USER'S GUIDE

P4-SPM-MDT
SCANNING PROBE MICROSCOPE

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NT-MDT

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The following labels are attached to the P4-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 P4-SPM-16 and P4-SPM-18 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.

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.

[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.

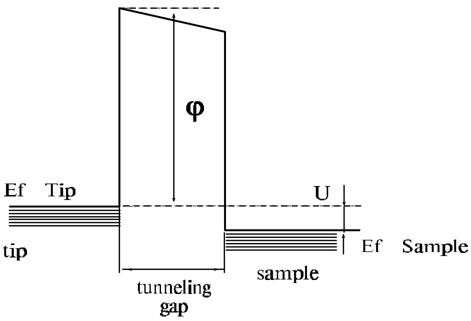


Fig. 1

Tunneling current in this approximation can be expressed as

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

u - bias voltage between tip and sample;

φ - average height of tunneling potential barrier s at zero bias;

z - width of tunneling potential barrier s (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.

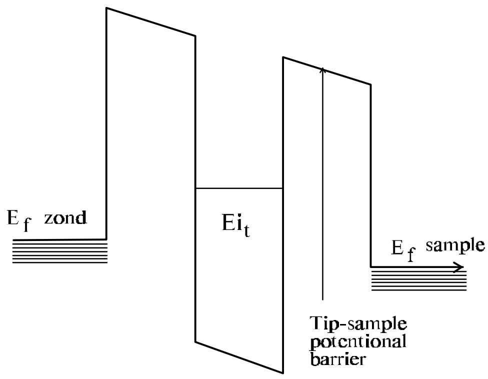


Fig. 2

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.

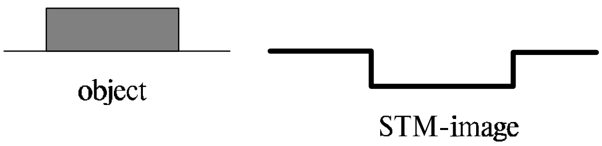


Fig. 3

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
Height imaging (topography)	Conductive surface topography imaging. Feedback system keeps tunneling current constant while scanning.
Current imaging	Tunneling current variations imaging. Tip height is kept constant relative to the sample base while scanning.
FB-error imaging	Imaging of tunneling current variations when feedback system tries to keep it constant while scanning. Similar to derivation of surface profile; steep surface features look emphasized.
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.
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.
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.

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.

1.2.2 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.
Semicontact	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.
	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.
Non-contact	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.
	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).

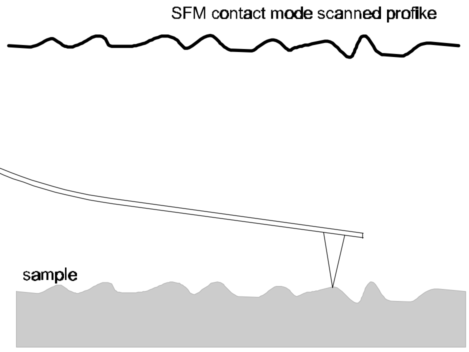


Fig. 4

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.

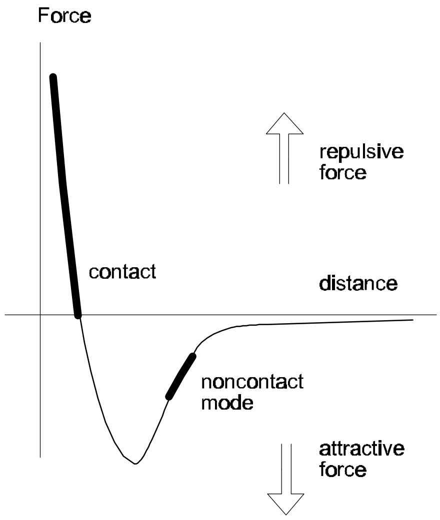


Fig. 5

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).

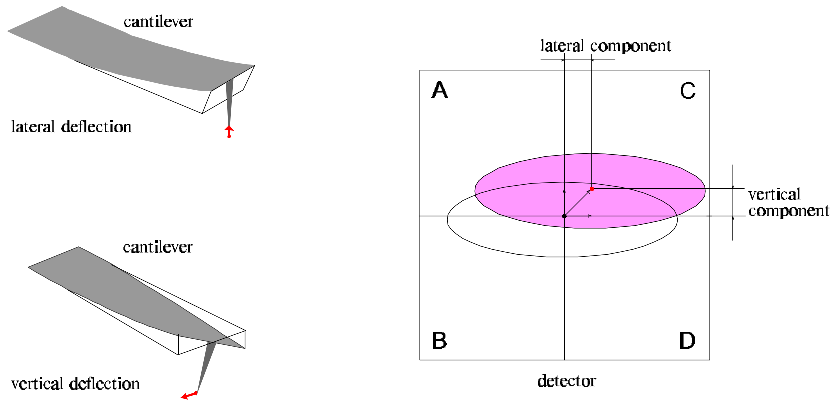


Fig. 6

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.

[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.

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° 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.

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.

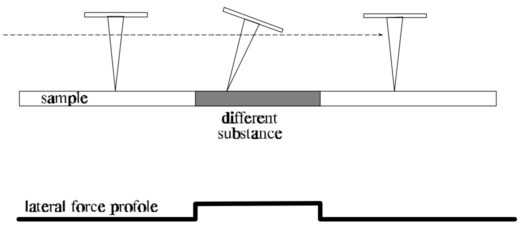


Fig. 7

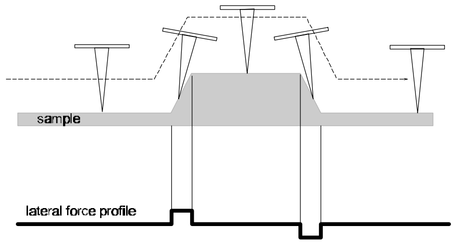


Fig. 8

In the lateral force mode getting atomic resolutionon mica is easy and on other layered materials. Notice that during measurement of topographywith 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 photodiodeis 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 resolutionis obtained. cautiously applying electronic adjustment of force on samples with small corrugations may be required.

1.2.3 Vibration and modulation SPM 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 noncontactand semicontact modes.
In the STM mode, a sample or tip vibration and corresponding modulation of tip-sample gap permits detection of tunneling currentoscillations to receive a signal dI/dz. This provides information on local height of a potential barrierfor 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.
In the AFM mode, sample vibration registers amplitudeof cantilever response that provides information on local elasticity of a sample.
Detecting an amplitude and/or phase of cantilever vibrations allows scanningof the surface topographyin 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.

1.2.3.1 Vibration and modulation STM techniques

Mode of local barrier height measurement

For the measurement of local height of a potential barrierfor 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 currentconstant 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 currentdependence 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.

1.2.3.2 Vibration AFM techniques. General information.

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).

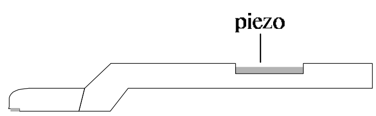


Fig. 9

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 ampli-

tude 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.

1.2.4 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).

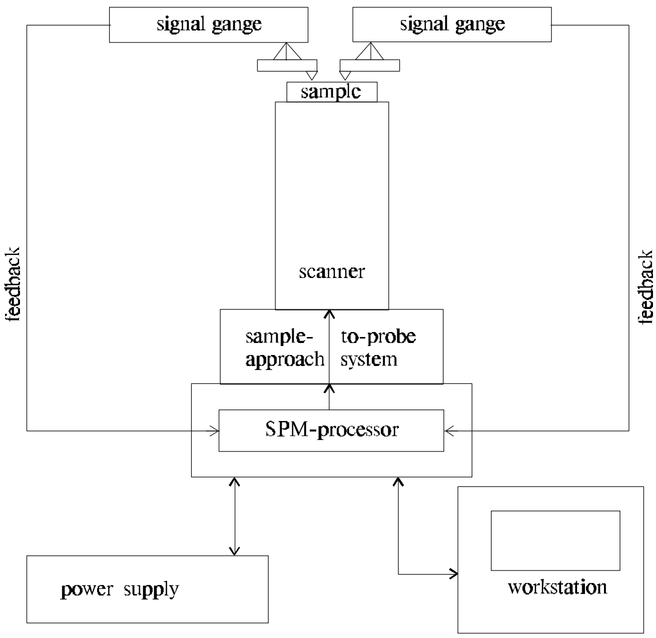


Fig. 10

1.2.5 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.

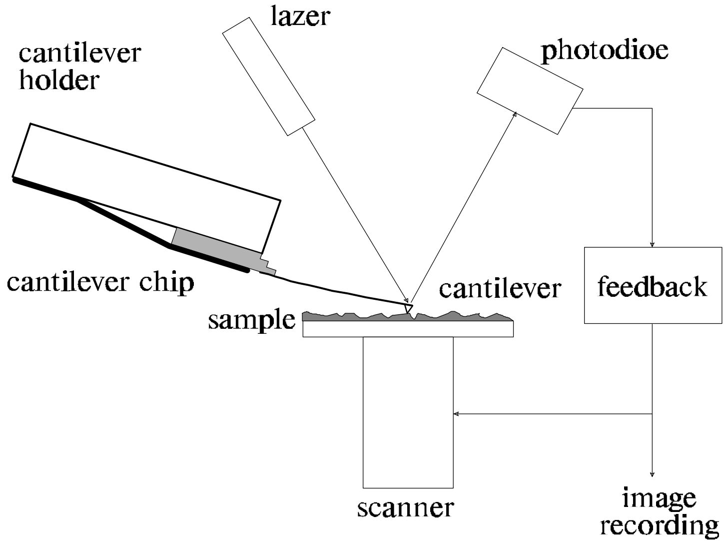

lazer
cantilever
holder
photodioe
feedback
cantilever chip
cantilever
sample
scanner
image
recording

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, and the power supply unit.
The piezotube scanner has a sample holder on its top. An SFM head (Fig. 12.C) or another head is placed on the suspended unit above the scanner. 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.

Fig. 12

2.1 SPM installation

2.1.1 Complete Solver SPM Unit Standard configuration

1) SPM Base with main electronics (processor) located within the base mount (Fig. 12.A; Fig. 13.A) and a three post suspension system mounted onto the mount.
2) Suspension system (Fig. 12.B) with piezoscanner and approach system.

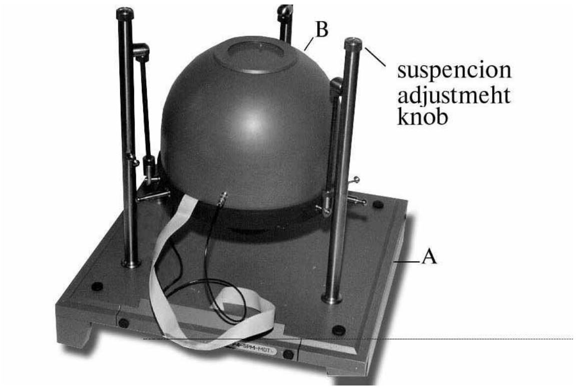


Fig. 13

Fig. 14

3) Protective cover for acoustic isolation and electromagnetic shielding (Fig. 13.B).
4) Power supply unit (Fig. 14).
5) Computer interface card (located within the computer if a computer is provided as part of the system).
6) Program diskette (control program is installed onto the hard disk if a computer is included within the deliverable system)

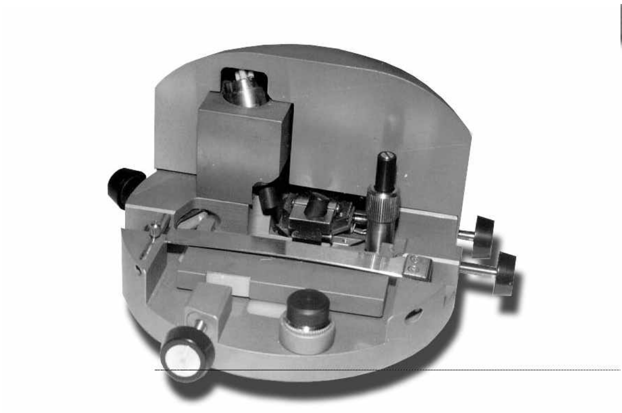


Fig. 15

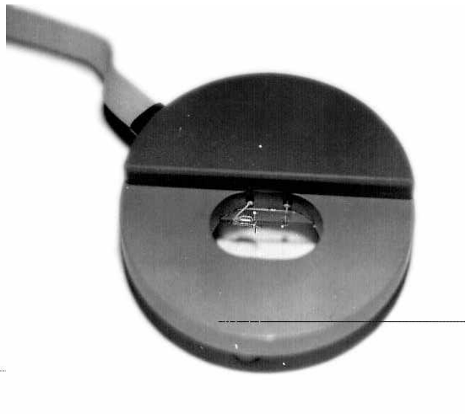


Fig. 16

7) AFM head with the standard adjustable part (Fig. 15).
8) STM head (Fig. 16).
9) Cables:

a) basic - PS-SPM (Fig. 17).
b) interface - SPM-IBM (Fig. 18).
c) two grounding wires (Fig. 19).
d) protective cover grounding wire (Fig. 20).
e) oscilloscope cable (Fig. 21).
f) power cord 220V.

Fig. 17

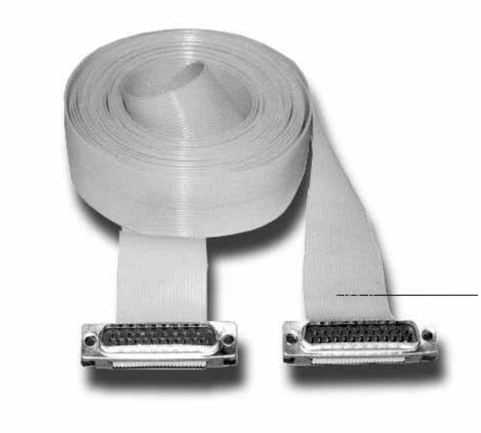


Fig. 18

Fig. 19

Fig. 20

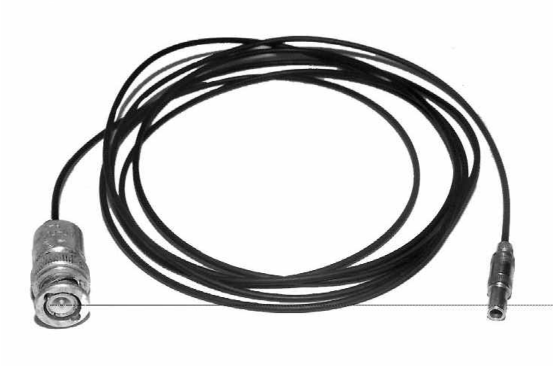


Fig. 21

10) Cantilever holders - two PCS (Fig. 24.1).
11) Tweezers for separation and mounting of cantileverchips (Fig. 26.4, 27.11).
12) Box with a strip of cantilever chips (Fig. 25).
13) Set of substrates:

a) with bias voltage contact strip for STM-study of thick samples (Fig. 22),
b) with bias voltage contact strip for STM-study of thin samples (Fig. 23, Fig. 27.7),

c) ordinary AFM substrates - four PCS (Fig. 27.4, 5, 6).

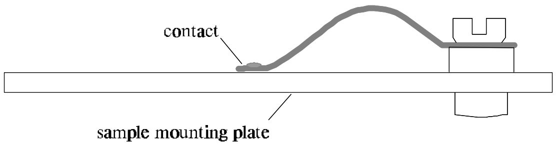


Fig. 22

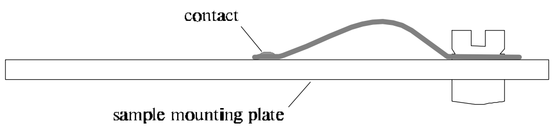


Fig. 23

14) HOPG (graphite) test sample and the set of test gratings TGS02.[4]

15) Pt-Ir or Pt-Rh wire for STM tips.

16) Wide tweezers and narrow tweezers (Fig. 26.2, 3, 27.9,10).

17) Special scissors for cutting STM tips (Fig. 26.1).

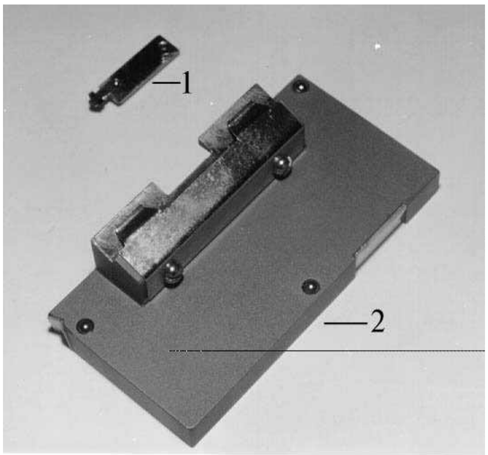


Fig. 24

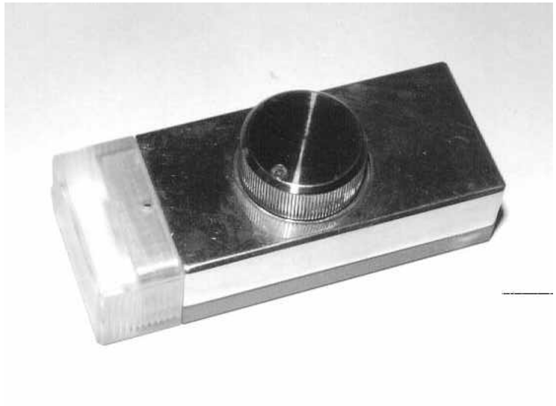


Fig. 25

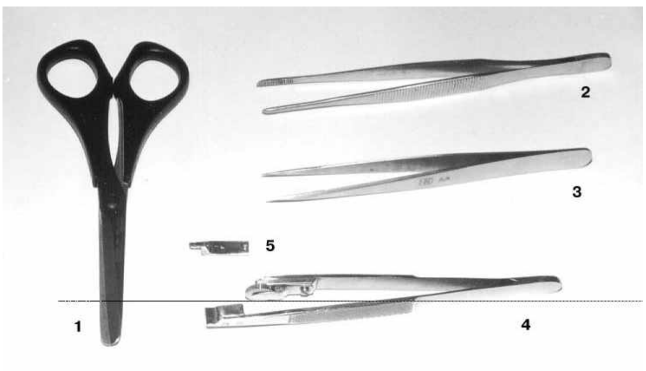


Fig. 26

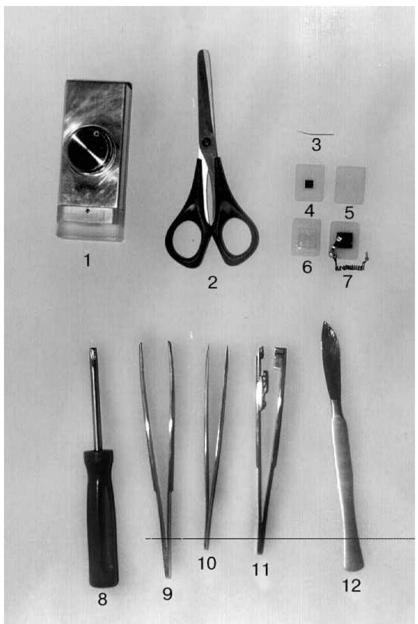


Fig. 27

[4] See Appendix 2.

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

18) Screwdriver (Fig. 27.8).
19) Scalpel (Fig. 27.12).
20) Double-stick tape for sticking samples.
21) Spare isolation band for suspension system.

2.1.2 Identification of components and inventory materials

See Appendix 1.

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 SPMoperation. 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 inFig. 28.

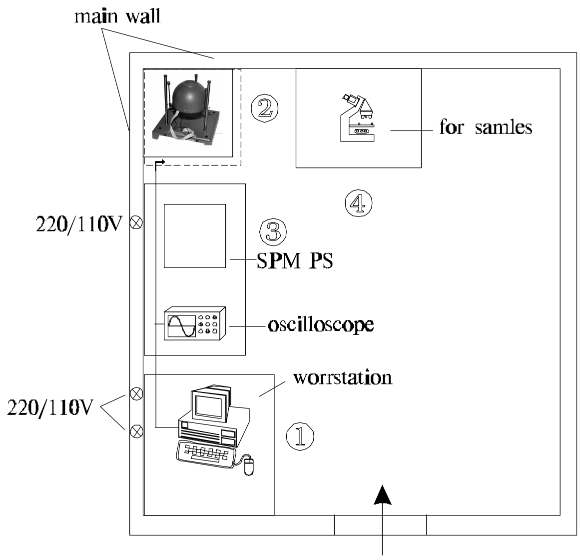


Fig. 28

The desired placement of the SPM unit is in a corner. An external oscilloscopewith 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 resolutionmust 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:

- ✓ On the front side of the housing (Fig. 30) exists common grounding (1), output (2) of STM bias voltage (+5V...-5V) and of lithography voltage (+12V...-12V), input (3) for Z-axis external modulation, connector (4) for linking to piezoscanner, and connector (5) for linking to one of the measuring heads;

- ✓ On the opposite side of the housing (Fig. 31) there are connectors for linking the computer (1) and the power supply (2);

- ✓ On the third side (Fig. 32) exists an input socket (1) for an external signal and output socket (2) of either tunneling current or photodiode current depending on the head in use.

2.3.2 Connecting the SPM without Resonant mode unit

CAUTION!
Don`t put the scanner unit on a table upside down and don`t exert any force to the sample holder on the scanner top. It will cause breakage of piezotube.

1) Place the main SPM unit on a hard, steady table, and put the power supply unit on another one (see item 2.2).
2) Put the scanner unit (Fig. 29) with its three supporting "fingers" onto three the supporting rods of the suspension system pillars (see e.g. Fig. 35) with the scanner up (and black round knob down), and with the rubber disk insert turned toward the front side of the main unit housing. Put the three isolation suspension bands on the three short "fingers" of the scanner unit and lock them into position with the "C" locking washers.
3) Pull out the two rubber plugs (Fig. 30.6) from the bar on the face side of the SPM housing, unfasten the screws, and remove the bar.
4) Connect the female 9-pin connector (3) (Fig. 29) of the longest cable of the scanner unit to the connector labeled "MOTOR" on the rear side of power supply.
5) Connect the male 9-pin connector (4) (Fig. 29) of the shorter cable of the scanner unit to the connector (4) (Fig. 30) on the front side of main board housing.
6) Insert connector (2) (Fig. 29) of the scanner unit cable to the bias voltage socket (2) (Fig. 30), marked "Ut", on the front panel of main board housing.

Fig. 29

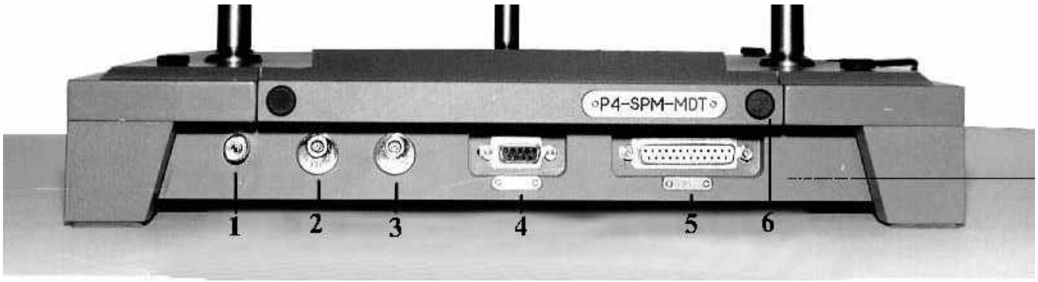


Fig. 30

Fig. 31

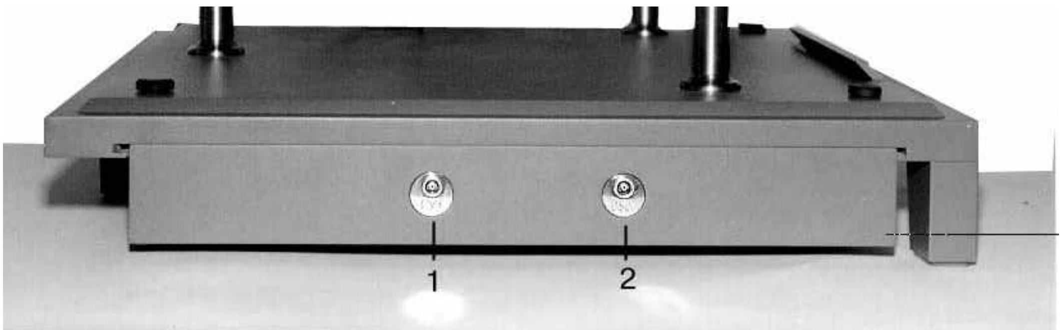


Fig. 32

2.3.2.1 Grounding connections

7) Unfasten a nut from the "ground screw" (1) (Fig. 30).

