Solver PRO Scanning Probe Microscope

Fluid Measurements
(AU028 measuring insert)

Instruction Manual

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Read me First!

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

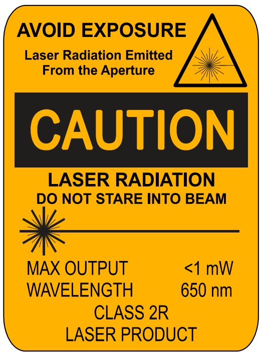

Fig. 1

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

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

Feedback

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

User’s documentation set

The following manuals are included into the user’s documentation set:

- **Instruction Manual** – is the guidance for the preparation of the instrument and other equipment for operation on various techniques of Scanning Probe Microscopy. The contents of the user’s documentation set may differ in dependence on the delivery set of the instrument.
- **SPM Software Reference Manual** – is the description of the control program interface functions, all commands and functions of the menu and, also a description of the Image Analysis module and the Macro Language “Nova PowerScript”.
- **Control Electronics. Reference Manual** – is the guide to SPM controller, Thermocontroller and Signal Access module.

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

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

Solver PRO Scanning Probe Microscope. Fluid Measurements (AU028 measuring insert)

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1. Basic information

To enable measurements in liquids the universal measuring head is supplied with an measuring insert model AU028 (Fig. 1-1) and a open liquid cell.

Fig. 1-1. Universal measuring head with the measuring insert model AU028

Fig. 1-2 shows the items included in AU028:

- adjustment unit;
- cable;
- cell;
- set of substrates;
- lever.

Fig. 1-2. Items included in the set of the measuring insert model AU028
with the open liquid cell
1 – adjustment unit; 2 – cell; 3 – substrates; 4 – lever

The measuring insert is mounted on the measuring head and is connected to it by cable. The measuring insert contains a probe holder. The design of the holder for operation in liquid environment is schematically shown in Fig. 1-3. The probe is installed on the beveled side of the glass pedestal and fixed by clip spring 1.

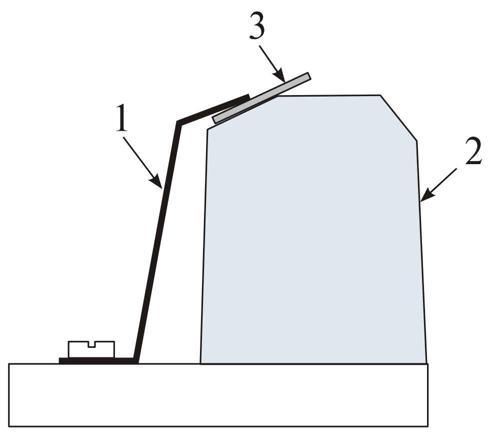


Fig. 1-3.
1 – clip spring; 2 – glass pedestal; 3 – probe

A sample can be installed either directly into the cell or preliminary fixed on a special polycrystalline substrate (see 3 in Fig. 1-2). Clips are intended to fix the sample in the cell.

Depending on the method chosen to mount the cell in the scanner two types of cells could be used:

- a cell with a metal base 15 mm in diameter can be mounted on the magnetic holder of the scanner;
- a cell with a polycrystalline base 19 mm in diameter can be fixed in the probe holder with clips.

Measurement techniques

The following techniques are available for measurements in liquids:

- Contact AFM;
- Semicontact AFM;
- Lateral Force Imaging;
- Adhesion Force Imaging;
- Force Modulation Mode;
- Phase Imaging Mode;
- AFM Lithography.

Specifications

Parameter	Value
Maximum sample dimensions	14.5x14.5x2.5 mm
Maximum volume of liquid	1 ml
Measuring insert dimensions	70x38.5x27.5 mm
Cell dimensions	∅32x8.5 mm
Connector cable length	170 mm
Material	Titan
Measuring insert mass	72 g
Cell mass	11 g
Inner diameter of the cell	21 mm
Cell depth	3.5 mm

2. Preparation for operation

The instrument is supposed to be prepared for operation in “Scanning-by-sample” configuration. All relevant connections are assumed to be made (see *Performing Measurements*):

1. The interface card is installed.
2. The software is set up.
3. The connection of the instrument electromechanical parts is made.
4. The elecromechanical parts of the instrument are configured for “scanning by sample”:
   - The scanner is installed in the positioning device of the approach unit;
   - The scanner and the measuring head are connected to the approach unit of the instrument.
5. The scanner calibration parameters are loaded.

Basic operations to be performed in preparation for using AU028 cell are:

1. Probe installation (see i. 2.1 on page 8).
2. Installation of the measuring insert on the measuring head (see i. 2.2 on page 10).
3. Laser beam alignment onto the cantilever (see i. 2.3 on page 11).
4. Centering the scanner (see i. 2.4 on page 14).
5. Sample installation (see i. 2.5 on page 14).
6. Installation of the measuring head (see i. 2.6 on page 16).
7. Adjusting the optical system for detecting the cantilever deflection (see i. 2.7 on page 17).
8. Installation of the protective hood (see i. 2.8 on page 21).

A description of the listed operations is given below.

2.1. Probe installation

1. Verify that the glass pedestal is clean. If required, wipe it with a pileless tissue, having loosened the clip fixing screw and drawn the clip aside.
2. Put the measuring insert on a flat surface with the probe holder up.
3. Use the special lever to install the probe. Grasp the clip spring with the lever brace and gently pressing the handle unbend the spring (Fig. 2-1).

ATTENTION! To avoid deformation of the spring unbend it only to the minimum angle required.

Fig. 2-1. Probe installation
The arrow shows the direction of applied force

4. Using tweezers put the probe on the beveled side of the glass pedestal under the spring. The length of the chip part extending above the upper side of the glass pedestal should be about 0.8 mm (i.e. the chip edge with the cantilever should be approximately above the centre of the glass pedestal upper side).
5. Lower the end of the clip spring and remove the lever (Fig. 2-2).

Fig. 2-2. Probe installed in the holder

2.2. *Installation of the measuring insert on the measuring head*

1. Using the cable connect the measuring insert to the measuring head (Fig. 2-3).

ATTENTION! To avoid damage of the electronic circuitry power off the computer and the controller before connecting or disconnecting the adjustment unit.

Fig. 2-3. The measuring insert is connected to the measuring head

2. Retract the hold-down by turning clockwise hold-down knob 2 (Fig. 2-4). If required, draw back the photodiode.

Fig. 2-4. Installation of the measuring insert on the measuring head
1 – XY screws for tuning the adjustment unit, 2 – hold-down

3. Install the measuring insert so that the supporting balls rest on the sapphire platforms of the measuring head.
4. Tune the XY screws until their stops rest upon the corresponding seats.
5. Clip the measuring insert to the stops by turning the down-hold knob counterclockwise.

2.3. Laser beam alignment onto the cantilever

1. Power on the computer and start the control program.
2. Turn on the SPM controller.
3. The laser switching-on and switching-off is controlled by Laser button (to the right on the main parameters panel). The laser is automatically switched on upon starting the program.
4. Take the measuring head and lift it about 10-15 cm above a sheet of paper using the latter as a screen for observing the shape of the laser spot (Fig. 2-5).

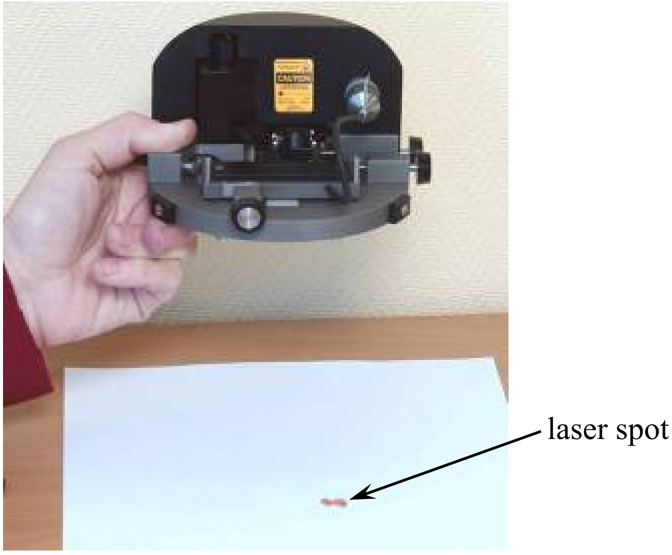


Fig. 2-5

Three are three possibilities here:

a. Undistorted image of the laser spot is observed on the screen (Fig. 2-6). This means that the laser beam hits neither the cantilever nor the probe tip;

b. The image of the laser spot is distorted. The beam partly hits against some part of the design or the cantilever (Fig. 2-7). It should be noted that quite a variety of images is possible (as, for instance, in Fig. 2-7);

c. No image of the spot is seen on the screen. This means that the laser beam hits either the chip (Fig. 2-8) or the probe holder and cannot leave the measuring head.

Undistorted image on the screen:

The beam passes free

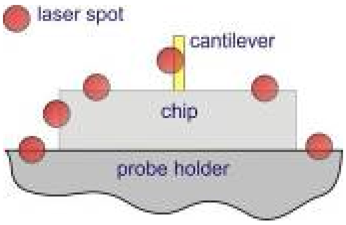
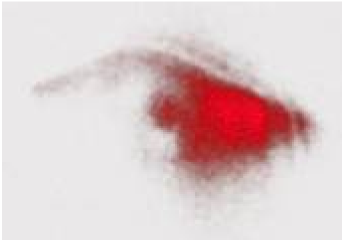


Fig. 2-6

Distorted image of arbitrary shape on the screen:



The beam touches an edge

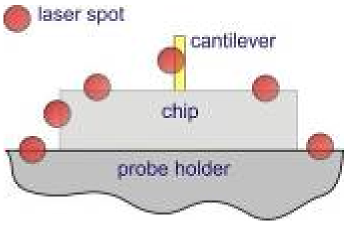


Fig. 2-7

No image on the screen:

The beam hits the chip or the holder

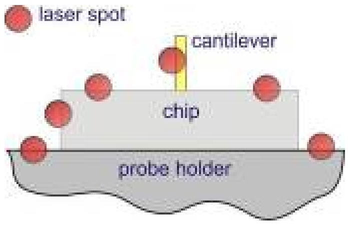


Fig. 2-8

Moreover, the design of the measuring head allows to visually examine the cantilever and the surrounding parts thus enabling the determination of the approximate location of the laser beam.

To align the laser beam onto the cantilever the following steps are required:

1. Rotating screw 2 (Fig. 2-9) achieve the undistorted shape of the laser beam. Generally, the laser spot would appear in position 1 (see Fig. 2-10).

