occur during scanning if the tip is displaced from the scanner axis.

NOTE. This procedure is optional.

Scanner centering procedure:

1. Turning the approach knob (Fig. 1-39) clockwise move the positioning system in its lowermost point.

2. Place the measuring head with its legs onto the seats of the exchangeable mount (Fig. 1-38), the measuring insert facing the operator (Fig. 1-39).
3. Watch the head from the top and, using the micrometer screws of the positioner, shift the scanner so that the probe is located at the scanner axis (Fig. 1-40).

![Fig. 1-40. The hole which is situated on the scanner axis](image)

4. Remove the measuring head from the base unit and placed it on an auxiliary table.

### 1.5.4. Mounting the Sample

Scanning-by-sample configuration allows to operate with samples satisfying the following conditions:

- Diameter  - up to 40 mm.
- Height      - up to 15 mm.
- Weight      - up to 100 g.

**Mounting the sample on the scanner without sensors**

Samples should be fixed to special polycrystalline sapphire substrates, which are contained in the hardware set of the instrument. Dimensions of these substrates are 24x19x0.5 mm. Fastening of the sample to the substrate can be done by using double-sided scotch tape or glue.

**NOTE.** Any plate of 0.5 mm thickness can be used as an adapter substrate. This plate should be wide enough to allow the spring clips of the sample holder (or the sample stage) to retain it firmly.

The substrate SU001 is recommended to use with small size samples (less than 10÷12 mm diameter). The substrate SU002 is recommended to use with bigger size samples (greater than 10÷15 mm diameter).
NOTE. To mount large samples, use the substrate SU001 with an about 2mm thick adapter plate glued to it. Thickness of the plate should be big enough to raise the sample above the spring clips of the sample stage, while its width should be less than the distance between the clips. The sample is fixed to the adapter plate.

When performing the techniques that require electrical contact between the sample and the instrument, it is recommended to use the substrate SU015 equipped with a spring contact device (Fig. 1-43).

ATTENTION! When performing measurements on thick samples, correct software settings for their height. Default settings are made for 0.5 mm height (this procedure is addressed in more detail on page 3-24).

When mounting the substrate with samples on the sample stage, the substrate is inserted under the spring clips from the side of two of the support balls. The clips press the top of the substrate down, while its bottom surface rests on the three support balls (Fig. 1-44).
Mounting the sample on the scanner with sensors

A special metal substrate is used to mount samples on scanners with built-in sensors (Fig. 1-45). Fastening of the sample to the substrate can be done by using double-sided scotch tape or glue.

The polycrystalline sapphire substrates SU001, SU002, SU0015 can also used to mount samples (see page 3-20). A special sample stage is used to mount these substrates on the scanner with built-in sensors (Fig. 1-46). The substrate is inserted under the spring clips from the side of two of the support balls. The clips press the top of the substrate down, while its bottom surface rests on the three support balls (Fig. 1-47).
ATTENTION! When performing measurements on thick samples, correct software settings for their height. Default settings are made for 0.5 mm height (this procedure is addressed in more detail on page 3-24).

The metal substrate (or the sample stage) is mounted on the magnetic holder of the scanner (Fig. 1-48). The substrate (or the sample stage) should be placed at the center of the magnetic holder and it should rest on the three support balls.

Fig. 1-48. Mounting the sample on the scanner with built-in sensors

**Specifics of mounting the sample on the substrate with the spring contact**

1. Mount the sample on the substrate (using double-sided scotch tape, for example).
2. Use tweezers to twist the contact spring so that it could touch the sample edge so that the center of the sample remains free (Fig. 1-49).

Fig. 1-49. Substrate with the spring contact

3. Insert the substrate into the sample holder so that the contact spring is on the farther side from the operator (the details of mounting the substrate to the sample holder see on pages 3-20, 3-22).
4. Connect the substrate with the bias voltage input socket (BV) 1 (Fig. 1-50), or with the grounding terminal 2.
Performing measurements

ATTENTION! Do not use the BV socket for grounding the sample as the signal BiasVoltage is simultaneously applied both to the probe and to the BV socket.

Setting the sample height value

Height of the sample above the scanner surface must be taken into account for more accurate results of scanning.

Before setting the height value, measure the overall thickness of the sample and the substrate. Then subtract thickness of the substrate that has been used for calibration. The resulting value should be used in the computer program as the value of the sample height.

To set the sample height value, perform the following steps:

1. Open the dialog box Scanner Calibration Setup (Fig. 1-51) by selecting the sequence Settings → Calibrations → Change Calibrations from the Main menu.
2. Set the sample height value in the text box Sample Height in either of the tabs (Fig. 1-51).
3. Click OK to save alterations and to close the dialog box.

When mounting another sample of a different thickness, change its height value in the text box Sample Height in the dialog box Scanner Calibration Setup.

When operating on samples of the same thickness, the value of thickness can be written into file Default.hrd in order to use it on the startup of the program automatically. File Default.hrd is stored in folder HRD.

**To set the sample height value in file Default.hrd, perform the following steps:**

1. Open file Default.hrd using any text editor.
2. Alter the line containing the sample height value (or append it if missing) in millimeters. Example: for samples of 1.5 mm height this line is Scanner_SampleHeight=1.5.
3. Save alteration to file Default.hrd.

Once this is done, the default value of the sample height loaded on the program startup is equal to the value written in this line.
1.5.5. Installing the Measuring Head

Before installing the measuring head it’s necessary to estimate the difference between the probe and the surface of the sample after the measuring head will be installed. It’s necessary to make sure that this distance is enough (not less than 1-2 mm) in order to avoid damaging the probe and the surface of the sample. If the distance is less than 1-2 mm, lower the sample by winding the approach knob.

1. Place the measuring head with its legs onto the seats of the exchangeable mount, the stage facing the operator (Fig. 1-52).

![Fig. 1-52. Installation of the measuring head.](image)

1 – approach knob; 2 – cable holder of the measuring head

2. Insert the cable of the head in the cable holder (Fig. 1-52).

1.5.6. Initial Approach

1. Looking from a side and turning the approach knob (pos.1 in Fig. 1-52) counterclockwise approach the sample to the probe at the distance of 0.5-1 mm.

2. Looking from above, make sure the probe is properly positioned with respect to the sample. Looking from the side, make sure the probe will not touch anything during the next approaching.

3. Check the adjustment of the detection system for detecting the cantilever deflections using the photodiode indicator and readjust if needed.

1.5.7. Installation of a Protective Hood

It is recommended to work with a hood in the following cases:

- if it is necessary to obtain a high resolution image in XY plane or along Z axis;
- when carrying out measurements at controlled temperature;
- for protection against temperature drifts;
- for reduction of acoustic noise level.
For installation of the protective hood proceed as follows:

1. Turn the videomicroscope support clockwise against the stop.
2. Take the protective hood with your left hand using handle 1, and with your right hand - using handle 2 (Fig. 1-53).

3. Bring the hood to the stand so that plastic inserts on handle 1 of the hood (see pos. 3 in Fig. 1-53) rest against the stand, and the slot in handle 2 is located above the base unit frame (Fig. 1-54). Put on the hood so that the frame gets into the slot. The plastic inserts should slide along the stand.

The general procedure of preparation and installation of the “scanning head” and a sample consists of the following sequence of basic operations:

1. Installing the Probe (see i. 1.6.1 on page 3-28).
2. Adjusting the Optical System for Detecting the Cantilever Deflections (see i. 1.6.2 on page 3-30).
3. Mounting the Sample (see i. 1.6.3 on page 3-36).
4. Installing the Measuring Head (see i. 1.6.4 on page 3-40).
5. Initial Approach (see i. 1.6.5 on page 3-41).
6. Installation of a Protective Hood (see i. 1.6.6 on page 3-41).

These operations are described in detail below.

### 1.6.1. Installing the Probe

To install or replace the probe, do the following:

1. Turn the scanning head over and put on the auxiliary stage (auxiliary platform) upside down.

![Fig. 1-55](image)

2. Lift the probe spring clip. To do this, turn downward the trapeziform lever, located on the outer side of the holder body, with the tweezers (Fig. 1-55).

⚠️ **ATTENTION!** Lift the clamping spring only by turning the lever. Do not bend it with the tweezers or by hand, as this can result in irreversible deformation.

⚠️ **ATTENTION!** The probe holder is attached to the scanner, so excessive stress applied to the holder can damage the scanner.
3. Take the probe from the box with the tweezers (Fig. 1-56) making sure that the working side of the chip with cantilevers is directed to you during the installation. Do not turn the chip over because probes in the box are placed with their tips pointing upwards.

![Fig. 1-56](image1)

![Fig. 1-57](image2)

4. Place the probe on the sapphire pedestal to the left of the working position (Fig. 1-57, Fig. 1-58), which is in the right corner under the clip.

![Fig. 1-58](image3)

![Fig. 1-59](image4)

5. Move the probe with the tweezers to the working position (Fig. 1-59) as shown schematically in Fig. 1-57. The probe working position is shown in Fig. 1-60.
Performing measurements

6. Once the probe is in working position, clamp it with the clip. To do this, turn the lever to the holding position using the tweezers (Fig. 1-60).

1.6.2. Adjusting the Optical System for Detecting the Cantilever Deflections

1.6.2.1. General information about the laser beam alignment onto the cantilever

The laser beam is focused in a point on the plane in which the cantilever lies. The beam can be moved relative to the cantilever using the adjustment screws 1 and 2 (Fig. 1-61, Fig. 1-62).

The purpose of the beam aligning is to steer the laser spot onto the cantilever's end using adjustment screws 1 and 2 as shown schematically in Fig. 1-62.
The scanning head is equipped with a special mirror for viewing the cantilever and the sample. The mirror is attached to the scanning head base near the probe holder. Viewing is performed through an opening in the cylinder tip of the scanner at an angle of 30° to the horizontal plane (sample plane) (Fig. 1-63, Fig. 1-64).

Setting-up of the detection system is performed by means of the control program.

Laser switch on and off is performed with the button located to the right in the main parameters panel (Fig. 1-27). The laser is automatically switched on upon starting the control program.

![Fig. 1-65](image)

ATTENTION! Do not stare into the laser beam! (The laser safety class of this laser warrant a safe contact between the beam and human eye during the period of time corresponding to a normal human reaction to irritation (about 0.25 s in this case). This time is enough for a person to blink and turn away. Extended exposure can lead to short-term eyesight disorder).

Use the tab **Aiming** for setting the detection system up (Fig. 1-66) (this tab is enabled by clicking the button on the **Main Operations Panel**).
Performing measurements

The panel of the detection system adjustment contains an indicator of the laser spot position relative to the photodiode segments (left side) and a panel for displaying the photodiode signals current values.

The displayed signals have the following meaning:

- **DFL**: the difference signal between top and bottom halves of the photodiode;
- **LF**: the difference signal between left and right halves of the photodiode;
- **Laser**: the total signal that comes from all four segments of the photodiode that is proportional to the intensity of the laser radiation reflected from the cantilever.

### 1.6.2.2. Laser Beam Alignment onto the Cantilever

1. Trace the laser beam path with respect to the cantilever. View the image of the laser beam passing through the scanning head. For that, lift the scanning head approx. 10÷15 cm above a sheet of white paper serving as a screen (Fig. 1-67).
Three cases are possible

a. **Undistorted** image on paper. (Fig. 1-68) This means that the laser beam is off the cantilever or the cantilever chip;

b. **Distorted** laser image on paper (see examples in Fig. 1-69). This means that the laser beam hits the cantilever or the edge of some other element. Note that image shape can be different from the one shown in Fig. 1-69;

c. No image at all. This means that the laser beam falls onto the chip or the probe holder (Fig. 1-70) and can not pass through the scanning head.
The design of the scanning head allows to visually observe the cantilever. Thus, it is possible to determine approximately position of the laser beam with respect to the cantilever.

**To point the laser beam onto the cantilever perform the following steps:**

2. Rotating the screw 2 (Fig. 1-71) make the laser spot appear undistorted. In general the laser spot will appear in position 1 (see Fig. 1-72).

3. Rotating the screw 2, move the beam perpendicularly to the front edge of the chip (1→2 in Fig. 1-72) until the laser spot becomes distorted.