8) Put a contact of the scanner unit grounding wire (1) (Fig. 29) on the "ground screw" (1) (Fig. 30).

9) Put a contact of the protective cover grounding wire (Fig. 20) on the "ground screw"(1) (Fig. 30).

10) Put contacts of grounding wires (Fig. 19) on the "ground screw" (1) (Fig. 30). If your supply line has a ground wire (i.e. you have usual 3-terminal supply line), use only one of two wires (Fig. 19).

11) Fasten the nut to the "ground screw" (1) (Fig. 30) again.

12) Insert connector of protective cover grounding wire (Fig. 20) into the socket on the cover (this can also be performed later, when the cover is ready to be set in place).

13) Connect the free end of ground wire (Fig. 19) to the power supply grounding screw ("GROUND", Fig. 33).

14) ONLY if you have 2-terminal supply line without grounding wire:

Connect the free end of the second ground wire (Fig. 19) to the computer housing.

2.3.2.2 Other connections

ATTENTION:
Be sure that the switch 110V/230V at the rear side of power supply unit (Fig. 33) is in proper position for your locale.

15) If you would like to use your external oscilloscope, connect it to the socket (2) (Fig. 32) by the cable (Fig. 21).

16) Link the measuring head (either STM or SFM) to the 25-pin connector (5) (Fig. 30) on the front side of the main board housing.

17) Put the front-side bar to its place. Be sure that the measuring head cable, protective cover grounding wire, and all the scanner unit cables pass through the slot between the bar and the housing. Fasten the bar with its screws and reinsert the rubber plugs.

2.3.2.3 Connecting the power supply unit

18) Connect the 25-pin connector "SPM" on the rear side of power supply (Fig. 33) by the black cable (Fig. 17) to the connector "PS" on the rear side of main SPM housing (2) (Fig. 31).

19) Connect the socket "POWER" on the rear side of power supply (Fig. 33) by power cord to supply line immediately or through an additional filter (the last is not included).

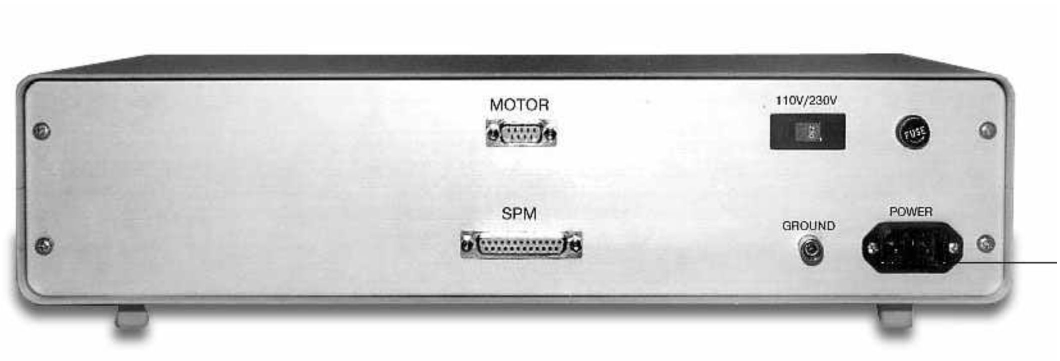


Fig. 33

2.3.2.4 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 diskette, read the file README.TXT and make recommended changes to files AUTOEXEC.BAT and CONFIG.SYS of your computer. Create a directory P7 on your hard disk and copy all the files from the diskette.
For the program operation 3 files are necessary:

P7_spm.exe
P7_spm.opt
P7_spm.hlp

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.

2.3.2.5 Connecting the main SPM board to the computer

20) Connect 25-pin connector "IBM" on the rear side of the main SPM housing (1) (Fig. 31) by the flat cable (Fig. 18) to the SPM interface board. Be sure that you insert the cable to the SPM interface board rather than to the computer LPT port.

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

2.3.2.6 Switching on the device

Turn on the computer and SPM power supply by the switch on its front side (Fig. 14) (in any order). Start the program P7_SPM.EXE.

2.3.3 Connecting SPM with Resonant mode unit

All procedures should be done according to paragraph 2.3.2 except for the connecting of the measuring head (operation 16), Resonant mode unit grounding, and its connecting to supply line.
Besides operations 16, a grounding wire for Resonant mode unit should also be connected to the common grounding place (1) (Fig. 30). The other end should be connected to the grounding screw on the rear side of Resonant mode unit.
Instead of operation 16, the measuring head should be linked to the connector (1) (Fig. 34) on the front side of Resonant mode unit. And another connector (2) (Fig. 34) should be linked to connector (5) (Fig. 30) by the short cable similar to that in Fig. 17.
If you want to use your external oscilloscope to look at the tunneling current or the signal from the photodiode before detection, you should connect it to the socket on the front side of Resonant mode unit by oscilloscope cable (Fig. 21).
The power cord should connect Resonant mode unit to supply line immediately or through a power surge filter (not included).
After switching on the power supply you should turn on the Resonant mode unit by the switch on its rear side near power cord socket. A red light on the front side indicates POWER ON state.

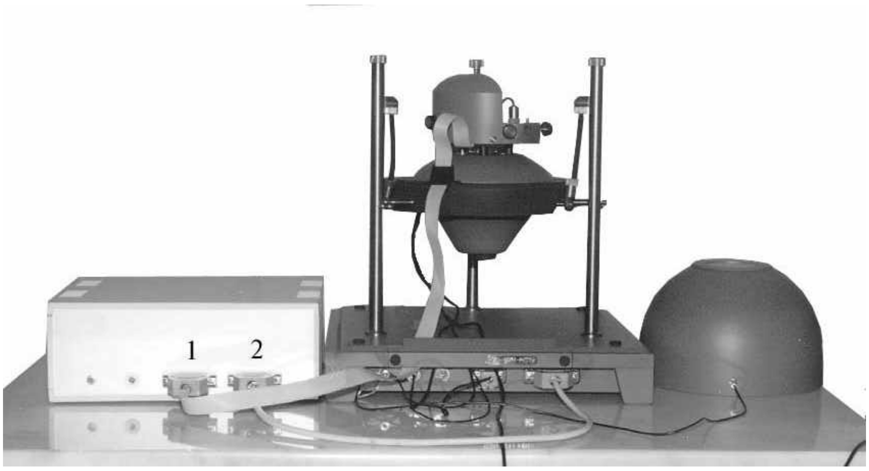


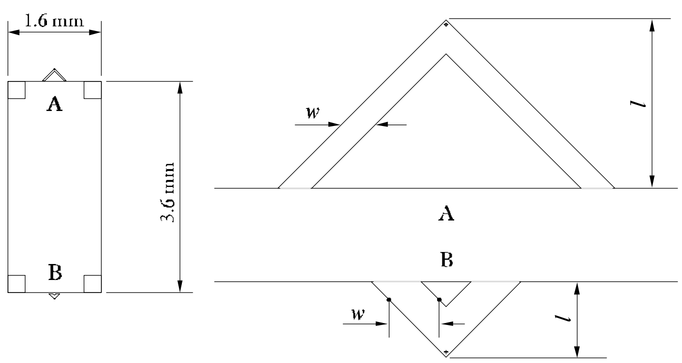
Fig. 34

3. Basic operations

3.1 SFM-mode

3.1.1 Choosing a cantilever

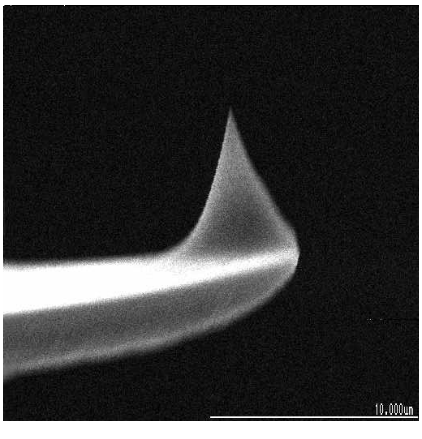
The cantilever choice depends on the task and on the sample to be studied. To get surface topography of a rigid sample, use fairly short triangular cantilevers that permit measurement of true topography and not distortion of lateral force influence. Shorter cantilevers do not suffer from a sort of self 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 cantilevers produced by NT-MDT are presented below.

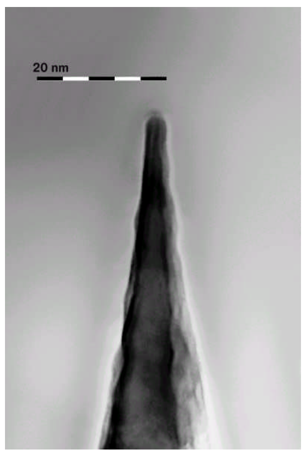


Cantilevers type	A			B		
	Min	Typical	Max	Min	Typical	Max
Cantilevers length, *l*, µm		200			90	
Cantilevers width, *w*, µm		40			60	
*Cantilevers thickness, µm	1.6	1.9	2.2	1.6	1.9	2.2
*Resonant frequency, kHz	40	50	60	300	350	400
*Force constant, N/m	1.8	3.0	4.2	24.0	30.0	36.0

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





3.1.2 Mounting a cantilever

Special mounting tweezers should be used to install a tip from the box of a cantilever strip into the SFM holder.

Simple tweezers could also be used if cantilevers are supplied in another package than the special box.

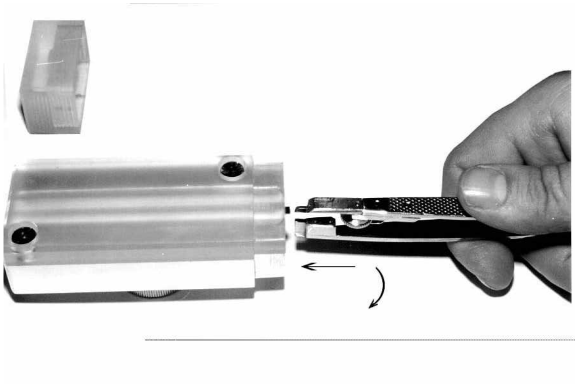


Fig. 35

3.1.2.1 Cantilever mounting tools

Box for a cantilever strip - consists of:

- Plexiglass part (1) (Fig. 36.A) with shallow flat groove to put a strip of cantilevers (6) (Fig. 36.A) with the tips upwards;
- \- An aluminum counterpart (2) (Fig. 36.B) with movable insert and a screw terminated in a knob (3) to retain the strip against the groove (rotating the knob clockwise loosens the strip, and rotating it counterclockwise clamps the strip);
- Two screws (5) (Fig. 36.B) fastening both parts together (in the other version of the box design the two screws are fastened from the plexiglass side (Fig. 40));
- Plexiglass dosing cover (4) (Fig. 36.B) with the pit of one-chip depth.

Mounting tweezers - consist of:

- A flexible bar (7) (Fig. 36.C, D) with the specially shaped end (10) to press against the spring clamp (12) of a cantilever holder (11) while breaking cantilever chip apart from the strip;
- A flexible bar (8) (Fig. 36.C, D) with the screw (9) to fasten cantilever holder;
- SFM cantilever holder (11) (Fig. 36.C, D) with flat spring (12) to clutch cantilever chip (6i).

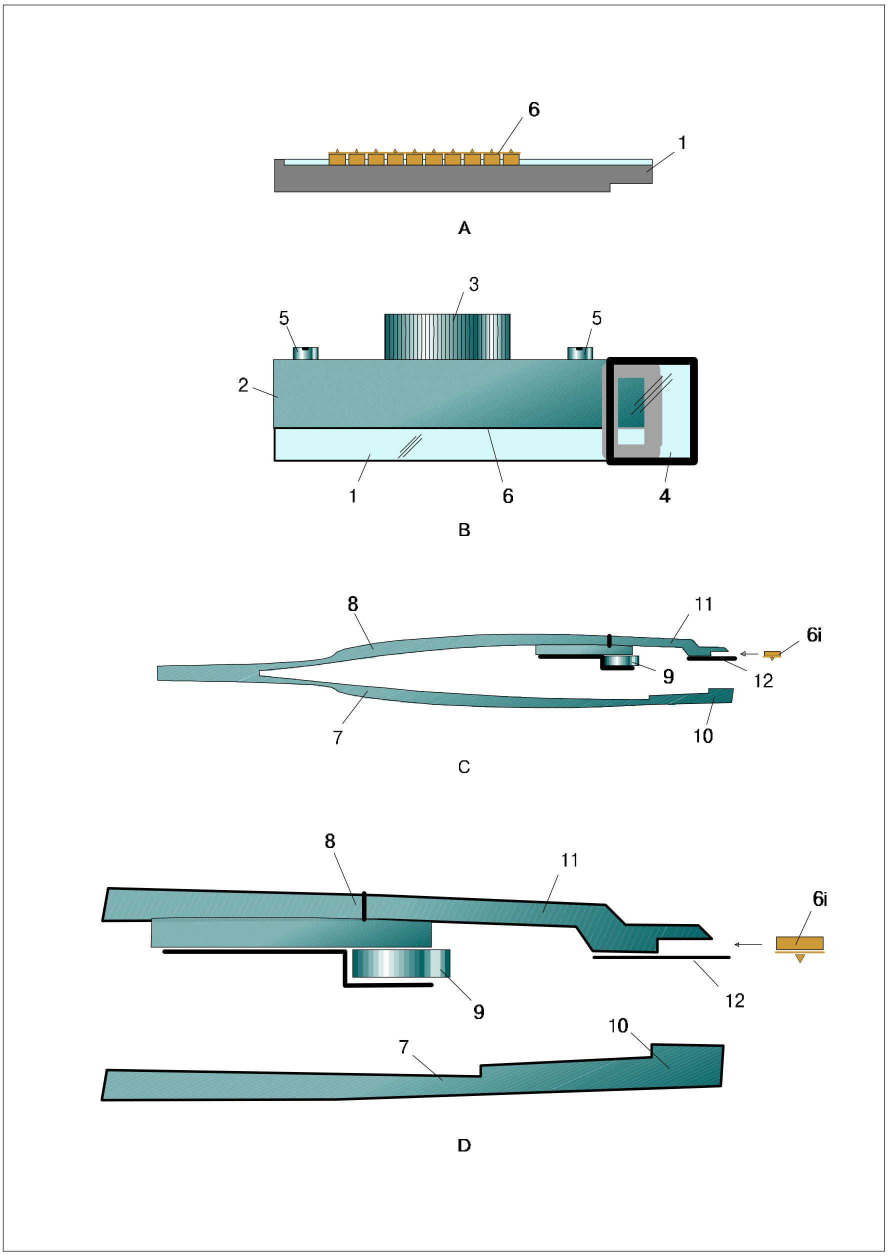
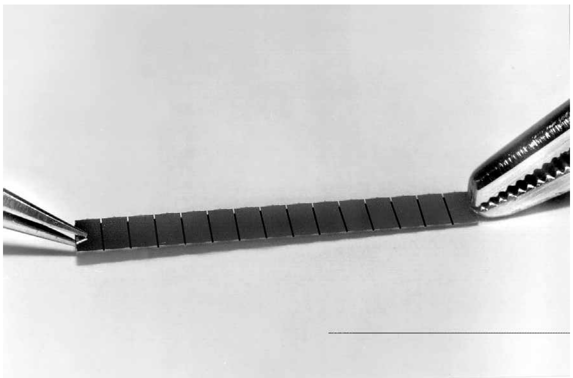


Fig. 36

Placing a strip of cantilever chips to the box

Remove the dosing cover from the box (Fig. 39). Unfasten two screws (5) (Fig. 36.B) connecting plexiglass and aluminum parts together (Fig. 40). Put the box with the knob upwards and pull up the aluminum part, holding the plexiglass one (Fig. 41). Verify that both inner surfaces are clean, otherwise wipe them with alcohol. Put a strip of cantileverchips to the flat shallow groove on the plexiglass part (Fig. 37, Fig. 38) with the tip side upwards. Rotate the knob clockwise tightly. Put the aluminum part against the plexiglass one and holding them together replace the dosing cover, then fasten the parts to each other with screws. The strip of cantilevers can loosely slide along the groove now. Tilt the box with the dosing cover downwards. The strip slides down to the stop (if it has already been in the box for a long time, it can be stuck a bit; then slightly shake the box). Rotate the knob counterclockwise to the stop. The strip is captured in the groove now. The box is ready for transportation and use.



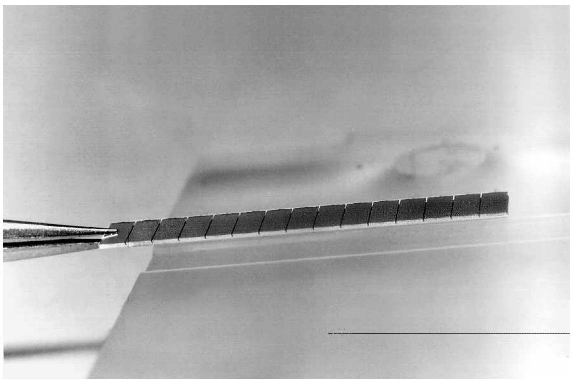
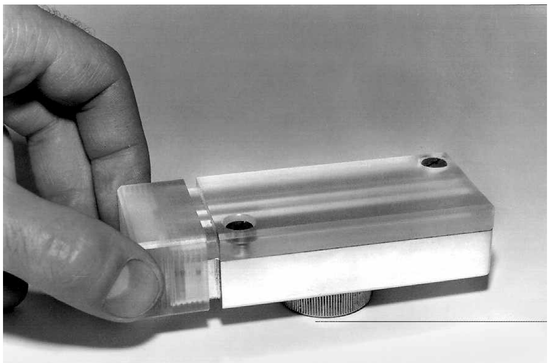


Fig. 37

Fig. 38



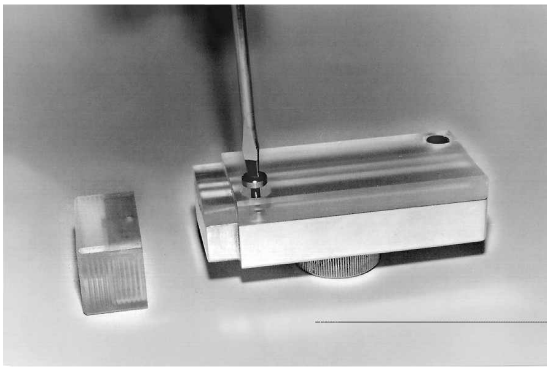


Fig. 39

Fig. 40

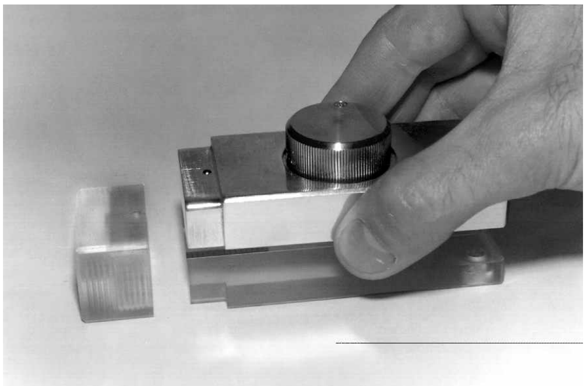


Fig. 41

ATTENTION:
Do not touch the cantilevers while working with cantilever chips. Any contact will contaminate or damage tips and cantilevers.

Mounting a cantilever onto the holder

The strip of cantilevers can now loosely slide along the groove. Tilt the box with the dosing cover downwards. The strip slides down to the stop (if it has been in the box for a long time, it can slightly stick; in that case gently shake the box). Rotate the knob counterclockwise to the stop. Remove the dosing cover. The strip is clutched in the groove now and only one cantilever tip extends beyond the front edge of the box.

ATTENTION:
When the strip of cantilevers is not clamped inside the box, dosing cover should be closed up to the stop, otherwise cantilevers can drop out.

Take mounting tweezers with cantileverholder attached to them. Carefully move up tweezers to put cantilever extending from the box into the gap between the holder itself and its flat spring (Fig. 35, Fig. 36.D). Cantilever chip should be oriented with the tipside toward the flat spring and should equally extend beyond both edges. Press tweezers to grip the cantilever and tilt them toward the bar (10) (Fig. 36.D) to break the tip from the strip (Fig. 35). Put the holder into the SFM adjustable insert (2) (Fig. 24). Unfasten the screw (9) (Fig. 36.C, D) to detach tweezers from the holder. Final state is shown in Fig. 47. When the SFM adjustable insert lies on a table upside down as shown in Fig. 47, chip should be approximately in the middle part of the insert and cantilever(s) should be the uppermost point of the assembly.

3.1.3 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. 43, Fig. 42). 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.

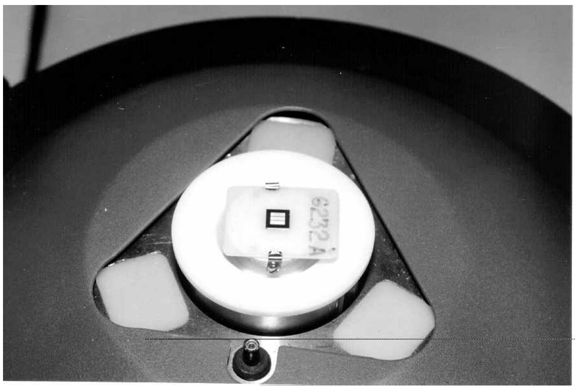


Fig. 42

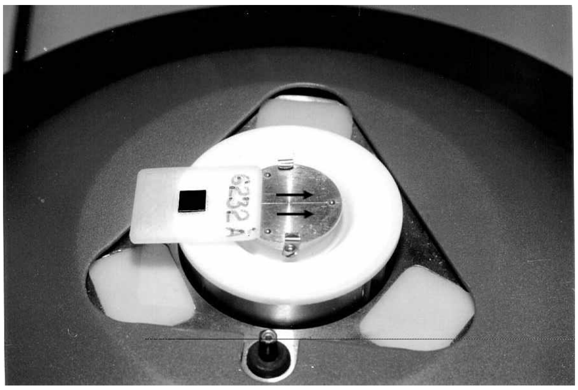


Fig. 43

3.1.4 Adjusting SFM head

3.1.4.1 Basic elements of the SFM head (Fig. 44, Fig. 45):

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 modeSFM 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

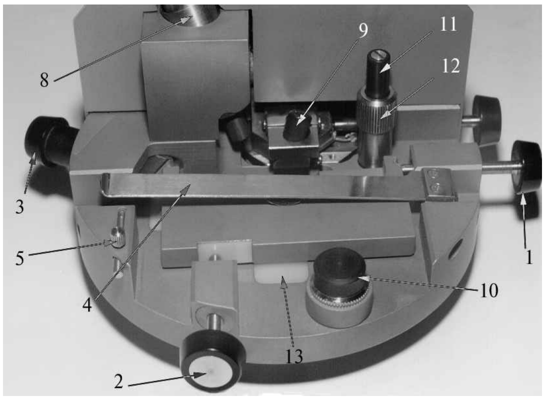


Fig. 44

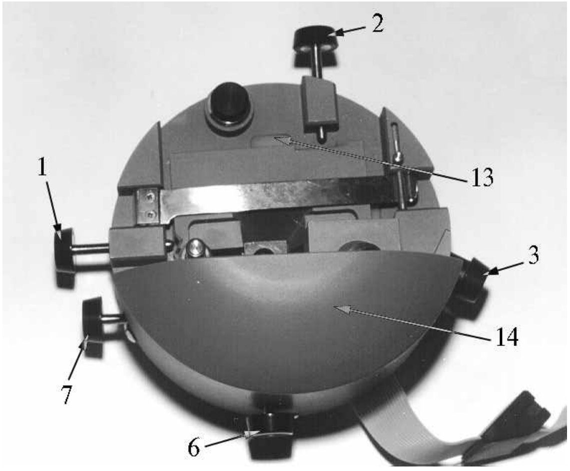


Fig. 45

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. 44, Fig. 45). Put the SFM movable insert with mounted cantilever(see section 3.1.2) (Fig. 47) into SFM head (Fig. 46). 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. 44, Fig. 45) to let spring plunger push the insert. Put the clamp bar (4) on the insert but do not fix it with the catch (5).