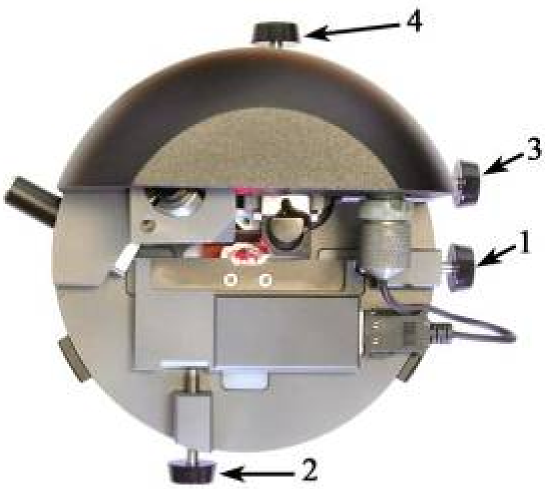


Fig. 2-9
1, 2 – XY screws for tuning the adjustment unit;
3, 4 – photodiode adjustment screws

2. Rotating screw 2 move the beam at the right angle to the front edge of the chip (**1→2** in Fig. 2-10) until the laser spot becomes distorted again.

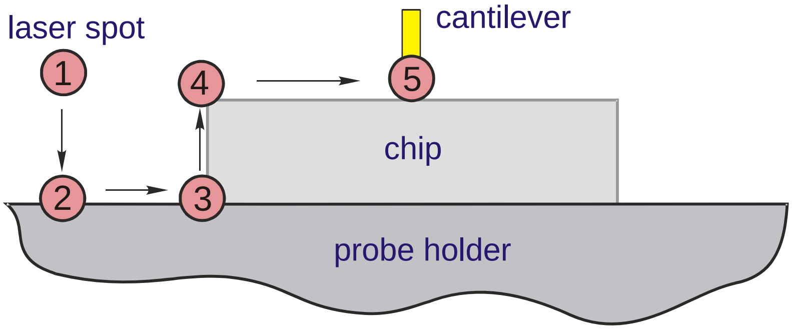
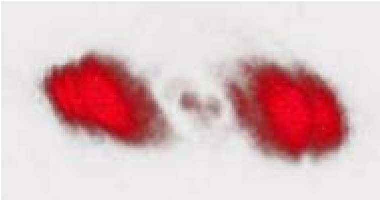


Fig. 2-10. Moving the laser spot in general case

3. Rotating screw 1 move the beam parallel to the front edge of the chip. The two alternatives are possible:
    a. The laser spot moves along the edge of the holder (2→3): in this case the spot disappears when it hits the chip;
    b. The laser spot moves along the edge of the chip (4→5): in this event, when the beam hits the cantilever an interference pattern is observed (Fig. 2-11). Now the laser beam is at the base of the cantilever. Move it towards the cantilever tip. The laser beam alignment onto the cantilever is complete.

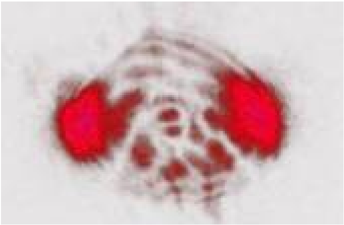
4. When the spot disappears rotate screw 2 moving the laser beam towards the end face of the chip (3→4) until the laser spot reappears. Now the laser beam is at the edge of the chip (position 4).
5. Use screw 1 to move the beam along the front edge of the chip (4→5) until an interference pattern is observed (Fig. 2-11). Now the laser beam is at the cantilever base.

6. Move the laser beam towards the cantilever tip.



a) rectangular cantilever

b) triangular cantilever, the beam hitting one of its arms



c) triangular cantilever, the beam hitting the cantilever tip

Fig. 2-11. Image of the spot when the laser beam hits the cantilever

2.4. Centering the scanner

Prior to the cell installation 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 manual approach screw on the manual approach unit (Fig. 2-12) move the positioning system in its lowermost point.
2. Place the measuring head with its legs onto the seats of the universal sample stage, the stage facing the operator (Fig. 2-12).

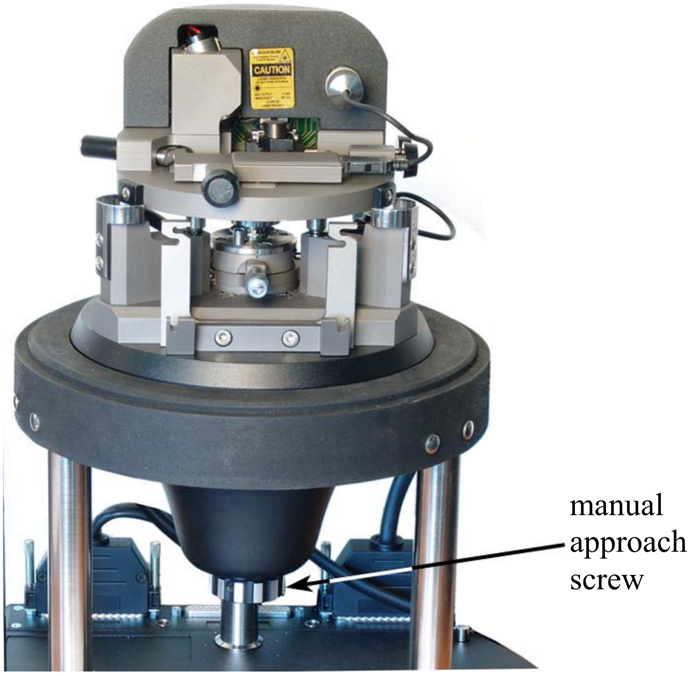


Fig. 2-12. Installation of the measuring head

3. Looking at the head from above, use the positioning system micrometer screws to align the probe with the scanner axis (Fig. 2-13).

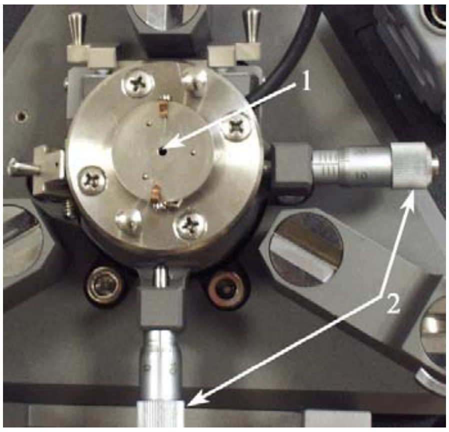


Fig. 2-13.
1 - the hole which is situated on the scanner axis;
2 – the positioning system micrometer screws

4. Remove the measuring head from the universal stage.

2.5. Sample installation

1. Place the cell on a flat surface.
2. Install the sample (or the substrate with the fixed sample) in the cell and secure it with the clips (Fig. 2-14).

Fig. 2-14. Substrate with the sample installed in the cell

NOTE. The sample should be positioned with the scan region as close to the center of the cell (i.e. to the scanner axis) as possible. Otherwise a surface tilt appears when scanning which limits the application of some techniques.

3. Fix the cell with the sample on the scanner sample holder so that after the measuring head has been installed the clips are at the right angle to the longest side of the measuring insert (Fig. 2-15 and Fig. 2-16):

- During installation on the sample holder the polycrystalline base of the cell should be pulled under the clips from the side containing two balls;
- When installing on the scanner with a magnetic holder a cell with a metal base should be used.

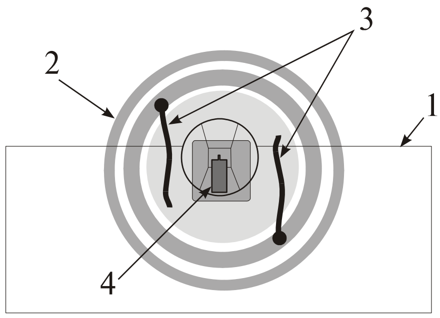


Fig. 2-15. Relative position of the measuring insert and the clips
1 – adjustment unit, 2 – cell,
3 – clips, 4 – probe

Fig. 2-16. The cell installed on the sample holder

2.6. Installation of the measuring head

1. Flood the cell. The level of liquid should be sufficient to cover the cantilever with the adjacent part of the glass pedestal after their immersion, and yet, not excessive to prevent spillage.

NOTE. If a certain region of the sample is to be imaged then prior to flooding the cell the measuring head should be installed and the sample region of interest should be positioned below the probe using the positioning system screws. Then remove the measuring head and flood the cell.

ATTENTION! Spilling liquid on the scanner can cause its damage.

2. Place the measuring head with its legs onto the seats of the universal stage.

NOTE. Before installing the measuring head it is recommended to put a drop of liquid on the glass pedestal below the projecting part of the probe. Thus trapping of air bubbles is avoided.

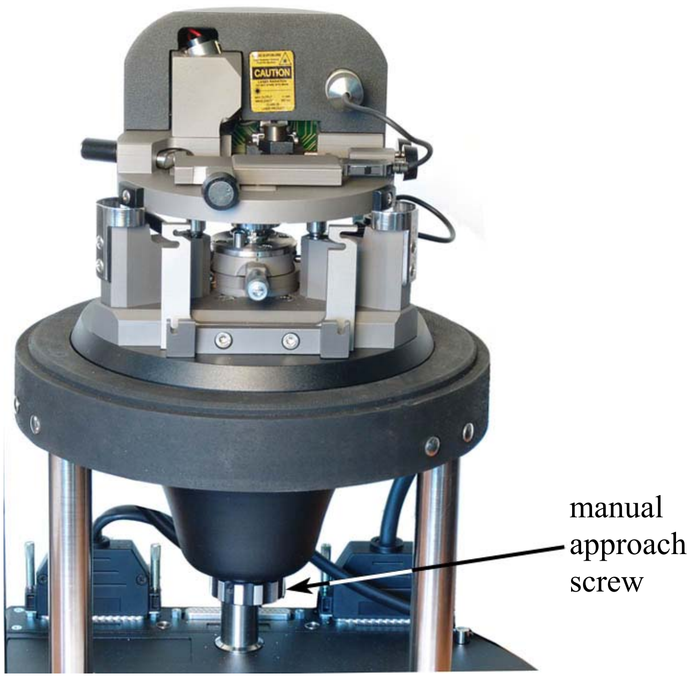


Fig. 2-17. Installation of the measuring head

3. Put the measuring head cable in its designated holder (Fig. 2-17).
4. While looking at the instrument from a side, use the manual approach screw to bring the cell to the probe until it is in one plane with the cell edge.

2.7. Adjusting the optical system for detecting the cantilever deflection

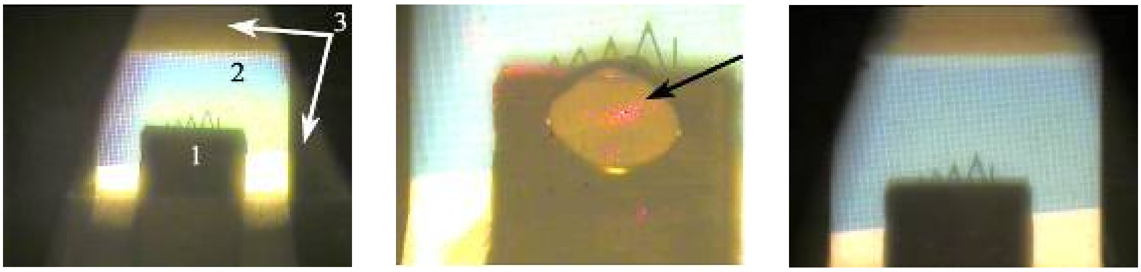
2.7.1. Adjusting with the optical viewing system

It is recommended to use the optical viewing system to adjust the detection system for operation in liquid.