4. Rotating the screw 1, move the beam in parallel to the front edge of the chip. Two variants are possible:
   a. The laser spot moves along the edge of the holder (2→3): in this case the spot will disappear when it hits the chip;
   b. The laser spot moves along the edge of the chip (4→5): in this case when it hits the cantilever, an interference pattern will appear (Fig. 1-73). The laser beam is located at the cantilever base.
5. When the spot disappears, rotate the screw 2, moving the laser beam towards the end face of the chip (3→4), until the laser spot appears. Now the laser beam is at the edge of the chip (position 4).

6. Rotating the screw 1, move the beam along the front edge of the chip (4→5) until an interference pattern appears (Fig. 1-73). The laser beam is at the cantilever base.

7. Move the laser beam, rotating screw 1, 2, in the direction of the cantilever free end.

![Fig. 1-73. The spot image at the laser beam hitting the cantilever](image)

a) a rectangular cantilever  
b) a triangular cantilever, (laser is on one of the cantilever ribs)  
c) a triangular cantilever, (laser is on the the vertex of triangle)

### 1.6.2.3. Accurate alignment of the detection system

When the laser beam is pointed onto the cantilever, some values different from zero appear on the photodiode indicator (Fig. 1-74).

![Fig. 1-74](image)

During the adjustment it is necessary that the laser beam, being reflected from the cantilever, hits the central part of the photodiode, equally illuminating all four segments of the photodiode.
Fine adjustment of the detection system is performed as follows:

1. Rotate photodiode adjustment screws 3 and 4 (Fig. 1-71) to center the laser spot on the photodiode indicator (Fig. 1-75). The values of DFL and LF signals shall be close to zero while the magnitude of the total Laser signal shall remain rather large.

![Figure 1-75](image)

When adjusting the photodiode position it is important to make sure that turning the photodiode vertical displacement screw causes the vertical motion of the laser beam and similarly for the horizontal screw. If the laser beam moves arbitrarily and the Laser signal decreases to zero when turning any of the photodiode adjustment screws then it means that the laser beam hits the photodiode edge. In this event the corresponding screw shall be turned in the opposite direction.

2. Verify that the laser beam really hits the cantilever. A characteristic signature of laser beam hitting the cantilever is a Laser signal decreasing during rotation of the screws 1 and 2 first in one direction and then in opposite direction.

3. Perform precise pointing of the laser beam onto the cantilever using the measured values of the photodiode signals. For that, turning the measuring insert X, Y screws 1 and 2, slightly move the beam with respect to the cantilever, adjusting to the maximal value of the photodiode signal (Laser). The Laser signal value should be within 20÷50 nA range.

4. Upon laser readjustment repeat the photodiode alignment procedure with photodiode adjustments 3, 4 because of the beam possible shifting from the center.

1.6.3. Mounting the Sample

Scanning-by-probe configuration allows to operate with samples satisfying the following conditions:

- Diameter - up to 100 mm.
- Height - up to 15 mm (up to 50 mm if special legs for measuring head are used).
- Weight - up to 300 g.
Mounting the sample on the sample holder

Samples should be fixed to special polycrystalline sapphire substrates, which are contained in the hardware set of the instrument. Dimensions of these substrates are 24x19x0.5 mm. Fastening of the sample to the substrate can be done by using double-sided scotch tape or glue.

NOTE. Any plate of 0.5 mm thickness can be used as an adapter substrate. This plate should be wide enough to allow the spring clips of the sample holder (or the sample stage) to retain it firmly.

The substrate SU001 is recommended to use with small size samples (less than 10±12 mm diameter). The substrate SU002 is recommended to use with bigger size samples (greater than 10±15 mm diameter).

NOTE. To mount large samples, use the substrate SU001 with an about 2mm thick adapter plate glued to it. Thickness of the plate should be big enough to raise the sample above the spring clips of the sample stage, while its width should be less than the distance between the clips. The sample is fixed to the adapter plate.

When performing the techniques that require electrical contact between the sample and the instrument, it is recommended to use the substrate SU015 equipped with a spring contact device (Fig. 1-43).

A special sample stage is used for mounting the substrate with the sample on it (Fig. 1-79). The base of this stage is made of a ferromagnetic material.
Performing measurements

The sample stage is mounted on the magnetic holder (Fig. 1-80), which is, in turn, mounted on the flange of the sample holder. This design makes it possible to rotate the sample stage around the vertical axis at any angle and, therefore, to set any required orientation of the sample in the XY plane.

When mounting the substrate with samples on the sample stage, the substrate is inserted under the spring clips from the side of two of the support balls. The clips press the top of the substrate down, while its bottom surface rests on the three support balls (Fig. 1-44).
Specifics of mounting the sample on the substrate with the spring contact

1. Mount the sample on the substrate (using double-sided scotch tape, for example).
2. Use tweezers to twist the contact spring so that it could touch the sample edge so that the center of the sample remains free (Fig. 1-49).
3. Insert the substrate into the sample holder so that the contact spring is on the farther side from the operator (the details of mounting the substrate to the sample holder see on pages 3-20, 3-22).
4. Connect the substrate with the bias voltage input socket (BV) 1 (Fig. 1-50), or with the grounding terminal 2.

ATTENTION! Do not use the BV socket for grounding the sample as the signal BiasVoltage is simultaneously applied both to the probe and to the BV socket.
1.6.4. **Installing the Measuring Head**

⚠️ **ATTENTION!** When using the optical viewing system the length of the measuring head supports should be adjusted to provide a gap of ~ 4 mm between the stage surface and the probe when the head is installed on the stage.

1. Before installing the measuring head it’s necessary to estimate the difference between the probe and the surface of the sample after the measuring head will be installed. It’s necessary to make sure that this distance is enough (not less than 1-2 mm) in order to avoid damaging the probe and the surface of the sample. For that:

   a. Put the measuring head front supports fixed by the lock-nuts in their seats 1 (see Fig. 1-85) without letting down the rear support;

   ![Fig. 1-85](image)

   1 – seats

   b. Looking from a side, carefully lower the rear support of the measuring head. If the gap between the holder and the sample is less than 2 mm then remove the measuring head;

   c. Loosen the lock-nuts and unscrew the threaded supports on the required length.

2. Install the measuring head, with the threaded supports resting on their seats in the exchangeable mount in such way that the front supports of the scanning head, fixed with contra-nuts, fit into the seats (one with a hollow and two with a groove).
3. During initial installation of the measuring head and, also, after altering the supports length, check if the plane of the measuring head base is parallel to the plane of the sample stage. This is done as follows:
   a. Measure the distance between the exchangeable mount surface and the measuring head base near each of the tree supports;
   b. Adjust the length of the supports so that these distances are equal. A ruler with one-millimeter-point scale would provide the adjustment accuracy of 0.5 mm which usually suffices;
   c. Fix the lock-nuts on the front supports.

4. Insert the cable of the head in the cable holder (see pos. 2 in Fig. 1-86).

1.6.5. Initial Approach

1. Looking from a side and turning the approach knob counterclockwise approach the sample to the probe at the distance of 0.5-1 mm.

2. Check the adjustment of the detection system for detecting the cantilever deflections using the photodiode indicator and readjust if needed.

1.6.6. Installation of a Protective Hood

It is recommended to work with a hood in the following cases:

- if it is necessary to obtain a high resolution image in XY plane or along Z axis;
- when carrying out at controlled temperature;
- for protection against temperature drifts;
- for reduction of acoustic noise level.
For installation of the protective hood proceed as follows:

1. Turn the videomicroscope support clockwise against the stop.

2. Take the protective hood with your left hand using handle 1, and with your right hand - using handle 2 (see Fig. 1-87).

3. Bring the hood to the stand so that plastic inserts on handle 1 of the hood (see pos. 3 in Fig. 1-87) rest against the stand, and the slot in handle 2 is located above the base unit frame (see Fig. 1-88). Put on the hood so that the frame gets into the slot. The plastic inserts should slide along the stand.
2. Contact Atomic Force Microscopy

Measurements of surface topography by means of the Constant Force Mode are the basis for operating the instrument using other contact techniques: for example, the Lateral Force Mode, the Spreading Resistance Imaging Mode, the Force Modulation Mode and the Contact Error Mode.

2.1. Constant Force Mode

2.1.1. Preparations for Measurements

Initial state

The following initial procedures are meant to be performed before operations:
- Launch the control program;
- Switch the instrument on;
- Install the probe and adjust the cantilever detection system;
- Mount the sample;
- Install the measuring head;
- Approach the sample to the probe at the distance of 0.5 ± 1 mm.

Main procedures performed during operations by Contact Microscopy Modes

Operations on Constant Force Modes can be divided into the following sequence of basic procedures:
1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).

A more detailed description of the enumerated above procedures is given below.
2.1.2. **Set the Electronic Configuration**

Set up the instrument for operating in the Contact technique by selecting **Contact** in the controller configuration list (Fig. 2-1) on the Main Parameters panel.

![Fig. 2-1. Selection of configuration](image)

Once the configuration **Contact** is set, all switching sequences required to operate on contact techniques are performed automatically.

2.1.3. **Set initial Level for the DFL Signal**

The value of the initial level for the **DFL** signal, which corresponds to the free state of the cantilever, is defined by the position of the photodiode with respect to the beam reflected from the cantilever. The zero value of the **DFL** signal corresponds to the position of the laser spot in between the top and bottom halves of the photodiode, and, therefore, the spot area is equally distributed between them.

The value of the **DFL** signal can be adjusted by means of screw 4 (Fig. 2-2), which provides mechanical translation of the photodiode in the transverse direction with respect to the laser beam reflected from the cantilever.

![Fig. 2-2. AFM measuring heads](image)

1, 2 – laser positioning screws; 3, 4 – photodiode positioning screws
DFL signal level adjustment procedure

1. Switch to the **Aiming** tab (click the ![Aiming button on the Main Operations panel) (Fig. 2-3).

![Fig. 2-3. Main Operations panel]

The tuning panel consists of a table, which contains real-time values acquired from the photodiode and the indicator of the laser spot position with respect to the sections of the photodiode (Fig. 2-4).

![Fig. 2-4. Tuning panel of the system for detecting the cantilever deflections](image)

2. Watch the signal level using the indicator and set the initial level for the DFL signal equal to 0 ±0.1 (see Fig. 2-4) by means of screw 4 (vertical translation screw of the photodiode, see Fig. 2-2).

2.1.4. **Approach the Sample to the Probe**

Carry out the following operations to perform approach of the sample to the probe:

1. Switch to the **Approach** tab (click the ![Approach button on the Main Operations panel) (Fig. 2-5).

![Fig. 2-5. Main Operations Panel]

2. Switch on the mode of automated setting of **Auto SetPoint** parameter by pressing of ![Auto SetPoint button (Fig. 2-6).](image)
3. Launch the approach procedure by clicking the button ![Landing].

The results of this procedure are:

- The feedback loop closes and the Z-section of the scanner extends at its maximum value, which is reflected in the scanner extension indicator in the bottom left corner of the main window of the program (Fig. 2-7). The magnitude of the scanner extension is characterized by the length of the colored bar;

![Fig. 2-7. Scanner extension indicator](image)

- The value of parameter **Set Point** is automatically set two units greater than the initial value of the signal **DFL** (i.e., **Set Point**=**DFL**+2);
- The stepper motor, which performs approach of the sample to the probe, is enabled.

Observe variations of the **DFL** signal and the state of the scanner extension indicator during the approach procedure using the software oscilloscope and wait for the completion of this procedure.

Providing the approach parameters are set correctly, the approach procedure is completed in about 10 to 30 seconds and the following actions take place (Fig. 2-8):

![Fig. 2-8. Approach process](image)

1 – scanner extension indicator; 2 – journal
- The signal DFL increases to the level of parameter Set Point, the feedback loop maintains the Z-scanner in the position where the value of DFL is equal to Set Point. Note that this position of the scanner is approximately equal to half of the scanner extension range;
- Length of the indicator bar decreases and occupies some intermediate position (see 1 in Fig. 2-8);
- The stepper motor is disabled;
- The increase of the DFL signal to the value of parameter Set Point is visualized in the DFL (t) graph on the software oscilloscope;
- The record “…Approach Done.” appears in the journal (see 2 in Fig. 2-8).

