USER'S GUIDE

P47-SPM-MDT
SCANNING PROBE MICROSCOPE

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NT-MDT

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The following labels are attached to the P47-SPM-MDT for user safety.

Their position and content should be noted as follows:

Laser Warning Logotype and Aperture Label - This label is located on the SFM head.

Complies with 21 CFR 1040.10

Certification Label - This label is located on the SFM head

Contents

1. Introduction

1.1 What is Scanning Probe Microscopy?

Scanning probe microscope (SPM) is a device that enables investigation of various surfaces with high resolution from micrometers down to atomic scale.

Three basic SPM techniques are classified by the type of probe used: scanning tunneling microscopy (STM), scanning force microscopy (SFM), and scanning near field optical microscopy (SNOM).

In STM bias voltage is applied between a sharp conductive tip and conductive sample, so when a sample is approached to a few angstroms from the tip, tunneling current occurs, that indicates proximity of the tip to the sample with very high accuracy.[1]

STM gives true atomic resolution on some samples even at ambient conditions.

In an ultrahigh vacuum atomic resolution was achieved on most conductive samples.

G.Binnig and H.Rohrer were awarded with the Nobel Prize in Physics in 1985 for the STM invention.

In SFM some force interaction is registered between sharp tip and sample when they are close enough to each other.[2]

SFM routinely gives the so-called lattice resolution on some samples in air, but achieving true atomic resolution is much more difficult. It has only been reported for some special conditions.

In SNOM a specially prepared sharpened optical fiber is set close to the sample and collects light from luminescent objects on the surface or collects an evanescent light wave from the surface of a transparent sample illuminated from inside. Here amplitude of lateral vibrations of the optical fiber is usually used as a proximity gauge (shear-force mode), and collected light intensity gives information about optical properties of the sample. Lateral resolution down to about 20 nm was reported for this technique.

Scanning probe microscopes Solver P47-SPM-MDT of NT-MDT allow the use of various techniques in STM, SFM and SNOM modes. What methods are suitable for the specific sample depends on its properties and features to be resolved (see item 1.2).

1.2 Modern SPM techniques

1.2.1 STM techniques

1.2.1.1 Objects of research

Scanning tunneling microscopy can be applied to study conductive surfaces or thin nonconductive films and small objects deposited on conductive substrates. Some commonly used substrates are highly oriented pyrolithic graphite (HOPG), gold or platinum layers on mica, on quartz, on polished silicon. As to many other conductors on air, most of them are not only covered with adsorbate layers, but are also oxidized. Tunneling probability is very small for such a oxide layer because of its thickness and electronic properties, so tunneling current occurs only when STM tip contracts and destroys that layer and therefore it is impossible to achieve high resolution. For example, a silicon surface can only be investigated with atomic resolution in an ultrahigh vacuum. Surface passivation methods can sometimes be applied to study such samples at ambient conditions.

As to STM applicability for thin nonconductive films and small objects investigation, the problem should be solved in each particular case.

Results can depend not only on object properties but also on those of substrate, and on deposition method. For example, STM has been successfully applied to study LB-films and some biological objects: DNA and protein molecules, viruses, and others.

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[1] Invention of STM was reported by G.Binnig and H.Rohrer in 1981.

[2] Invention of SFM was reported by G.Binnig, C.F.Quate and Ch.Gerber in 1986.

1.2.1.2 Basic principles of STM operation

Schematic of electron tunneling through a potential barrier between the tip and sample is shown in Fig. 1 for the most simple one-dimensional model.

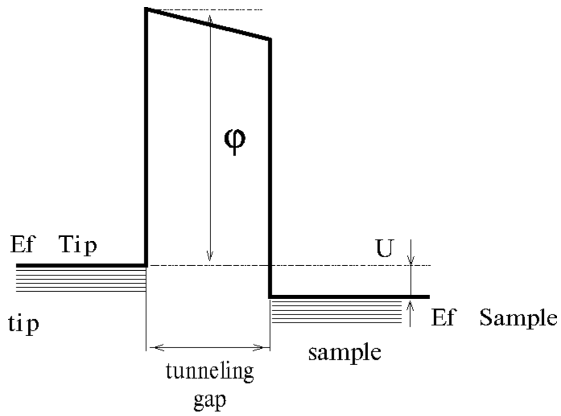

Fig. 1

Tunneling current in this approximation can be expressed as

$$I_t = f(u) * \exp(-(A\sqrt{(j - \frac{u}{2})} * z))$$

u - bias voltage between tip and sample;
φ - average height of tunneling potential barriers at zero bias;
z - width of tunneling potential barriers (tunneling gap).

Exponential dependence of tunneling current on tip-sample separation ensures very high spatial resolution of STM.

However, this model is too simple for real surfaces, especially in air.

Current value is also influenced by tip and surface geometry, by nature of adsorbed layers, and so on. For example, shape of a potential barrier can be dramatically modified due to presence of an adsorbed layer or deposited film (Fig. 2). Tip-sample current can also have an ionic component for many samples especially due to water adsorption from atmosphere.

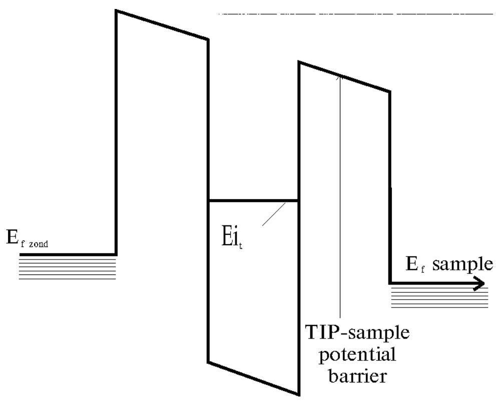

Fig. 2

Sometimes these effects interfere with the true topography, but in other cases they enable to study objects and films deposited onto conductive substrates.

In any case STM-image of an inhomogeneous surface cannot be treated as an image of topography but should be considered as some combination with the map of electronic properties. A nonconductive object can be imaged as a pit instead of prominence (Fig. 3), pikes of the image of a surface atomic lattice do not coincide with surface atoms positions, and so on.

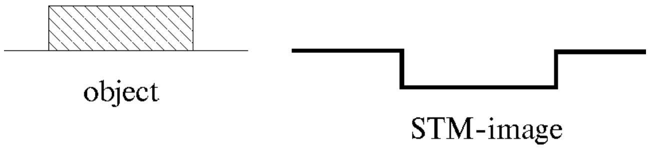

Fig. 3

Information on the surface electronic properties can be extracted from I-V and I-z dependence. I-V curve shape is mostly determined by spectral electron density of states in the tip and sample. The I-z curve shape in the first approximation represents the potential barrier height. The image of dI/dV and dI/dz correspondingly provides a map of spectral density of states and potential barrier height.

1.2.1.3 Modes of operation

STM operation is based on measuring of tunneling current while scanning the sample under the tip. The feedback system can move the sample toward the tip or backwards thus maintaining tunneling current (or some related value) constant. The scanning mode is specified by what values are kept constant and what signal is registered. Various modes of operation can provide different type of information.

Table 1

Method	Description	Example
Height imaging (topography) I=const	Conductive surface topography imaging. Feedback system keeps tunneling current constant while scanning.	See item 3.1.5.1
Current imaging Z=const	Tunneling current variations imaging. Tip height is kept constant relative to the sample base while scanning.	See item 3.1.5.3
FB-error imaging	Imaging of tunneling current variations when feedback system tries to keep it constant whilescanning. Similar to derivation of surface profile; steep surface features look emphasized.	See item 3.1.5.2
Scanning Tunneling Spectroscopy (STS)	I-V (current-voltage) curves measurement in some points or imaging of dI/dV distribution over the surface. I-V curves can also be measured over some area. These data give information on locally spectral density of states for tunneling electrons.	See item 3.1.5.4 3.1.5.6
Local barrier height measurement	I-z (current-distance) curves measurement in some points or imaging of dI/dz distribution over the surface. I-z curves can also be measured over some area. These data give information on local height of a potential barrier (local work function) for tunneling electrons.	See item 3.1.5.5 3.1.5.6
Lithography	Local influences on a surface by voltage pulses. Can be used to study or modify some properties of top layers or to change a surface profile.	See item 3.5

Height imaging mode (topography mode, I=const.)

The most frequently used mode is topography.

In this mode feedback maintains I=const., changing tip height Z relative to sample. For example, when the device detects an increase of tunneling current, it changes voltage applied to the piezotube scanner thus moving the sample away from the tip, and so on. Two-dimensional array of the piezotube voltage values in different scan points are recorded and represent surface topography. To the first approximation real surface heights are linearly related to those voltages by the piezotube transmission module. Scanning parameters for this mode should be chosen to ensure sufficiently quick response of feedback system and high accuracy of tunneling current maintenance. It can be kept constant within a few percent, so tip-sample separation can be constant to about 0.1 A.

Current imaging mode (Z=const.)

Current imaging mode is most useful to get images of atomically flat surfaces. In this mode, feedback is switched off, and tip moves at a constant distance from the sample basement rather than from its surface. So the tip-surface separation can vary during scanning. Two-dimensional array of tunneling current values at different scan points represents both surface topography and its electronic properties. Scanning rates can be much higher here than in topography mode, since it is not limited by the speed of feedback operation. When a sample surface is too rough, tip and sample can be damaged during scanning in current imaging mode.

Feedback error imaging mode (FB-error)

Feedback error imaging mode can be used to register small objects on sloped or curved surface. Feedback system operates similarly in topography mode and current values are registered. Feedback parameters however should be fitted to trace only smooth surface features but skip small abrupt objects. Those objects will therefore be imaged as current deviations from set point value.

Feedback error imaging can also be used to check whether feedback system operates properly in height imaging mode or some parameters should be changed.

In the last two modes, third coordinate of the images is expressed in units of current.

The described three modes are used depending on sample nature and conditions of experiment.

1.2.1.4 Vibration and modulation STM techniques

Based on various principles of probe microscopy there were developed many techniques for getting information on surface properties using vibration of a probe or a sample or modulation of some parameter.

Use of vibration or modulation on rather high frequency allows, on the one hand, to register the differential characteristics while maintaining constant average values. On the other hand it permits reduction of 1/f noise (noise with frequency dependence 1/f) due to transfer of a signal spectrum from the region about 0 Hz to higher frequencies.

Some vibration techniques exhibit advantages that use resonant properties for comparison with static measurement and diminishing interaction lateral forces between probe and surface in non-contact and semicontact modes.

In the STM mode, a sample or tip vibration and corresponding modulation of tip-sample gap permits detection of tunneling current oscillations to receive a signal dI/dz. This provides information on local height of a potential barrier for electrons (local work function). Bias voltage modulation in STM mode allows to register a signal dI/du, determined by local spectral density of state of tunneling electrons.

Mode of local barrier height measurement

For the measurement of local height of a potential barrier for tunneling electrons (also called local work function), a driving signal is applied to Z-electrodes of piezotube. Feedback system keeps low-frequency component of tunneling current constant while scanning. Amplitudes of high-frequency oscillations of tunneling current, caused by piezotube vibration, are registered.

The simplest one-dimensional approximation of electron tunneling through a rectangular potential barrier of height 'φ' gives exponential tunneling current dependence on barrier width z:

$$I \approx \exp\left(-A\sqrt{j}\,z\right)$$

Derivative of this expression with respect to z:

$$I \approx A\sqrt{j}\,\exp\left(-A\sqrt{j}\,z\right)$$

And, hence

$$\frac{dI/dz}{I} \approx \sqrt{j}$$

Therefore, i.e., derivative of tunneling current with respect to gap width, normalized to current itself, gives information on the potential barrier height.

The average value of tunneling current during scanning is kept constant and the amplitude of piezotube vibration does not vary. So the scanned map of the amplitude of current oscillation contains information on distribution of the value $\sqrt{j}$, and therefore about chemical properties of the surface.

The real situation is not so simple, and the amplitude of tunneling current oscillations also depends on surface geometry, on composition of adsorbates that change the shape of a potential barrier, etc.

The real situation is not so simple, and the amplitude of tunneling current oscillations also depends on the surface geometry, on the composition of adsorbates that change the shape of a potential barrier, etc.

Furthermore, repulsive forces exist between tip and surface when scanning on air, since the tip has to force through a layer of adsorbates to afford appreciable tunneling current. Therefore, the scanned profile depends on the local elasticity of a sample. So in the soft regions of the scanned area, piezotube vibrations deform the surface itself rather than a tip-surface gap consisted of an adsorbate layer. The decreased amplitude of tunneling current oscillation thus simulates lower work function value. This effect should be taken into account when explaining the results.

Spectroscopy mode

For the spectroscopy mode, bias voltage V between tip and sample is modulated, and amplitude of tunneling current response is registered.

Low-frequency components of bias voltage remain constant, and feedback maintains the average value of tunneling current constant. Thus, the result of measurement represents derivative dI/dV at a given point of I-V curve.

The name “spectroscopy” arises from the fact that the shape of I-V curve is determined by the energy spectrum of surface and bulk electron states of a sample.

For the spectroscopy mode, and for local barrier height measurement, feedback has to keep tunneling current constant with sufficient accuracy (unless the logarithmic amplifier is used). Otherwise, deviations of current oscillation amplitude due to low-frequency variations of tunneling gap and current, caused by inaccurate tracing of a surface profile, can be much more pronounced than the deviations due to different surface properties.

1.2.2 SFM techniques

Table 2

Mode	Method	Description
Contact	Height imaging (topography, constant force mode)	Conductive and non-conductive surface topography imaging. Feedback system keeps normal force of a surface-probe interaction constant while scanning.
	Force imaging (deflection imaging)	Normal force variations imaging. The sample base is kept at a constant height while scanning. The measurable value is a cantilever deflection.
	Feedback error imaging	Normal force variations imaging when the feedback system tries to keep it constant while scanning. Similar to a derivation of a surface profile, the steep surface features look emphasized.
	Lateral force imaging	Cantilever twisting angle variations imaging when the feedback keeps a normal force constant while scanning. Lateral force measurements are useful to research frictional properties of surfaces.
	Local elasticity imaging	Vibration amplitude variations imaging when the feedback keeps a normal force constant while scanning and sample or probe vibrates. Distinguishing materials is possible if they have different mechanical properties.
	Height imaging (topography)	Semicontact mode differs from contact one by smaller surface damage. Here no lateral forces are between a sample and a probe tip, so soft and jelly-like objects can be investigated. The probe performs vibrations on its resonant frequency and touches a sample surface. Feedback system keeps a cantilever vibration amplitude constant while scanning.

Semicon-tact	Feedback error imaging	Imaging of vibration amplitude variations when feedback system tries to keep an amplitude constant while scanning. Similar to derivation of a surface profile, a steep surface feature looks emphasized. Phase imaging Phase variations imaging when feedback system keeps a cantilever vibration amplitude constant while scanning. Distinguishing materials is possible if they have different mechanical properties.
	Height imaging (topography)	Constant gradient surface of Van der Waals forces is imaged. The probe vibrates within its resonant frequency without touching a sample surface. Feedback system keeps a cantilever vibration amplitude constant while scanning.
Non-contact	Electric force imaging	Cantilevers with a high dielectric permittivity tip are used and a constant gradient surface of electric field is imaged. The SPM operates the same way as in the height imaging.
	Magnetic force imaging	Cantilevers with high a magnetic permeability tip are used and a constant gradient surface of magnetic field is imaged. The SPM operates the same way as in the height imaging.
	Feedback error imaging	Vibration amplitude variations imaging when feedback system tries to keep an amplitude constant while scanning. Similar to derivation of a surface profile, a steep feature looks emphasized.

1.2.2.1 Contact mode

In this mode the cantilever directly touches a sample surface by a tip. In ideal conditions the probe experiences an elastic force of the cantilever, a Van der Waals attraction to the sample and a repulsive force between the sample and the probe tip. The elastic force is defined by a deflection and a rigidity of the cantilever. While scanning the cantilever deflection is used as a surface displacement sensor. The probe displacement is measured with an optical system that consist of a semiconductor laser and a foursection diode (Fig. 4).

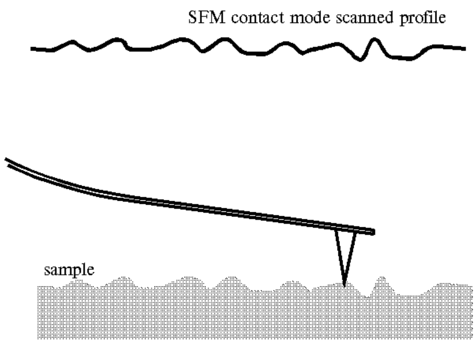

Fig. 4

The contact mode of SFM can be divided depending on an environment of atmospheric or liquid. The atmspheric model is convenient and easier to operate. However, in liquid, smaller interaction forces are possible of a cantilever with a sample without destruction to softer samples. The liquid model is only possible as some objects can only be observed in their natural environment, such as cells and other biological objects, organic solutions and others. The air variant of SFM will be considered here.

The air contact SFM has produced impressive results in research of rigid objects, such as integrated circuit chips, nanostructures, various inorganic material films and many other. With the air contact SFM obtaining sufficiently good results in research of biological objects are possible (cells, viruses), LB-films and, some organic materials.

Forces, acting between the cantilever and the sample

In this item, an interaction force between the cantilever and the sample will be briefly considered. When the cantilever approaches to the sample surface, the Van der Waals force begins to attract it to the sample (Fig. 5). It is sufficiently long-range and is appreciable from a distance of tens of angstroms. Then from distance in a few angstroms a repulsive force becomes perceptible. In moist air a layer of water on a sample surface exists. Capillary forces arise and give an additional pressing cantilever to the sample, so increasing minimum achievable force of interaction while scanning. The force curve can appreciably differ with different samples and cantilevers.

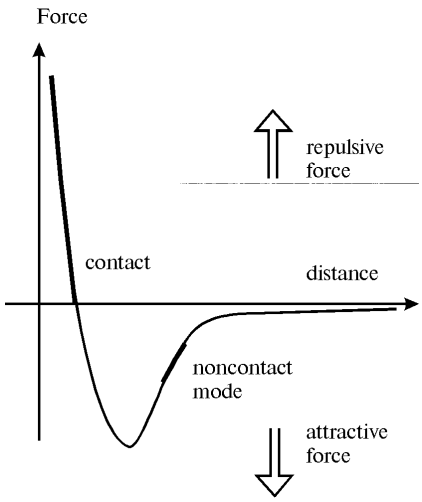

Fig. 5

The electrostatic interaction between the probe and the sample is often possible. It can be both repulsion and attraction. In the repulsion case the situation is feasible when a cantilever approach stops before touch of the probe and the sample. Here increasing an initial force at the repeated approach is possible or to leave the device for some time (hours) for leakage of static electricity.

In the region of a tip-surface contact there are appreciable deformations of both the tip and the sample. Avoiding deformations is possible if forces are about 10^{-11} N [ ], but operating it in liquid is necessary.

1.2.2.2 Height imaging

The surface topography measurement while keeping a constant force of probe-sample interaction is the basis for measurement of local elasticity and local friction force. Let us consider an optical scheme to measure a deflection angle of a cantilever. (Fig. 11).

The radiation of the laser is focused with a lens into an elliptical spot with a maximum dimension approximately 50 microns on a reflecting cantilever surface. The reflected beam falls on the four-section photodiode. The vertical deflection is measured by a differential signal (A + C) - (B + D) (Fig. 6). The lateral forces cause a twist deformation of the cantilever and the reflected beam is displaced in perpendicular direction. The lateral twist is measured with a differential signal (A + B) - (C + D) (Fig. 6).

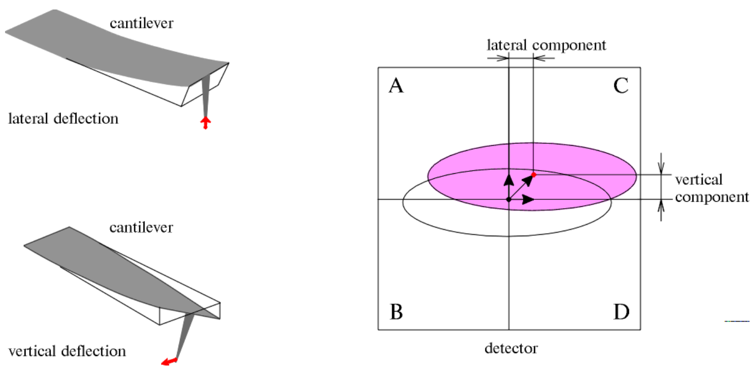

Fig. 6

The functional scheme of SFM during height imaging can be described as follows.

The differential signal from recording system is amplified and is given to an integrator. If a deviation from a set point occurs, it is treated as an error signal and is integrated. It provides a correction to the feedback system of constant piezodrive displacement. The signal from the integrator is given to the high-voltage amplifier and from it to the piezodrive that compensates an arising error. The feedback supports a differential signal near to the given set point. The voltage from the integrator is given to the adjustable amplifier, because ensuring different sensitivity of a measuring part of the device is necessary while operating with an atomic resolution and on rough relief samples. Then the signal is given to an analog-digital converter, and therefrom through an interface unit written down in a computer memory and is interpreted as a relief of the sample. The pressing force of a cantilever to the sample is regulated at an initial adjustment of the photodiode. The additional unit for displacement adjustment provides an opportunity to change of pressing force in an approached state. The remote force adjustment increases convenience of operating with the device.[3]

The operating accuracy of the integrating feedback depends on a feedback loop amplification. The achievement of a maximum scanning speed requires the operation of the fast feedback. To increase the processing speed of the feedback error signal it is advantageous to set the maximum feedback loop amplification. However, a generation threshold can be achieved through a large feedback amplification. The operation near to the generation threshold is characterized by a large overshooting and consequently the monitoring accuracy decreases. On the other hand if the feedback amplification is too small, the feedback cannot track sharp relief changes, that also reduces accuracy of measurements. Therefore, an optimum feedback amplification for each probe-sample system provides maximum feedback operation accuracy and data reliability.

A few reasons influence the feedback loop amplification. Depending on a cantilever used and other parameters else being equal it can be changed several times. The amplification factor is changed in an inverse proportion to a cantilever length, and therefore the cantilever is shorter the amplification factor is higher. Besides the amplification factor can appreciably change depending on a cantilever adjustment. The operator can control the feedback loop amplification factor with an adjustment of the amplifier with the variable amplification factor in the integrator.

The generation arises on frequencies of the first piezodrive resonance due to a large feedback amplification value. For the scanner with a field 11 x 11 mm^2 it is about 10 kHz and with a field 25x25 mm^2 it is about 7.5 kHz.

The generation frequency depends on a sample weight. To eliminate the generation, reduce the amplification factor of the adjustable amplifier.

Meanwhile, the oscillation amplitude will decrease without frequency change up to a disappearance.

With a large friction between the sample and the tip, a different kind of generation can also arise. It is characterized by a frequency decrease almost without amplitude change while the feedback loop amplification factor is reduced, and the frequency can reach tenths of a Hertz, but the generation is always present. To avoid this type of generation, reduce the friction force with a decrease of the tip-sample interaction force or using short cantilevers. The generation amplitude considerably decreases while scanning. Therefore, often its presence does not affect an image quality.

The distorted lines can arise on the image as a scanning result.

Distortion lines look similar to separate lines in a scanning direction with a distinction in height from the general relief. They are caused by a tip clinging to a relief corrugation, slippage on the sample, or by damage of the sample. Avoiding the distortions with a selection of a scanning direction is possible, pressing force decrease or scanning speed decrease.

Difference between the scanning directions is due to cantilever asymmetry and its inclined orientation that cause different interaction of the cantilever and the sample with arising relief changes. While scanning along a positive "Y" direction (+ Y) thinking that the cantilever moves up compared with the surface image on a monitor is possible (in fact the scanning is realized with the sample that moves in the opposite direction).

Thus it goes over obstacles with a smaller slope of the tip and easily surmounts them. Here the distorted lines do not often arise while scanning.

If it goes over obstacles with steep sides tilted at a 75^0 angle, it clings more often to the relief corrugations and the distorted lines arise on the image more often. Overall selecting a scanning direction depending on a sample is necessary. In special cases scanning along + X or -X, may be advantageous or necessary for example, while imaging lateral force.

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[3] As the differetial signal deviates from zero, an intensity noise of the laser appears. Therefore it is necessary cautiously to apply electronic adjustment of force on samples with small corrugations, for example, when the atomic resolution is obtained.

Characteristic traces connected with relief features in a fast scanning direction can arise on the image. They are caused with the final feedback speed. Their dimensions depend on scanning speed, feedback loop amplification and relief features. If the feedback error signal is registered while scanning, places with the large feedback error will be seen well. The image obtained contains practically whole information lost while height imaging. Using results of scanning in the topography mode and in the feedback error registration mode, restoring the topography of a surface is possible.

1.2.2.3 Force imaging.

The SFM operation with feedback, results in an increase of a noise level, a partial loss of the topography information or a scanning speed restriction.

Sometimes operating it at scanning mode when the feedback is broken is useful, the piezodrive "Z" displacement is fixed and a photodiode differential signal is directly registered. It is a force imaging mode, sometimes called constant Z. Here the pressing force of a cantilever to a surface changes while scanning.

However, if a sample is rigid enough, the image found reflects surface topography well. Calculating a height image from registered current image using dependence of a cantilever deflection on a Z-displacement is possible.

However remembering that the dependence of the differential signal on is necessary "Z" displacement can be nonlinear at large deviations from the zero point. An approximate linear range depends on a cantilever choice. The shorter the cantilever, the less the range.

Dynamics of surface tracing in this mode are limited by frequency properties of a cantilever rather than feedback ones. The resonant frequencies of cantilevers are much higher than characteristic feedback frequency that is a few kilohertz. It enables to scan with higher speeds.

1.2.2.4 Feedback error imaging.

The feedback error arising in a height imaging contains an additional information about topography. It can be used to restore surface topography more precisely. However this mode can be considered as an intermediate one between height imaging and force imaging if the feedback speed is adjusted to trace smooth changes of relief and to skip steep changes. Then while crossing by probe, a small corrugation, the scanning will occur at an almost constant displacement of a piezodrive. As a result the steep changes will appear highly contrasted with respect to the smooth one. Searching fine features over a large field on a background of large smooth relief peculiarities can be useful.

1.2.2.5 Lateral force imaging

While scanning along +X or -X directions there are additional twist deformation of the cantilever. It is caused by a moment of forces applied to the tip along scanning direction. The twist angle at small deviations is proportional to the lateral force. The measuring system of the microscope allows registration of the twist of the cantilever. Here the laser beam reflected from the cantilever has an additional displacement in a lateral direction (Fig. 6) and a signal (A + B) - (C + D) is registered. To measure lateral force SFM operates in height imaging mode while keeping a normal force constant.

The cantilever twist changes when scanning over regions with different friction (Fig. 7). This allows interpretation as a measurement of local friction. If a relief exists, such interpretation is impossible (Fig. 8). Nevertheless, this kind of measurement allows acquisition of the image, on which fine relief peculiarities are seen well, and to simplify their search.

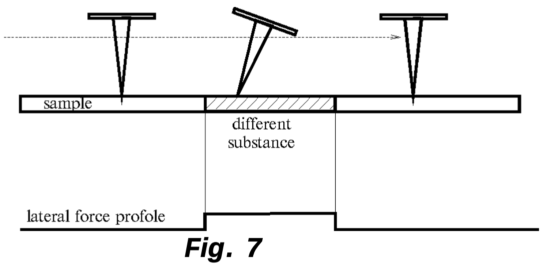

Fig. 7

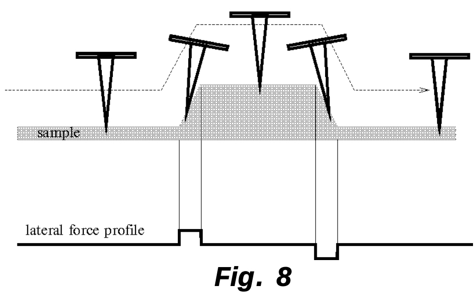

Fig. 8

In the lateral force mode getting atomic resolution on mica is easy and on other layered materials. Notice that during measurement of topography with the atomic resolution, an atomic corrugation ranges several angstroms, whereas the real corrugation is a fraction of an angstrom. In particular, the large size of corrugation is explained by an influence of the twist deformation due to an imperfect behavior of recording system. The cantilever twist is treated as its longitudinal bend. For example, it even arises under a longitudinal bend of the cantilever when the photodiode is rotated to a very small angle with respect to a direction of a beam movement.

As the differential signal deviates from zero, a noise of the laser appears.

Therefore when atomic resolution is obtained. cautiously applying electronic adjustment of force on samples with small corrugations may be required.

1.2.2.6 Vibration and modulation AFM techniques. General information

Based on various principles of probe microscopy there were developed many techniques for getting information on surface properties using vibration of a probe or a sample or modulation of some parameter.

Use of vibration or modulation on rather high frequency allows, on the one hand, to register the differential characteristics while maintaining constant average values. On the other hand it permits reduction of 1/f noise (noise with frequency dependence 1/f) due to transfer of a signal spectrum from the region about 0 Hz to higher frequencies.

Some vibration techniques exhibit advantages that use resonant properties for comparison with static measurement and diminishing interaction lateral forces between probe and surface in noncontact and semicontact modes.

In the AFM mode, sample vibration registers amplitude of cantilever response that provides information on local elasticity of a sample.

Detecting an amplitude and/or phase of cantilever vibrations allows scanning of the surface topography in noncontact and semicontact mode. Even for such samples that cannot be investigated in contact mode, they can be easily deformed or destroyed by cantilever tip. These modes permit use of cantilevers with thin and very sharp tips that can be easily broken in contact mode.

Among the vibration AFM techniques there exist noncontact, semicontact modes, local elasticity and local viscosity modes.

Noncontact mode.

Noncontact mode provides measurement of Van der Waals, electrostatic, magnetic forces near the surface. Interaction force can be very small (about 10^{-12} N) that allows investigation of very sensitive objects or those loosely coupled to a substrate surface, without any destruction or displacement. A cantilever holder contains piezoceramic plate (Fig. 9).

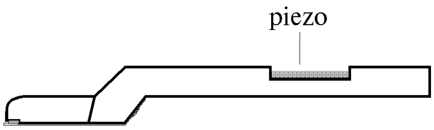

Fig. 9

Piezo and holder vibrations induce cantilever oscillations at a necessary frequency. All variations of this technique imply the choice of frequency within one of a cantilever resonant peaks. Driving the signal is formed with a digital synthesizer, containing a stable quartz generator, that allows maintenance of the signal frequency with relative accuracy about 10^{-5}.

High-frequency component of a signal from the four-sectional photodiode, caused by cantilever vibrations, is amplified and applied to the synchronous detector, that forms the following signals:

- signal proportional to the amplitude of the main frequency or of one of harmonics;
- signal of a phase shift of cantilever vibrations relative to the driving signal;
- signal of amplitude multiplied by sine or cosine of phase shift.

Any of the listed signals can be brought into a feedback loop.

Cantilever, vibrating with a small amplitude, experiences an influence of a non-uniform force field near the sample surface. A force gradient causes a frequency shift of the resonance peak. Therefore both amplitude and phase change in a non-uniform field, if the driving signal does not change. If the feedback system moves a sample toward the probe and backwards while scanning to keep amplitude or phase of cantilever vibrations constant (topography mode), then the feedback output signal represents the surface of constant force gradient.

Registering an amplitude or phase change during scanning without changing the distance between a probe and sample base is also possible (constant height mode). Another mode is also possible that implies preliminary scanning of topography in contact or semicontact mode.

Then the some area is scanned again. Some chosen tip-sample distance is kept constant during the second scanning with respect to surface topography already known from the first scanning.

Amplitude or phase is being registered during the second scanning. This mode allows separation of information on magnetic and electrostatic properties of a surface from topographic data. Since the distance between probe and surface does not vary, van der Waals attraction of the cantilever and surface remains practically constant during repeated scanning. It means that the changes of amplitude and phase are caused by other long-range forces - electrostatic or magnetic.

The minimum possible distance between cantilever tip and sample surface in noncontact mode is determined by tip and surface properties and by force constant of the cantilever beam. If the attraction force exceeds a cantilever force constant as the surface is approached, then the cantilever will "stick" to the surface. Therefore the minimum working distance should exceed this critical distance. Usually the most significant reason of attraction is capillary effect which besides has large inherent hysteresis.

And even in the absence of capillary attraction (for example in the case of hydrophobic surfaces), the effect of "sticking" can still be observed due to electrostatic, magnetic and even van der Waals attractive forces. Therefore the larger force constant of the cantilever, the smaller working distance can be used, and the better resolution can be achieved (at distances commensurable or exceeding tip radius of curvature). But the interaction force in this case is also increased.

The situation is also possible, when the gradient of attractive force does not exceed cantilever force constant down to the contact of tip and surface, i.e. down to approach of extreme atoms of tip and sample to the region of repulsive force. What this means is that whatever a small working distance is used, the cantilever will not "stick". Such a situation is transitional between noncontact and semicontact mode.

Semicontact mode

The characteristics feature of semicontact mode is that cantilever tip does not touch a surface during the major part of the cycle and does not almost interact with it. Only when the tip hits the surface (i.e., experiences remarkable gradient of repulsive force), the cantilever loses excessive energy that the cantilever got during the previous part of a cycle. The phase shifts of the cantilever response concerning the driving signal at the main frequency and amplitude and phase of higher harmonics can vary depending on character of interaction. Basic effect is that the vibration amplitude is limited at a level approximately equal to the distance between sample surface and tip apex of the undisturbed cantilever. In an example, as a rough guide, cantilever vibration amplitude can only increase up to the moment when tip starts touching the surface at extreme positions during a cycle and not farther.

In semicontact mode, in contrast to noncontact one, the cantilever force constant can be less than the maximum gradient of attractive force near the surface. Then amplitude of vibration should be high enough to prevent "sticking" of a cantilever tip. In an example, maximum attraction exerted to the tip when it touches the surface should not exceed the retracting force from the cantilever beam. In noncontact mode it is impossible since the amplitude for that mode should be small enough in comparison with the interval where a gradient force appreciably varies.

Local elasticity mode

For measurement of local elasticity, a driving signal is applied to the Z-electrodes of the piezotube. The results being the cantilever tip touching the surface, vibration of the sample is transferred to the cantilever beam, and the amplitude of beam vibrations is registered while scanning. Coefficient of vibration transfer from sample to cantilever is proportional to the ratio of tip-surface elasticity in the given point and beam force constant.

In one limiting case of an absolutely flexible cantilever with a rigid tip touching a rigid sample, the piezotube vibrations are completely transferred to the beam. In a very stiff cantilever on flexible or easily deformable sample and/or with a flexible tip, piezotube vibrations cause only tip and surface deformations, whereas the beam does not move.

It is necessary that the elasticity of tip-sample system depends not only on Young's modulus of both substances, but also on their geometry, in particular on curvature radii. The greatest stiffness of the contact region can be reached with surfaces that have equal absolute values of a curvature and opposite sign. Therefore the local elasticity mode will contrast surface steps and show small hillocks as areas with lower stiffness, even if they consist of the same material as the entire sample.

1.2.3 SPM components' interaction scheme

Basic SPM components are schematically represented in Fig. 10:
- Sample under investigation,
- STM tip,
- SFM cantilever,
- Cantilever deflection monitoring system,
- Piezotube scanner
- Rough approach system that brings sample to within normal displacement range of piezoscanner,
- Processor,
- Power supply unit,
- Computer (workstation).

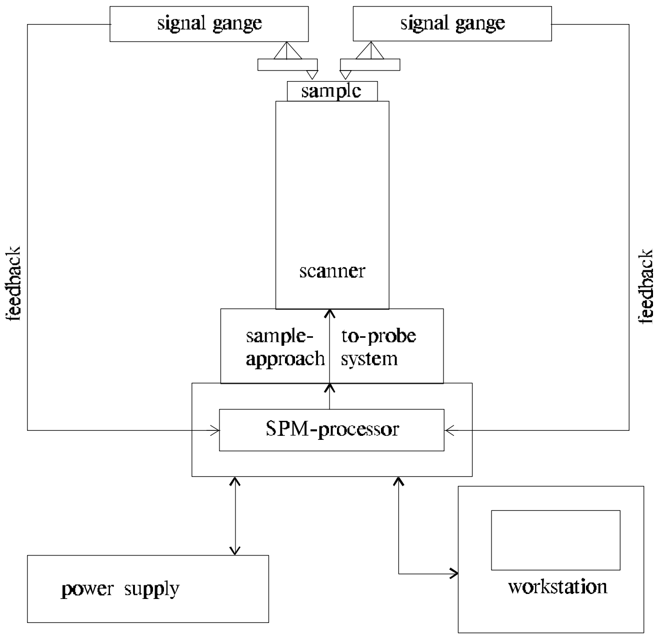

Fig. 10

1.2.4 Cantilever deflection monitoring system

The SFM head uses a beam deflection scheme to monitor the cantilever displacement (Fig. 11). This scheme is quite simple and permits registration of both normal deflection of the cantilever with sub-angstrom resolution and its twisting angle, so normal and lateral force can be measured simultaneously.

A semiconductor laser is used as a light source with wavelength 670nm and optical power 0.9mW. A laser beam is focused onto the back surface of cantilever close to tip position, and reflected beam falls onto the quadrant photodiode. Cantilever deflection causes displacement of the reflected beam over sections of the photodiode. An amplified differential signal from the quadrant photodiode permits measurement of angular deviation with the accuracy of less than 0.1″, that corresponds to normal cantilever deflection of the order of 0.1nm.

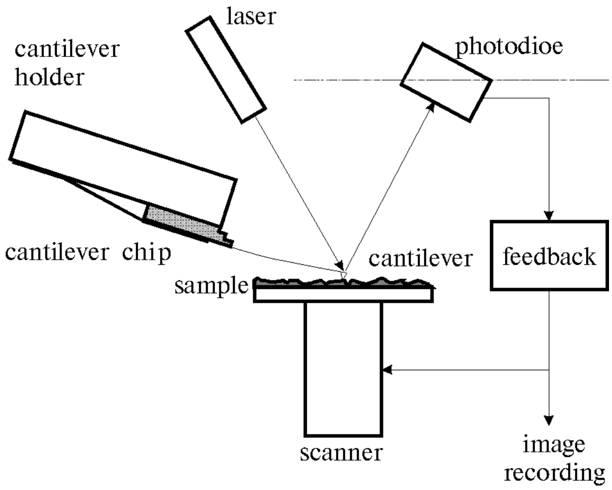
laser
cantilever
holder
photodioe
cantilever chip
cantilever
feedback
sample
scanner
image
recording

Fig. 11

2. SOLVER SPM installation

SOLVER SPM contains a housing with electronic circuitry and processor within it (Fig. 12.A), vibration-protection system, a suspended piezoscanner (Fig. 12.B), a set of measuring heads, computer interface card. The piezoscanner is provided with a mounting for samples being investigated and an outlet for plugging in the thermal table. The basement of the microscope contains a power unit and an electronic printed-circuit board i.e. processor controlling the operation of the microscope in different modes: STM and SFM. Various heads of the SEM: STM, AFM, NearField etc. (Fig. 12, C) are mounted on the platform. Depending upon this the microscope can operate in different modes.

The processor amplifies measured the signal value and converts it to a digital form and then transmits it to the computer through the interface card (mounted within the computer) for further recording.

Due to modular structure of the Solver SPM, a user can easily extend the basic configuration and change it according to the specific requirements. The electronic block is provided with external terminals for connecting user sensors and thanks to the flexible switching the electronic circuit makes it possible to develop and use new techniques based on both modulating and measuring the signals of the microscope itself and using external signals.

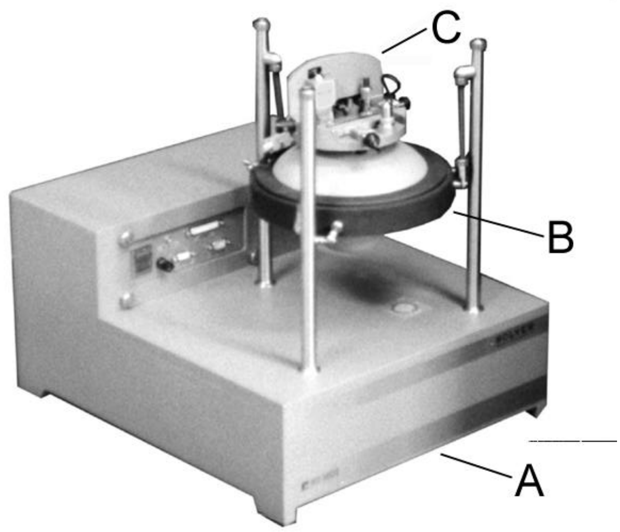

Fig. 12

2.1 SPM installation

2.1.1 Complete Solver SPM Unit Standard configuration

1) P47-SPM - base, power unit and an electronic board built-in the cabinet (Figure 12.A), suspension system (Fig. 12.B) with piezoscanner and approach system, protective cover for acoustic isolation and electromagnetic shielding (Fig. 14).
2) Computer interface card (located within the computer if a computer is provided as part of the system).
3) Program diskette (control program is installed onto the hard disk if a computer is included within the deliverable system).
4) AFM head with the standard adjustable part (Fig. 13).
5) STM head (Fig. 15).

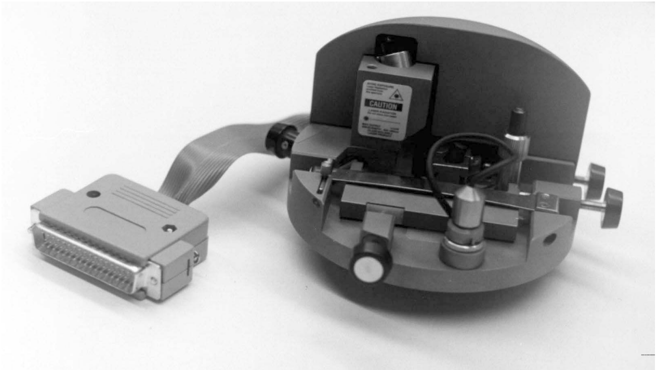

Fig. 13

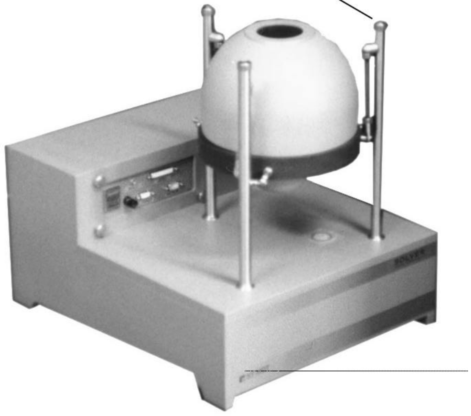

Fig. 14

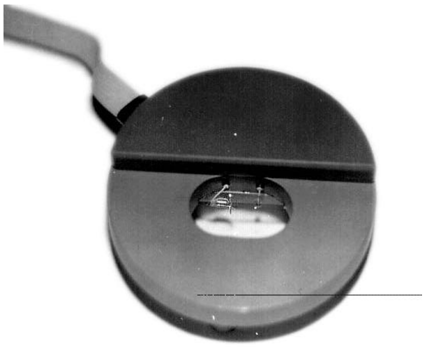

Fig. 15

6) Cables:

   a) interface - SPM-IBM (Fig. 16).
   b) oscilloscope cable (Fig. 17).
   c) power cord 220V.

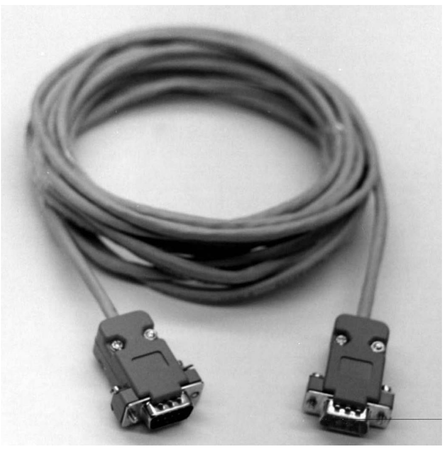

Fig. 16

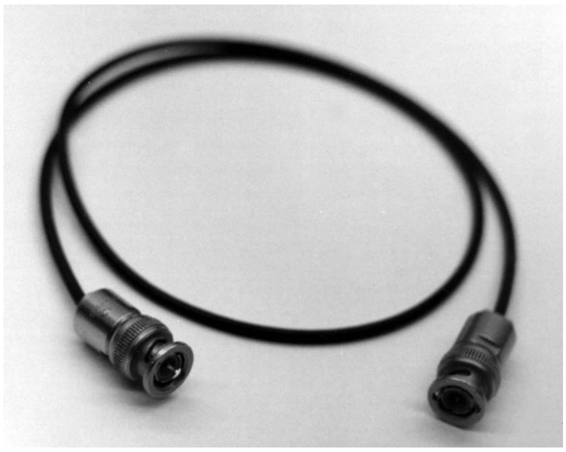

Fig. 17

7) Cantilever holders - two PCS (Fig. 20. 6).

8) Set of substrates:

   a) with bias voltage contact strip for STM-study of thick samples (Fig. 18),
   b) with bias voltage contact strip for STM-study of thin samples (Fig. 19),
   c) 10 ordinary AFM substrates - four PCS (Fig. 21. 2, 3, 4).

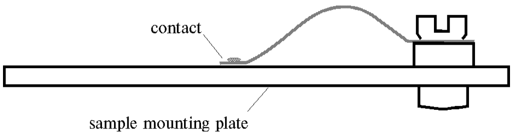

Fig. 18

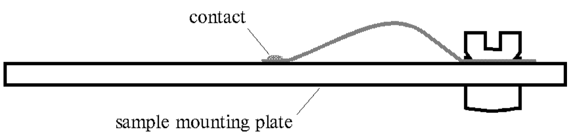

Fig. 19

9) HOPG (graphite) test sample.
10) Pt-Ir or Pt-Rh wire for STM tips (Fig. 21. 1).
11) Wide tweezers and narrow tweezers (Fig. 20. 2, 3).
12) Special scissors for cutting STM tips (Fig. 20. 4).
13) Screwdriver (Fig. 20. 1).
14) Scalpel (Fig. 20. 5).
15) Double-stick tape for sticking samples.

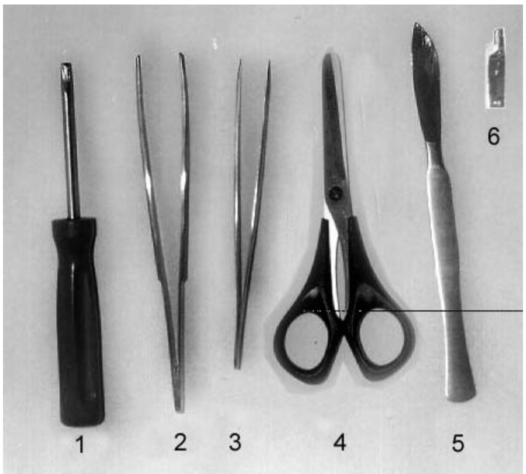

Fig. 20

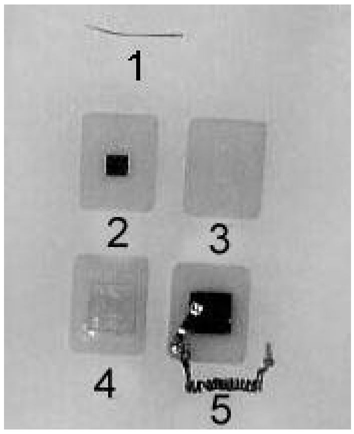

Fig. 21

ATTENTION:
Scissors for cutting tips should not be used for other purposes.

2.2 Placement of SPM

Before arranging the SPM components, it is desirable to provide the most suitable conditions for successful an SPM operation.

In an operational room there should be no other devices capable of producing mechanical vibrations and acoustic noise. The room should also be protected from external acoustic noise. Quick temperature and humidity changes also adversely affect and influence the SPM operation. It is necessary to have at least two tables, one of which which should be a solid, steady, and stable unit and used only for the SPM unit. The other table(s) can be used for placement of the power supply, computer, auxiliary oscilloscope, and so on. The computer monitor should be placed some distance from the main unit, thereby minimizing any electromagnetic noise. An example of equipment layout is shown in Fig. 22.

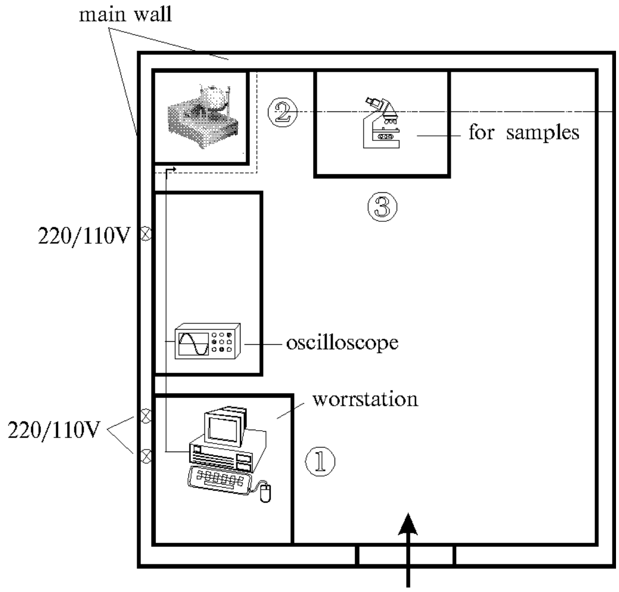

Fig. 22

The desired placement of the SPM unit is in a corner. An external oscilloscope with sensitivity less than 5 mV/Div is useful for seeing the tunneling current (or some other signal) behavior while scanning. The room should be provided with 110 V or 220V wall outlets. If other devices that can produce electric noise are connected to the same supply line, it is desirable to connect the SPM through surge protectors or additional electronic filters.

The SPM can also be supplied with a cable up to 20 m long, that allows placement of the main unit and the computer in separate rooms. An advantage, for example, would be if atomic resolution must be demonstrated to a large audience, or if operator must be separated from the SPM main unit for some reason.

2.3 Connecting SPM

2.3.1 Destination of connectors of the main SPM board

Housing of the main SPM board has a number of connectors for connections to other units:

✓ the following is located on the front panel (Fig. 23): three terminals to connect a scanner («Scanner»), a measuring head («Head») and external devices («Extension») as well as the power switch («Power») and the grounding terminal.

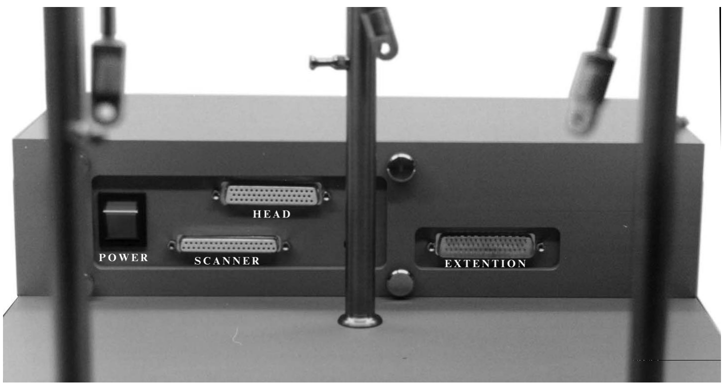

Fig. 23

- ✓ the following is located on the rear panel (Fig. 24): terminals to connect an external oscilloscope («OSC») an interface cable «Computer» and a power connection for 110-220 V («Power»).

Fig. 24

Fig. 25

2.3.2 Connecting Microscope

1) Install the device on a heavy and stable table.
2) Connect the cable 1 (Fig. 25), going out of the scanner cabinet to the SCANNER terminal (Fig. 23).
3) Connect the cable (Fig. 13), going out of the measuring head to the HEAD terminal (Fig. 23).
4) If you are going to use an external oscilloscope, connect it with a cable to the OSC terminal (Fig. 24) located on the rear panel of the unit (Fig. 17).
5) Connect the power connection terminal on the cabinet (Fig. 24) to a wall outlet by a power cord.

2.3.2.1 If computer is not included to delivery set

Installation of an interface board in the computer

If a computer is not included with the delivery set, you must mount an interface board to your computer and install the SPM software program to its hard disk yourself. Mounting the SPM interface board to your computer. Disconnect your computer from the electrical power line and remove its cover. Choose an appropriate empty slot and remove the corresponding cover from the frame. Accurately insert the interface board up to the stop. Verify that the board is inserted squarely. Fasten it into position with the cover screws. Gently try to swing the board to ensure that it is mounted reliably and nothing around can touch it. Replace the computer cover.

Installation of the SPM control program

Insert the supplied diskette into the drive. Run the installation file (there is only one installation file on the diskette), follow the instructions displayed on the screen in the course of the installation.

A directory will be created on your hard drive containing the following program files:

- s7_spm.exe - the executed program;
- s7_spm.opt - the file of a configuration;
- s7_spm.hlp - the file of the context-sensitive help;

The file s7_spm.256 containing a palettes set is also supplied with the program.

You should also have enough free space on the hard disk to be able to save data files that can contain from 100 Kbyte to 8Mbyte.

2.3.2.2 Connecting the main SPM board to the computer

6) Connect connector "COMPUTER" on the front side of the SPM base (Fig. 24) by the interface cable to the SPM interface board (beforehand inserted in the computer - see item 2.3.2.1).

Attention!
Computer and SPM have to be switched off before connecting or disconnecting any cable. Otherwise electronic components will be damaged.

2.3.2.3 Switching on the device

Turn on the computer and SPM by the switch on (POWER) its front side (Fig. 23). Start the program s7_spm.exe (this program had to installed - see item 2.3.2.1).

3. Basic operations

3.1 STM-mode

The scanning tunnel microscopy mode is designed for investigation of conductive surfaces at the accuracy up to atomic resolution. The tunnel current registered in the course of the measurement is sufficiently small - up to 0.1 nA (with a special STM head - up to 0.02 nA), so it is possible to investigate also low conductivity surfaces, in particular biological objects.

Using special techniques it is also possible to investigate the on-surface distribution of zones having different electron characteristics i.e. exit behavior, charge density etc.

Among the STM disadvantages one can mention the complexity of the results interpretation for some surfaces since the surface image received in the STM investigation mode is determined not only by the surface relief but also by various electrostatic parameters of surface areas.

3.1.1 Installing the tip

The suggested technique for the STM tips preparation is presumed to meet the following requirements to the resulting STM probe tips.

Requirements of a tip:

- the point's sharpness stability over time;
- as small as possible point's curvature radius;
- repeatability of manufacturing method.

When standard objects like, for instance, highly orientated pyrolytic graphite (HOPG) are investigated the substrate's behavior is known and the results make it possible to judge the tip's quality.

Shearing:

The simplest technique for STM tips preparation consists of shearing with scissors.

Wire made of PtIr or PtRh (Fig. 21. 1) is appropriate for the tips manufacture by means of shearing. The wire diameter must be 0.25 to 0.5 mm.

It is convenient to use two pairs of tweezers when manufacturing and installing the tips: one pair of tweezers should be with sharp points and the other with serrated teeth on the point's inner surface.

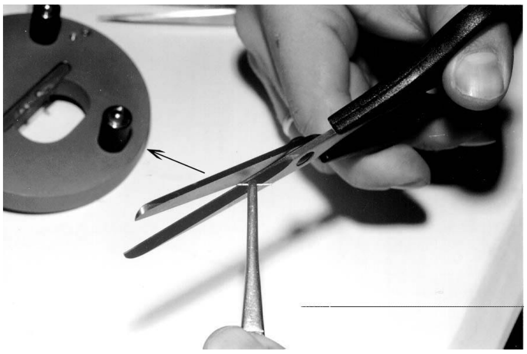

Fig. 26

The wire is clamped in the clip so that it comes out on one side for 2-3 mm (Fig. 26). On the other side the wire must be cut with scissors having sharp cutting edges. The cutting should be done with the nearest part of the edge at the angle of 10 - 15 degrees to the wire's longitudinal axis. The cut must be done to avoid contact between the shear and the freshly split off point.