Proceed as follows:

1. Prepare the optical viewing system for operation as specified *Performing Measurements*, Appendix.
2. Focus the videomicroscope on the cantilever.
3. Turning the manual approach screw and monitoring the sample position on the display (Fig. 2-18) bring the sample to the probe so that the edge of the glass pedestal with the probe dips in the liquid.

If bubbles appear (see Fig. 2-18 b), remove the measuring head and carefully wipe the liquid off the probe and its holder using a pileless tissue. Remount the measuring head on the universal stage.



a) probe in the air;
1 – probe, 2 – sample,
3 – chamfered sides of the glass pedestal

b) the probe submerged in the liquid; the arrow shows an air bubble

c) the probe submerged in the liquid; air bubbles are absent

Fig. 2-18. The image on the display of the optical viewing system during approach

4. Bring the sample to the probe at the distance of 0.5÷1 mm by carefully turning the manual approach screw until the sample surface comes to focus.
5. Align the laser beam onto the cantilever using screws 1 and 2 (Fig. 2-19).

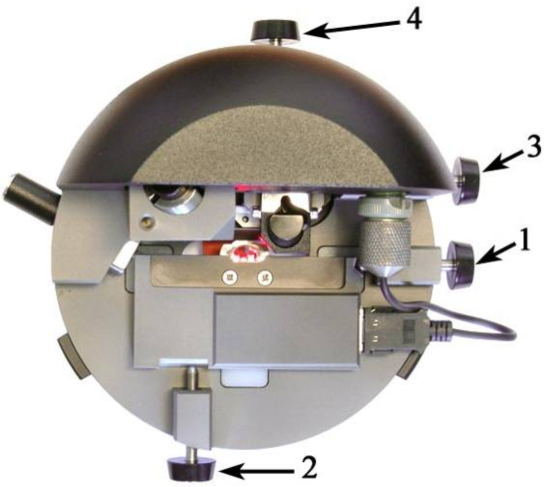


Fig. 2-19. Measuring head
1, 2 – XY screws for tuning the adjustment unit;
3, 4 – photodiode adjustment screws

Further, precise alignment of the laser beam onto the cantilever tip shall be performed based upon the magnitude of the total photodiode signal. This procedure is implemented in the control program:

1. Switch to the **Aiming** tab (click the 

button on the Main Operations panel).
2. Carefully turning XY screws 1 and 2 for tuning the adjustment unit, achieve the maximum value of the **Laser** signal (17÷25 nA).

NOTE. If there is no signal on the photodiode indicator but the laser beam is aligned onto the cantilever, as could be seen on the optical viewing system display, then the photodiode might be in a position where the laser beam misses it. If this is the case, rotate the photodiode adjustment screws until the signal appears.

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

*NOTE. If the **Laser** signal decreases to zero when turning any of the 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.*

2.7.2. Adjusting without using the optical viewing system

1. Switch to the **Aiming** tab (click the 

button on the Main Operations panel). Usually, after pointing the laser beam on the cantilever the photodiode indicator starts displaying nonzero readings.
2. Turning the manual approach screw bring the cell and the holder closer together until the probe dips into the liquid. At this the value of **Laser** signal on the photodiode indicator would fall to zero.
3. Turn screw 2 (Fig. 2-20) 10÷25° **clockwise** (at this the laser spot moves towards the chip) until some **Laser** signal appears.

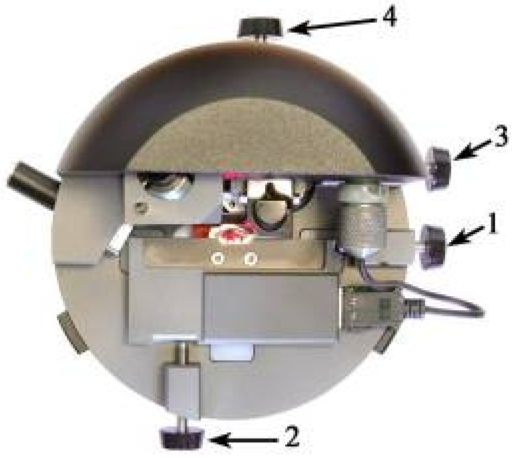


Fig. 2-20. Measuring head
1, 2 - XY screws for tuning the adjustment unit; 3, 4 – photodiode adjustment screws

4. Then turn screw 1 **clockwise** while observing the variation in the **Laser** signal. A criterion for hitting the cantilever is the presence of maximum in Laser signal when turning screw 1. In case of a triangular cantilever the laser beam hits one of its arms.
5. Turn photodiode adjustment screws 3,4 to achieve the maximum increase in **Laser** signal.

*NOTE. 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 **Laser** signal decreases to zero it means that the laser beam hits the photodiode edge. In this event the corresponding screw shall be turned in the opposite direction.*

6. Move the laser beam in the direction away from the chip by alternately turning the adjustment screws. Tune on the maximum value of **Laser** signal (17÷25 nA).
7. Turn photodiode adjustment screws 3 and 4 (Fig. 2-20) center the laser on the photodiode indicator. The values of **DFL** and **LF** signals shall be close to zero while the magnitude of the total **Laser** signal shall remain fairly large.

For a triangular cantilever it is advised to ensure that the laser beam hits the tip of the triangle rather than one of its arms. To do this:

1. Shift the laser beam closer to the chip without changing the laser horizontal position in order to reduce **Laser** signal approximately twice. The laser adjusted correctly, the laser beam now passes between the cantilever arms reflecting to some extent from both of them.
2. When turning the laser horizontal displacement screw left and right two maxima of **Laser** signal shall be observed corresponding to the two arms of the triangular cantilever.
3. To return the laser beam to the triangle tip position the laser beam between the arms to obtain minimum **Laser** signal and then move the laser beam from the chip to the triangular tip.

A criterion for the right alignment of the laser beam onto the triangular cantilever tip is that the **Laser** signal decreases upon turning the laser adjustment screws.

When using the procedure described above for multy-beam probes the laser beam is likely to be aligned onto an outer beam. For more precise laser beam positioning use the optical viewing system. Moreover, the laser beam can be aligned onto the required probe beam by moving the former along the front edge of the chip and noting the number of maxima in the **Laser** signal, each of which corresponds to an arm of the triangular cantilever or to a rectangular cantilever.

2.8. Installation of the protective hood

The protective hood shall be used in the following cases:

− when high resolution in XY-plane or Z-direction is desired;
− for temperature measurements;
− for protection from temperature shocks;
− to reduce acoustic noise.

To install the protective hood proceed as follows:

1. Insert the measuring head cable clip in the special holder on the approach unit (Fig. 2-21).

Fig. 2-21. The measuring head cable is secured in the holder

2. Place the protective hood on the supporting chuck of the approach unit.
3. Ground the protective hood by plugging the special cable of the approach unit into the grounding jack on the hood (Fig. 2-22).

Fig. 2-22. The protective hood is installed on the approach unit
1 – grounding jack

4. Turn on the Vibration Isolation system.

The instrument is ready for performing the measurements.

3. Features of the performing measurements in liquid

3.1. Contact AFM

In contact AFM mode of operation all the procedures are similar to performing the measurements in air (see *Performing Measurements*, part 3, chapter "Contact AFM").

3.2. Semicontact AFM

Nitride cantilevers with in-liquid resonance frequency in the range 20÷40 kHz are recommended for use with measuring insert model AU028.

As opposed to the cantilever resonance curve in air which contains only one working peak, the curve in liquid features a multitude of peaks. Some of them are suitable for operation while the others are not. The working frequency shall be chosen correctly in order to ensure successful operation.

We suggest two procedures for choosing the working frequency: one is manual and the other is automated.

When using cantilevers of a new type for the first time the manual adjustment of the working frequency is preferable. This manual procedure (described below) allows finding the working frequencies for cantilevers of practically any type.

Subsequently, the automatic adjustment can be used (see Appendix "Semicontact AFM. Setting the working frequency in automatic mode" on page 34) provided it has been verified that the resulting frequencies are the same.

The basic steps for the preparation of the instrument and performing the measurements of the piezodriver working frequency in the manual mode are:

1. Approach the Sample to the Probe (see i. 3.2.1 on page 23).
2. Setting the piezodriver working frequency (see i. 3.2.2 on page 26).
3. Preparation for scanning (see i. 3.2.3 on page 31):
   − Selection of working point (**Set Point**);
   − Setting of the working level of the feedback gain (**FB Gain**).
4. Scanning (see i. 3.2.4 on page 33).
5. Saving results (see i. 3.2.5 on page 33).
6. Finishing the operation (see i. 3.2.6 on page 33).

3.2.1. Approach the Sample to the Probe

When applying semicontact microscopy techniques for operation in liquid the approach procedure for contact-mode measurements should be used.

Contact-mode approach procedure:

1. Select **Contact** in the controller configuration list on the main parameters panel (Fig. 3-1).

Fig. 3-1

2. Switch to the **Approach** tab (click the button 

on the Main Operations panel) (Fig. 3-2).

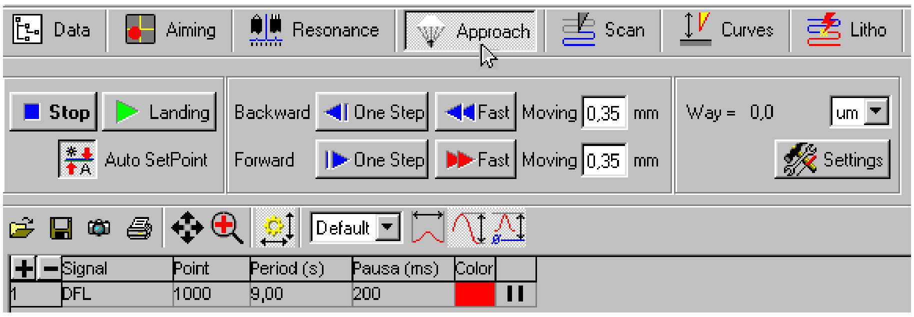


Fig. 3-2

3. Check that **Auto SetPoint** button responsible for the automatic adjustment of the **Set Point** parameter is pressed (Fig. 3-3).

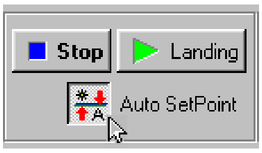


Fig. 3-3

4. Start the approach procedure by clicking 

button.