NOTE. In the case of scanning by probe, the laser lights the probe being reflected from the mirror that is firmly fixed to the scanner. Therefore some misalignment of the optical detection system may occur when closing the feedback loop (while extending the Z-scanner). As a result, the position of the reflected laser spot on the photodiode changes and this causes a small feature (discontinuity) in the signal behavior on the software oscilloscope. This effect can be minimized by performing the following:
1. Open the feedback loop using the button .
2. Open the area of additional operations using the button .
3. Switch to the tab Scheme.
4. Click the control button of the Z-section of the scanner and set the slider in the middle position.
5. Switch to the tab Aiming and repeat tuning of the signal Laser value to maximum. Also, adjust the position of the photodiode.

Selection and manual setting of parameter Set Point

Setting of parameter Set Point requires the following:
- switch off the mode of automated setting of Auto SetPoint parameter (the button is not pressed in).
- Type in a value of parameter Set Point into the data text box available from the main parameters panel.

As an initial value, it is recommended to set the value of parameter Set Point equal to the value of the DFL signal plus approximately 5÷10 % of the value of signal Laser (i.e. SetPoint=DFL+(0.05÷0.1)*Laser).

While selecting an optimal value of parameter Set Point, consider the following:
- The difference between the value of Set Point and the initial level of the DFL signal defines the magnitude of interaction between the probe’s tip and the sample surface. The greater the difference between the value of Set Point and the initial level of the signal DFL, the greater deflection of the cantilever and, respectively, the greater magnitude of interaction between the probe’s tip and the sample surface. Therefore, it
is possible to set and alter the magnitude of interaction between the probe’s tip and the sample surface by varying the value of parameter **Set Point** with respect to the initial value of the signal **DFL**;

- If the value of the difference between the value of **Set Point** and the initial level of **DFL** is set too big, which corresponds to a too tight interaction between the probe and the sample surface, then this may cause damage to both the probe and the surface under study during scanning;

- If the value of the difference between the value of **Set Point** and the initial level of **DFL** is set too small, which corresponds to an inadequately weak interaction between the probe and the sample surface, then this may result in an unsteady mode of operation of the feedback system;

- The value of parameter **Set Point** should not be set less than the value of initial level of the signal **DFL** and should not be set greater than the value of the signal **Laser**.

2.1.5. Setting the Feedback Gain Factor Working Level

The greater the value of the feedback gain factor (parameter **FB Gain**), the greater the feedback loop data processing speed. Nevertheless, at some big value of the feedback gain factor (let us call it threshold value), the mode of operation of the feedback loop becomes unsteady and some noise generation occurs. A significant variable component of the signal **DFL** appears during such a mode of operation (Fig. 2-9).

![Fig. 2-9. Noise generation in the feedback loop](image)

For steady operation, it is recommended to set the value of the feedback gain factor to be not greater than 0.5÷0.7 of the threshold value where the noise generation occurs. Adjustment of the feedback gain factor is performed by means of the text box of **FB Gain**.
Setting of the operating level for the feedback gain factor requires the following:

1. Double-click the mouse in the text box of parameter FB Gain from the Main Parameters panel. The slider for setting the feedback gain factor will appear (Fig. 2-10).

2. Increase the value of FB Gain and watch the value of signal DFL by means of the software oscilloscope.

3. Determine the value of the parameter FB Gain that corresponds to the beginning of noise generation. The beginning of the generation is identified by the appearance of a significant variable component in the signal DFL (see Fig. 2-9).

4. Decrease the value of the parameter FB Gain and set it equal to 0.5÷0.7 of the threshold value to be used as the value for operating.

2.1.6. Set parameters of Scanning

Switch to the Scan tab (the Scan button on the Main Operations panel) (Fig. 2-11).

The top part of the Scan tab contains a panel which provides control over scanning (Fig. 2-12).

Another panel is located below. This panel contains a 1D image of the signal measured during scanning line-by-line. Also another panel containing 2D images of the scanning data is located below.
Performing measurements

Selection of AFM mode

Select the mode **Contact Topography (Constant Force)** from the list **Mode** (scan mode list) of the control panel (Fig. 2-13). The controller performs all the corresponding switching sequences automatically during this selection.

![Fig. 2-13. Selection of the Constant Force Mode from the control panel of the Scan tab](image)

Selection of an area for scanning

The following recommendations on the selection of preliminary dimensions of the scanning area can be made:

- If there is some preliminary information on surface properties of the sample under investigation and it is certain that the expected surface topography overfalls are within the limits of the Z-scanner range. In this case it is recommended to set the maximum field of scanning;

- If there is no preliminary information on surface properties of the sample under investigation. In this case it is recommended to begin scanning with a small size area, for example about 0.5÷1.0 μm. Then, on the results of scanning of that area, it is possible to set and optimize such parameters as speed of scanning, **Set Point**, **FB Gain**. Then the scanning area can be resized.
The following actions should be performed to select and resize the scanning area:

1. Enable the option of selection and resizing of the scanning area by clicking the button on the panel of 2D images of the scanning data (Fig. 2-14).

![Fig. 2-14. Panel of 2D images of the scanning data](image)

- 1 – boundaries of the selected scanning area;
- 2 – marker indicating the position of the probe

2. Use the mouse to alter the size and position of the scanning area (see 1 in Fig. 2-14).

   NOTE. Alterations of the scanning area size are automatically reflected in the text boxes of the parameter Scan Size.

3. Click the button . Verify that, within the scanning area selected, the probe reaches the surface without “burying” into it. For that, click the left button of the mouse and, holding it down, move the cursor (see 2 in Fig. 2-14) within the boundaries of the selected scanning area. Displacement of the cursor reflects the true travel of the probe with respect to the sample surface. The level of extension of the piezo-scanner can be controlled by means of the indicator in the bottom part of the window (see Fig. 2-7).
Performing measurements

**Setting of the scanned image size, number of pixels, pace of scanning**

The number of pixels along the X- and Y-axes (parameter **Point Number**), the size of the image scanned (parameter **Scan Size**) and the pace of scanning (parameter **Step Size**) are set by means of selecting the corresponding parameter from a list (Fig. 2-15).

![Fig. 2-15](image)

When setting parameters **Point Number**, **Scan Size** and **Step Size**, consider the following:

- While altering **Point Number**: **Scan Size** alters; **Step Size** does not alter.
- While altering **Scan Size**: **Step Size** alters; **Point Number** does not alter.
- While altering **Step Size**: **Scan Size** alters; **Point Number** does not alter.

**Setting of scanning speed**

Selection of the optimal value for scanning speed depends on surface properties of the sample under study, scanning area dimensions and external conditions. Surface with smooth topography can be scanned at higher speed than that with uneven topography and high overfalls.

At the start, it is recommended to set the line scanning frequency (parameter **Frequency**) within 0.5÷2.0 Hz (see Fig. 2-15).

**Changing the measurement signal**

When using the measuring head equipped with displacement sensors, it is recommended to use **Sensor Height** as the signal for acquisition for its higher measurement accuracy. To change the measurement signal perform the following:

1. Open the dialog window **Scan Setting** (Fig. 2-16) by clicking the button from the control panel of the **Scan** tab.
2. Click the mouse in the text box of the first channel and select SensorHeight from the drop-down list that appears.

3. Click the button OK to save all the alterations made and to close the dialog window.

2.1.7. Scanning

Consider the scanning process on the example of a sample in the form of a rectangular grating (standard grating TGQ-1, 3 μm resolution).

Trigger scanning

Scanning of the sample surface should be started after 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 of the Scan tab (Fig. 2-17).
Performing measurements

The following actions are the results of clicking the button **Run**:

- Line-by-line scanning of the sample surface is triggered and an image of the scanned area appears line-by-line in the panel of 2D images of the scanning data (Fig. 2-18). In the example under consideration, this is an image of the rectangular grating:

![Surface topography image](image1.png)

Fig. 2-18. Surface topography image

- A corresponding image, which reflects the signal measured in real time, appears on the panel of 1D images of the scanning data line-by-line (Fig. 2-19);

![Signal Height](image2.png)

Fig. 2-19. Signal Height

- Some buttons disappear from the control panel of the **Scan** tab, while a number of new buttons appear: **Pause, Restart, Stop** (Fig. 2-20).

![Control panel](image3.png)

Fig. 2-20

Should the scanning procedure be interrupted for some reason, click the button **Stop** or use the key <Esc> of the keyboard.
 Alterations of parameters in the process of scanning

Tilt subtraction

Consider the given above example (see Fig. 2-18, Fig. 2-19). It is seen that the sample has some tilt along the X-axis.

This tilt can be subtracted directly in the process of scanning by using the button Subtract. By default, this button is in the position None (see Fig. 2-20).

Click this button and select the option Plane from the corresponding list (Fig. 2-21). This entails subtraction of the plane tilt, and the original image, illustrated in Fig. 2-18, is transformed into the image shown in Fig. 2-22.

Respectively, the panel of 1D images of the scanning data will be visualizing the measured signal with tilt subtracted from it. Therefore the resulting signal will be transformed as shown in Fig. 2-23, instead of the signal illustrated in Fig. 2-19.
Performing measurements

Fig. 2-23. Signal Height

A more detailed description of the function **Subtract** is provided in *SPM Software*, part 1 “SPM Control Program”.

NOTE. Transformations of the scanned image performed by means of the function **Subtract** are not saved in the frames obtained.

**Tuning of parameters during the process of scanning**

Quality of the obtained surface images essentially depends on such parameters as scanning frequency, **Frequency** (Fig. 2-24), the value of the operating point **Set Point** (Fig. 2-25) and the feedback gain factor, **FB Gain** (Fig. 2-25). All these parameters can be altered directly in the process of scanning.

The special function **Pause** is used for tuning of scanning parameters. Once this function is enabled, scanning along the slow axis is halted, while scanning along the fast axis goes on continuously. This mode of scanning can be used for optimization of scanning parameters. Profile of the scanned line can be visually monitored and it varies while altering corresponding parameters: scanning frequency, **Frequency**, the value of **Set Point** or the feedback gain factor **FB Gain**, for example.

Once the function **Pause** is disabled, the process of scanning continues from the same line.

The button **Restart** is used to restart scanning again.

NOTE. It is recommended to restart scanning (using the button **Restart**) after using the function **Pause** and altering scanning parameters.
Some recommendations on optimization of scanning parameters

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

Surfaces with smooth topography can be scanned at a speed higher than those with sharp features and rapid overfalls.

It is recommended to start scanning at lower scanning speed, increasing it gradually until distortions start altering the topography profile.

Scanning speed should be reduced when surface features are not read in the direction of scanning.

In measurements on soft materials, the images may be affected by “dragging effects” caused by surface features in the direction of scanning. In the event of such effects, it is recommended to reduce the speed of scanning and, also, to decrease the value of Set Point in order to reduce pressure on the sample.

2.1.8. Saving of Measurement Data

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

1. Select File → Save command from the main menu.

2. A dialog box will appear. Choose a folder to store the data (by default, it is the folder C:\Program Files\NT-MDT\Nova).

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

NOTE. By default, the images obtained are stored in files “NoNameXX.mdt”, where XX is the file index in the folder Nova.

2.1.9. Completion of Measurements

To complete operation, perform the following steps:

1. Open the feedback loop (the button is not pressed in).

2. Take the sample aside from the probe. For that, perform the following steps:

   a. Switch to the Approach tab (the button on the Main Operations panel) (Fig. 2-26).
Performing measurements

b. Double-click in the text box **Moving** for **Backward** (Fig. 2-27) on the control panel of the **Approach** tab. Set the value 2÷3 mm using the slider.

![Fig. 2-27]

\[
\begin{array}{|c|c|c|c|c|c|}
\hline
\text{Stop} & \text{Landing} & \text{Backward} & \text{One Step} & \text{Fast} & \text{Moving} \\
\hline
\text{Auto SetPoint} & \text{Forward} & \text{One Step} & \text{Fast} & \text{Moving} \\
\hline
\end{array}
\]

\begin{itemize}
\item \text{Slider:} \(2.00\) mm
\end{itemize}

Fig. 2-27

c. Click the button \(\text{Fast}\) for **Backward** to take the sample aside from the probe (Fig. 2-28).