To do this when shearing an effort must be applied in the direction from the tweezers. The total tip's length must not be over 12-15 mm. Check the point's cut surface with an optical microscope with a 200 X magnification. If necessary, repeat the cut.[4]

After the cutting take the tip out of the clamp and heat it in the flame (e.q.: of an alcohol lamp) for 1-2 seconds to remove organic matters from the tip's point. Check the tip with an optical microscope: the cut must be bright and have no traces of soot or dust. After heating, insert the blunt point of the tip into the tip holder (Fig. 28).

Turn the STM head over so its legs are facing upward. Use your left hand to force the spring clamp apart and your right hand to insert the blunt point of the tip so that its sharp point comes out for not more than 3-4 mm (Fig. 27). If during the installation the sharp point of the tip comes by accident in contact with any object the cutting must be done again. The tip must be rigid and tightly fixed in the tip holder. To prevent damage of the input amplifier during the installation, the tweezers must be grounded or the power supply unit must be turned off.

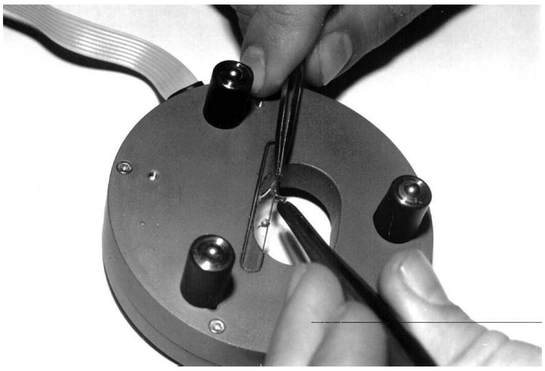

Fig. 27

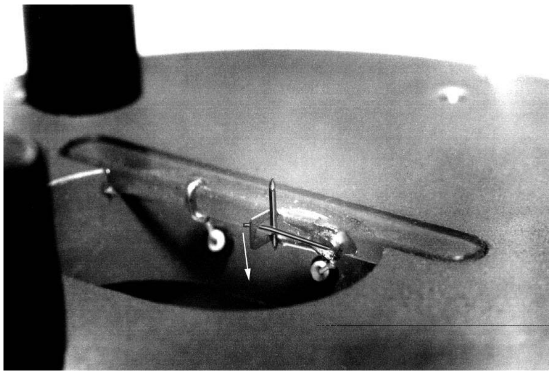

Fig. 28

The tip's quality and the clamping pressure within the tip holder are among the main factors ensuring good results in the STM operation.

Electrochemical etching:

CAUTION:
Following procedure has to be performed in accordance with organizational safety standards.

Sharp tips can be made using the electrochemical etching technique. Etching ensures higher repeatability of the general tip shape than cutting. We have experienced and can recommend the following technique for tips manufacture from platinum group metals. The etching is done in the installation schematically shown in the figure below in $CaCl_2$ water solution, high- purity graphite acts as a counter electrode. The etching is done in two stages. At the first stage the voltage between the electrodes is about 25 V, when etching Pt group metals alternating voltage is used, possibly, with the supply line frequency of 50-60 Hz. The tip that has been pre-fired in alcohol flame is placed in the solution at the depth of about 0.5 mm, a violent etching reaction accompanied by partial solution splashing starts immediately. The etching process stops automatically as soon as the tip's point comes above the solution surface. At the second stage at a voltage of 6 V between the electrodes the tip's point is slightly placed in the solution for 3-5 seconds. After etching the tip is washed in a NH_4OH water solution and then fired in an alcohol flame.

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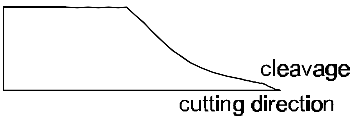
[4] In fact this technique of tip preparation implies tearing the wire at the last moment rather than cutting it.



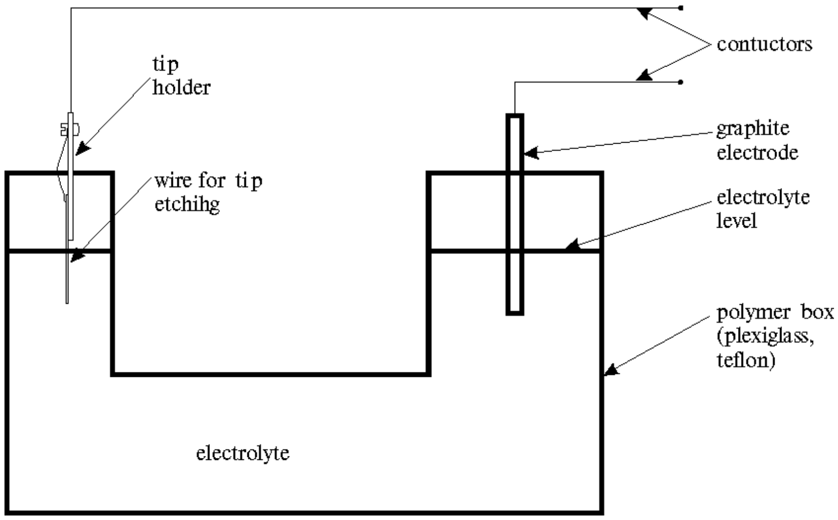


Fig. 29 Installation for tip etching.

The etching speed and quality depends on the wire diameter. If the wire diameter is 0.1 mm etching is done for about 20 sec., a wire having 0.15 mm in diameter must be etched during two - three minutes. It is not possible to prepare a STM tip from a thicker wire using this method as in the process of etching too much heat is generated, the solution is violently splashed over and the meniscus does not stand still. The tip must be welded or soldered to a wire of about 0.25-0.5 mm in diameter to fit the tip holder.

3.1.2 Preparing and installing the sample

Substrates having electrical contacts for bias voltage application are appropriate for STM investigations (Fig. 21. 5).

Take a clean substrate. Cut off a piece of double-sided adhesive tape larger than the sample. Stick a piece of adhesive tape on the substrate, smooth it out with the pointed tweezers to prevent any air-bubbles from forming between the substrate and the adhesive tape. Put the sample - a graphite plate - over the adhesive tape and carefully press it down with the tweezers in several places (but do not touch the areas to be studied). After a sample has been fixed into position with adhesive tape, the vertical drift can be appreciable for about an hour.

This should be taken into consideration with the type of your samples.

Prepare the sample at least one hour before measurements.

Adhesive tape is used to obtain a clean and smooth graphite surface. Stick a piece of adhesive tape that is larger than the graphite over the graphite surface and smooth it out to prevent air-bubbles from forming under the tape. Then remove the tape off together with the upper graphite layer stuck to the tape. The graphite molecular layers have a slight inclination with respect to the outer surface. Therefore the smoothness of the surface depends on what edge of the tape was lifted first.

Check what edge of the stuck tape should be lifted first to make a smoother graphite surface and remember it for future cleavage of that piece of HOPG.

Insert the substrate with the sample into the holder at the scanner unit.

Make sure the tunneling voltage contact attachment does not come in touch with the metal parts of sample holder (Fig. 30).

Turn the tunneling voltage contact and fix it at the sample's edge leaving the center free. Insert the contact's plug into the appropriate socket on the scanner unit. (Fig. 30).

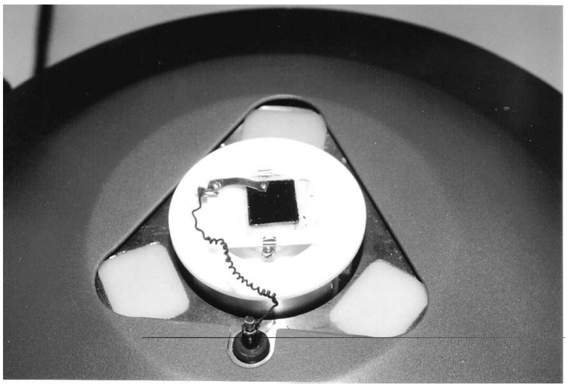


Fig. 30

3.1.3 Installing the STM head

With the device turned off, connect the STM head cable to the appropriate socket.

Take the metal hood off the scanning unit, if necessary disconnect the grounding wire from it. Move the sample down manually turning the rotor of the scanner's step motor clockwise if viewed from bottom (Fig. 31).

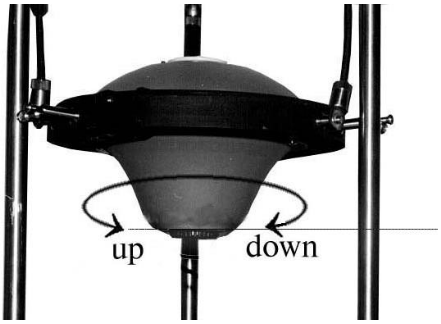


Fig. 31

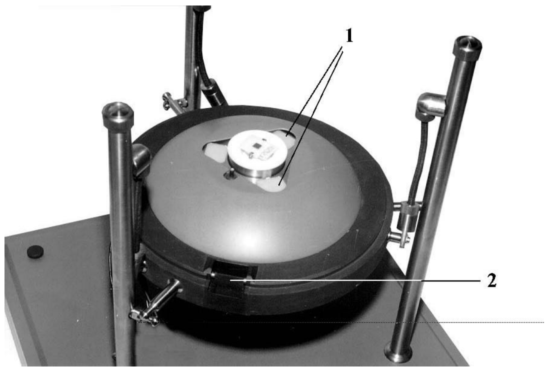


Fig. 32

Install the legs of the measuring head on the supporting plates so (1) that cable going from the head is in front of the socket (2) designed for it (Fig. 32). Insert the holder on the cable into the socket (Fig. 33). Watching from the side, approach the sample manually to a distance of 2-3 mm from the probe. Looking down on the head, place the probe over the area selected for investigation, then again from the side watch the approach of the sample to a distance of 0.5-1 mm (Fig. 34). It is advisable to have the investigated area of the sample on the scanner axis i.e. in the middles of the line connecting the substrate clips. Should the investigated sample be located away from the scanner axis there will be a parasitic incline of the surface in the course of scanning. It limits the use of some features and makes the operation more difficult.

Fig. 33

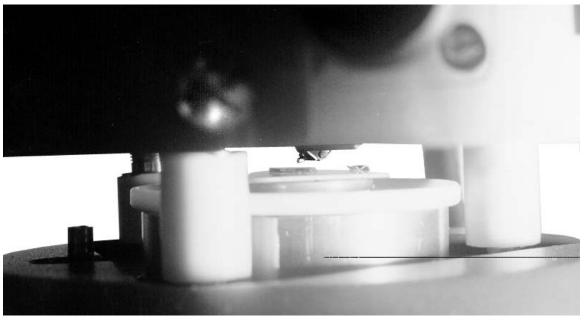


Fig. 34

Cover with the hood so that the grounding wire attachment on the hood is not coincident with the cable attachment socket. Ground the hood. Lift and turn the scanner unit to suspend it by the hangers and using the suspension adjustment knobs bring it into horizontal position (Fig. 14).

3.1.4 Approaching the sample to the probe in automatic mode

Switch the device on and Start the program. Click the button in the middle top of the screen and select the STM mode from the open menu by pressing the appropriate button and press the left mouse button (Fig. 35). The window for the selection confirmation will be displayed, press "Yes" (Fig. 36).

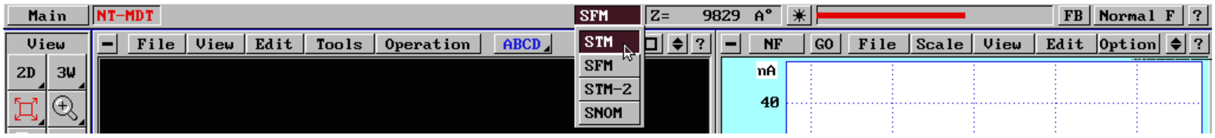


Fig. 35

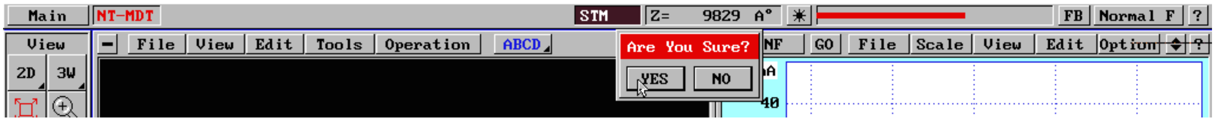


Fig. 36

In the upper right corner choose the feedback input "Ipr - Low" (Fig. 37). Move the mouse cursor to the "Operation" button. In the opened menu choose the "Approach" item (Fig. 38) and press the left mouse button. In "Probe" label set up the parameters for the sample landing "Set Point" = 0.1 (Fig. 39), "Bias Voltage" = 0.1 (Fig. 40), "Feed Back Gain" is about 1.5 (Fig. 41). The set up values may be saved on the hard drive by clicking on the "Save" button, for future reading and application (Fig. 42).

Fig. 37

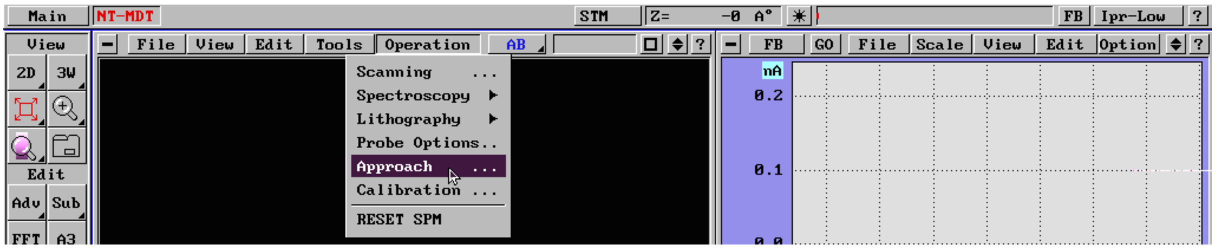


Fig. 38

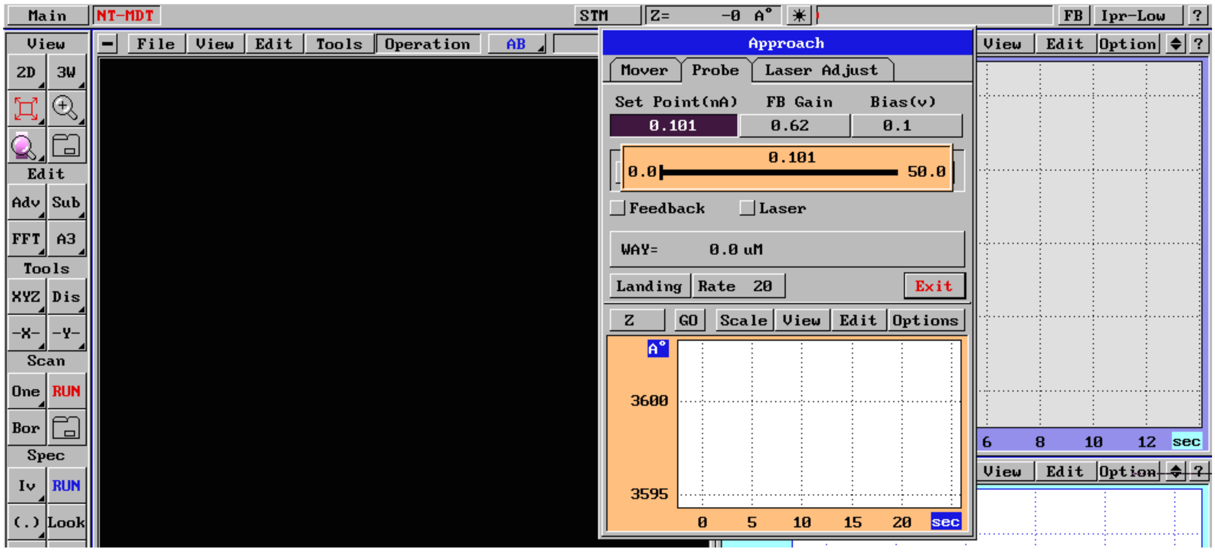


Fig. 39

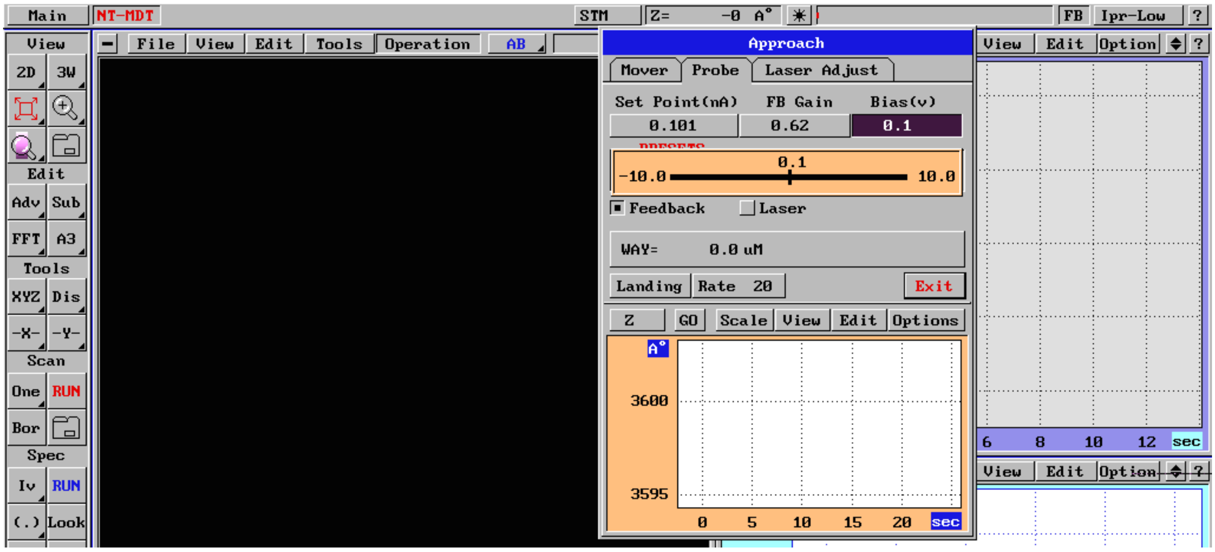


Fig. 40

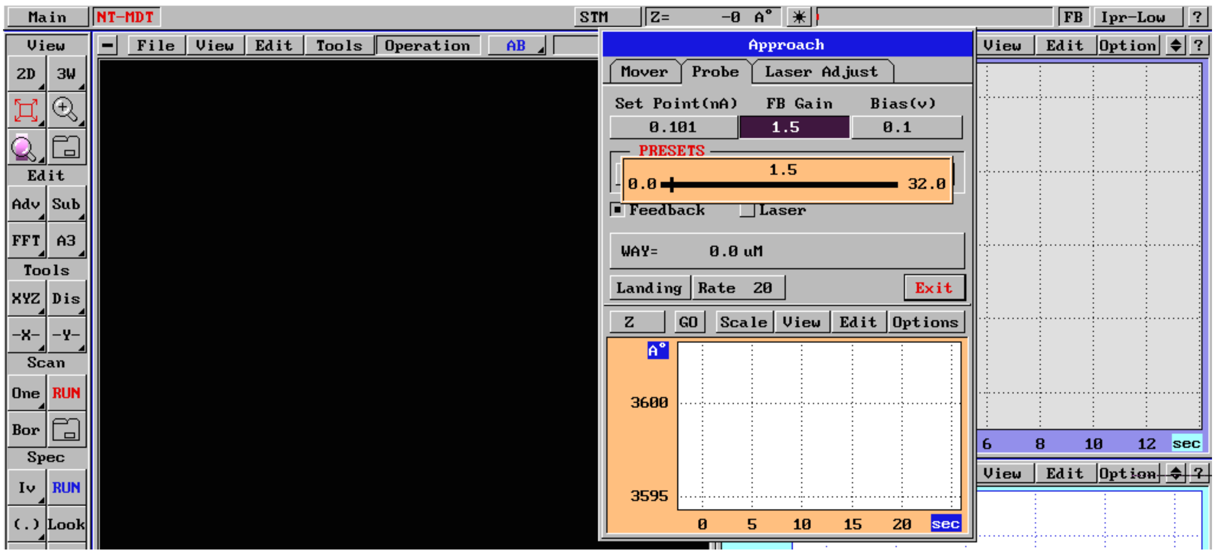


Fig. 41

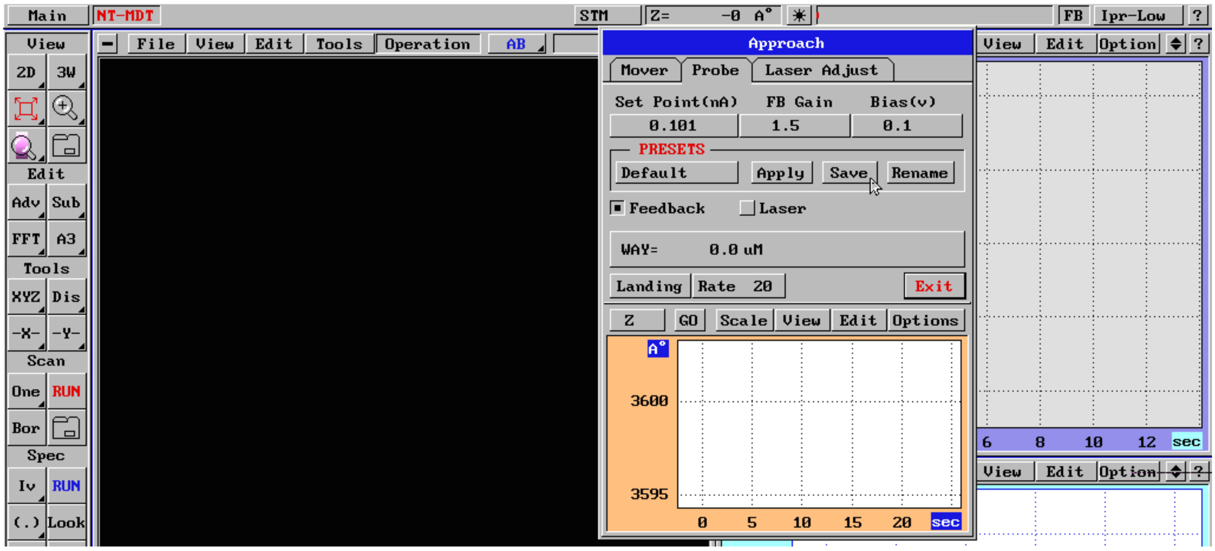


Fig. 42

Before landing it is advisable to turn on the oscilloscope ("FB" input terminal (Fig. 43) - Feedback) by pressing the "GO" button (Fig. 44) and to watch the feedback signal during the landing.

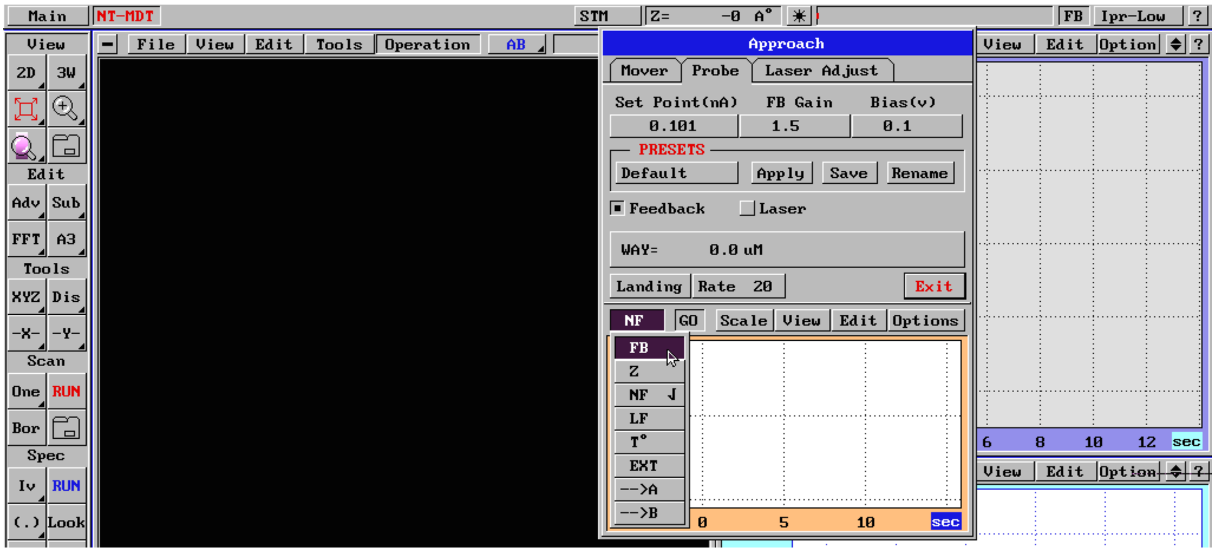


Fig. 43

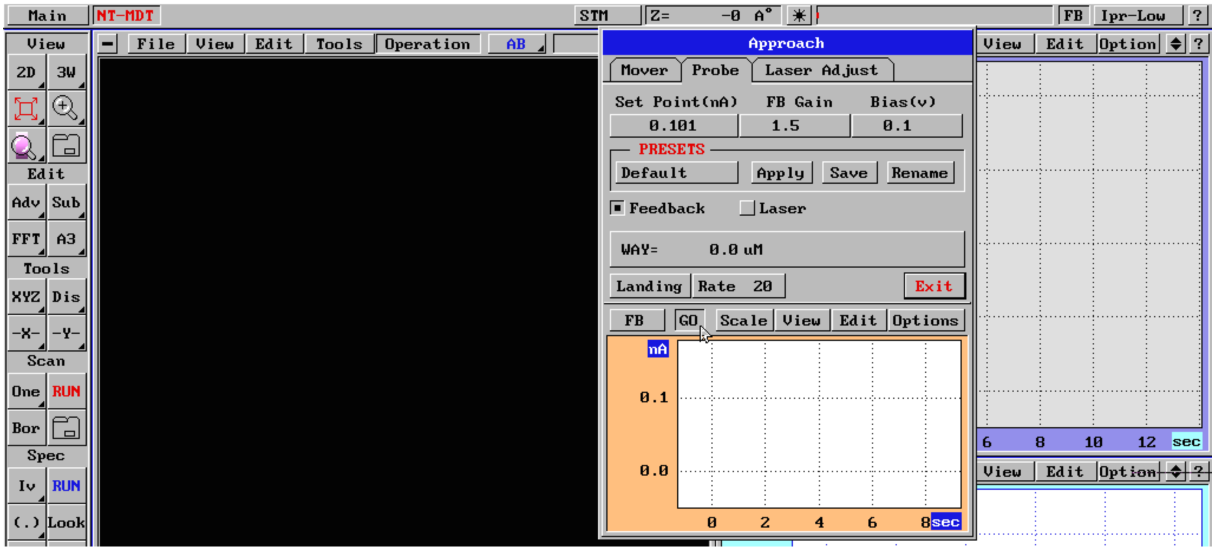


Fig. 44

Set the maximum Landing Rate (Fig. 45).

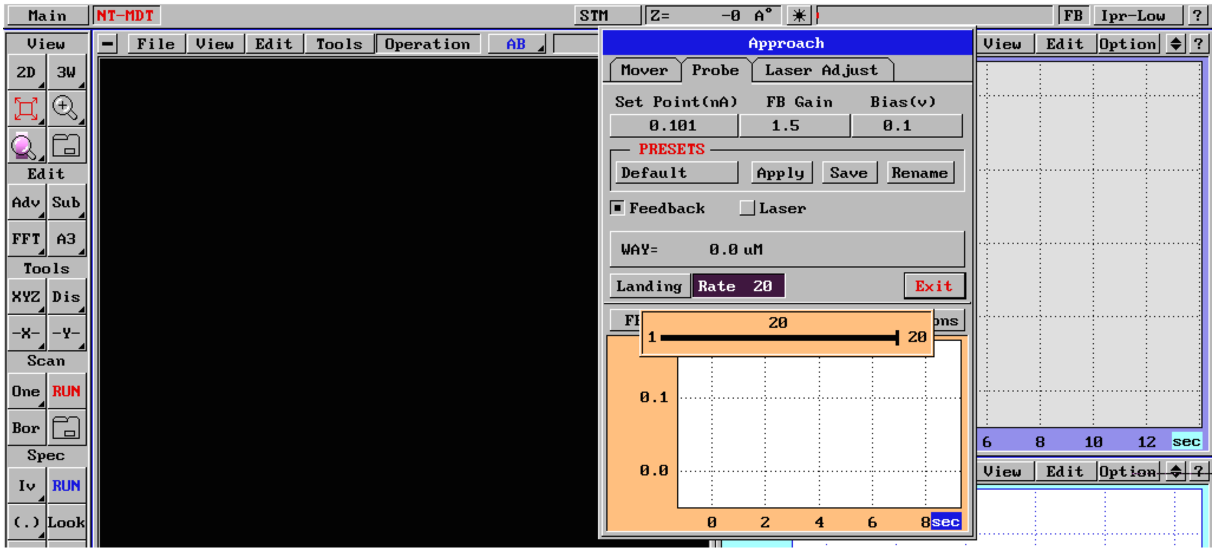


Fig. 45

It is advisable to turn off the measurement of currently unnecessary signals in oscilloscopes as computer time is assigned for the signal measurement and also switching of the automatic centering device between signals to be measured may create interference. In order to turn off the measurement of a currently unnecessary signal it is necessary to open the list of the oscilloscope inputs (Fig. 46), in front of the signals being measured there is a check sign. Choose the signal you want to measure and press the “GO” button (Fig. 47). Should several signals be turned off, repeat this procedure for all currently unnecessary signals.

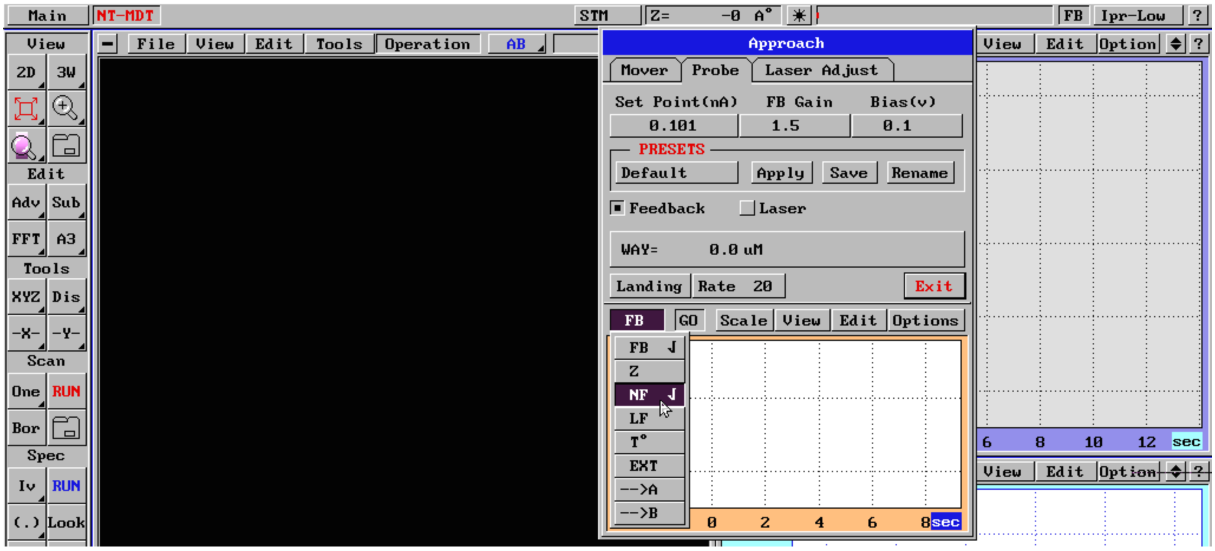


Fig. 46

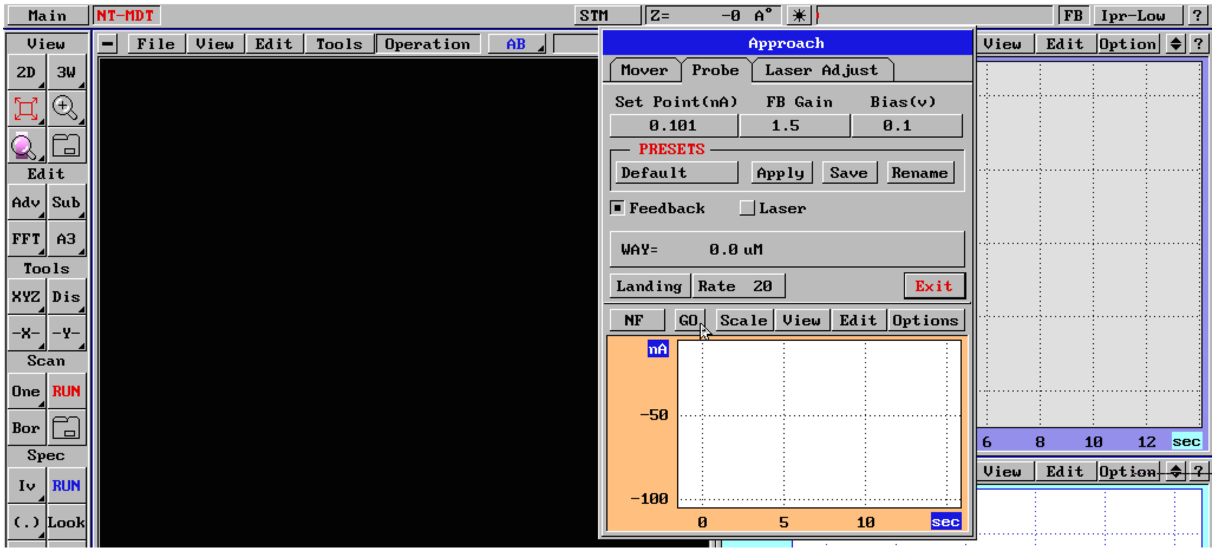


Fig. 47

Click on the "Landing" button (Fig. 48). The landing is done automatically and this is indicated by an audible signal and an on-screen message. At the same time the feedback should be enabled (the "FB" button in the top right corner of the screen will look pressed and the scanner must move to the maximum front position which is indicated by a red bar in the top right corner of the screen. The reading of step engine counter will increase. In case the feedback is enabled but the scanner has been moved and the reading of the counter starts decreasing, stop the landing by pressing the ESC key or the right mouse button in any position.

For the operation in the contact STM mode the value set in "Set Point" should be higher than the current value of the FB or NF signal. For example, SP=0, current value of FB=-5nA.

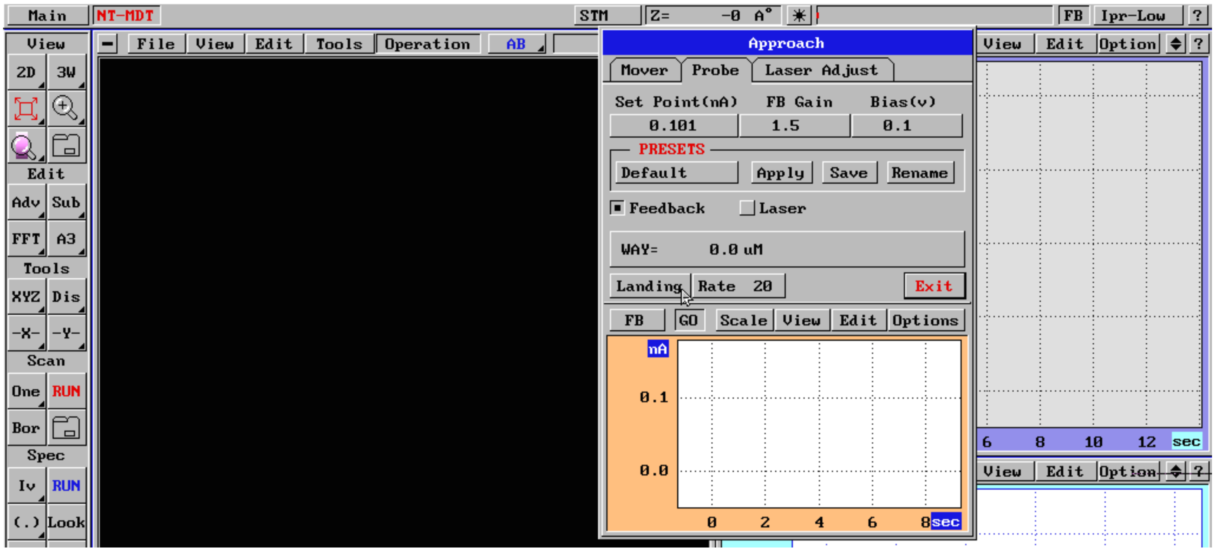


Fig. 48

For fine investigations (nanometric objects) set the mover in the backlash mode. To do this go to the Mover item in the Approach menu. After decreasing the Forward Rate to 4-6 (Fig. 49) make one or more steps forward by pressing the "Forward One step" button (Fig. 50) while watching the red bar in the top right corner of the screen which indicates the scanner extension. It is not recommended to set the scanner in the backlash mode for rough relief samples the scanner position fine setting with the step engine is impossible. The bar must decrease to one forth of its maximum length. After it click on "Backward Until ESC" (Fig. 51) and wait until the counter counts back about 100 microns. As soon as this value is reached, stop the step engine by pressing the ESC key on the keyboard.

In the backlash mode the scanner is mechanically linked to the step engine and the single pressing of the "Backward"/"Forward"-"One step" button do not change the scanner extension (red bar in the top right corner of the screen) within the limits of the current noise.

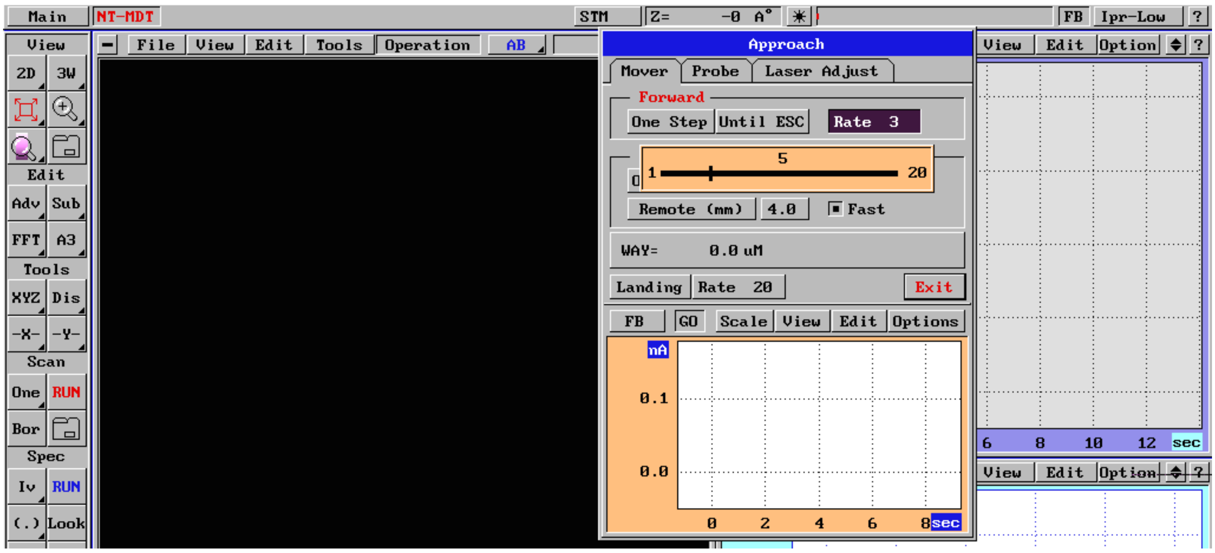


Fig. 49

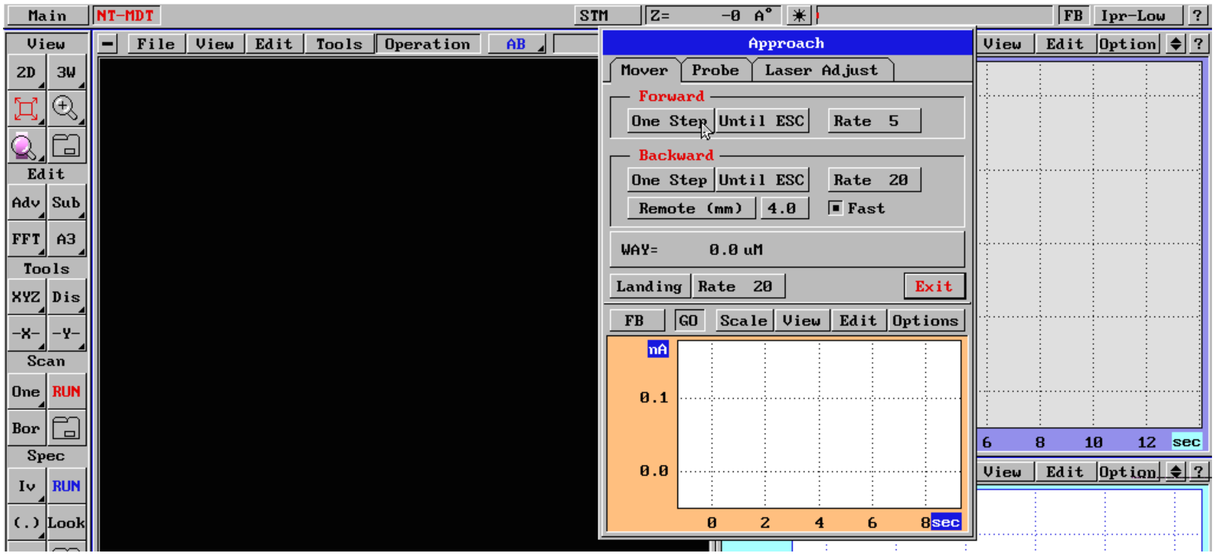


Fig. 50

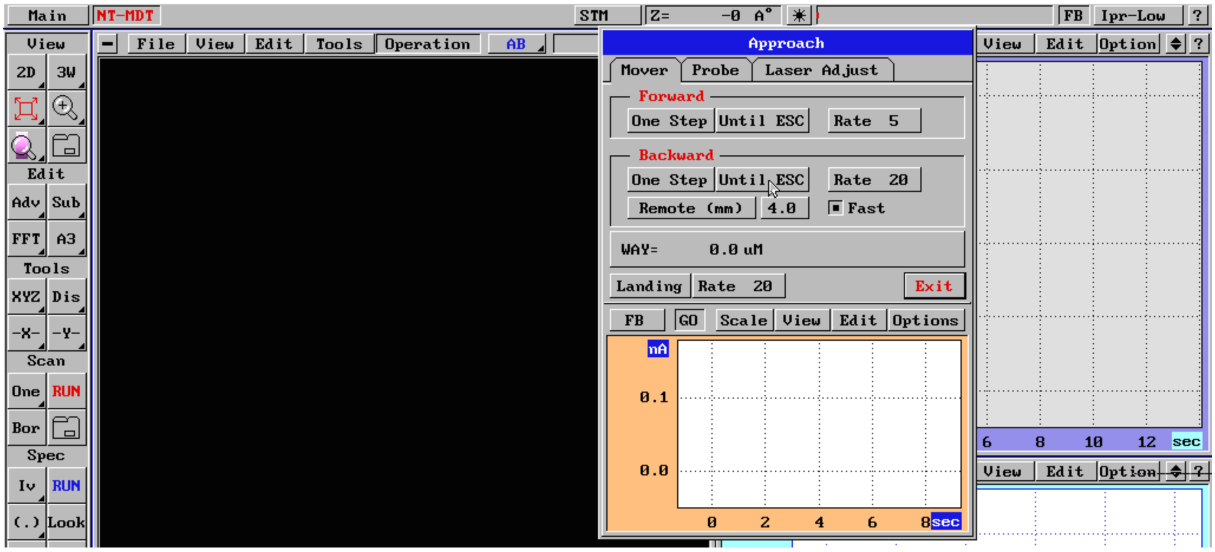


Fig. 51

3.1.5 Getting test scan

The SPM window on the left side of the screen is designed for scanning. The microscope control is done through the “Operation” menu (Fig. 52). Select the "Scanning" (Fig. 53) item in the opened menu and press the left mouse button.

The scanning parameters are set in the opened menu.

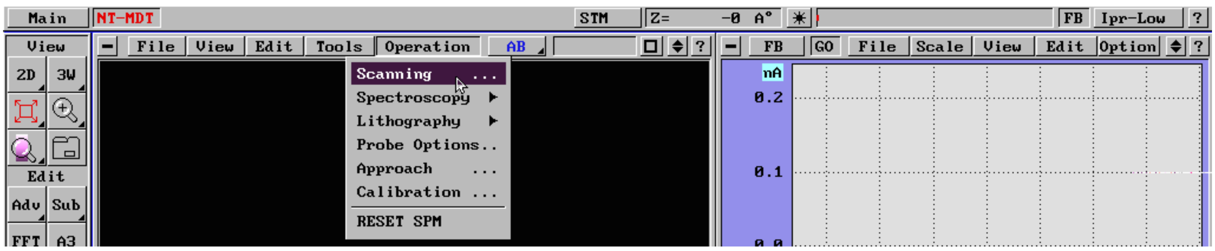


Fig. 52

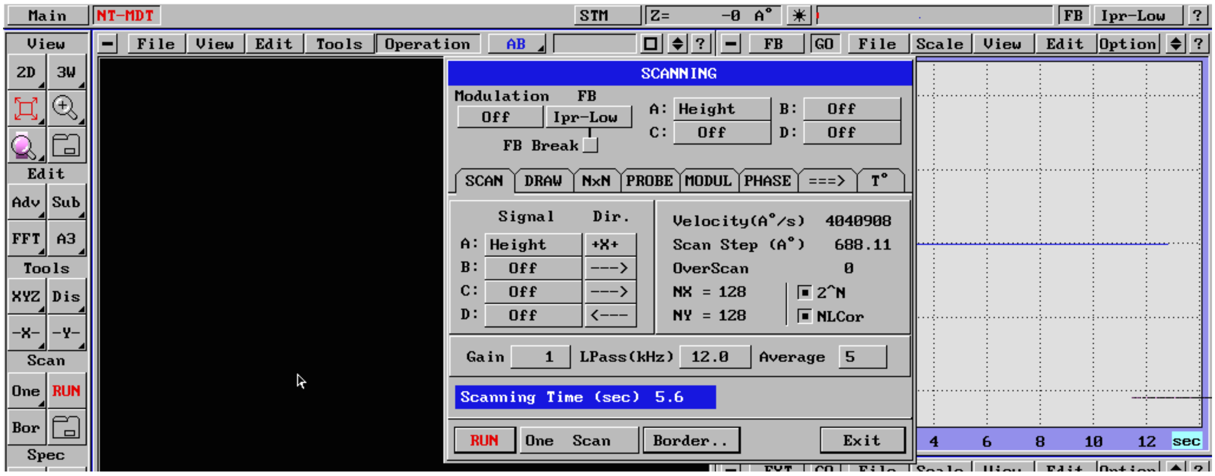


Fig. 53

3.1.5.1 Getting topography on graphite

First of all it is necessary to receive the graphite topographic image.
Set the following parameters:

Modulation - off (Fig. 54)
FB - Ipr - Log (Fig. 55)
Scan A - Height (Fig. 56)
Scan B, C, D - off (Fig. 56)
Velocity = 200000 (Fig. 57)
Step (A) - 20 - 40 (Fig. 57)
NX xNY - 256 õ 256 (Fig. 57)
NL Corr - on (Fig. 57)
A: Scan dir - +X+ (Fig. 57)
Gain = 10 (Fig. 57)
Lpass(kHz) = 3 (Fig. 57)
Average = 10 (Fig. 57)
Drawing - on (Fig. 58)
Subplane - on+ (Fig. 58)
SP - 0.5 (Fig. 59)
FB Gain - 0.1 (Fig. 59)

The "Sp" and "FB" parameters are chosen so that they are sufficiently big but without producing generation.

Select the single scanning mode - One scan (Fig. 60).

Click RUN to start scanning (Fig. 61).

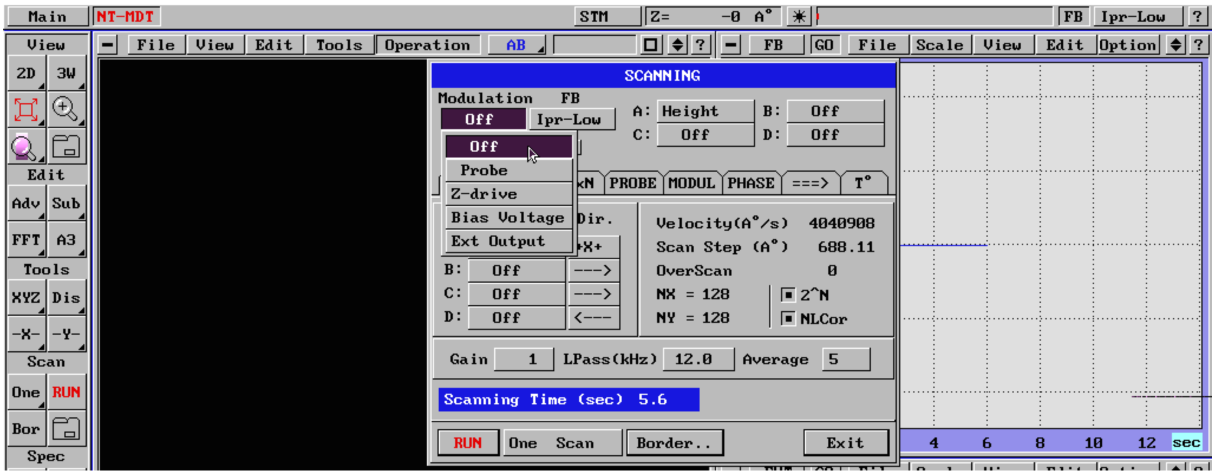


Fig. 54

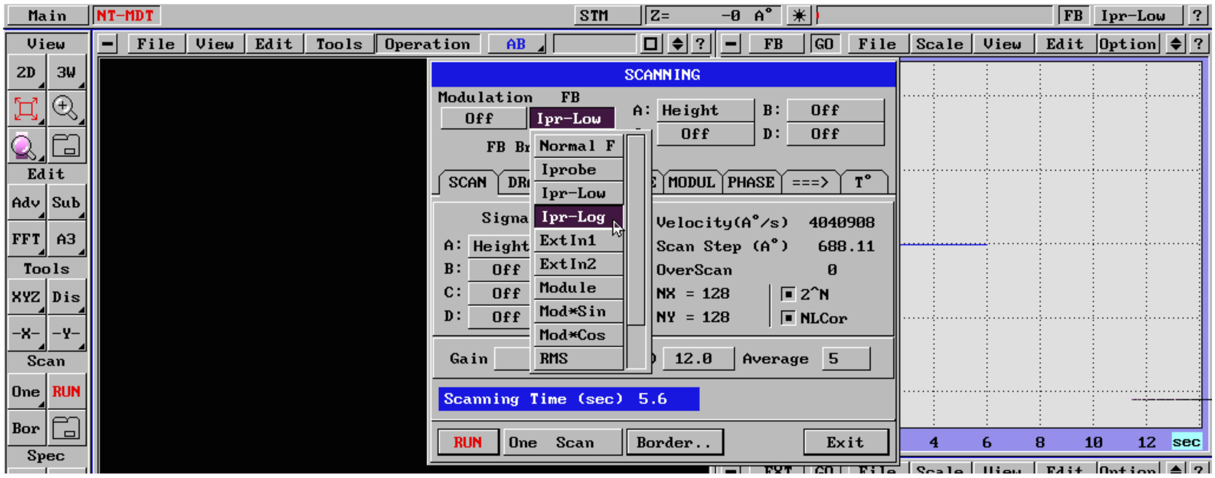


Fig. 55

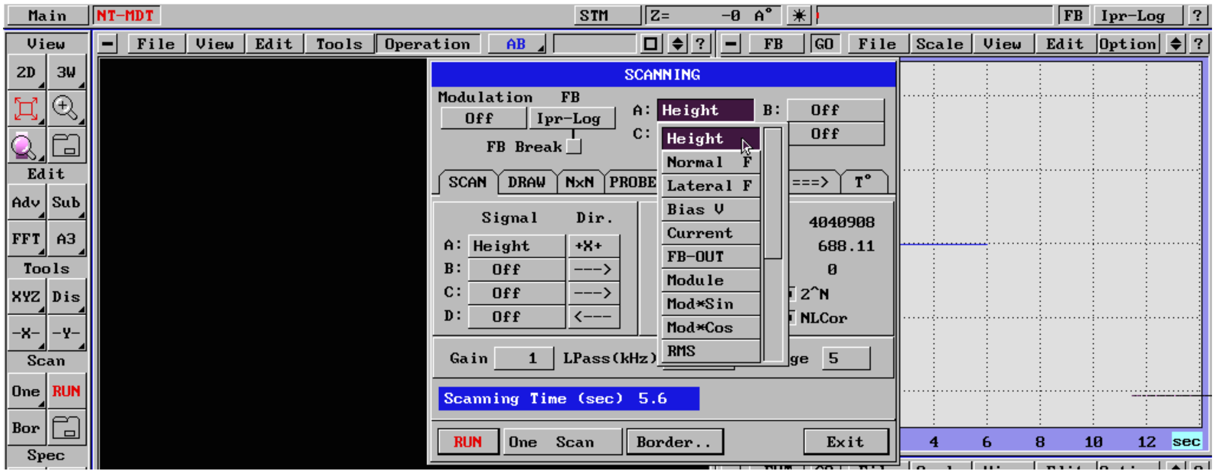


Fig. 56

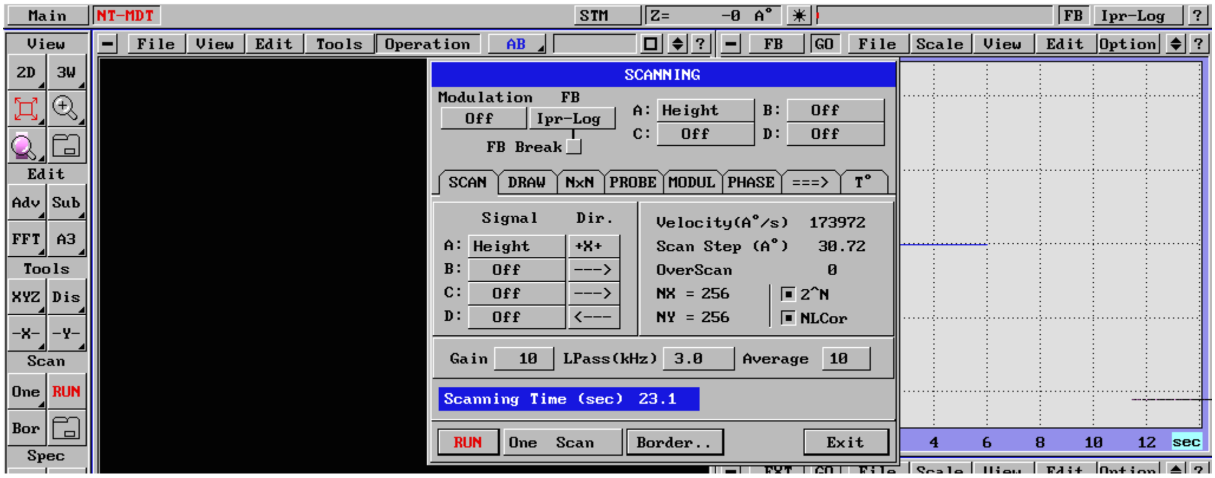


Fig. 57

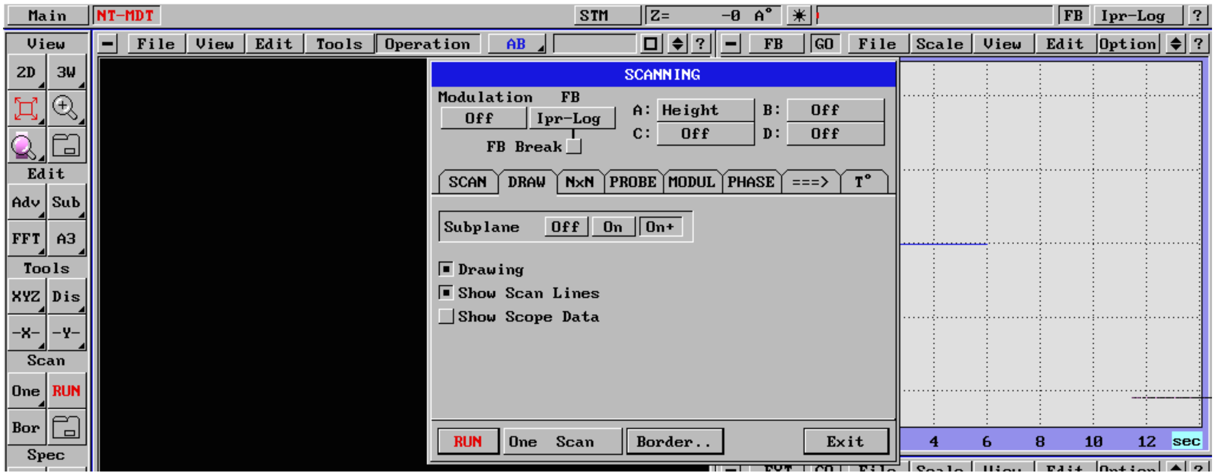


Fig. 58

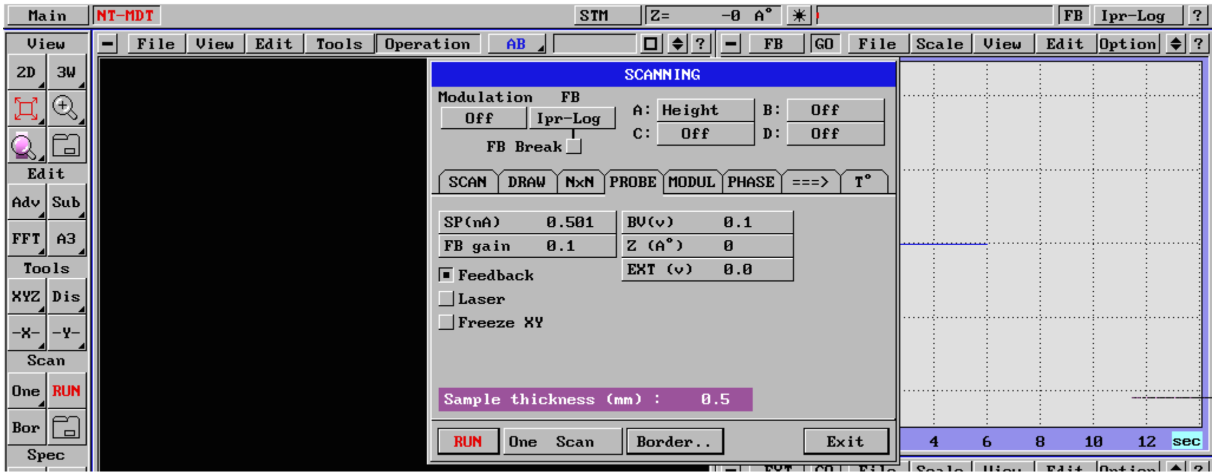


Fig. 59

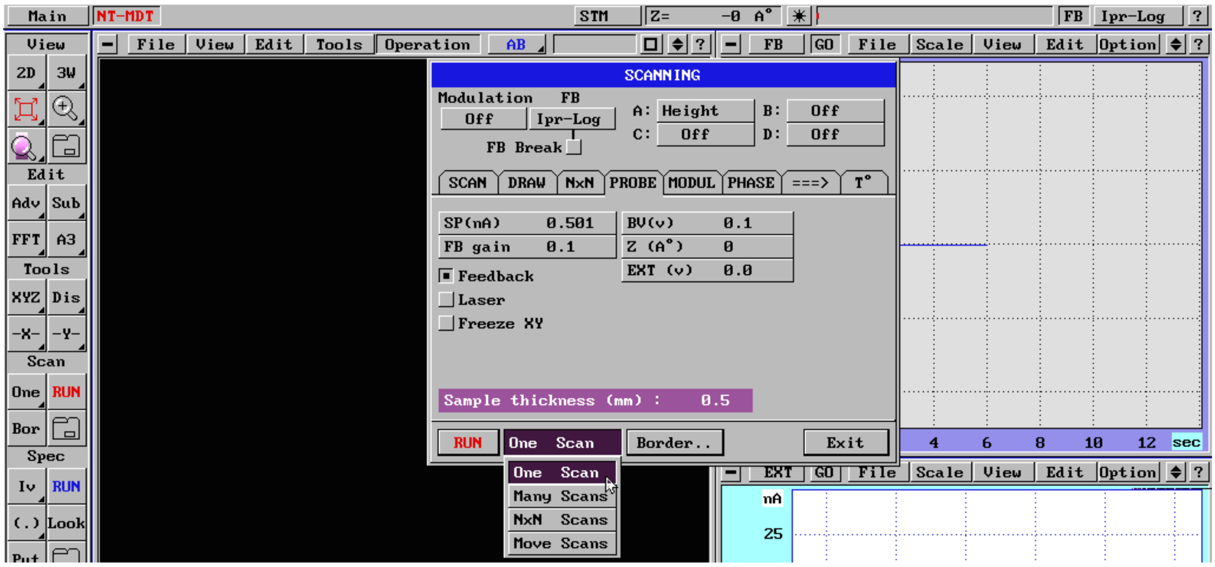


Fig. 60

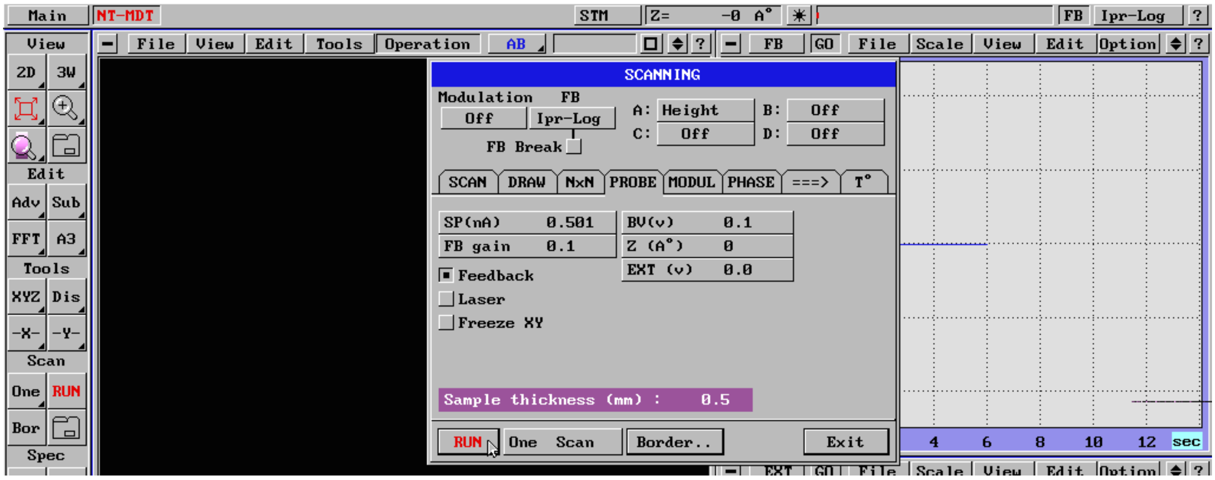


Fig. 61

3.1.5.2 Getting atomic resolution on graphite

Set the following parameters:

Modulation - off (Fig. 62)
FB - Ipr - Log (Fig. 63)
Scan A - Height (Fig. 64)
Scan B, C, D - off (Fig. 64)
Velocity = max (Fig. 65)
Step (A) - 0.2 - 0.5 (Fig. 65)
NX xNY - 128 õ 128 (Fig. 65)
NL Corr - on (Fig. 65)
A: Scan dir - +X+ (Fig. 65)
Gain = 100 (Fig. 65)
Lpass(kHz) = 12 (Fig. 65)
Average = 1 (Fig. 65)
Drawing - off (Fig. 66)
Subplane - on (Fig. 66)
SP - 0.5-1 (Fig. 67)
FB Gain - 0.2-1 (Fig. 67)

The minimum scanning step and the maximum speed may be different for different devices. Select a smooth area on the previously received image. Using the “Board” - “Relative command (Fig. 69) position the frame of the new scanning area on the smooth zone. Enable “Moving” in the Board menu (Fig. 68).