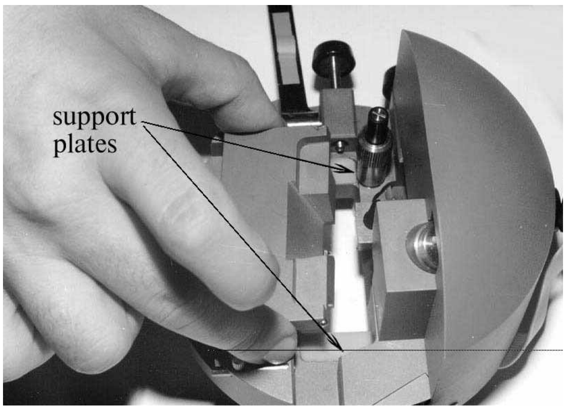


Fig. 46

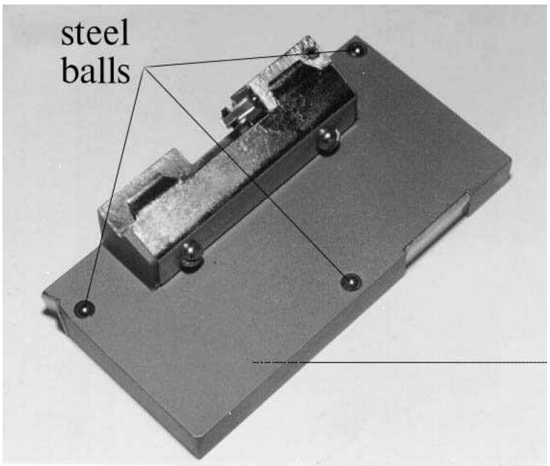


Fig. 47

Turn on power supply (Fig. 33).
Start SPM control program **P7_SPM.EXE**.
In the left top corner of the screen (Fig. 48) 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. 49) and click it. You will see three windows: scan window "SPM" and two windows of program oscilloscopes "LFM" and "I" (Fig. 50). Window "LFM" shows photodiodedifferential current corresponding to the position of a reflected laser beam in lateral force direction and its changes caused by cantilevertwist. A window "I" reveals the photodiode differential current corresponding to normal force direction and its changes caused by normal bending of cantilever.

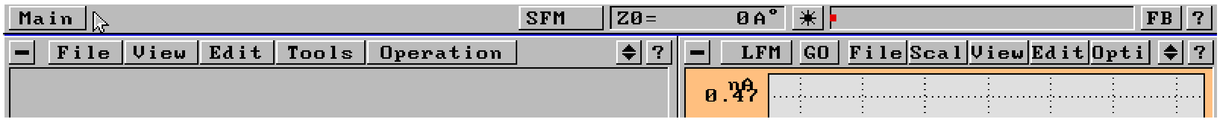


Fig. 48

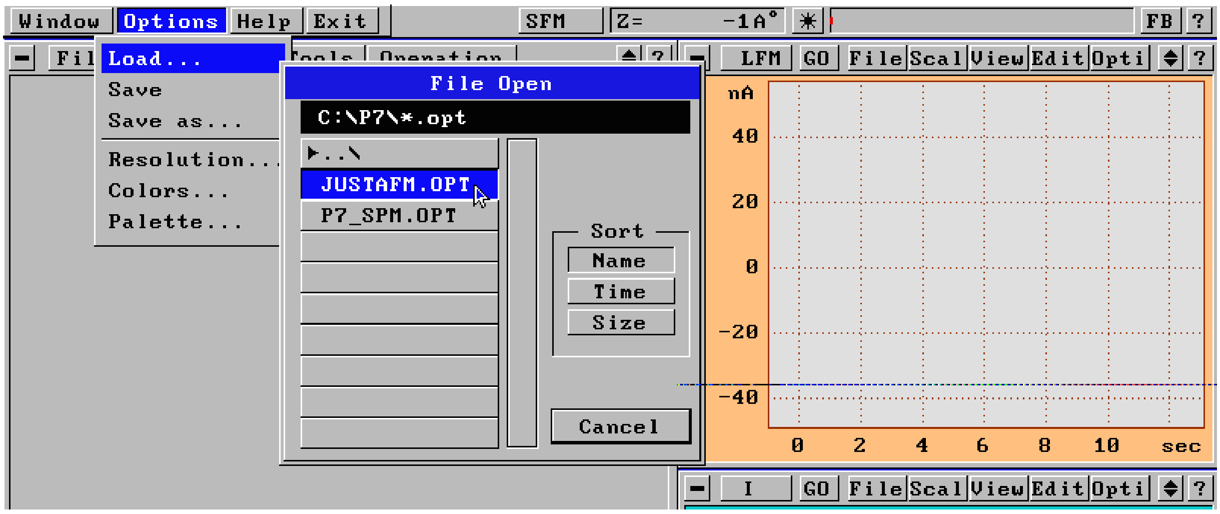


Fig. 49

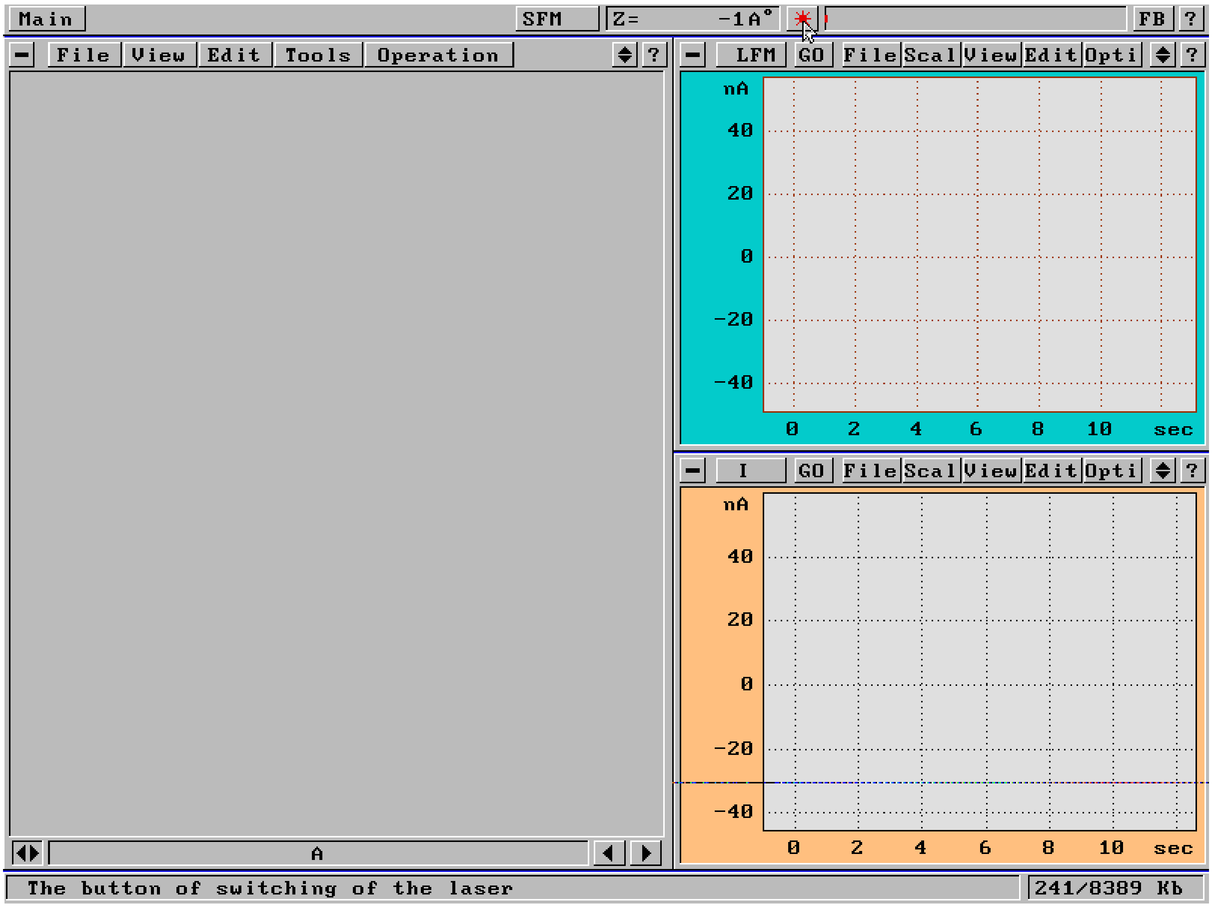


Fig. 50

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. 50, Fig. 51), and laser on the SFM heads turns on.

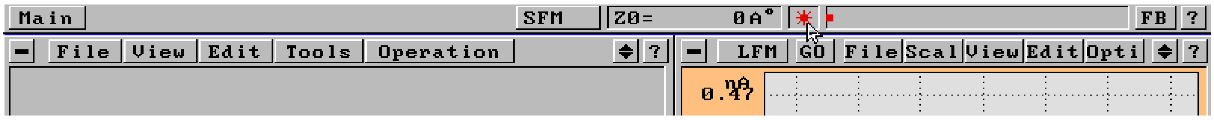


Fig. 51

Press buttons GO in oscilloscope windows "LFM" and "I" 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. 44, Fig. 45) 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. 52). 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 photodode and the spot (Fig. 53).

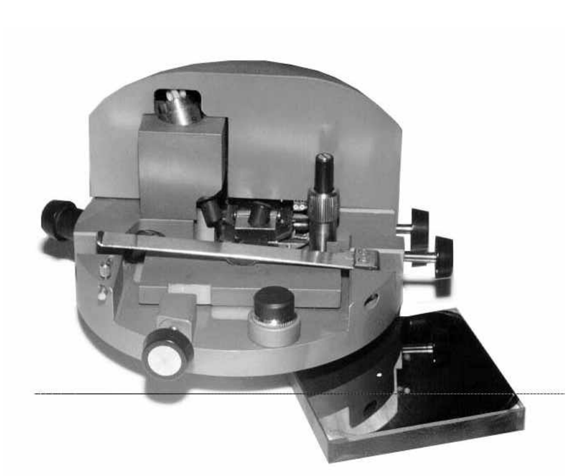


Fig. 52

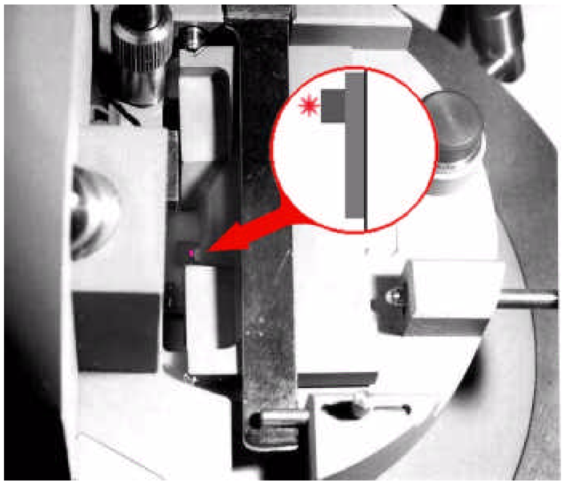


Fig. 53

CAUTION:
Don`t stare into beam!

Signal values in oscilloscope windows "LFM" and "I" will deviate from zero (Fig. 54).
Set some positive values in both windows (Fig. 55) by means of photodiode adjusting screws (6, 7) (Fig. 45). Slightly move cantilever about focused beam by rotating adjusting screw (1) (Fig. 45) to get the maximum signal value in any window (Fig. 56). Then slightly move it along another direction by the screw (2) (Fig. 45) to get maximum signal value (with cantilever with triangular area at its end) again in the same window (Fig. 57).

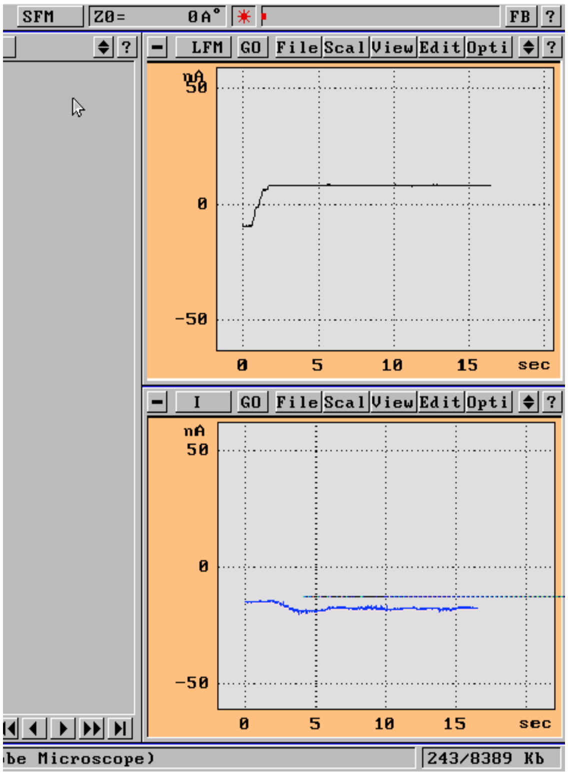


Fig. 54

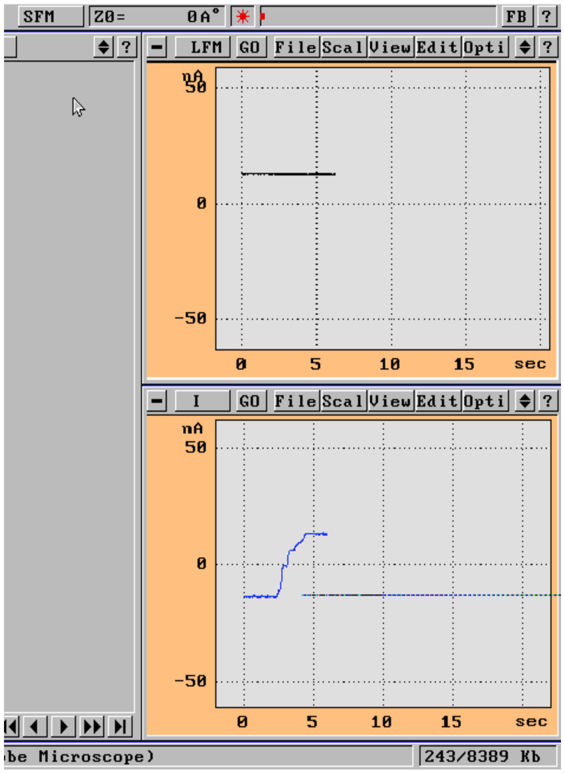


Fig. 55

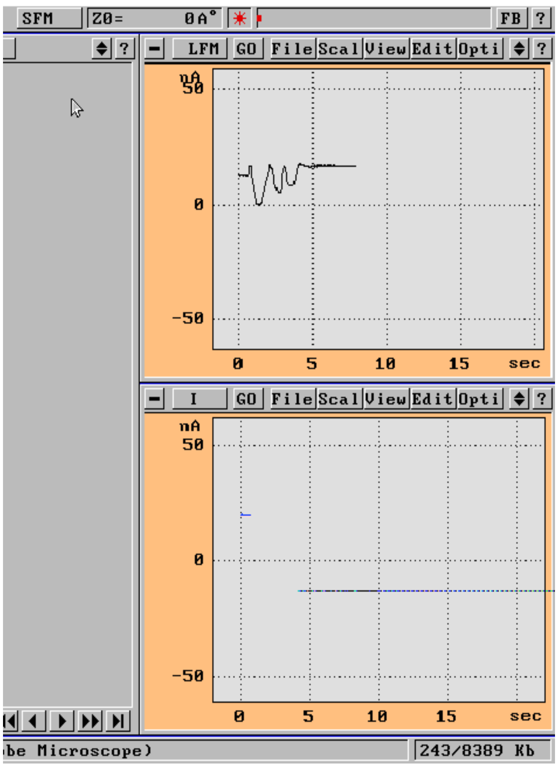


Fig. 56

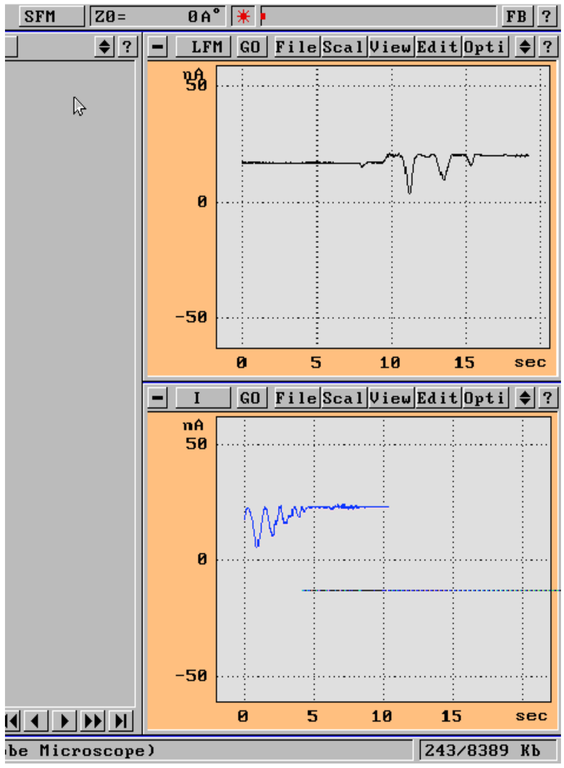


Fig. 57

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. 58) around the spot of the laser beam.

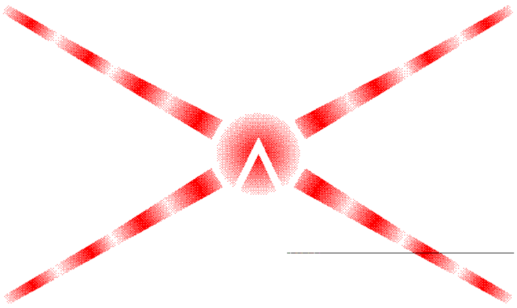


Fig. 58

Put the head back on a table. Press the movable insert with the clamp bar (4) and fix it with the catch (5) (Fig. 45).
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) clockwise 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. 45) are used to align it with the reflected beam.
Rotate the screw (7) to set signal value in the window "LFM" equal to zero (Fig. 59). 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 photodiodequadrant.
Rotate the screw (6) to set signal value in the window "I" equal to zero (Fig. 60).Check the signal value by slight rotation of the screw for indication of positive and negative values. When both "LFM" and "I" 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 "I" 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 "I" by screw adjustment (6), than set it to about a few nA.

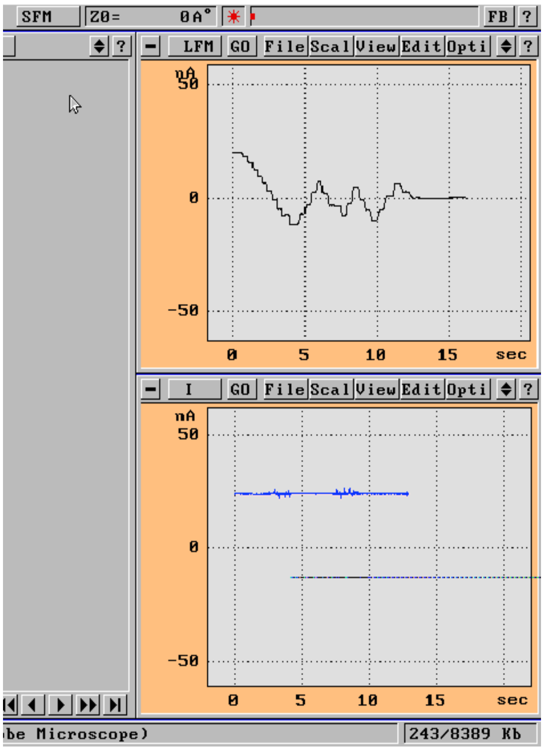


Fig. 59

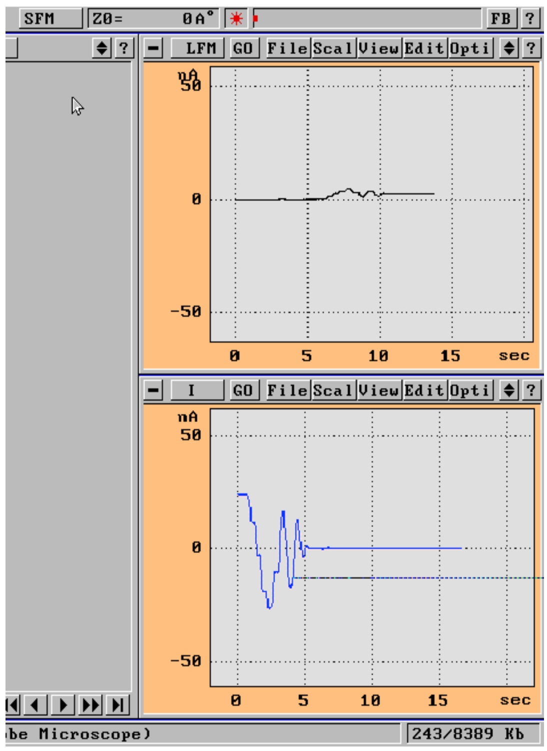


Fig. 60

ATTENTION:
Signal value in the window "LFM" should always be adjusted to zero to have the least possible noise value while getting atomic lattice resolution in lateral force mode.

3.1.5 Setting SFM head

Put aside protective cover (B) (Fig. 13) and disconnect its grounding wire if necessary. Move down the scanner with a sample on its top by rotating black knob (5) (Fig. 29) clockwise (Fig. 62). The knob is attached to a screw, and the sliding cylinder with piezotube behaves as a nut; so when the knob is rotated clockwise (bottom view) the cylinder moves down, and vice versa.

Fig. 61

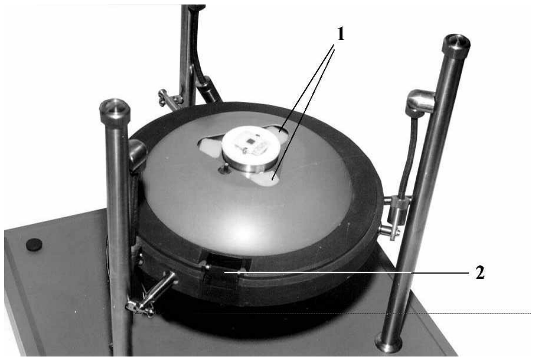


Fig. 62

Set SFM measuring head with its legs onto the support plates (1) (Fig. 62) on top of the scanner unit. The cable should be opposite the pocket (2) (Fig. 62) in the rubber disk. Insert the black holder on the cable into the pocket (Fig. 63). Move the scanner up by rotating the knob counterclockwise (bottom view) to approach the sample to about 0.5 - 1.0 mm from the probe (Fig. 64) observing it from the side. Then move the SFM head in the lateral direction to align the probe with the piezotube axis.

Fig. 63

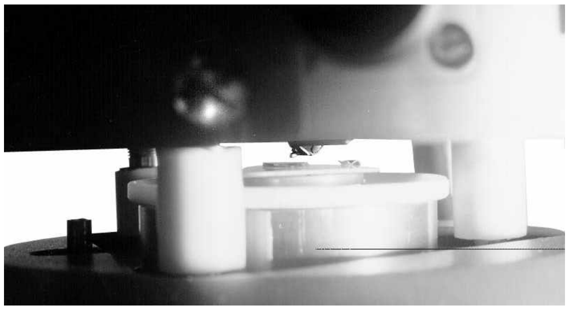


Fig. 64

Check signal values in the program oscilloscope windows "I" and "LFM" to be sure that the adjusting screws have not been accidentally moved.
Place the protective cover (B) (Fig. 13) 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. 13).