<u>**The above procedure should yield the following results**</u>

- the value of **Set Point** parameter is automatically set exceeding the current value of **DFL** signal by two units (i.e. **Set Point**=**DFL**+2);
- the feedback is switched on and Z-direction piezo scanner is protracted to the maximum. The protraction of Z-direction scanner is illustrated by the indicator of scanner protraction, located in the left lower field of the program main window (Fig. 3-4). The degree of scanner protraction is shown by the length of the color bar;
- the step motor is switched on, moving the scanner with the sample in the direction of the tip.

Fig. 3-4

Watch the **DFL** signal changes in the software oscilloscope during the approach procedure, while monitoring the progress of the scanner protraction indicator. Wait till the procedure is completed.

If the approach parameters are set correctly, the procedure is completed after some time, leading to the following (see Fig. 3-5):

- **DFL** signal increases to the value of **Set Point** parameter with the feedback maintaining *Z*-direction scanner in the position where **DFL** signal equals **Set Point** with the position corresponding approximately to one half of the scanner protraction range;
- the length of the indicator's color bar decreases and stops somewhere in the middle (pos. 1 in Fig. 3-5);
- the step motor stops;
- the increase of **DFL** signal to the value of **Set Point** parameter is represented on **DFL (t)** trace on the oscilloscope (Fig. 3-5);
- the message **"…Approach Done."** will appear in the journal (pos. 2 in Fig. 3-5).

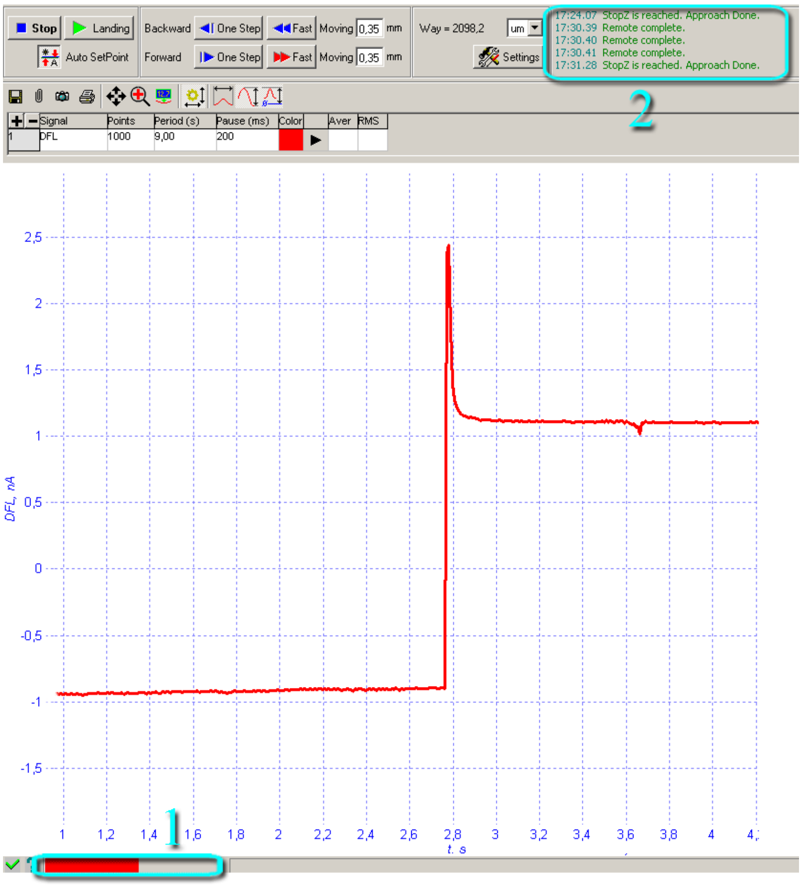


Fig. 3-5. Completing of approach procedure
1 – indicator of scanner protraction; 2 – journal

Special cases

Self-oscillation

It may happen that after the approach procedure is performed and **DFL** signal is increased to the value of **Set Point** parameter, the **DFL(t)** trace shows a considerable increase of **DFL** signal noise (as shown in Fig. 3-6 for example). This means that an oscillation occurs in the feedback loop due to the fact that the value of **FB Gain** parameter is too high. In this case the value of **FB Gain** parameter should be decreased to 0.5-0.7 of the current value. The procedure of **FB Gain** parameter adjustment is described below in item 3.2.3 “Preparation for scanning” on page 32.

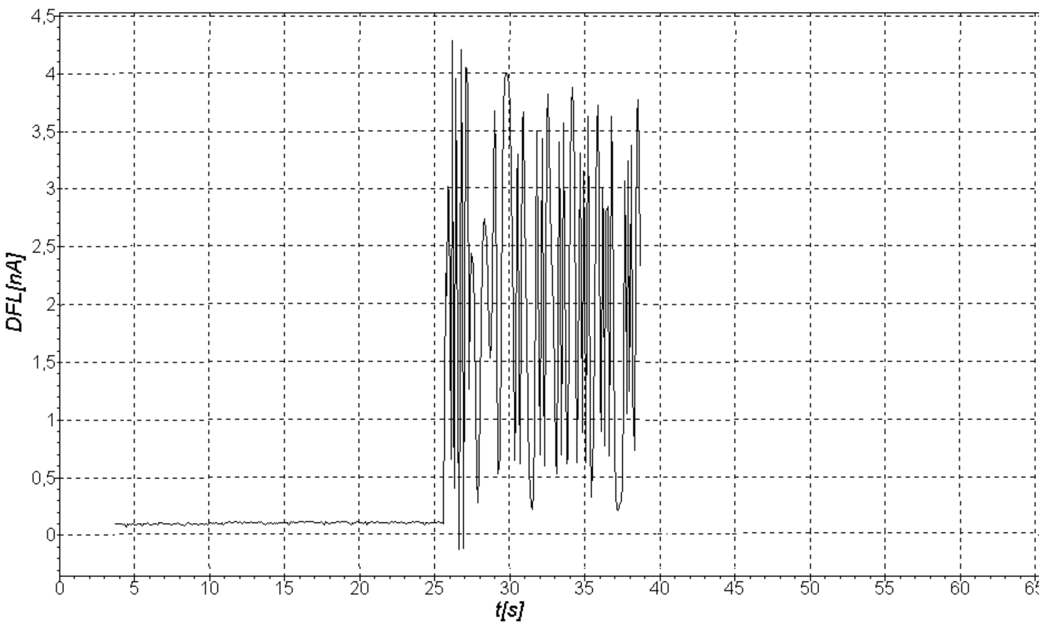


Fig. 3-6

Selecting and setting up the “Set Point” parameter manually

In order to manually enter the value of **Set Point** parameter:

- switch off the **Auto SetPoint** option;
- enter the **Set Point** parameter value in the text box in the main parameters panel.

The recommended initial value of **Set Point** parameter should be equal to the sum of the following: **DFL** signal plus (5÷10) % of laser signal value (i.e. **Set Point=DFL+(0.05÷0.1)×Laser**).

When selecting an optimal value of **Set Point** parameter the considerations presented below should be taken into account:

– The difference between the value of **Set Point** parameter and the initial value of **DFL** signal defines the value of the interaction between the apex of the tip and the sample surface. The greater is the difference between the value of **Set Point** and the initial value of **DFL** signal, the greater is the value of the cantilever deviation and the greater is the force of the tip-surface interaction. Thus, modifying the value of **Set Point** parameter with respect to the initial value of **DFL** signal results in modifying the force of the tip-surface interaction, which provides the possibility to obtain surface topography with different values of the tip-surface interaction.

– If the difference between the value of **Set Point** parameter and the initial value of **DFL** signal is too great, corresponding to a tip-surface interaction of considerable force, it can result in the destruction of the tip, as well as in surface damage during scanning.

– If the difference between the value of **Set Point** parameter and the initial value of **DFL** signal is too little, which corresponds to maintaining the tip-surface interaction of medium force, the operation of the tracking system may turn unstable.

– The value of **Set Point** parameter cannot be less than the value of the initial level of **DFL** signal or cannot exceed the value of laser signal (**Laser**).

3.2.2. Setting the piezodriver working frequency

Contrary to the cantilever resonance frequency response in air the response in liquid contains many peaks. Hence, the determination of the cantilever resonance frequency is performed in a manual mode.

The procedure for setting the piezodriver working frequency is:

1. Turn off the feedback (FB button on the main parameters panel is not pressed in).
2. Select **SemiContact** in the menu for choosing the electronic configuration on the main parameters panel (Fig. 3-7).

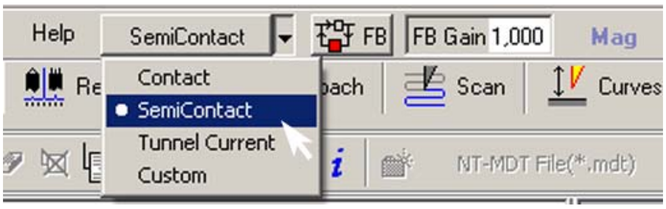


Fig. 3-7

3. Switch to **Resonance** tab (Resonance button on the Main Operation panel) (Fig. 3-8).
4. In the synchronous detector settings panel select the frequency range **Pass Band** <55kHz.

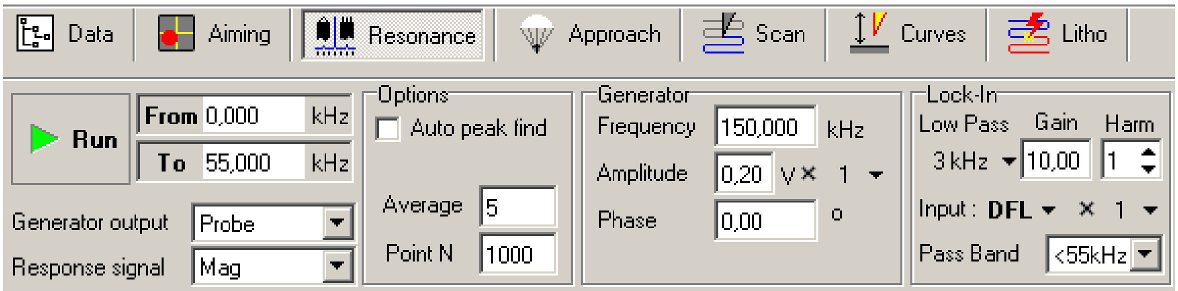


Fig. 3-8

5. Set the range for measuring the frequency response of the cantilever oscillation amplitude (text boxes **From** and **To**) so that it contains the cantilever resonance frequency. It should be noted that the cantilever resonance frequency in liquid is 5÷6 smaller than that in air. The recommended values are **From**=5 kHz, **To**=50 kHz.

6. In **Options** panel (Fig. 3-9) clear the **Auto peak find** check box (the option for automatic setting of the resonance frequency).