Select the single scanning mode - One scan (Fig. 70).

Click RUN to start scanning (Fig. 71).

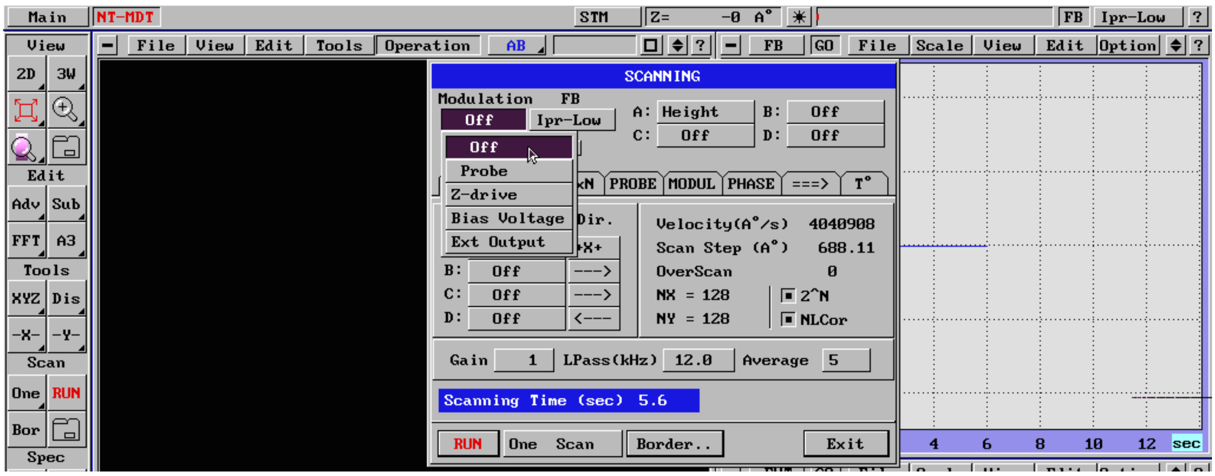


Fig. 62

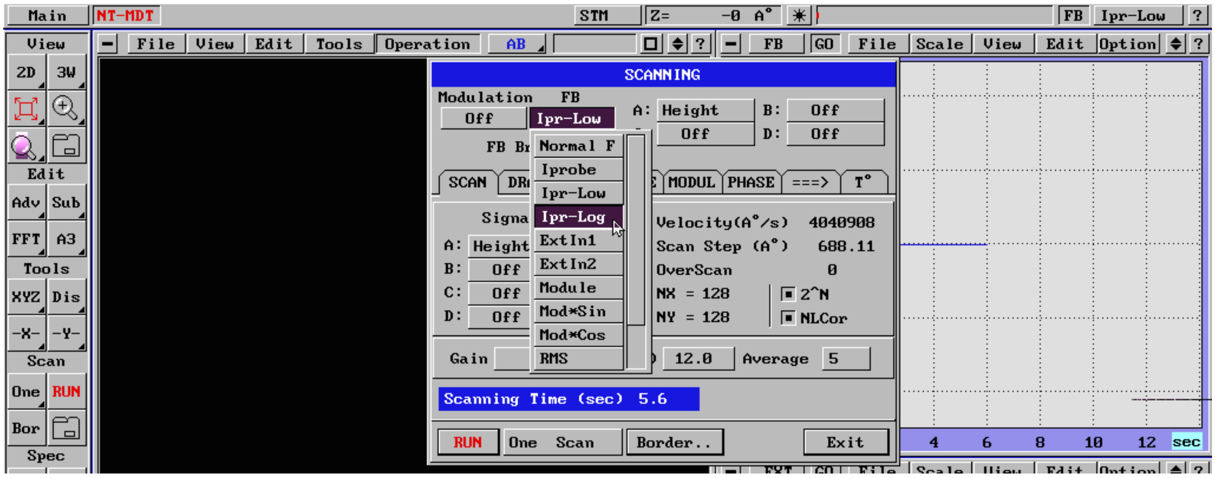


Fig. 63

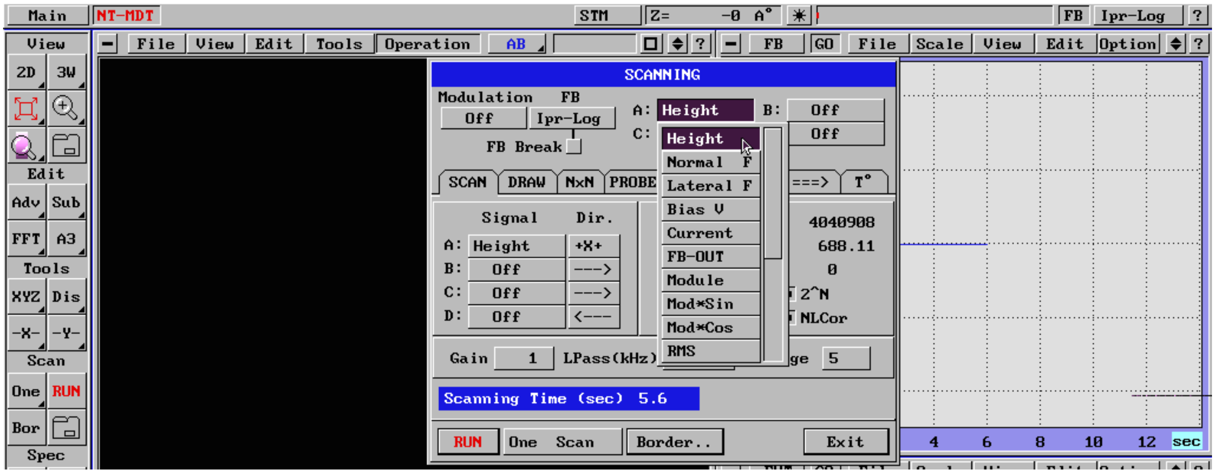


Fig. 64

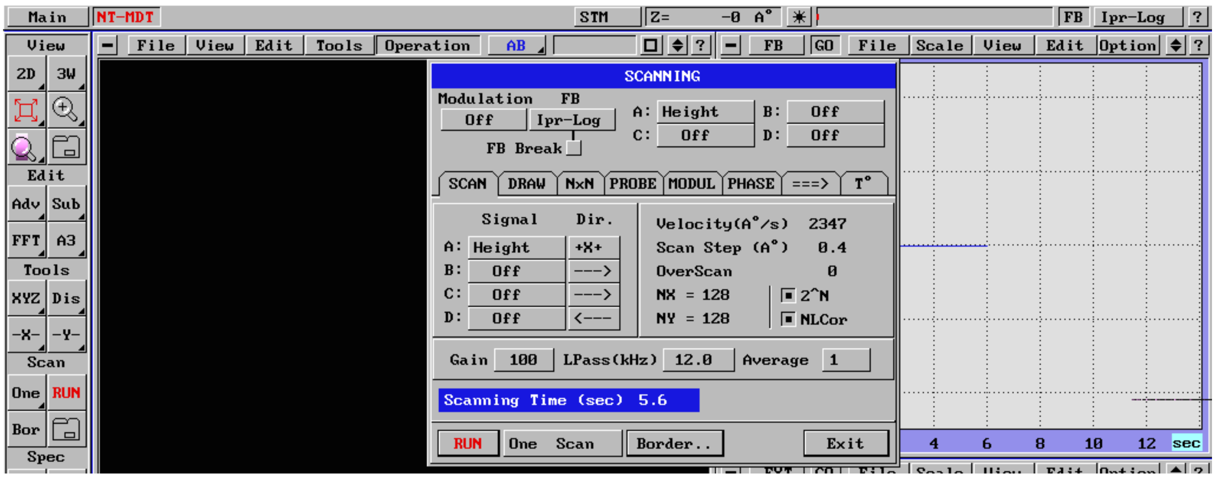


Fig. 65

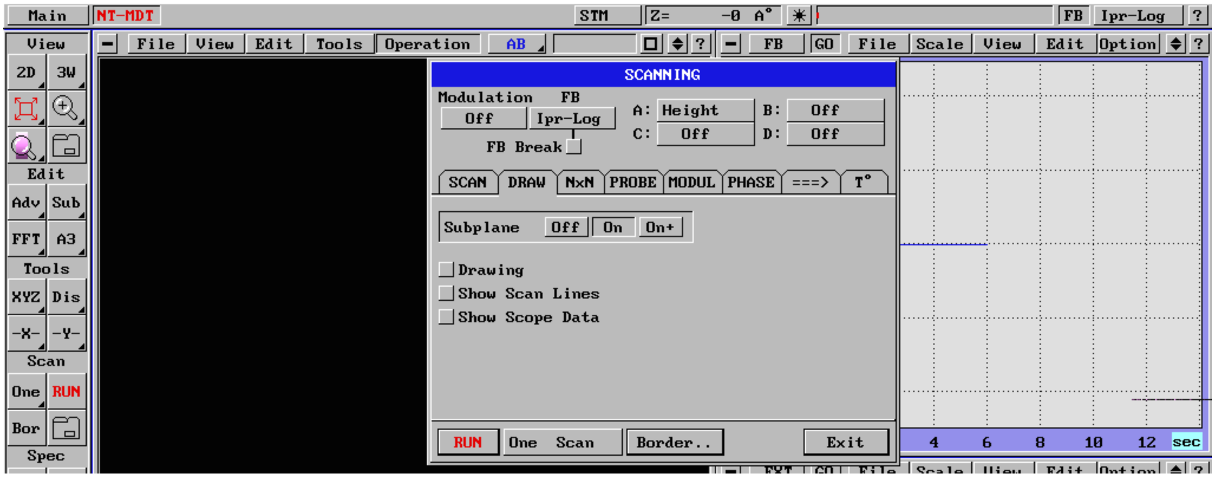


Fig. 66

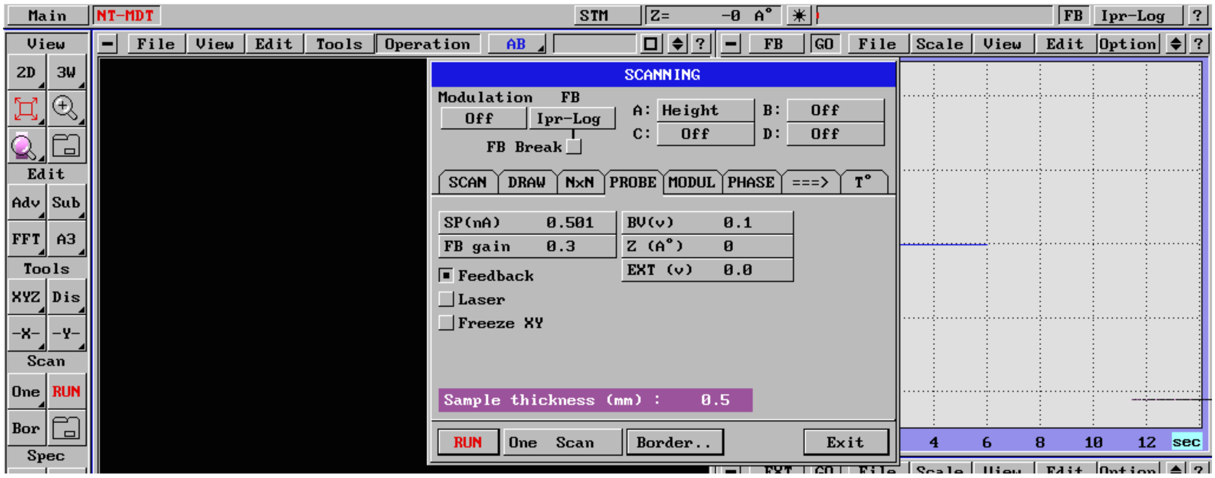


Fig. 67

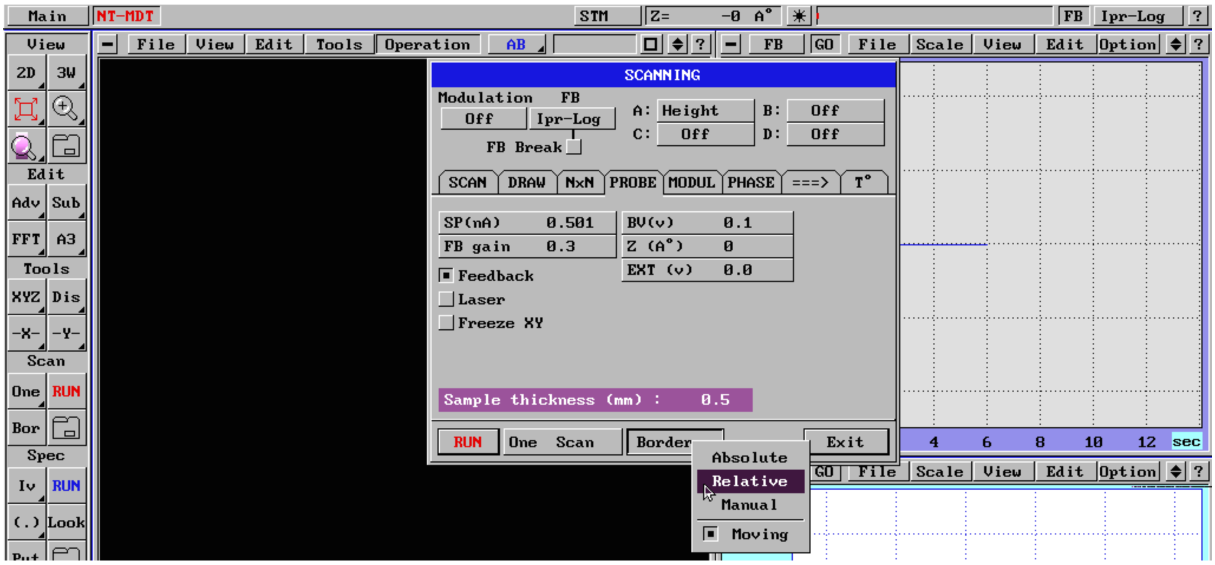


Fig. 68

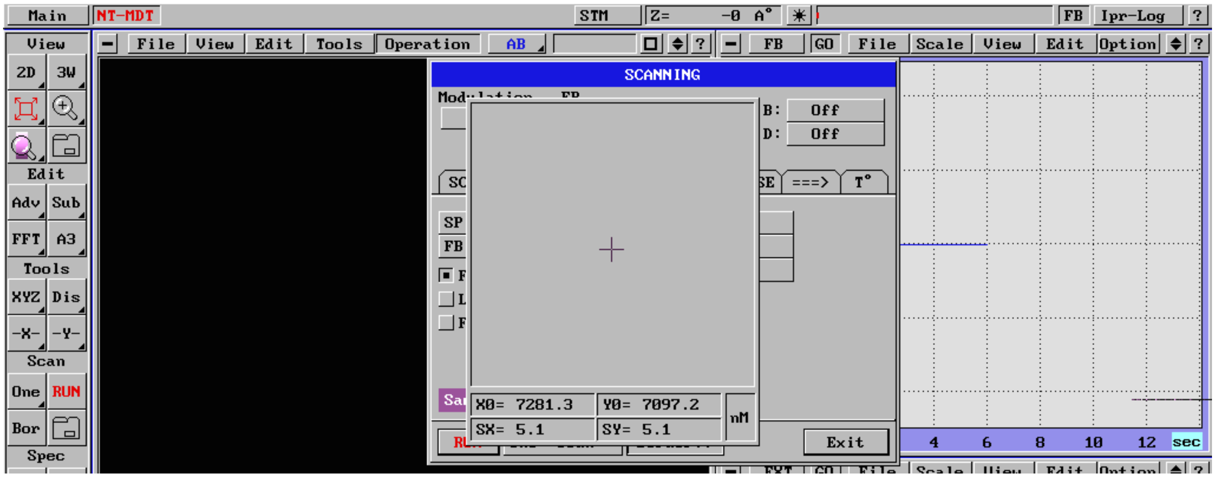


Fig. 69

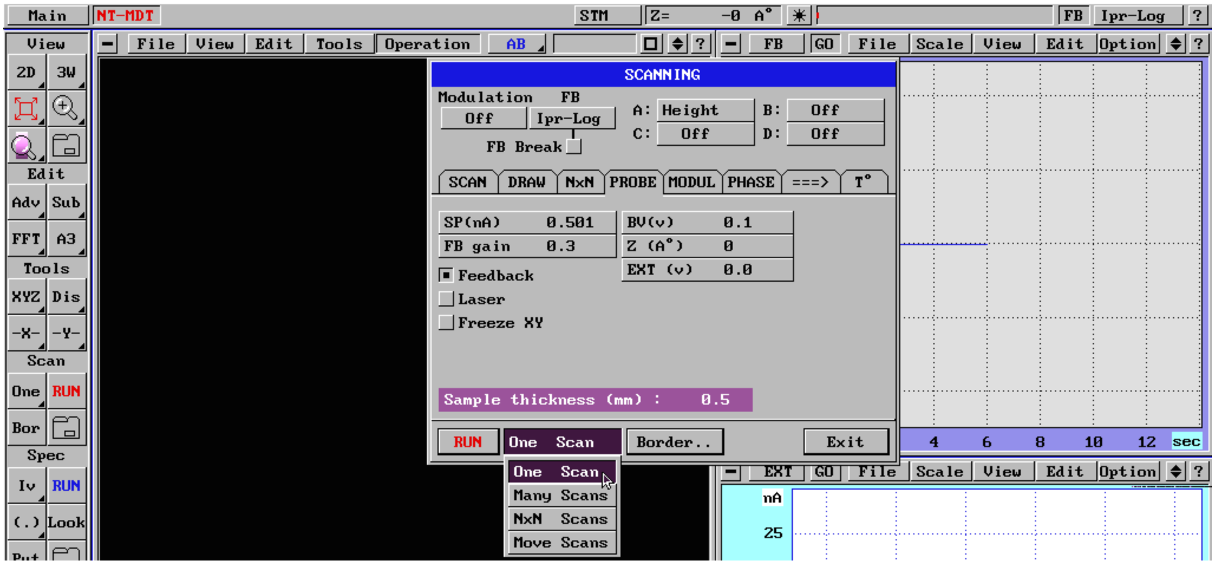


Fig. 70

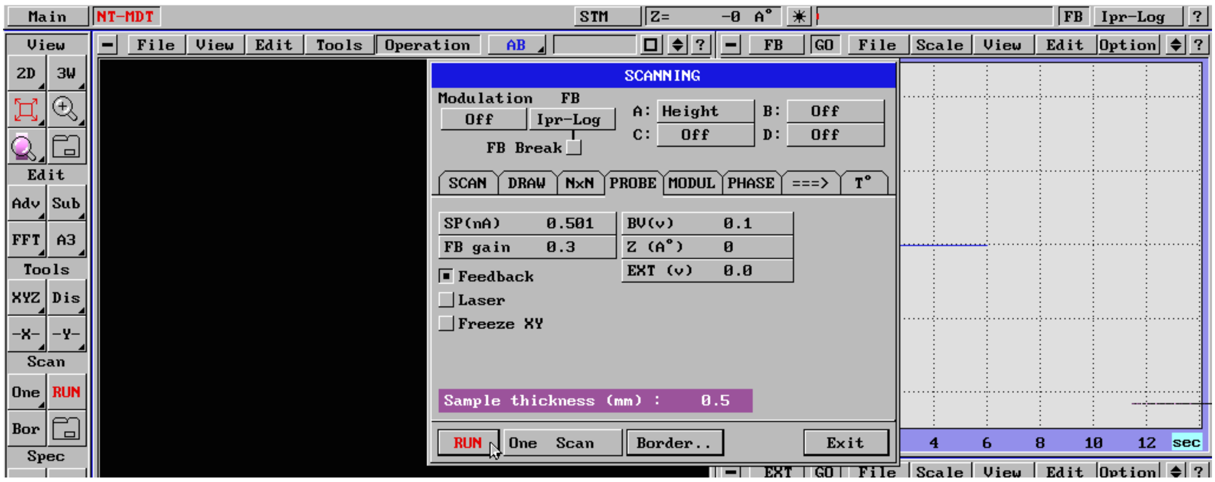


Fig. 71

To obtain the best possible result you can modify the "Step", "Scan dir", "SP", "FB gain" parameters.

3.1.5.3 Measuring in the current registration mode (Z=const)

☝ **In this mode it is possible to scan only very smooth surfaces without any substantial inclination because the feedback is not supported in the course of scanning and the tip may run into the sample.**

Set up the parameters:

Modulation - off (Fig. 72)
FB - Ipr-Low (Fig. 73)
Scan A - Current (Fig. 74)
Velocity - max (Fig. 75)
Step (A) - 0.2 - 0.5 (Fig. 75)
NX xNY - 128 õ 128 (Fig. 75)
NL Corr - on (Fig. 75)
A: Scan dir - +X+ (Fig. 75)
Gain = 1 (Fig. 75)
Lpass(kHz)=12 (Fig. 75)
Average = 1 (Fig. 75)
Drawing - off (Fig. 76)
Subplane - on (Fig. 76)
SP - 0.1 (Fig. 77)
FB Gain - 0.2 - 1 (Fig. 77)

and the additional parameter:

FB Break - on (Fig. 78)

The minimum scanning step and the maximum speed may be different for different devices.
Select the single scanning mode - One scan (Fig. 79).
Click RUN to start scanning (Fig. 80).

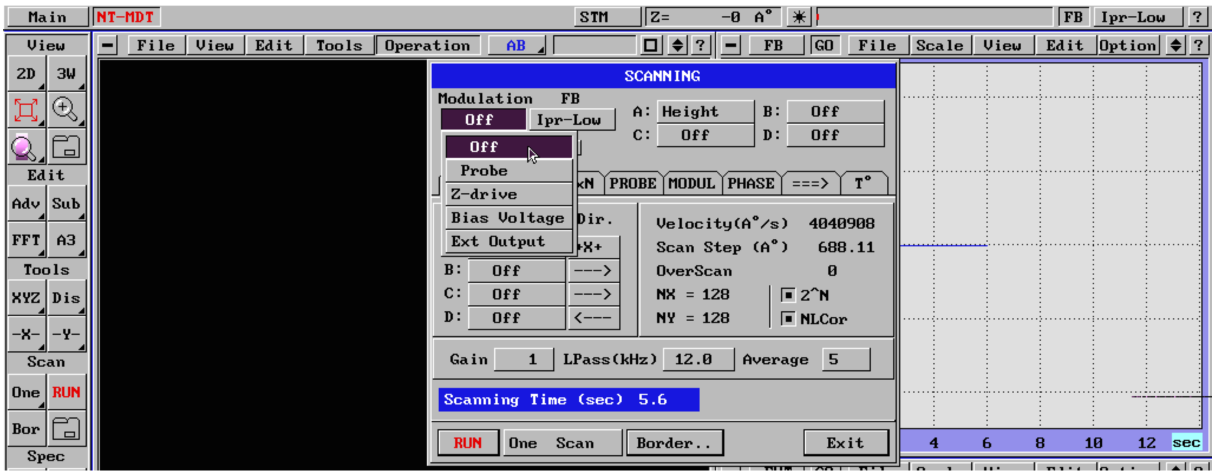


Fig. 72

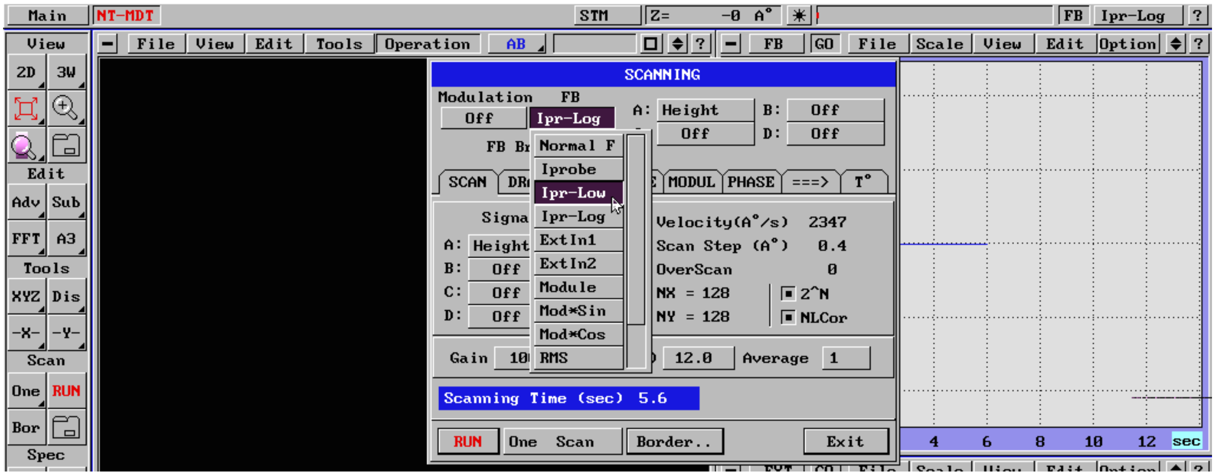


Fig. 73

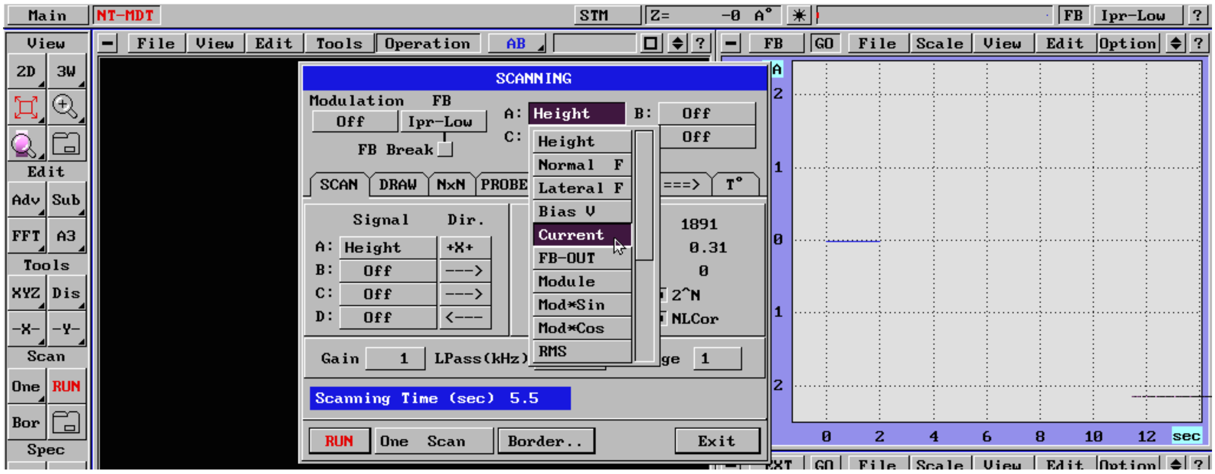


Fig. 74

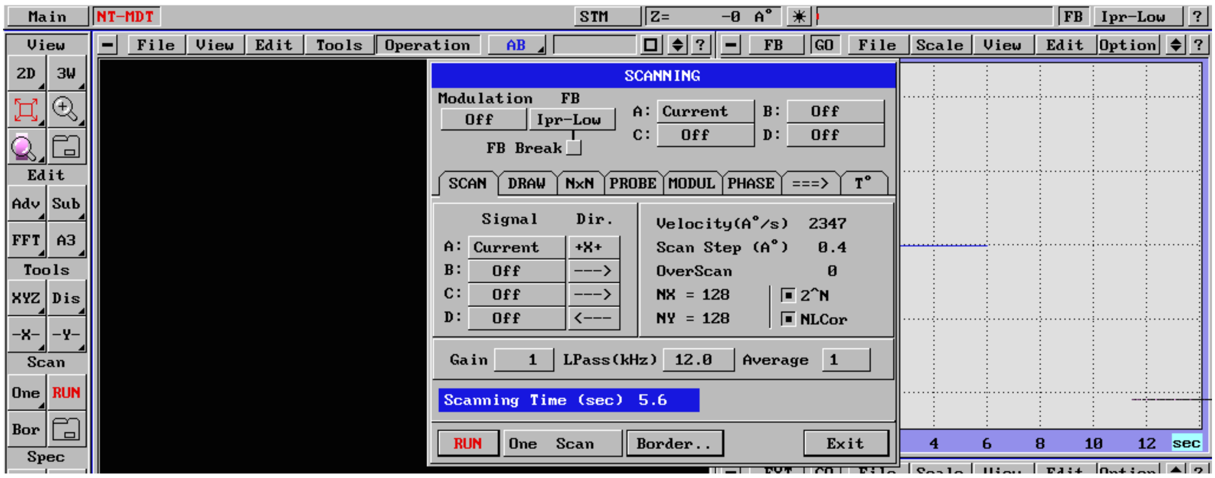


Fig. 75

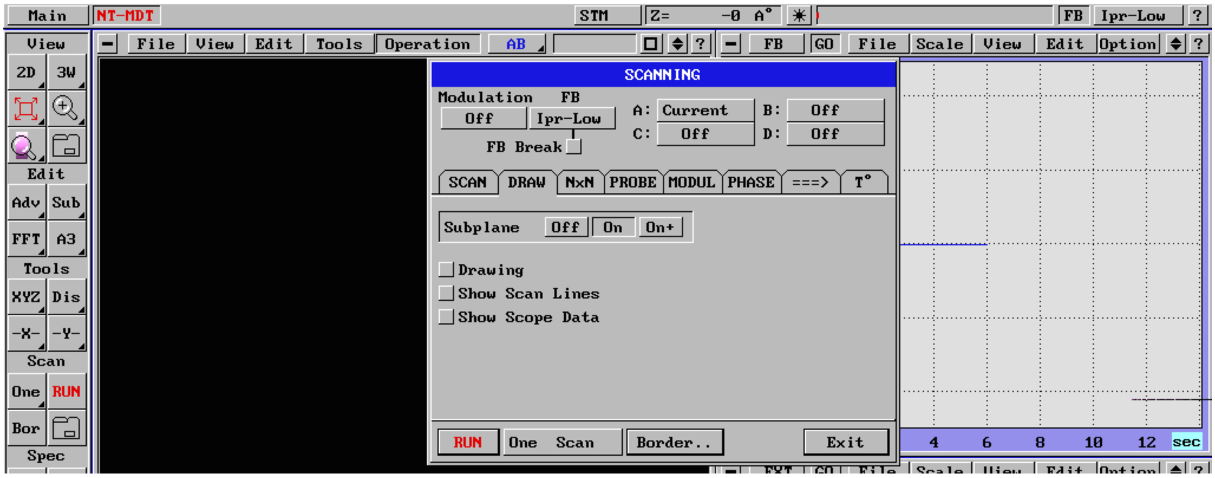


Fig. 76

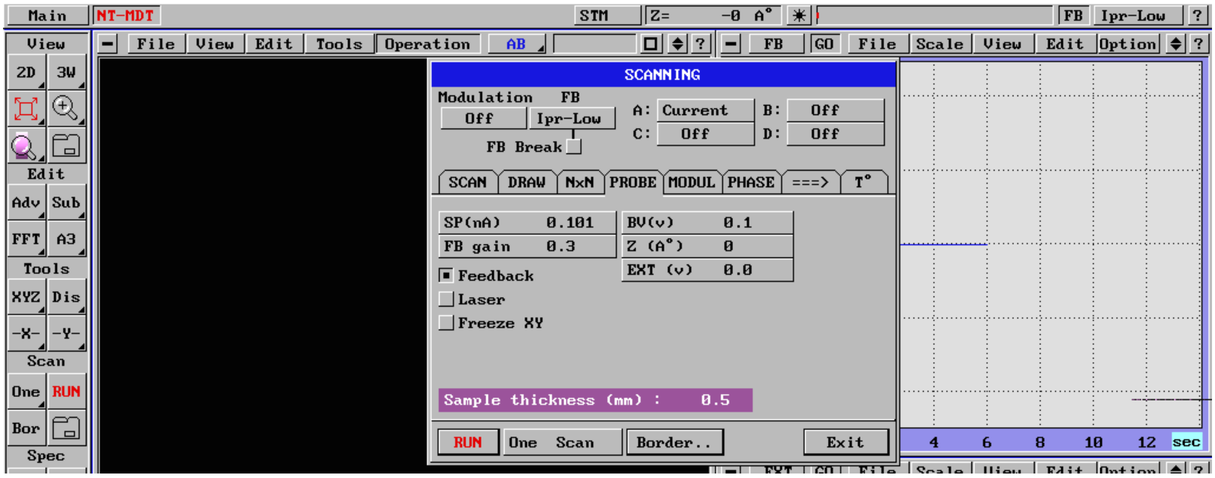


Fig. 77

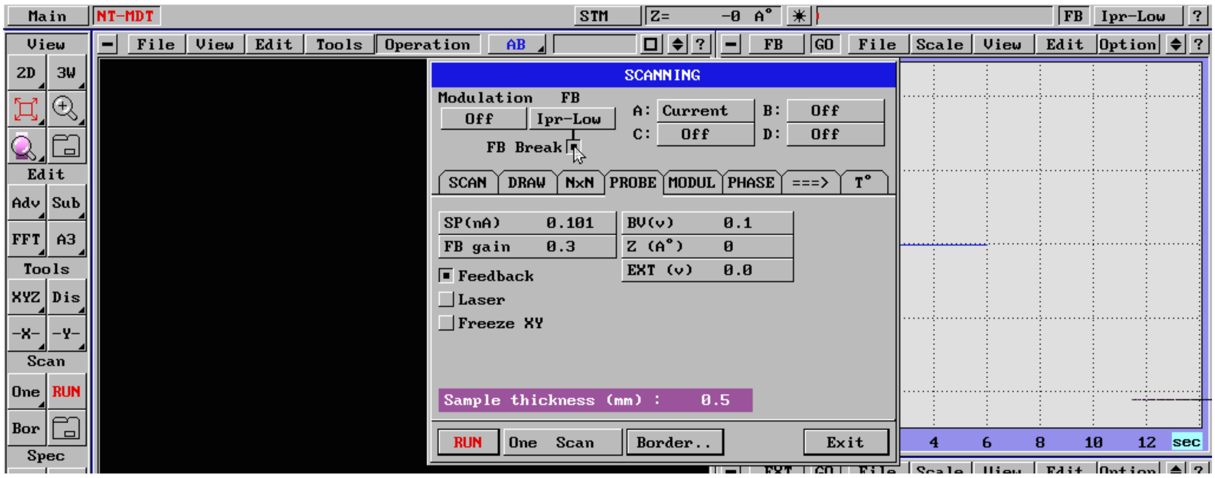


Fig. 78

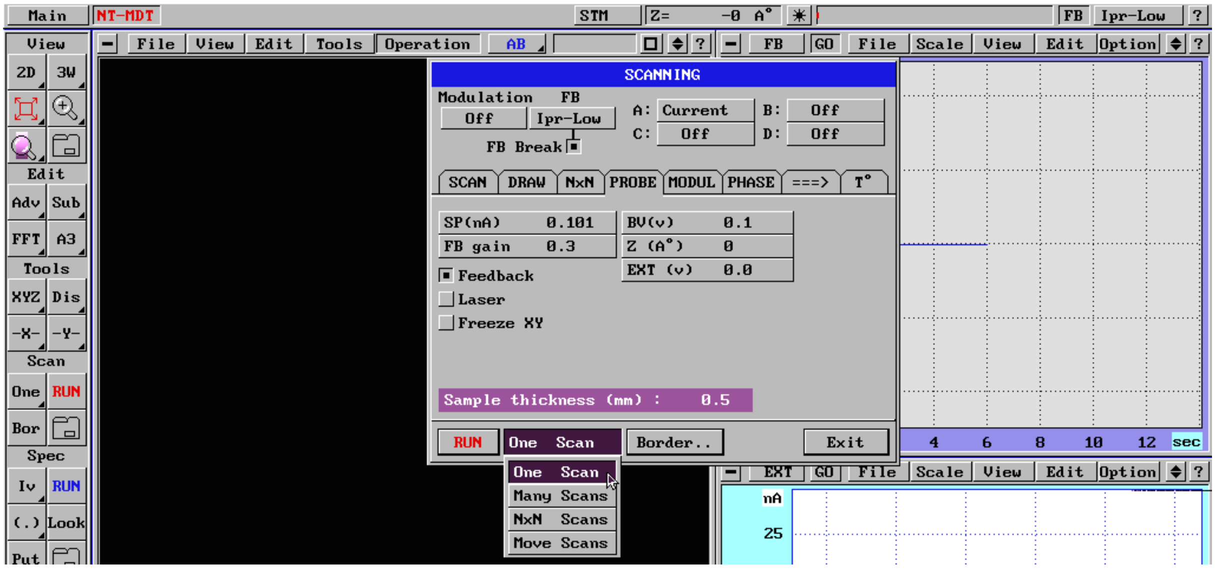


Fig. 79

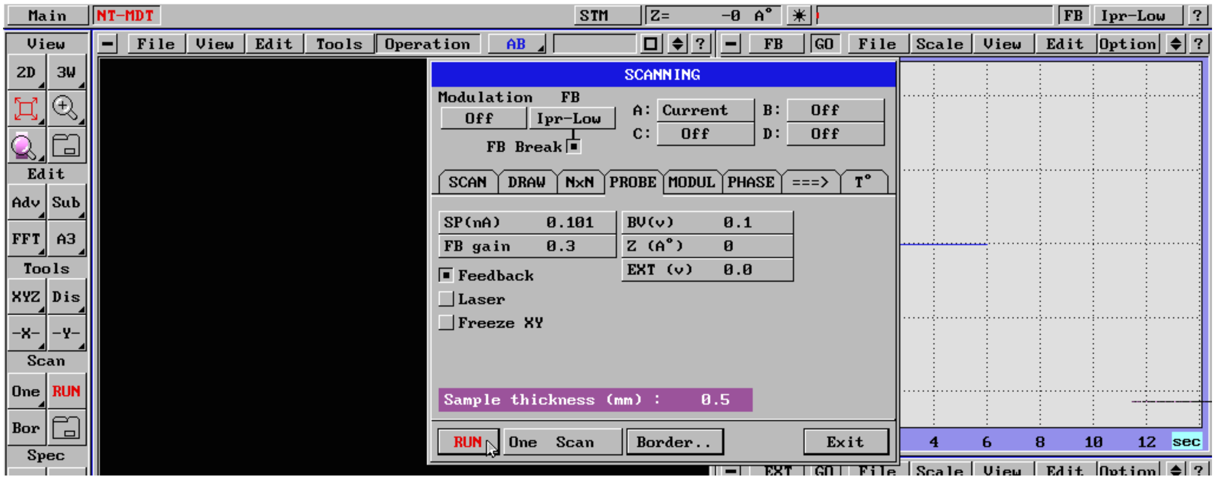


Fig. 80

3.1.5.4 Spectroscopy I(U)

Spectroscopy I(U) consists of taking the voltage and amperage parameters of the tunnel tip-sample passage (for the STM mode) or the conductive probe-sample contact (for the AFM mode).

In order to carry out the I(U) spectroscopy in the STM mode first it is necessary to scan the required object.

Then enter the spectroscopy parameters menu and select the spectroscopy mode (Fig. 81).

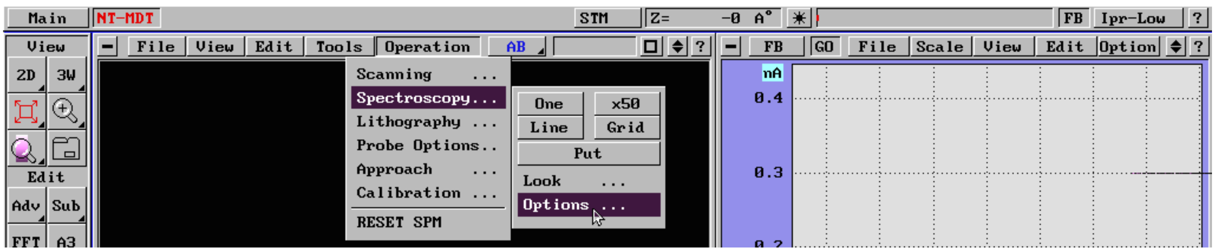


Fig. 81

Note that the voltage and amperage parameters can be obtained by two methods: I(U) and Fb(U). However, the latter method is suitable only if the feedback is supported using the "Iprobe" or "Ipr-low" signals. If the feedback is supported using the "Ipr-low" signal or in case of AFM, "Normal Force", the I(U) mode only is suitable for taking the voltage and amperage parameters (Fig. 82).

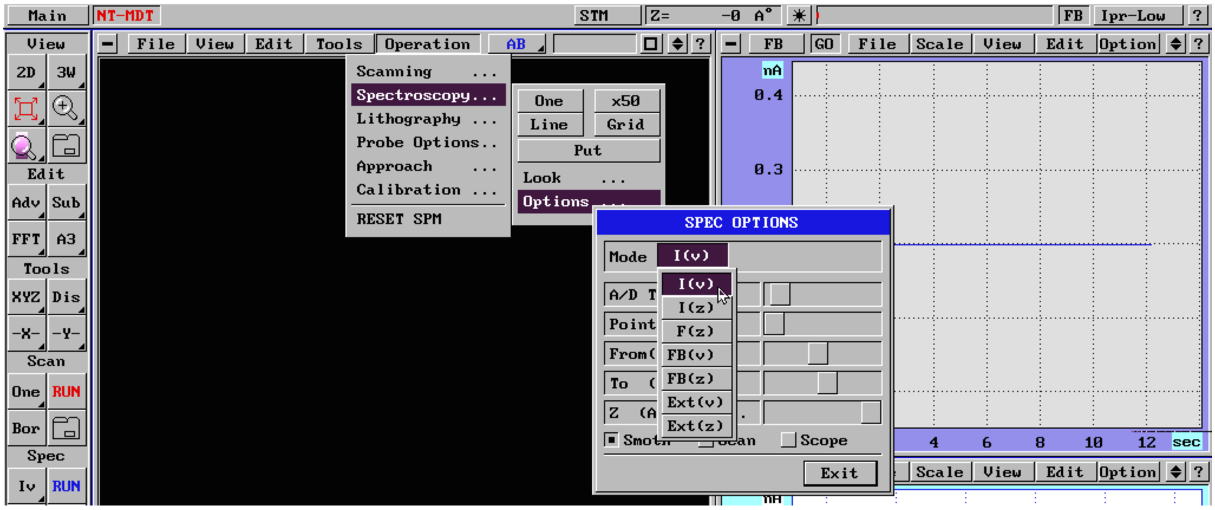


Fig. 82

The other parameters of the spectroscopy have the following meaning:

"A/D Times" - number of measurements in each point of the voltage scanning with subsequent averaging;

"Points" - number of points where the voltage and amperage parameters are to be taken;

"From(U)", "To(U)" - range of the voltage applied;

"Z(A)" - disabled in this case;

"Smooth" - smoothing of the parameter;

"Scan" - taking the spectroscopy parameter simultaneously with scanning data (topography).

For the first taking of the voltage and amperage parameters enable the Scan option and after setting up the required parameters (for example, (Fig. 83): Mode I(U), A/D Times=20, Point=100, From -1.0V, To 1.0V) enter the spectroscopy menu.

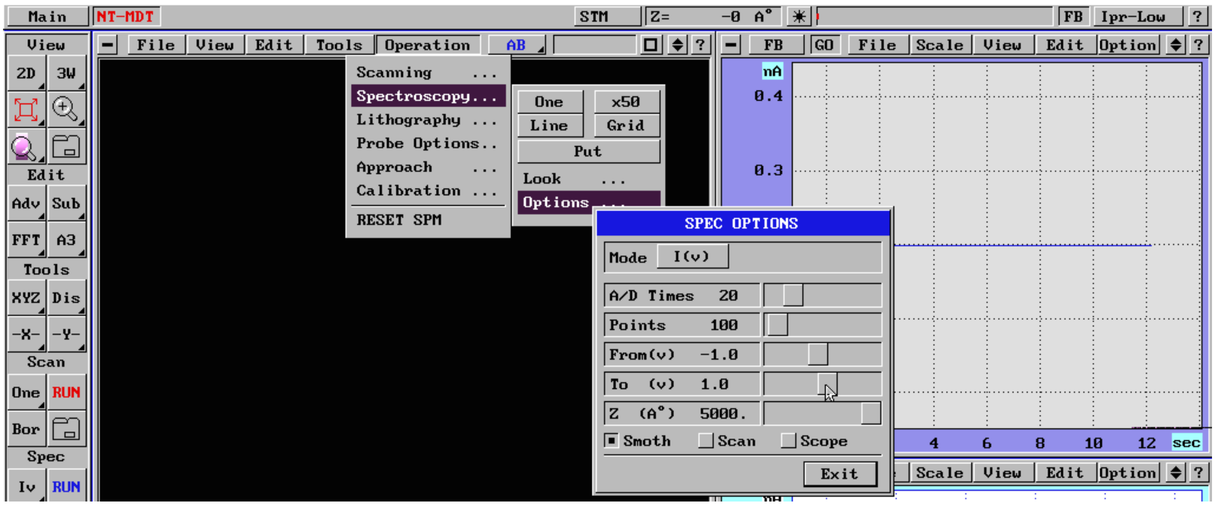


Fig. 83

First let us carry out single scanning of the voltage and amperage parameters "ONE" (Fig. 84).

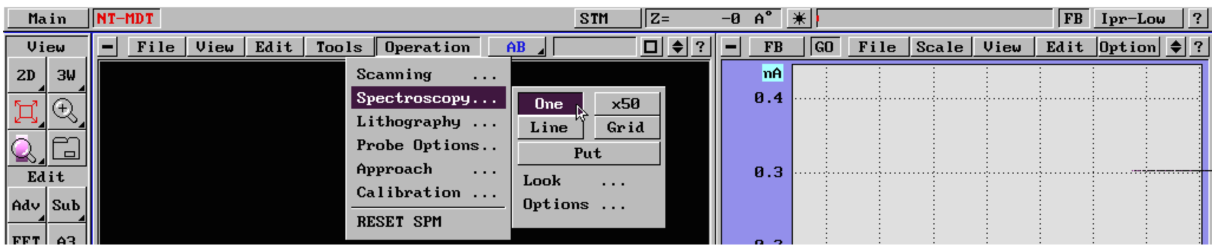


Fig. 84

After the “ONE” button is pressed a marker appears on the scanning field. Place it with the mouse on the required place and press Enter. The voltage and amperage parameter will be displayed in the oscilloscope field as well as the point where it has been taken will be displayed on the scanning field and the marker will remain on the scanning field.

You can take one or more parameters in various points or exit this mode by pressing the right mouse button or the "ESC" key.

The last taken parameter remains in the random-access memory until the next parameter is taken. If you want to leave it to view later or save in a file press the "Put" button (Fig. 85).

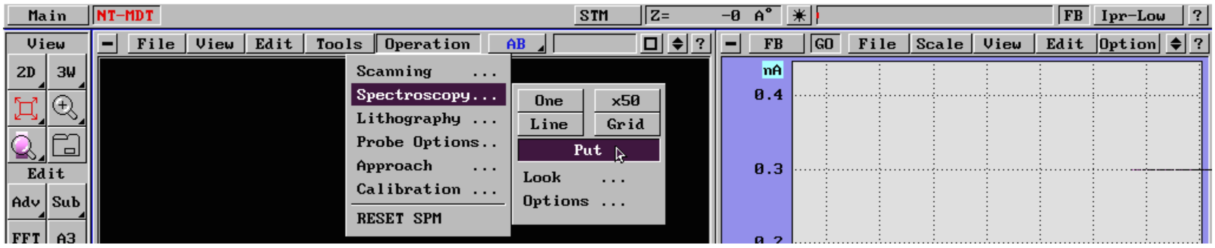


Fig. 85

Other commands of spectrometry execution: "x50", "Line", "Grid" create three-dimensional spectroscopy data where the XZ plane contains the voltage and amperage parameter and the Y axis contains the parameter number.

When the "x50" command is used one point must be selected, “Line” - two end points of a line, “Grid” - number of points in a grid. The number of parameters taken using the Line command corresponds to the number of points in the scan image along the selected line.