3.1.6 Automatic approach of sample to probe

Further control of SPM operation is executed through a control program.
Point the button "Operation" in the top of the "SPM" Window. Click the item "Probe Options" in the appeared menu and set proper parameters in the table "Probe Options" (Fig. 65). 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"=1 (maximum value) (Fig. 66). As for value of bias voltage "Bias Voltage (v)", it is not important in SFM mode if the sample is not connected to the Ut socket.

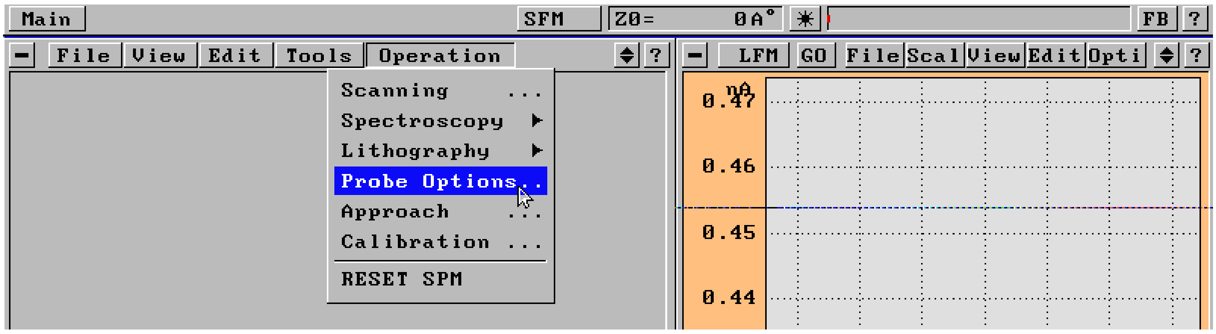


Fig. 65

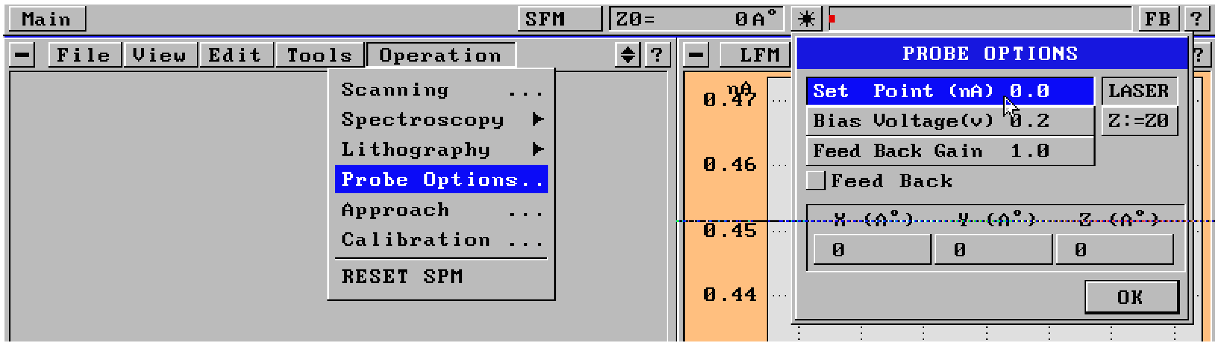


Fig. 66

Close the table of parameters and open the menu "Approach" (Fig. 67). Use sliders to set the maximum velocity of sample to probe approach with feedback state monitoring ("Landing"=20), without monitoring ("Forward"=20) and retraction velocity ("Backward"=20). Click "Landing" to start approaching the sample to probe (Fig. 68). It will be completed automatically and then marked with beep sound and notice on the screen. Press ESC button.
Click buttons GO in both oscilloscope windows to stop measuring of signals "I" and "LFM" (Fig. 69).

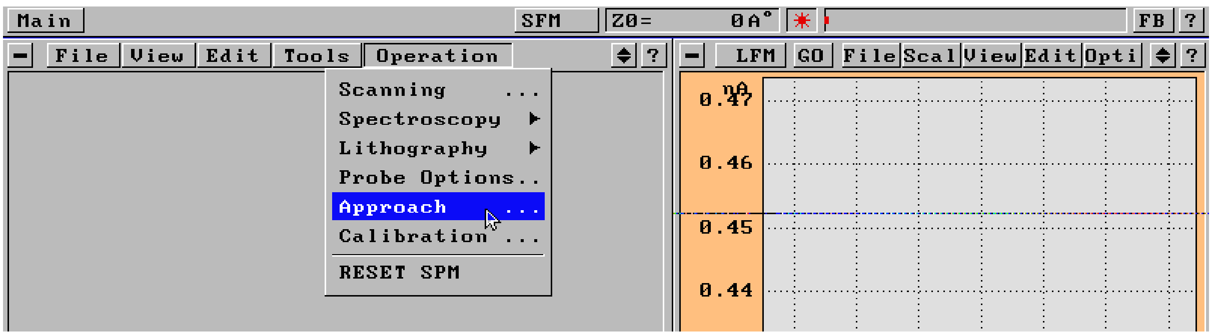


Fig. 67

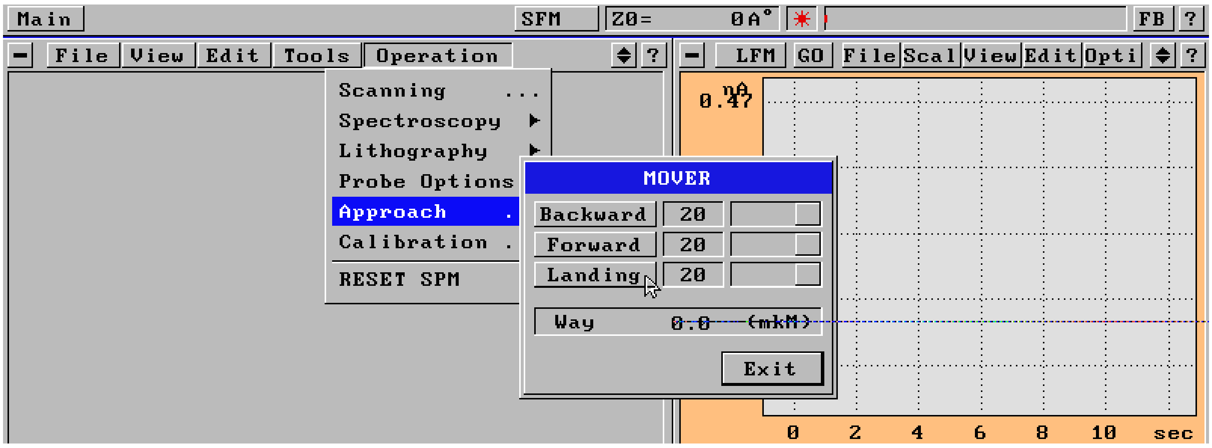


Fig. 68

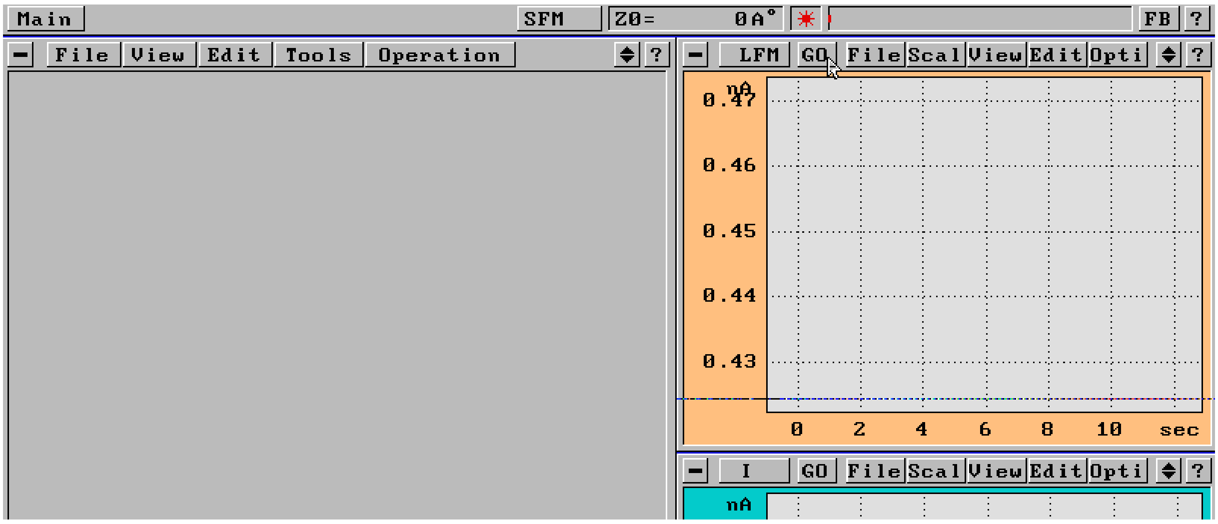


Fig. 69

It is useful to withdraw part of approach system to reduce thermal drift caused by elements of the system. Click "Backward" while holding SHIFT pressed and wait until the counter shows about 100 um back (Fig. 71). Then press ESC to stop moving.

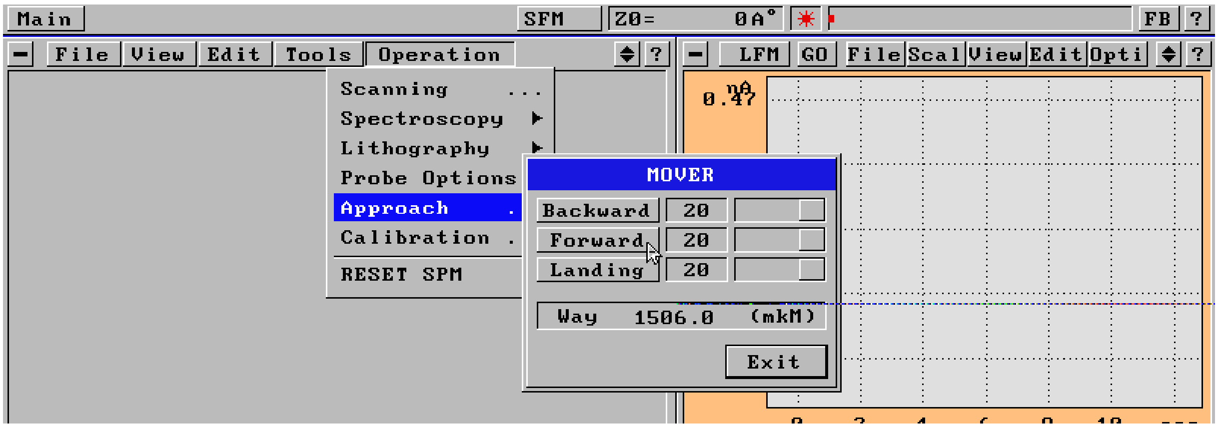


Fig. 70

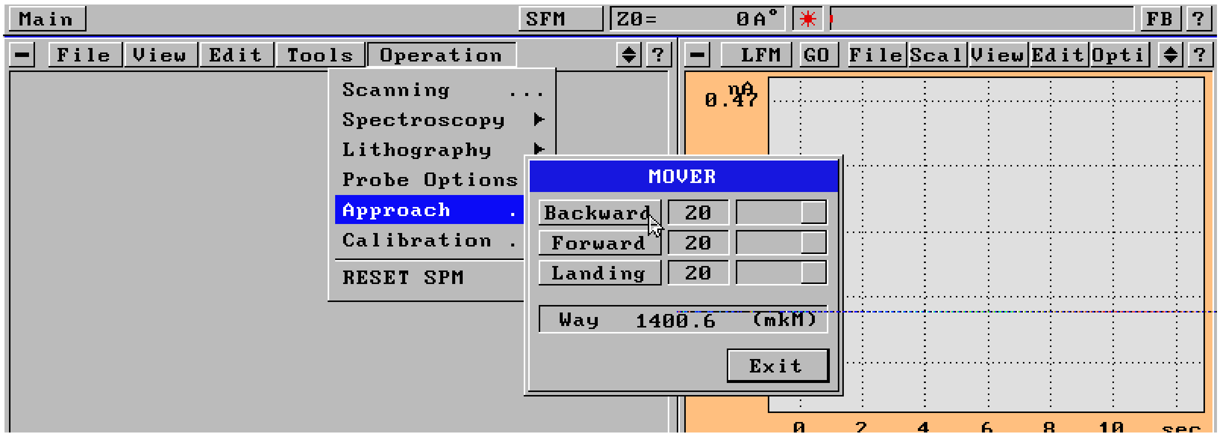


Fig. 71

3.1.7 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” (Fig. 72) to open corresponding menu and then click the item “Scanning” to open its menu. Now click “Scanning” and set the values of scanning parameters (Fig. 73): velocities of forward and backward motions, number of measurements in a point, digitizing accuracy of the measured signal value, scanning steps, number of steps in both directions, scanning mode. You should also specify the second signal to be recorded while scanning and decide whether each string should be imaged just after it has been scanned, whether fitted plane should be subtracted, and whether nonlinearity should be corrected while scanning (Fig. 73, Fig. 74, Fig. 75, Fig. 76, Fig. 77, Fig. 78, Fig. 79).