Fig. 3-9

7. Set the maximum amplitude of the generator output signal (**Amplitude** parameter):

   a. On **Generator** panel select the gain for **Amplitude** parameter equal to 10 (Fig. 3-10);

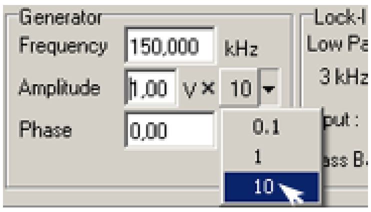


Fig. 3-10

   b. Double-click in **Amplitude** text box and, using the slider that appears, (Fig. 3-11) increase the value of **Amplitude** to its maximum.

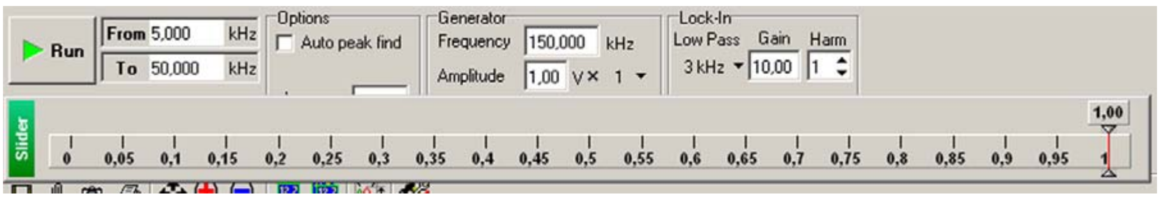


Fig. 3-11

8. Click **Run** button (on the left on the control panel) to obtain the frequency response of the cantilever oscillations amplitude (**Mag** signal). As can be seen in Fig. 3-12 the cantilever resonance curve in liquid contains numerous peaks.

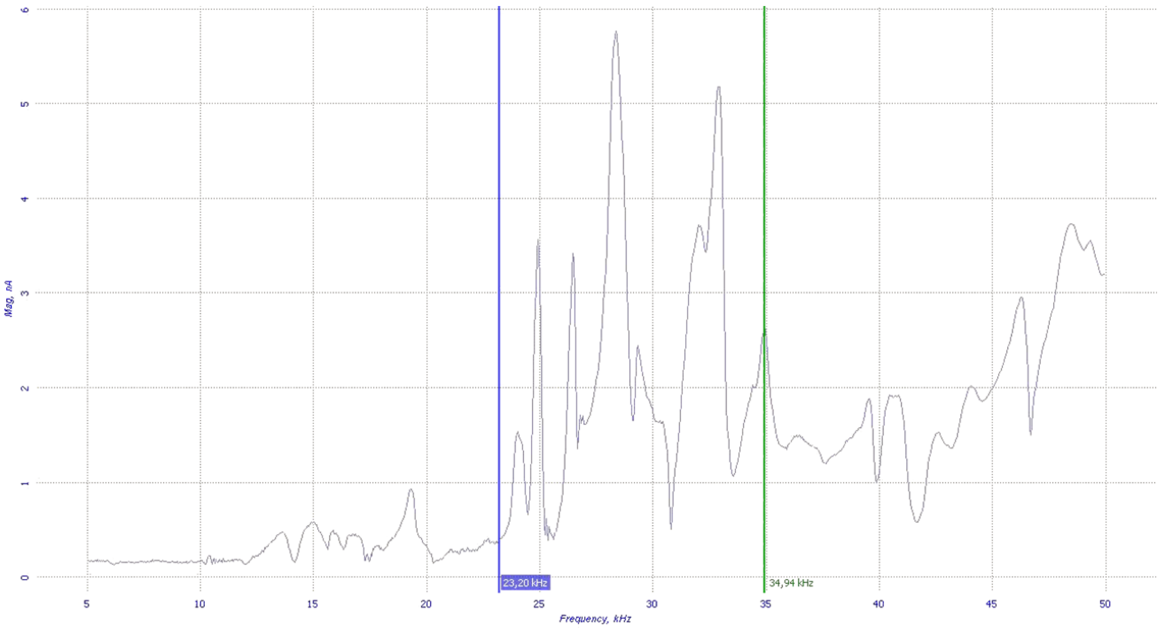


Fig. 3-12

9. Select a range with the highest resonance peaks and input a new range for plotting the frequency response of **Mag** signal. This can be done by two ways:
   - by changing the values of **From** and **To** on the control panel;
   - using the markers that appear on the plot (see Fig. 3-12).

10. Click **Run** button to plot the resonance curve for the selected range (Fig. 3-13).

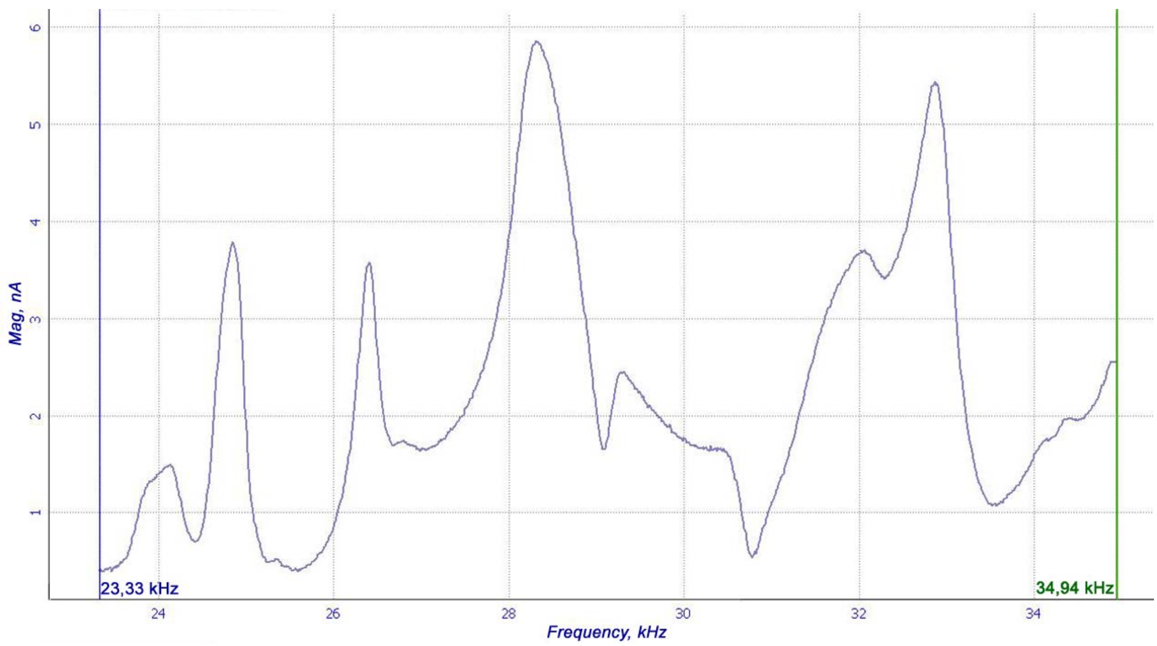


Fig. 3-13. Cantilever resonance curve in the free state

11. In the main parameters panel set **DFL** as a feedback input signal (Fig. 3-14).

ATTENTION! Before switching the signal applied to the feedback input make sure that the feedback is off (FB button is not pressed in).

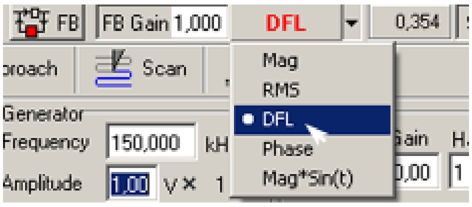


Fig. 3-14

12. Enter a value of **Set Point** parameter that exceeds the current **DFL** value by 1.5÷2 nA.
13. Click FB button to turn the feedback on. The piezotube will advance by half of its length, as can be seen on the piezotube extension indicator, and the cantilever will approach the sample surface.
14. Click **Run** button to repeat the resonance curve plotting procedure for the same range but with the feedback on. (Fig. 3-15).

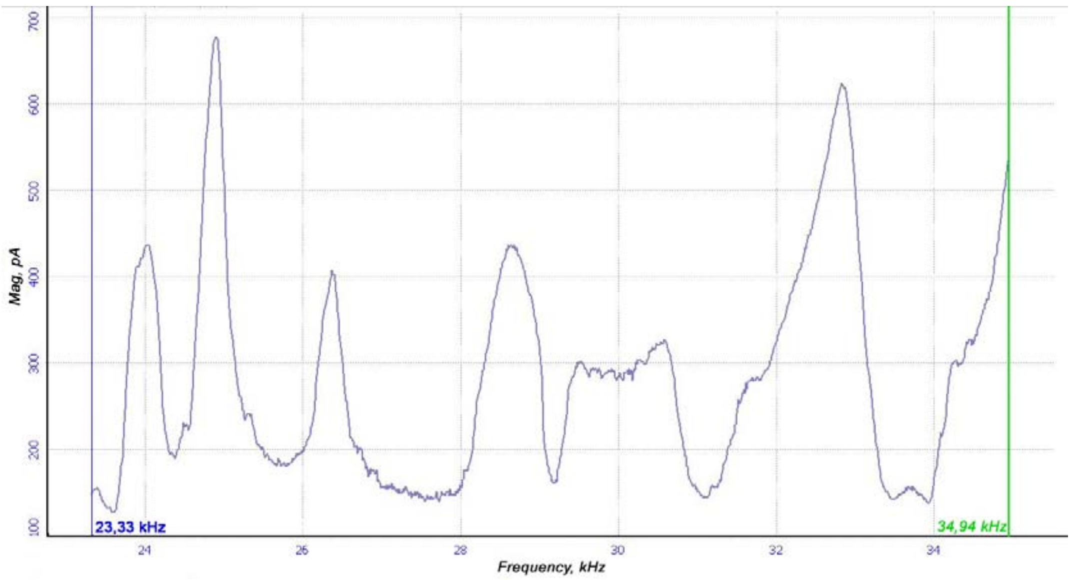


Fig. 3-15. Resonance curve for the cantilever propped to the sample surface

Compare the resonance curve corresponding to free cantilever with that for the propped cantilever. Some of the resonance peaks decreased significantly, others remained practically the same but some increased.

Any frequency at which the cantilever oscillation amplitude increases when the cantilever is propped can be chosen for the measurements. Select a peak which best meets the following two requirements:

– the cantilever oscillation amplitude is the largest when the feedback is off;
– with the feedback turned on (when the cantilever is propped against the sample surface), the drop in the cantilever oscillation amplitude is most pronounced.

15. Turn off the feedback ( button is not pressed in).
16. Replot the cantilever resonance peak in the free state (**Run** button).

17. On the plot obtained find the selected peak. Set the piezodriver resonance frequency (**Frequency** parameter) equal to that of the peak:
    a. In **Frequency** parameter text box on **Generator** panel enter the value corresponding to the maximum of the selected peak. At this a marker will appear on the plot (see Fig. 3-16);
    b. Moving the marker (with the mouse left button pressed) set the frequency value related to the maximum **Mag** signal for the selected peak. **Frequency** text box will display a value at the current marker position.