You can view the spectroscopy results using the "Look" menu (Fig. 86) (it can be pulled down in the SPM window).

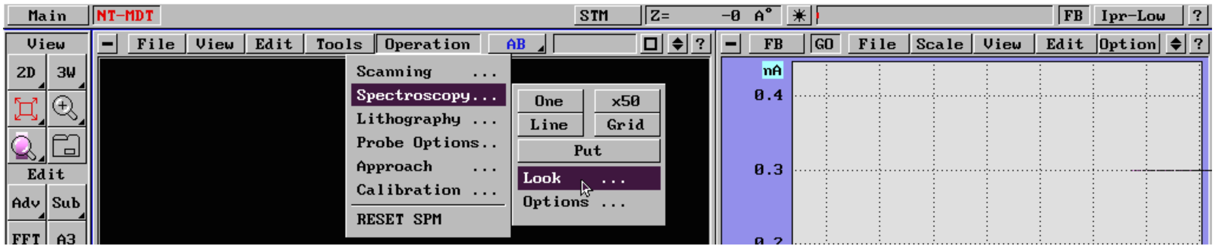


Fig. 86

The "View" command displays in the OSC window one of the spectrometry parameters, the “All” command displays all the parameters one by one. Using the commands "<<" and ">>" you can view other spectrometry data for this picture (out of those who have been saved earlier using the “Put” command) and the "Scan" and "Spec" commands display in the SPM window either the scanning or spectrometry data (Fig. 87).

Fig. 87

In case the scanning data show drifting along the X, Y axis (caused, for example, by a recently glued sample) it is useful to use the “Scan” parameter in the spectrometry options.

To do this enable the “Scan” parameter in the spectrometry options (Fig. 88) and specify for the last scan results spectrometry along a line by drawing it across the required area. In this case this area will be scanned and spectrometry will be done in the selected points. This will result in a scan image and spectrometry data that have been determined with an absolute accuracy in relation to the scan.

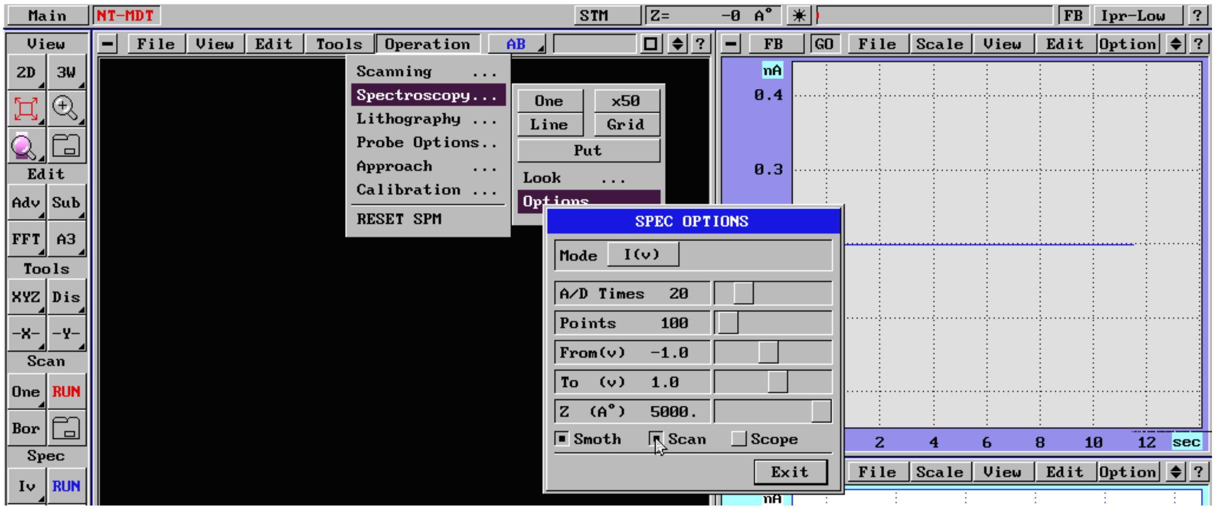


Fig. 88

☝ **The parameters determining the scanning area ("Step", "Nx", "Ny", "Border") should not be changed as compared to the previous scan.**

3.1.5.5 Spectroscopy I(z)

Spectroscopy I(z) is taking the relation between the change of the tunnel current when the probe is being removed from the sample (for the STM mode). This parameter shows very well the quality of the tip point. I(z) parameter taking is described in this section as an example of estimating the sharpness of the probe.

In order to carry out spectroscopy I(z) in the STM mode first make a scan with the required object.

After it enter the spectrometry parameters menu and select a spectroscopy mode (Fig. 89).

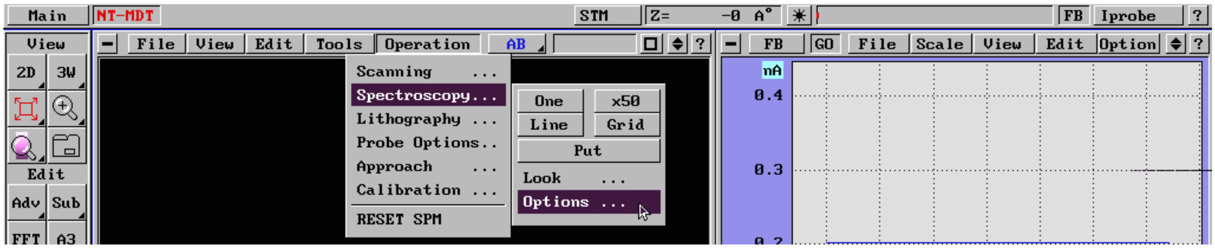


Fig. 89

Note that this parameter can be obtained by two methods: I(z) and Fb(z). However, the latter method is suitable only if the feedback is supported using the "Iprobe" or "Ipr-low" signal. If the feedback is supported using the "Ipr-log" signal, then I(z) mode only is suitable for taking this parameter (Fig. 90).

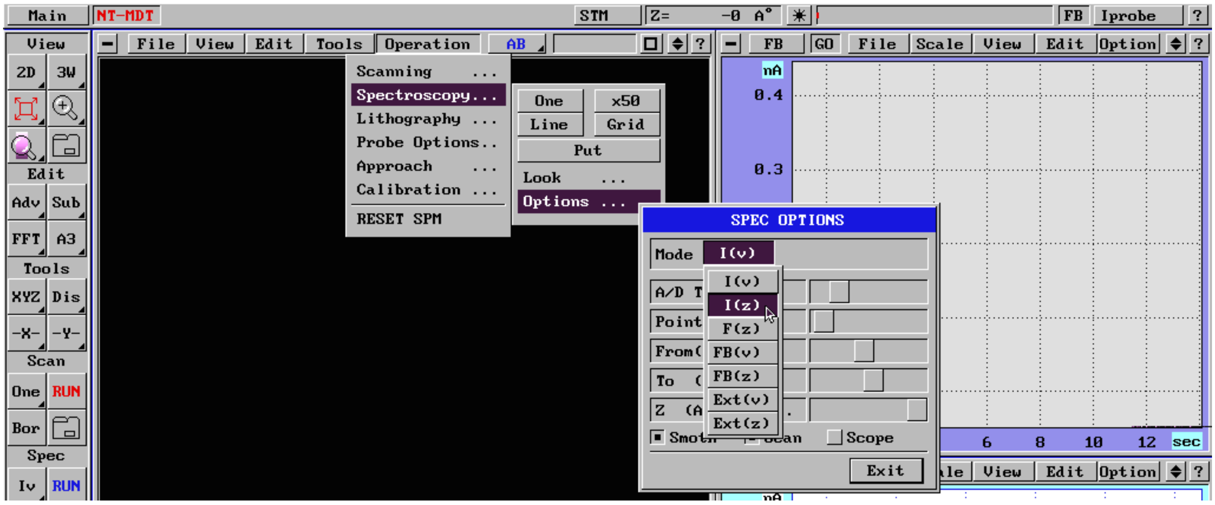


Fig. 90

The other parameters of the spectroscopy have the following meaning:

"A/D Times" - number of measurements in each point of the voltage scanning with subsequent averaging;

"Points" - number of parameter points;

"From(v)", "To(v)" - disabled in this case;

"Z(A)" -move-out of probe from the surface when taking the parameter (in this case not more than 100);

"Smooth" - smoothing of the parameter;

"Scan" - taking the spectroscopy parameter simultaneously with scanning data (topography).

For the first taking of this parameter disable the Scan option and after setting up the required parameters (for example, (Fig. 91): Mode I(z), A/D Times=20, Point=100, Point=100, Z=60A), enter the spectroscopy menu.

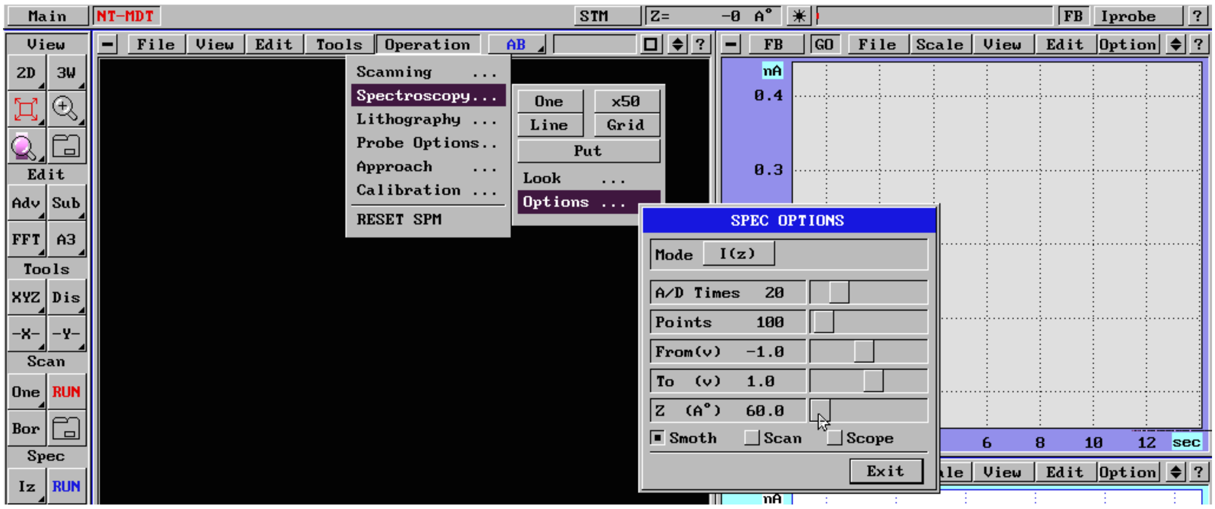


Fig. 91

First let us carry out single scanning of the parameter “ONE” (Fig. 92).

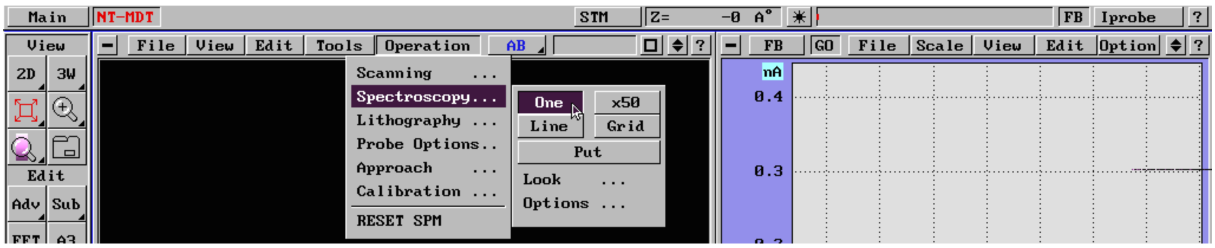


Fig. 92

After the “ONE” button is pressed a marker appears on the scanning field. Place it with the mouse on the required place and press Enter. The I(z) parameter will be displayed in the oscilloscope field as well as the point where it has been taken will be displayed in the scanning field and the marker will remain in the scanning field.

You can take one or more parameters in various points or exit this mode by pressing the right mouse button or the "ESC" key.

The last taken parameter remains in the random-access memory until the next parameter is taken. If you want to leave it to view later or save in a file press the "Put" button (Fig. 93).

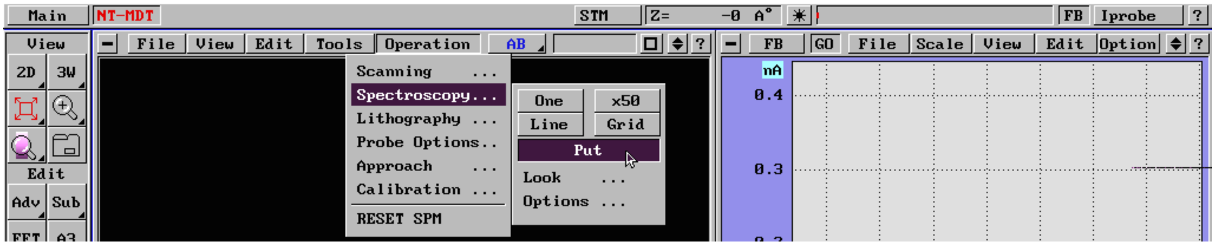


Fig. 93

Other commands of spectrometry execution: "x50", "Line", "Grid" create three-dimensional spectroscopy data where the XZ plane contains the selected parameter and the Y axis contains the parameter number.

When the "x50" command is used one point must be selected, “Line” - two end points of a line, “Grid” - number of points in a grid. The number of parameters taken using the Line command corresponds to the number of points in the scan image along the selected line.

You can view the spectroscopy results using the "Look" menu (Fig. 94) (it can be pulled down in the SPM window).

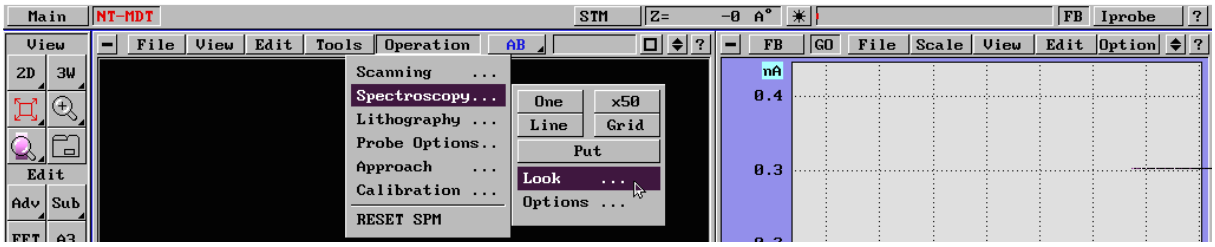


Fig. 94

The "View" command displays in the OSC window one of the spectrometry parameters, the “All” command displays all the parameters one by one. Using the commands "<<" and ">>" you can view other spectrometry data for this picture (out of those who have been saved earlier using the “Put” command) and the "Scan" and "Spec" commands display in the SPM window either the scanning or spectrometry data (Fig. 95).

Fig. 95

3.1.5.6 Modulation methods of the STM mode.

¶I/¶Z

∂I/∂Z - local potential barrier height for electrons (exit behavior) (local barrier height).

In order to measure ∂I/∂Z first take topography and then select the scanning modes (step, rate, Set Point, Bias Voltage, FB Gain) to obtain good topography (see item 3.1.5.1).

Set one of the oscilloscopes for the input "→A" (Fig. 96) measurement and turn the measurement on (Fig. 97).

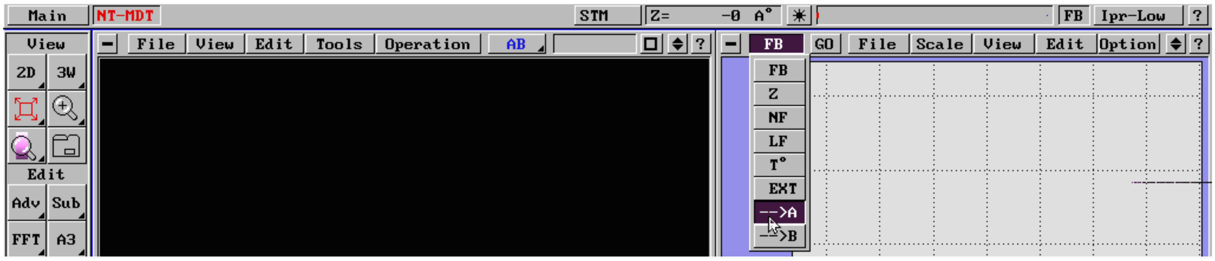


Fig. 96

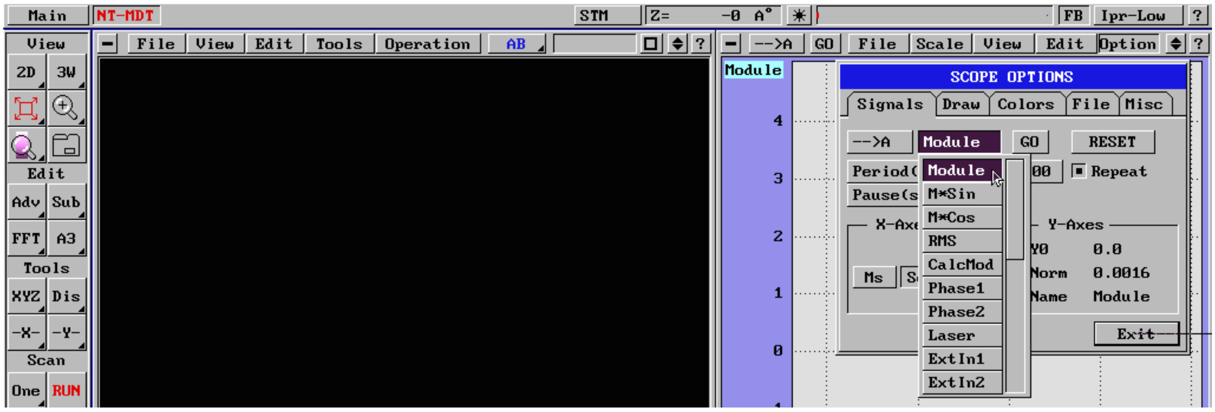


Fig. 97

Then set up the modulation parameters:
Operation → Scanning → Modul (Fig. 98)
Frequency: Main = 21 êÃö (Fig. 98);
Hi/Low - Low (Fig. 98);
SD Input - I probe (Fig. 98);
Gain - 10-20 (Fig. 98);
Amplitude - 0.5 (Fig. 98);
Scan - Module (Fig. 98).

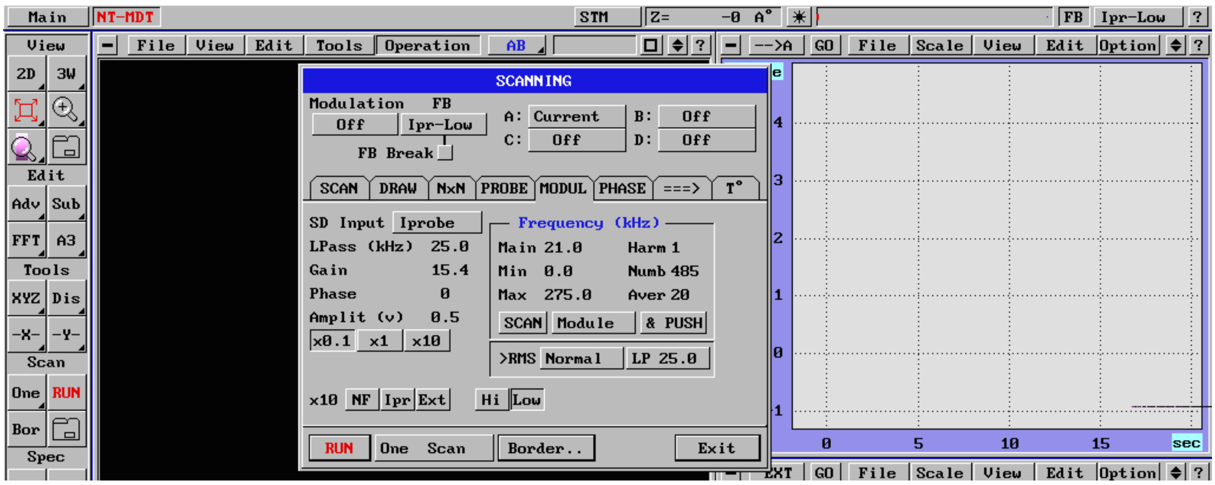


Fig. 98

Turn the scanner modulation on:
Modulation - Z drive (Fig. 99).

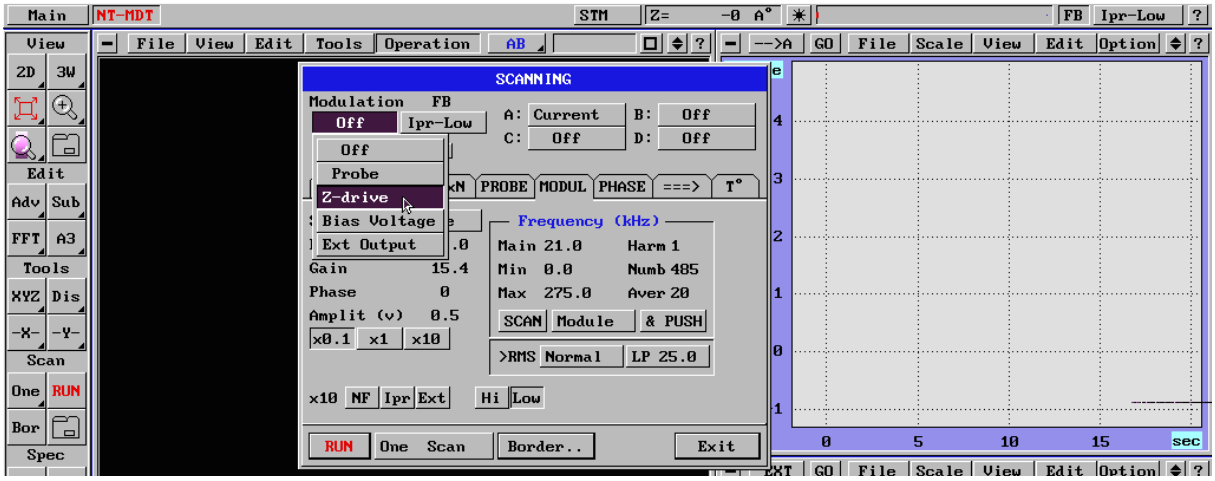


Fig. 99

A non-zero signal may be displayed by the oscilloscope (Fig. 100) (for example, 1 nA).

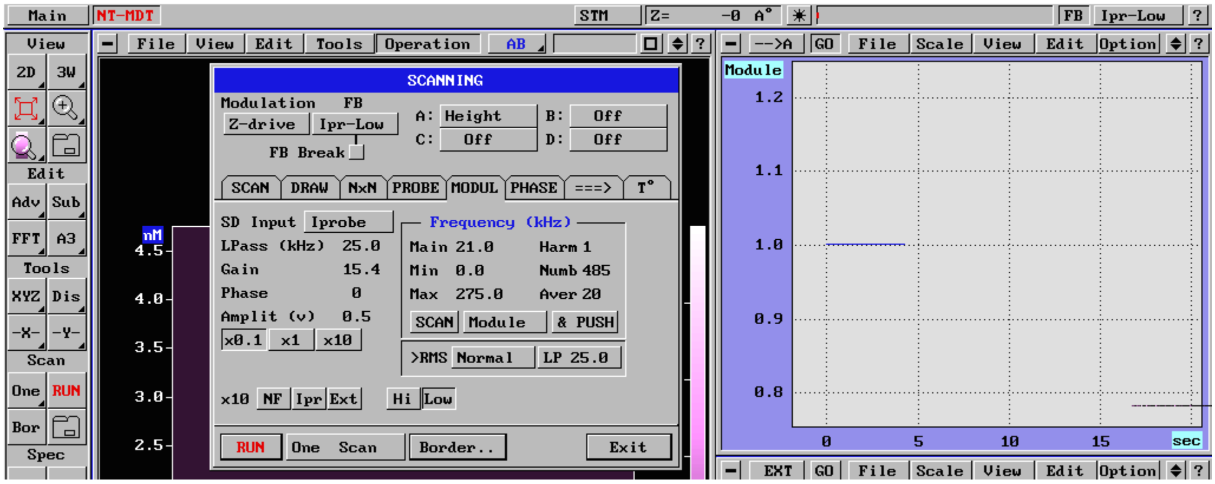


Fig. 100

Use the “Amplit” parameter to tune a linear range (when the amplitude is changed two times the signal on the oscilloscope must also change two times). Also note the change in the signal noise when the "Gain" parameters are changed (if the signal is small the gain of the Ipr channel may be enabled (Fig. 101) the ("x10 Ipr") coefficients of the amplitude multiplication "x0.1", "x1", "x10" do not apply in case of scanner modulation.

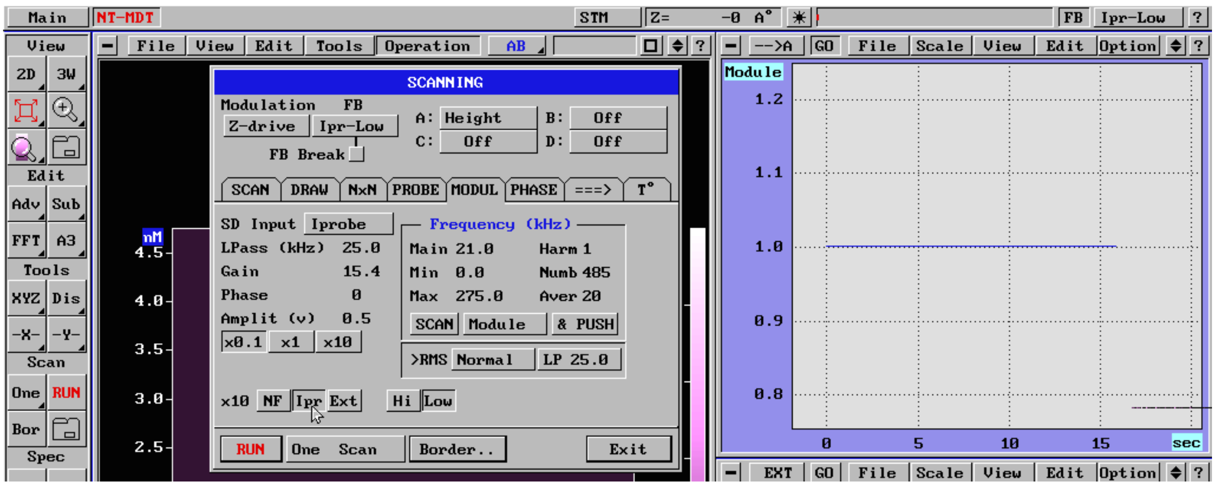


Fig. 101

It is not recommended to increase the signal over 30 nA (in this case disable "x10 Ipr" or decrease “Gain”).

After selecting the parameters go to the Scan label. In order to register in the “B” window select the “Module” signal, scanning direction "→" (Fig. 102), set the SPM window splitting to register both signals (in upper part “AB” (Fig. 103), in the lower part "<>" (Fig. 104) and run the scanning (“**RUN**”) (Fig. 105).

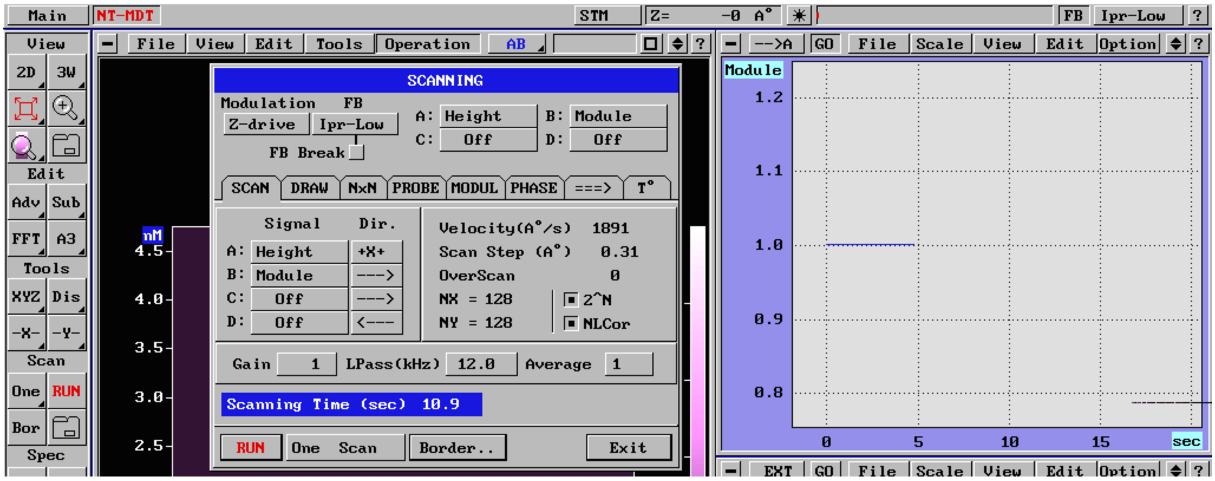


Fig. 102

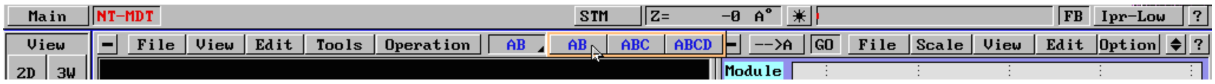


Fig. 103

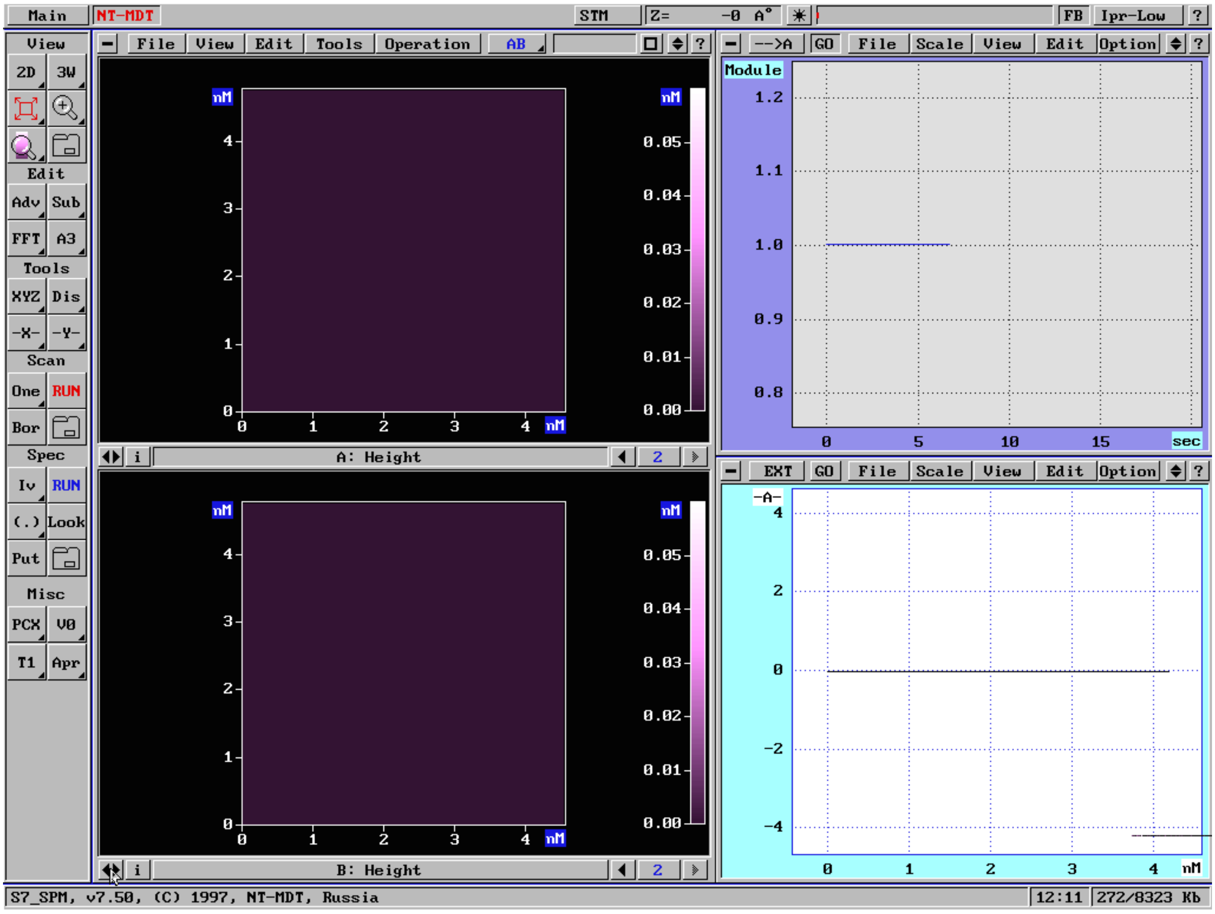


Fig. 104

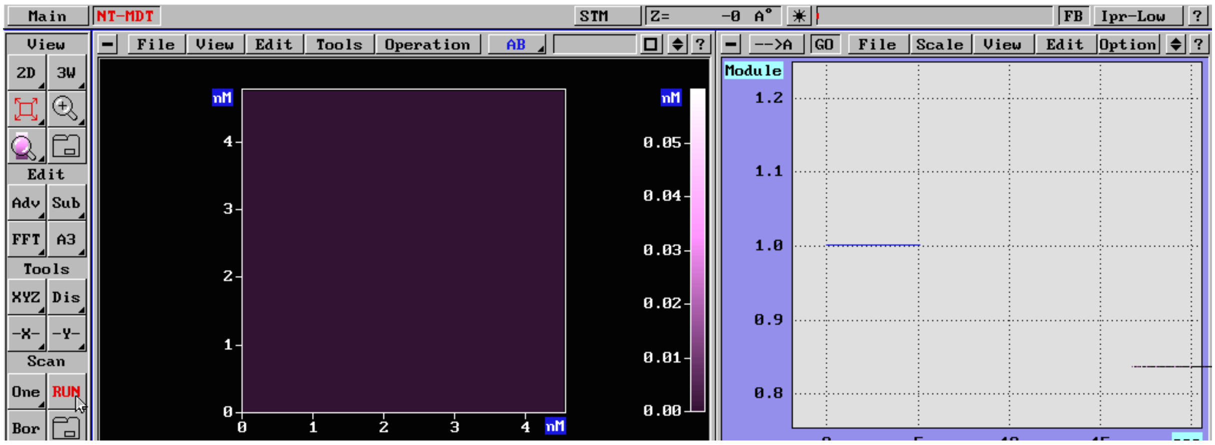


Fig. 105

Note that the ∂I/∂Z measurement data are determined not only by the local height of a potential barrier for electrons but also by the local hardness.

¶I/¶U

∂I/∂U - local spectral states density for electrons.

In order to measure ∂I/∂Z first take topography and then select the scanning modes (step, rate, Set Point, Bias Voltage, FB Gain) to obtain good topography (see item 3.1.5.1).

Set one of the oscilloscopes for the input "→A" (Fig. 106) measurement and turn the measurement on (Fig. 107).

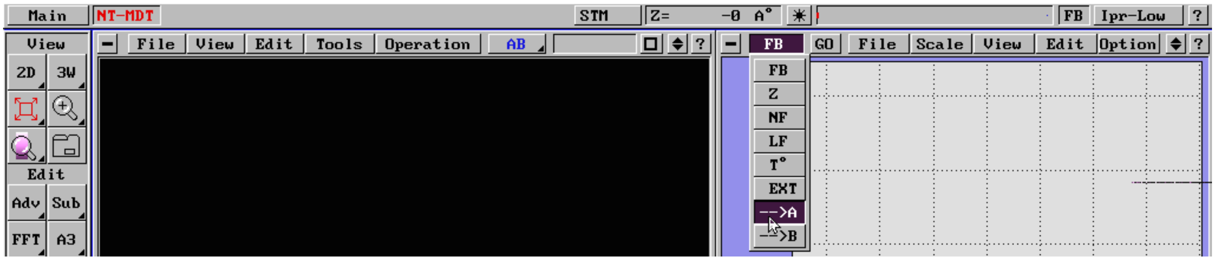


Fig. 106

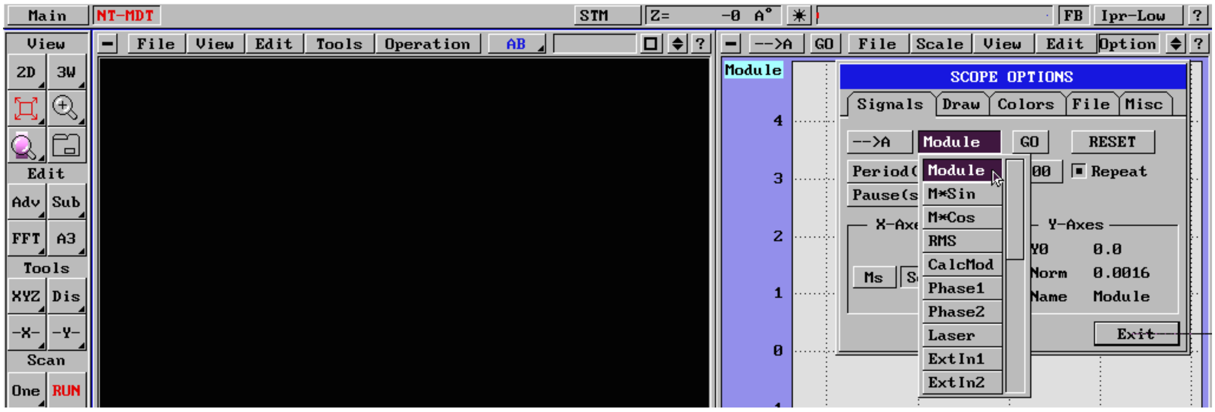


Fig. 107

Then set up the modulation parameters:
Operation → Scanning → Modul (Fig. 108)
Frequency: Main = 21 êÃö (Fig. 108);
Hi/Low - Low (Fig. 108);
SD Input - I probe (Fig. 108);
Gain - 10-20 (Fig. 108);
Amplitude - 0.5 (Fig. 108);
Scan - Module (Fig. 108).

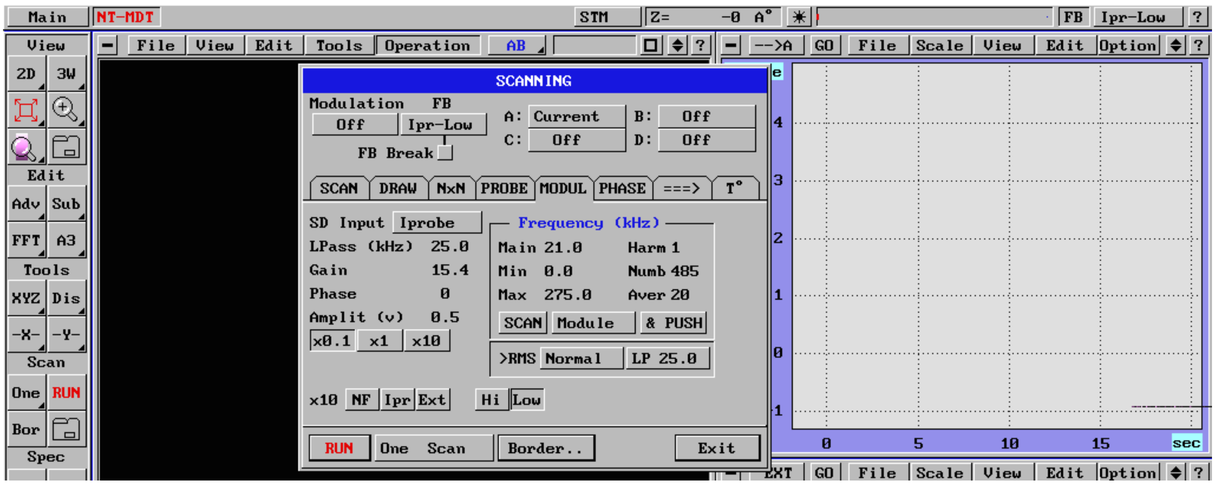


Fig. 108

Turn on the bias voltage modulation:
Modulation - Bias Voltage (Fig. 109).

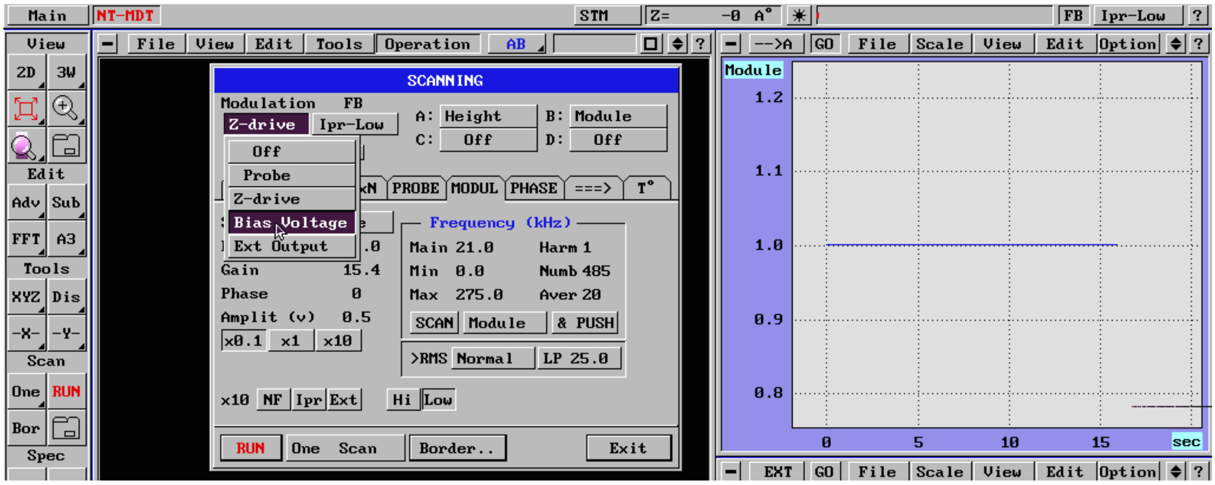


Fig. 109

A non-zero signal must be displayed by the oscilloscope (Fig. 110) (for example, 1 nA).

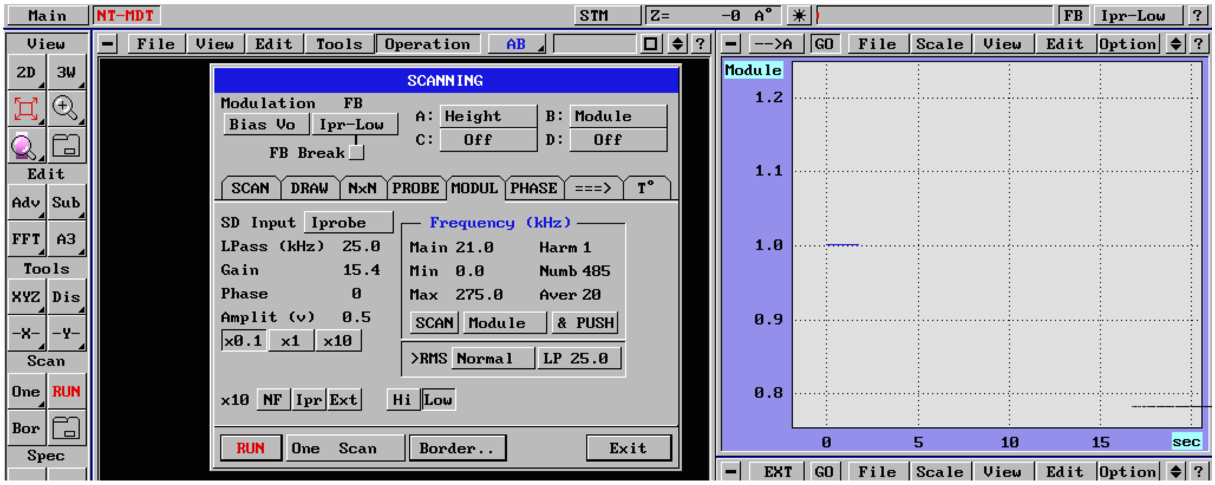


Fig. 110

Use the “Amplit” parameter to tune a linear range (when the amplitude is changed two times the signal on the oscilloscope must also change two times). Also note the change in the signal noise when the "Gain" parameters are changed (if the signal is small the gain of the "Ipr" channel may be enabled (Fig. 111) the ("x10 Ipr") coefficients of the amplitude multiplication "x0.1", "x1", "x10" do not apply in case of voltage modulation.

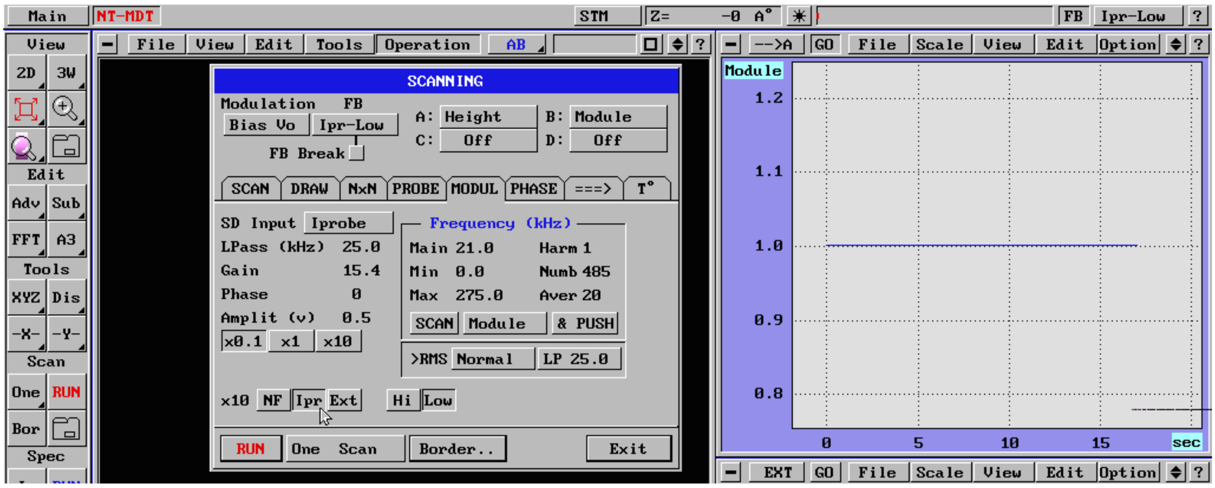


Fig. 111

It is not recommended to increase the signal over 30 nA (in this case disable "x10 Ipr" or decrease “Gain”).

After selecting the parameters go to the Scan label. In order to register in the “B” window select the “Module” signal, scanning direction "→" (Fig. 112), set the SPM window splitting to register both signals (in upper part “AB” (Fig. 113), in the lower part “<>“ (Fig. 114) and run the scanning (“**RUN**”) (Fig. 115).

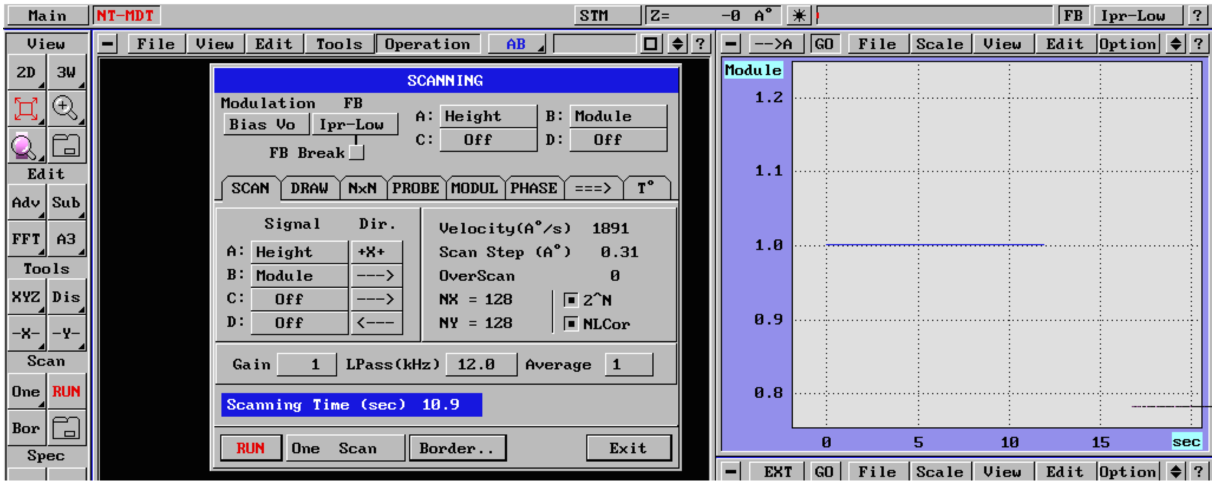


Fig. 112

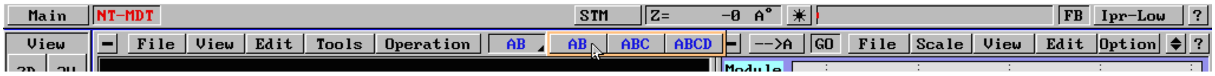


Fig. 113

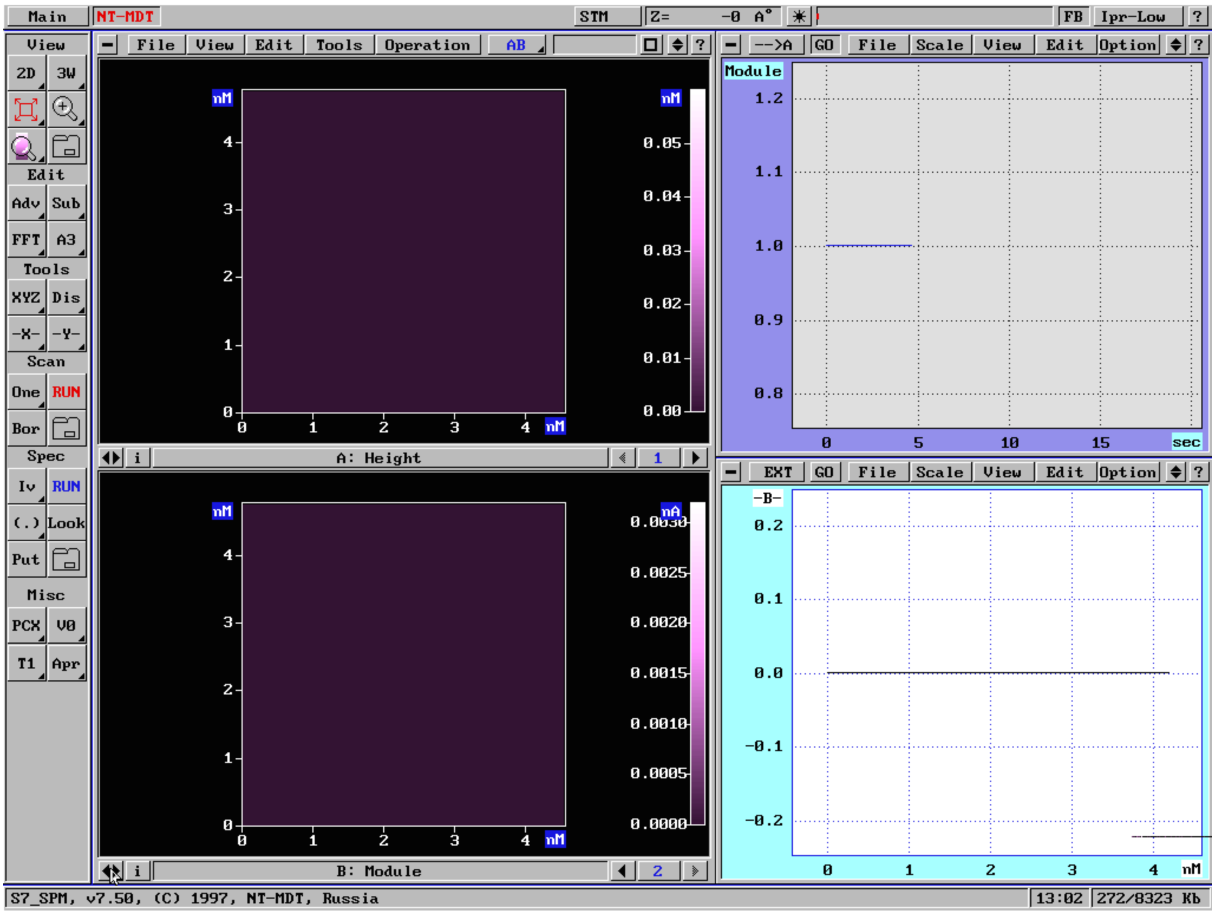


Fig. 114

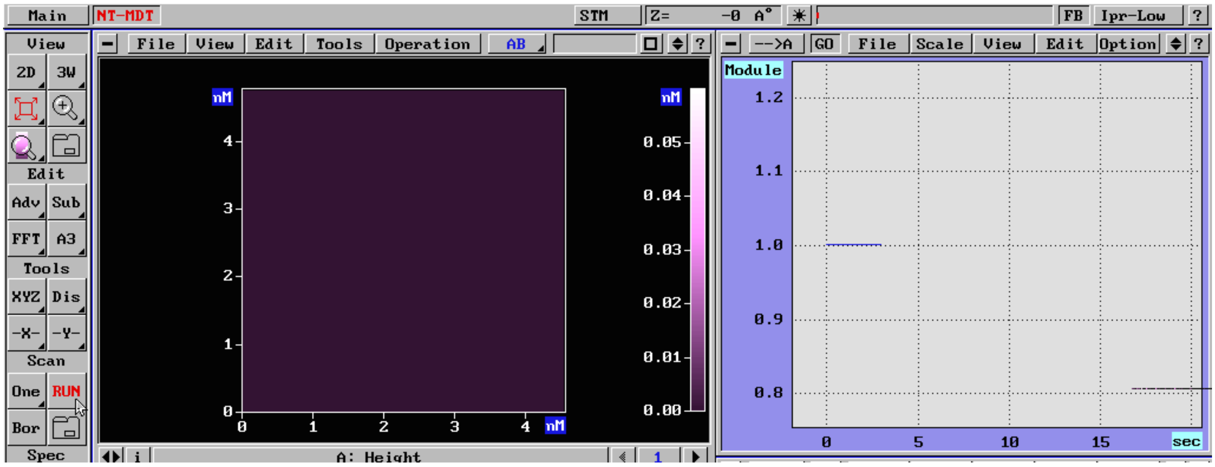


Fig. 115

This picture shows the states density at one of the energy levels as determined by the applied voltage (Bias Voltage). A rather comprehensive picture can be obtained with a series of such scans at various tunnel voltages.

3.1.6 Checking the tip quality

Though the shape of the tip point in tunneling microscopy is casual, exponential dependance of the tunneling current It on the distance z

$$It = f(u) * \exp\left(-\left(A\sqrt{\left(j-\frac{u}{2}\right)} * z\right)\right)$$

makes it possible to obtain similar results in quantitative terms using different tips. The quality of the microscope operation and the maximum resolution depend on the tip's quality.

To control the tip's quality it is recommended to find out the I(Z) dependance. To do this first it is necessary to select an area on the sample with a low level of tunneling current noise.

The area selection is done by means of the "Ctrl"+"Border" operation (select in the "Operation" menu item "Scanning" Fig. 116 and press the left mouse button, in the opened menu select the 'Border' button and in the pulled-down menu select "Absolute" Fig. 117). The designation of the area available for scanning and the frame inside of which scanning will be done will displayed (Fig. 118). When moving the frame in this mode in reality you are moving the tip located in the center of this frame.

Controlling the noise level through the oscilloscope you can find a place with a low noise level.