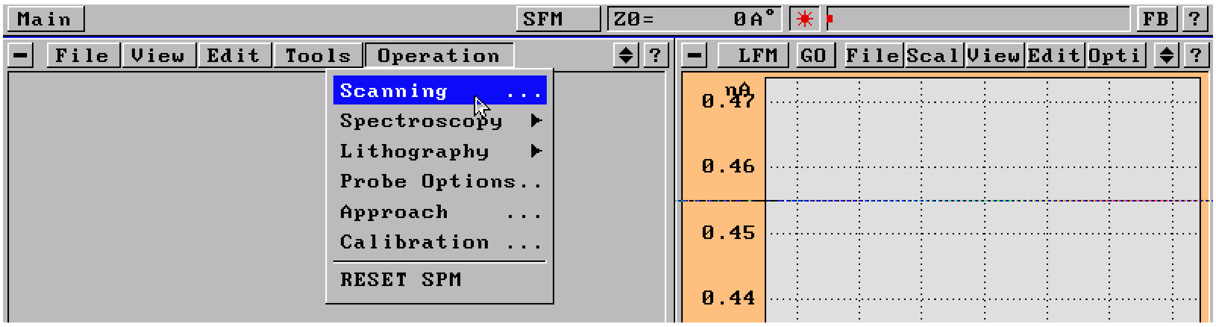


Fig. 72

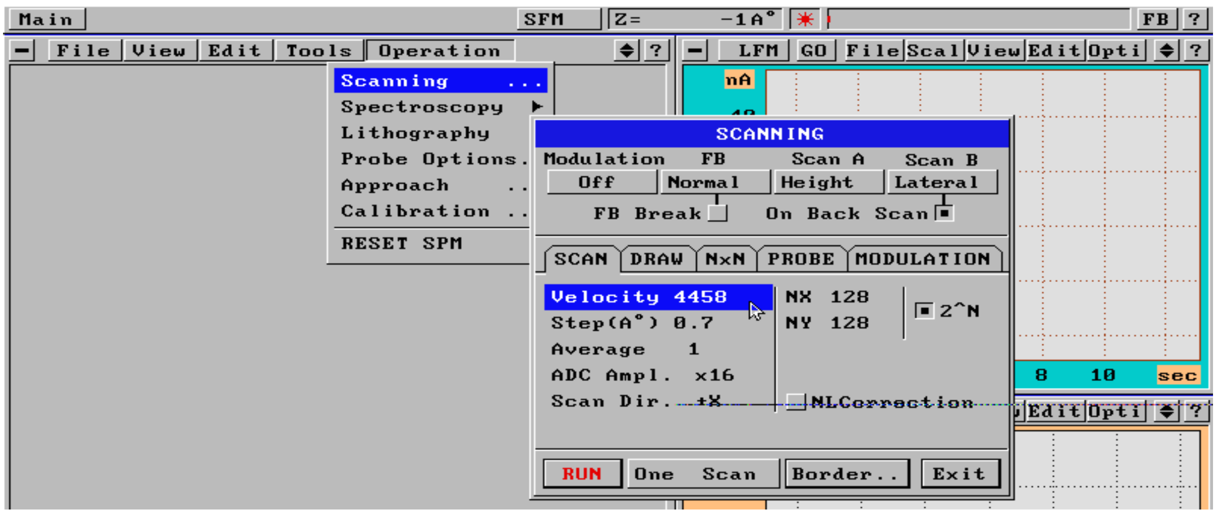


Fig. 73

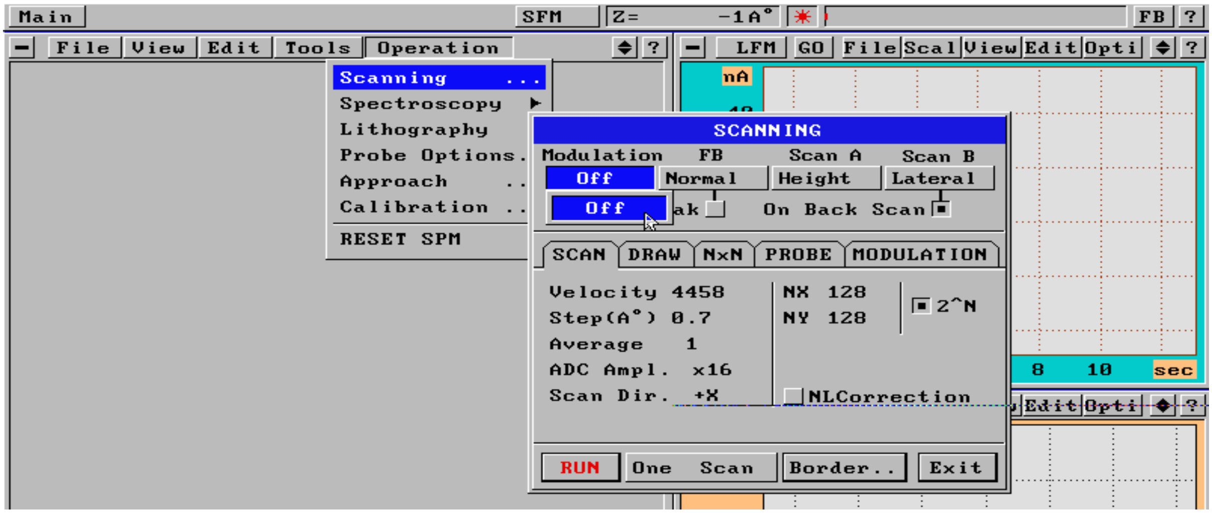


Fig. 74

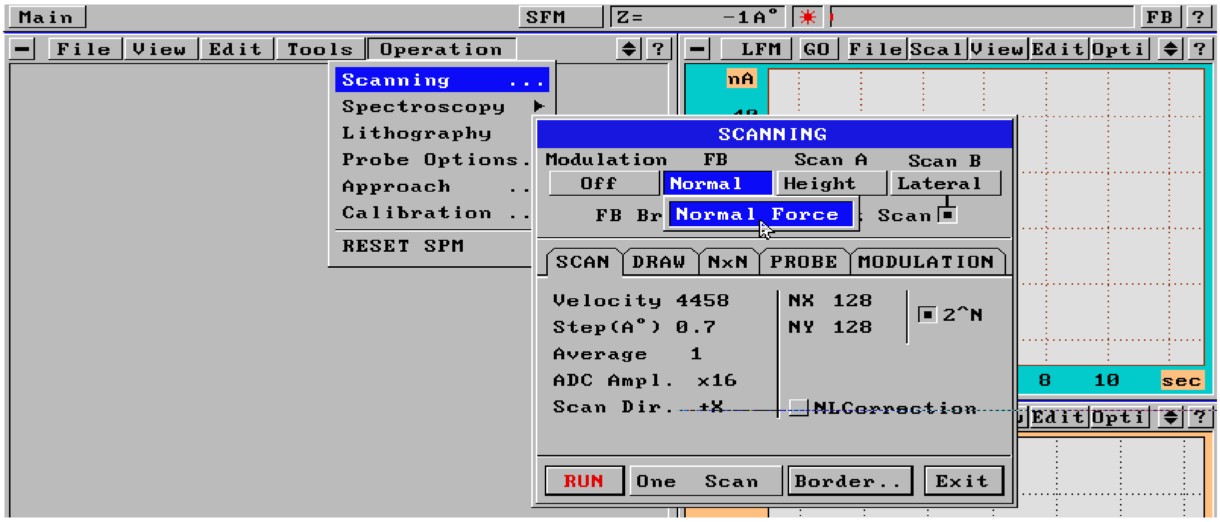


Fig. 75

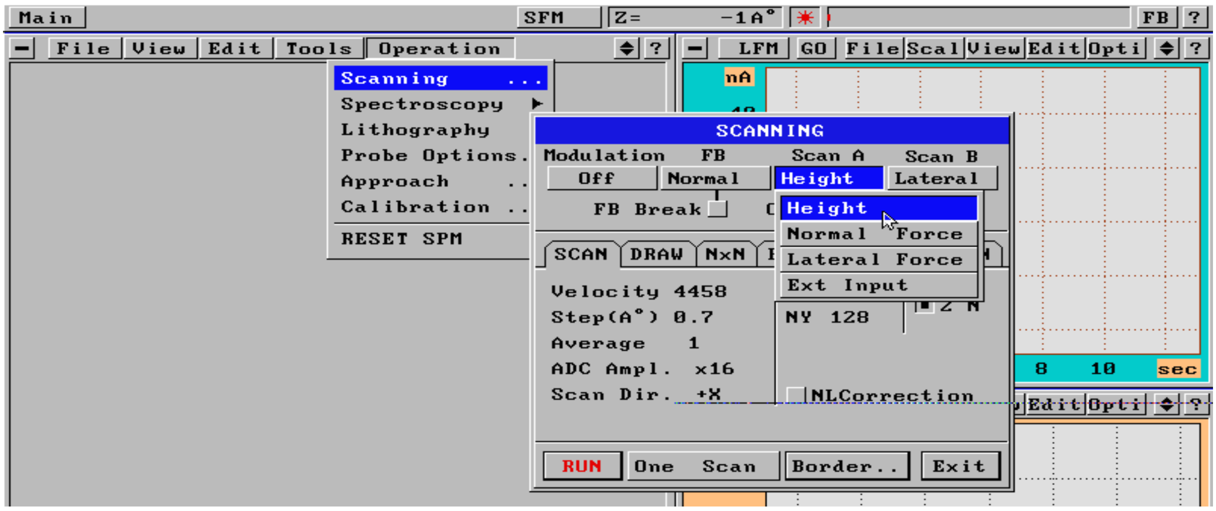


Fig. 76

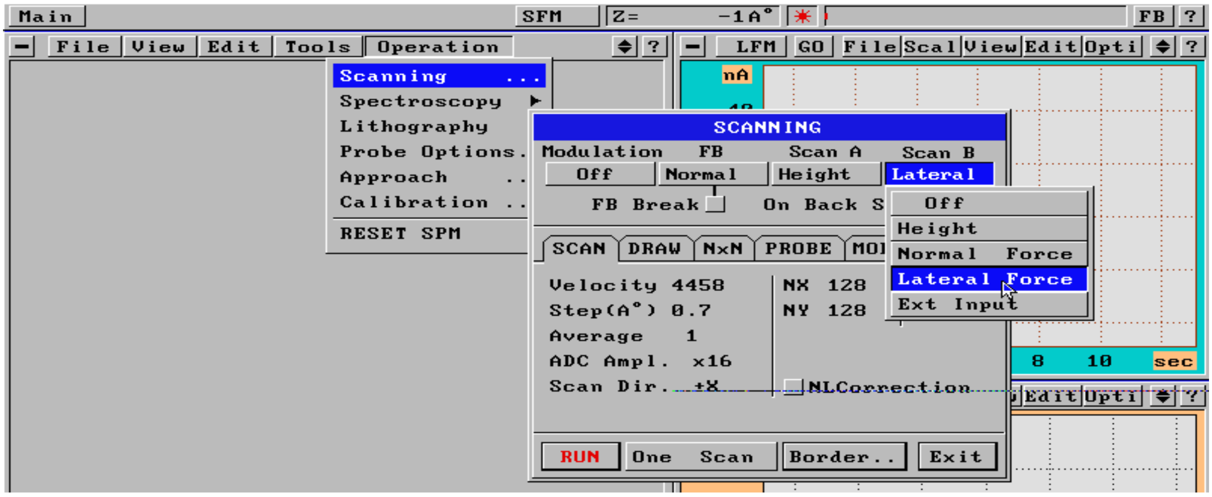


Fig. 77

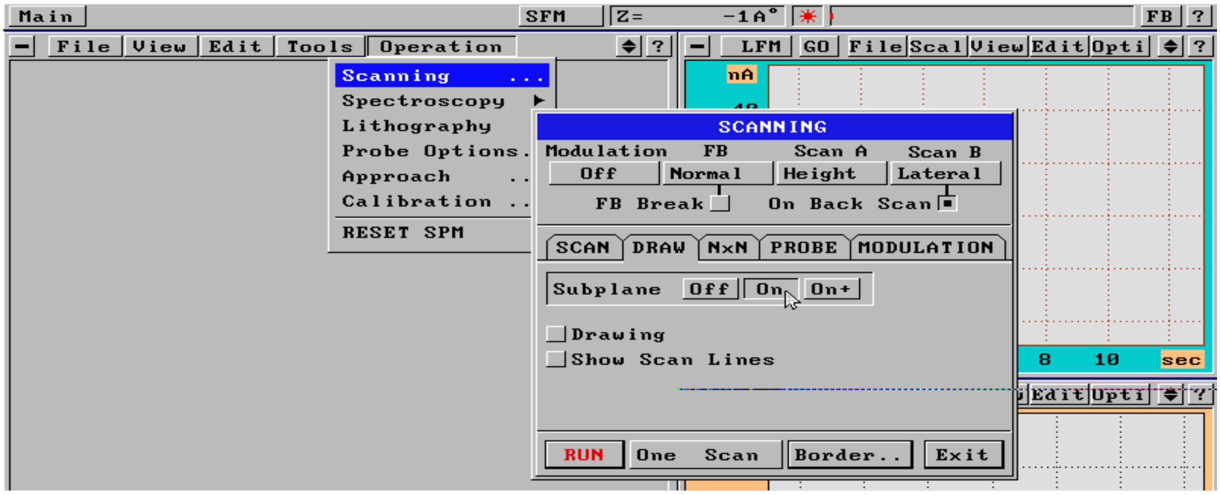


Fig. 78

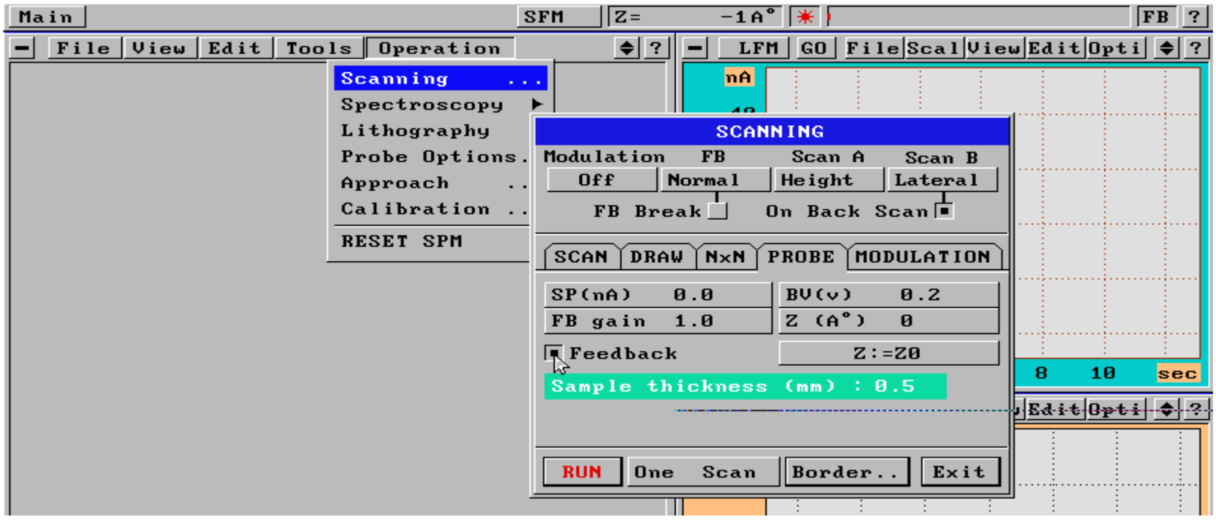


Fig. 79

3.1.7.1 Getting atomic lattice resolution (for 7um and 14um-range scanners)

Set the following values of parameters:

Modulation - off (Fig. 74)
FB - Normal Force (Fig. 75)

Scan A - Height (Fig. 76)
Scan B - Lateral Force (Fig. 77)
Velocity = max (Fig. 73)
One Back Scan = on (Fig. 73)
ADC Ampl = õ16 (Fig. 73)
Average = 1 (Fig. 73)
Step (A) = min (Fig. 73)
NX xNY = 128 õ 128 (Fig. 73)
Scan dir - +X (Fig. 73)
Drawing - off (Fig. 78)
Subplane - on (Fig. 78)
NL Correction - off (Fig. 73)
SP = 0 (Fig. 79)
FB Gain = 1 (Fig. 79)

The minimum step of scanning and maximum velocity depend on the specific configuration of SPM. Prior to scanning carry out the operation Z:=Z0 (Fig. 80).

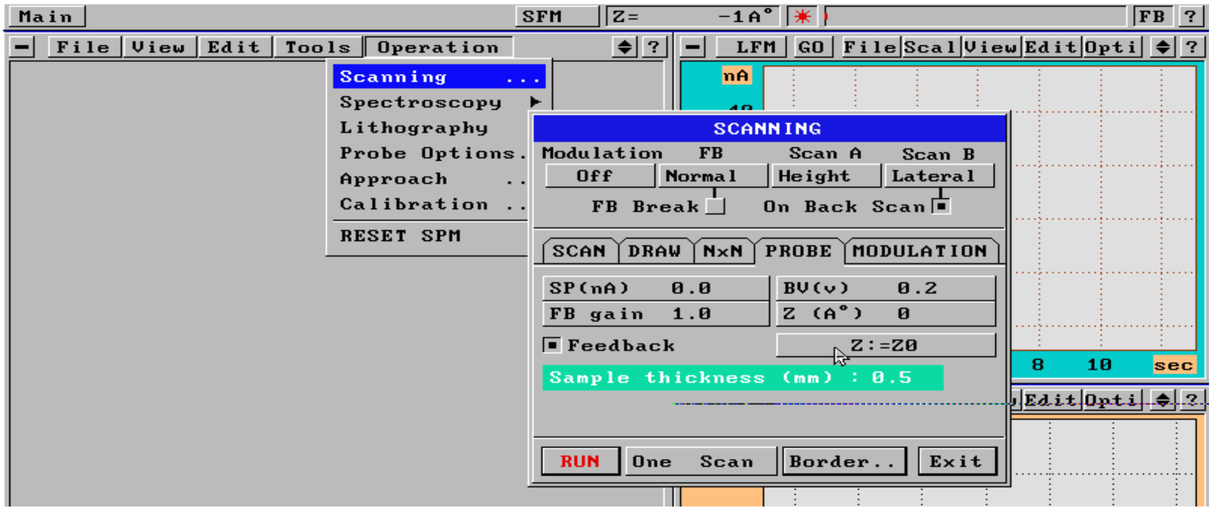


Fig. 80

Select the single scanning mode - One scan (Fig. 81).

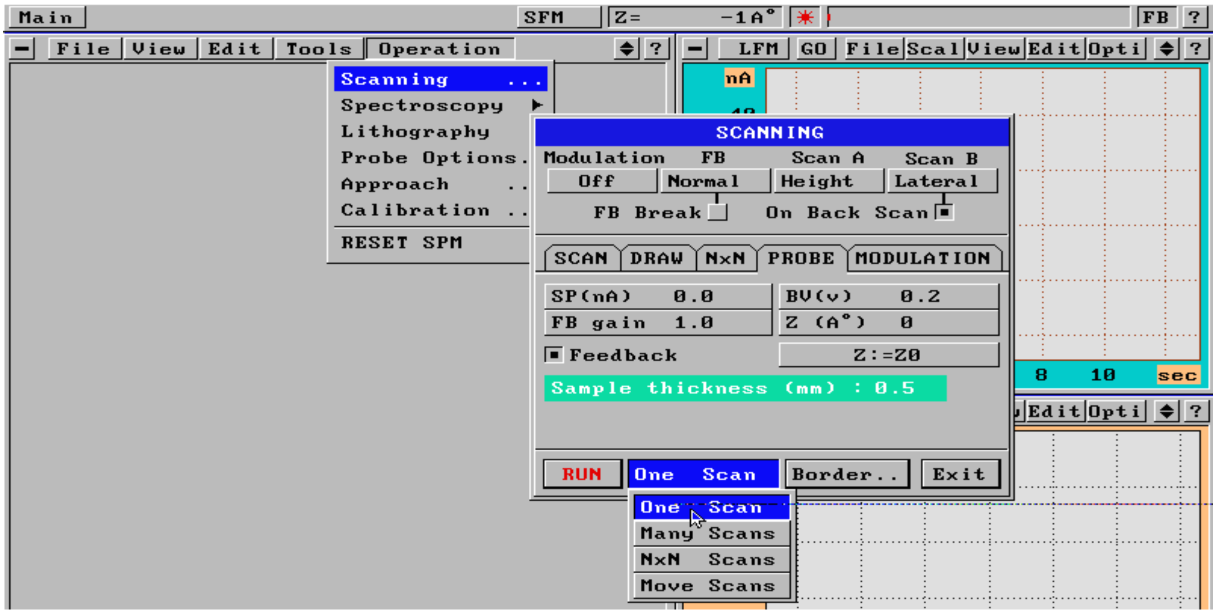


Fig. 81

Click RUN to start scanning (Fig. 82).

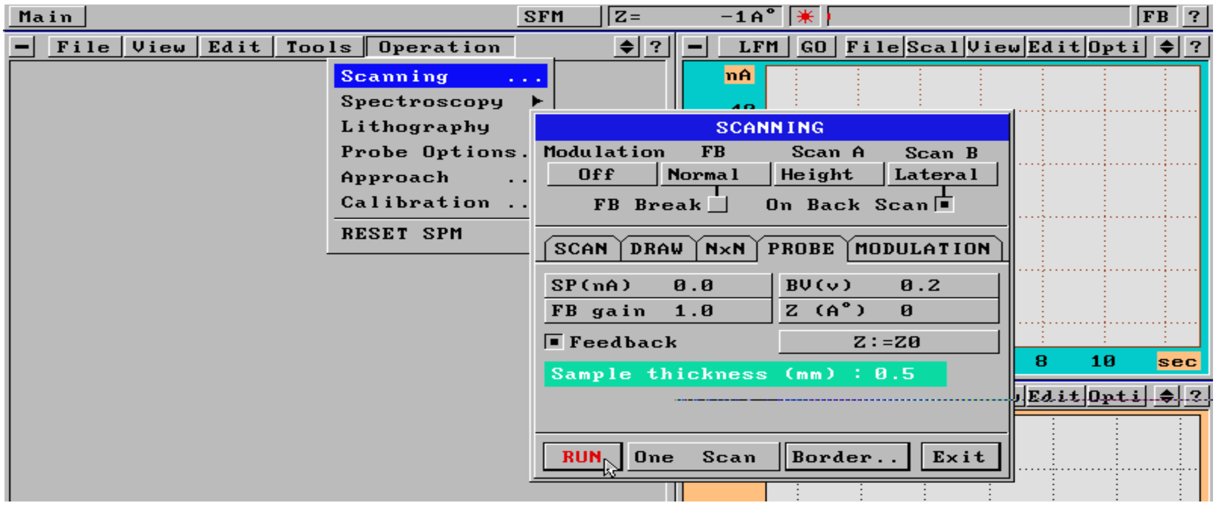


Fig. 82

3.1.7.2 Getting scan of a test grating

Set the following values of parameters:

Modulation - off (Fig. 83)
FB - Normal Force (Fig. 84)
Scan A - Height (Fig. 85)
Scan B - Lateral Force (Fig. 86)
Velocity = 500000 A/ñåê (Fig. 87)
Average = 10 (Fig. 87)
ADC Ampl - x1 (Fig. 87)
NX x NY = 128 õ 128[5] (Fig. 87)
Step (A) = max (Fig. 87)
Scan dir - +X (Fig. 87)
Drawing - on (Fig. 88)
Subplane - on+ (Fig. 88)
NL Correction - on (Fig. 87)
FB Gain = 1 (Fig. 89)
SP = 0 (Fig. 89)

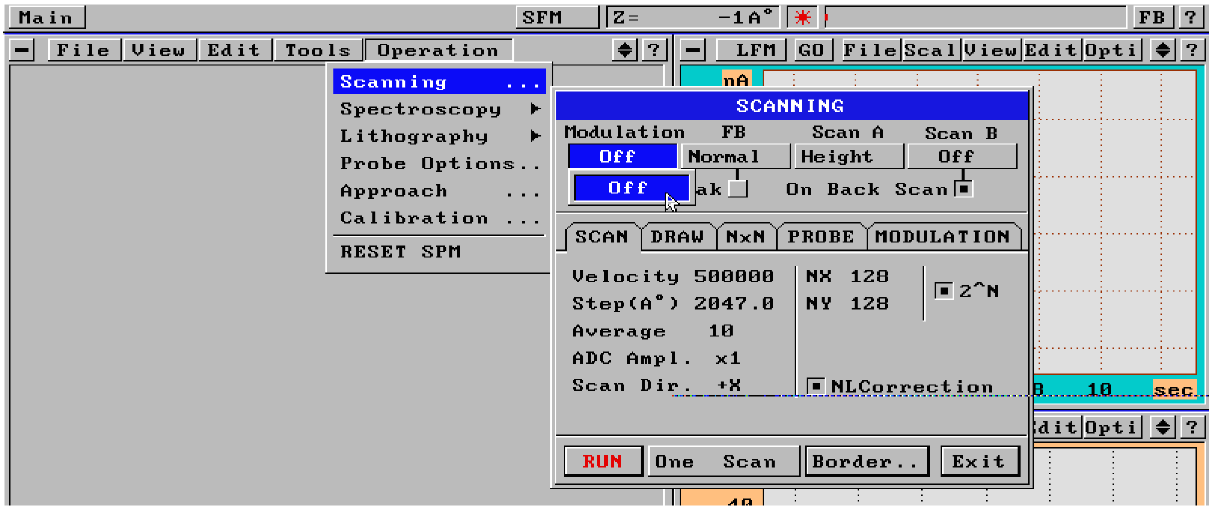


Fig. 83

[5] 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.

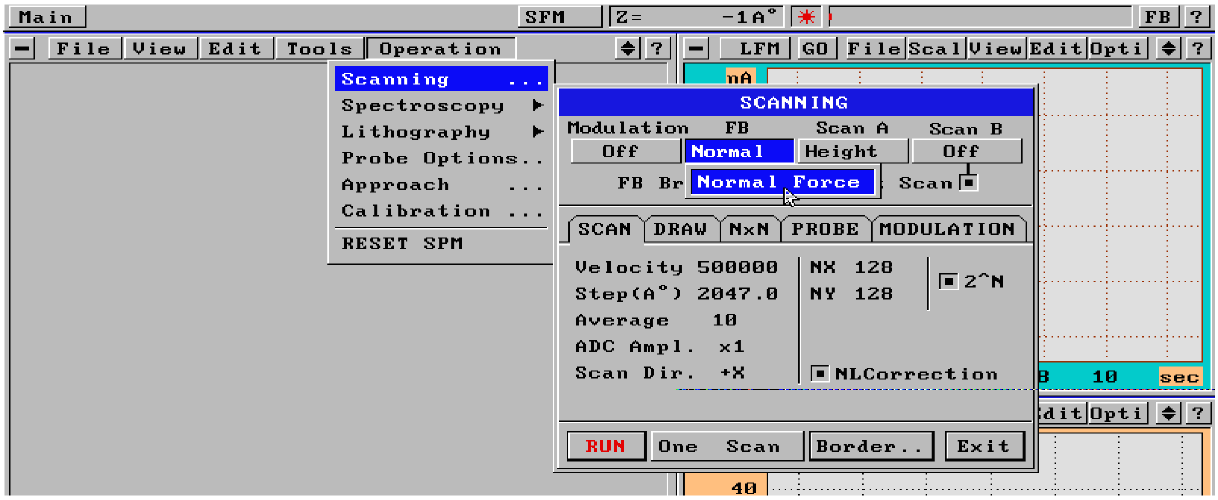


Fig. 84

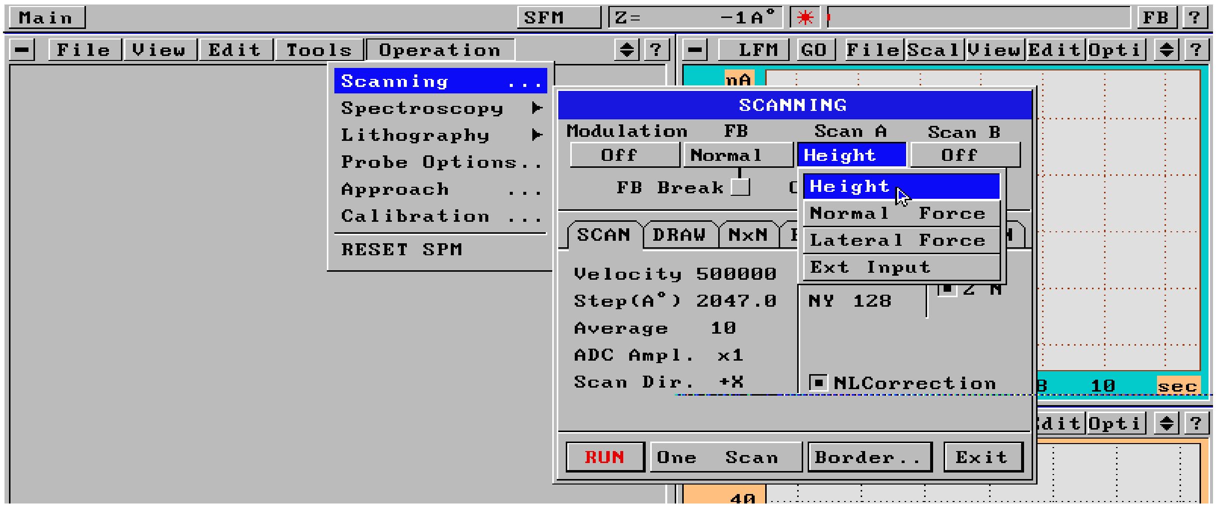


Fig. 85

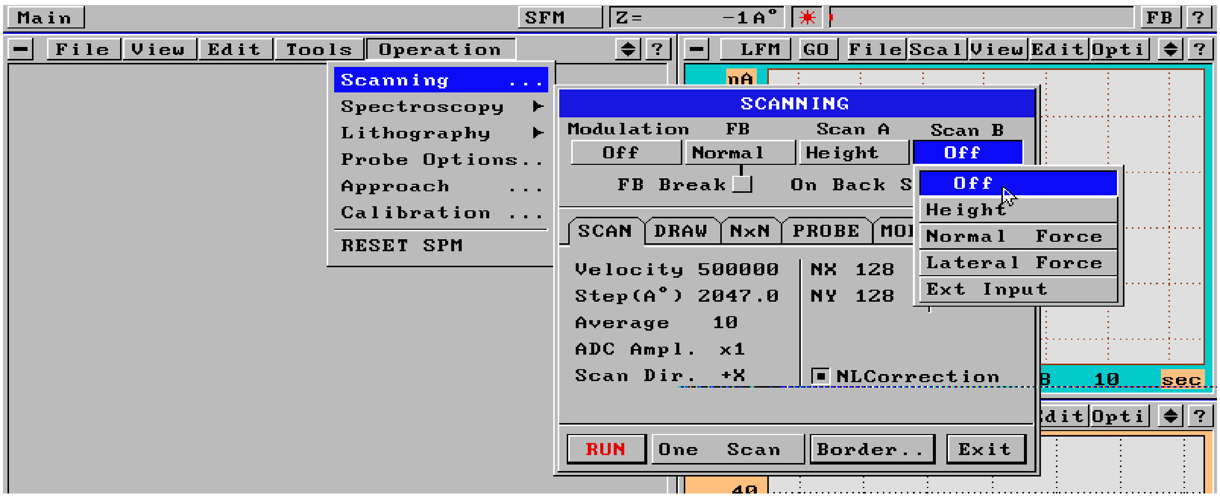


Fig. 86

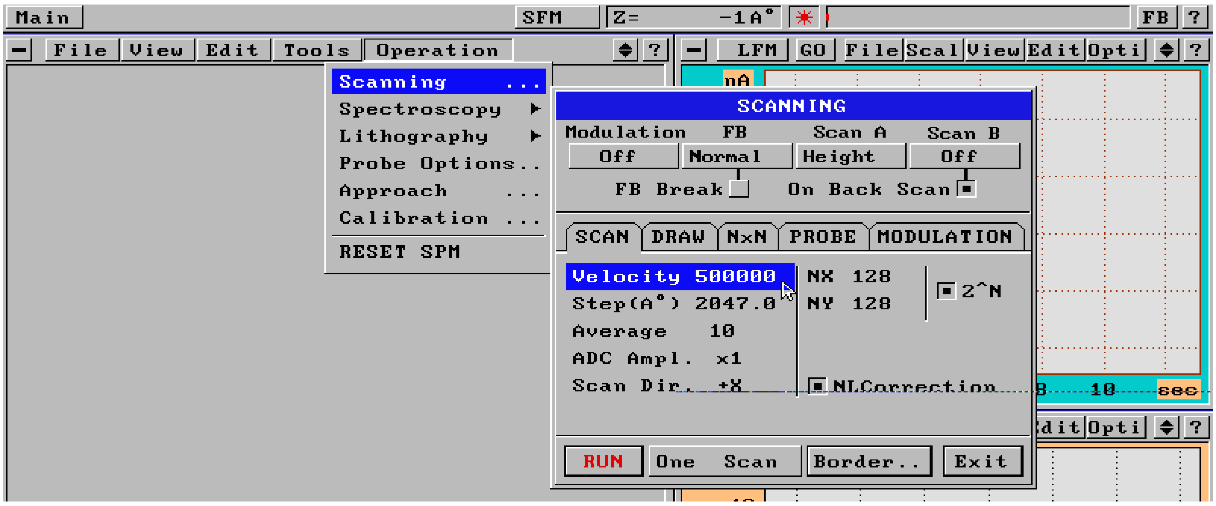


Fig. 87

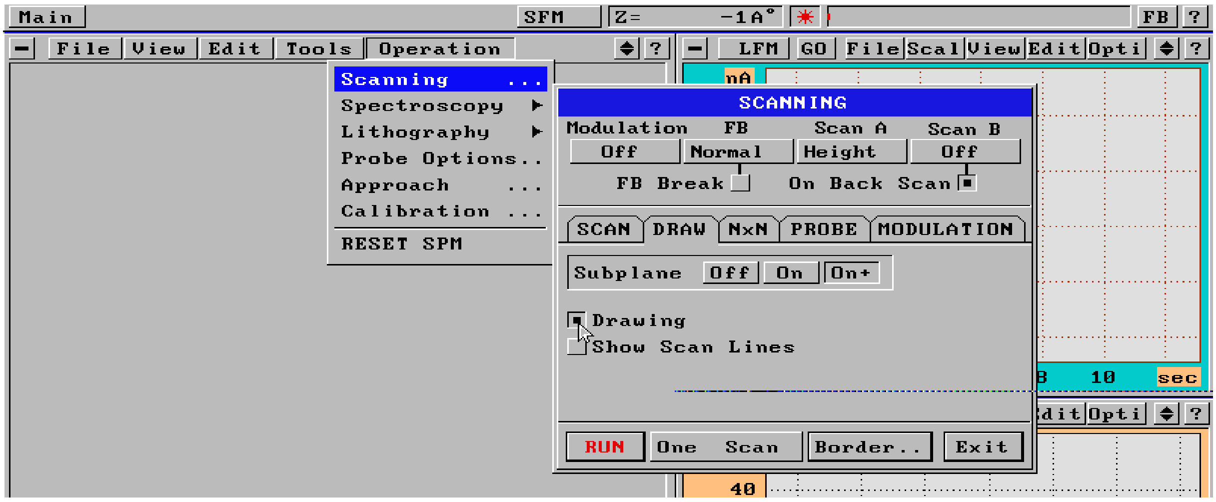


Fig. 88

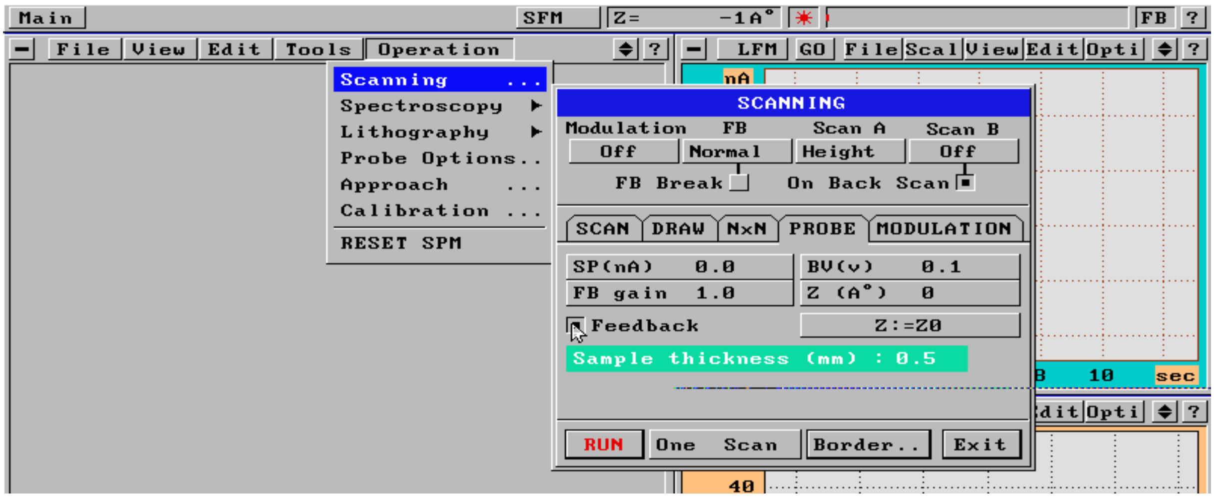


Fig. 89

Select the single scanning mode - One scan (Fig. 90).