![Fig. 2-28]

\[
\begin{array}{|c|c|c|c|c|c|}
\hline
\text{Stop} & \text{Landing} & \text{Backward} & \text{One Step} & \text{Fast} & \text{Moving} \\
\hline
\text{Auto SetPoint} & \text{Forward} & \text{One Step} & \text{Fast} & \text{Moving} \\
\hline
\end{array}
\]

\begin{itemize}
\item \text{Slider:} \(2.00\) mm
\item \text{Slider:} \(3.00\) mm
\end{itemize}

Fig. 2-28

\section*{2.2. \textit{Lateral Force Microscopy}}

\subsection*{2.2.1. Brief Description of the Mode}

The Lateral Force Microscopy makes it possible to discriminate between areas with different coefficients of friction and, in addition, it visualizes specifics of surface topography. This mode can be useful in research of semiconductors, polymers, film coatings, shape-memory materials, in the study of surface properties, chemical properties in particular (contamination, for example) and tribology characteristics.

The physical essence of the Lateral Force Microscopy is as follows. While scanning in this mode in the direction perpendicular to the longitudinal axis of the cantilever, some torsion deflection of the probe, which is additional to the normal deflection, occurs (Fig. 2-29). It is caused by momentum of the force acting on the tip of the probe.

![Fig. 2-29]

![Fig. 2-30]
At small angles, the angle of twist of the probe is proportional to lateral forces. The magnitude of the twist is measured with the optical detection system of the microscope. This detection system forms the signal $LF$, whose variations are proportional to the value of the torsion deflection of the cantilever. This signal is then used to obtain images of the local force of friction on the surface of the sample.

While moving the probe along a flat surface, which has segments with different friction coefficients, the twist angle of the probe will change from one segment to another (Fig. 2-30). This contains information on distribution of local friction properties. However, such interpretation is no longer valid in the case of complicated topography.

Nevertheless this type of measurement techniques makes it possible to obtain good images with highly detailed small surface features. For example, it is easy to determine parameters of the atomic lattice of mica and other lamellar materials in measurements for lateral forces.

### 2.2.2. Preparation for Measurements

The Lateral Force Mode is based on the Constant Force Mode, which is described in detail in i. 2.1 “Constant Force Mode” on page 3-43.

**Main procedures performed during operations by the Lateral Force Mode**

1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the $DFL$ (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49). Choose the mode $Lateral \ Force$.
6. Scanning (i. 2.1.7 on page 3-53).

The main difference of this mode from the operations in the Constant Force Mode is that in i. 2.1.6 “Set parameters of Scanning” on page 3-49 the mode $Lateral \ Force$ is set instead of $Contact \ Topography$ ($Constant \ Force$). To perform this setting, select mode $Lateral \ Force$ in the list **Mode** available from the control panel of the **Scan** tab (Fig. 2-31). Correspondingly, all necessary for this mode switching sequences are done in the system automatically.

![Fig. 2-31. Selection of the Lateral Force Microscopy](image)
2.2.3. Scanning

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 and 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 lateral forces (signal LF) (Fig. 2-32);

![Fig. 2-32. Images of surface topography and distribution of lateral forces](image)

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-33).

![Fig. 2-33. Signal LF](image)

2.3. Constant Height Mode

2.3.1. Brief Description of the Mode

In the Constant Height Mode the probe’s tip is in contact with the surface. During scanning, the end of the cantilever, which is fixed to the chip, is maintained at a constant height above the sample surface (scanning with the open feedback loop). Consequently, deflections of the cantilever follow surface features of the sample under investigation.

The process of reading the surface with the cantilever is limited by frequency properties of the cantilever. This is the difference from the Constant Force Mode, which is limited by
properties of the feedback loop. Resonance frequencies of the cantilever are much higher than characteristic frequencies of the feedback loop, whose value is measured in the units of kHz. This gives the opportunity to perform scanning at higher speeds.

If the relationship between deflection of the cantilever used in measurements and the distance from the chip to the sample surface is known (see Fig. 2-34) (dependence of the signal DFL versus the distance between the chip and the surface), then it is possible to employ this relationship to transform the distribution of the DFL signal into a surface topography image.

Fig. 2-34

In addition, providing that the spring constant of the cantilever employed and its geometrical sizes are known, the image of the normal deflection distribution of the cantilever (signal DFL) can be converted into an image of distribution of local forces acting on the cantilever.

However it is necessary to remember that the dependence of the DFL signal becomes non-linear for big values of deflection of the cantilever. An approximate range of linearity depends on the cantilever: the shorter the cantilever, the smaller the range.

The requirement of smoothness to the surface is amongst the disadvantages of this mode. In addition, when investigating soft samples (like polymers, biological objects, Langmuir-Blodgett films etc.), the samples may become damaged as the probe is in direct mechanical contact with the surface. When scanning relatively soft samples with complicated surface topography, the pressure exerted by the probe on the surface varies, and, at the same time, the surface twists unevenly. This may result in distortions of the true topography image.
2.3.2. Preparation for Measurements

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

Main procedures performed during operations by the Constant Height Mode

1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).

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

2.3.3. Setting of Parameters

1. Switch to the Scan tab (the button on the Main Operations panel) (Fig. 2-35).

Fig. 2-35. Main Operations panel

2. Choose the mode Contact Constant Height from the list Mode on the control panel of the Scan tab (Fig. 2-36).

Fig. 2-36. Selection of the Constant Height Mode

NOTE. When choosing the Constant Height Mode, the value of the parameter FB Gain (gain coefficient of the feedback loop) is automatically set close to zero. This is equivalent to opening the feedback loop and fixing the position of the tip of the probe at a certain height above the surface. Simultaneously, the signal DFL is applied to the ADC input.
2.3.4. Scanning

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 is triggered and an image of the scanned area appears line-by-line in the panel of 2D images of the scanning data (Fig. 2-37);

![Fig. 2-37. Surface topography image](image)

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-38).

![Fig. 2-38. Signal DFL](image)

2.4. Spreading Resistance Imaging

2.4.1. Brief Description of the Mode

The Spreading Resistance Imaging mode is a very efficient AFM mode. It is employed for various measurements. For example, to determine defects in conductive and low conductive films, materials characterization in terms of local resistance etc. Realization of this mode is based on the use of a conductive probe, which comes in contact with the sample surface. A bias voltage is applied to the probe, and measurements of the resulting current flow through the sample are performed as a function of the probe simultaneously with surface topography measurements. The latter are performed using the Constant Force
Performing measurements

mode. It is easy to prove that, in the assumption of constant contact probe-surface resistance for a given bias voltage, the magnitude of the measured current flow is proportional to local resistance of the sample under investigation.

This mode is typically used for finding the real geometrical size of the source and sinks regions in MOS transistors, for localization of p-n junctions, for study of semi-conductor and composite materials.

2.4.2. Preparation for Measurements

The Spreading Resistance Imaging Mode is based on the Constant Force Mode, which is described in detail in i. 2.1 “Constant Force Mode” on page 3-43.

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

Measuring of the Spreading Resistance Imaging is performed either with the scanning measuring head or with the universal head with the measuring insert AU006.

A sample should be mounted on the substrate with a spring contact. The contact is connected to the BV socket of the base unit.

The measuring head is equipped with the conducting contact probe.

ATTENTION! When mounting the probe, ensure that the lever of the probe holder is turned counterclockwise up to the stop. Otherwise, the probe will be electrically grounded.

Main procedures performed during operations by the Spreading Resistance Imaging Mode
1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).

After the completion of preliminary measurements of surface topography by the Constant Force Mode, perform setting of parameters for operating by the Spreading Resistance Imaging Mode.
2.4.3. Setting of Parameters

1. Switch to the Scan tab (the button on the Main Operations panel) (Fig. 2-39).

   ![Fig. 2-39. Main Operations panel](image)

2. Choose the mode **Spreading Resistance** from the list Mode on the control panel of the Scan tab (Fig. 2-40).

   ![Fig. 2-40. Selection of the Spreading Resistance Imaging Mode](image)

   **NOTE.** The control program performs all necessary switching automatically.

3. Open the Additional operations area (button).

4. Get an image of the signal $I_{pr\, low}$ on the software oscilloscope.

5. Double-click the mouse in the text box of parameter **Bias Voltage**. The slider for setting an electrical potentials difference between the sample and probe will appear (Fig. 2-41).

   ![Fig. 2-41. Slider for setting the parameter Bias Voltage](image)

6. Move the marker and set voltage **Bias Voltage** so that the signal $I_{pr\, low}$ would be within 1÷10 nA range on the software oscilloscope.
2.4.4. **Scanning**

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 and 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 spreading current (signal **lpr low**);

![Fig. 2-42. Images of surface topography and distribution of spreading current](image1)

**NOTE.** The lighter areas in the signal **lpr low** image correspond to higher conductivity.

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-43).

![Fig. 2-43. Signal **lpr low**](image2)
2.4.5. **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 the value of the electrical potentials difference between the probe and the sample (parameter **Bias Voltage**) (see Fig. 2-41). Too high voltage may cause local oxidization of the surface;
- Replace the probe (with different elasticity or another type of conductive coating).

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

2.5. **Force Modulation Mode**

2.5.1. **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. 2-44).

![Cantilever deflection amplitude](image)

Image of elasticity

**Fig. 2-44**

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.
2.5.2. **Preparation for Measurements**

The Force Modulation Mode is based on the Constant Force Mode, which is described in detail in 2.1 “Constant Force Mode” on page 3-43.

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

**Main procedures performed during operations by the Force Modulation Mode**

1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).

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

2.5.3. **Setting of Parameters**

1. Switch to the Scan tab (the \[\text{Scan}\] button on the Main Operations panel) (Fig. 2-45).

   ![Fig. 2-45. Main Operations panel](image)

2. Choose the mode **Force Modulation** from the list Mode on the control panel of the Scan tab (Fig. 2-46).

   ![Fig. 2-46. Selection of the Force Modulation Mode](image)

   **NOTE.** The program automatically connects the voltage generator with the Z-section of the scanner, sets a range for the search for the resonance frequency of the system (scanner, sample and probe), switches to the low frequency input of the lock-in amplifier and sets all signals required for the measurement.
The resonance frequency search is performed as follows:

1. Switch to the **Resonance** tab (the button on the Main Operations panel) (Fig. 2-48).

2. Clear the **Auto peak find** check box (Fig. 2-48).

3. Set a frequency range **From** 5 kHz **To** 25 kHz.

4. Start the procedure of resonance frequency search by clicking the button **Run** (the feedback loop should be closed).

   On the completion of this procedure, a frequency dependence of the cantilever oscillations (signal **Mag**) will be visualized (Fig. 2-49).

5. Set parameter **Frequency** (Fig. 2-50), which corresponds to one of the peaks.
Performing measurements

6. Note that the height of the selected peak should be within 2÷5 nA. To achieve that, you can vary the generator voltage (parameter **Amplitude**) (see Fig. 2-48), or the gain coefficients of the lock-in amplifier (parameter **Gain**) and preamplifier (parameter **Preamplifier**) (see Fig. 2-48).

2.5.4. **Scanning**

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 and 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**) (Fig. 2-51);
NOTE. Lighter areas of the signal Mag distribution correspond to harder surface.

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-52).

2.5.5. 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 Amplitude) (see Fig. 2-48), gain coefficients of the lock-in amplifier (parameter Gain) and preamplifier (parameter Preamplifier) (see Fig. 2-48);
- Replace the probe (with different elasticity and resonance frequency).
2.6. **Contact Error Mode**

2.6.1. **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.

2.6.2. **Preparation for Measurements**

The Contact Error Mode is based on the Constant Force Mode, which is described in detail in i. 2.1 “Constant Force Mode” on page 3-43.

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

**Main procedures performed during operations by the Force Modulation Mode**

1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).
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.

2.6.3. Setting of Parameters

Measurements on the Contact Error Mode require enabling the signal DFL. For that, perform the following steps:

1. Open the dialog box **Scan Settings** (Fig. 2-53) by clicking the button on the control panel of the **Scan** tab.

   ![Scan Settings dialog box](image)

   **Fig. 2-53. Scan Settings dialog box**

2. Select the signal DFL as the second signal for registration during the direct pass.
3. Click **OK** to save alterations and to close the dialog box.

2.6.4. Scanning

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 and 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. 2-54);
Fig. 2-54. Images of surface topography and the Error signal

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-55).