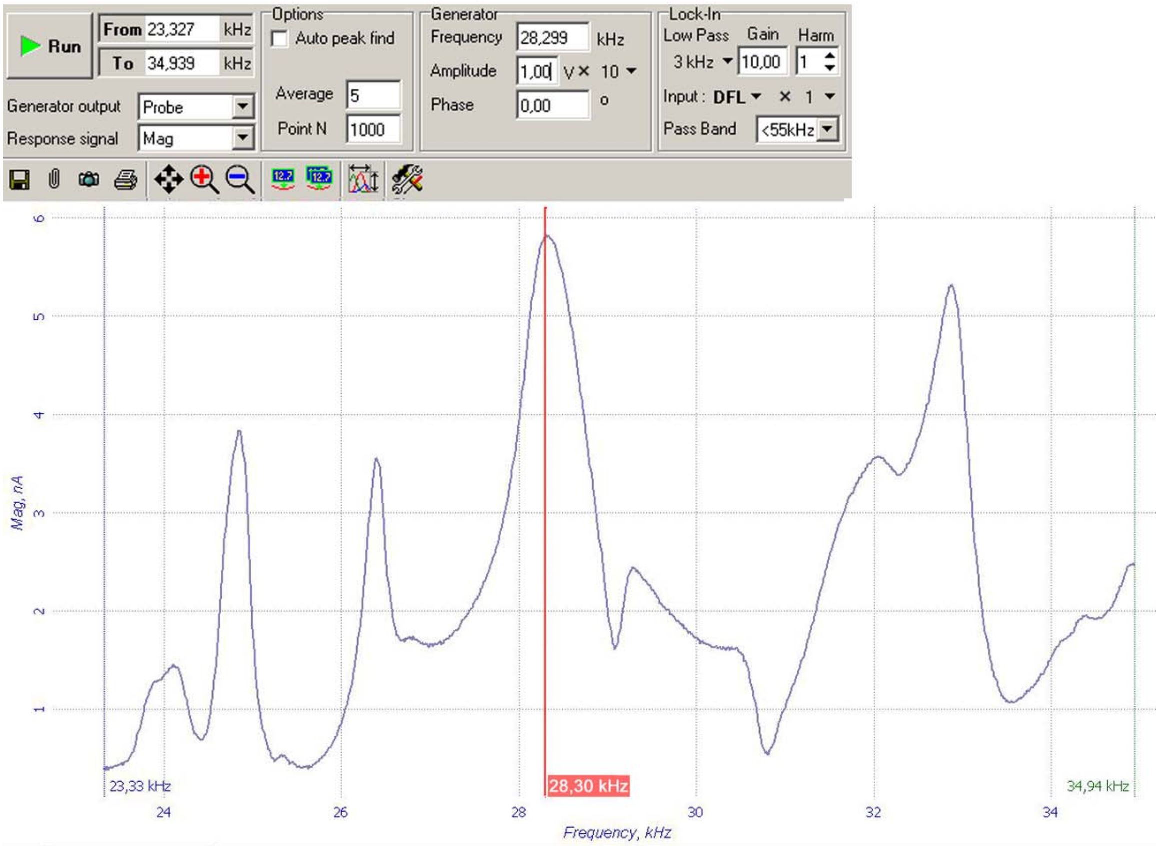


Fig. 3-16

NOTE. The resonance peak may have a flat top. In this case decrease the value of the ***Amplitude*** *parameter.*

Setting the piezodriver working frequency is complete.

3.2.3. Preparation for scanning

Selection of working point (Set Point)

1. On the main parameters panel select **Mag** as a feedback input signal (before switching make sure that the feedback is off).

Fig. 3-17

2. Input the value of **Set Point** equal to approximately half of the **Mag** signal value.
3. Reduce the **FB Gain** value to 0.3÷0.5 in order to avoid excitation after turning on the feedback.
4. Switch to **Approach** tab (
 button on the main operations panel).

5. Turn on the feedback (
 button on the main parameters panel is pressed in). Once the feedback is on, **Mag** signal will fall to the level of **Set Point** (Fig. 3-18).

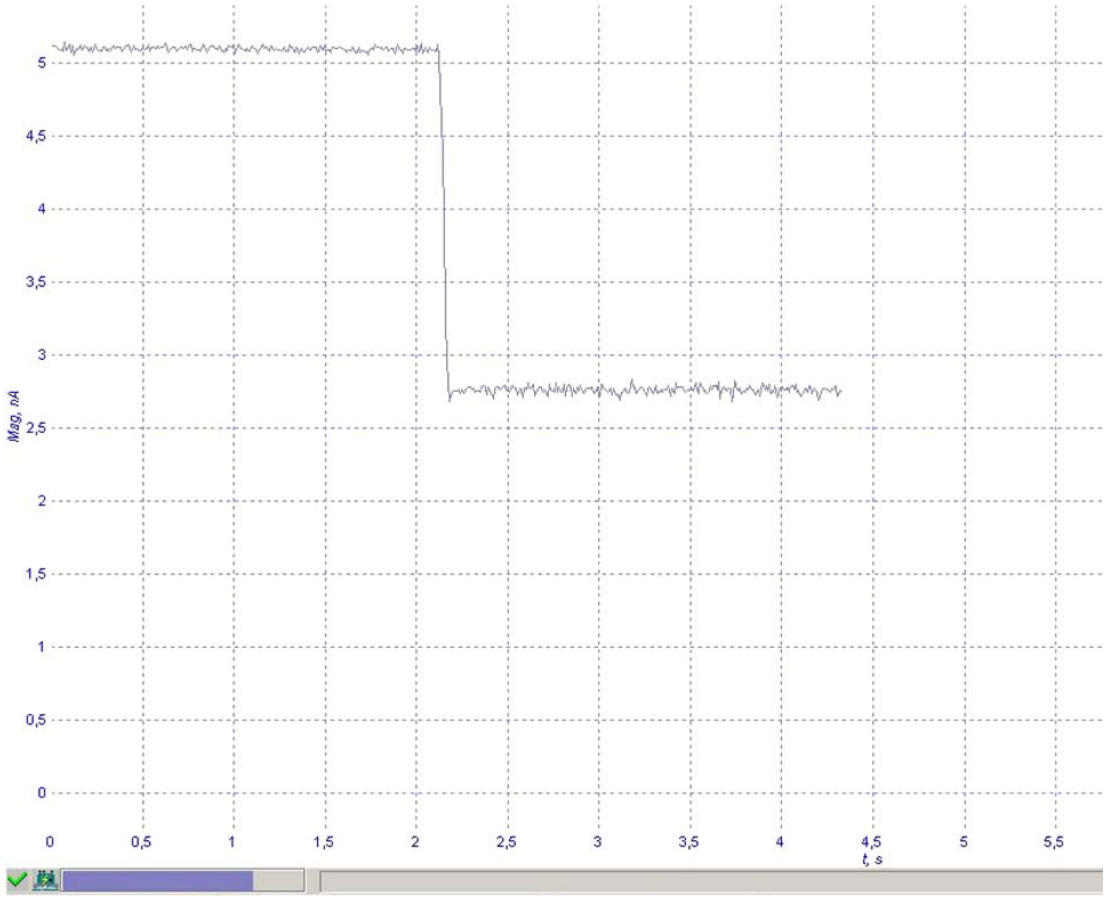


Fig. 3-18

6. Verify that the probe was brought to contact with the sample surface by changing the value of **Set Point** in the range ±20% using the slider and monitoring the piezotube extension on the corresponding indicator. Minor variations in **Set Point** shall not result in any piezotube displacement.

   Otherwise, repeat the approach procedure for a smaller value of **Set Point**:

   1). Reduce **Set Point** by a factor of 0.5÷0.7;

   2). Deactivate **Auto SetPoint**;

   3). Start the approach procedure by clicking  button.

   Once the approach is finished recheck the piezotube behavior with variations in **Set Point**.

*NOTE. During retract from the surface **Mag** signal at a chosen frequency may drop making its use for another approach impossible. Otherwise, the same signal can be reused.*

Setting the working level of the feedback gain

1. Double click with the mouse left button in **FB Gain** text box located on the main operations panel. Using the slider that appears increase **FB Gain** while monitoring the **Mag** signal level on the software oscilloscope (in **Approach** tab).
2. Determine **FB Gain** value at which excitation starts.
3. The beginning of excitation is detected by a sudden rise in the **Mag** signal a.c. component (Fig. 3-19).

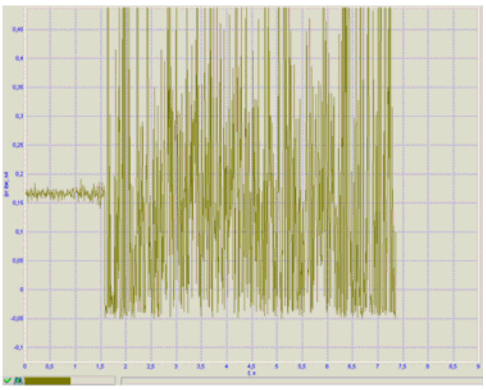


Fig. 3-19. Generation

4. Set **FB Gain** equal to 0.6÷0.7 of the value at which excitation starts.

   A typical **FB Gain** value in liquid is 0.6÷1.2.

   If the excitation is still present after decreasing **FB Gain** to 0.2÷0.3 try setting the piezodriver frequency on another peak. Also, the excitation may result from poor fixing of the sample or the cell.

3.2.4. Scanning

Setting up the scanner parameters (selection of SPM mode, scan region and scanning speed) and the measuring procedures are similar to those in air. See *Performing Measurements* for details.

Given below is the sequence of the basic procedures performed when starting the scan process:

1. Switching to **Scan** tab.
2. Selection of SPM mode.
3. Selection of scan region and the scanning speed.
4. Starting scanning (**Run** button).

3.2.5. Saving results

In the main menu select subsequently **File → Save**.

In a dialog that appears choose a folder for saving the results, enter a name of the file and save if with ***.mdt** extension.

3.2.6. Finishing the operation

1. Switch to **Approach** tab.
2. Retract the sample from the probe at approximately 2÷3 mm (

button for **Backward** on the control panel).

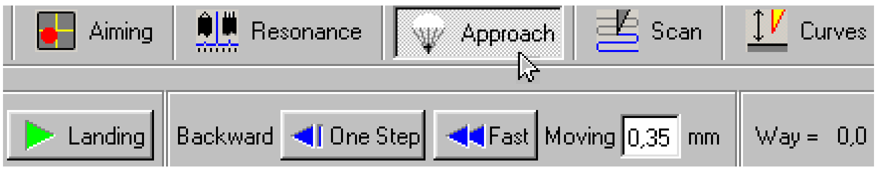


Fig. 3-20

3. Turn off the feedback (

button is not pressed in).
4. Turn off the SPM controller.
5. Turn off the Vibration Isolation system.
6. Close the control program.