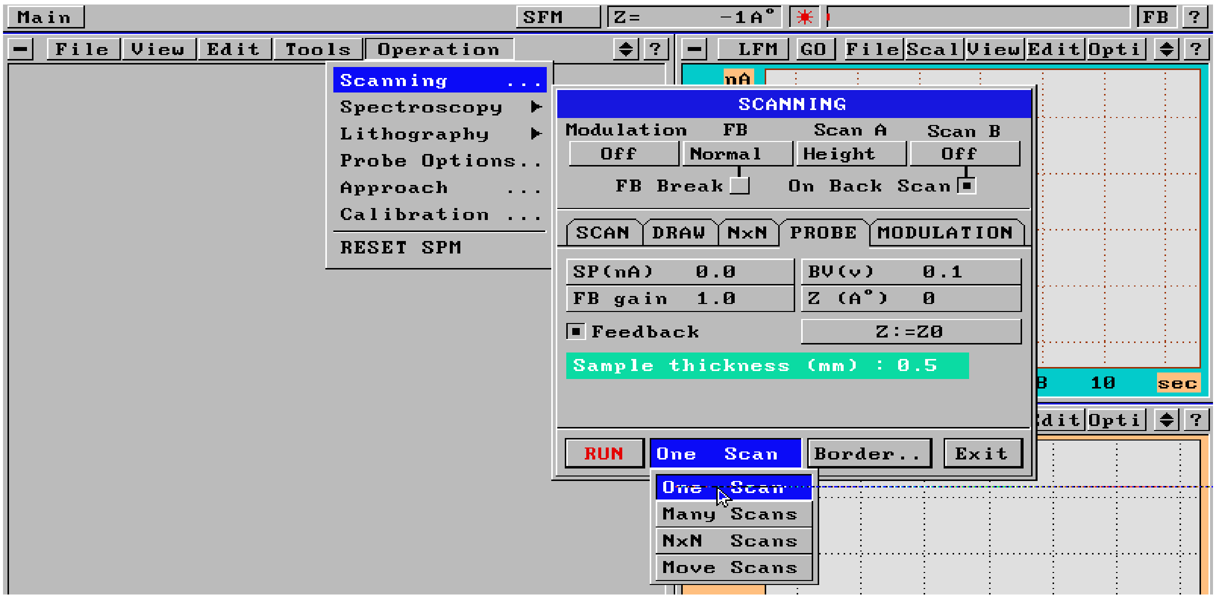


Fig. 90

Click RUN to start scanning (Fig. 91).

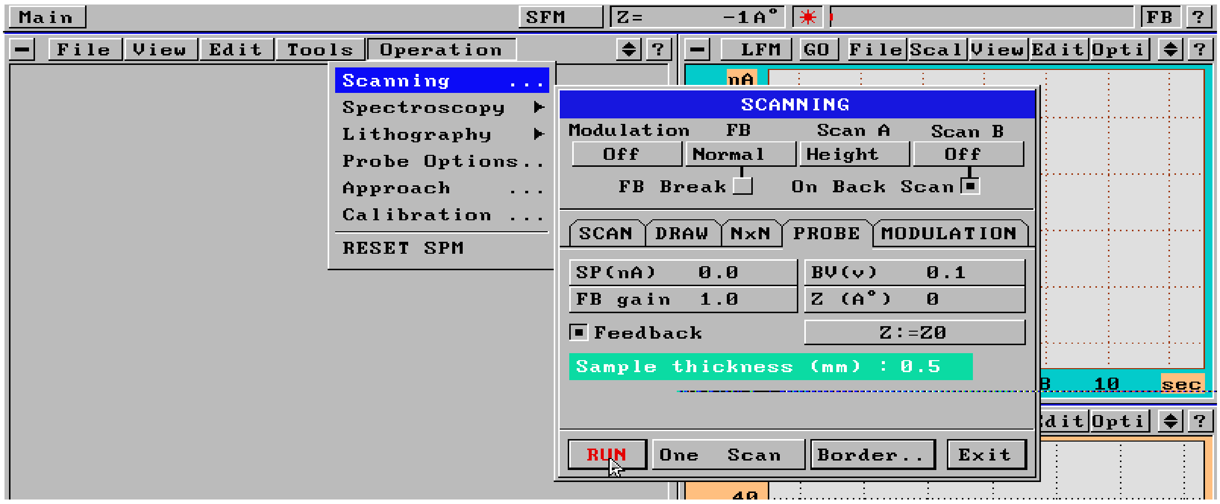


Fig. 91

After getting about 5-10% of the scan lines, click “RESTART” in the top string menu that appears as soon as scanning has been started (use keyboard ENTER to click “RESTART” and keyboard arrows to choose items of that string and to change their values). This operation will reduce residual nonlinearity in the slow scanning direction (Fig. 92).

Fig. 92

3.1.8 Shutdown procedure

Point the button "Operation" in the top of the “SPM” Window and click “Approach” to open the menu "Mover" (Fig. 93). Click ”Backward” while pressing SHIFT on the keyboard (Fig. 94). Step motor will retract sample from the probe until you press ESC on the keyboard.

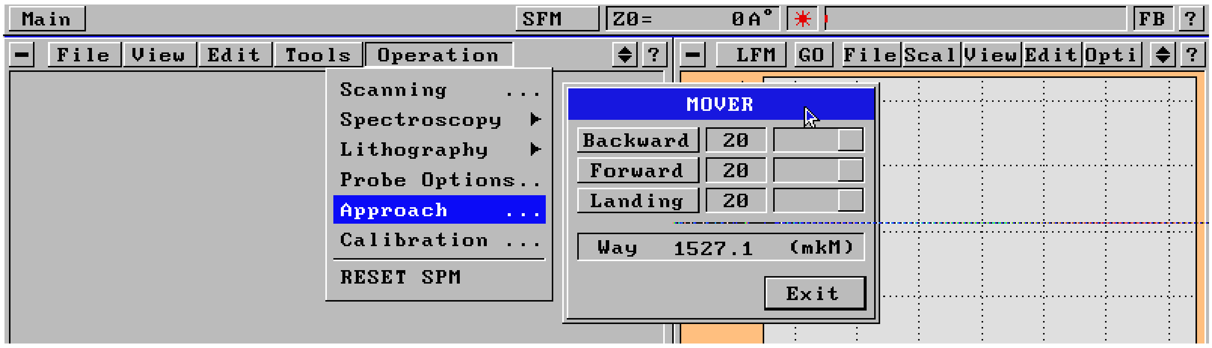


Fig. 93

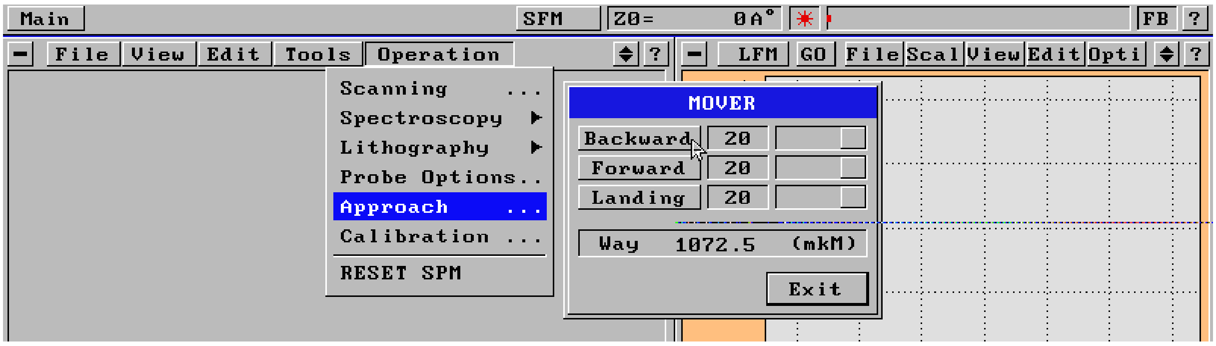


Fig. 94

ATTENTION

Be careful: Don't click FORWARD while pressing SHIFT if sample is close to the probe. Probe and sample can be damaged if collide with each other.

As soon as the counter shows about 500-1000 um back (Fig. 94), you can press ESC to stop moving.

1) Click indicator of the piezotubelength in the top right corner of the screen ("red line") to open the menu "Probe options". Turn off feedbackand laser emission (Fig. 95, Fig. 96). Lift scanner unit, slightly rotate it clockwise and put it onto three supporting rods (Fig. 62). You can also further retract sample from probe manually by rotating approach knob (Fig. 61) clockwise (bottom view).

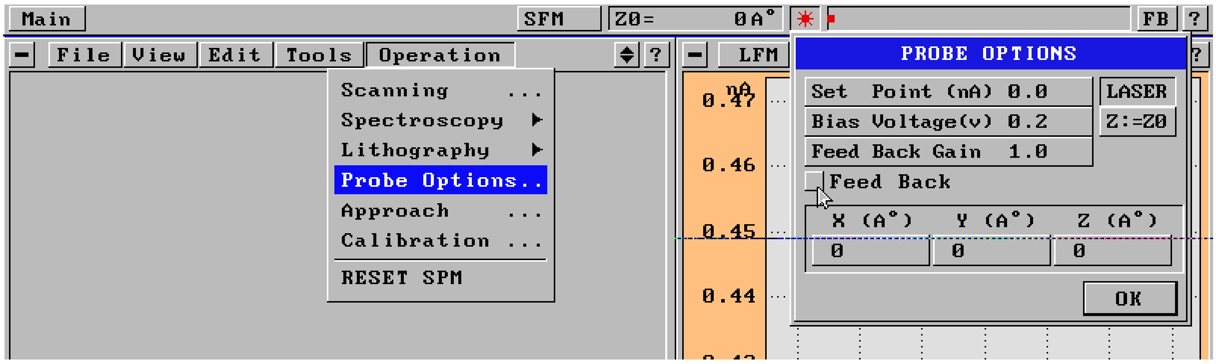


Fig. 95

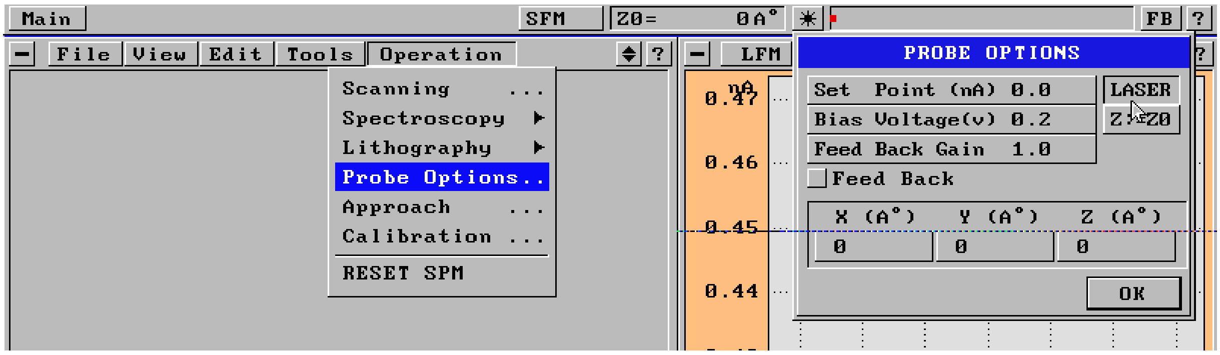


Fig. 96

2) Put aside the SPM head.

3) Carefully remove sample from the holder. Don't exert big force: it can damage the holder or break the piezotube.

4) Turn off the switch on the front side of power supply unit.

5) Click the button "MAIN" in the top left corner of the screen and then click "EXIT" (Fig. 97) to quit the program. Keyboard button F10 can also be used for this purpose. All open menus should be closed by pressing ESC before quitting the program, otherwise F10 doesn't work.

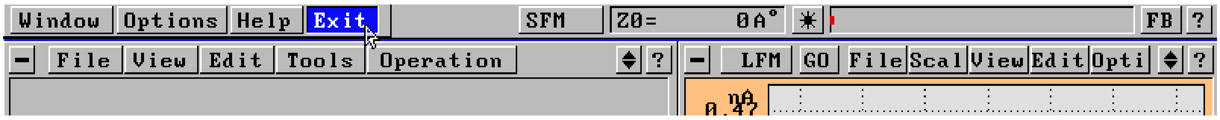


Fig. 97

3.2 STM-mode

3.2.1 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. 27.3) 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.

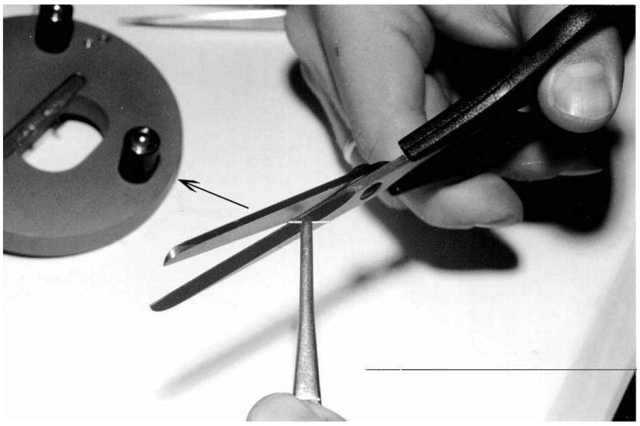


Fig. 98

The wire is clamped in the clip so that it comes out on one side for 2-3 mm (Fig. 98). 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.[6]
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. 100).
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. 99). 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.

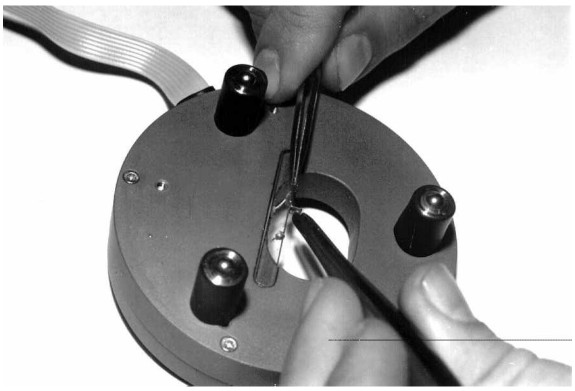


Fig. 99

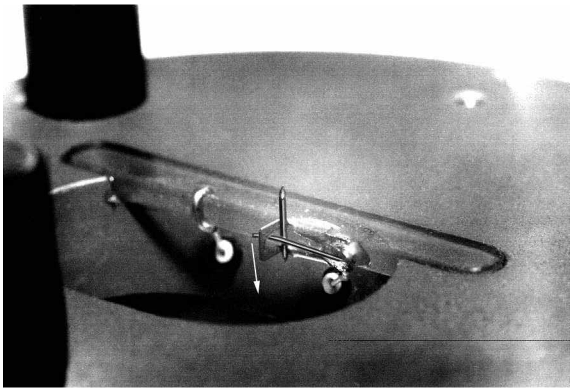
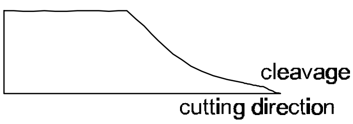


Fig. 100

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:

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



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.

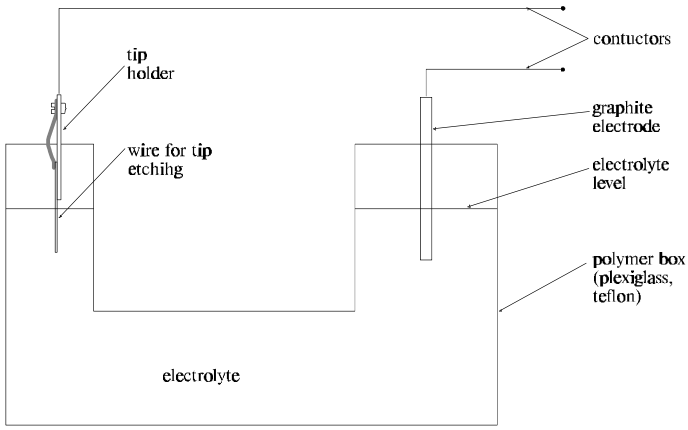


Fig. 101 Installation for tip etching.

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.

3.2.2 Preparing and installing the sample

Substrates having electrical contacts for bias voltage application are appropriate for STM investigations (Fig. 27.7).
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. 102).
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. 102).

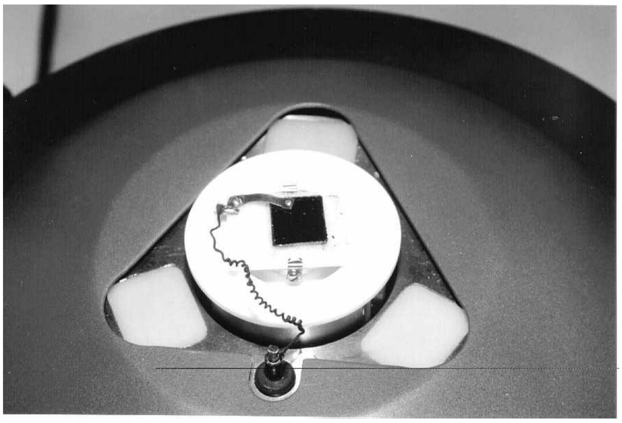


Fig. 102

3.2.3 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. 61).
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. 62). Insert the holder on the cable into the socket (Fig. 63).
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. 61, Fig. 64).
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. 13).

3.2.4 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. 103). The window for the selection confirmation will be displayed, press “Yes" (Fig. 104).

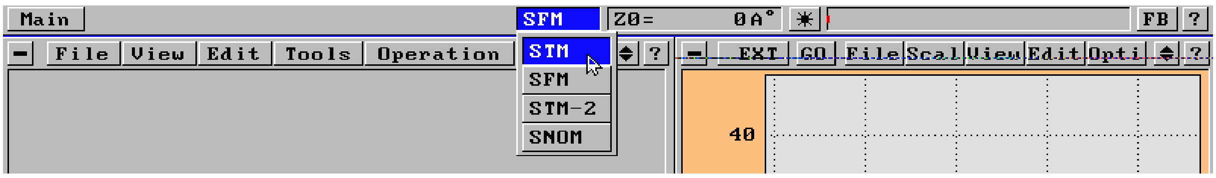


Fig. 103

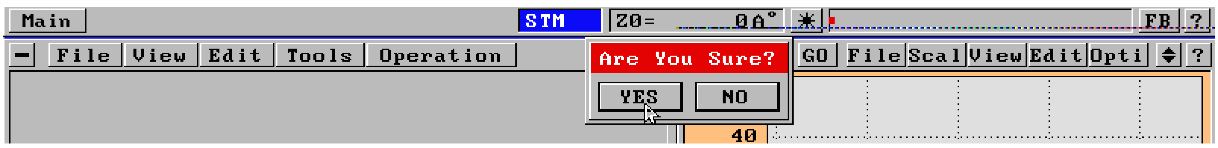


Fig. 104

Move the mouse pointer to the Operation button. In the opened menu select the “Probe Options” item (Fig. 105) and press the left mouse button. Set the sample's approach parameters "Set Point" = 0,1nA, "Bias Voltage" = 0,1B, "Feedback Gain"=maximum (Fig. 106).

After closing the menu enter the 'Approach' menu (Fig. 107). Set the maximum approach speed (Landing) = 20 and turn on "Landing" (Fig. 108). The approach is done automatically. To carry out fine investigations (atomic-skale objects) bring the approach system into the backlash position. To do this the "Forward" speed must be decreased to the value 3-4 (Fig. 109). Then press "Forward" (Fig. 110) while watching the analog indicator Z. When the analog indicator red band reduces to 1/3 - 1/4 of its normal length stop the movement by pressing the ESC key. Then press "Shift" + "Backward" and after reversing for about 100 um (Fig. 111), stop the movement by pressing the ESC key.