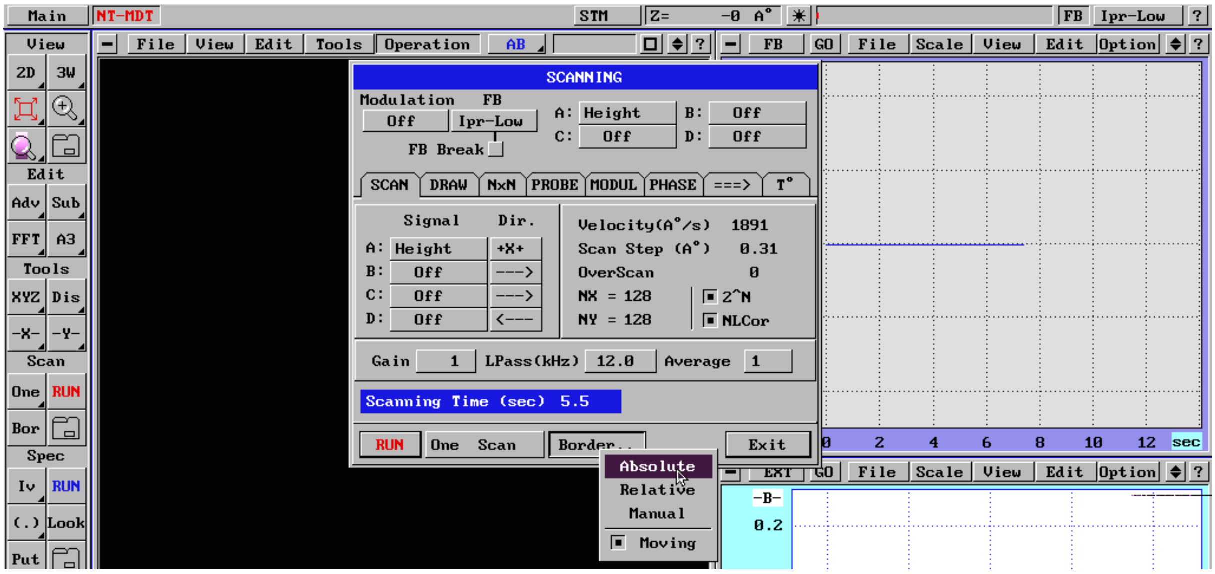


Fig. 116

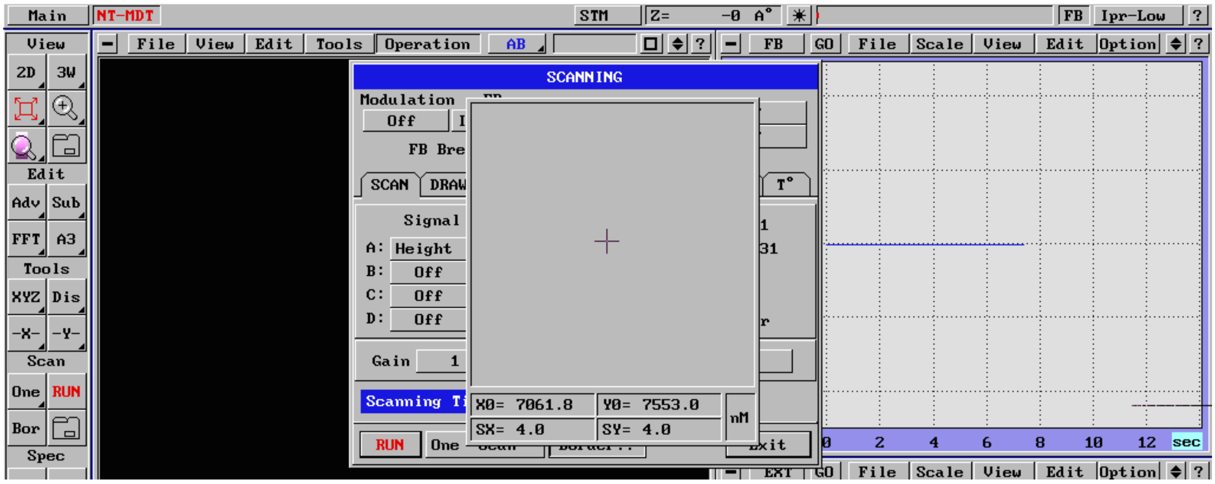


Fig. 117

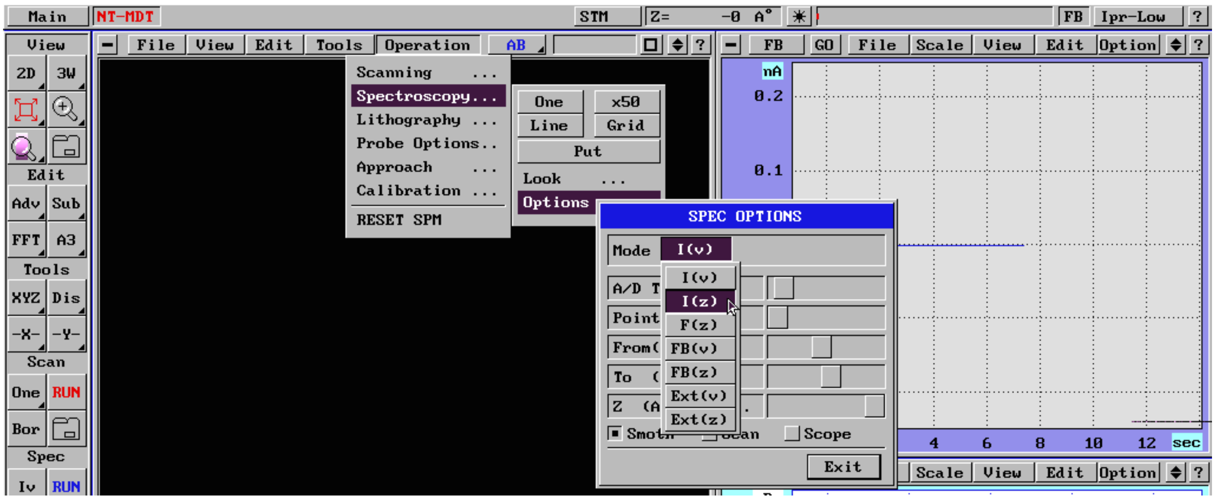


Fig. 118

After it the following must be inserted:
“Operation” - “Spectroscopy” - “Options” - Mode I(z), Z(A)=60 (Fig. 119, Fig. 120)

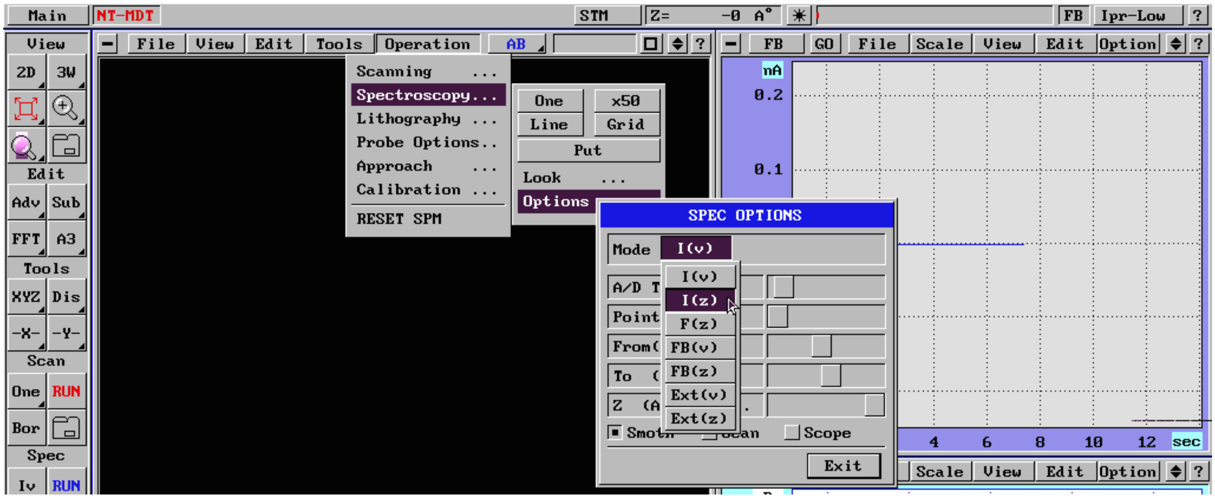


Fig. 119

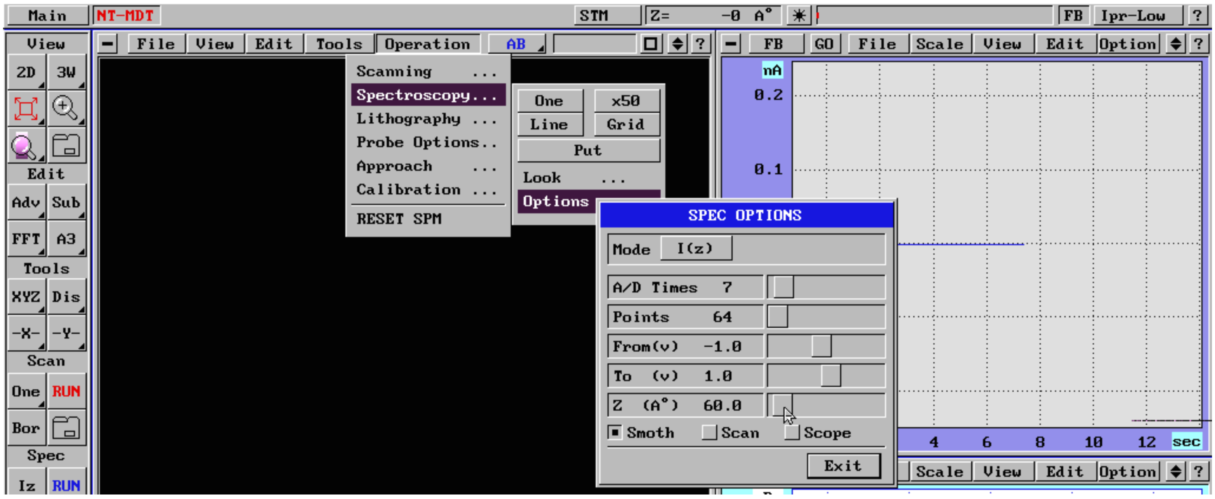


Fig. 120

and do single spectroscopy (Fig. 121).

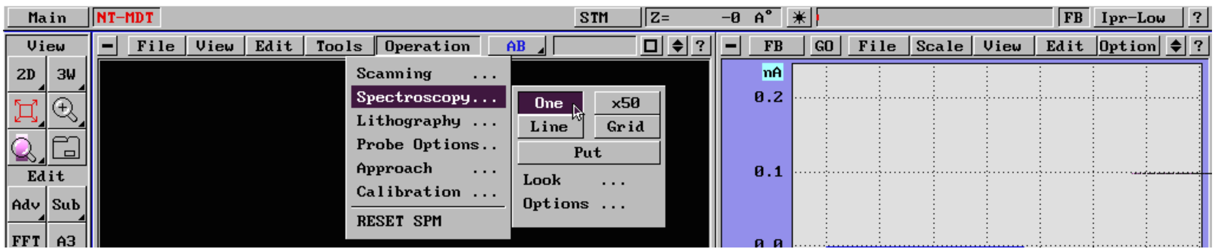


Fig. 121

In the oscilloscope window we have a graphic representation of the I(Z) dependence. An example of the dependance for a 'very good' and 'satisfactory' tip (Fig. 122, Fig. 123).

If the It drop to one-half takes place with Z < 3 A the tip is considered to be very good (Fig. 122), if with Z < 10 A (Fig. 123), then using this tip it is possible to have an atomic resolution on HOPG. If this takes place with Z > 20 A this tip should not be used and must be replaced.

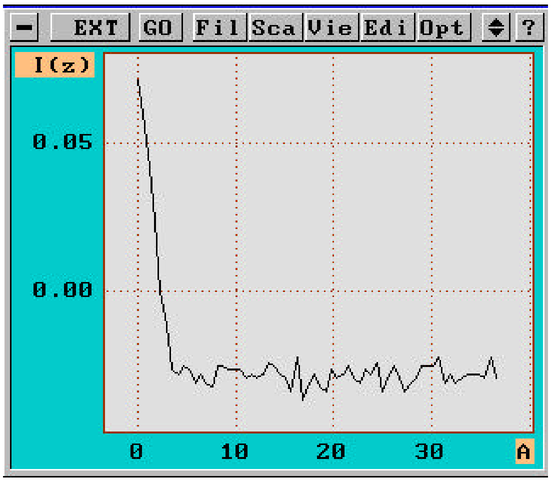


Fig. 122

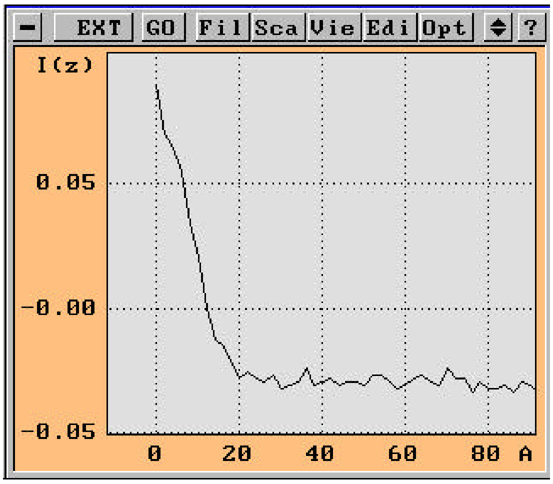


Fig. 123

3.1.7 Evaluation of the tip's stability over time

Evaluation of the tip's stability over time is done via tunneling current control at the oscilloscope. If the tunneling current noise level is 3-4 times as large as the noise level observed with the retracted tip despite the scanning area, it means that the tip is either dirty or there is a 'burr' on the tip point. The tip can be considered good if when scanning with currents less than 1 nA the noise level is as high as with the tip moved away. Tips made of a platinum group metal are very stable over time. An experienced operator can work with one tip over two weeks on some samples. When the tip becomes dirty it is enough to fire it in an alcohol flame. Tips made of tungsten should be replaced once per day.

3.1.8 Shutdown procedure

Open the microscope control menu - “Operation” → "Approach" → "Mover" (Fig. 124) and press "Remote" on 4 mm (Fig. 125).

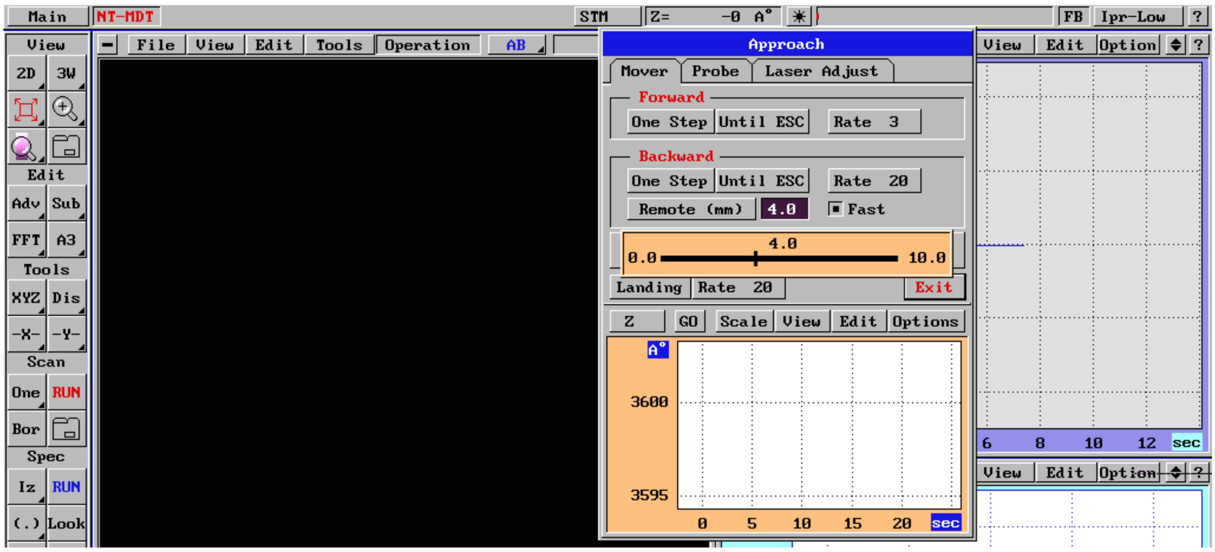


Fig. 124

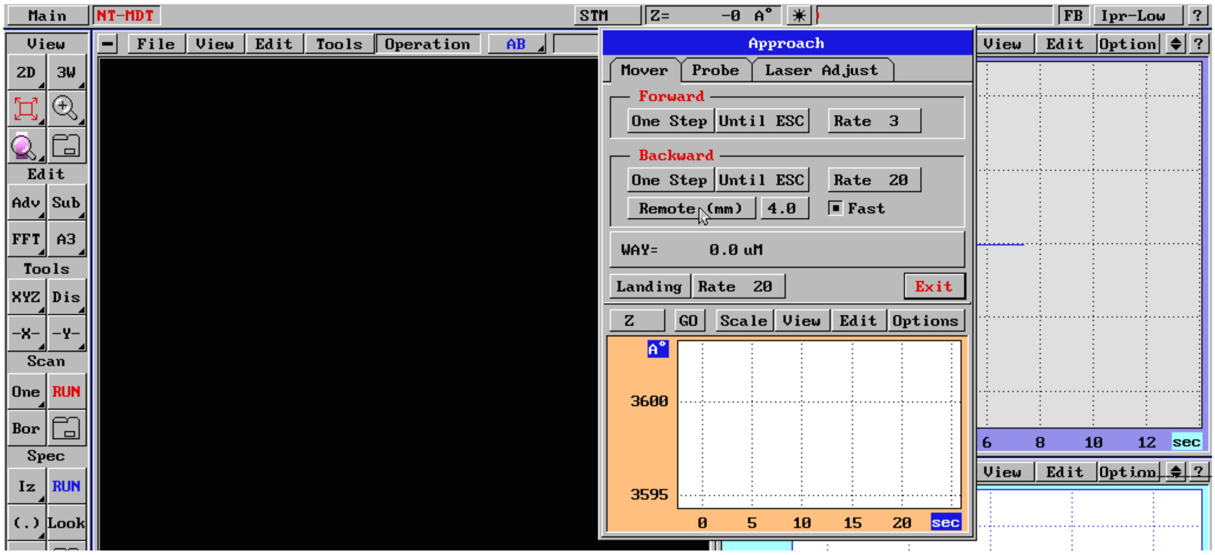


Fig. 125

WARNING:
Be especially careful when executing the "Forward" + "Until ESC" command. Don't use unless absolutely necessary.

In the "Probe options" menu disable the feedback (Fig. 126). Lift the scan unit and install retainers on it.

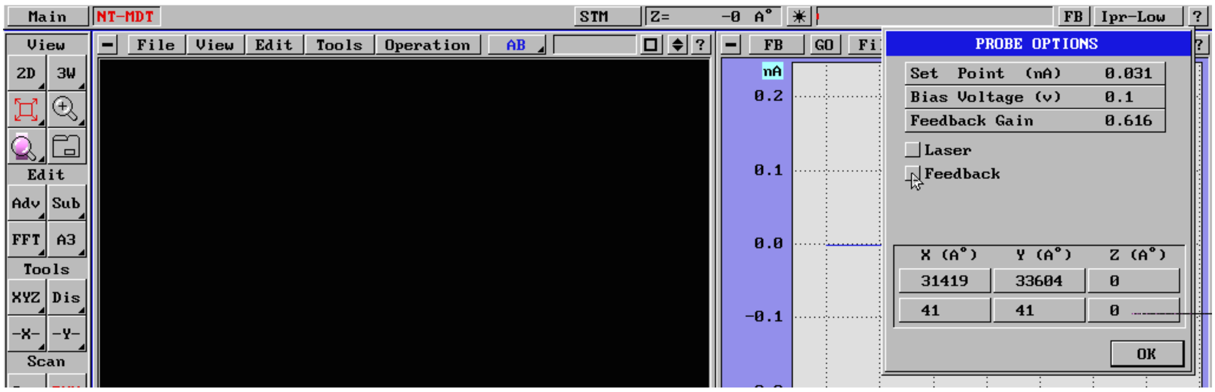


Fig. 126

Then:

- Remove the STM head.
- Carefully take the sample from the site on the piezoscanner avoiding any considerable pressure on the sample holder.
- Turn off the power supply of the microscope using the power switch on the unit.
- Exit the control program by pressing the Exit button in the Main button menu in the left corner of the screen (Fig. 127) or using the F10 functional key (after closing all the opened menus using the "ESC" key, otherwise the F10 is not activated).

Fig. 127

3.2 SFM-mode

The contact scanning force microscopy mode is designed for investigation of sufficiently hard surfaces, both conductive and dielectric. For instance, metals, semiconductors, crystal structures.

This mode feature sufficiently simple setting of operating parameters, high (up to atomic) resolution. When using this mode in addition to surface topography it is possible to obtain data on the difference of friction coefficients on various areas of the investigated surface as well as on the distribution of local hardness and softness. When using conductive cantilevers it is also possible to measure in parallel with topography various electrical parameters of the surface and if the cantilever is sufficiently hard you can switch over to the STM mode and investigate the same surface using two methods.

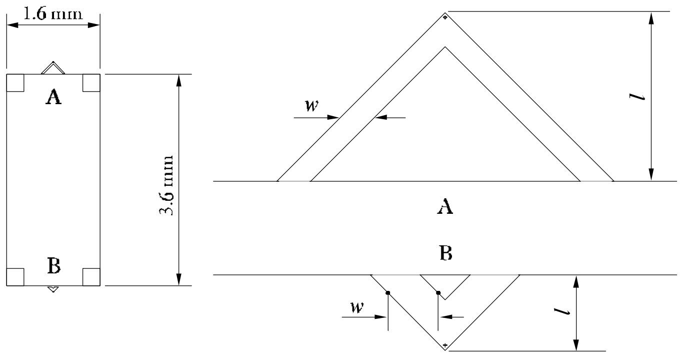
The use of this mode is limited by high attraction force between the probe and the sample which makes it impossible to investigate soft samples such as many types of polymers and most biological objects.

3.2.1 Choosing a cantilever

The cantilever choice depends on the task and on the sample to be studied. To get surface topography of a rigid sample, use fairly short triangular cantilevers that permit measurement of true topography and not distortion of lateral force influence. Shorter cantilevers do not suffer from a sort of self oscillation connected with the lateral force induced cantilever bending during feedback operation. On the other hand, if the map of friction distribution over the sample surface is being strived for, using either a narrow rectangular cantilever or long triangular one is preferred. Longer cantilevers with small spring constants are also useful for soft samples, to exert only a small force even when feedback fails to trace some surface features. Nevertheless, the least possible force between tip and sample is determined by their properties and tip curvature radius rather than by cantilever spring constant. On the air tip-sample interaction force less than about 10^{-9} N can hardly be achieved.

For local elasticity and local viscosity measurements, cantilever resonant frequency should remarkably exceed the modulation frequency.

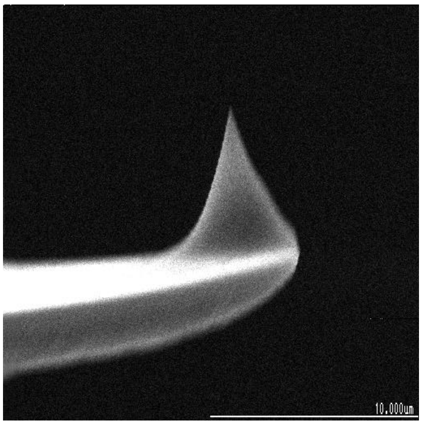
As an example, parameters of one of the types of silicon cantilevers produced by NT-MDT are presented below.

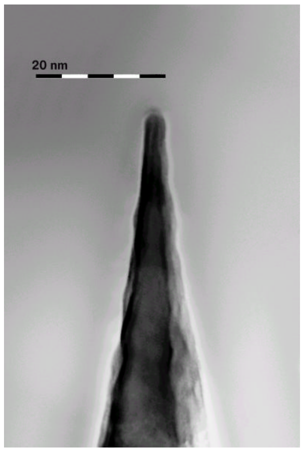


Cantilevers type	A			B		
	Min	Typical	Max	Min	Typical	Max
Cantilevers length, *l*, µm		200			90	
Cantilevers width, *w*, µm		40			60	
*Cantilevers thickness, µm	1.6	1.9	2.2	1.6	1.9	2.2
*Resonant frequency, kHz	40	50	60	300	350	400
*Force constant, N/m	1.8	3.0	4.2	24.0	30.0	36.0

Tip parameters:

- radius the radius of curvature less than 10 nm
- tip height 7 um
- full cone angle near the apex 22°
- tip is conductive





3.2.2 Mounting a cantilever

The cantilever is installed in the holder with fine sharp forceps. Note that cantilevers are placed in the box with their tips up (Fig. 128), they should be inserted into the holder with their tips facing the holding spring (Fig. 129).

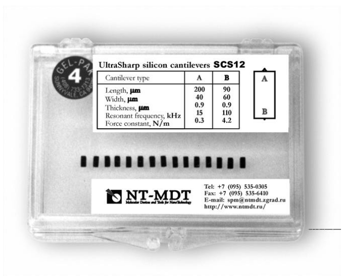


Fig. 128

Fig. 129

3.2.3 Preparing and mounting a sample

Very smooth and flat surfaces should be used as substrates for SFM research of nanometer-size objects. Polished silicon, fused quartz, or layered materials with atomically flat surfaces - graphite, mica - meet this requirement.

The most appropriate lateral size of a sample should not exceed about 10x15 mm^2, otherwise it should be mounted with additional plate interposed between Sample and mounting plate. Take a clean mounting plate from the set and a piece of double-stick tape that slightly exceeds the sample size. Try to stick the piece of tape over the plate gradually from one edge to another to ensure absence of air bubbles. Then put the sample on the tape and press it's edges to stick the sample to the tape. If the sample is very flexible (like a film), also try to put it gradually from one edge to another to have no air bubbles in between. Bubbles can cause long-term drift of the sample.

Don`t touch the surface to be studied. However if you want to study fresh surface of a layered material, its top layers can be cleaved with a thin blade or with a stick tape. Insert mounting plate into the sample holder on the top of piezoscanner (Fig. 131, Fig. 130). It should easily slide over the three small steel balls, being pressed against them with two spring clamps. Do not exert a large force onto the sample holder: it can break the piezotube.

Verify that the mounting plate lies on all three balls.

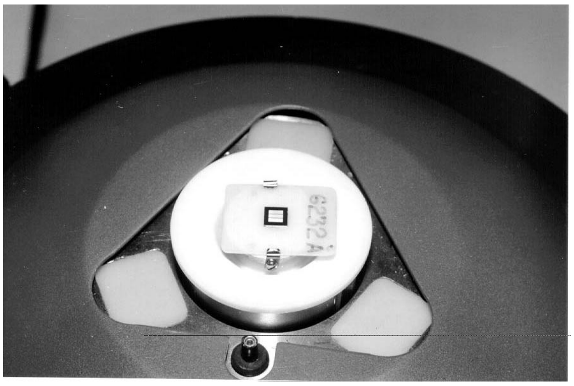


Fig. 130

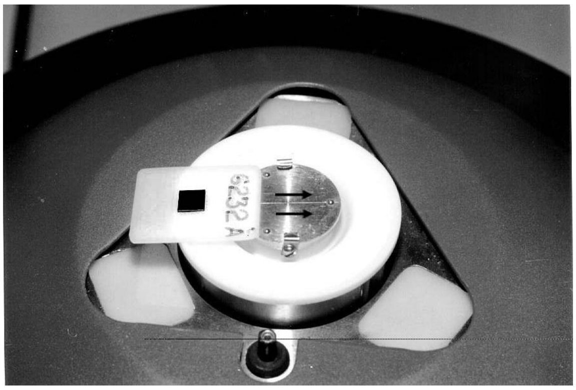


Fig. 131

3.2.4 Adjusting SFM head

3.2.4.1 Basic elements of the SFM head (Fig. 132, Fig. 133):

1. Adjusting screw for movable insert
2. Adjusting screw for movable insert
3. Spring plunger
4. Clamp bar
5. Catch of the clamp bar
6. Adjusting screw of the quadrant photodiode (normal direction)
7. Adjusting screw of the quadrant photodiode (lateral direction)
8. Laser
9. Quadrant (4-sectional) photodiode
10. Connector (covered with cap) for Resonant mode SFM movable insert and for STM movable insert
11. Screw for adjustment of the head slope
12. Fixing nut of the screw 11
13. Support plate (polycrystalline sapphire)
14. Head protective cover

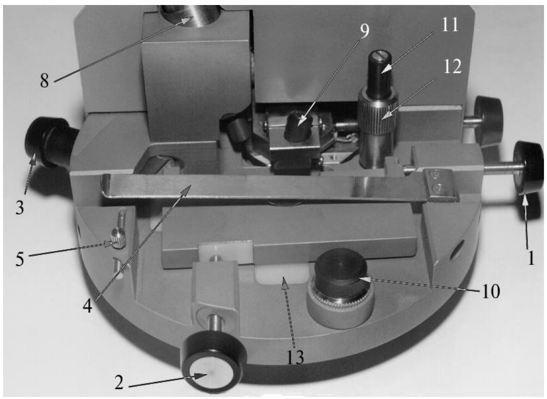


Fig. 132

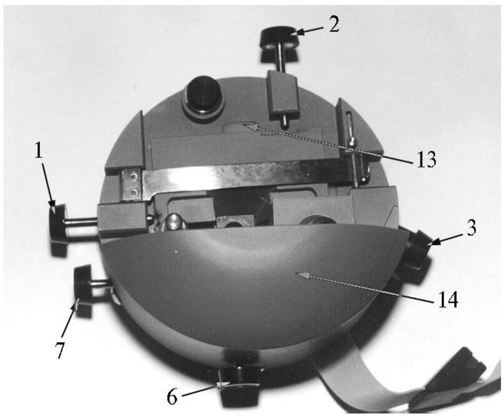


Fig. 133

Put the SFM-head on a level surface over a sheet of a white paper. Loosen the screws (1,2) and open the clamp bar (3) (Fig. 132, Fig. 133). Put the SFM movable insert with mounted cantilever (see section 3.2.2) (Fig. 135) into SFM head (Fig. 134). Three steel balls of the movable insert should stand on three support plates of SFM head. Fasten the adjusting screws (1, 2) to touch the movable insert and correct the insert position to touch screws with the V-shaped groove and flat plate correspondingly. Then rotate the knob (3) (Fig. 132, Fig. 133) to let spring plunger push the insert. Put the clamp bar (4) on the insert but do not fix it with the catch (5).

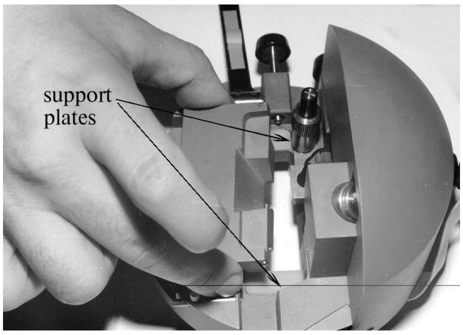


Fig. 134

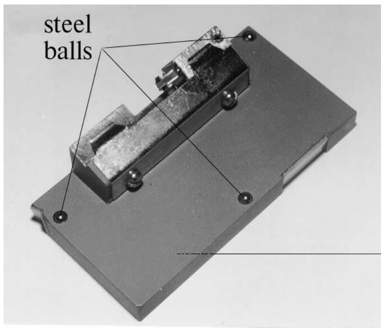


Fig. 135

Turn on power switch («Power») on the front panel of SPM (Fig. 23).

Start SPM control program S**7_SPM.EXE**.

In the left top corner of the screen (Fig. 136) point the button “MAIN” by the mouse cursor and click - string menu opens. Press “OPTIONS” button, then the command “LOAD”. Choose the file JUSTAFM.OPT with program presets for adjustment procedure (Fig. 137) and click it. You will see three windows: scan window "SPM" and two windows of program oscilloscopes "NF" and "LF" (Fig. 138). Window "NF" shows photodiode differential current corresponding to the position of a reflected laser beam in lateral force direction and its changes caused by cantilever twist. A window "LF" reveals the photodiode differential current corresponding to normal force direction and its changes caused by normal bending of cantilever.

Fig. 136

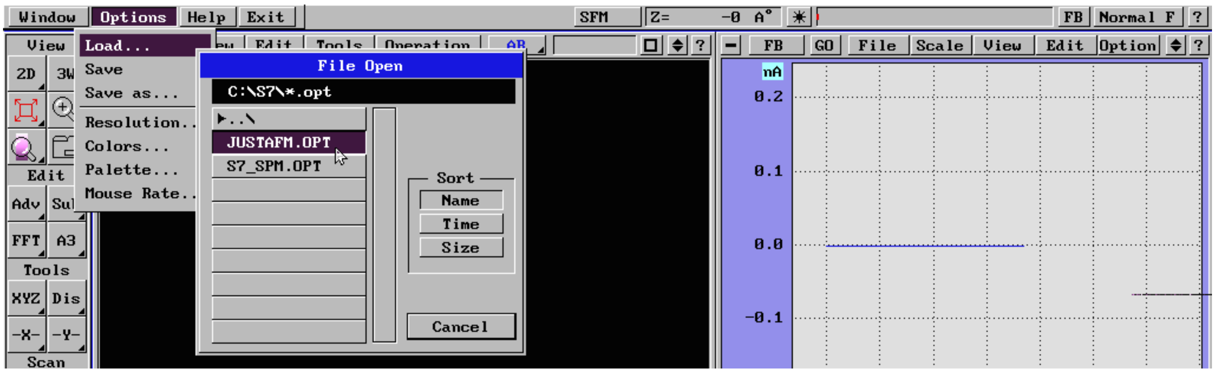


Fig. 137

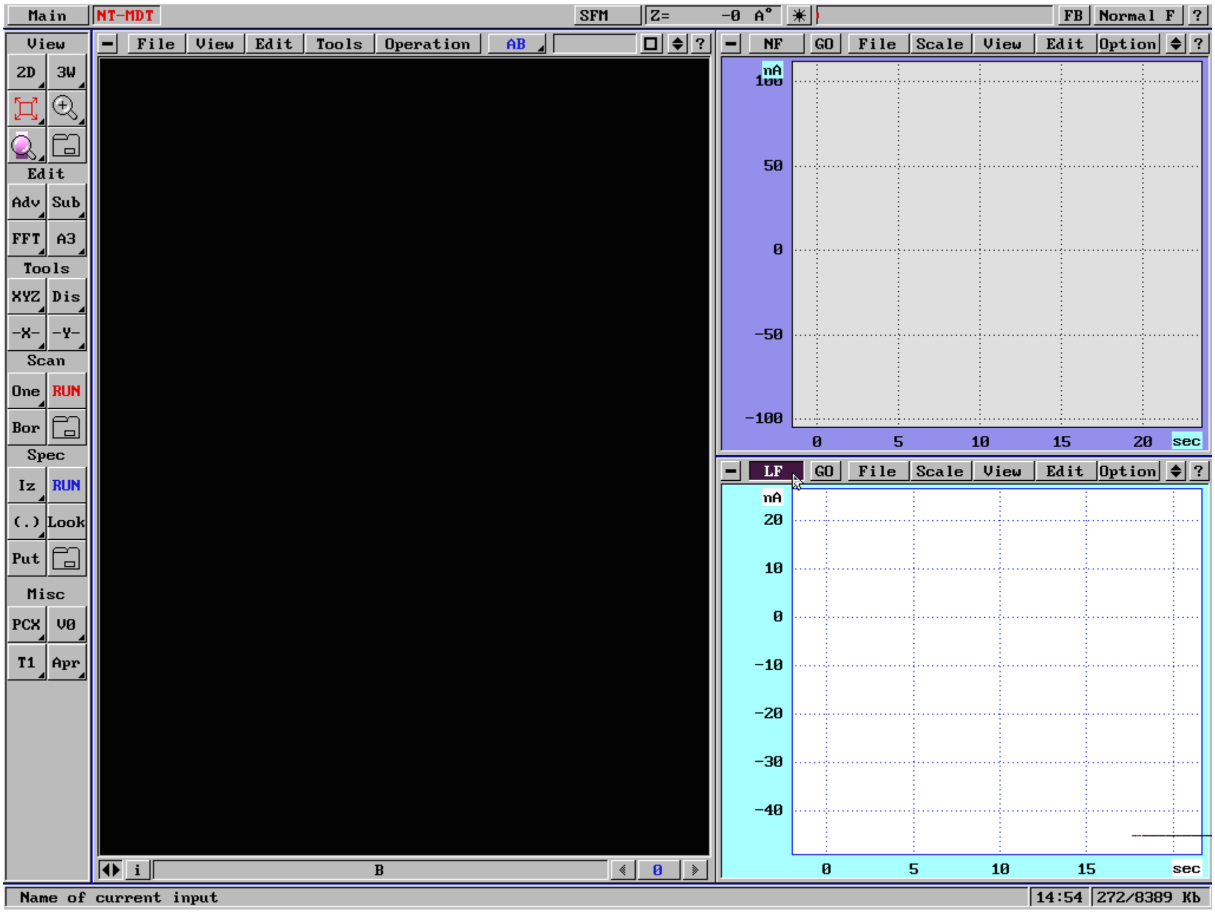


Fig. 138

Click "laser" button in the top right part of the screen to switch on the laser. The button changes its color from grey to red (Fig. 138, Fig. 139), and laser on the SFM heads turns on.

Fig. 139

Press buttons GO in oscilloscope windows "NF" and "LF" to start measuring signals.

Rotate the computer monitor if necessary to see it while adjusting SFM head. Having a long working distance optical microscope or lens can also be useful. Set the optical microscope above the SFM head to see the cantilever beam.

Move the insert by the screws (1, 2) (Fig. 132, Fig. 133) to adjust the cantilever to the focused laser beam. You should see a bright spot on the end of cantilever.

Put a dark mirror under the SFM head (Fig. 140). Adjust photodiode position with respect to the spot of a laser beam reflected from cantilever. The dark mirror can be used to see mutual position of the photodiode and the spot (Fig. 141).

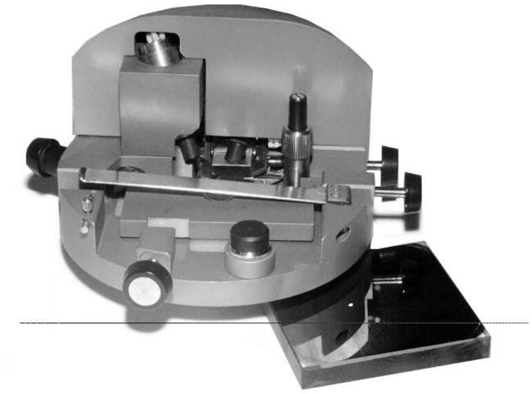


Fig. 140

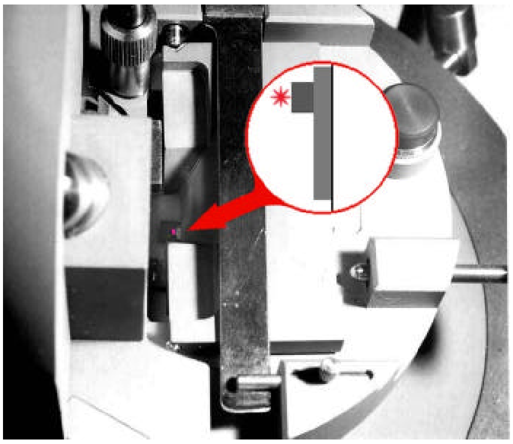


Fig. 141

CAUTION:
Don`t stare into beam!

Signal values in oscilloscope windows "NF" and "LF" will deviate from zero (Fig. 142).

Set some positive values in both windows (Fig. 143) by means of photodiode adjusting screws (6, 7) (Fig. 133). Slightly move cantilever about focused beam by rotating adjusting screw (1) (Fig. 133) to get the maximum signal value in any window (Fig. 144). Then slightly move it along another direction by the screw (2) (Fig. 133) to get maximum signal value (with cantilever with triangular area at its end) again in the same window (Fig. 145).

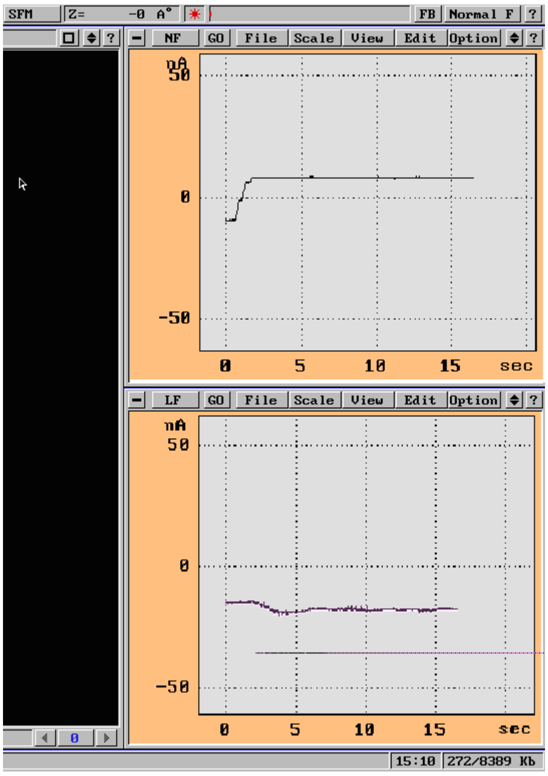


Fig. 142

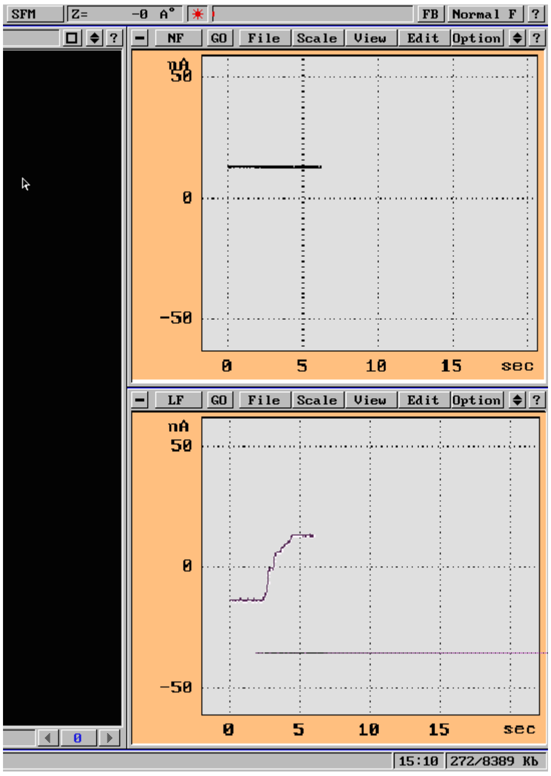


Fig. 143

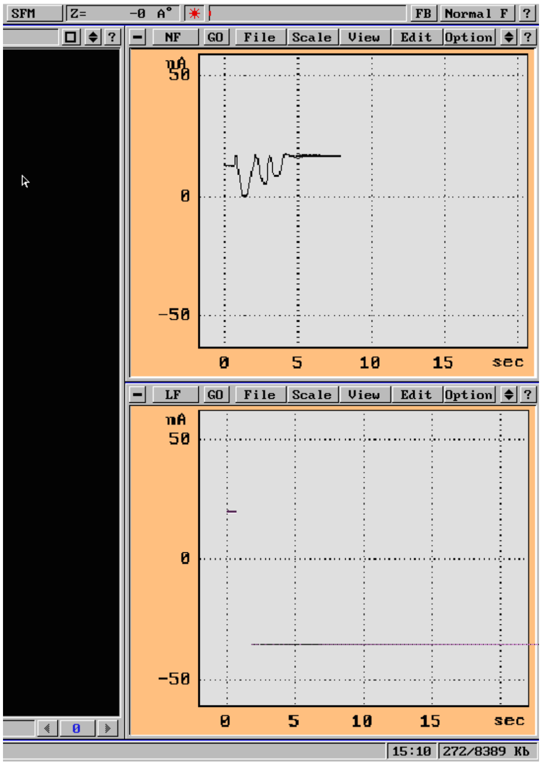


Fig. 144

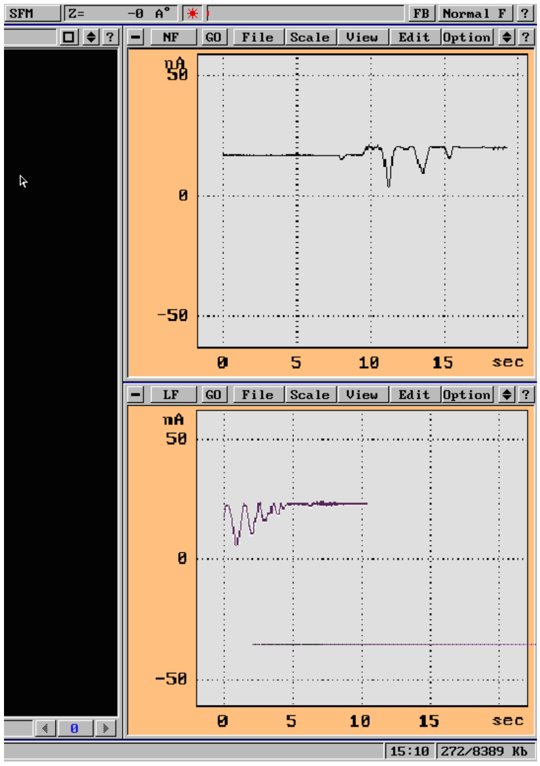


Fig. 145

Repeat the last two operations to be sure that the triangular area at the end of cantilever is well adjusted to the laser spot.

To check the results, lift the SFM head a little and look at the table. On a sheet of a white paper you should see a characteristic diffraction pattern from the cantilever (Fig. 146) around the spot of the laser beam.

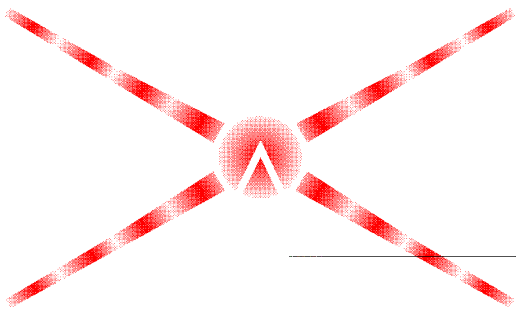


Fig. 146

Put the head back on a table. Press the movable insert with the clamp bar (4) and fix it with the catch (5) (Fig. 133).

It could also be useful to withdraw the adjusting elements from the movable insert to further diminish the thermal drift when you want to study atomic-scale objects. Rotate the knob (3) clock-wise to pull the spring plunger back. Then withdraw the adjusting screws (1,2) from the insert. Look at the diffraction pattern again to be sure that the insert has not moved accidentally.

Photodiode adjusting screws (6,7) (Fig. 133) are used to align it with the reflected beam.

Rotate the screw (7) to set signal value in the window “NF” equal to zero (Fig. 147). Check the signal value by slight rotation of the screw for indication of positive and negative values: it shows that the laser beam is approximately within the middle photodiode quadrant.

Rotate the screw (6) to set signal value in the window “LF” equal to zero (Fig. 148).Check the signal value by slight rotation of the screw for indication of positive and negative values. When both “NF” and “LF” signals are equal to zero and adjustments produce positive and negative values, the spot is in the center of photodiode. Now set some negative signal value in the window “LF” by the screw (6). The value should be a few times less than the saturation signal. So if you can reach maximum values +30nA -30nA in the window “LF” by screw adjustment (6), than set it to about a few nA.

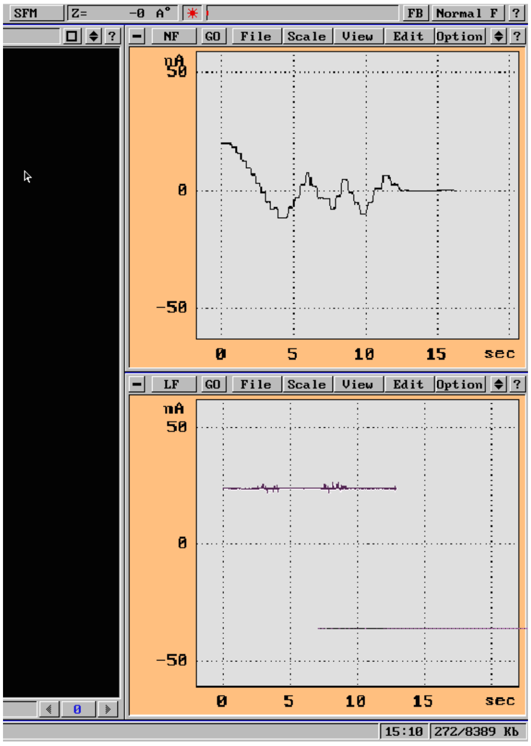


Fig. 147

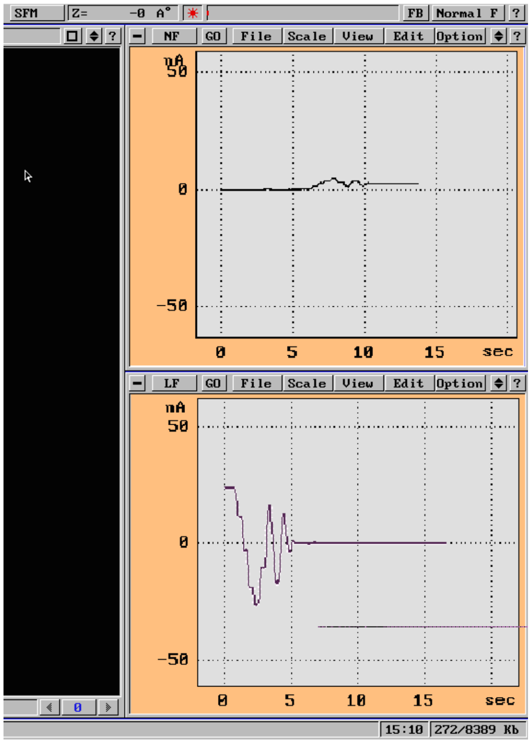


Fig. 148

ATTENTION:
Signal value in the window “LF” should always be adjusted to zero. Signal value in the window “NF”, in case of work in a contact mode, before landing is shifted by the screw 6 (Fig. 133) into NEGATIVE area. The magnitude “NF” is proportional to a value of a repulsive force between a probe and surface of a sample and depends both on a type and geometry of a cantilever and investigated sample. Value “ NF ” can change from -3 nA up to -20 nA. For operation in the modulation mode the “NF” signal must be set to the zero position.

3.2.5 Setting SFM head

Put aside protective cover and disconnect its grounding wire if necessary. Move down the scanner with a sample on its top by rotating black knob clockwise (Fig. 149). The knob is attached to a screw, and the sliding cylinder with piezotube behaves as a nut; so when the knob is rotated clock-wise (bottom view) the cylinder moves down, and vice versa.

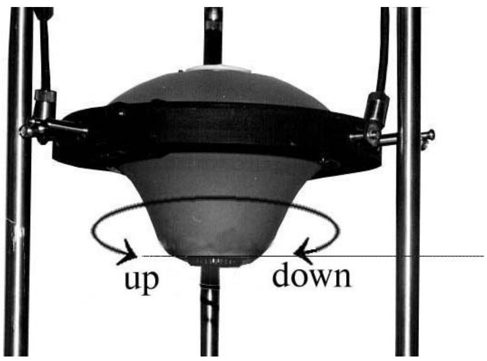


Fig. 149

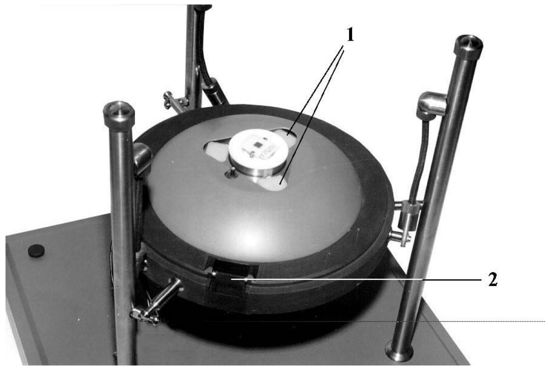


Fig. 150

Set SFM measuring head with its legs onto the support plates (1) (Fig. 150) on top of the scanner unit.

The cable should be opposite the pocket (2) (Fig. 150) in the rubber disk. Insert the black holder on the cable into the pocket (Fig. 151). Move the scanner up by rotating the knob counter-clockwise (bottom view) to approach the sample to about 0.5 - 1.0 mm from the probe (Fig. 152) observing it from the side. It is advisable to have the investigated area of the sample on the scanner axis i.e. in the middles of the line connecting the substrate clips. Should the investigated sample be located away from the scanner axis there will be a parasitic incline of the surface in the course of scanning. It limits the use of some features and makes the operation more difficult.

Fig. 151

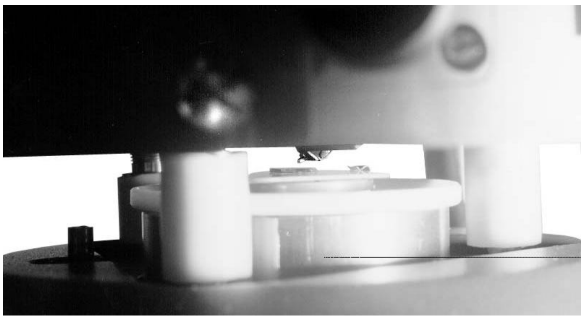


Fig. 152

Check signal values in the program oscilloscope windows "LF" and "NF" to be sure that the adjusting screws have not been accidentally moved.

Place the protective cover (Fig. 14) onto the scanner unit. Plug the protective cover grounding cable into its jack. Carefully take the scanner unit, lift it slightly from the arresting rods while rotating it clockwise (top view), and lower it to suspend it from the rubber isolation bands. Then set it horizontally within 2 - 3 degrees with adjusting screws of suspension system (Fig. 14).

3.2.6 Automatic approach of sample to probe (contact mode)

Further control of SPM operation is executed through a control program.

In a right upper angle establish an input of a feed back "Normal F" (Fig. 153).

Point the button "Operation" in the top of the "SPM" Window. Click the item "Approach" (Fig. 154) in the appeared menu and set proper parameters in the label "Probe" (Fig. 155). To choose a parameter value, click its button. Set the value by dragging a slider with the mouse or moving it with keyboard arrows.

Set photodiode differential current set point "Set Point (nA)"=0 and feedback gain "Feed Back Gain"=10 (Fig. 155). As for value of bias voltage "Bias Voltage (v)", it is not important in SFM mode. The set up values may be saved on the hard drive for future reading and application (Fig. 156).