After the operation is finished remove drops of liquid from the surface of the glass pedestal using a soft pileless tissue. Also remove the liquid from the cell.

Appendix

1. Semicontact AFM. Setting the working frequency in automatic mode

Nitride cantilevers with in-liquid resonance frequency in the range 20÷40 kHz are recommended for use with measuring insert model AU028.

Before using the procedure for setting the piezodriver working frequency in the automatic mode it is recommended to compare the resonance frequency values determined in both modes for a chosen cantilever type. If the results agree then the automatic procedure should be used for the cantilevers of the given type.

The basic steps for the preparation of the instrument and performing the measurements of the piezodriver working frequency in the automatic mode are:

1. Setting the piezodriver working frequency (see Appendix i. 1.1 on page 35).
2. Approach (see Appendix i. 1.2 on page 38).
3. Preparation for scanning (see Appendix i. 1.3 on page 40):
    a. correction of the working frequency;
    b. selection of **Set Point**;
    c. setting **FB Gain**.
4. Scanning (see i. 3.2.4 on page 33).
5. Saving results (see i. 3.2.5 on page 33).
6. Finishing the operation (see i. 3.2.6 on page 33).

1.1. Setting the piezodriver working frequency

Prior to determining the resonance frequency in the automatic mode the probe shall be manually brought to the sample surface at a distance of 1÷2 mm. The optical viewing system can be used for this purpose. With the videomicroscope focused on the cantilever, proceed with manually approaching the sample to the probe until the sample surface comes to focus.

To set the piezodriver working frequency proceed as follows:

1. Switch to the **Resonance** tab ( button on the main operations panel) (Fig. 1-1).

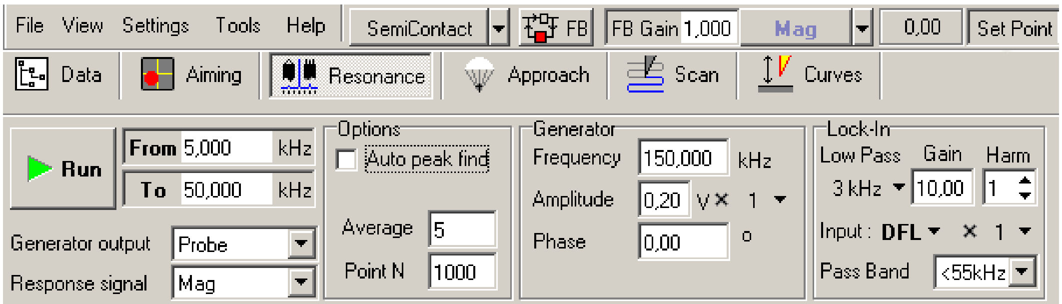
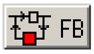


Fig. 1-1

2. Set the range to perform search for the resonance frequency (text box **From**, **To**).

   In the case when the resonance frequency in liquid is unknown for the given probe, define an approximate frequency range by measuring thermal noise of the cantilever. This is done as follows:

   a. Disable the feedback loop (the button  is not pressed in).

   b. Set the following parameters from the control panel of the tab **Resonance** (Fig. 1-2):

      – Clear the check box **Auto peak find**;
      – **Average** ≈ 20;
      – **Point N** ≈ 3000;
      – **Amplitude** 0;
      – **Gain** 100;
      – Gain coefficient of the synchronous detector **×10**.

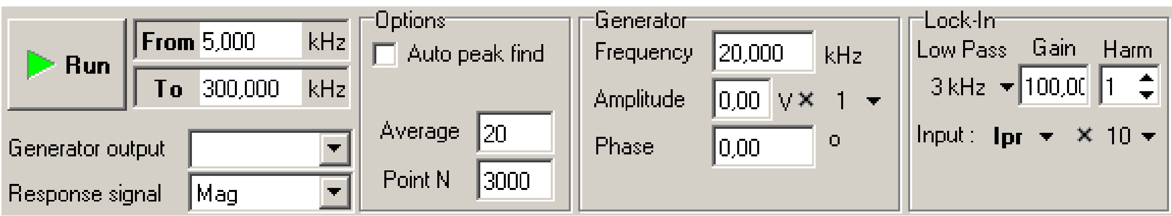


Fig. 1-2

c. Set a frequency range to measure thermal noise:
   - **From** ≈ 5 kHz;
   - **To** ≈ 300 kHz.

d. Using the Block Scheme of the instrument, set the key into the position where voltage from the generator is applied to the probe (Fig. 1-3).

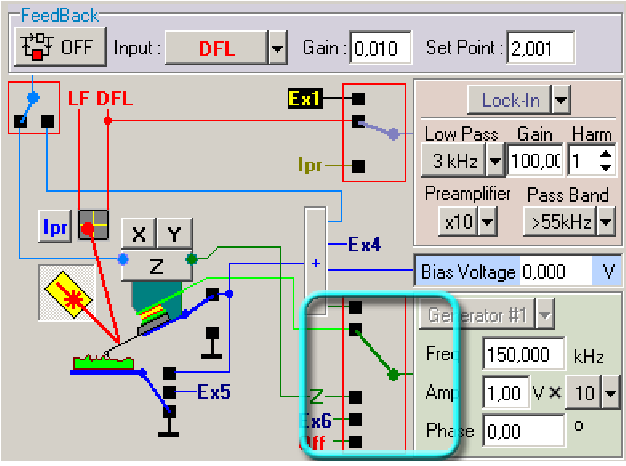


Fig. 1-3

e. Click the button **Run** to build a frequency dependence of the cantilever oscillation amplitude**.**

f. Set the range of values **From**, **To,** which is used to perform search for resonance frequency, so that it would contain the peak of thermal noise corresponding to the first harmonic.

3. In the main menu select subsequently **Tools → Script → Scripts → Res_Liq_Find** (Fig. 1-4).

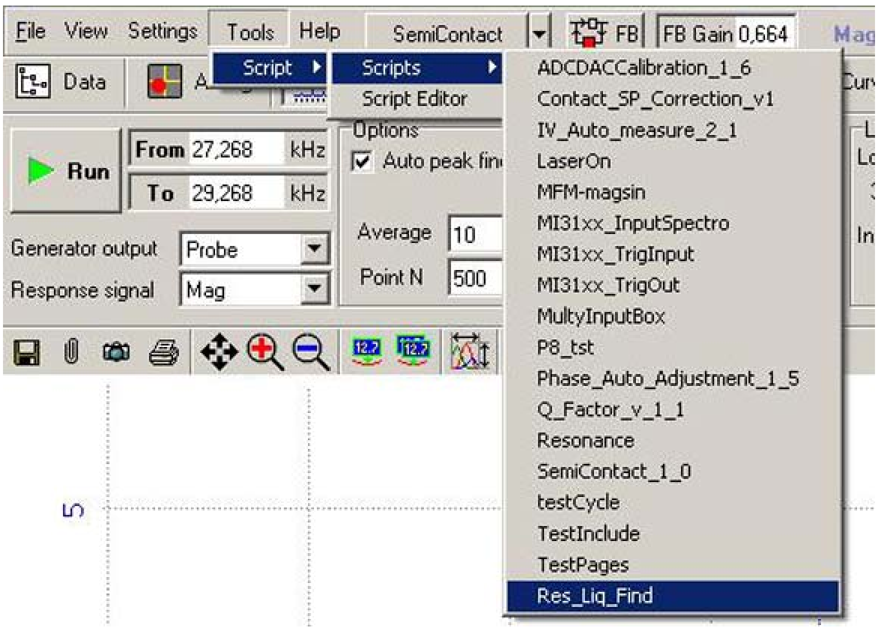


Fig. 1-4

Upon choosing **Res_Liq_Find,** the automatic determination of the cantilever resonance frequency for operation in liquid will be performed:

- cantilever thermal noise will be measured (Fig. 1-5);

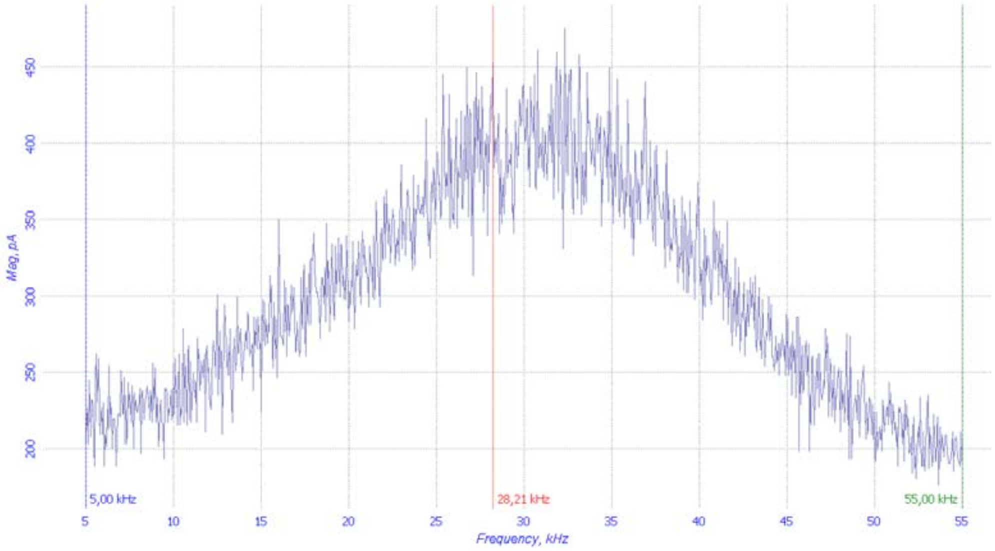


Fig. 1-5

- the frequency response of the cantilever oscillation amplitude (**Mag** signal) will be obtained in the range corresponding to the maximum of thermal noise;
- finally, the frequency response of **Mag** signal will be plotted near the maximum (Fig. 1-6);

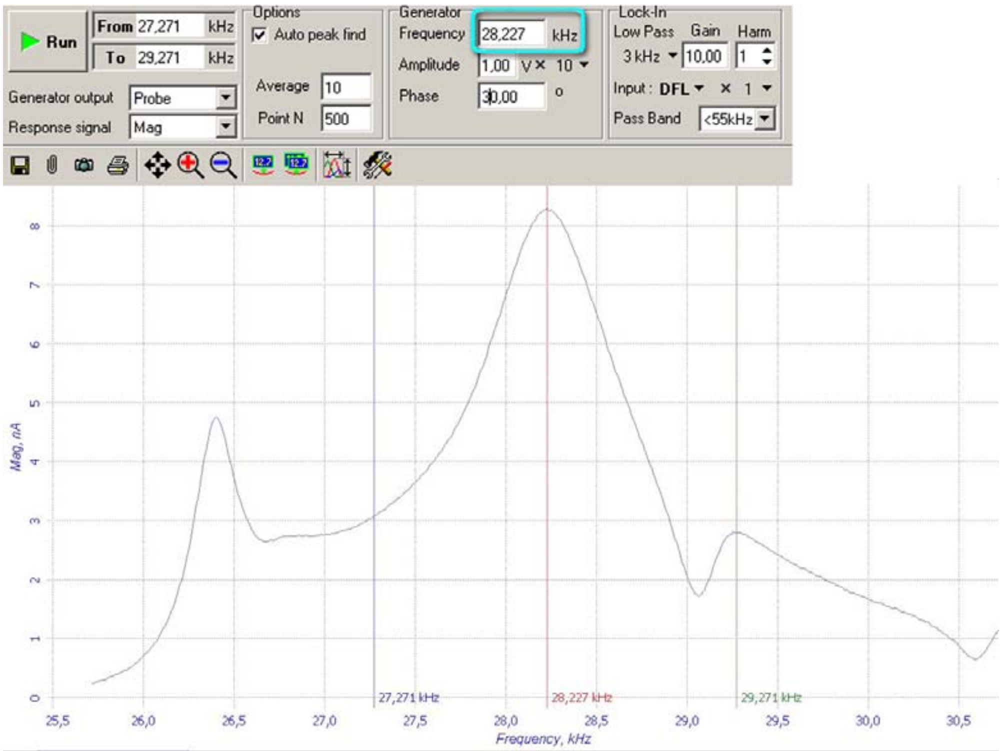


Fig. 1-6

The search for resonance frequency results in the following:

- the piezodriver working frequency will be adjusted to a peak optimal for the operation in the semicontact mode;
- the operating level **Set Point** will be selected;
- the feedback loop will be enabled.