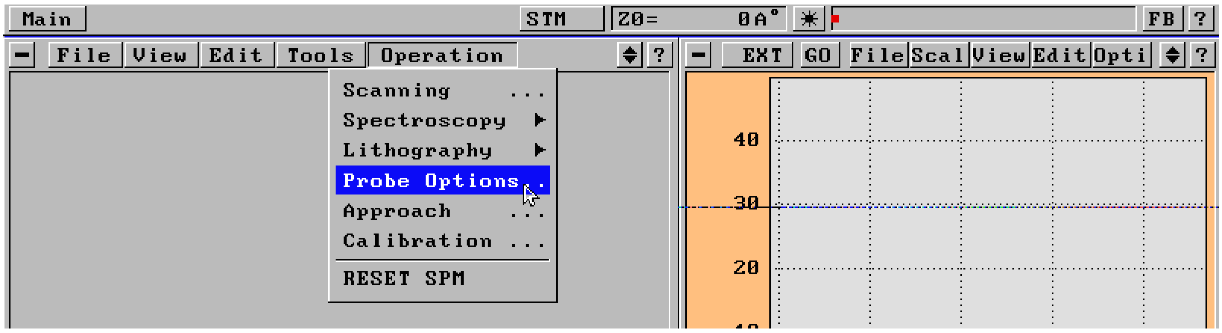


Fig. 105

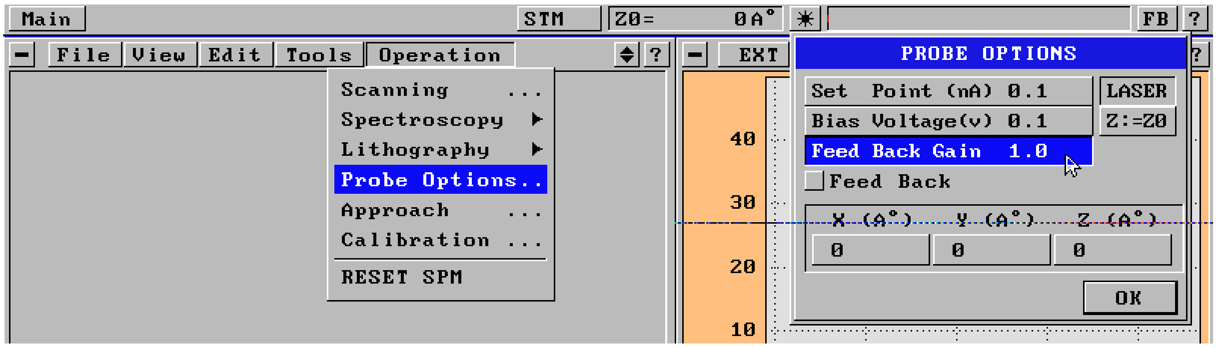


Fig. 106

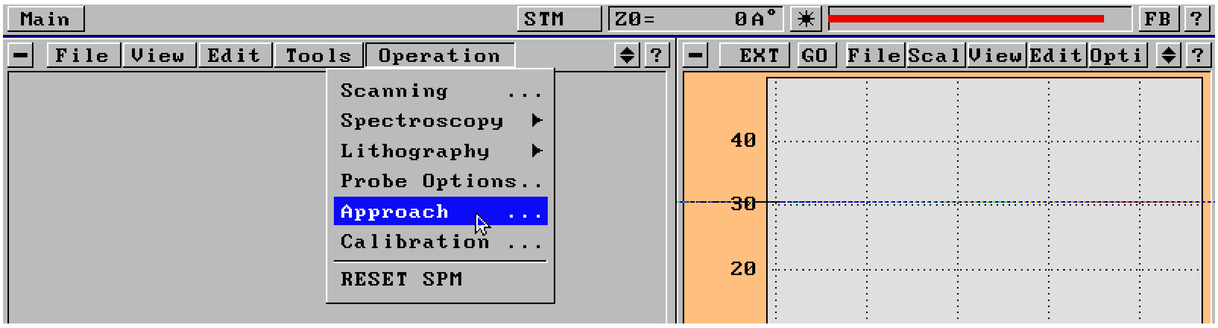


Fig. 107

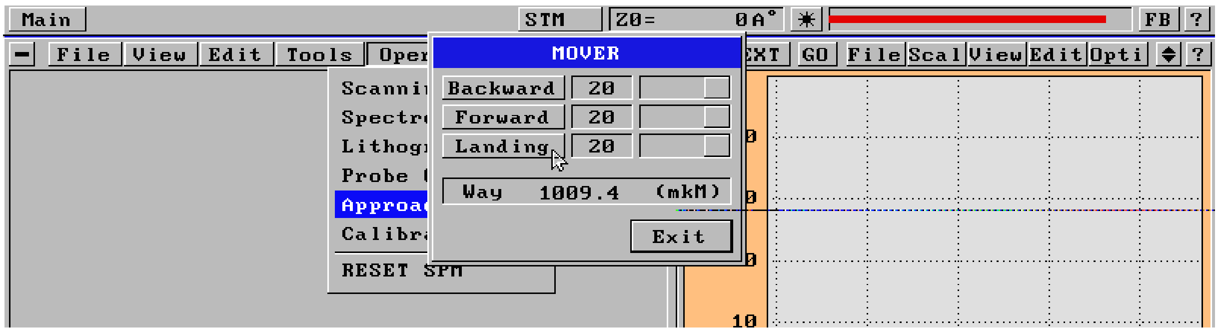


Fig. 108

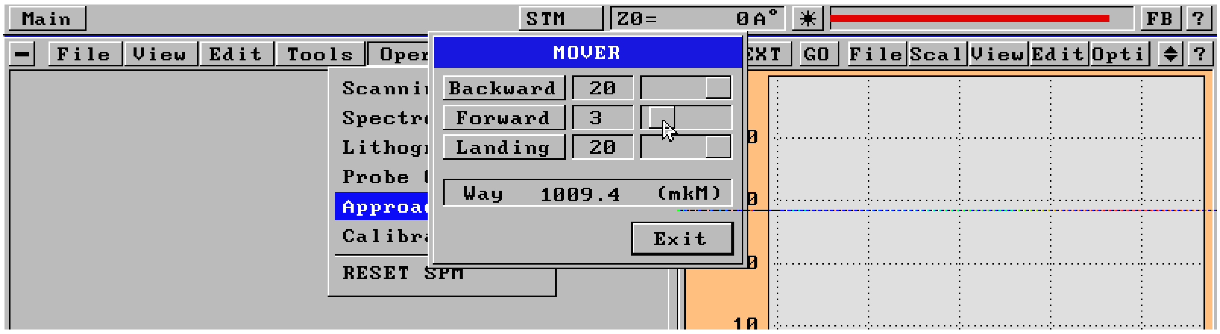


Fig. 109

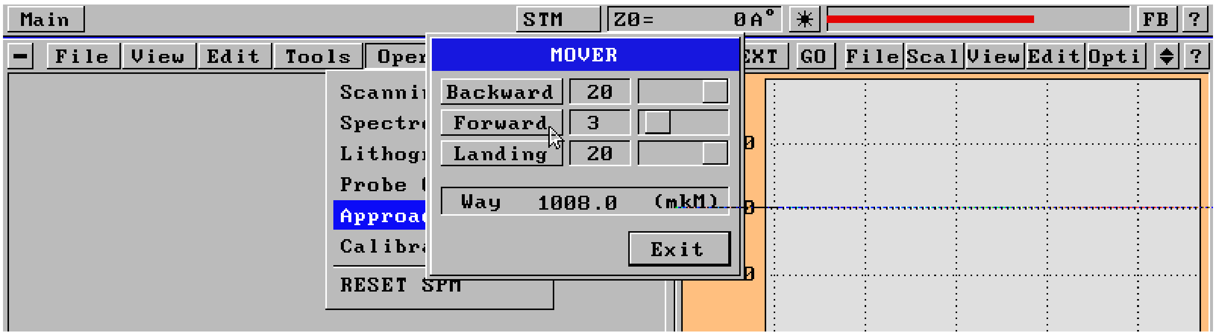


Fig. 110

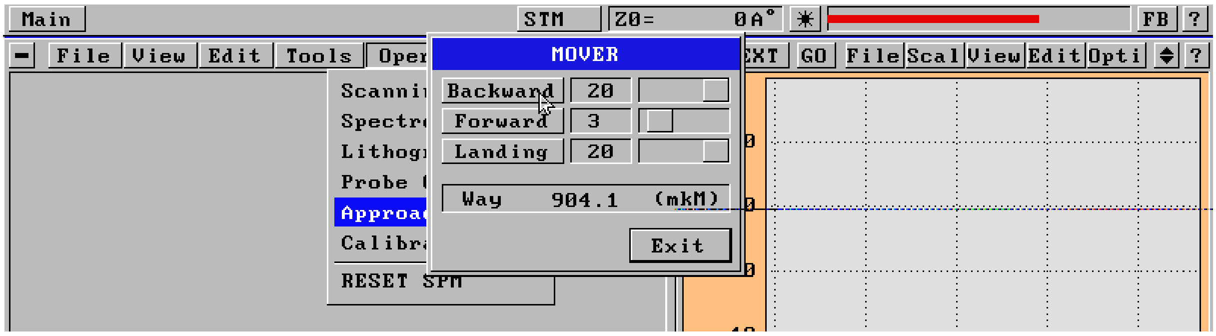


Fig. 111

3.2.5 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. 112). Select the "Scanning" (Fig. 113) item in the opened menu and press the left mouse button.
The following scanning parameters are set in the opened menu - speed, number of measurements in one point, reverse speed, signal amplification along the Z axis, scanning steps, scan dimensions in points, second signal to be registered, drawing operation when scanning, fitted plane subtraction, nonlinearity correction (Fig. 113, Fig. 114, Fig. 115, Fig. 116, Fig. 117, Fig. 118, Fig. 119).

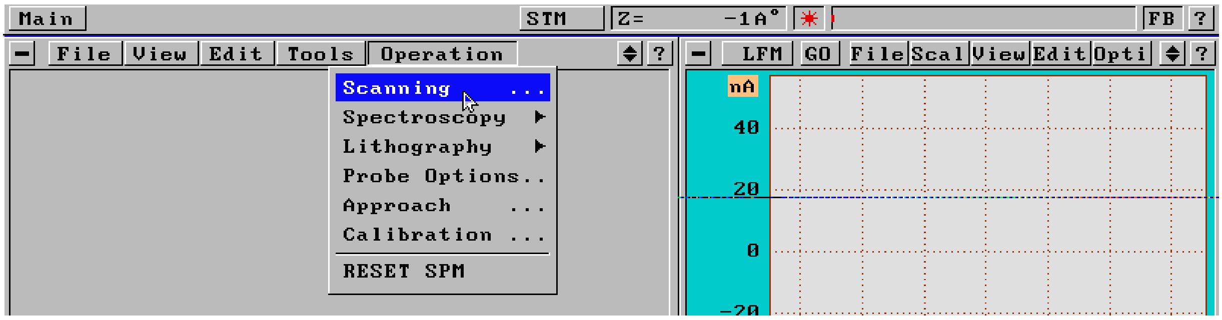


Fig. 112

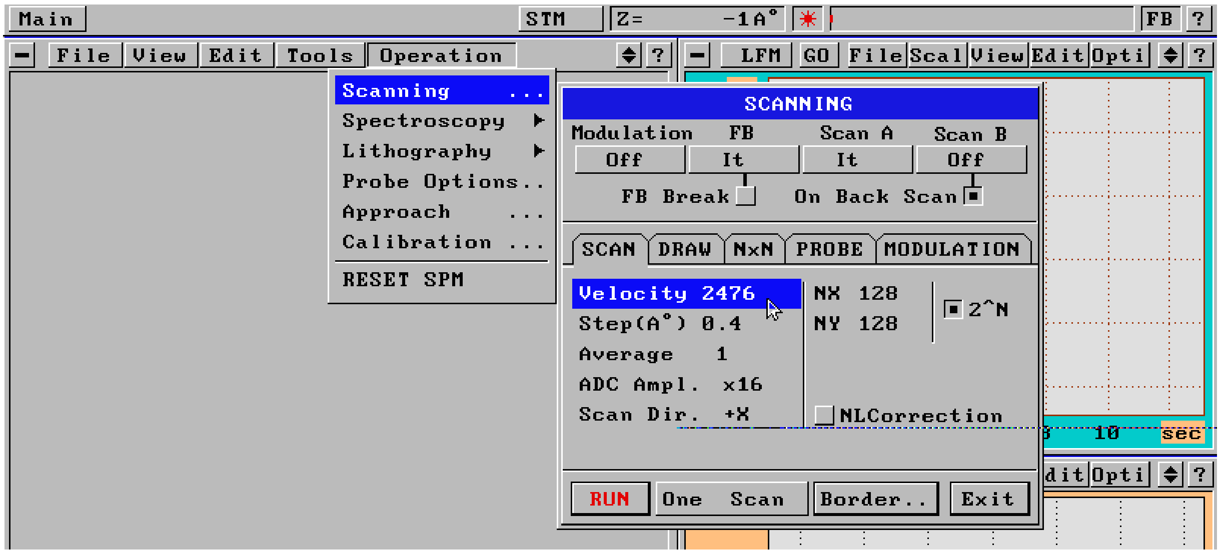


Fig. 113

3.2.5.1 Getting atomic resolutionon graphite

Set the following parameters:

Modulation - off (Fig. 114)
FB - It (Fig. 115)
FB Break - on (Fig. 113)
Scan A - It (Fig. 116)
Scan B - off (Fig. 117)
Velocity = max (Fig. 113)
ADC Ampl = õ16 (Fig. 113)
Average = 1 (Fig. 113)
Step (A) = min (Fig. 113)
NX xNY = 128 õ 128 (Fig. 113)
Scan dir - +X (Fig. 113)
Drawing - off (Fig. 118)
Subplane - on (Fig. 118)
NL Correction - off (Fig. 113)
FB Gain = 0.03-0.05 (Fig. 119)
SP = 1,0 - 1,5 (without letting generation appear) first decrease FB Gain, then increase the current (Fig. 119).
BV = 0.1 (Fig. 119)

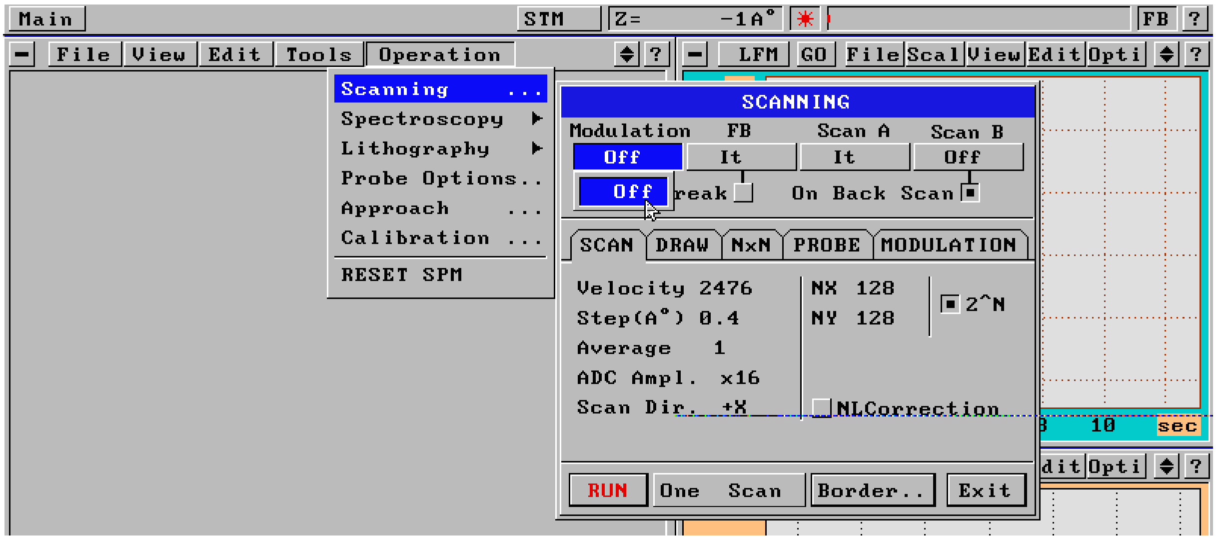


Fig. 114

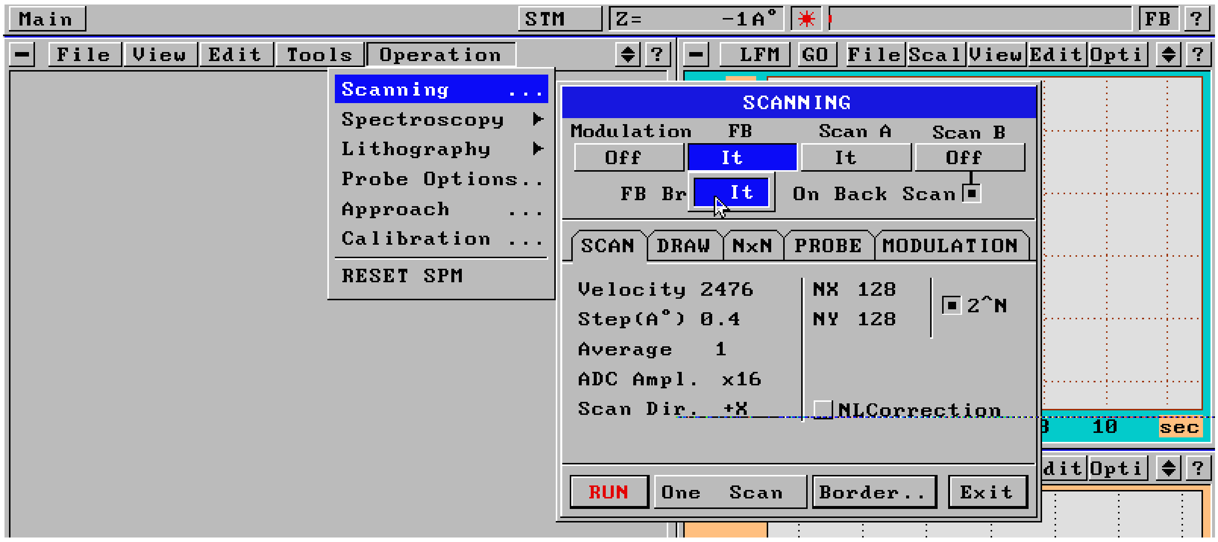


Fig. 115

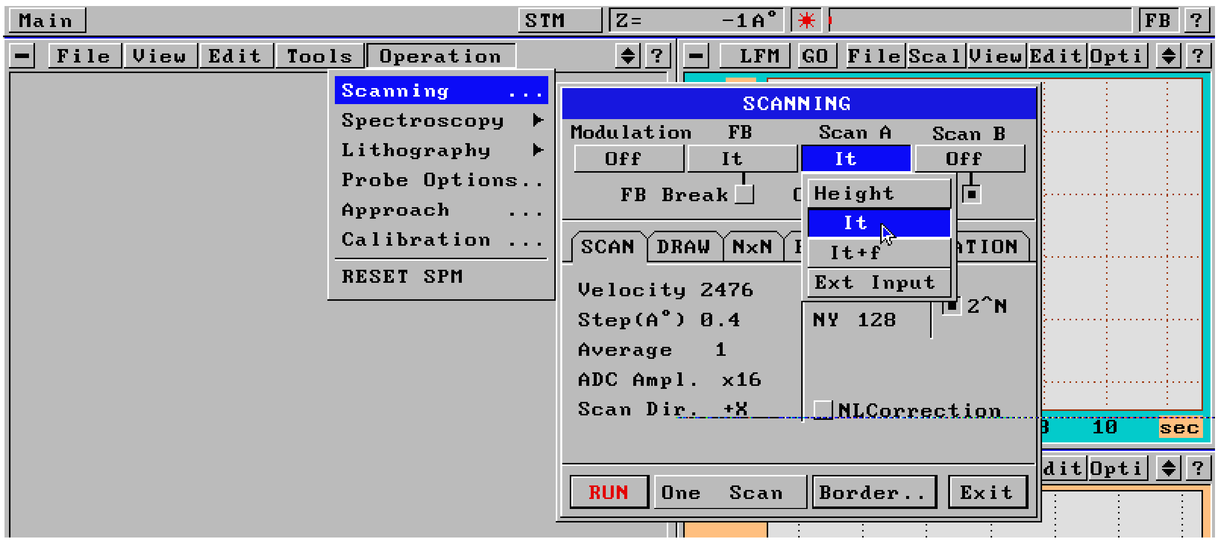


Fig. 116

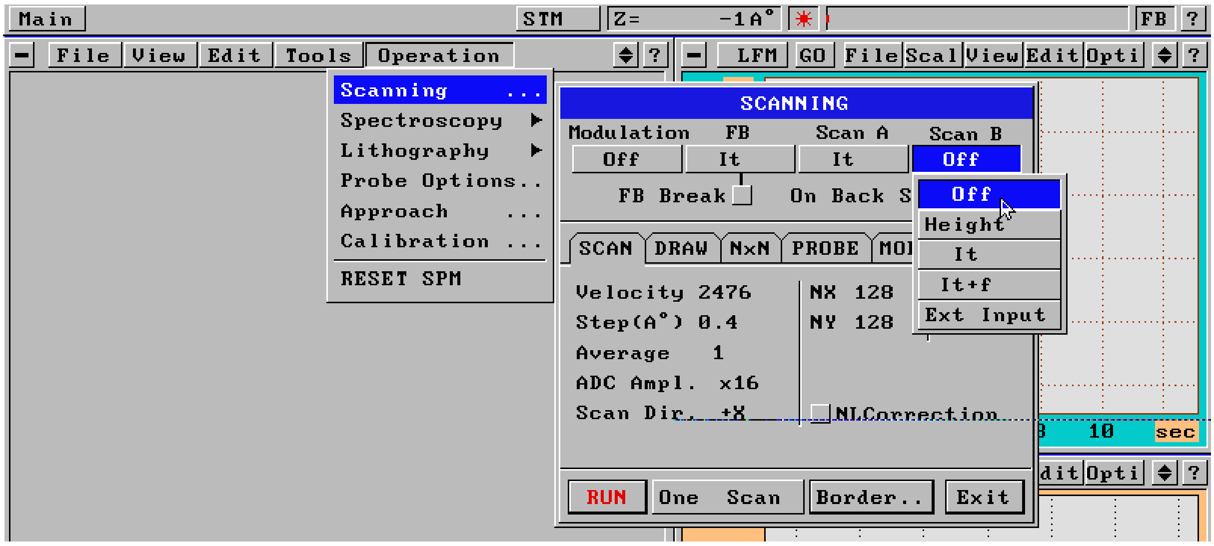


Fig. 117

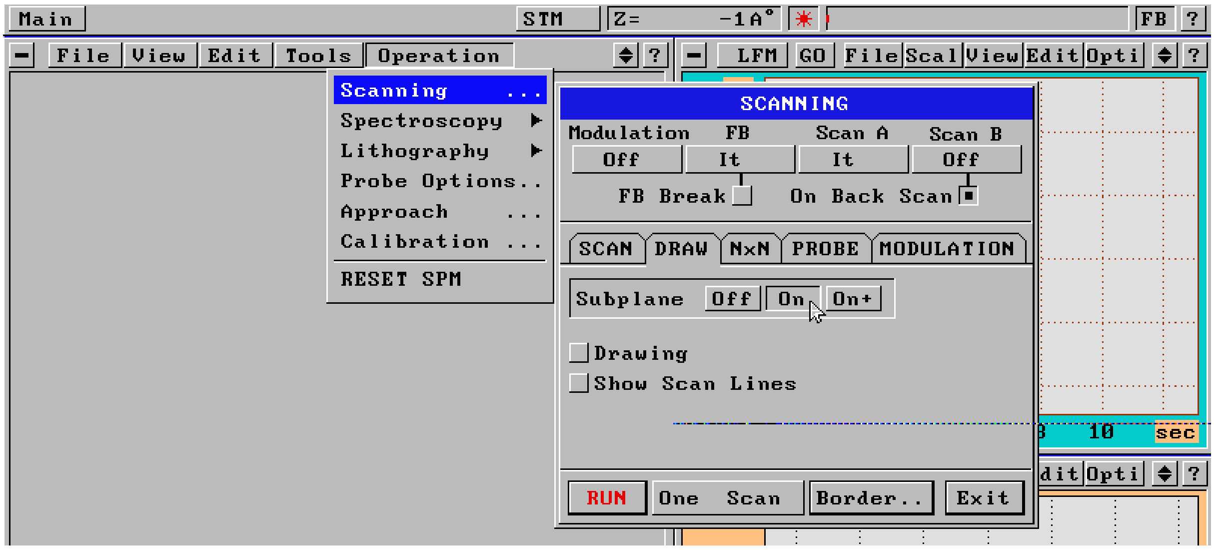


Fig. 118

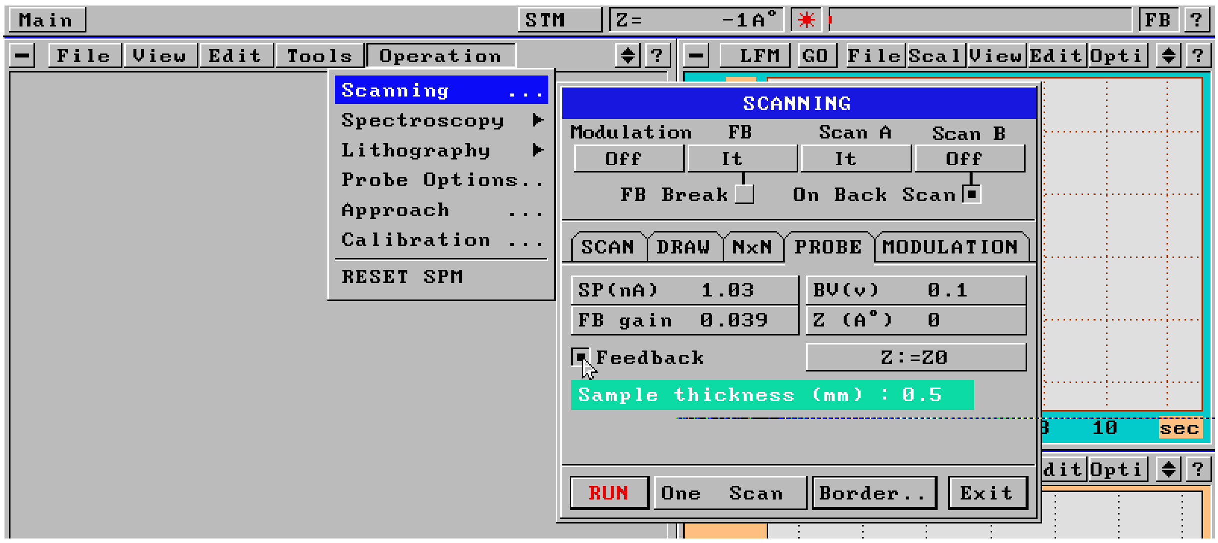


Fig. 119

Prior to scanning carry out the operation Z:=Z0 (Fig. 120).