---

2.7. **Piezoresponse Force Microscopy**

2.7.1. **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. Special 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. 2-56 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.

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. 2-57). This information makes it possible to study the domain structure of the sample under investigation.
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.

### 2.7.2. Preparation for Measurements

Measuring of the Piezoresponse Force Microscopy is performed either with the scanning measuring head or with the universal head with the measuring insert AU006 or AU007. The measuring head is equipped with the conducting contact probe.

**ATTENTION!** When mounting the probe, ensure that the lever of the probe holder is turned counterclockwise up to the stop. Otherwise, the probe will be electrically grounded.

A sample should be mounted on the substrate with a spring contact (see Part 3, i. Mounting the Sample).
For Piezoresponse Force Microscopy, ac voltage is applied in one of the following ways:

- to the tip;
- to the sample.

The scanning measuring head can operate with ac voltage applied either to the tip or to the sample. For the universal measuring head, the measuring insert AU007 or AU006 is used for ac voltage applied to the tip or to the sample, respectively.

Voltage is applied to the tip when the contact of the substrate is connected to the grounding socket of the base unit.

Voltage is applied to the sample when the contact of the substrate is connected to the BV socket of the base unit.

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

### Main procedures performed during operations on Piezoresponse Force Microscopy

1. Set the Electronic Configuration (i. 2.1.2 on page 3-44).
2. Set initial Level for the DFL (i. 2.1.3 on page 3-44).
3. Approach the Sample to the Probe (i. 2.1.4 on page 3-45).
4. Setting the Feedback Gain Factor Working Level (i. 2.1.5 on page 3-48).
5. Set parameters of Scanning (i. 2.1.6 on page 3-49).
6. Scanning (i. 2.1.7 on page 3-53).

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

### 2.7.3. Setting of Parameters

1. Switch to the **Scan** tab (the **Scan** button on the Main Operations panel) (Fig. 2-58).

   ![Fig. 2-58. Main Operations panel](image)

2. Choose the mode **Piezoresponse Force Microscopy** from the list **Mode** on the control panel of the **Scan** tab (Fig. 2-59).
NOTE. When selecting this mode, the system automatically sets the maximum value for gain coefficients of the lock-in amplifier, Gain, and the Preamplifier. The signal DFL is automatically connected to the input (Fig. 2-60).

Also, the following signals will be selected for measurement during scanning:
- **Height** and **Mag**, on the forward pass, (Fig. 2-61);
- **Phase**, on the backward pass, (Fig. 2-61).
3. Verify that the tip and the probe on the block scheme of the instrument are biased as follows: the sample is grounded while the tip is connected to Bias Voltage (see Fig. 2-62).

4. If ac voltage will be applied to the sample, the tip should be grounded. For grounding the tip, press the IPR button in the block scheme (Fig. 2-62).
2.7.4. Scanning

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 and 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) (Fig. 2-63).

![Images of the surface under investigation](image)

Fig. 2-63. Images of the surface under investigation

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 2-64).

![Signal Mag](image)

Fig. 2-64. Signal Mag

**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 (signal LF), switch the inputs of the lock-in amplifier and the phase detector from DFL to LF (Fig. 2-65).

![Switching inputs](image)

Fig. 2-65
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. 2-66).

Fig. 2-66. Images of the surface under investigation
3. **Semicontact Atomic Force Microscopy**

Operations of the instrument for measurements of surface topography my means of Semicontact Atomic Force Microscopy modes are also the basis for the realization of a number of other techniques based on the use of resonance oscillations of the cantilever.

### 3.1. **Semicontact Mode**

#### 3.1.1. Preparation for Measurements

Measuring in the Semicontact Mode is carried out with the universal or scanning measuring head. When measuring with the universal measuring head, it can be supplied with any measuring insert except AU020 and AU030. The measuring head is equipped with the semicontact probe.

**Initial state**

The following initial procedures are meant to be performed before operations:

- Launch the control program;
- Switch the instrument on;
- Install the probe and adjusting the system for detecting the cantilever deflections (detection system);
- Mount the sample;
- Install the measuring head;
- Approach the sample to the probe at the distance of 0.5÷1 mm.

**Main procedures performed during operations by Semicontact Microscopy Modes**

Operations in the Semicontact Modes mode can be divided into the following sequence of basic procedures:

1. Set the Electronic Configuration (i. 3.1.2 on page 3-83).
2. Set the Piezodriver Operating Frequency (i. 3.1.3 on page 3-83).
3. Set Initial Level for the Signal Mag (i. 3.1.4 on page 3-87).
4. Approach the Sample to the Probe (i. 3.1.5 on page 3-88).
5. Setting the Feedback Gain Factor Working Level (i. 3.1.6 on page 3-90).
6. Set parameters of Scanning (i. 3.1.7 on page 3-92).
7. Scanning (i. 3.1.8 on page 3-95).

A more detailed description of the enumerated above procedures is given below.
3.1.2. Set the Electronic Configuration

Set up the instrument for operating in the semicontact modes by selecting SemiContact in the controller configuration list (Fig. 3-1) on Main Parameters panel.

![Fig. 3-1. Selection of configuration](image)

Once the configuration SemiContact is set, all switching sequences required to operate on semicontact techniques are performed automatically.

3.1.3. Set the Piezodriver Operating Frequency

The following actions are performed for setting the piezodriver operating frequency:

1. Switch to the Resonance tab (the button on the Main Operations panel) (Fig. 3-2).

![Fig. 3-2. Main Operations panel](image)

2. Select the Auto peak find check box (Fig. 3-3).

![Fig. 3-3. Control panel of the Resonance tab](image)

3. Set the range for measuring the frequency response of the cantilever oscillation amplitude From 90 kHz To 500 kHz.

4. Start the procedure of the automated resonance frequency search by clicking the button Run.

The following are the results of the procedure:

- Determination of frequency dependence of the cantilever oscillations amplitude (signal Mag);
- Determination of the resonance frequency of the cantilever;
- Setting of the piezodriver operating frequency, which is equal to the resonance frequency of the cantilever;
- Visualization of the frequency dependence of the signal Mag near its maximum (Fig. 3-4).

5. Pay attention to the shape of the resonance curve, especially near its peak. If the shape of the dependence obtained (especially near the peak) is approximately similar to the one given in the example in Fig. 3-4, then the automated search procedure can be considered successful. Move on to the next step (i.e., **3.1.4 “Set Initial Level for the Signal Mag”** on page 3-87).

Usually, the resonance curve has a good shape and the automated search procedure is performed successfully.

**Special cases**

It is quite possible that, on the completion of the automated search, the visualized resonance curve has a peak whose shape is affected by distortions. These distortions might appear in different forms. For example, the resonance peak can be broadened, as shown in Fig. 3-5, or have several local maximums as shown in Fig. 3-9.

The nature of these distortions can be linked, for example, with too high oscillation amplitude of the cantilever (too high value is set for the generator voltage), or too big gain coefficient is set (parameter Gain of the lock-in amplifier, parameter Amp Gain of the preamplifier), or both of these factors take place simultaneously.
I. The resonance curve peak is distorted and the near-peak area lies above 35÷40 units.

If the resonance peak is distorted and the near-peak area lies above 35÷40 units (Fig. 3-5), do the following:

1. Double-click the mouse in the text box of the lock-in amplifier gain coefficient **Gain**. The slider will appear (Fig. 3-6).

2. Alter the value of parameter **Mag** to 20÷25 units by reducing the value of parameter **Gain** by means of the slider. Check the value of signal **Mag** by the readings available from the panel of main parameters (Fig. 3-7).

3. After reducing the signal **Mag** to 20÷25 units, repeat the procedure of the automated resonance frequency search by clicking **Run**.

4. If distortions of the resonance peak disappear, move on to the next procedure (i. 3.1.4 on page 3-87), otherwise repeat points 1-3 or reinstall the probe.
Performing measurements

Also, the preamplifier gain coefficient can be changed (Fig. 3-8) (parameter Preamplifier can have two values: 1 or 10).

II. The resonance curve peak is distorted and the near-peak area lies below 35÷40 units.

If the resonance peak is distorted and the near-peak area lies below 35÷40 units (Fig. 3-9), do the following:

1. Double-click the mouse in the text box of the generator voltage Amplitude. The slider will appear (Fig. 3-10).
2. Reduce the value of parameter \textbf{Amplitude} using the slider.

3. After reducing the signal, repeat the procedure of the automated resonance frequency search by clicking \textbf{Run}.

4. If distortions of the resonance peak disappear, move on to the next procedure (i. \textbf{3.1.4 “Set Initial Level for the Signal Mag” on page 3-87}), otherwise repeat points 1-3 or reinstall the probe.

\textbf{3.1.4. \ Set Initial Level for the Signal Mag}

It is recommended to set the signal \textbf{Mag} within 20÷25 units.

Altering voltage from the generator does setting of the signal \textbf{Mag} level. This is performed as follows:

1. Double-click the mouse in the text box of the generator voltage \textbf{Amplitude}. The slider will appear (Fig. 3-11) (voltage can vary within the range 0÷1 V).

2. Set the value of parameter \textbf{Mag} to 20÷25 units by altering the value of parameter \textbf{Amplitude} by means of the slider. Check the value of signal \textbf{Mag} by the readings available from the panel of main parameters (Fig. 3-12).

3. If necessary, change the value of the gain coefficient of the generator output signal amplitude (it can have either of the three values: \textbf{0.1, 1, 10}) (Fig. 3-13).
NOTE.

- Level of the signal \( \text{Mag} \) can be altered by altering the gain coefficients of the lock-in amplifier (parameter \( \text{Gain} \)) and preamplifier (parameter \( \text{Preamplifier} \));
- When adjusting the signal \( \text{Mag} \) level by altering the gain coefficients of the lock-in amplifier and preamplifier with the generator voltage fixed (parameter \( \text{Amplitude} \)), the cantilever oscillation amplitude remains constant;
- Otherwise, when adjusting the signal \( \text{Mag} \) level by altering the generator voltage with the fixed gain coefficients of the lock-in amplifier and preamplifier, the cantilever oscillation amplitude will be altering.

3.1.5. Approach the Sample to the Probe

1. Switch to the Approach tab (click the \( \text{Approach} \) button on the Main Operations panel) (Fig. 3-14).

![Fig. 3-14. Main Operations panel](image)

2. Switch on the mode of automated setting of Auto SetPoint parameter by pressing of \( \text{Auto SetPoint} \) button (Fig. 3-15).

![Fig. 3-15. Control panel of the Approach tab](image)

3. Launch the approach procedure by clicking the button \( \text{Landing} \).

The results of this procedure are:

- The feedback loop closes and the Z-section of the scanner extends at its maximum value, which is reflected in the scanner extension indicator in the bottom left corner of the Main window of the program (Fig. 3-16). The magnitude of the scanner extension is characterized by the length of the colored bar;

![Fig. 3-16. Scanner extension indicator](image)

- The value of parameter Set Point is automatically set equal to half of the current value of signal \( \text{Mag} \) (i.e. \( \text{Set Point}=\text{Mag}/2 \));
- The stepper motor, which performs approach of the sample to the probe, is enabled.
Observe variations of the $\text{Mag}$ signal and the state of the scanner extension indicator during the approach procedure using the software oscilloscope and wait for the completion of this procedure.

Providing the approach parameters are set correctly, the approach procedure is completed in about 10 to 30 seconds and the following actions take place (Fig. 3-17):

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

- Length of the indicator bar decreases and occupies some intermediate position (see 1 in Fig. 3-17);
- The stepper motor is disabled;
- The decrease of the signal \( \text{Mag} \) to the value of parameter \( \text{Set Point} \) is visualized in the \( \text{Mag}(t) \) graph on the software oscilloscope;
- The record “\(...\text{Approach Done.}\)” appears in the journal (see 2 in Fig. 3-17).

\[ \text{NOTE. In the case of scanning by probe, the laser lights the cantilever being reflected from the mirror that is firmly fixed to the scanner. Therefore some misalignment of the optical detection system may occur when closing the feedback loop (while extending the Z-scanner). As a result, the position of the reflected laser spot on the photodiode alters and this causes a small feature in the signal behavior on the software oscilloscope. Setting an optimal position of the laser spot on the cantilever can minimize this effect. This position is typically the point where the \( \text{Mag} \) signal reaches its maximum.} \]

**Selection and manual setting of parameter \( \text{Set Point} \)**

Setting of parameter \( \text{Set Point} \) requires the following:

- switch on the mode of automated setting of \( \text{Auto SetPoint} \) parameter (the button \( \text{A} \) is not pressed in);
- type in a value of parameter \( \text{Set Point} \) into the text box available from the panel of main parameters (Fig. 3-18).