Fig. 153

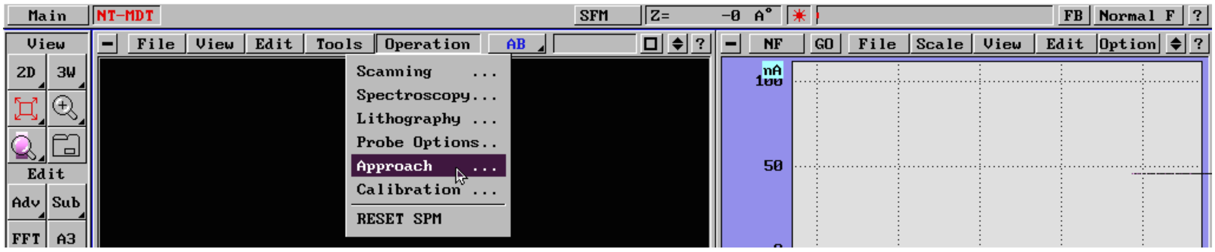


Fig. 154

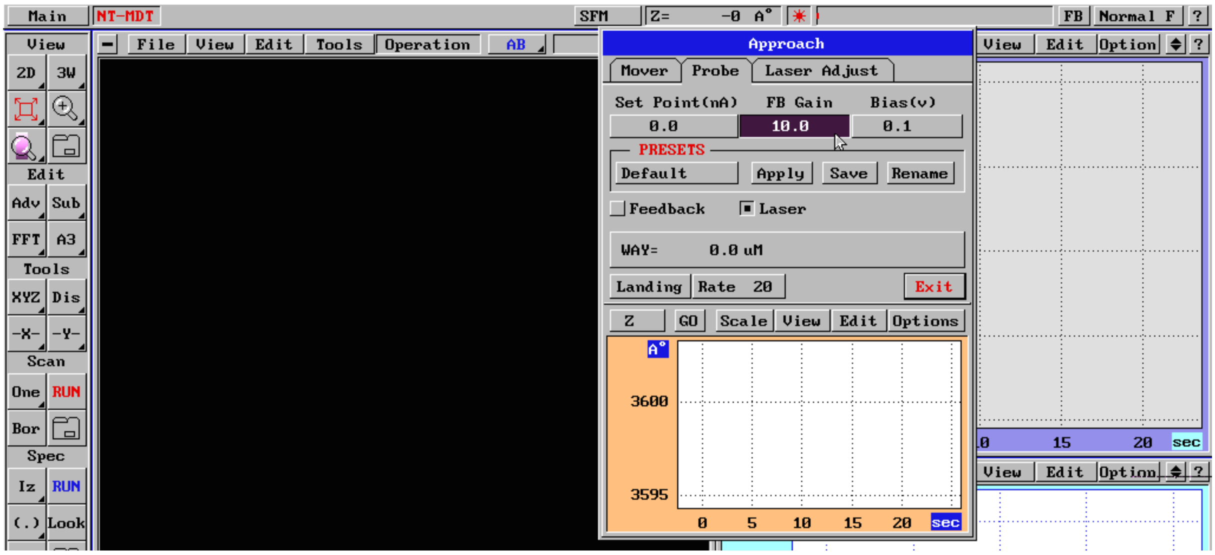


Fig. 155

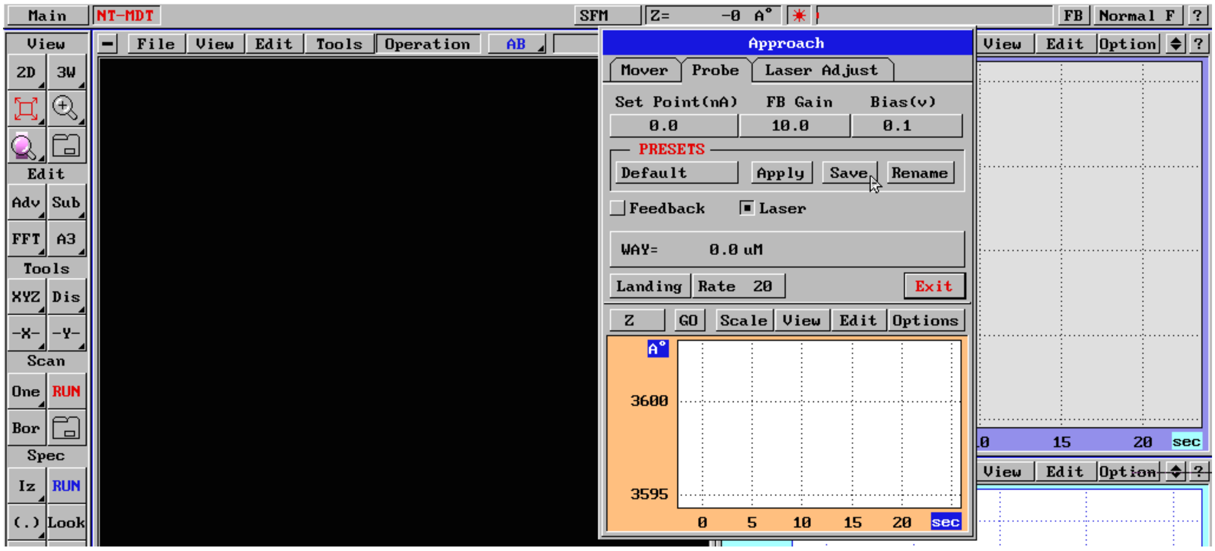


Fig. 156

Before landing it is advisable to turn on the oscilloscope (“FB” input terminal (Fig. 157) - Feedback) by pressing the “GO” button (Fig. 158) in order to check whether the signal has been set up for the negative zone (see “!” on page 3-67) and to watch the feedback signal during the landing.

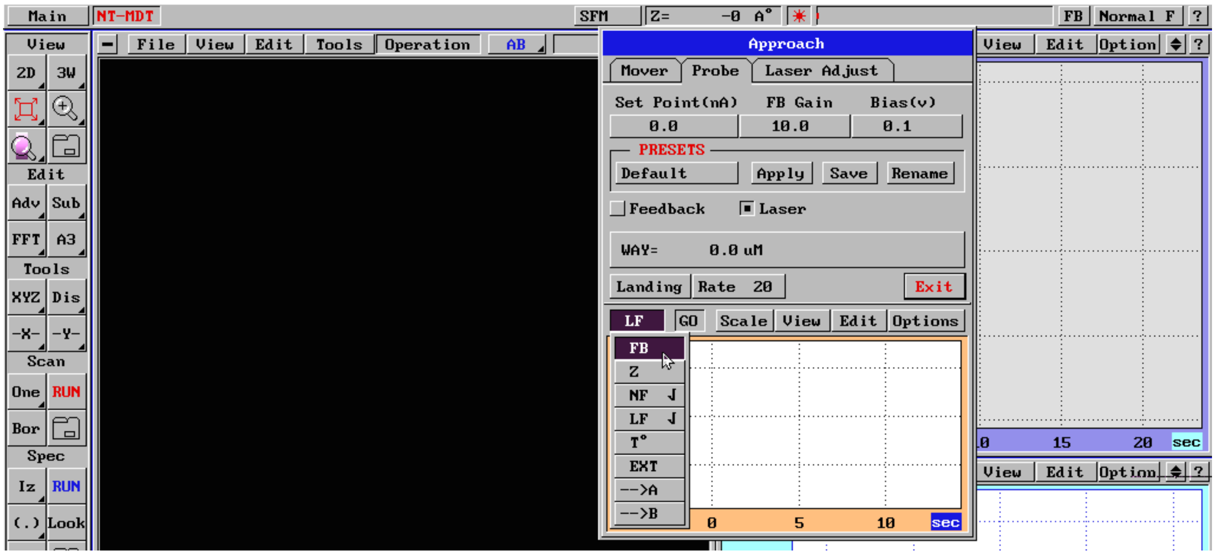


Fig. 157

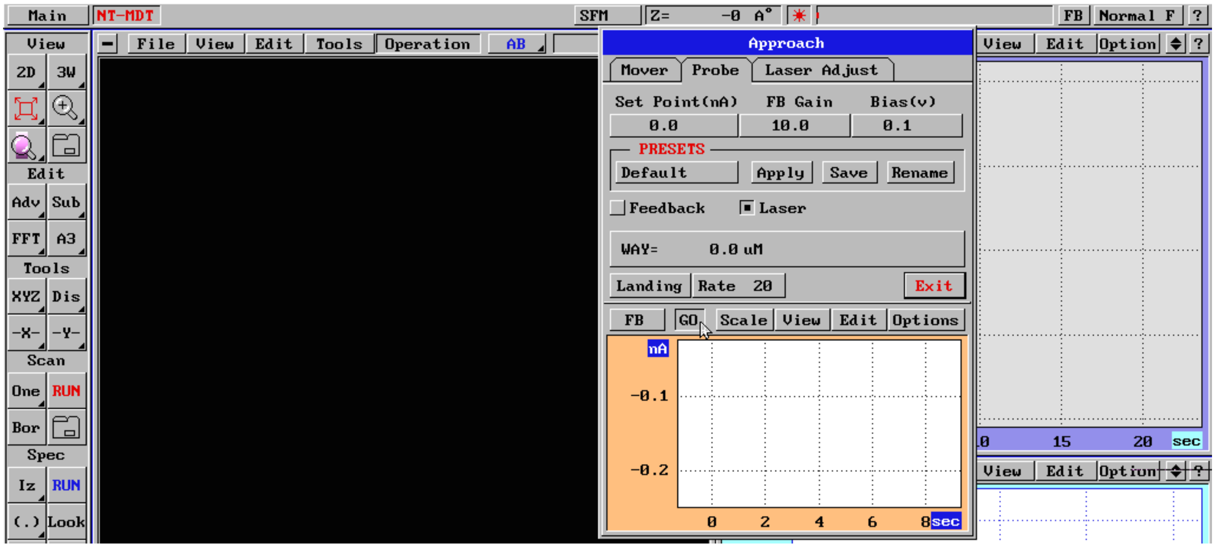


Fig. 158

Set up the maximum "Landing Rate" (Fig. 159).

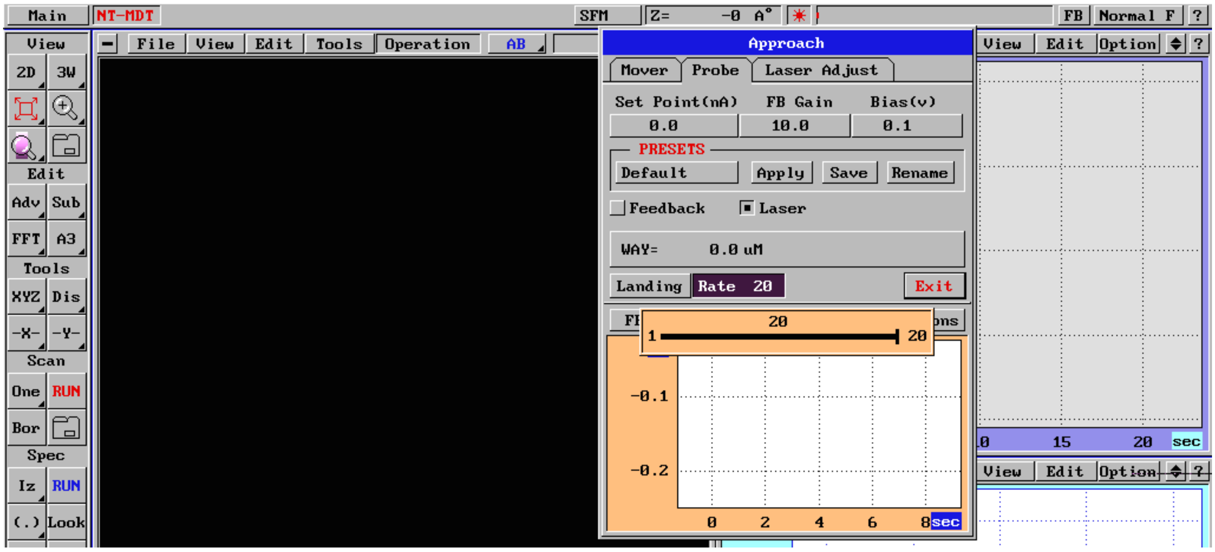


Fig. 159

It is advisable to turn off the measurement of currently unnecessary signals in oscilloscopes as computer time is assigned for the signal measurement and also switching of the automatic centering device between signals to be measured may create interference. In order to turn off the measurement of a currently unnecessary signal it is necessary to open the list of the oscilloscope inputs (Fig. 160), in front of the signals being measured there is a check sign. Choose the signal you want to turn off and depress the “GO” button (Fig. 161). Should several signals be turned off, repeat this procedure for all currently unnecessary signals.

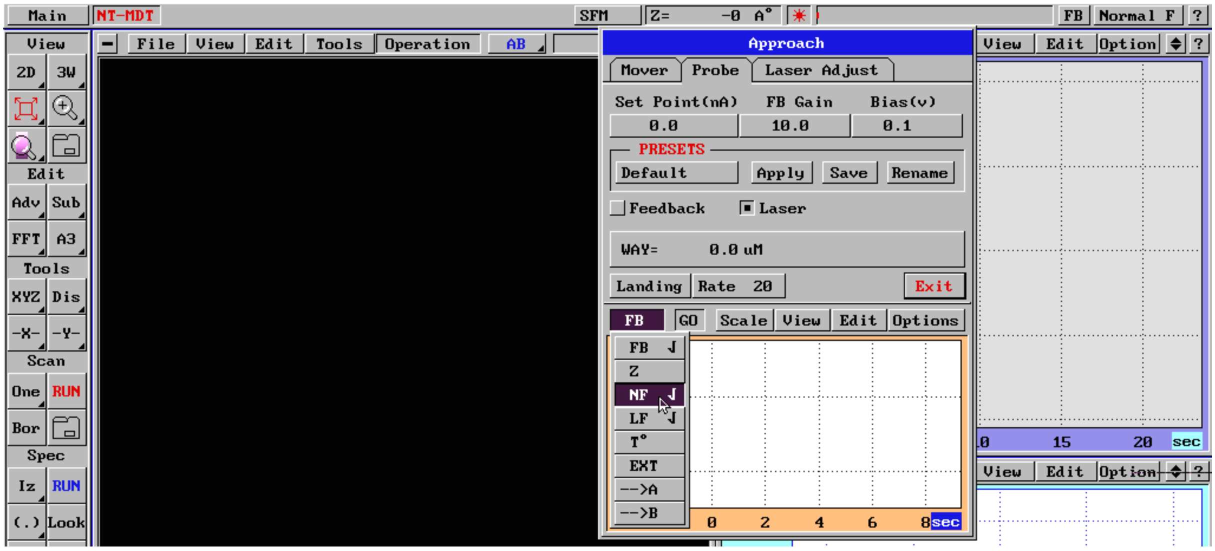


Fig. 160

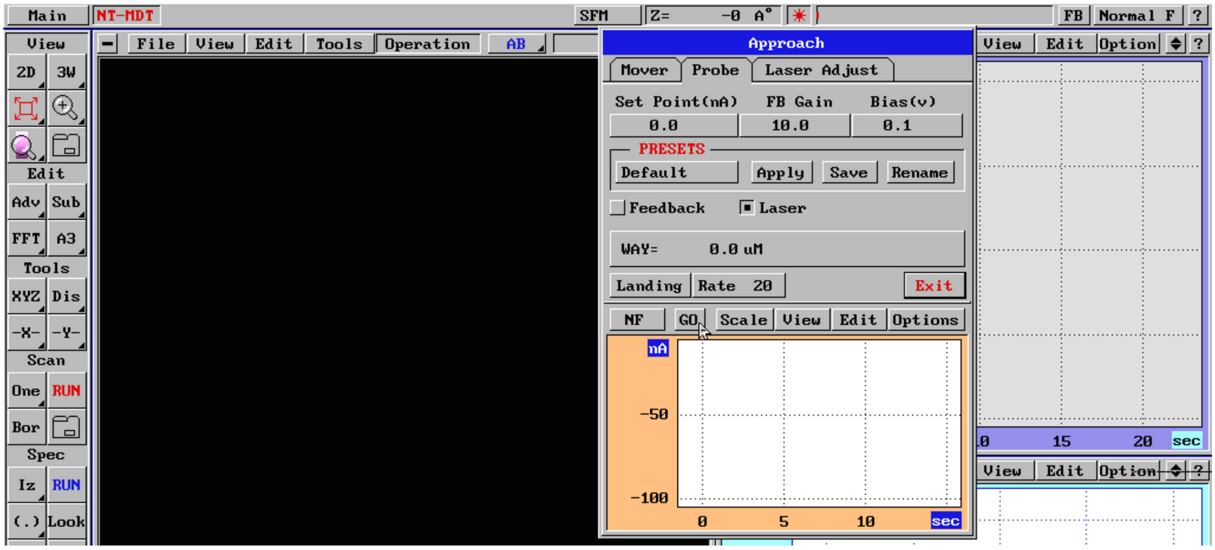


Fig. 161

Click on the "Landing" button (Fig. 162). The landing is done automatically and this is indicated by an audible signal and an on-screen message. At the same time the feedback should be enabled (the "FB" button in the top right corner of the screen will look pressed and the scanner must move to the maximum front position which is indicated by a red bar in the top right corner of the screen. The reading of step engine counter will increase. In case the feedback is enabled but the scanner does not move and the reading of the counter starts decreasing, stop the landing by pressing the ESC key or the right mouse button in any position, and check if the current parameter has been set up correctly ("Set Point").

For the operation in the contact CCM mode the value set in "Set Point" should be higher than the current value of the "FB" or "NF" signal. For example, SP=0, current value of FB=-5nA.

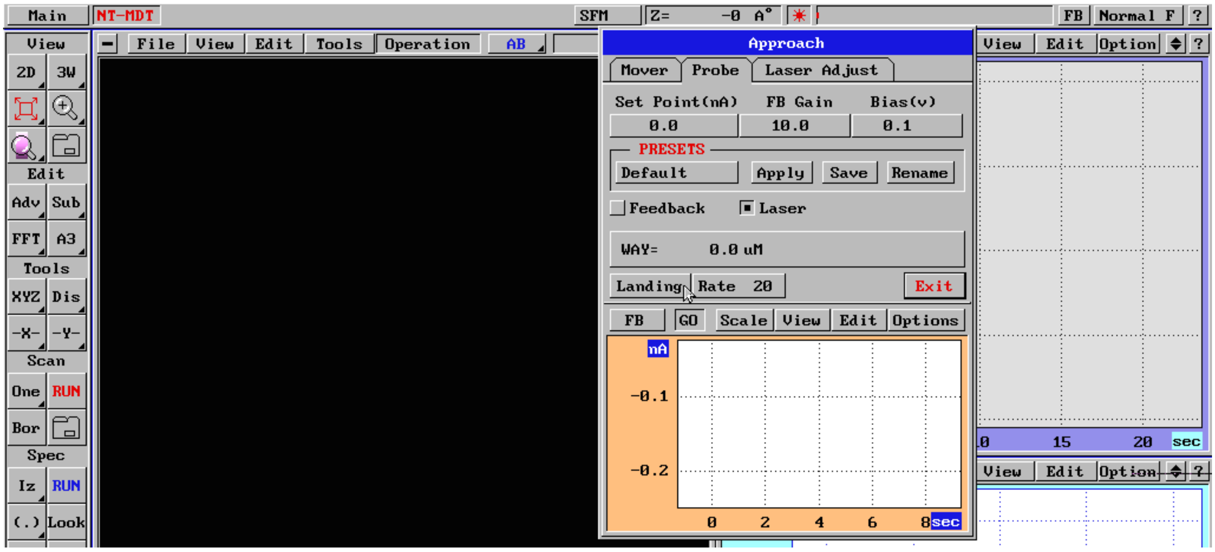


Fig. 162

It is useful to withdraw part of approach system to reduce thermal drift caused by elements of the system. To do this go to the "Mover" label in the "Approach" menu (Fig. 163). After decreasing the "Forward Rate" (Fig. 164) make one or more steps forward by pressing the "Forward One step" button (Fig. 165) while watching the red bar in the top right corner of the screen which indicates the scanner extension. The bar must decrease to one forth of its maximum length. After it click on "Backward Until ESC" (Fig. 166) and wait until the counter counts back about 100 microns. Then press button "ESC" on keyboard to stop moving.

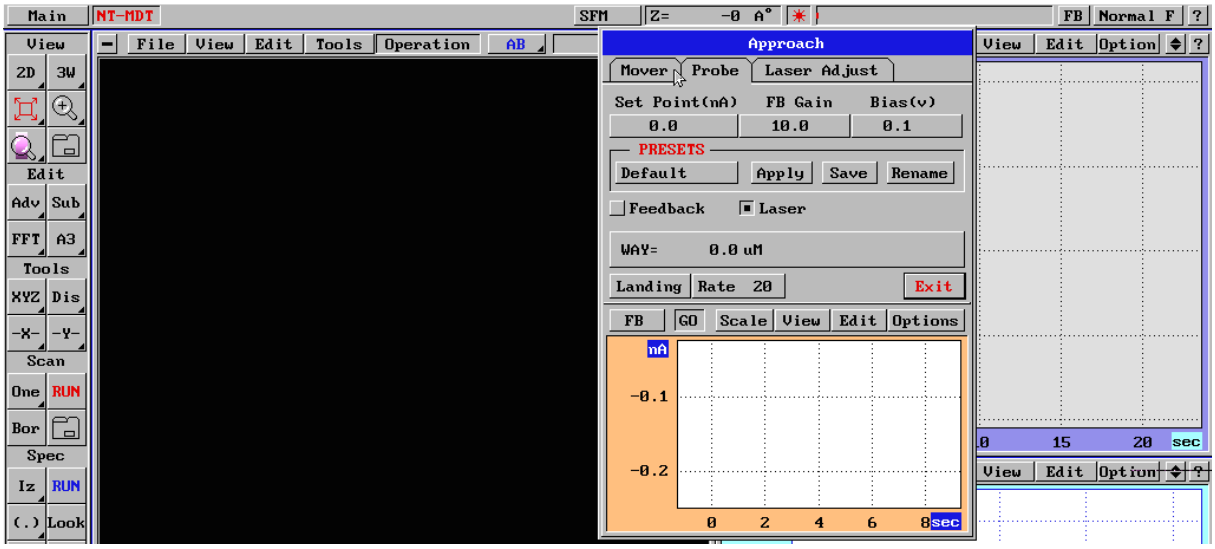


Fig. 163

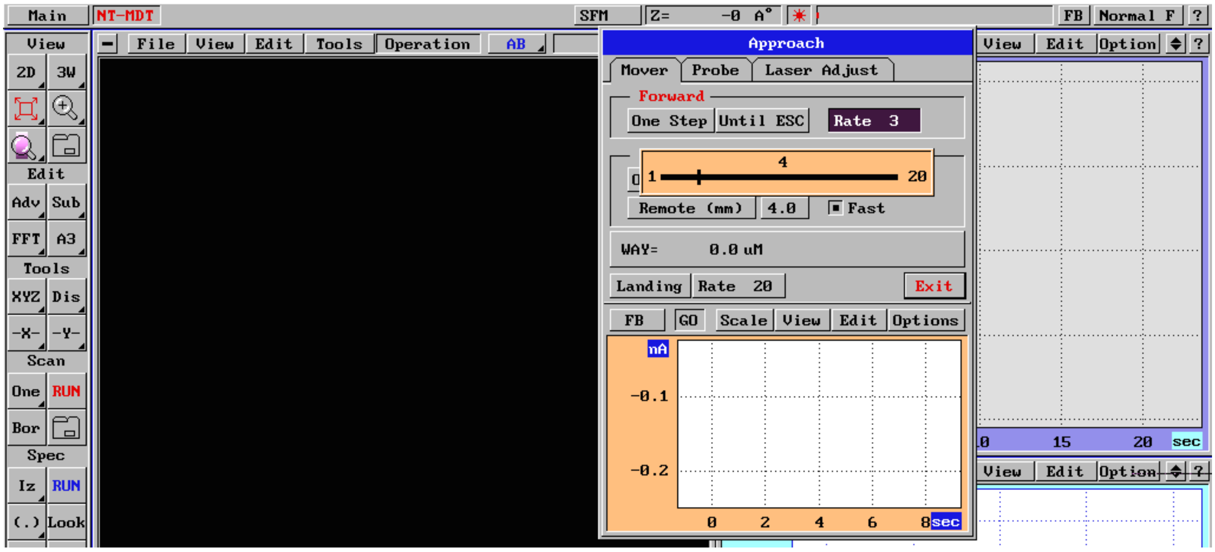


Fig. 164

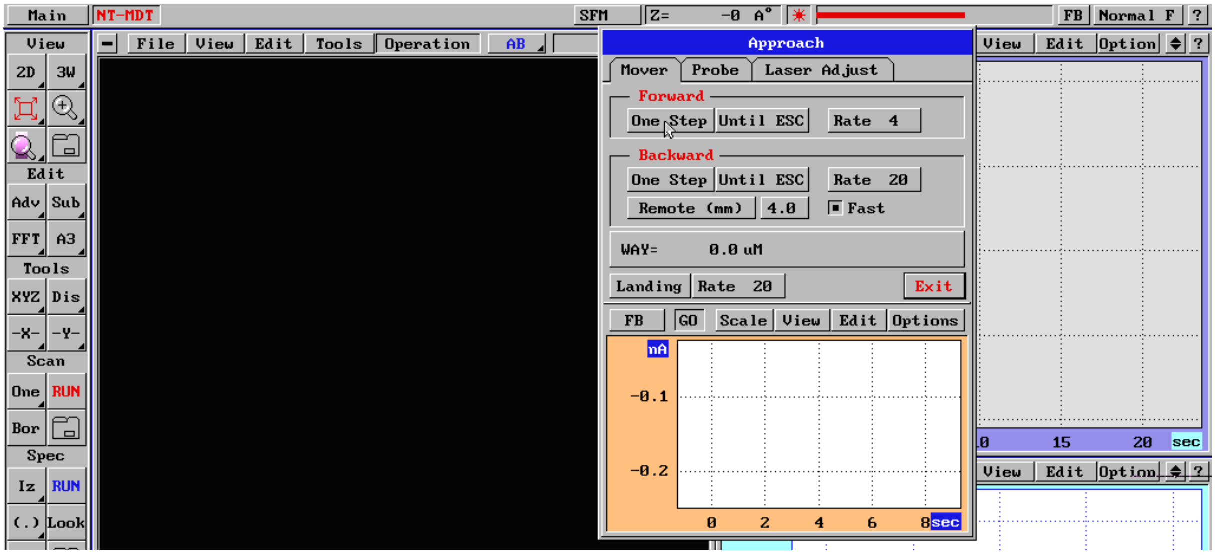


Fig. 165

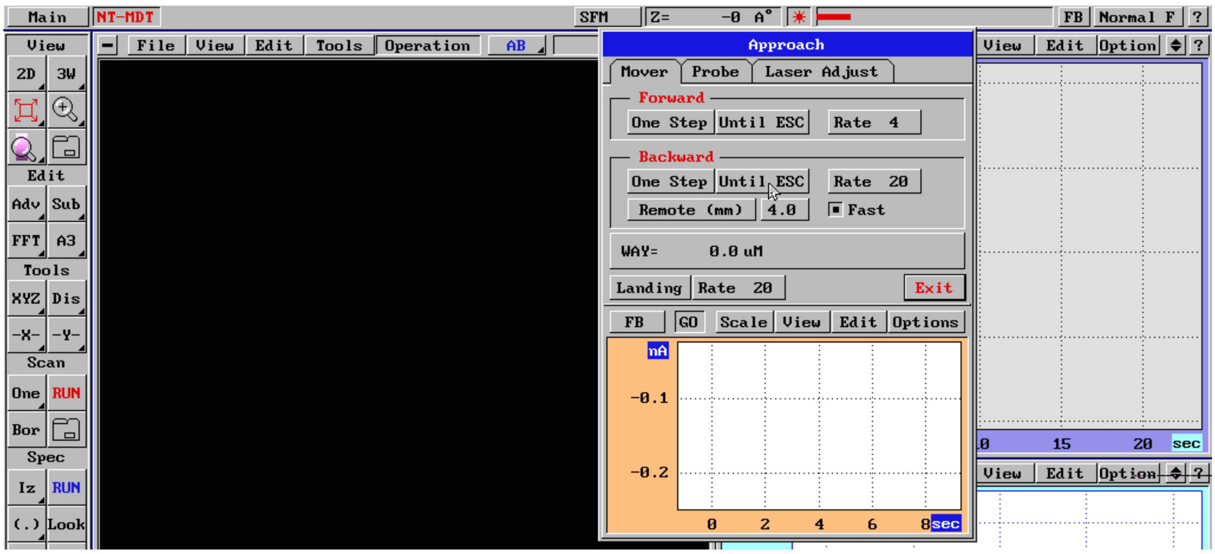


Fig. 166

3.2.7 Getting a test scan

Operations of setting scan parameters, getting image, and its processing are executed in "SPM" window in the left part of the screen. Point the button “Operation” to open corresponding menu and then click the item “Scanning” (Fig. 167) to open its menu. Scanning parameters are set up in the displayed window.

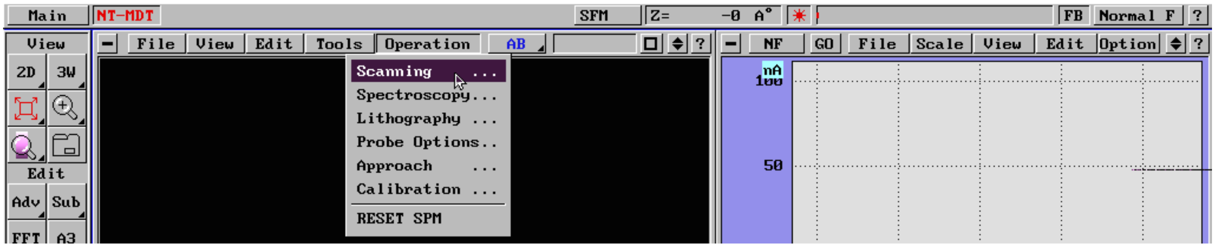


Fig. 167

3.2.7.1 Getting scan of a test grating

Set the following values of parameters:

- Modulation - off (Fig. 168)
- FB - Normal Force (Fig. 169)
- Scan A - Height (Fig. 170)
- Scan B - Lateral Force (Fig. 171)
- Velocity - 500000 A/ñåê (Fig. 172)
- NX xNY - 256 õ 256[5] (Fig. 172)
- Step (A) - max[6] (Fig. 172)
- NL Corr - on (Fig. 172)
- Scan dir - +X+ (Fig. 172)
- Gain = 1 (Fig. 172)
- Lpass(kHz)=3 (Fig. 172)
- Average = 1 (Fig. 172)
- Drawing - on (Fig. 173)
- Subplane - on+ (Fig. 173)
- SP - 0 (Fig. 174)
- FB Gain - 1-5 (Fig. 174) in case generation takes place decrease FB Gain.

Select the single scanning mode - One scan (Fig. 175).

Click RUN to start scanning (Fig. 176).

---

[5] If the software does not let you increase the scan size up to 128x128, then the scanning step "Step" should be decreased at least by two times. After that increase the scan size again.

[6] First set the scan size NXxNY at a small step of scanning and then set the biggest step size possible for this number of steps.

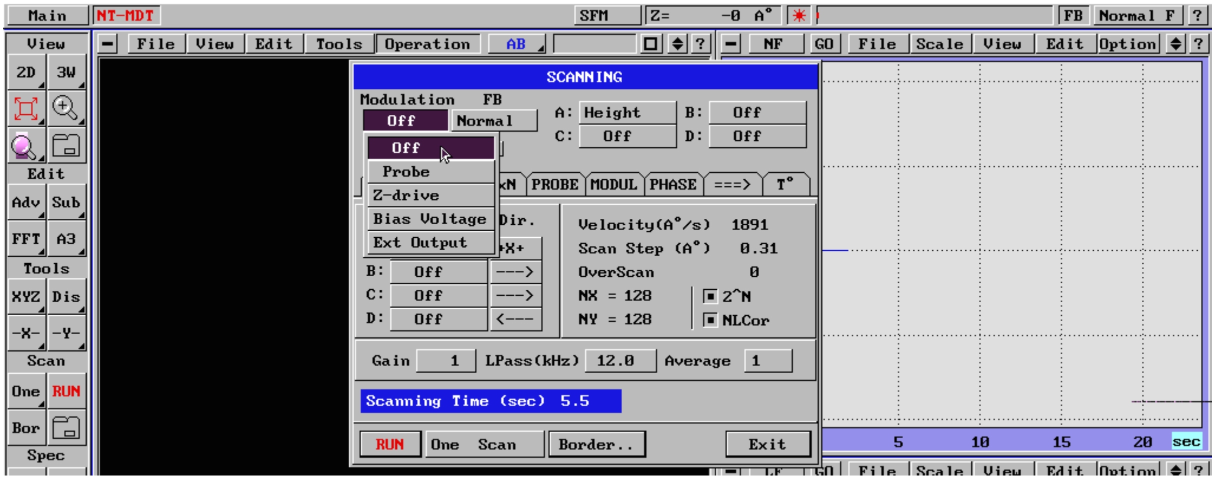


Fig. 168

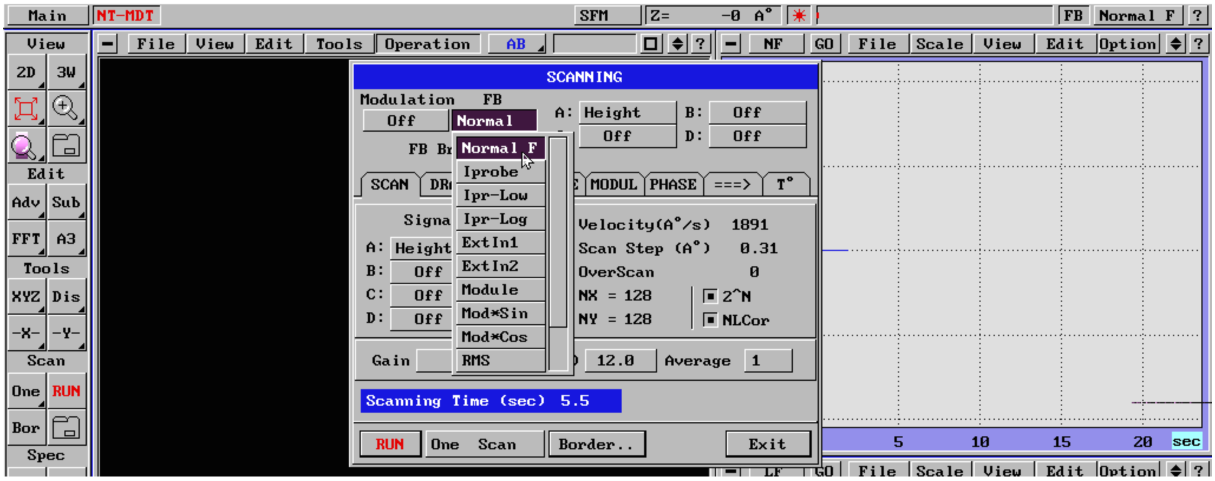


Fig. 169

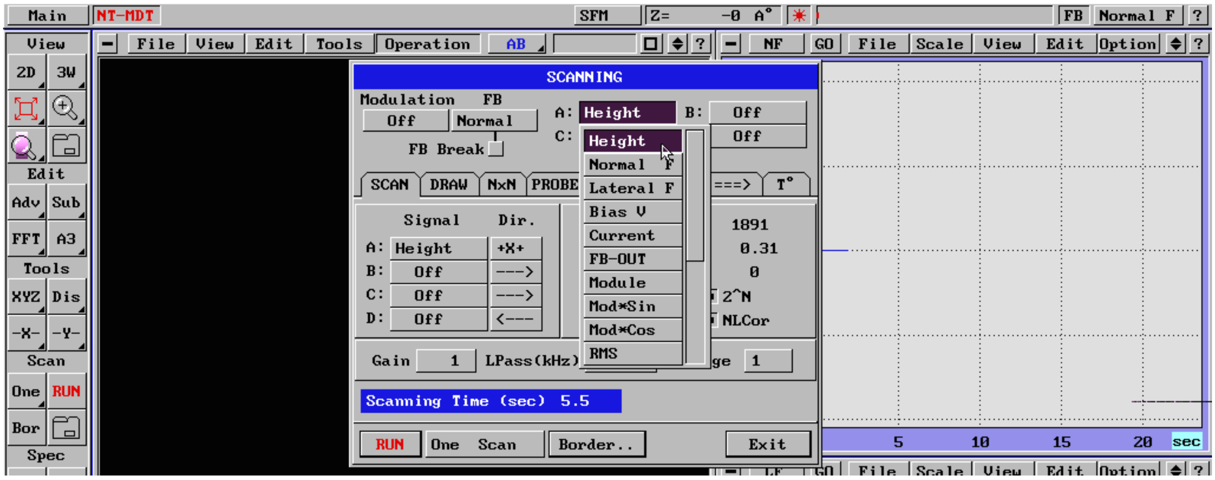


Fig. 170

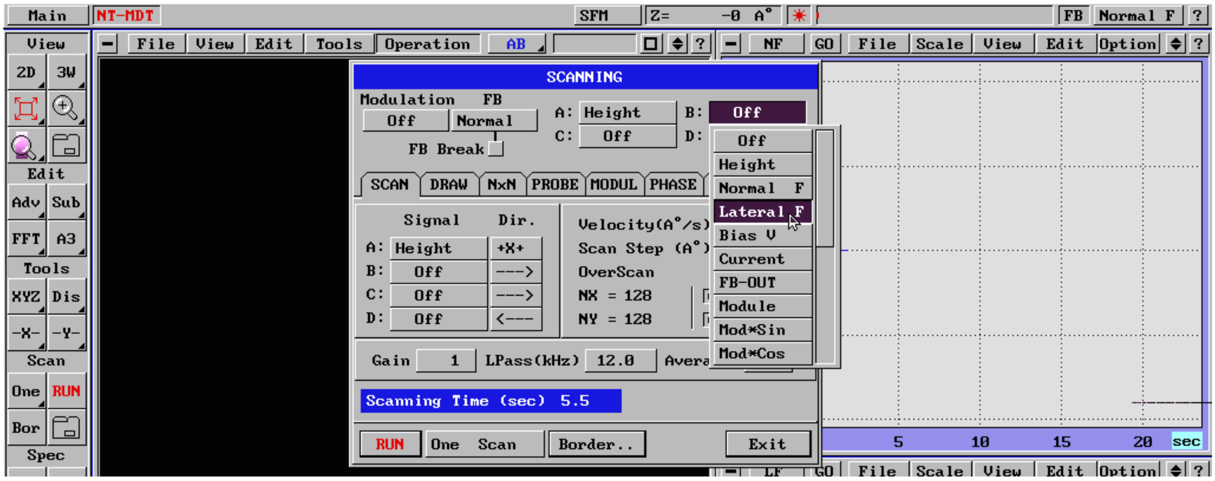


Fig. 171

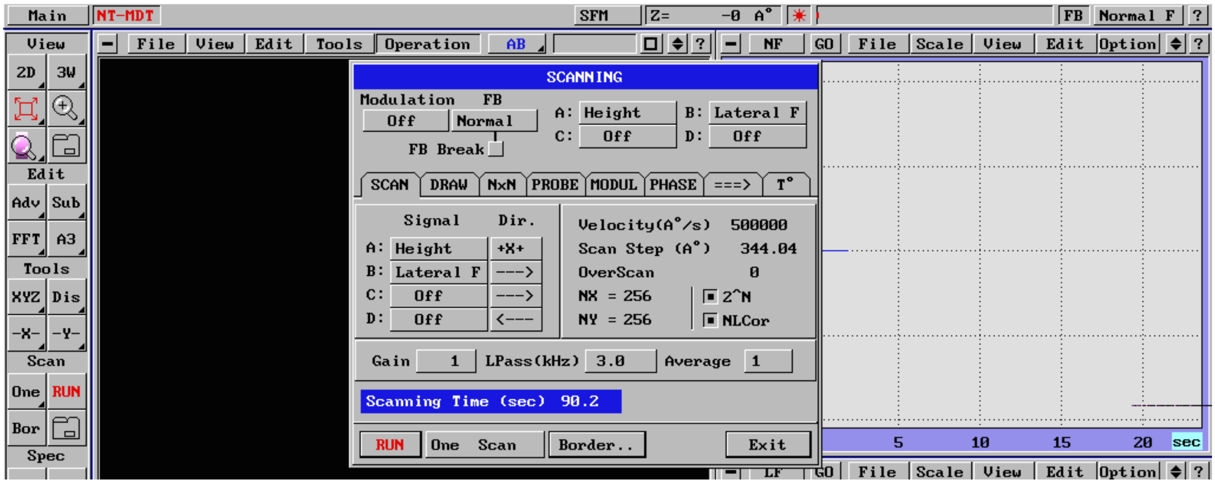


Fig. 172

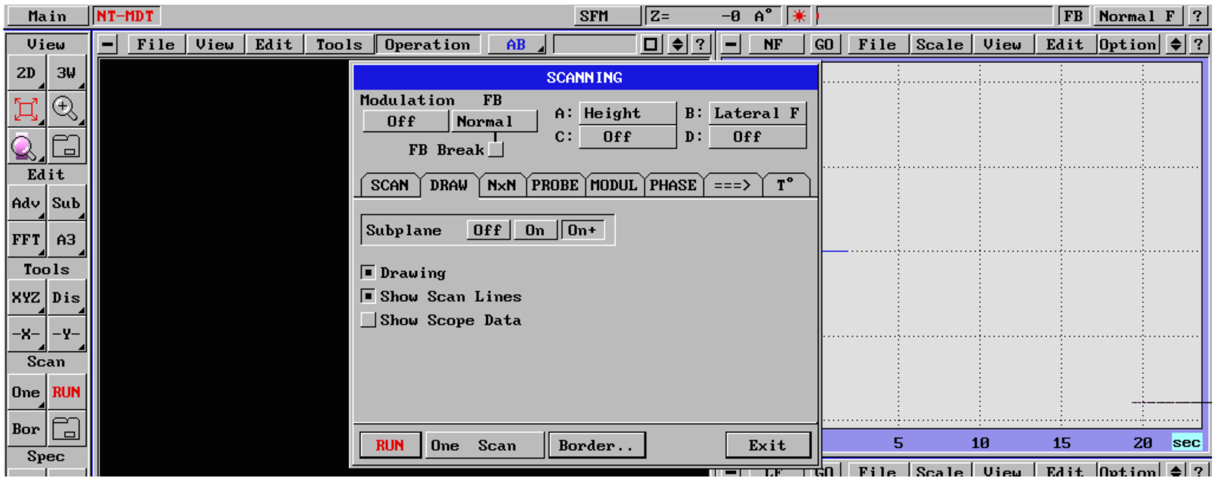


Fig. 173

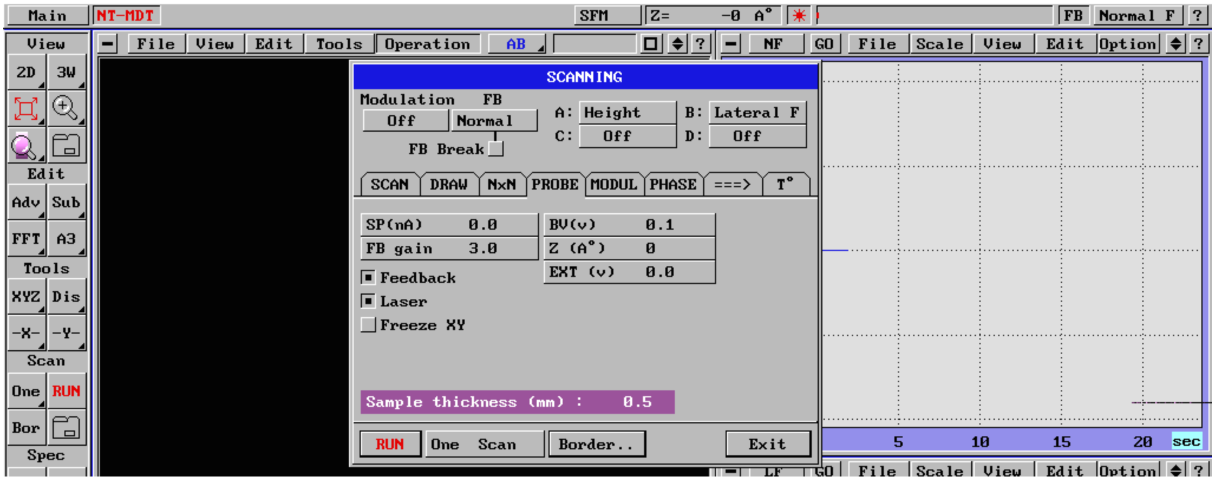


Fig. 174

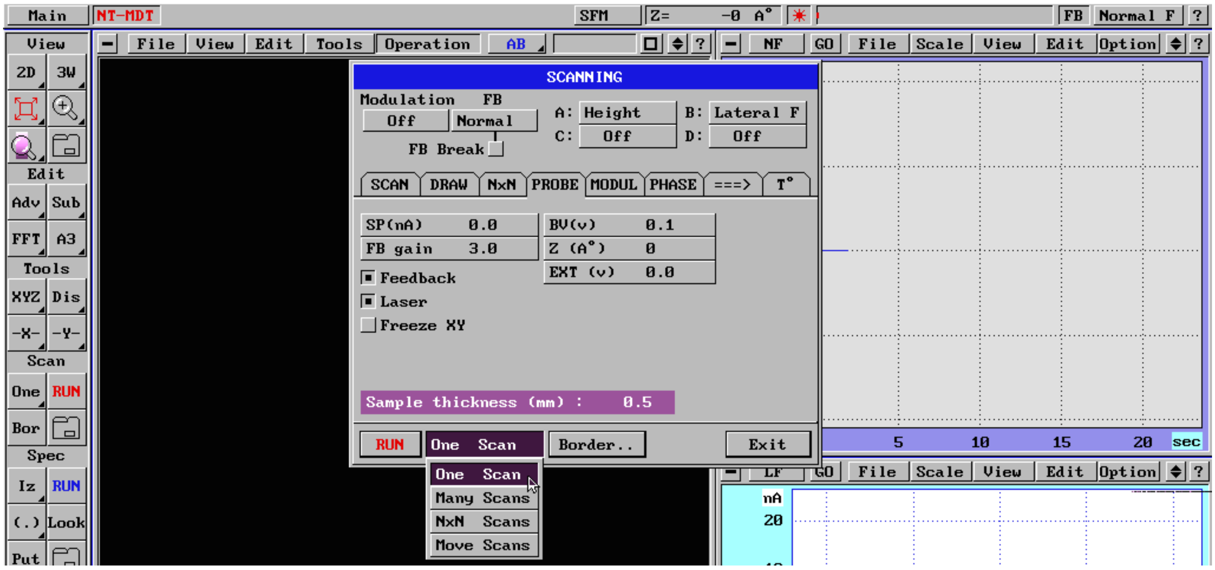


Fig. 175

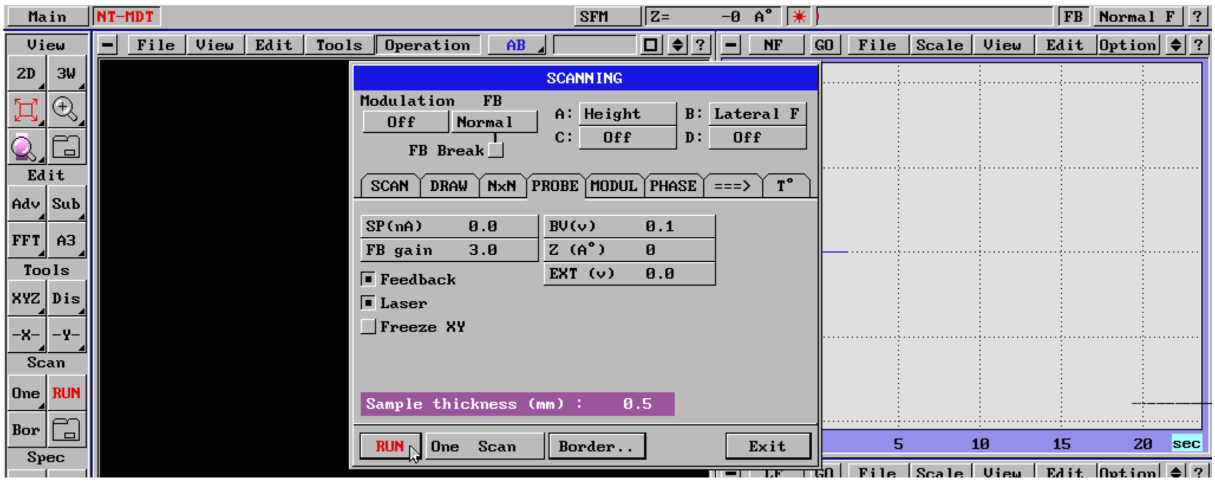


Fig. 176

To improve the result in the course of scanning it is possible to change the "Velocity", "FB gain" parameters in the top line of the menu selecting options by the "→" "←" keys and changing values by the "↑" "↓" keys. After the above values have been changed and the first scan lines have been taken execute the Restart command in the menu displayed in the upper part of the screen during the scanning.

3.2.7.2 Getting atomic lattice resolution

A fresh chip of mica plate is used for getting a test atomic resolution (see item 3.2.3.a).
Set the following values of parameters:

Modulation - off (Fig. 177)
FB - Normal Force (Fig. 178)
Scan A - Height (Fig. 179)
Scan B - Lateral Force (Fig. 180)
Velocity = max (Fig. 181)
Step (A) - 0.2-0.5 (Fig. 181)
NX xNY = 128 õ 128 (Fig. 181)
NL Corr - on (Fig. 181)
A: Scan dir - +X+ (Fig. 181)
B: Scan dir - → (Fig. 181)
Gain = 100 (Fig. 181)
Average = 1 (Fig. 181)
Drawing - off (Fig. 182)
Subplane - on (Fig. 182)
SP = 0 (Fig. 183)
FB Gain = 0.03-0.1 (Fig. 183)

The minimum step of scanning and maximum velocity depend on the specific configuration of SPM.

Select the single scanning mode - One scan (Fig. 184).

Click RUN to start scanning (Fig. 185).