If required, once the automatic tuning is done, manual tuning of the generator and the synchronous detector is possible.

1.2. Approach

1. Switch the instrument in the semicontact AFM measuring mode by selecting **SemiContact** (Fig. 1-7) in the controller configuration list on the main parameters panel.

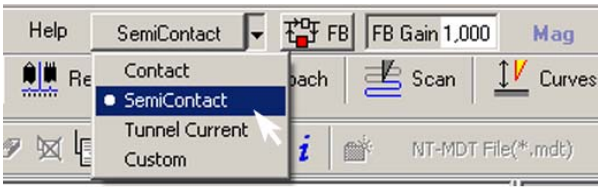


Fig. 1-7

2. Switch to **Approach** tab (

button on the main operation panel) (Fig. 1-8).

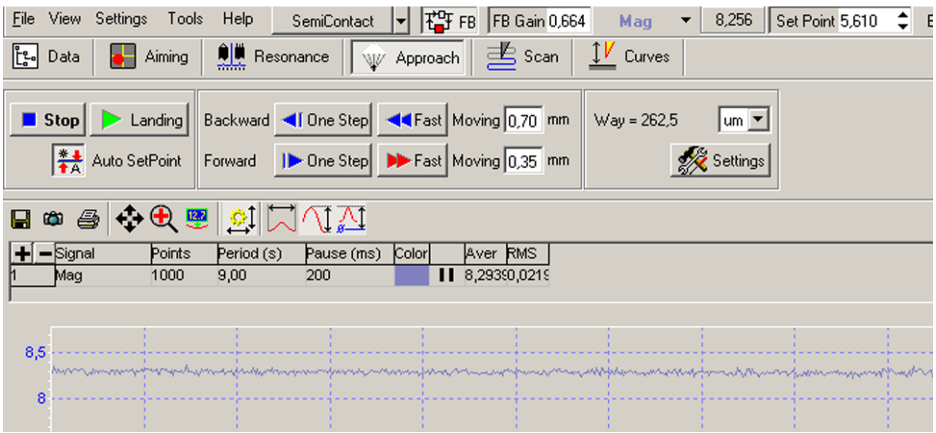


Fig. 1-8

3. Click 

button to start the approach procedure.

   During approach, monitor the changes of **Mag** signal in the oscilloscope's screen as well as the state of the scanner extension indicator and wait for the conclusion of the approaching process.

   After a while, if the tuning of the approaching parameters was correct, the approaching process will be completed and the system will perform the following:

– the **Mag signal** will be reduce down to the value of **Set Point** parameter, the feedback will keep the Z-scanner in the position where **Mag signal** is equal to **Set Point**, moreover that position of the scanner will be approximately one half of the scanner extension range;
– the length of the indicator colored strip will be reduced and would be in some intermediate position;
– the step motor will stop;
– the **Mag(t)** signal trace in the oscilloscope will display a step-down of the **Mag signal** to the value of **Set Point** parameter (Fig. 1-10, Fig. 1-11);
– The message **"…Approach Done."** will appear in the journal (Fig. 1-9).

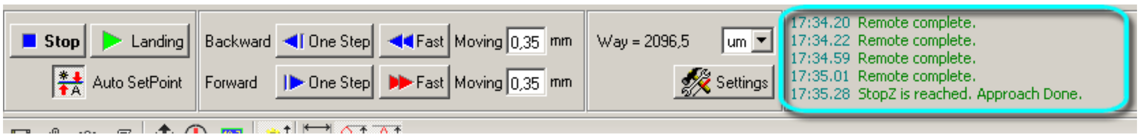


Fig. 1-9

A detailed description of the operations performed by the program during the approach is given in *Performing Measurements*, section "Semicontact AFM".

A criterion for finishing the approach, i.e. for the contact between the probe and the sample surface, is the sudden drop of the **Mag** signal value (a nearly vertical line in Fig. 1-10).

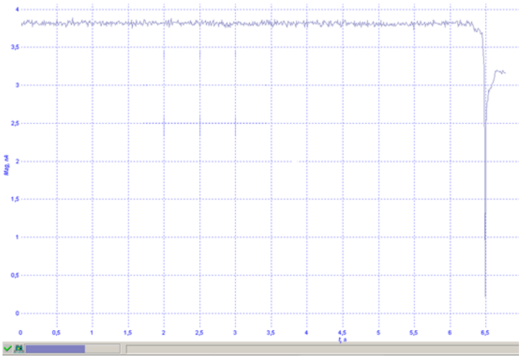


Fig. 1-10. Mag(t) function in case of a successful approach of the probe to the sample surface

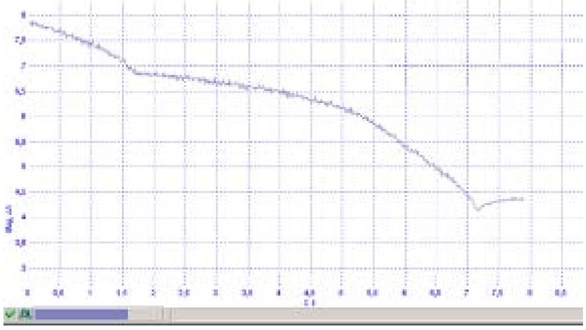


Fig. 1-11. Mag(t) function for the case when the approach procedure was incomplete

The absence of a sharp dip on the plot, as in the example shown in Fig. 1-11, is the evidence that the approach was incomplete. In this event the value of **Set Point** shall be decreased:

a. switch to **Resonance** tab;

b. turn off the feedback ( button is not pressed in);

c. click **Run** button to plot the resonance curve;

d. using the marker fine-tune the piezodriver working frequency;

e. switch to **Approach** tab;

f. Check that the option for the automatic setting of **Set Point** parameter is disabled (button is not pressed in);

g. decrease **Set Point** by a factor of 0.7÷0.8 (at this the scanner shall advance on its full length);

h. start the approach procedure by clicking Landing button.

Once the approach procedure in liquid is finished, it is recommended to verify that the probe has actually approached the sample:

1. Double-click in **Set Point** signal text box.
2. Using the slider that appears change the value of **Set Point** in the range ±0.5÷1 nA, while observing the piezotube extension by the indicator.

   The indicator shall not change its position. Otherwise, the approach shall be performed with a smaller value of **Set Point**.

1.3. Preparation for scanning

Correction of the piezodriver working frequency.

Near the surface the shape of the resonance peak can change hence after finishing the approach the piezodriver working frequency shall be corrected:

1. Switch to **Resonance** (Resonance tab on the main operations panel).
2. Turn off the feedback (FB button on the main parameters panel is not pressed in).
3. Click **Run** button to plot the resonance curve.
4. Using the marker correct the piezodriver working frequency.

<u>Selecting the working point (Set Point)</u>

The working point for scanning shall be chosen 0.8÷0.9 of the cantilever free-oscillations amplitude measured after the approach.

The amplitude curve shall be measured (Mag(z) function) in order to determine the maximum amplitude of **Mag** signal:

1. Switch to **Curves** tab (Curves button on the main operations panel).
2. In the control panel of **Curves** tab (Fig. 1-12) select **Mag** for the signal to be measured (**f1(a)** parameter) and **Height** signal as its argument (**a** parameter).

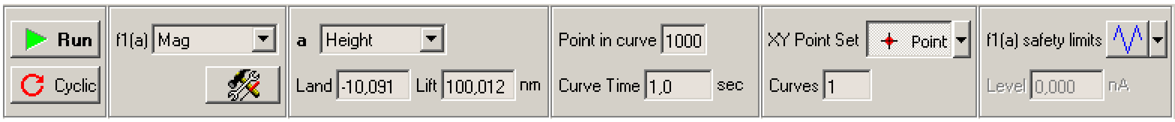


Fig. 1-12

3. Set the height variation range (**Land** and **Lift** parameters) from –10 to +100 nm.
4. Using the cursor select a point in the scanning region where **Mag** (z) function is to be measured.
5. Click **Run** button. Once the measurements are finished an additional panel appears displaying the measured response.
6. Carefully adjust the height variation range to obtain a curve similar to that in Fig. 1-13.

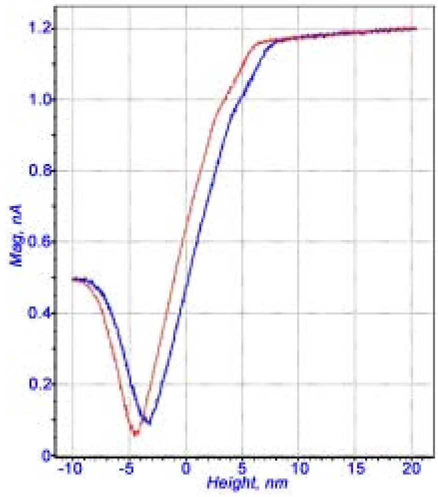


Fig. 1-13

7. Assign **Set Point** a value 0.8÷0.9 of the maximum **Mag** signal amplitude.

   A typical value for the maximum **Mag** signal amplitude is within the range of 5÷10 nm.

Setting the working level of FB Gain

Set the feedback gain as described in item 3.2.3 on page 32.

If the excitation is still present after decreasing **FB Gain** to 0.1÷0.2 try setting the piezodriver frequency on another peak. Also, the excitation may result from poor fixing of the sample or the cell.

Setting up the scanning parameters and scanning are similar to those described in i. 3.2 “Semicontact AFM” on page 22.