![Fig. 3-18](image)

As an initial value, it is recommended to set the value of parameter \( \text{Set Point} \) equal to half of the signal \( \text{Mag} \) value (i.e. \( \text{SetPoint}=\text{Mag}/2 \)).

\[ \text{NOTE. The value of parameter \( \text{Set Point} \) should not be set greater than the value of the signal \( \text{Mag} \).} \]

**3.1.6. Setting the Feedback Gain Factor Working Level**

The greater the value of the feedback gain factor (parameter \( \text{FB Gain} \)), the greater the feedback loop data processing speed. Nevertheless, at some big value of the feedback gain factor (let us call it threshold value), the mode of operation of the feedback loop becomes unsteady and noise generation occurs. A significant variable component of the signal \( \text{Mag} \) appears during such a mode of operation (Fig. 3-19).
For steady operation, it is recommended to set the value of the feedback gain factor to be not greater than 0.5÷0.7 of the threshold value. Adjustment of the feedback gain factor is performed by means of the text box of **FB Gain**.

**Setting of the operating level for the feedback gain factor requires the following:**

1. Double-click the mouse in the text box of parameter **FB Gain** from the **Main Parameters panel**. The slider for setting the feedback gain factor will appear (Fig. 3-20).

![Fig. 3-20. Slider for setting the parameter FB Gain](image)

2. Increase the value of **FB Gain** and watch the value of signal **Mag** by means of the software oscilloscope.

3. Determine the value of the parameter **FB Gain** that corresponds to the beginning of the noise generation. The beginning of the generation is identified by the appearance of a significant variable component in the signal **Mag** (see Fig. 3-19).

4. Decrease the value of the parameter **FB Gain** and set it equal to 0.5÷0.7 of the threshold value to be used as the value for operating.

NOTE. *It is not recommended to set the value of FB Gain less than 0.3 as the measured signal becomes less informative.*
Performing measurements

If the generation cannot be eliminated even after reducing the FB Gain parameter to 0.3 and below, perform the following actions:

1. Open the feedback loop (the button $\text{FB}$ is not pressed in).

2. Decrease the signal Mag by 20÷50 % by varying the gain coefficients of the lock-in amplifier (parameter $\text{Gain}$) and the pre-amplifier (parameter $\text{Preamplifier}$).

3. Set the value of the signal Mag to 20÷25 by increasing the value of the generator voltage (parameter $\text{Amplitude}$).

   \[\text{NOTE. If small oscillation amplitude of the cantilever is required for scanning of the sample, set the value of the signal Mag to be less 20.}\]

4. Close the feedback loop (the button $\text{FB}$ is pressed in).

5. Once the generation has been eliminated, go to the next procedure (i. 3.1.7 “Set parameters of Scanning” on page 3-92). Otherwise repeat points 1–4.

3.1.7. Set parameters of Scanning

Switch to the Scan tab (the $\text{Scan}$ button on the Main Operations panel) (Fig. 3-21).

![Fig. 3-21. Main Operations panel](image)

The top part of the Scan tab contains a panel which provides control over scanning (Fig. 3-22).

![Fig. 3-22. Control panel of the Scan tab](image)

Another panel is located below. This panel contains a 1D image of the signal measured during the line-by-line scanning. Also another panel containing 2D images of the scanning data is located below.
Selection of AFM mode

Select the mode **Semicontact Topography** from the list **Mode** (scan mode list) of the control panel (Fig. 3-23). The controller performs all the corresponding switching sequences automatically during this selection.

![Mode selection screen](image)

**Fig. 3-23. Selection of the Semicontact Mode**

Selection of an area for scanning

The following recommendations on the selection of preliminary dimensions of the scanning area can be made:

- If there is some preliminary information on surface properties of the sample under investigation and it is certain that the expected surface topography overfalls are within the limits of the Z-scanner range. In this case it is recommended to set the maximum field of scanning;

- If there is no preliminary information on surface properties of the sample under investigation. In this case it is recommended to begin scanning with a small size area, for example about 0.5÷1.0 μm. Then, on the results of scanning of that area, it is possible to set and optimize such parameters as speed of scanning, **Set Point**, **FB Gain**. Then the scanning area can be resized.

The following actions should be performed to select and resize the scanning area:

1. Enable the option of selection and resizing of the scanning area by clicking the button on the panel of 2D images of the scanning data (Fig. 3-24).

![2D images panel](image)

**Fig. 3-24. Panel of 2D images of the scanning data**

1 – boundaries of the selected scanning area; 2 – marker indicating the position of the probe
Performing measurements

2. Use the mouse to alter the size and position of the scanning area (see 1 in Fig. 3-24).

   \textit{NOTE. Alterations of the scanning area size are automatically reflected in the text boxes of parameter \textit{Scan Size}.}

3. Click the button \textcolor{darkred}{\textbullet{I}}. Verify that, within the scanning area selected, the probe reaches the surface without “burying” into it. For that, click the left button of the mouse and, holding it down, move the cursor (see 2 in Fig. 3-24) within the boundaries of the selected scanning area. Displacement of the cursor reflects the true travel of the probe with respect to the sample surface. The level of extension of the piezo-scanner can be controlled by means of the indicator in the bottom part of the window (see Fig. 3-16).

\textbf{Setting of the scanned image size, number of pixels, pace of scanning}

The number of pixels along the X- and Y-axes (parameter \textit{Point Number}), the size of the image scanned (parameter \textit{Scan Size}) and the pace of scanning (parameter \textit{Step Size}) are set by means of selecting the corresponding parameter from a list of these parameters (Fig. 3-25).

When setting parameters \textit{Point Number}, \textit{Scan Size} and \textit{Step Size}, consider the following:

- While altering \textit{Point Number}: \textit{Scan Size} alters; \textit{Step Size} does not alter.
- While altering \textit{Scan Size}: \textit{Step Size} alters; \textit{Point Number} does not alter.
- While altering \textit{Step Size}: \textit{Scan Size} alters; \textit{Point Number} does not alter.

\textbf{Setting of scanning speed}

Selection of the optimal value of scanning speed depends on surface properties of the sample under study, the scanning area dimensions and external conditions. Surface with smooth topography can be scanned at higher speed than that with uneven topography and high overfalls.

At the start, it is recommended to set the line scanning frequency (parameter \textit{Frequency}) within $0.5\div2.0$ Hz (see Fig. 3-25).
Changing the measurement signal

When using the measuring head equipped with displacement sensors, it is recommended to use **Sensor Height** as the signal for acquisition for its higher measurement accuracy. To change the measurement signal perform the following:

1. Open the dialog window **Scan Setting** (Fig. 2-16) by clicking the button from the control panel of the **Scan** tab.

![Scan Settings dialog window]

Fig. 3-26

2. Click the mouse in the text box of the first channel and select **Sensor Height** from the drop-down list that appears.

3. Click the button **OK** to save all the alterations made and to close the dialog window.

### 3.1.8. Scanning

Consider the scanning process on the example of a sample in the form of a rectangular grating (standard grating TGQ-1, 3 μm resolution).

**Trigger scanning**

Scanning of the sample surface should be started after 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 of the **Scan** tab (Fig. 3-27).
Performing measurements

Fig. 3-27

The following actions are the results of clicking the button **Run**:

- Line-by-line scanning of the sample surface is triggered and an image of the scanned area appears line-by-line in the panel of 2D images of the scanning data (Fig. 3-28);

![Fig. 3-28. Surface topography image](image)

- A corresponding image, which reflects the signal measured in real time, appears on the panel of 1D images of the scanning data line-by-line (Fig. 3-29);

![Fig. 3-29. Signal Height](image)

- Some buttons disappear from the control panel of the **Scan** tab, while a number of new buttons appear (Fig. 3-30).

![Fig. 3-30](image)
Should the scanning procedure be interrupted for some reason, click the button **Stop** or use the key <Esc> of the keyboard.

### Alterations of parameters in the process of scanning

#### Tilt subtraction

Consider the given above example (see Fig. 3-28, Fig. 3-29). It is seen that the sample has some tilt along the X-axis.

This tilt can be subtracted directly in the process of scanning by using the button **Subtract**. By default, this button is in the position **None** (Fig. 3-30).

Click this button and select the option **Plane** from the list (Fig. 3-31). This entails subtraction of the plane tilt, and the original image, illustrated in Fig. 3-28, is transformed into the image shown in Fig. 3-32.

![Figure 3-31](image)

![Figure 3-32](image)
Performing measurements

Respectively, the panel of 1D images of the scanning data will be visualizing the measured signal with tilt subtracted from it. Therefore the resulting signal will be transformed as shown in Fig. 3-33, instead of the signal illustrated in Fig. 3-29.

Fig. 3-33. Signal Height

A more detailed description of the function Subtract is provided in the SPM Software, part 1 «SPM Control Program».

NOTE. Transformations of the scanned image performed by means of the function Subtract are not saved in the frames obtained.

Tuning of parameters during the process of scanning

Quality of the obtained surface images essentially depends on such parameters as scanning frequency, Frequency (Fig. 3-34), the value of the operating point, Set Point (Fig. 3-35), and the feedback gain factor, FB Gain (Fig. 3-35). Any of these parameters can be altered directly in the process of scanning.

Fig. 3-34

The special function Pause is used for tuning of scanning parameters. Once this function is enabled, scanning along the slow axis is halted, while scanning along the fast axis goes on continuously. This mode of scanning can be used for optimization of scanning parameters. Profile of the scanned line can be visually monitored and it varies while altering corresponding parameters: scanning frequency, Frequency, the value of Set Point or the feedback gain factor, FB Gain, for example.

The button Restart is used to restart scanning again.
Some recommendations on optimization of scanning parameters

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

Surfaces with smooth topography can be scanned at a speed higher than those with sharp features and rapid overfalls.

It is recommended to start scanning at lower scanning speed, increasing it gradually until distortions start altering the topography profile.

Scanning speed should be reduced when surface features are not read in the direction of scanning.

In measurements on soft materials, the images may be affected by “dragging effects” caused by surface features in the direction of scanning. In the event of such effects, it is recommended to reduce the speed of scanning and, also, to increase the value of Set Point in order to reduce pressure on the sample.

3.1.9. Saving of Measurement Data

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

1. Select File → Save command from the main menu.

2. A dialog box will appear. Choose a folder to store the data (by default, it is the folder C:\Program Files\NT-MDT\Nova).

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

NOTE. By default, the images obtained are stored in files “NoNameXX.mdt”, where XX is the file index in the folder Nova.

3.1.10. Completion of Measurements

To complete operation, perform the following steps:

1. Open the feedback loop (the button is not pressed in).

2. Take the sample aside from the probe. For that, perform the following steps:

   a) Switch to the Approach tab (click the button on the Main Operations panel) (Fig. 3-36).

   Fig. 3-36. Main Operations panel

   b) Double-click in the text box Moving for Backward (Fig. 3-37) on the control panel of the Approach tab. Set the value 2÷3 mm using the slider.
3.2. **Semicontact Error Mode**

3.2.1. **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 being 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.
3.2.2. Preparation for Measurements

The Semicontact Error Mode is based on the Semicontact Mode, which is described in detail in i. 3.1 “Semicontact Mode” on page 3-82.

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

Measuring in the Semicontact Error Mode is carried out with the universal or scanning measuring head. When measuring with the universal measuring head, it can be supplied with any measuring insert except AU020 and AU030. The measuring head is equipped with the semicontact probe.

Main procedures performed during operations by the Semicontact Error Mode

1. Set the Electronic Configuration (i. 3.1.2 on page 3-83).
2. Set the Piezodriver Operating Frequency (i. 3.1.3 on page 3-83).
3. Set Initial Level for the Signal Mag (i. 3.1.4 on page 3-87).
4. Approach the Sample to the Probe (i. 3.1.5 on page 3-88).
5. Setting the Feedback Gain Factor Working Level (i. 3.1.6 on page 3-90).
6. Set parameters of Scanning (i. 3.1.7 on page 3-92).
7. Scanning (i. 3.1.8 on page 3-95).