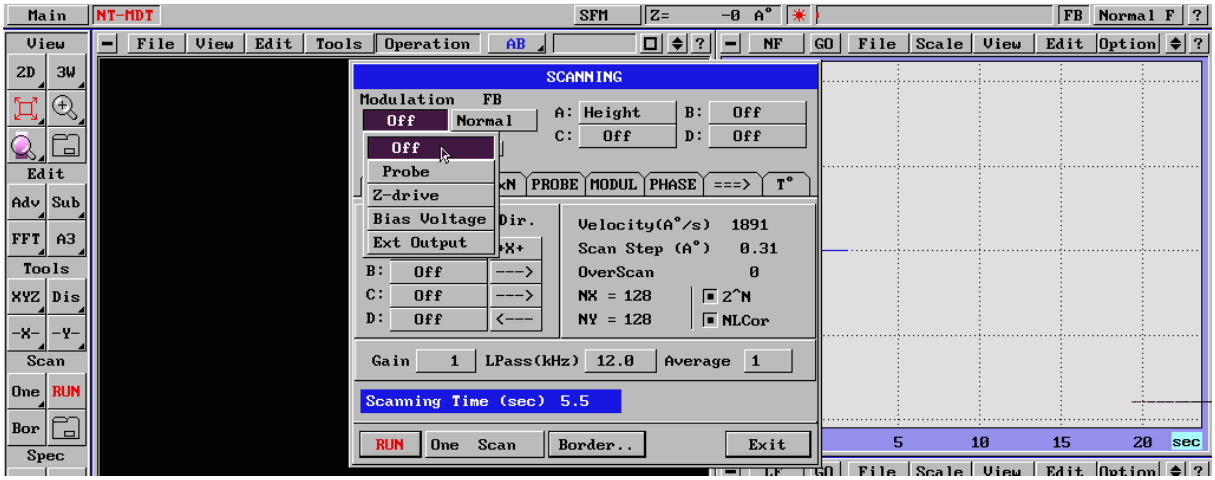


Fig. 177

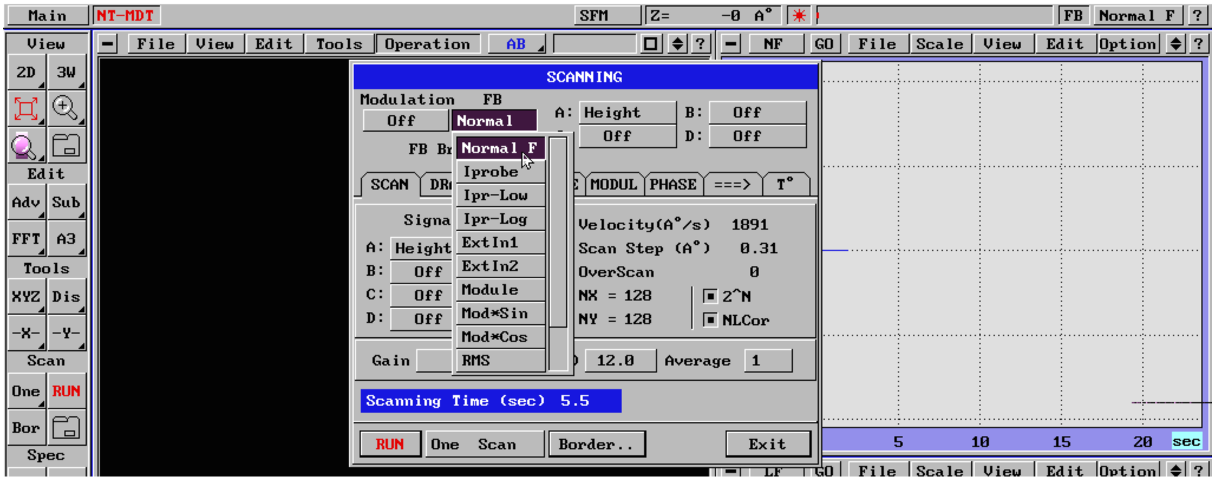


Fig. 178

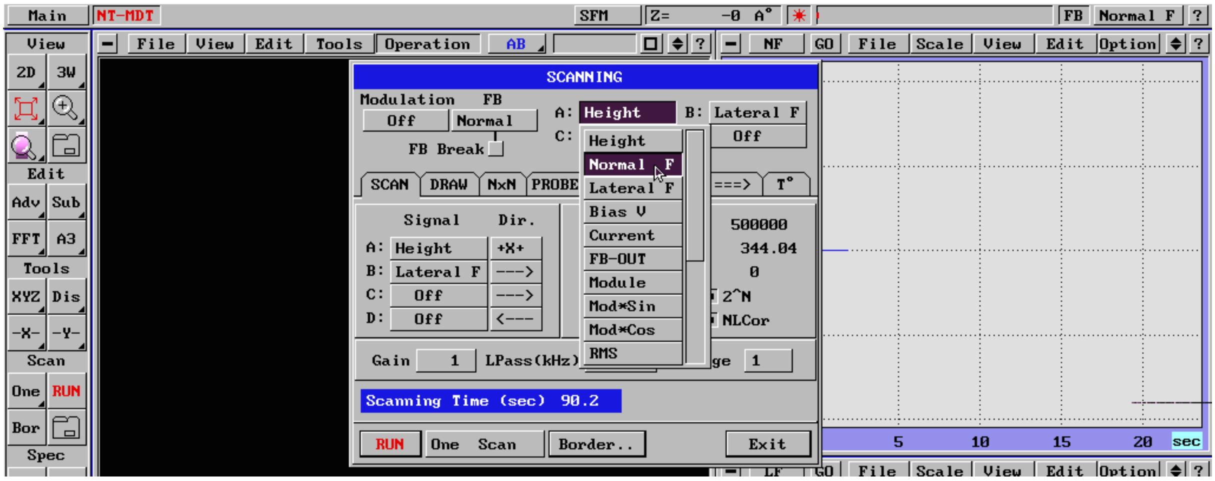


Fig. 179

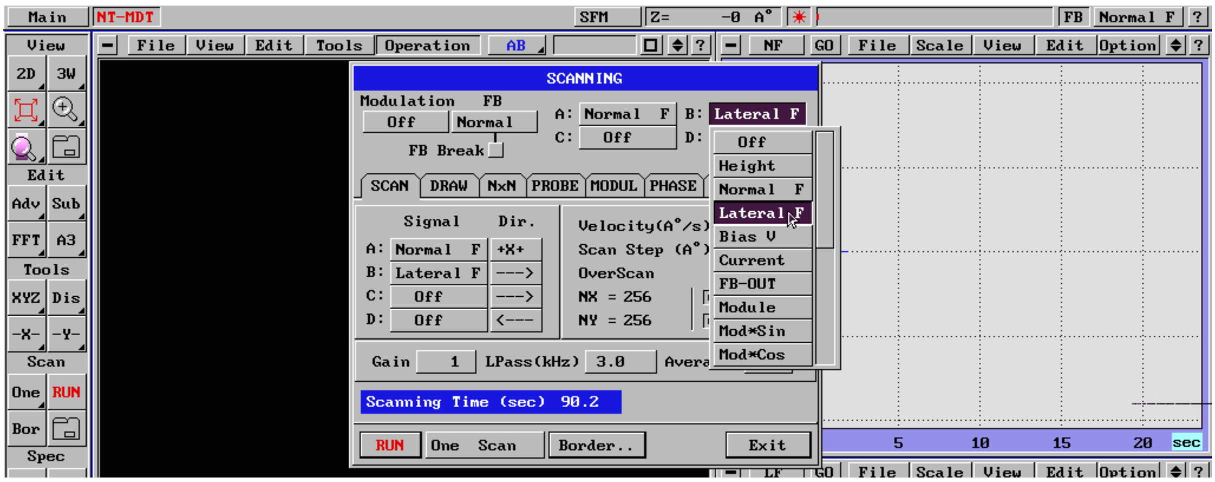


Fig. 180

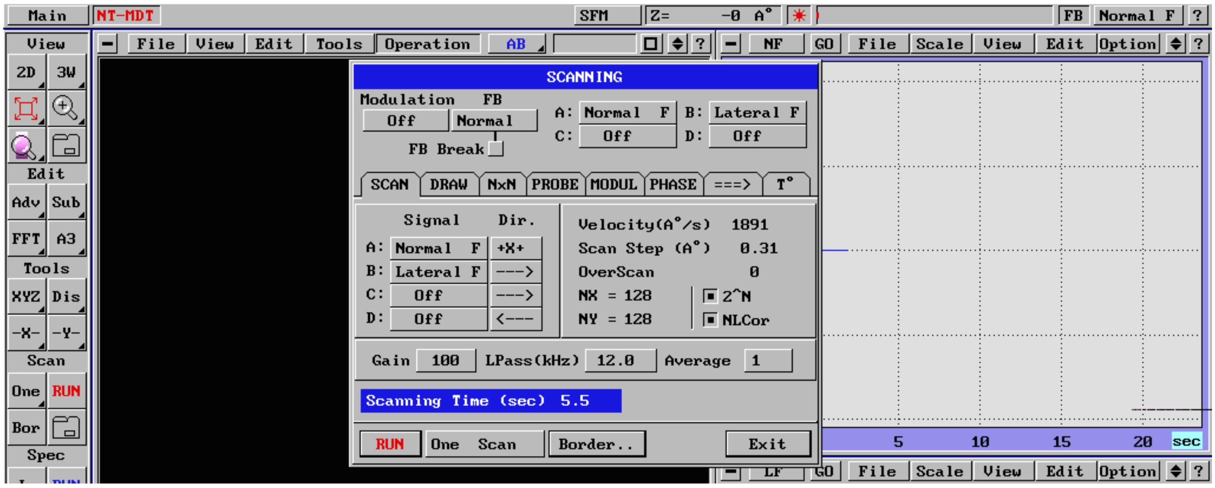


Fig. 181

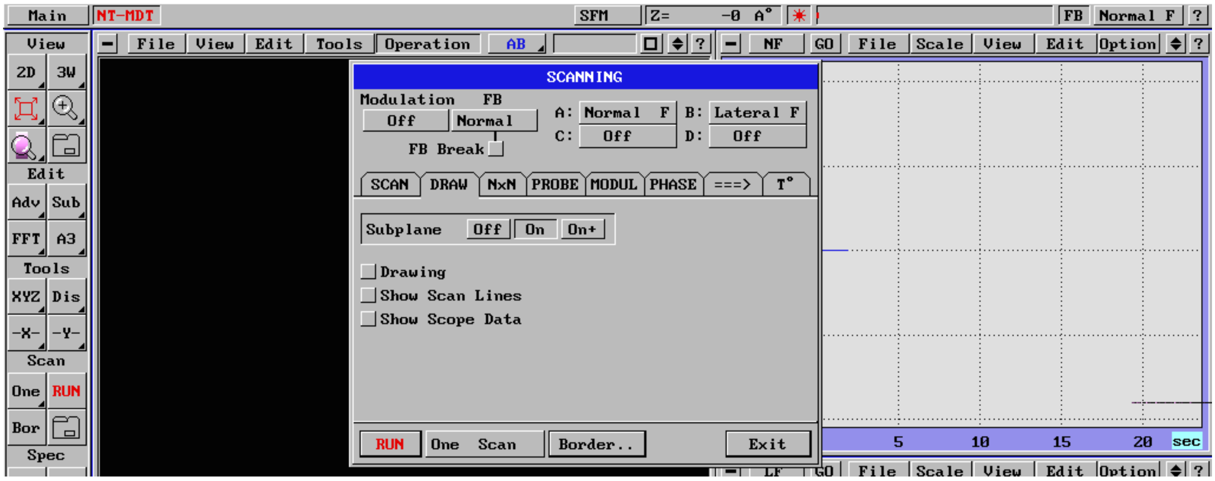


Fig. 182

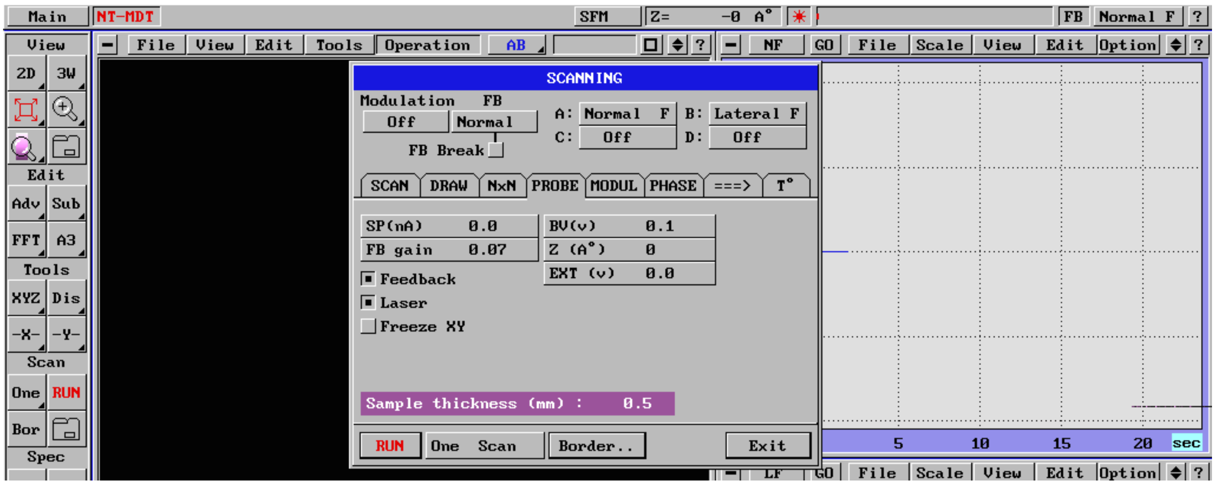


Fig. 183

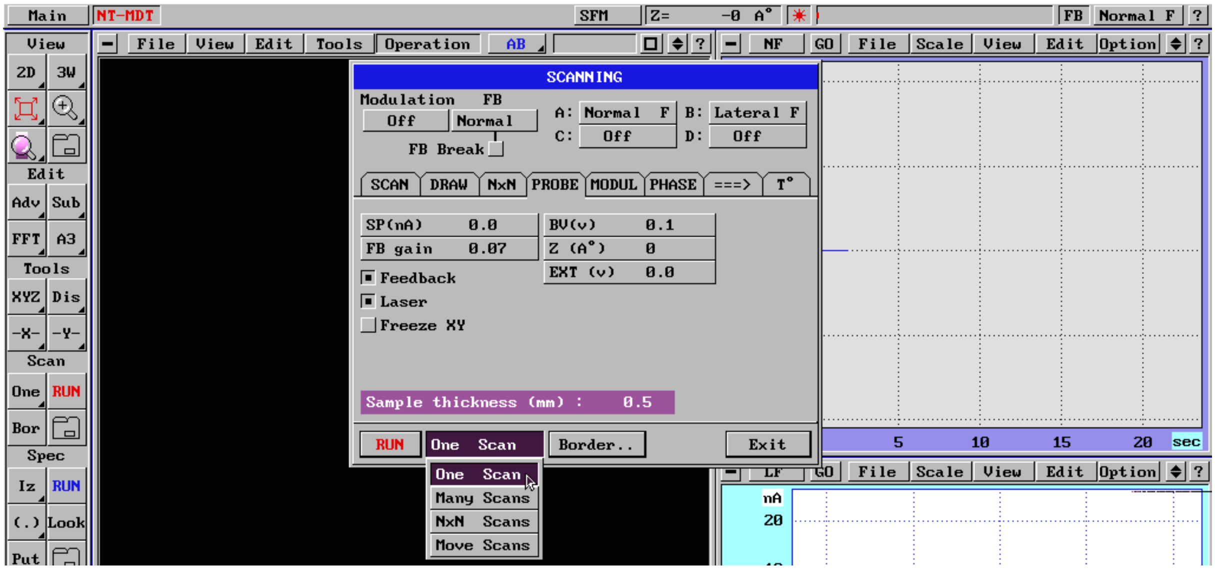


Fig. 184

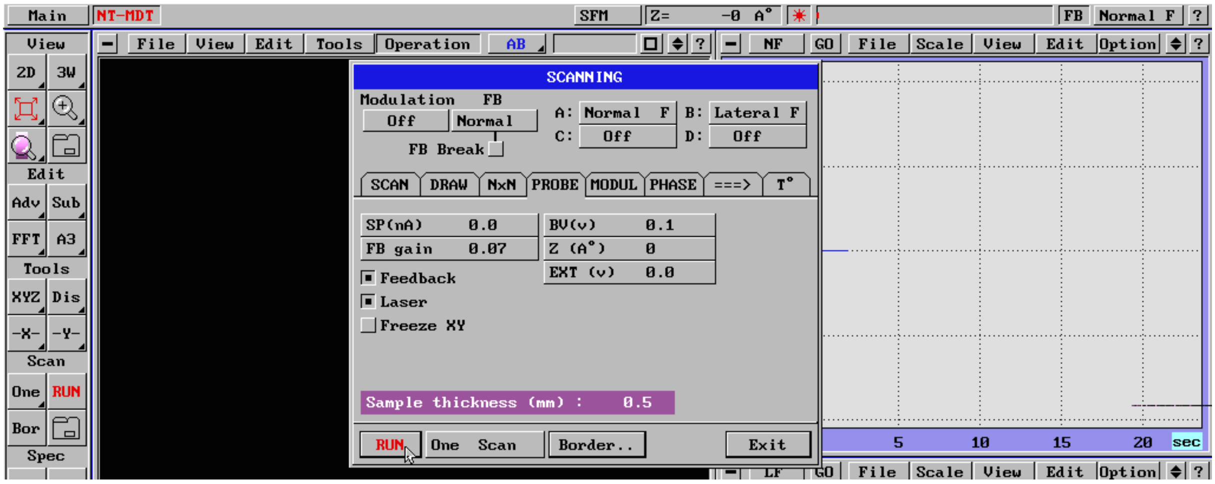


Fig. 185

In order to have the best result you can change the "Aver", FB qain", "Nx", "Ny" parameters.

3.2.8 Switching over from the contact to semicontact mode

To switch over between any modes it is necessary to turn off the feedback, set the new signal to the feedback input, determine the current value of this signal and set a new "Set Point" value in accordance with this value and only then the feedback may be turned on.

In this case after turning off the feedback it is necessary to determine the resonance frequency of the cantilever.

Enable the FB input on one of the oscilloscopes and send the "Module" signal to the feedback input. Turn on the probe modulation ("Operation", "Scanning", "Modulation" (top left) - "Probe"). Then search for the resonance frequency (see item 3.3.5) and select the "Amplit", "Gain" parameters (see item 3.3.5).

In the "Probe" label set up the "Set Point" value slightly (10%) higher than the current value of the "Module" signal. Turn on the feedback at the same place. Select the appropriate "Set Point" parameter (as described in item 3.3.6). Now you can scan in the semicontact mode.

3.2.9 Shutdown procedure

Using the step engine enter the Control menu ("Operation" -"Approach" - "Mover" (Fig. 186)) and turn on "Remote" for 4 mm (Fig. 187).

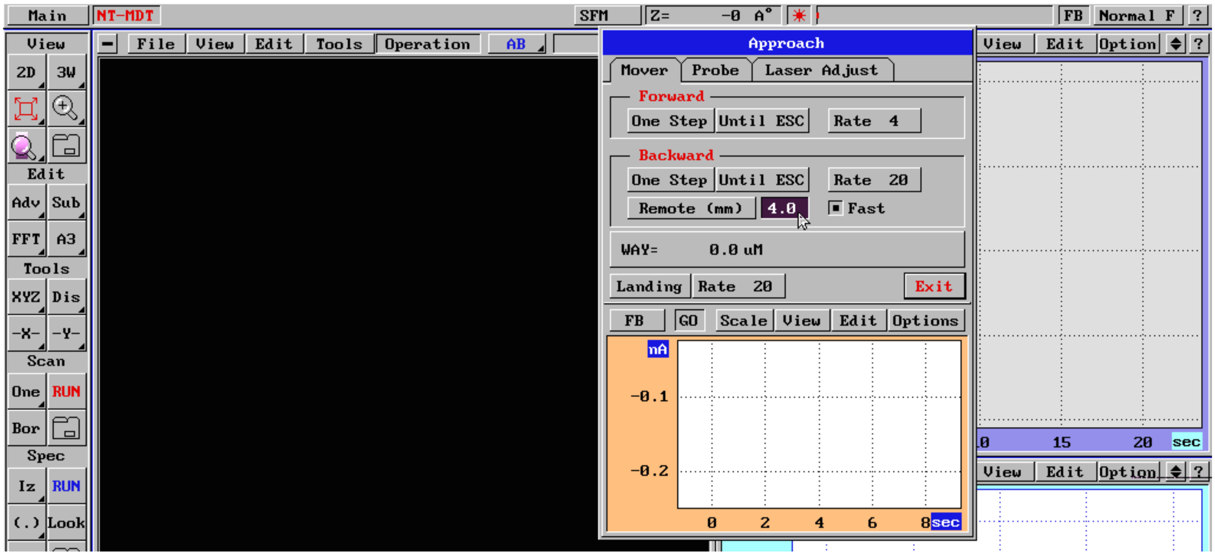


Fig. 186

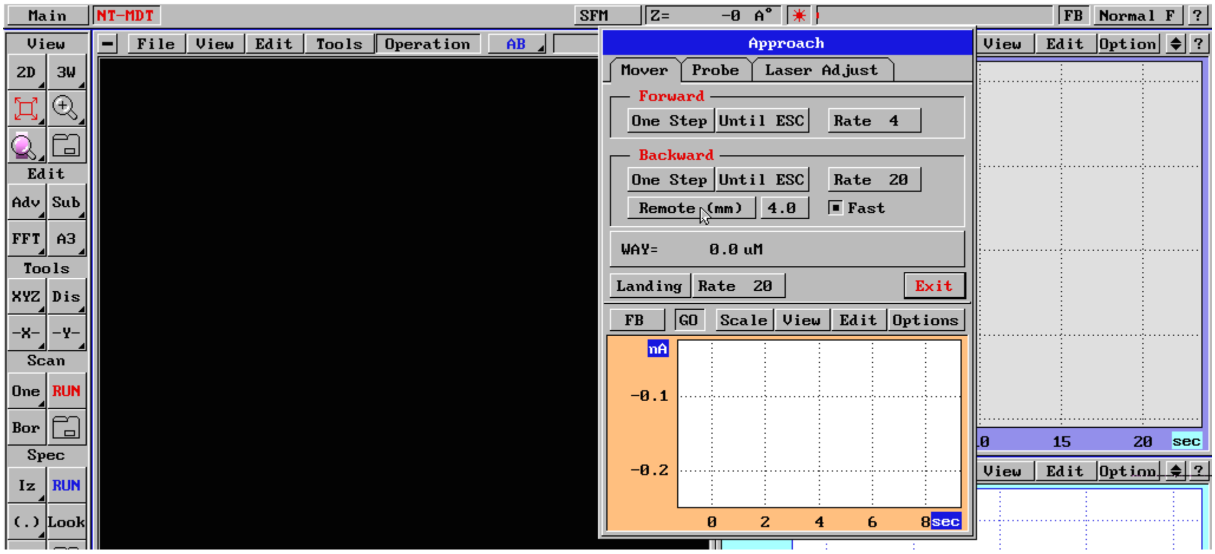


Fig. 187

WARNING:
Be especially careful when executing the "Forward" + "Until ESC" command. Don't use unless absolutely necessary.

In the "Probe options" menu disable the "Feedback" (Fig. 188) and "Laser" (Fig. 189). Lift the scan unit and install retainers on it.

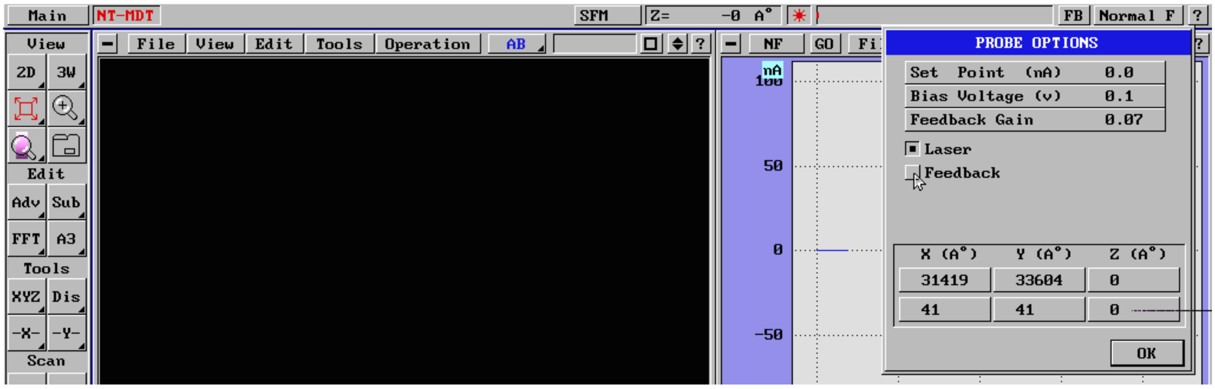


Fig. 188

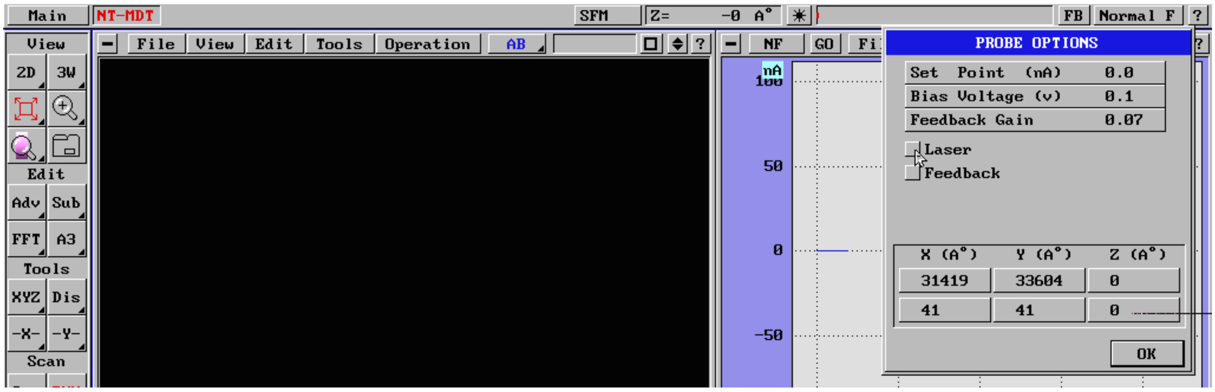


Fig. 189

Then:

- Remove the SFM head.
- Carefully take the sample from the site on the piezoscanner avoiding any considerable pressure on the sample holder.
- Turn off the power supply of the microscope using the power switch on the unit.
- Exit the control program by pressing the "Exit" button in the "Main" button menu in the left corner of the screen (Fig. 190) or using the F10 functional key (after closing all the opened menus using the "ESC" key, otherwise the F10 is not activated).

Fig. 190

3.3 Semicontact mode.

The semicontact mode of atomic force microscopy features some advantages as compared to the contact mode.

First of all this mode reduces pressure on the surface which makes it possible to investigate softer and more tender materials, such as polymers and biological organic objects. Also the semicontact mode is more ore sensitive to forces of interaction with surface which allows investigation of some surface properties - magnetic and electric domains distribution, hardness and viscosity of surface.

The principle of the semicontact mode is that the cantilever is swung by a mechanical swinging generator (in our unit this is the piezoceramic generator located in the cantilever holder) and when approaching the surface the oscillations of the cantilever (normally the amplitude of these oscillations is about 10-50 nm) are limited by collision with the surface. Usually this amplitude of oscillations limited by collision with the surface is used to support feedback. Most often the cantilever is swung on its resonance frequency.

3.3.1 Selecting the cantilever

Cantilevers having a resonance frequency over 80 kHz are normally used for the semicontact mode. In our unit the parameters of the resonance amplifier have been selected in the way to have the lowest noise in the band over 100 kHz. Usually silicon cantilevers have such resonance frequency as well as small nitride ones. The higher is the resonance frequency of a cantilever, the higher is its quality and therefore its sensitivity which allows operation at very small forces of interaction with the surface without any deformation of the surface being investigated. Magnetic cantilevers (cobalt sprayed) are required for investigation of magnetic domains and to investigate electrostatic interaction with the surface one should use conductive cantilevers (high silicon alloys or metal sprayed).

3.3.2 Mounting a cantilever

The cantilever is installed into a special holder having a piezoelement for the semicontact mode.

The special holder is installed in a special microscope table designed for the semicontact mode. The table terminal is connected to the head terminal.

3.3.3 Adjusting

Adjusting is carried out in the same way as in the SFM mode (see item 3.2.4).

When adjusting a cantilever, after obtaining the maximum intensity signal from the laser beam reflected by the cantilever it is recommended to shift the laser beam toward the narrow part of the cantilever even if it results in a slight (not more than 20%) decrease in the signal intensity. To do this slightly turn knob 2 (Fig. 133) counter clockwise.

As opposed to the SFM mode, it is necessary to obtain a zero signal. Turning photodiode alignment screws 6 and 7 (Fig. 133) set up 0 (Fig. 191) signal values in both oscilloscope windows (Fig. 192).

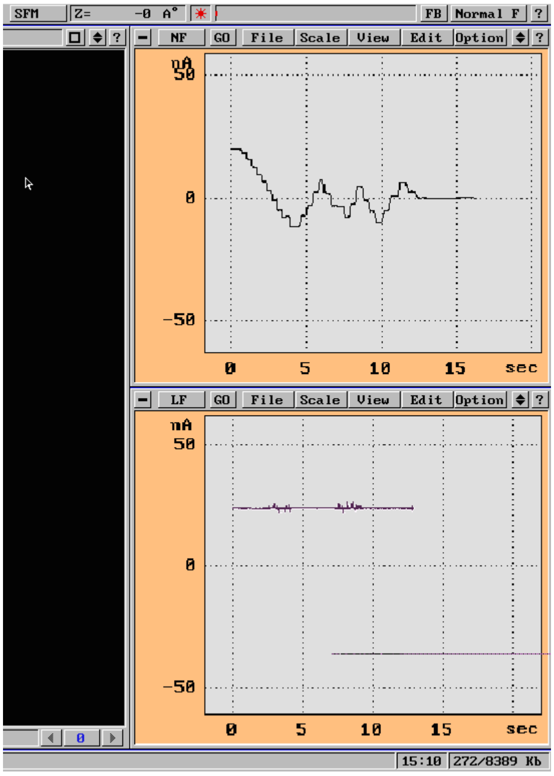


Fig. 191

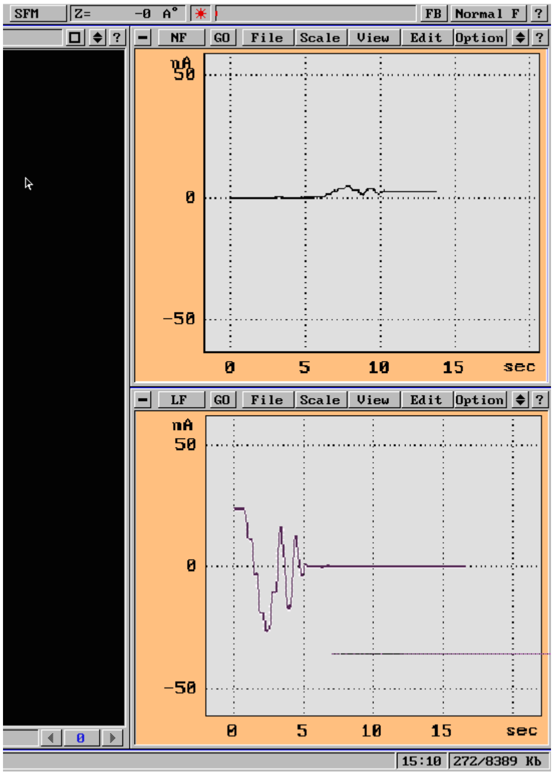


Fig. 192

3.3.4 Setting SFM Head

The installation of SFM head with a special table provided with a pizoelement for the semi-contact mode is done exactly in the same way as for a SFM head with ordinary table (see item 3.2.5).

3.3.5 Setting up parameters for the semicontact mode.

Further control over the unit's operation is done from the computer. To make the adjustment easier we recommend to set up the input terminals of the built-in oscilloscopes as follows: one will be used to register the feedback signal "FB" (Fig. 193) and the other will have an input for viewing two-dimensional data "Ext" (Fig. 194).

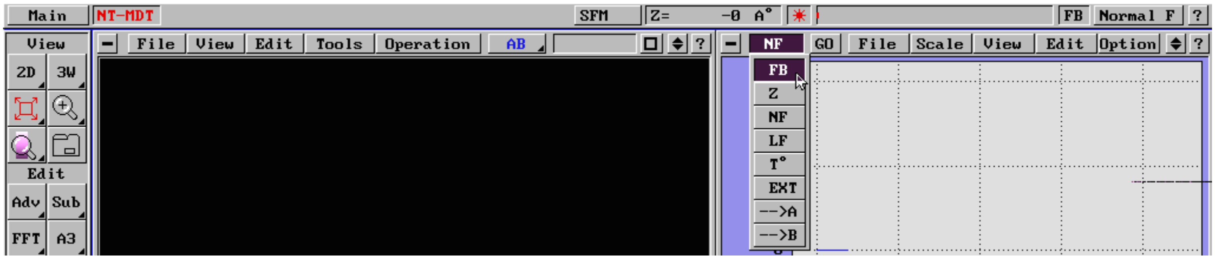


Fig. 193

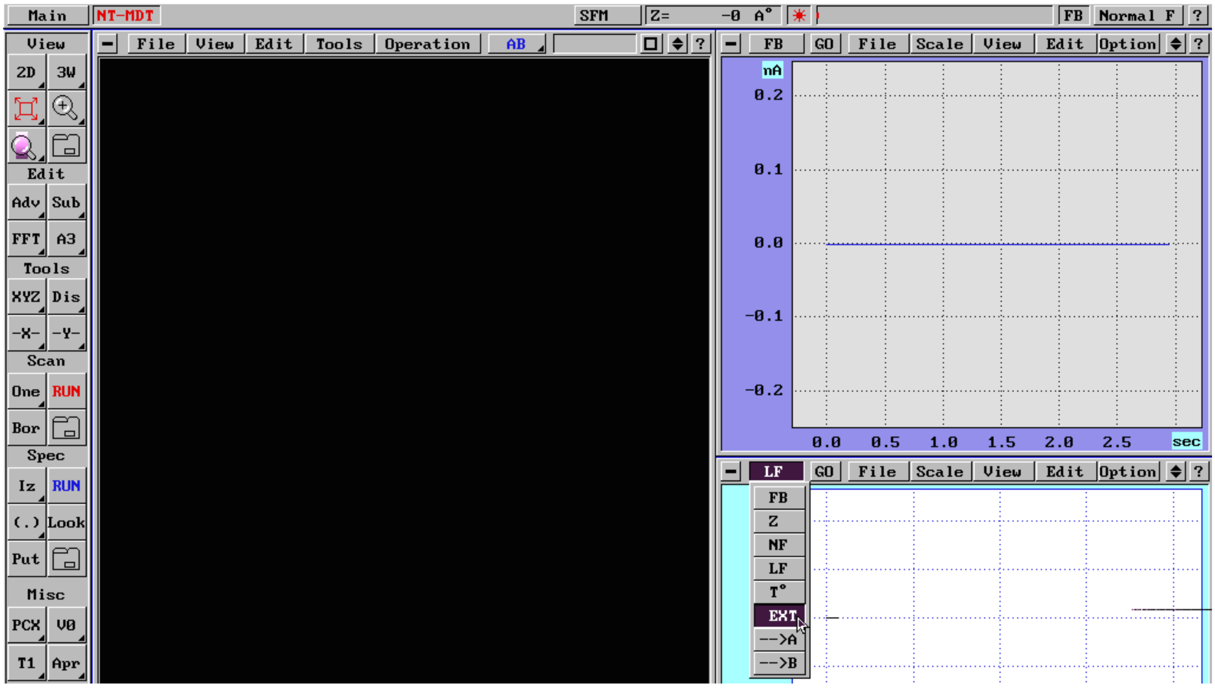


Fig. 194

Move the mouse cursor to the "Operation" button. Select "Scanning" in the opened menu (Fig. 195) and press the left mouse button.

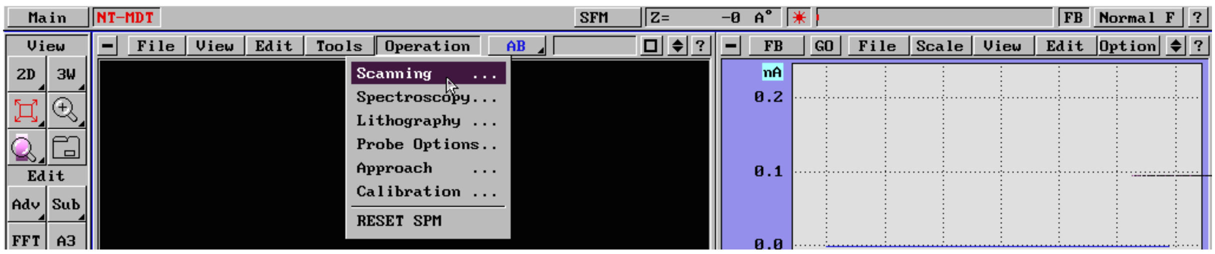


Fig. 195

In the “Scanning” menu set up the following parameters:
“Modulation” - Probe (Fig. 196);
“FB” - Module (Fig. 197) (feedback on the oscillation amplitude).

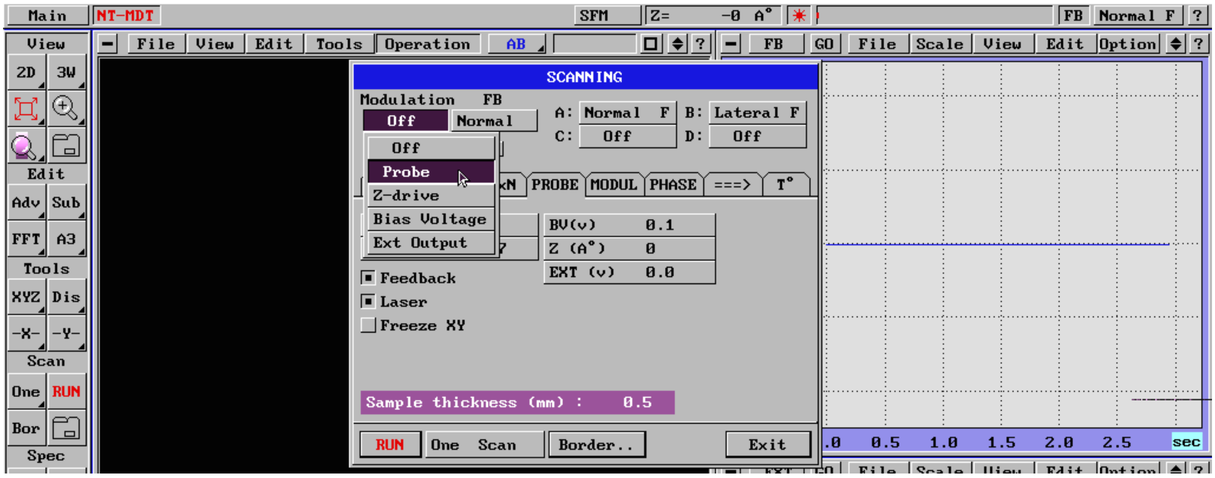


Fig. 196

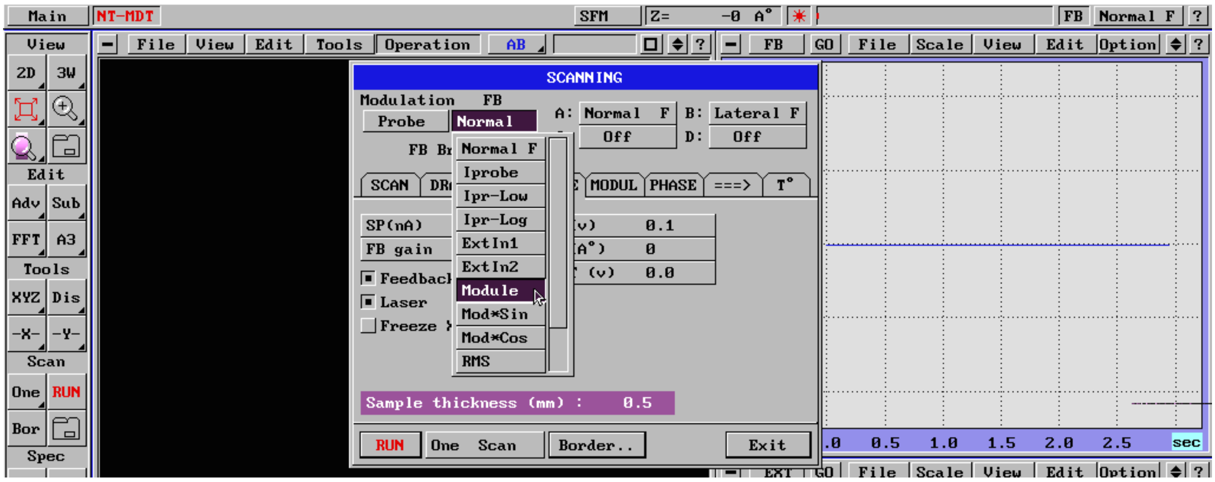


Fig. 197

In the “Scanning” menu select the “Modulation” label.

In the “Modulation” label we set up the following parameters (Fig. 198):

“Hi” - (amplifier band) if you want to use resonance frequency and the resonance frequency of the selected cantilever is over 55 kHz;

“Amplit” - 0.5x0.1 (amplitude of the mechanical oscillation generator)[7];

“Gain” - 3-15 (gain coefficient of the synchronous generator)[8];

“Frequency” - “Min” = 5;

“Max” = 1000 (frequency range to search for the cantilever resonance frequency);

“Numb” - set the required number of scanning points (500 are recommended).

---

[7] Point “1” of a generator amplitude is a special one: in this point the jump of amplitude is possible.

[8] The product of factors “Amplit” and “Gain” determines magnitude of a signal “Module”, by which the feed-back is in this case supported, but these factors have a different nature and different character of noise; it is necessary to set small factor “Amplit” (0.05 - more often 0.5), and by factor “Gain” to set a necessary level of a signal “Module”.

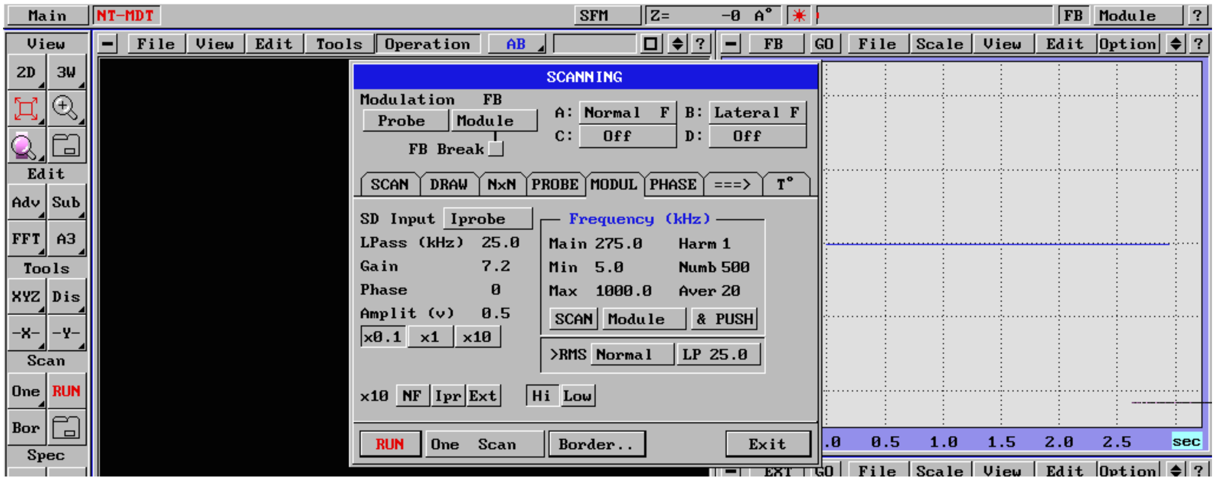


Fig. 198

Run the scanning - “SCAN” (Fig. 199).

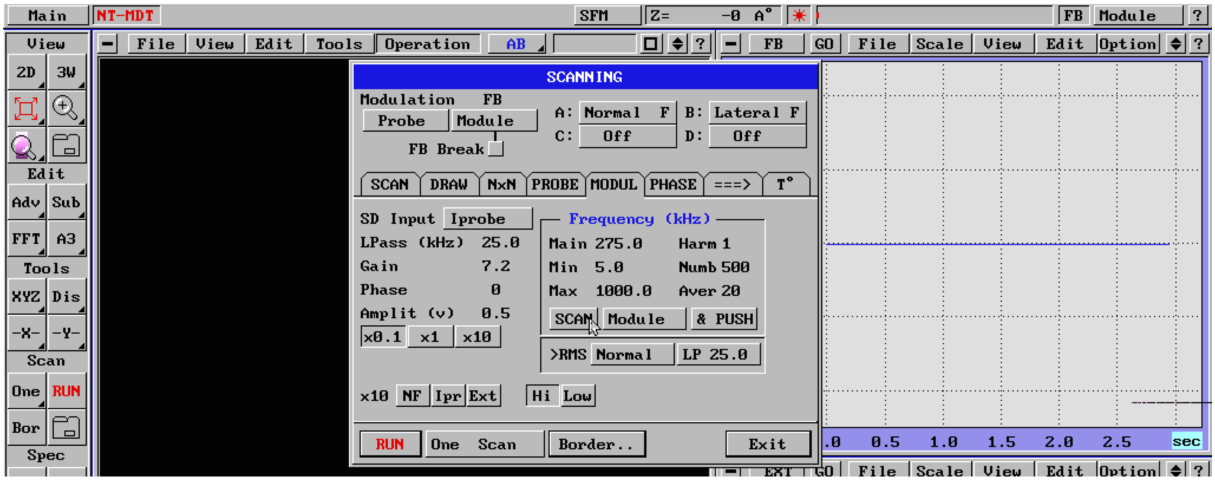


Fig. 199

A curve is formed as a result of scanning (Fig. 200), it shows that there is a big resonance on a particular frequency.

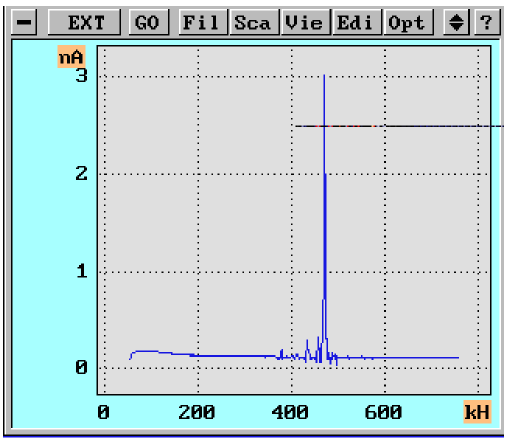


Fig. 200

Then this resonance should be viewed in more detail and to do this set the interval between “Min” and “Max” as close as possible but so that the resonance will fall within it.

Then start the scanning once again - “SCAN”. As a result of the scanning you will get a more detailed curve with resonance. This operation is to be repeated the number of times required to determined the exact resonance frequency (Fig. 201).

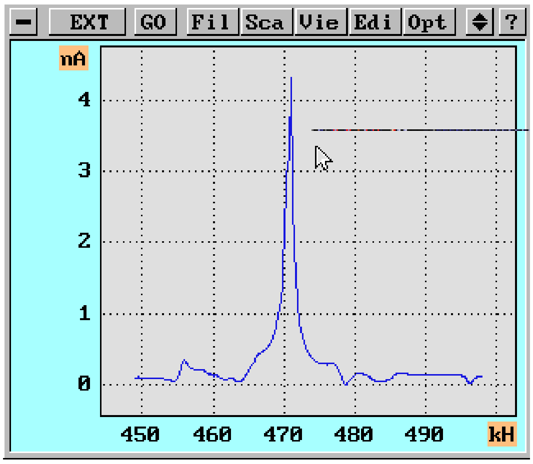


Fig. 201

Set the found resonance value in the “Scanning"-"Modulation"-"Frequency"-"Main" menu, this will be your working frequency (Fig. 202).

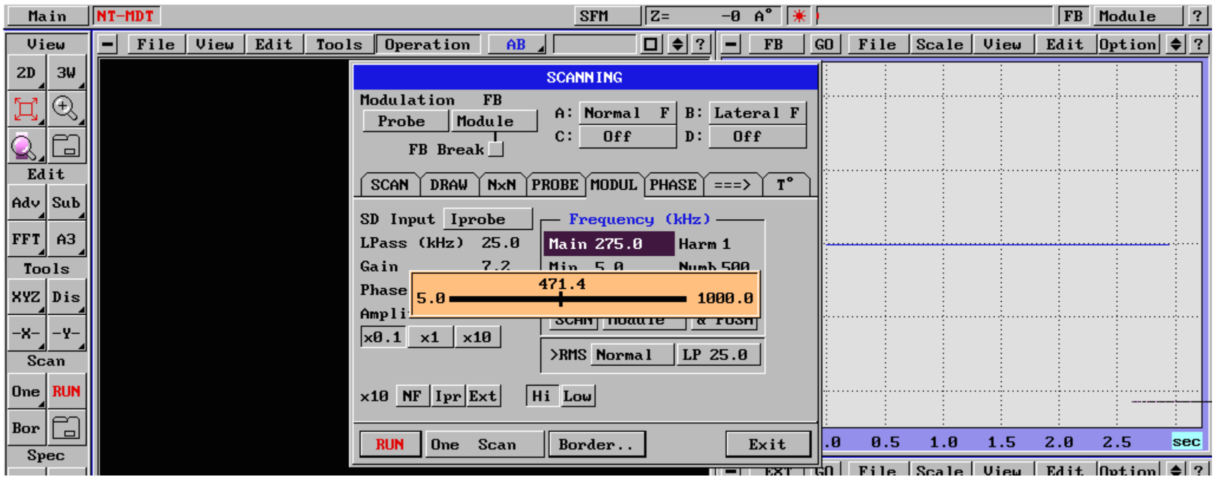


Fig. 202

Then using amplitude (“Amplit”) and gain (“Gain”) set the initial level of the oscillation amplitude. Based on the readings of the upper oscilloscope showing the level of the feedback signal response, the initial level of the oscillation amplitude must be selected so that it is not over 10 and not less than 3 (since the working value of the amplitude may lower than one third of the initial one). The values for the amplitude and gain are set up on individually for each specific case and material, they are determined by experimental matching (it is better to set a lower amplitude and a higher gain).

3.3.6 Automatic approach of sample to probe

Having closed the “Scanning” menu, enter the “Approach” menu, the "Probe" label (Fig. 203).

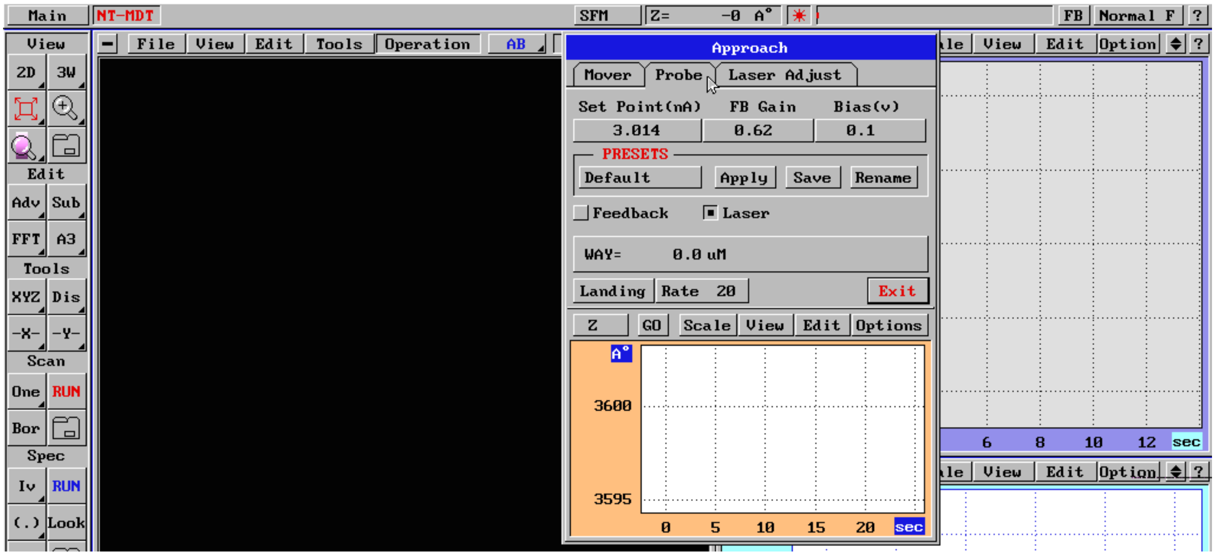


Fig. 203

Set up the following parameters: the “Set Point” value should be slightly lower than the initial oscillation amplitude level (i.e. closer to “0”, for example, if the initial level is 4.5, the “Set Point” level must be set to around 4.0, the “Feedback Gain” to 10 (Fig. 204).

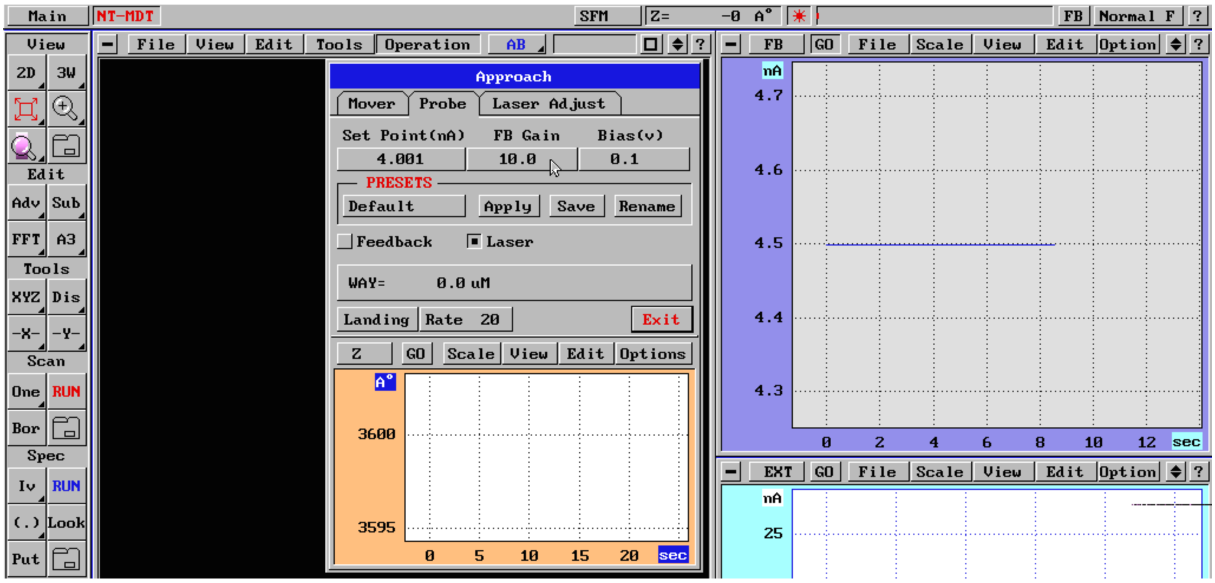


Fig. 204

Now begin to approach the sample.

Set up the maximum "Landing Rate" (Fig. 205).

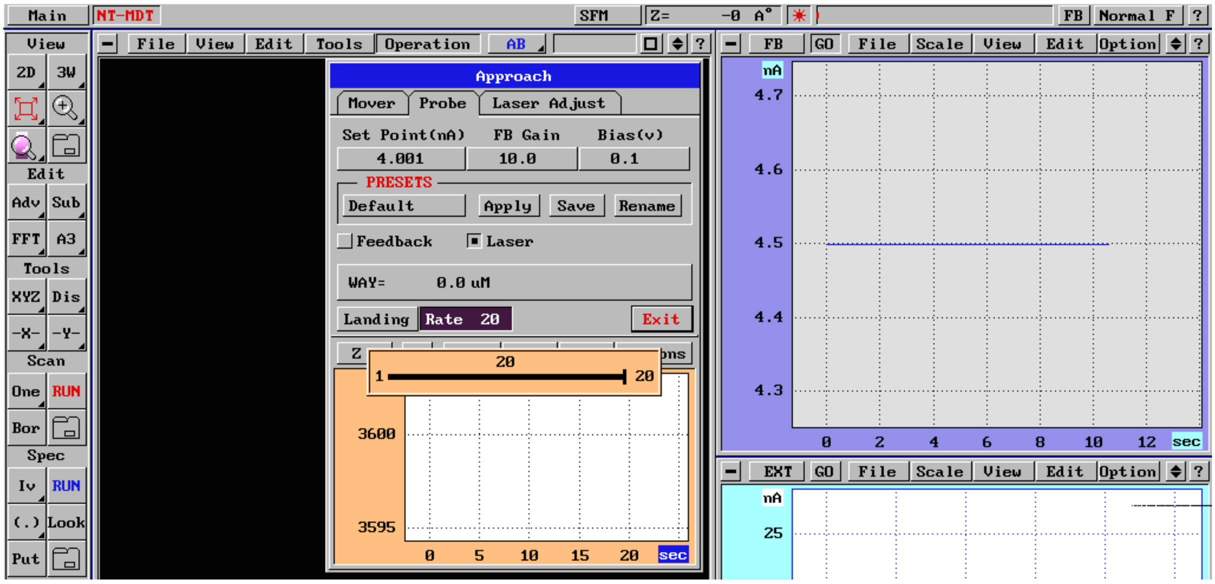


Fig. 205

Enable “Landing” (Fig. 206).

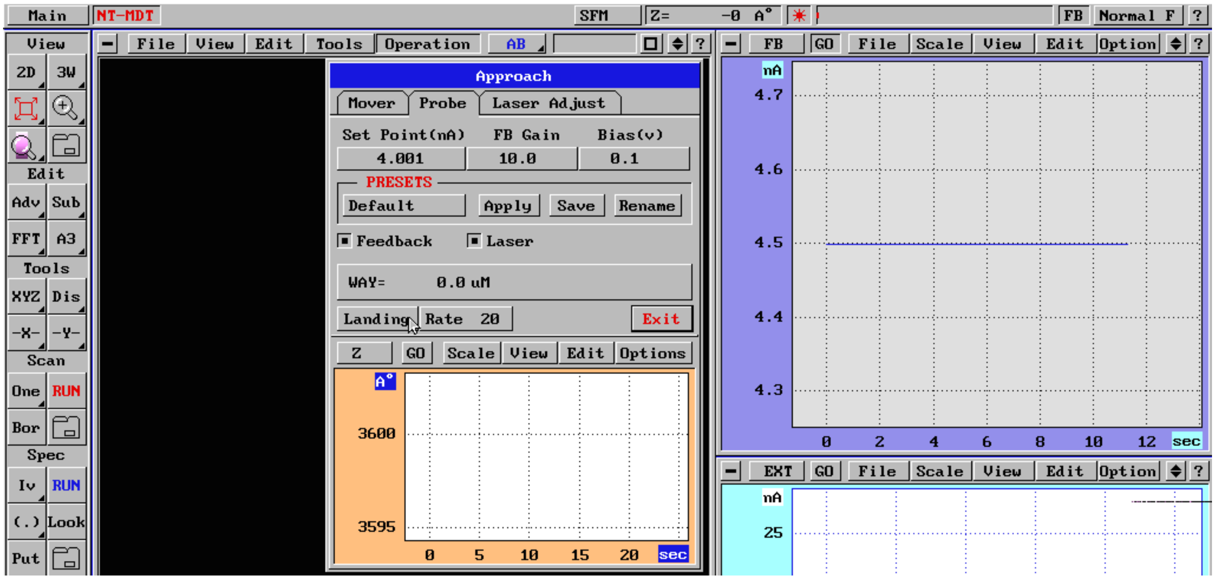


Fig. 206

The level of the oscillation amplitude will gradually decrease and will become equal to the value set up in the “Set Point”. After enter a new value in the “Set Point”. This value is selected based on the following: when you gradually increase the “Set Point” value, a red bar showing the scanner move-out starts growing in the scanner extension analog indicator. Finally, at a certain “Set Point” value this bar will go beyond the limit of the analog indicator and this value should be left for future operation (Fig. 207).

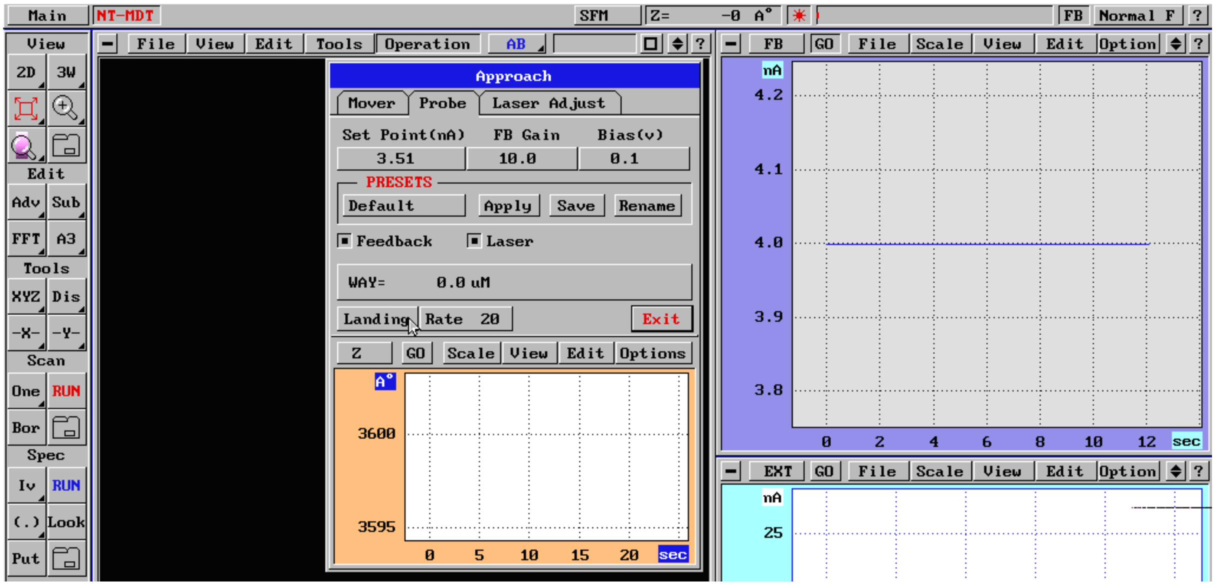


Fig. 207

Start "Landing" once again. The level of the oscillation amplitude will gradually decrease and will become equal to the value set up in the "Set Point". These two operations must be performed several times until such time when increasing the "Set Point" value stops making the scanner move-out bar in the analog indicator grow. This is the working point.

This procedure can be explained as follows: when the oscillating probe approaches the surface its oscillation amplitude goes slightly down due remote interaction with the surface, but this decrease is small and it is very difficult to operate maintaining this value. When approaching further the probe starts striking the surface and its oscillation amplitude is limited by the contact with the surface. In this case any slightest change in height (Z coordinate of the scanner) results in substantial changing of the oscillation amplitude.