After the completion of preliminary measurements of surface topography by the Semicontact Mode, perform setting of parameters for operating by the Semicontact Error Mode.
3.2.3. Setting of Parameters

Measurements on the Semicontact Error Mode are performed with the signal \textit{Mag} read through the second measurement channel. Do the following to connect the channel:

1. Open the dialog box \texttt{Scan Setup} (Fig. 3-39) by clicking the button \texttt{Scan Setup} on the control panel of the \texttt{Scan} tab.

![Scan Settings dialog box]

Fig. 3-39. Scan \texttt{Setup} dialog box

2. Select the signal \textit{Mag} as the second signal for registration during the direct pass.

3. Click \texttt{OK} to save alterations and to close the dialog box.

3.2.4. Scanning

Click the button \texttt{Run}, which is on the control panel of the \texttt{Scan} tab, to trigger the process of scanning.

The following actions take place:

- Line-by-line scanning of the sample surface starts and two images appear in the field of 2D visualization of the scanning data. One of the images is an image of surface topography (signal \textit{Height}), while the other one is the error signal (signal \textit{Mag}) (Fig. 3-40);
Fig. 3-40. Images of surface topography and the *error* signal (TGQ-1 grating)

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 3-41).

**3.3. Phase Imaging Mode**

**3.3.1. 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. 3-42).
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 mode is employed, for example, in the study of biological objects, samples with electrical and magnetic properties and a number of other areas.

### 3.3.2. Preparation for Measurements

Before the Phase Imaging measurements, prepare for the measurements and perform measurements of surface topography by the Semicontact mode.

Measuring in the Phase Imaging Mode is carried out with the universal or scanning measuring head. When measuring with the universal measuring head, it can be supplied with any measuring insert except AU020 and AU030. The measuring head is equipped with the semicontact probe.

**Main procedures performed during operations by the Phase Imaging Mode**

1. Set the Electronic Configuration (i. 3.1.2 on page 3-83).
2. Set the Piezodriver Operating Frequency (i. 3.1.3 on page 3-83).
3. Set Initial Level for the Signal Mag (i. 3.1.4 on page 3-87).
4. Approach the Sample to the Probe (i. 3.1.5 on page 3-88).
5. Setting the Feedback Gain Factor Working Level (i. 3.1.6 on page 3-90).
6. Set parameters of Scanning (i. 3.1.7 on page 3-92).
7. Scanning (i. 3.1.8 on page 3-95).

After the completion of preliminary measurements of surface topography by the Semicontact Mode, perform setting of parameters for operating by the Phase Imaging Mode.
3.3.3. Setting of Parameters

Select the mode **Phase Contrast** from the list **Mode** (scan mode list) of the control panel (Fig. 3-43). The controller performs all the corresponding switching sequences automatically during this selection.

![Fig. 3-43. Selection of the Phase Imaging Mode](image)

If manual setting of the initial phase is required, perform the following:

1. Open the additional operations area (button in the right top corner of the screen).
2. Select the signal **Phase** on the software oscilloscope (Fig. 3-44).

![Fig. 3-44](image)

The signal **Phase** varies from 0 to 180 degrees depending on the initial phase of the generator, which in turn can vary from 0 to 360 degrees.

3. Switch to the **Resonance** tab. Vary parameter **Phase** (Fig. 3-45) (initial phase of the generator) and get the value of phase to be approximately in the middle of its range on the oscilloscope (Fig. 3-46). It is not recommended to set its value near the boundaries of the range, 0 or 180 degrees. In this case, all possible variations of the measured phase will have the same sign.
3.3.4. **Scanning**

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 and 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. 3-47).
Fig. 3-47. Images of surface topography and phase distribution of polyethylene

- The panel of 1D images visualizes the signal measured line-by-line (Fig. 3-48)

Fig. 3-48. Signal **Phase**

### 3.3.5. 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 **Phase**);
- Alter the feedback gain factor (parameter **FB Gain**);
- Replace the probe (with different elasticity and resonance frequency).
4. AFM Spectroscopies

4.1. Introduction

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(Height). 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.

4.2. Force-distance Spectroscopy DFL(Height)

In Force-distance spectroscopy cantilever deflection is measured as a function of the scanner z-piezotube extension, i.e. the DFL(Height) curve is obtained.

After the probe has touched the surface any variation in the voltage applied to the z-contact of the scanner (the Height signal) results in a proportional change in the DFL signal. Using the DFL(Height) 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”.

4.2.1. Basic Operations

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the contact mode (refer to Performing Measurements, Part 3, Chapter 2, i. «Constant Force Mode»).

Basic operations:
1. Switching to Spectroscopy Tab (see i. 4.2.2 on page 3-109).
2. Selecting the Function to be Measured, i.e. selecting a function and its argument (see i. 4.2.3 on page 3-109).
3. Selecting Spectroscopy Points (see i. 4.2.4 on page 3-111).
4. Starting the Measurements (see i. 4.2.5 on page 3-113).
5. Viewing Spectroscopy Data and processing them (see i. 4.2.6 on page 3-114).
6. Calculating Adhesion Force (see i. 4.2.7 on page 3-116).

A detailed description of these basic operations is given below.
4.2.2. Switching to Spectroscopy Tab

Switch to the Curves tab (the button on the main operations panel, see Fig. 4-1).

![Fig. 4-1. Control panel of the Curves tab](image)

4.2.3. Selecting the Function to be Measured

1. Select an argument for the function using list \(a\) on the control panel, see Fig. 4-2. In the example below the argument is the \(\text{Height}\) signal.

![Fig. 4-2](image)

\[\text{NOTE.}\]

In spectroscopy measurements one of the following signals can be selected as the argument:

- \(\text{Height}\) – the parameter corresponding to the change of the scanner piezotube length in the Z axis;
- \(\text{SetPoint}\) – the feedback parameter which specifies the magnitude of the feedback loop signal maintained when the feedback is on;
- \(\text{BiasVoltage}\) – the bias voltage applied either to the probe or to the sample depending on the type of the instrument and circuitry settings.

2. In the \(f_1(a)\) list select the quantity to be measured. In the example below the measured signal is \(\text{DFL}\) (Fig. 4-3).

![Fig. 4-3](image)

\[\text{NOTE.}\text{ For each argument }a\text{ and for each group of methods there is a specific set of signals which can be used as the }f_1(a)\text{ measured signal.}\]
3. In text boxes Land and Lift define the range of the argument (Fig. 4-4).

![Image](image_url)

Fig. 4-4

The values are calculated relative to the current piezoscanner position.

- **Land** (in absolute values) – is the distance on which the scanner will extend, i.e. the amount on which the sample will approach the probe.

- **Lift** – is the distance the piezoscanner will retract on, or the distance on which the sample will be withdrawn from the probe.

For the first measurement we recommend to set Land = 0.

**NOTE.** The Height value changes according to the following scheme (Fig. 4-5):

- **curve 1** – the Height value abruptly reaches its maximum set in the Lift box;
- **curve 2** – it linearly decreases to the value defined by the Land box;
- **curve 3** – it linearly increases to the value set in the Lift box;
- **curve 4** – Height returns to its original value.

![Image](image_url)

Fig. 4-5

4. In the control panel set the number of points per curve (the Points in curve parameter) and the time interval for measuring the curve (the Curve Time parameter).

The recommended value for **Point in curve** is 1000, and for **Curve Time** is 1÷5 s.

![Image](image_url)

Fig. 4-6
5. If necessary, set the limits for the signal variation (the \textit{f1(a) safety limits} parameter).

\textbf{NOTE.} When force spectroscopy \textit{DFL(Height)} is made using a probe designated for contact modes the limiting value (the \textit{Level} parameter) should be defined so that it exceeds the \textit{Set Point} value by 5–10 nA. When the probe for semicontact modes is used the limiting value should be 3–5 nA greater than \textit{Set Point}.

\textbf{Suggestions for improving the quality of measurements}

Due to inertia inherent in piezoceramics there is no exact correspondence between the variations of the \textit{Height} value and real piezoscanner displacements. The real value could be obtained when a measuring head with displacement sensors is used. The feedback loop on the \textit{Z-sensor} can be closed by the button \(\bigcirc\) (Fig. 4-7).

![Fig. 4-7. The feedback on the Z-displacement sensor is closed](image)

If the available scanner does not include displacement sensors, then the time for measuring the curve, i.e. the \textit{Curve Time} parameter, should be increased.

\textbf{4.2.4. Selecting Spectroscopy Points}

The software facilitates “binding” spectroscopy data with scanned surface topography. To use this feature choose a desired frame in the \textit{Select Frame} list (Fig. 4-8). As a result the selected scan image will appear in the 2D-data view region.

![Fig. 4-8](image)

Spectroscopy measurements can be conducted:
- In a single point;
- In several points along the same line;
- In nods of a grid.
Performing measurements

In the example that follows measurements in one point will be considered. The procedures for defining coordinates of spectroscopy points for each mode are given below.

**Spectroscopy in a single point**

In this mode the selected curve will be measured the specified number of times in one point:

1. In the **Y Point Set** list select **Point** mode (Fig. 4-9).

   ![Fig. 4-9](image)

2. Define the position of the measurement point by left-clicking in the desired point of the scan region.

3. Set the required number of measurements in the **Curves** text box.

**Spectroscopy in several points along the same line**

In this mode the measurements are performed sequentially in points of the surface along a selected cut.

1. In the **XY Point Set** list select **Line** mode (Fig. 4-10).

   ![Fig. 4-10](image)

2. In the 2D-data view region specify the position of the line along which the measurements will be made (Fig. 4-11). To do this draw a line using a cursor while holding the <Ctrl> key and the left mouse button, or change the position of the line which has been drawn using the mouse.

   ![Fig. 4-11](image)

3. Set the number of points to be measured in the **Positions** text box.
Spectroscopy in nods of grid

In this mode the measurements are performed sequentially in nodes of a selected grid.

1. In the XY Point Set list select Grid (Fig. 4-12).

![Fig. 4-12](image)

By default the grid size is set equal to that of the scan image.

2. While holding the <Ctrl> key use the mouse to specify the size and position of the grid (Fig. 4-13). The size and position of an existing grid can be changed in a usual way using the mouse.

![Fig. 4-13](image)

3. In the Positions text box (Fig. 4-12) define the number of grid nodes along the X- and Y-axes.

4.2.5. Starting the Measurements

To initialize the measurements click the Run button in the left part of the control panel.

When spectroscopy is conducted in several points the point corresponding to the current probe position is marked by a cross.

To interrupt the measurements press the <Esc> key or click the Stop button.
4.2.6. Viewing Spectroscopy Data

The results of spectroscopy measurements will be displayed on a plot (Fig. 4-14). In the example being discussed the \textit{DFL(Heigth)} curve was measured in a point.

If the measurements were repeated several times or in a number of points then a set of plots will be displayed as a 2D map (Fig. 4-15). Each segment corresponds to a measurement in one point. In the example below the measurements were made on a grid consisting of 25 points.

The curves in Fig. 4-14 correspond to the functions obtained while the argument was changed from its maximum value to minimum (red curve) and in the opposite direction (blue curve).

\begin{itemize}
  \item \textbf{NOTE.} When the argument is \textit{Height}, the curves are called approach (red) and retract (blue) curves.
  \item \textbf{NOTE.} The button \textit{ enables either one of the curves or both curves to be displayed in the plot.}
\end{itemize}

Using a too “soft” cantilever at high humidity may cause “sticking”. If this is the case the \textit{DFL(Heigth)} curve will look as shown in Fig. 4-16.
Spectroscopy frame is automatically appended to a data file. To save spectroscopy data in a separate text file click the button on the control panel of the spectroscopy frame region (Fig. 4-17).

The acquired data can be viewed on the Data tab. The images can be further analyzed and processed using the image processing software tool (refer to the manual «SPM Software», Part 2).

If a scan was selected for obtaining spectroscopy curves then there is a further possibility to view the point in which the spectroscopy curve was measured.
Performing measurements

To use the option, proceed as follows:

1. Switch to the **Data** tab.
2. Select a desired spectroscopy frame (pos. 1 in Fig. 4-18).

3. In the Frame View and Modification window the selected spectroscopy frame (pos. 2) will be displayed together with the scanned surface image (pos. 3) to which the spectroscopy frame was “bound”.