In this mode it is possible to evaluate the real cantilever oscillation amplitude. To do this the point must be noted where with increasing the "Set Point" value the movement of the red bar in the analog indicator from left to right will sharply slow down (almost stop). Memorize the Z coordinate and the "Set Point" value at this point. Then decrease the absolute value of the "Set Point" (for example, from -2.0 to -1.0) and note for how much the Z coordinate has changed. This is roughly a half of the cantilever oscillation amplitude.

3.3.7 Getting a test scan

Operations of setting scan parameters, getting image, and its processing are executed in "SPM" window in the left part of the screen. Point the button "Operation" to open corresponding menu and then click the item "Scanning" (Fig. 208) to open its menu. The scanning parameters are entered in the displayed window:

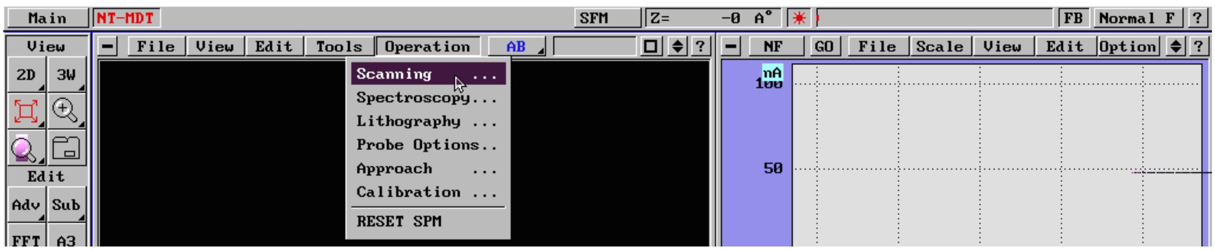


Fig. 208

3.3.7.1 Getting scan of a test grating

Set the following values of parameters:

Modulation - Probe (Fig. 209)

FB - Module (Fig. 210)

Scan A - Height (Fig. 211)
Velocity - 60000 A/ñåê (Fig. 212)
NX xNY - 128 õ 128[9] (Fig. 212)
Step (A) - max[10] (Fig. 212)
NL Corr - on (Fig. 212)
Scan dir - +X+ (Fig. 212)
Gain = 1 (Fig. 212)
Lpass(kHz)=10 (Fig. 212)
Average = 10 (Fig. 212)
Drawing - on (Fig. 213)
Subplane - on (Fig. 213)
Show scan lines - on (Fig. 213)
SP - by results of approach (Fig. 214)
FB Gain - by results of approach (Fig. 214)

Select the single scanning mode - One scan (Fig. 215).
Click RUN to start scanning (Fig. 216).

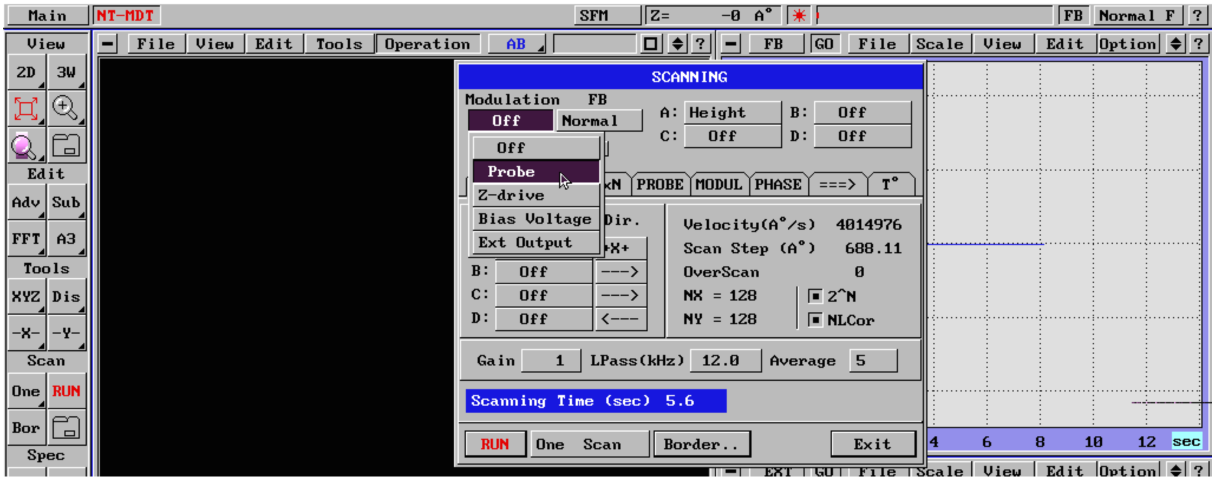


Fig. 209

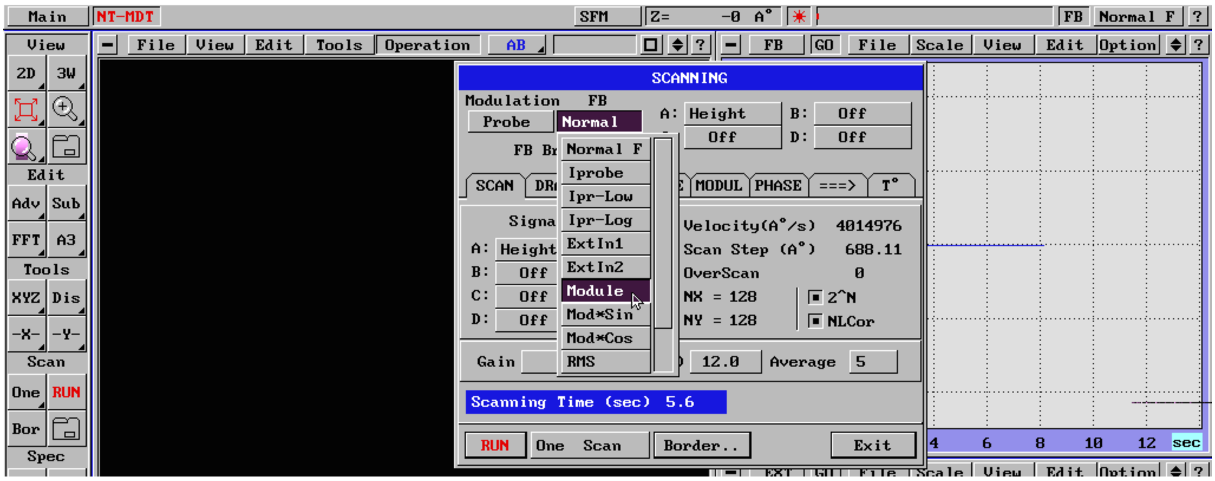


Fig. 210

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9 If the software does not let you increase the scan size up to 128x128, then the scanning step "Step" should be decreased at least by two times. After that increase the scan size again.

10 First set the scan size NXxNY at a small step of scanning and then set the biggest step size possible for this number of steps.

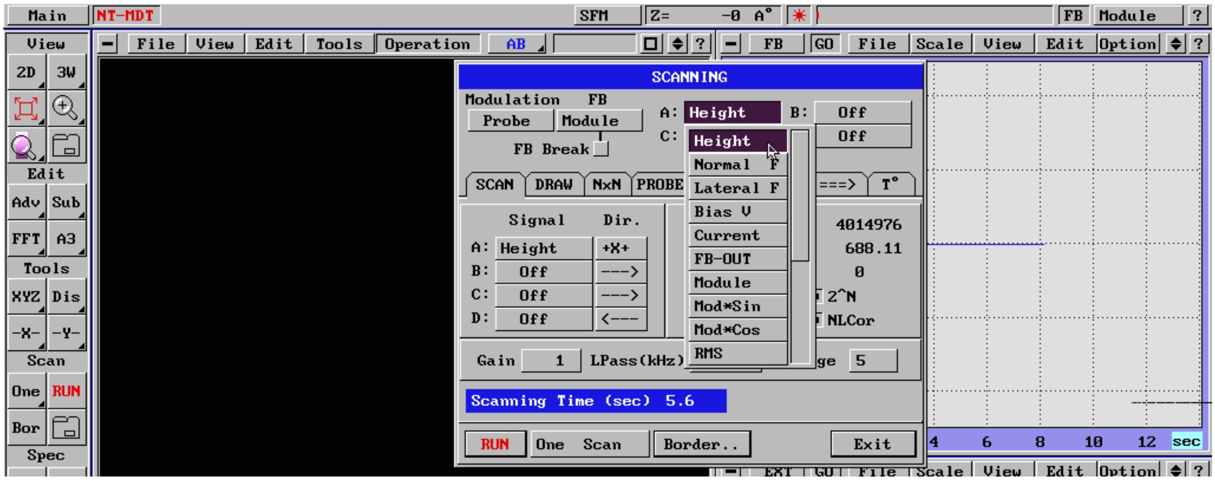

Fig. 211

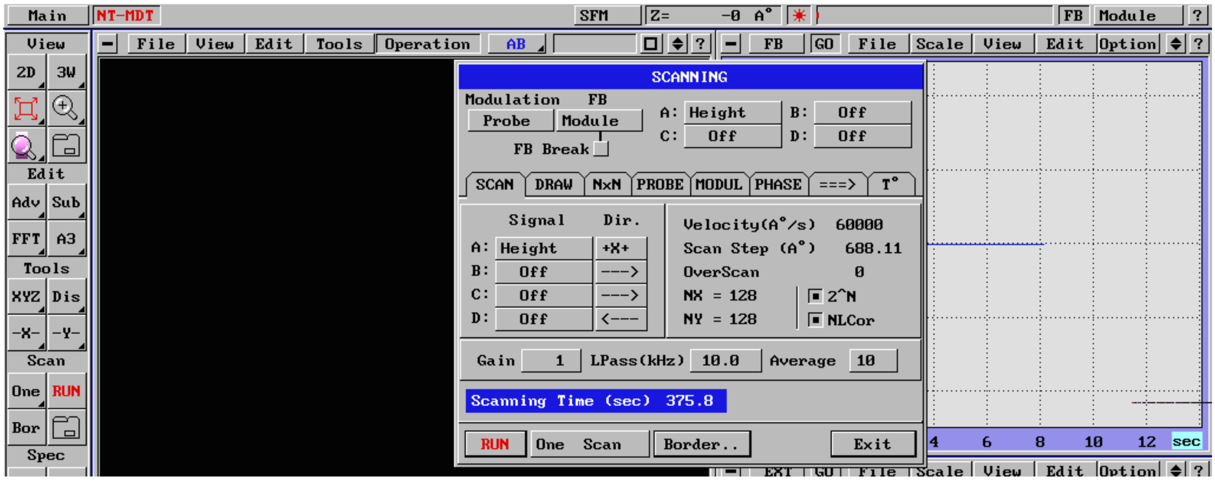

Fig. 212

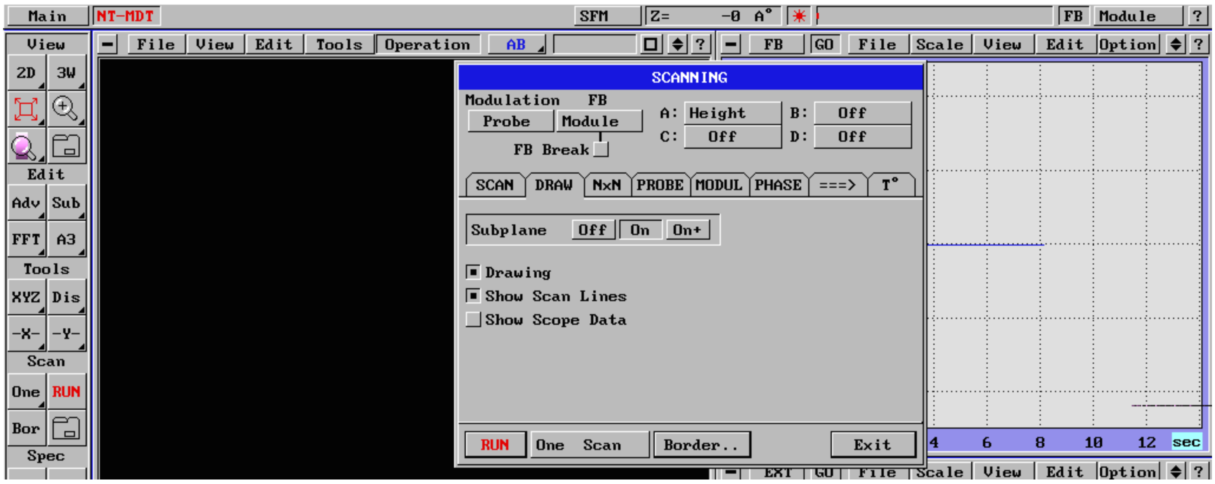

Fig. 213

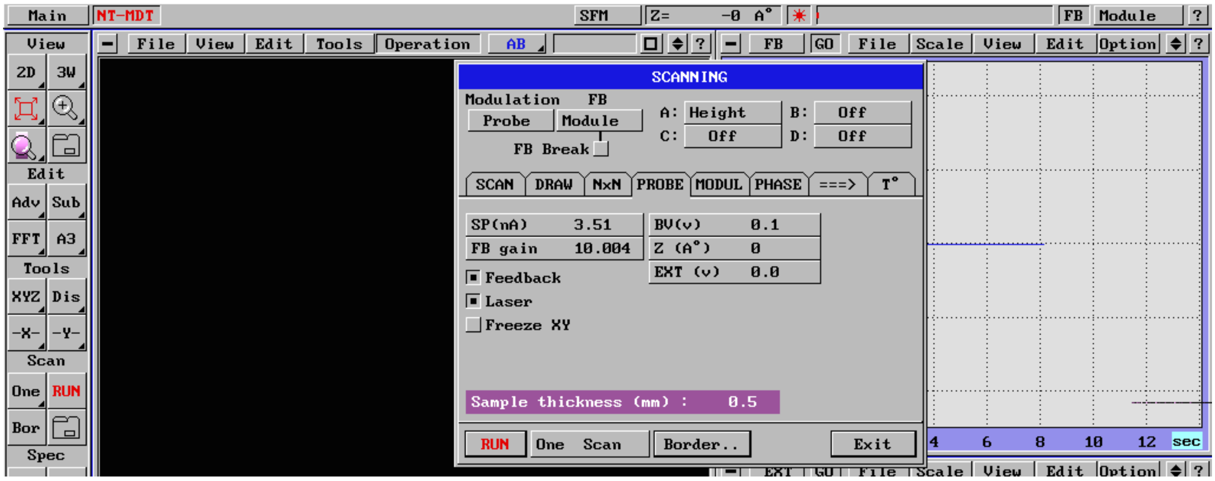

Fig. 214

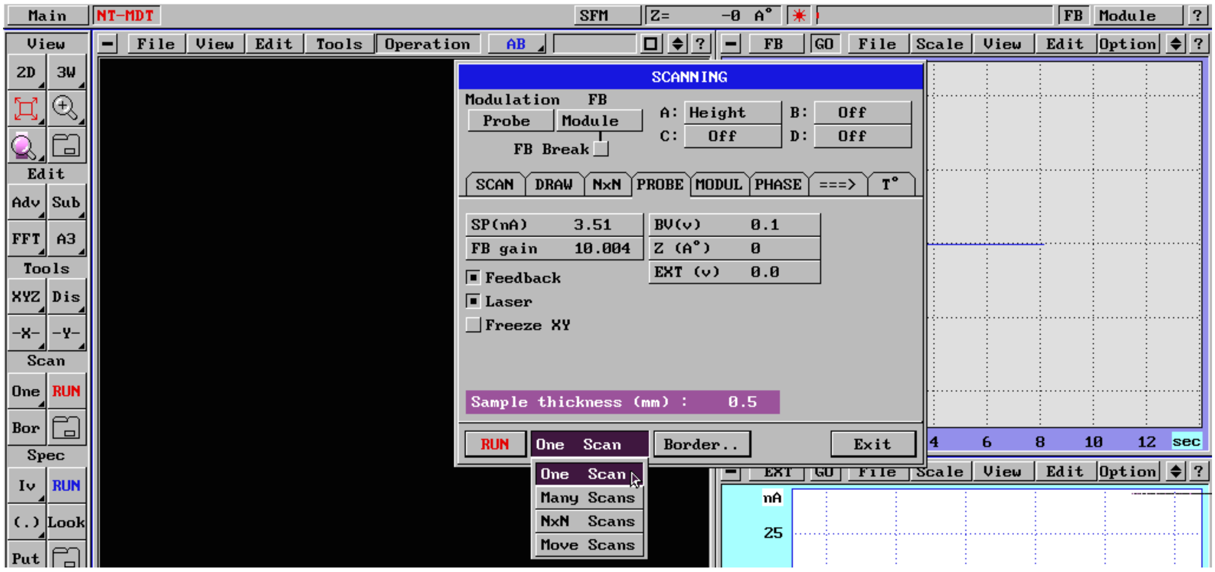

Fig. 215

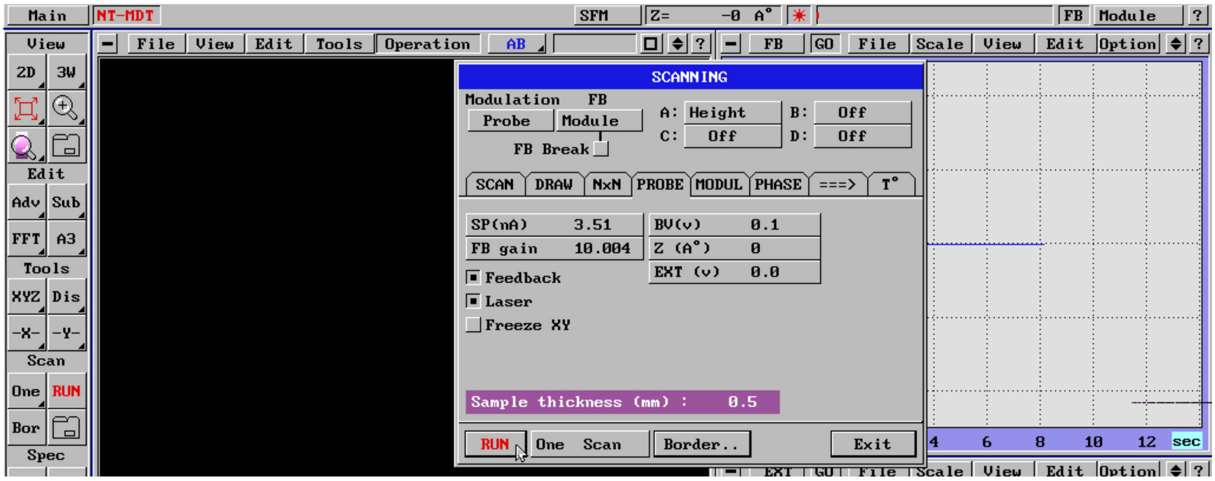

Fig. 216

After the first scan lines try to modify slightly the "Set Point" parameter in the menu displayed in the upper part of the screen in the course of scanning, while watching the changes in the scanning results using the data for each scanning line displayed in the OSC window and execute the

Restart command. Perform these operations several times until you find such "Set Point" value when the sharpest surface picture can be seen.

3.3.8 Switching over from semicontact to contact mode

To switch over between any modes it is necessary to turn off the feedback, set the new signal to the feedback input, determine the current value of this signal and set a new "Set Point" value in accordance with this value and only then the feedback may be turned on.

Turn off the feedback, disable the modulation, switch the feedback input over to "Normal Force" and turn one of the oscilloscopes on for measuring the "FB" signal. Set the "SP" parameter slightly (3-5 nA) higher than the current value of "Normal Force" signal. Now you can enable the feedback and continue the operation in the contact mode.

3.3.9 Turning off the microscope

The microscope is turned off in the same way as in the SFM mode (see 3.2.9).

3.3.10 Selecting parameters for the semicontact mode

- When approaching the sample to the probe the "Module" signal generation can occur. Try to determine more precisely the point where the cantilever touches the surface. Often generation does not occur immediately after touching but only after the "Set Point" parameter is decreased further (its absolute value). Should generation occur immediately after touching, it is necessary to further decrease the "Set Point" parameter (its absolute value). At a specific point the generation will stop. You can decrease the "Set Point" parameter slightly more to ensure a better stability but precaution should be taken to prevent the "Module" signal from spontaneous resetting to zero.
- If the cantilever, however, resets to zero try to increase the amplitude of the mechanical oscillation generator. To do this disable the feedback, enter the "Scanning" menu, "Modulation" label and, while watching the "NF"[11] signal on the turned-on built-in generator, increase slightly the amplitude of the mechanical oscillation generator - "Amplit". After this decrease the "Gain" parameter to keep the signal at the previous level. Enable the feedback and try once again to select a parameter for the "Set Point". It is likely that you will have to move slightly out and in (500-700 nm on the counter).
- If in the course of scanning a very sudden drop occurs along the Z coordinate it can mean that the phase changing has an effect on the "Module" signal change. In this case the "Amplit" parameter must be decreased.
- When working with soft materials artifacts can occur in the scanning direction on surface elevations. In this case the scanning rate and the holding force must be decreased (while increasing "SP").
- The scanning rate should also be decreased if an elevation slope on the side of a hole is not scanned well.

3.4 Two-pass technique (lift mode)

In order to investigate the remotely acting forces (as opposed to Van-der-Vaals forces) such as magnetic or electrostatic it is useful to employ the two-pass technique or the lift mode.

In our unit it is possible to set up not only two-pass but also triple- and four-pass techniques if necessary.

Let us describe the simplest way of using the two-pass technique. Naturally, appropriate probes are necessary to register remotely acting forces (for magnetic forces magnetic cantilevers will be required and for electrostatic forces cantilevers having high dielectric penetrability). It would be better if such cantilever is suitable for operation in the resonance mode as well i.e. has resonance frequency in the range of 100-2000 kHz.

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[11] Actually "NF" signal on the built in oscilloscope shows the signal by which the feed-back is being maintained: for STM it is a real tunneling current, for Contact Mode AFM it is a photodiode unbalance cu r-rent or a normal force of cantilever; in this case it is amplitude of oscillations of the cantilever.

The approaching to the sample and getting of the first topography samples is done in the semicontact mode (see item 3.3). When selecting options of the semicontact mode it is necessary to take into account that such parameters of the modulation technique as the amplitude of the mechanical oscillation generator and gain of the synchronous detector could be both increased and decreased.

After having selected parameters for the scanning mode to receive a stable topography picture it is necessary to set up parameters for the lift mode. Set the SPM window in the two image mode (Fig. 217). Set the topography registration mode in window A (Fig. 218). Set in window B (Fig. 219) the registration mode of amplitude (“Module”, “Theor mod”) or signal phase changing (“Mod-sin”, “Mod-cos”, PH1 or PH2).

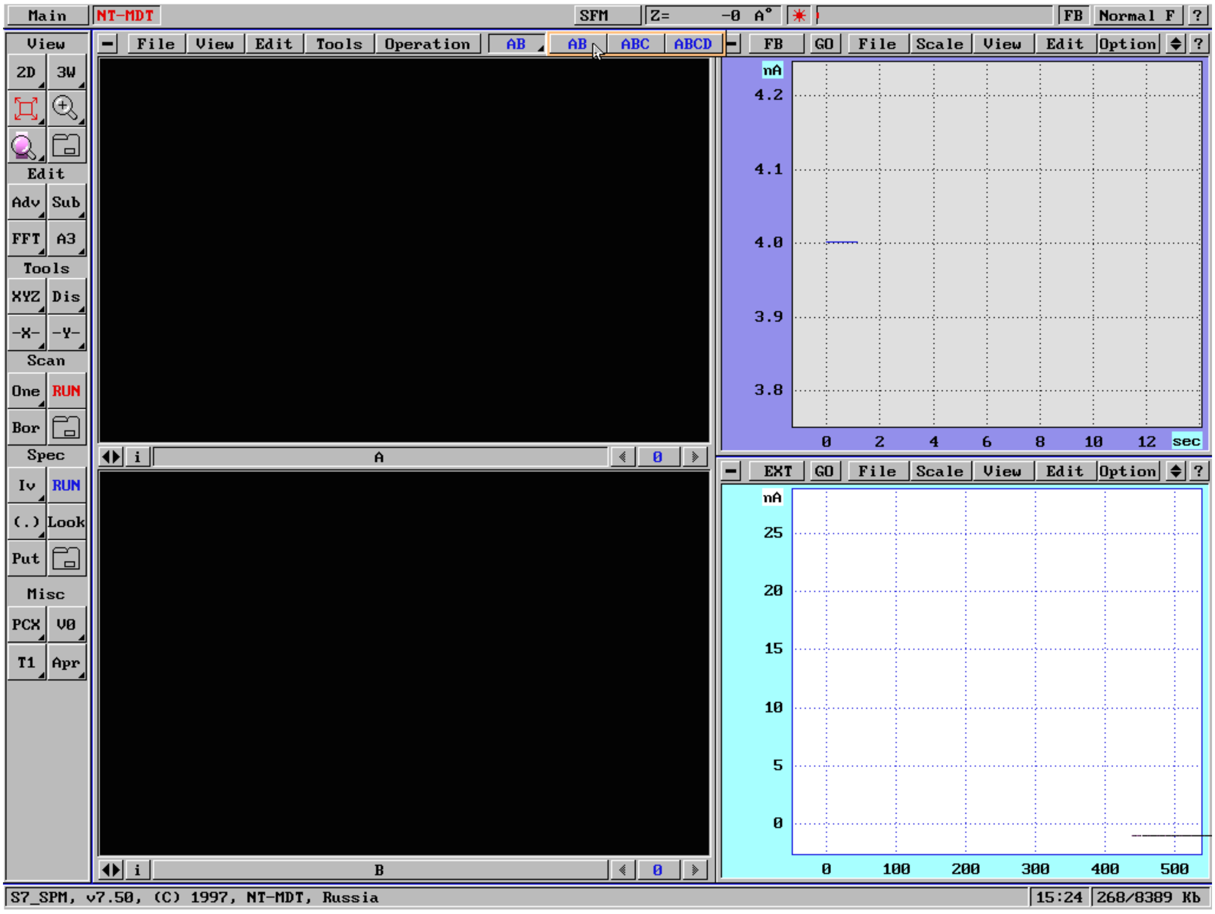

Fig. 217

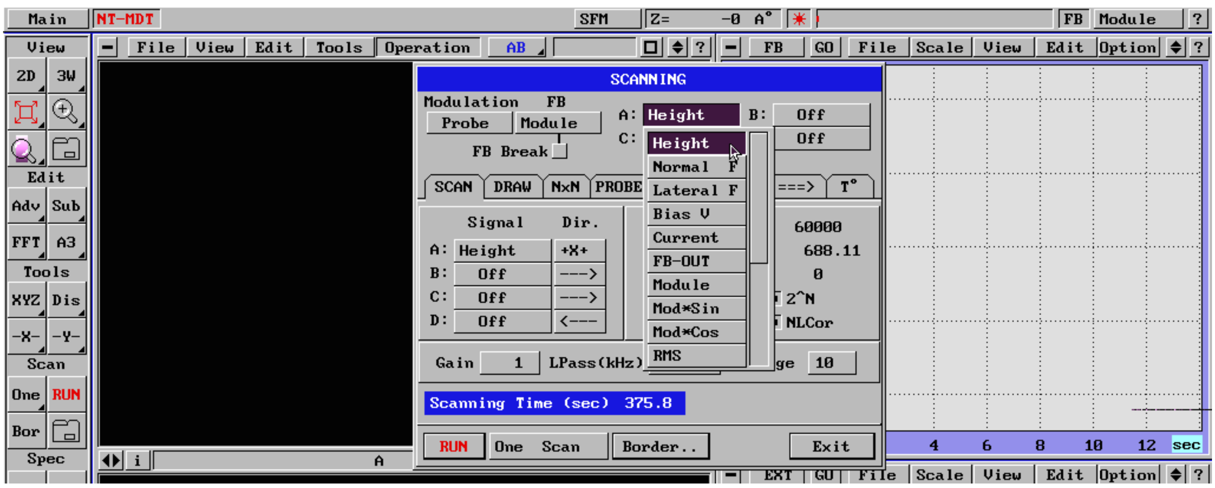

Fig. 218

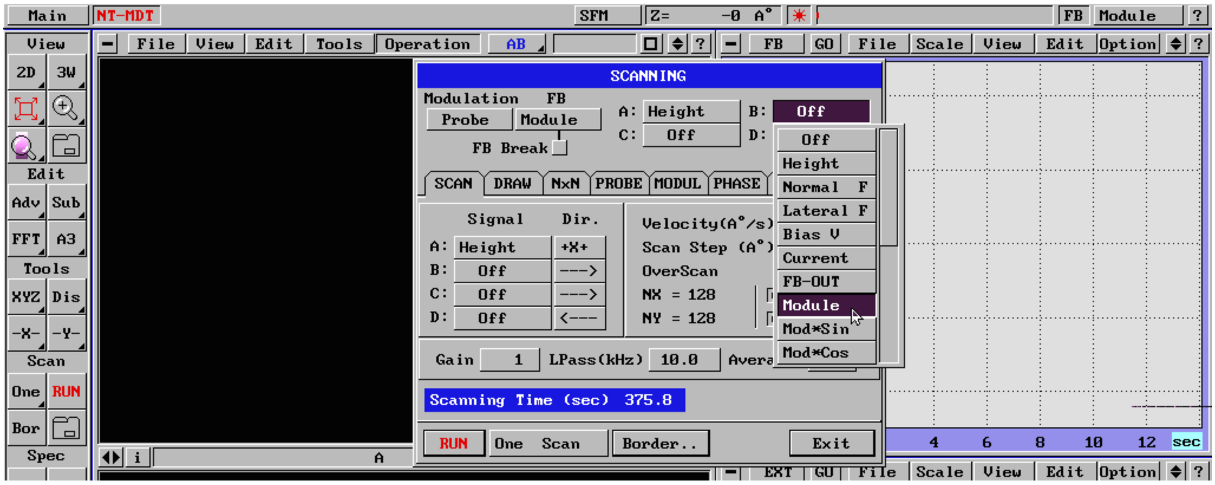

Fig. 219

Select "=>" in the movement direction selection menu in the second window (Fig. 220). This means that after taking one line of the topography image the probe returns to the initial point of the line and passes it again with the feedback disabled, and the height of the probe pass over the surface is controlled by the central processor on the basis of the already taken topography data and the signal selected by you is measured and registered in this window (amplitude or oscillation phase). It is not recommended to take the signal at the reverse pass "<=" immediately after the topography measurement - the system may not have had enough time to set itself up for other parameters.

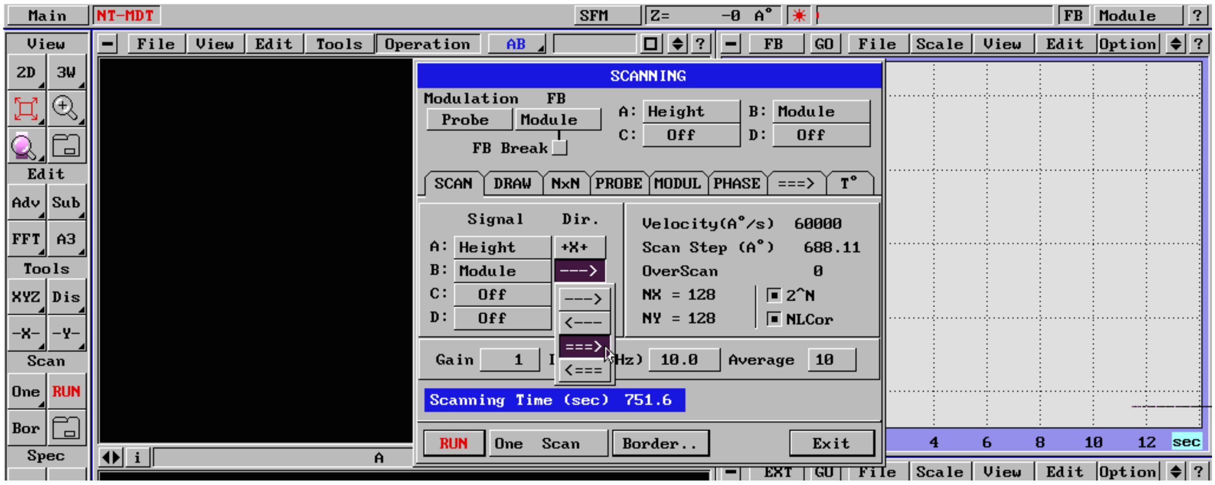

Fig. 220

When selecting the phase change registration make sure the signal is close to zero. To do this set up the “À →” (Fig. 221) input in one of the windows of the internal oscilloscope. In this case the signal frequency selected in the scanning menu will be sent to the input terminal of the internal oscilloscope. Set in this menu the signal you want to register, for example “Mod-sin” (Fig. 222). While watching the signal in the internal oscilloscope set the signal to zero using the “Phase” (Fig. 223) parameter.

Fig. 221

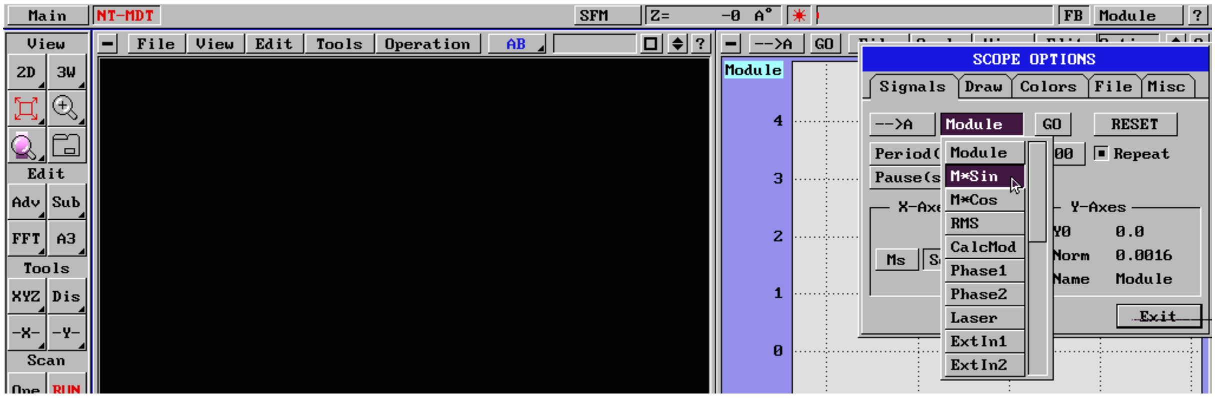

Fig. 222

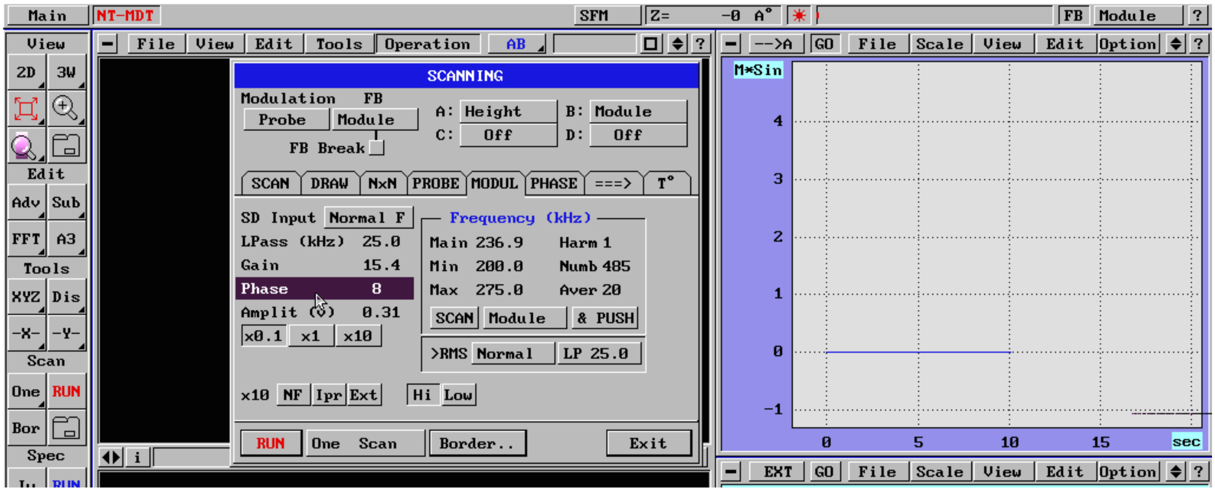

Fig. 223

Having enabled the scanning (Fig. 224) pay attention to the menu displayed in the upper lines of the screen during the scanning. The parameters on the left of the “?” item relate to the lift mode. For the second pass (in the lift mode) it is possible to specify height shifting “ΔZ”, increase or decrease the amplitude of the mechanical oscillation generator “Ampl”, increase or decrease the gain of the synchronous detector “Gain”, detect the second signal with frequency “ΔF” or phase “ΔĐ Í ” shifting or event on a different harmonic. The best image of the investigated forces is received with the right selection of these coefficients.

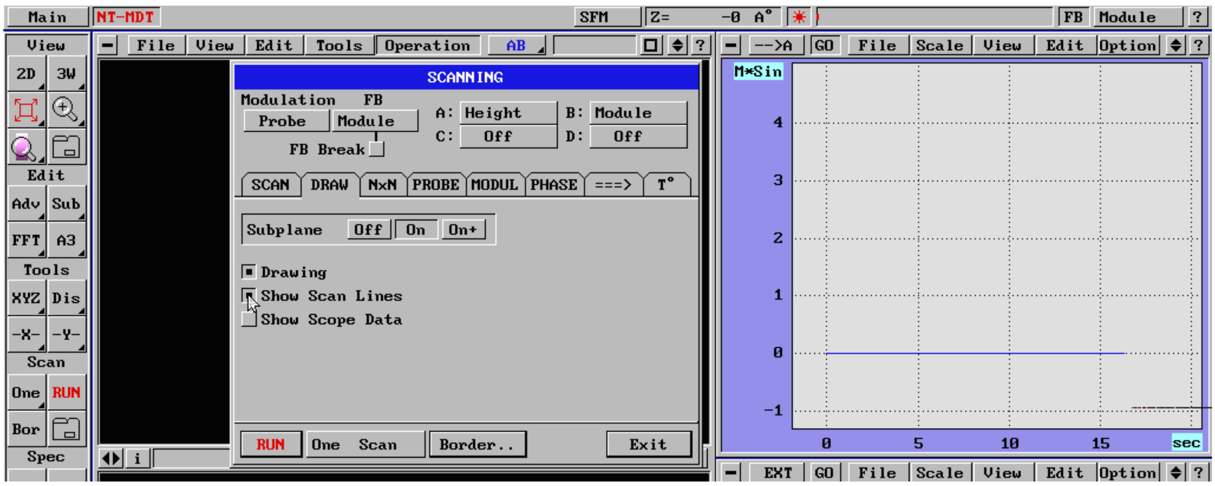

Fig. 224

Two simplest methods:

- move the probe away from the surface ΔZ ~ 1000Å, increase "Gain" and oscillation amplitude " Ampl"
- decrease the oscillation amplitude and increase gain and approach the probe to the surface (ΔZ < 0).

While the parameters are being selected scanning may be suspended by selecting the "Pause" menu item. The scanning in this case will be done along one line.

In this case the scanning process can watched in the internal oscilloscope window (if prior to the scanning the "Show scan lines" option has been selected in the "Draw" parameters), switching the output of scanning lines to the oscilloscope window from different scanning windows with the following keys "Ctrl - À", "Ctrl - B" etc.

3.5 Carrying out lithographic operations

Lithographic operations are designed to change topography or surface properties in scales comparable to the probe dimensions.

Depending upon the operation mode lithography can be carried out by two methods - by applying a short pulse (≅15 mcs) or a series of pulses at voltage up to 10 V between the probe and the sample - to operate in the STM mode or with a conductive cantilever; or by "scratching" the surface by the probe - for non-conductive surfaces and probes).

Lithography is done using the scan that has just been taken (scanning parameters cannot be changed after taking the scan and before performing lithography). The lithography parameters are set up in the "Operation" - "Lithography" - "Options" menu (Fig. 225).

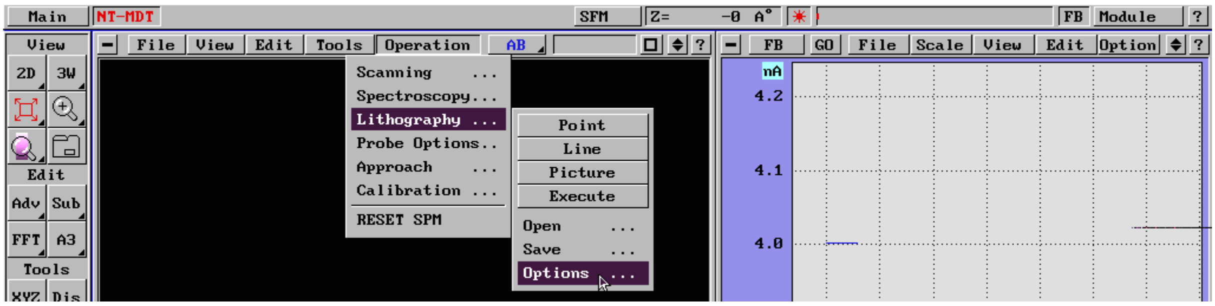

Fig. 225

Here you can specify the voltage to be applied to the sample in relation to the probe "Voltage" (Fig. 226), number of pulses in a series to be performed in a single point "Number" (Fig. 226).

The "Z-shift" parameter (Fig. 226) determines the execution of one of the above methods:

– if "Z-shift">0 - the probe is removed to the given distance and a voltage pulse is applied;
– if "Z-shift"<0 - voltage is applied, the sample is moved forward to the given distance increasing the force of interaction between the sample and the probe.

The "Rate" parameter (Fig. 226) determines how big is the pause between pulses and "Step" (Fig. 226) determines the step of the probe movement in relation to the scanning step (1-lithography in every scanning point, 2-in every second point etc.).

The option "Inp - on" means a cycling, constant voltage otherwise moves.

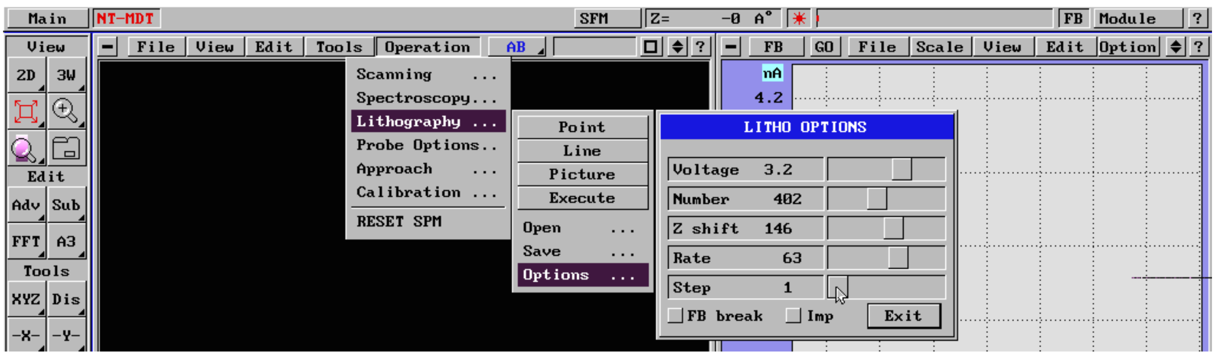

Fig. 226

Lithography can be performed both in a single point and along a line or a picture. The “Point” (Fig. 227), “Line” (Fig. 227), “Picture” (Fig. 227) commands are executable commands. When the appropriate buttons are pressed the user is prompted to select a point, a line or a picture to be lithographed.

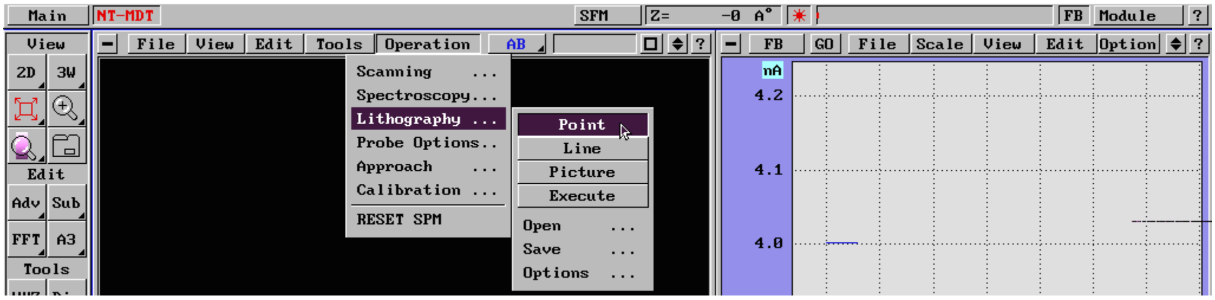

Fig. 227

The picture that has been lithographed can be saved in a separate file - “Save” (Fig. 228) and then loaded and compared with the result or lithography can be performed once again - “Execute” (Fig. 229). With “Number” = 0 lithography will not be performed and then you can simply save the picture in a file.

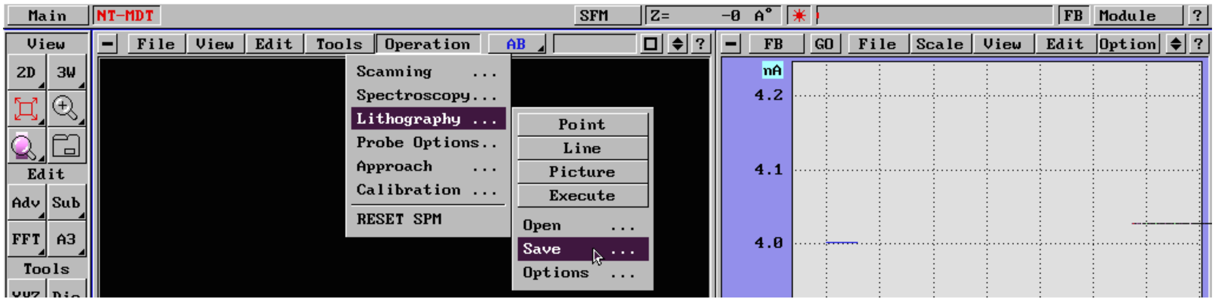

Fig. 228

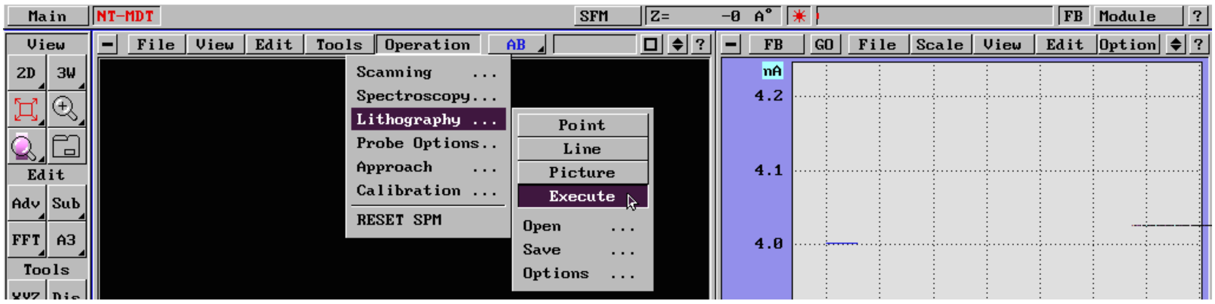

Fig. 229

If correct parameters have been selected (according to the probe and the material) it is possible to view the result by repeated scanning after the lithography has been performed.

One can consider as a lithography method the switching over from semicontact to contact mode and back (to work with soft surfaces). Such method, for example, is often used to determine the thickness of a soft film applied to a hard surface (LB-film on graphite).

To do this let us make a sufficiently large scan (1x1 micron) using the semicontact method. The semicontact mode parameters can be saved on the hard drive with the “Main” - “Options” - “Save as” command (Fig. 230).

Fig. 230

After this we switch over to the contact mode (see item 3.2.7) and before starting scanning using the “Border” - “Relative” command (Fig. 231) we will select an area in the center of an area scanned with a smaller size - 200x200 nm (the size can be modified by changing the scanning step in the “Border” mode by moving while holding the Shift key).

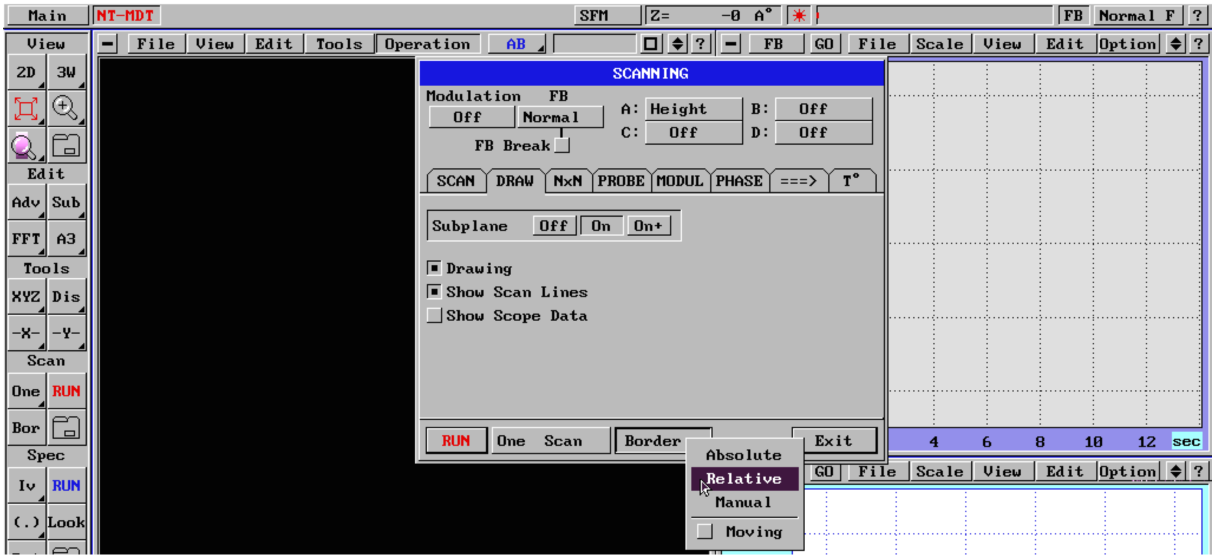

Fig. 231

To make sure that the pressing force is sufficient you can increase the “Set Point” by 3-5 nA. Scan the selected area in the contact mode. Most likely, you will not obtain any satisfactory result i.e. picture. After this switch over to the semicontact mode (see item 3.3.7) load the parameters you have saved and after having displayed the first picture (size of 1x1 micron) in the “Border” - “Relative” set once again the same scanning field. When scanning you are to obtain a result similar to the attached one. Using the scan cross-section obtained by the “Tools” - “X-section” command you can determine the film thickness.

4. Trouble-shooting

4.1 In STM mode

¹	Symptoms	Possible cause	Remedy
1.	The indicator on the microscope is not lit.	The microscope is not connected to the power supply. A fuse has blown. The microscope is out of order.	Connect the microscope to the power supply. Replace the fuse. Contact your dealer.
2.	When microscope is turned on the device does not respond to the program's control commands (digital indication of the scanner position does not change, the step motor does not rotate).	The microscope was turned on after the program's start and the command of the electronic circuits Reset to the initial position has not been executed. The microscope is not properly connected to the computer, the connecting plugs are not fully inserted. There is power failure. The interface board is out of order.	Execute the Reset command as indicated in the User's guide. Turn the microscope off. Check if the device is properly connected. See point 1. Contact your dealer.
3.	When the feedback is turned on without approaching the sample trembling is observed along Z (over 100 A).	Too low voltage in the power supply system.	Check the voltage in the supply line.
4.	In the process of the sample automatic approach the motor does not stop and the sample collides with the tip.	No U_T voltage at the sample: a) poor contact between the sample and the spring contact; b) the 'one-pole' plug U_T is not inserted into the socket at the scanner unit; c) the U_T cable plug is not inserted in the appropriate socket on the board. The U_T value equal to 0 has been specified in the Options. Too high value for I_T has been specified in the Options. Too low value “FB Gain” has been specified. The sample is not conductive.	a) check the contact; b) insert the plug into the socket; c) insert the cable into the U_T socket on the board; Set the U_T value as indicated in the User's guide. Set the I_T value as indicated in the User's guide. Set the “FB Gain” value as indicated in the User's guide. Change the measurement mode for this sample to the STM mode.
5.	Significant drift of the sample with respect to the tip is observed after approaching.	The scanner is not hung horizontally and the head slides slowly on the surface. The substrate with the sample is not properly inserted into the holder.	Use the adjusting knobs of suspension system to bring the scanner into horizontal position within ±3 degrees. Re-insert the substrate into the holder.

6.	After the sample is approached autooscillation at about 0.5...2 kHz is observed at the oscilloscope that disappears when the "FB Gain" value is reduced (see the User's guide).	The substrate with the sample is not properly inserted into the holder (it is supported only by two balls). The tip is not properly inserted into the STM head clamp.	Re-insert the substrate into the holder. Check if the tip is properly clamped in the holder. If necessary re-install.
7.	When the I(U) - curve is measured it has a rectangular form.	The spring contact U_T at the sample comes in touch with the metal sample holder.	Eliminate the contact between the holder and the spring contact.

4.2 In the SFM mode

¹	Symptoms	Possible cause	Remedy
1.	The indicator on the microscope is not lit.	The microscope is not connected to the power supply. A fuse has blown. The power supply unit is out of order.	Connect the microscope to the power supply. Replace the fuse. Contact your dealer.
2.	When the microscope is turned on the device does not respond to the program's control commands (digital indication of the scanner position does not change, the step motor does not rotate).	The microscope was turned on after the program's start and the command of the electronic circuits Reset to the initial position has not been executed. The microscope is not properly connected to the computer, the connecting plugs are not fully inserted. There is power failure. The interface board is out of order.	Execute the Reset command as indicated in the User's guide. Turn the microscope off. Check if the device is properly connected. See point 1. Contact your dealer.
3.	When the feedback is turned on without approaching the sample trembling is observed along Z (over 100 A).	Too low voltage in the power supply system.	Check the voltage in the supply line.
4.	No signal from the photodiode (I, LFM) at the program and external oscilloscopes.	The laser is not lit. The laser beam does not reach the cantilever. The reflected beam does not reach the photodiode. The photodiode is out of order.	Turn the laser on. Point it to the cantilever as indicated in the User's guide. Point the reflected beam to the photodiode as indicated in the User's guide. Contact your dealer.
4à.	No signal from the photodiode (I, LFM) at the program oscilloscope but the signal is present at external oscilloscope.	The STM operation mode is on instead of the SFM mode.	Switch over to the AFM mode.
5.	In the process of the sample automatic approach in the AFM mode the motor does not stop and the sample collides with the probe.	The STM mode is on. The operating current has been set too high in the Options. Too low value has been specified for "FB Gain".	Switch over to the AFM mode. Set the operating current as indicated in the User's guide. Specify the value for "FB Gain" as indicated in the User's guide.

6.	The signal changes smoothly up to the setpoint value (rather than jump-like at the last moment) while approaching the sample to the cantilever tip (see the User's guide)	The probe or the sample surface is dirty. Sample surface is charged and exerts electrostatic force to the cantilever.	Replace the probe with a new one. Place the probe over a different area of the sample or replace the sample.
7.	Significant drift of the sample with respect to the tip is observed after the sample approaching.	The scanner is not hung horizontally and the head slides slowly on the surface. The sample is not attached properly to the substiate. The substrate with the sample is not properly inserted into the holder.	Use the adjusting knobs of suspension system to bring the scanner into a horizontal position within ±3 degrees. Re-insert the substrate into the holder.
8.	After the sample is approached auto oscillation at about 0.5...2 kHz is observed at the oscilloscope that disappears when the is reduced (see the User's guide).	The substrate with the sample is not properly inserted into the holder (it is supported only by two balls). The tip is not properly inserted into the STM head clamp.	Re-insert the substrate into the holder. Check if the tip is properly clamped in the holder. If necessary re-install.