4. If spectroscopy was performed in several points then moving between the segments of the 2D map (pos. 4) you can observe the point on the surface image (highlighted in yellow) the measurements were made in.

### 4.2.7. 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. 4-19).
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:
\[ F = k \times \Delta \text{Height}, \]

where \( k \) – is the cantilever stiffness (see the calibration chart).

To determine the adhesion force measure the value of \( \Delta \text{Height} \). Press the button (Pair Markers) and then use the mouse to set the two markers on the part of the curve containing a slope, as shown in Fig. 4-20.

The values of \( DX \) and \( DY \) measured with the markers are \( \Delta \text{Height} \) and \( \Delta \text{DFL} \), respectively. In the example \( \Delta \text{Height} = 25 \text{ nm} \).

In our case the cantilever stiffness was \( \sim 0.03 \text{ N/m} \). Thus the adhesion force in the spectroscopy point is:

\[ F = 0.03 \text{ N/m} \times 25 \text{ nm} \approx 0.8 \text{ nN} \]
Performing measurements

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 \( \text{Set Point} = 2 \text{nA} \). This force results in the \( \Delta \text{Height} \) value measured as shown in Fig. 4-21. The position of the first marker corresponds to the \( \text{Set Point} \) value, while that for the second marker reflects the level of the DFL signal immediately before the cantilever detached from the sample surface.

![Fig. 4-21](image)

In our example, with the parameter \( \text{Set Point} = 2 \text{nA} \), constant pressing force is maintained:

\[
F = 0.03 \text{ N/m} \times 81 \text{ nm} \approx 2.4 \text{ nN}.
\] (3)

4.3. **Current Spectroscopy Ipr-low(Bias Voltage)**

In current spectroscopy the current through the probe (the Ipr-low signal) is measured as a function of the voltage applied between the probe and the sample (the parameter Bias Voltage). For obvious reasons, current measurements are only applicable to conducting samples.

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the contact mode (refer to the Performing measurements, Part 3, Chapter 2, Section «Constant Force Mode».) The sample shall be installed on a substrate with a spring contact. The connector shall be attached to the BV socket of the base unit.

4.3.1. **Configuring and Making Measurements**

The preparatory and measurement procedures are similar to those discussed in item 4.2 “Force-distance Spectroscopy DFL(Height)” on page 3-108. Hence, only a brief algorithm is given below. Only the stages that differ from the standard procedure are considered in detail.
1. Check that **Contact** is chosen in the controller configuration list (Fig. 4-22).

![Fig. 4-22](image)

2. Configure the block scheme of the instrument for performing electrical measurement. Proceed as follows:

   a. Switch to the **Scan** tab (the button ![Scan](image) on the main operations panel).

   b. In the **Mode** list select **Spreading Resistance** mode (Fig. 4-23).

![Fig. 4-23](image)

These will result in the following connections on the block scheme (Fig. 4-24).

![Fig. 4-24](image)

a) Positions of switches:
   - a) for «Scan by probe» configuration;
   - b) for «Scan by sample» configuration

1. Switch to the spectroscopy tab (the button ![Curves](image) on the main operations panel).

2. In list a on the control panel select **Bais Voltage**. In the f1(a) list choose **lpr-low** (Fig. 4-25).

![Fig. 4-25](image)
Performing measurements

3. Define the range of the argument (using the values of From and To).

4. Set the number of points per curve and the time interval for measuring the curve (Fig. 4-26). The recommended value for time interval Curve Time is 1 second.

5. Choose a spectroscopy mode (in point, along a line or grid nodes, see Fig. 4-27).

6. Define the points for spectroscopy.

7. Start the measurements by clicking the Run button.

4.3.2. Viewing Spectroscopy Data

The results of spectroscopy measurements will be displayed in a plot (Fig. 4-15). In the example being discussed the Ipr-low(Bias Voltage) curve was measured in a point. If the measurements were repeated several times or in a number of points then a set of plots will be displayed as a 2D map.
The curves correspond to the functions obtained while the argument was changed from its maximum value to minimum (red curve) and in the opposite direction (blue curve).

NOTE. The button enables either one of the curves or both curves to be displayed in the plot.

Spectroscopy frame is automatically appended to a data file. To save spectroscopy data in a separate text file click the button on the control panel of the spectroscopy frame region (Fig. 4-29).

The acquired data can be viewed on the Data tab. The images can be further analyzed and processed using Image Processing software tool (refer to the manual «SPM Software», Part 2).
4.4. **Amplitude Spectroscopy Mag(Height)**

In amplitude spectroscopy the amplitude of the cantilever oscillations is measured as a function of the scanner z-tube extension, i.e. the Mag(Height) curve is obtained, where Mag – is the signal proportional to the cantilever oscillation amplitude and Height – 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 contact mode (refer to *Performing measurements*, Part 3, Chapter 2, Section «Semicontact Mode».)

4.4.1. **Configuring and Making Measurements**

The preparatory and measurement procedures are similar to those discussed in item 4.2 “Force-distance Spectroscopy DFL(Height)” on page 3-108. 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. 4-30).

   ![Fig. 4-30](image)

2. Switch to the spectroscopy tab (the button **Curve** on the main operations panel).

3. In list a select **Height**. In the f1(a) list choose **Mag** (Fig. 4-31).

   ![Fig. 4-31](image)

4. Define the range of the argument (using the values of **Land** and **Lift**). The values are calculated relative to the current piezoscanner position.

5. Set the number of points per curve and the time interval for measuring the curve (Fig. 4-32):
NOTE. When completing amplitude spectroscopy it is critical to limit the minimum value of \( \text{Mag} \) (the \( \text{Land} \) parameter) to avoid excessive impact on the probe and the sample. The minimum \( \text{Mag} \) value should be set about 10\% of the corresponding \( \text{Mag} \) value for a probe fully withdrawn from the surface (with typical settings, \( \text{Level} \) amounts 0.5 – 2 nA). With the limits thus set, any range for the \( \text{Height} \) parameter could be specified without danger of damaging the sample, the cantilever or the scanner.

6. Choose a spectroscopy mode (in point, along a line or grid nodes, see Fig. 4-33).

7. Define the points for spectroscopy.

8. Start the measurements by clicking the button \( \text{Run} \).

**4.4.2. Viewing Spectroscopy Data**

The results of spectroscopy measurements will be displayed in a plot (Fig. 4-34). In the example being discussed the \( \text{Mag(Height)} \) curve was measured in a point. If the measurements were repeated several times or in a number of points then a set of plots will be displayed as a 2D map.
The curves correspond to the functions obtained while the argument was changed from its maximum value to minimum (red curve) and in the opposite direction (blue curve).

NOTE. When the argument is *Height*, the curves are called approach (red) and retract (blue) curves.

NOTE. The button enables either one of the curves or both curves to be displayed in the plot.

Spectroscopy frame is automatically appended to a data file. To save spectroscopy data in a separate text file click the button on the control panel of the spectroscopy frame region (Fig. 4-35).

The acquired data can be viewed on the Data tab. The images can be further analyzed and processed using Image Processing software tool (refer to the manual «SPM Software», Part 2).
4.4.3. **Calibration of Cantilever Oscillations Amplitude**

Using the measured Mag(Height) 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 z-tube extension (Δ Height):

\[ Δ \text{ Amplitude} = Δ \text{ Height} \]  

(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:

\[ Δ \text{ Height} = K × Δ \text{ Mag} \]  

(2)

where K – is proportionality constant.

To calculate the calibration coefficient measure the values of Δ Mag and Δ Height. Press the button (Pair Markers) and use the mouse to set the markers on the sloped part of the curve, see Fig. 4-36. The values of DX and DY measured using the markers are Δ Height and Δ Mag, respectively.

![Graph showing calibration](image)

**Fig. 4-36**
Thus, following equation (2) the proportionality constant connecting the cantilever oscillations amplitude with the value of the $Mag$ signal is given by:

$$K = \frac{\Delta \text{Height}}{\Delta \text{Mag}} = \frac{DX}{DY} = 31.7 / 4 = 7.93 \text{ [nm/nA]}$$  \(3\)

The actual amplitude of the cantilever oscillations can be calculated from the following equation:

$$\text{Amplitude} = K \times \text{Mag}$$  \(4\)

In the example, when performing a scan with the parameter $\text{Set Point} = 8 \text{ nA}$, the amplitude of oscillations is 64 nm.

NOTE. It should be mentioned that the actual probe oscillations amplitude is independent from the $\text{Gain}$ parameter specified in the $\text{Lock-In}$ set of parameters on the instrument block scheme (the $\text{Scheme}$ tab on the Additional operations panel). At the same time, a modification of the $\text{Lock-In}$ circuit parameters also changes the calculation coefficient. Therefore, the amplitude signal will have to be recalibrated.

### 4.5. Phase Spectroscopy Phase(Height)

In phase spectroscopy the phase of the cantilever oscillations is measured as a function of the scanner $z$-tube extension, i.e. the $\text{Phase (Height)}$ curve is obtained.

Using the dependence of the phase from the piezotube extension one can judge the stability or, vice versa, instability of a given measurement mode and choose the optimal parameters for making measurements.

Spectroscopy measurements could be started as soon as the probe has been approached to the surface using the contact mode (refer to $\text{Performing Measurements}$, Part 3, Chapter 2, Section «Semicontact mode».)

#### 4.5.1. Configuring and Making Measurements

The preparatory and measurement procedures are similar to those discussed in item 4.2 (page 3-108). Hence, just a brief algorithm is given below. Only the stages that differ from the standard procedure are considered in detail.

To make the measurements complete the following procedures:

1. Check that $\text{SemiContact}$ is chosen in the controller configuration list $\text{SemiContact}$ (Fig. 4-37).
2. Switch to the spectroscopy tab (the button on the main operations panel).

3. In list a select **Height**. In the f1(a) list choose **Phase** (Fig. 4-38).

4. Define the range of the argument (using the values of **Land** and **Lift**). The values are calculated relative to the current piezoscanner position.

**NOTE.** For the minimum limit of the argument (the **Land** parameter) we recommend to set a **Height** value which corresponds to the oscillations amplitude 10–20 % of the **Mag** value observed for a probe fully withdrawn from the sample surface, see Fig. 4-39. This would require the **Mag(Height)** curve to be measured in advance (refer to item 4.4 "Amplitude Spectroscopy Mag(Height)" on page 3-122). In the example below the corresponding **Height** value is about ~50 nA.

5. Set the number of points per curve and the time interval for measuring the curve (Fig. 4-40).
Performing measurements

6. Choose a spectroscopy mode (in point, along a line or grid nodes, see Fig. 4-33).

7. Define the points for spectroscopy.

8. Start the measurements by clicking the button Run.

4.5.2. Viewing and Processing Spectroscopy Data

The results of spectroscopy measurements will be displayed in a plot (Fig. 4-42). In the example being discussed the \( \text{Phase(Height)} \) was measured in one point. If the measurements were repeated several times or in a number of points then a set of plots will be displayed as a 2D map.

The curves correspond to the functions obtained while the argument was changed from its maximum value to minimum (red curve) and in the opposite direction (blue curve).

\[ \text{NOTE. When the argument is } \text{Height the curves are called approach (red) and retract (blue) curves.} \]
NOTE. The button enables either one of the curves or both curves will be displayed in the plot.

From the shape of the Phase(Height) curve one can decide whether the measurements are stable or, vice versa, unstable. The example in Fig. 4-42 shows a curve without hysteresis, which is an indication of a stable mode.

Fig. 4-43 shows a spectroscopy curve typical for an unstable mode. There is an evident hysteresis. A stable mode can be achieved by reducing the real amplitude of cantilever oscillations, i.e. the voltage applied from the generator to the piezodriver (the Amplitude parameter).

Spectroscopy frame is automatically appended to a data file. To save spectroscopy data in a separate text file click the button on the toolbar of the spectroscopy frame region (Fig. 4-44).
Performing measurements

The acquired data can be viewed on the **Data** tab. The images can be further analyzed and processed using Image Processing software tool (refer to the manual «SPM Software», Part 2).