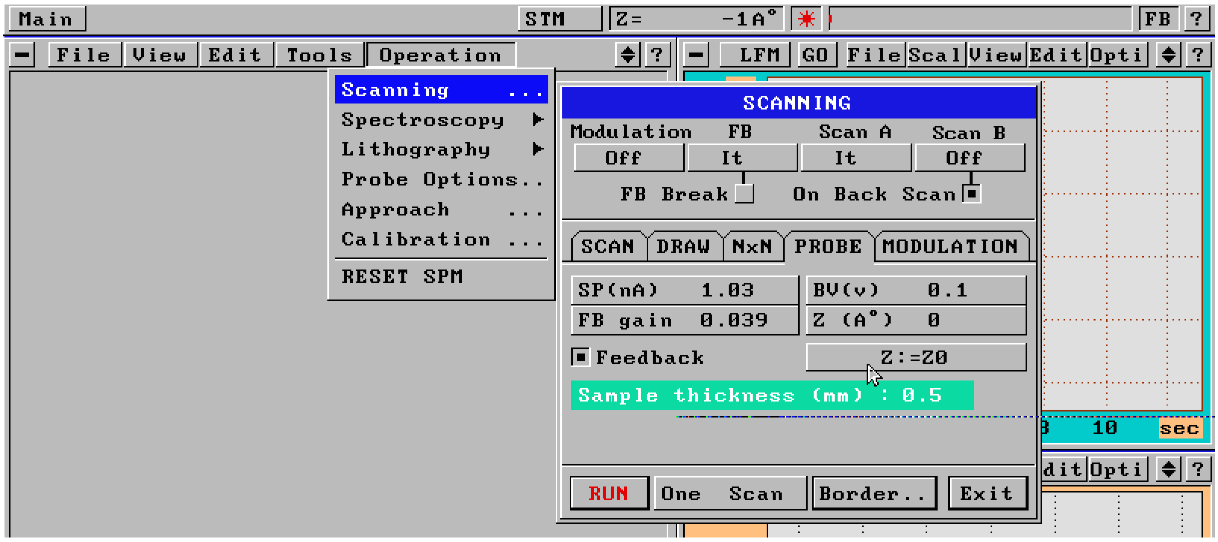


Fig. 120

Select the single scanning mode - One scan (Fig. 121).

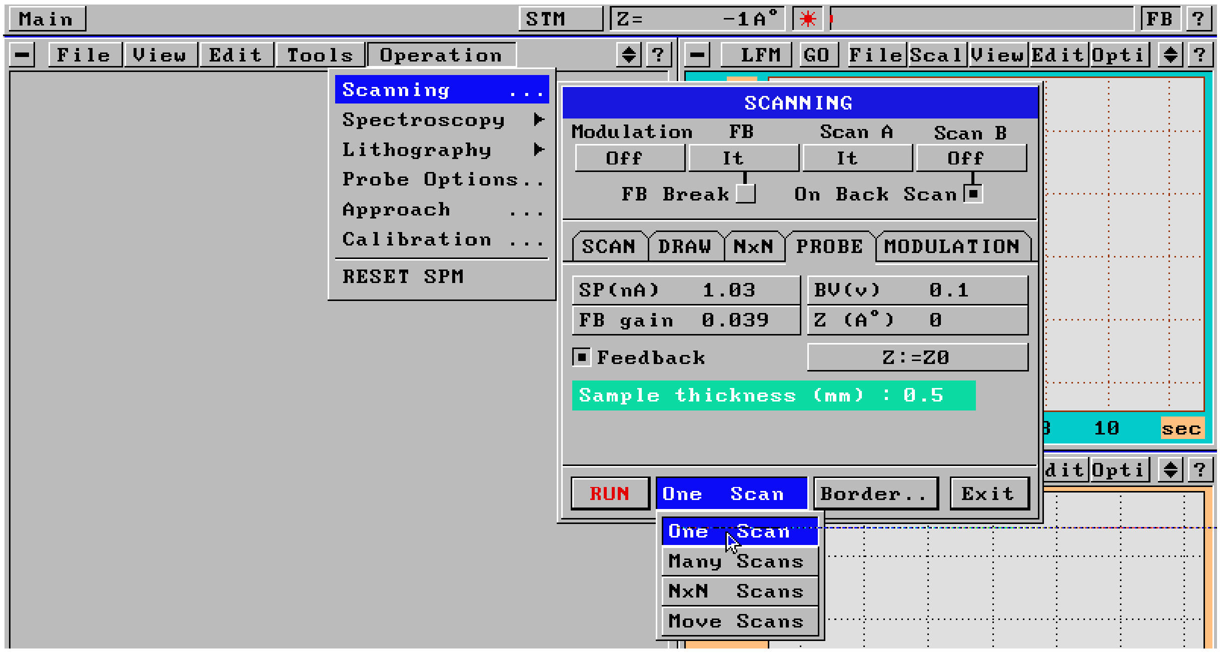


Fig. 121

Click RUN to start scanning (Fig. 122).

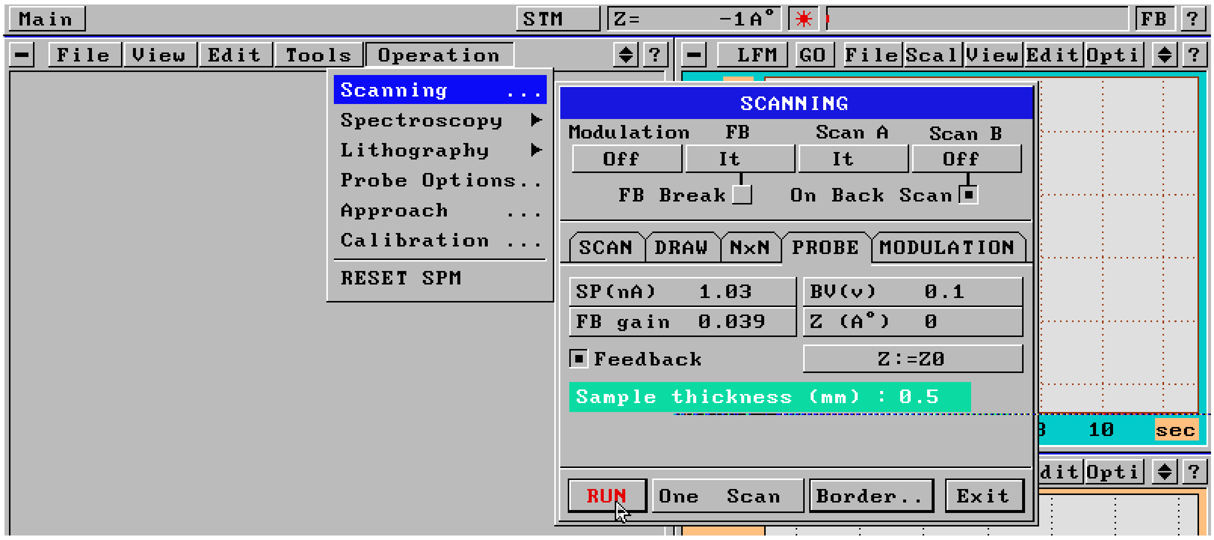


Fig. 122

3.2.6 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. 123 and press the left mouse button, in the opened menu select the 'Border' button and in the pulled-down menu select "Absolute"Fig. 124). The designation of the area available for scanningand the frame inside of which scanning will be done will displayed (Fig. 125). 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.

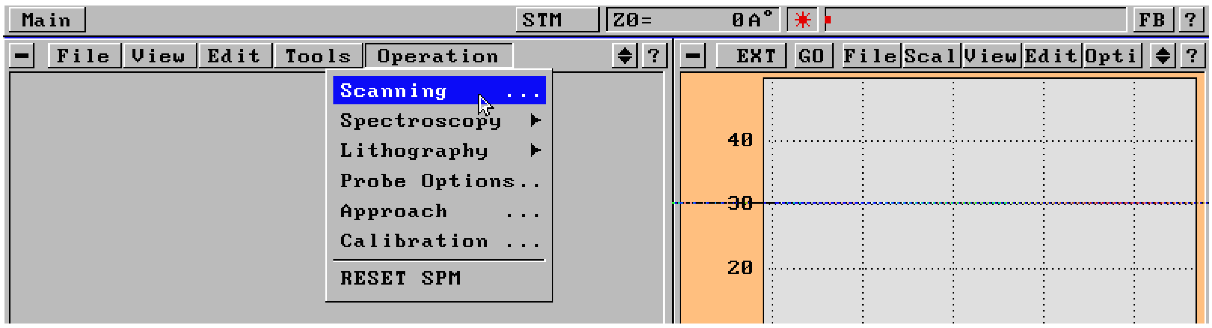


Fig. 123

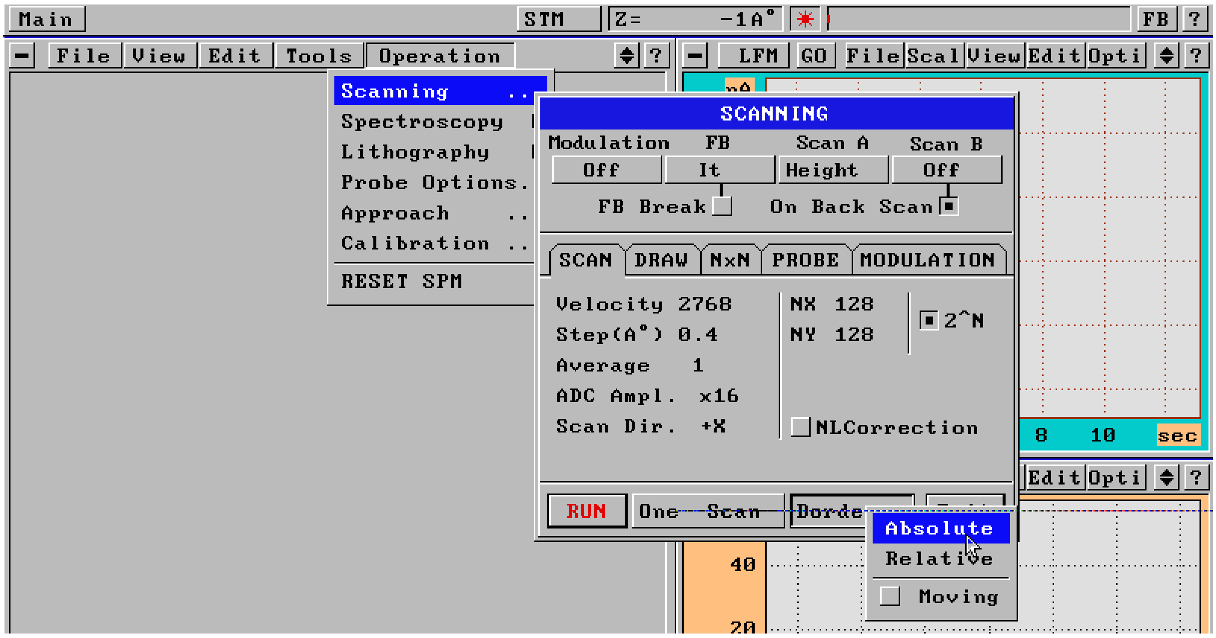


Fig. 124

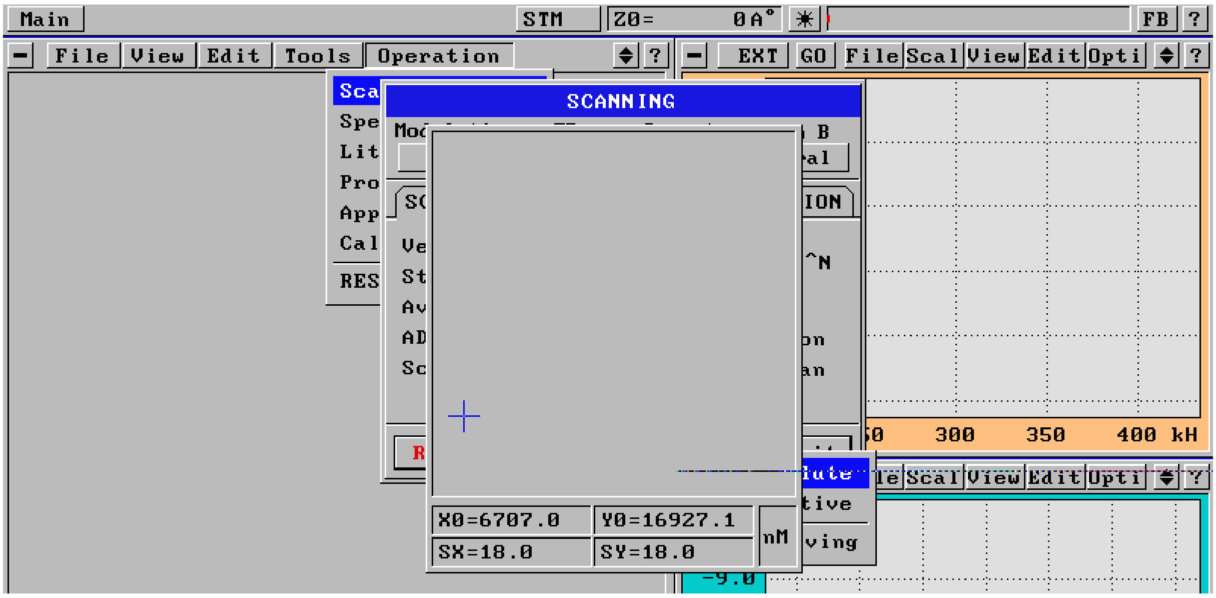


Fig. 125

After it the following must be inserted:
"Operation" - "Spectroscopy" - "Options" (Fig. 126)- Mode I(z), Z(A)=40 (Fig. 127, Fig. 128)

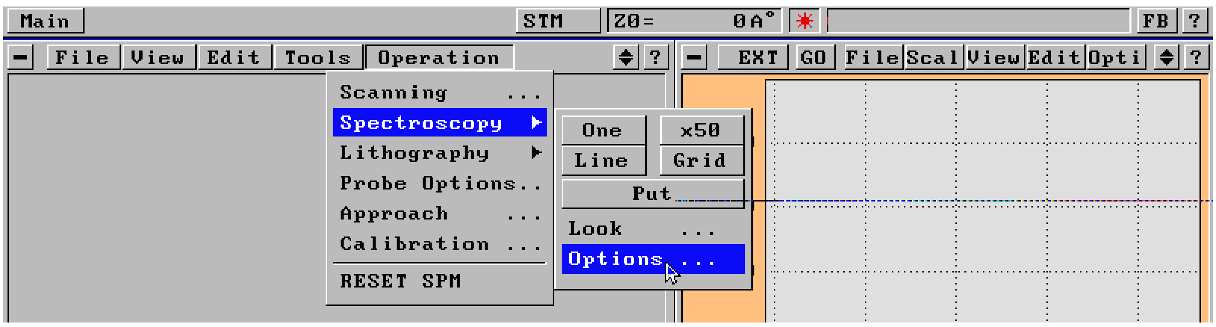


Fig. 126

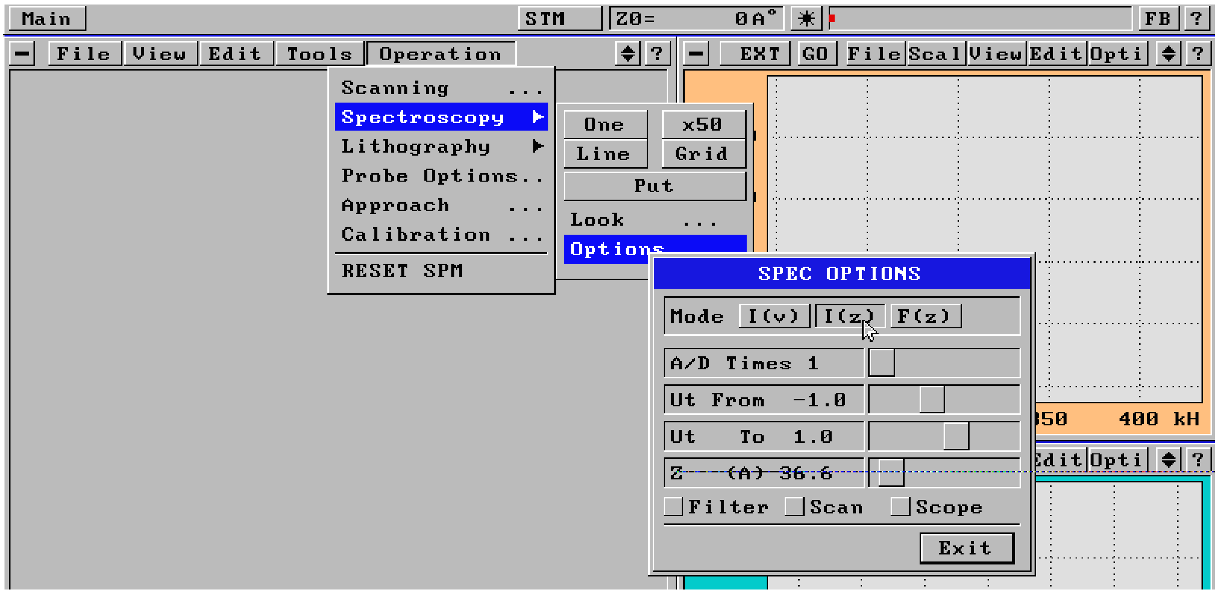


Fig. 127

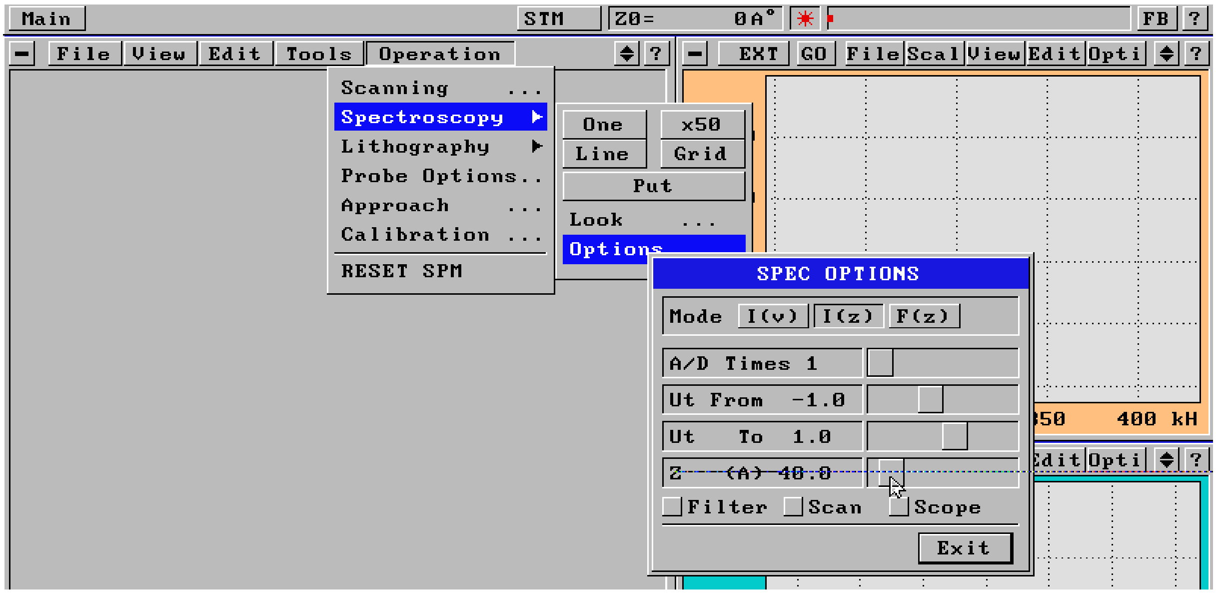


Fig. 128

and do single spectroscopy (Fig. 129).

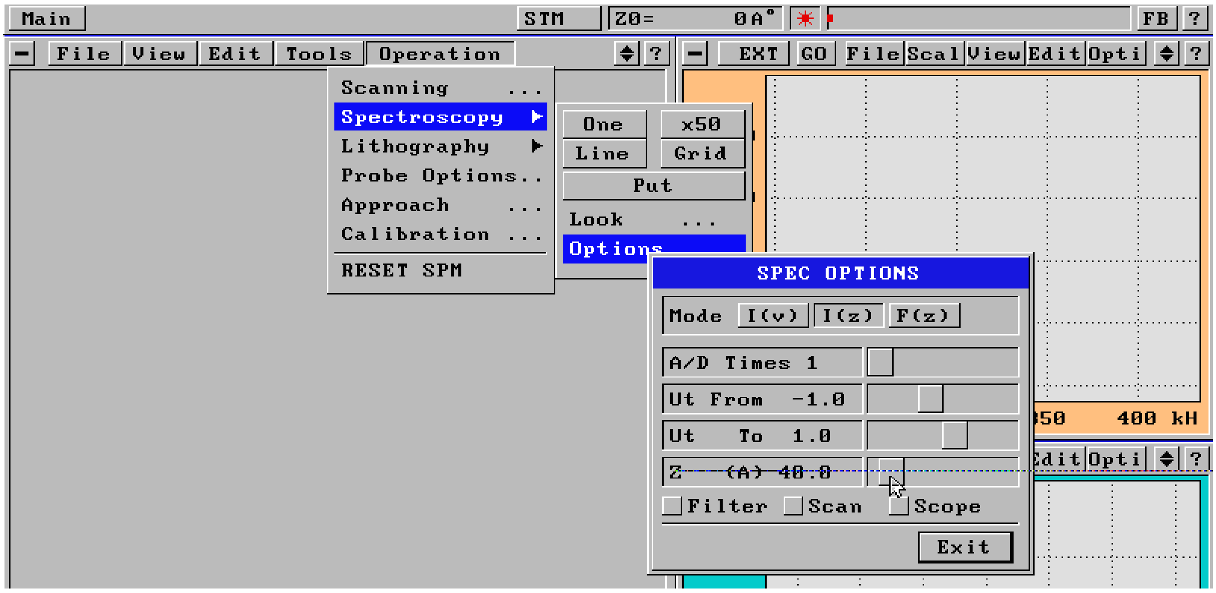


Fig. 129

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. 130, Fig. 131).
If the It drop to one-half takes place with Z < 3 A the tipis considered to be very good (Fig. 130), if with Z < 10 A (Fig. 131), then using this tip it is possible to have an atomic resolutionon HOPG. If this takes place with Z > 20 A this tip should not be used and must be replaced.

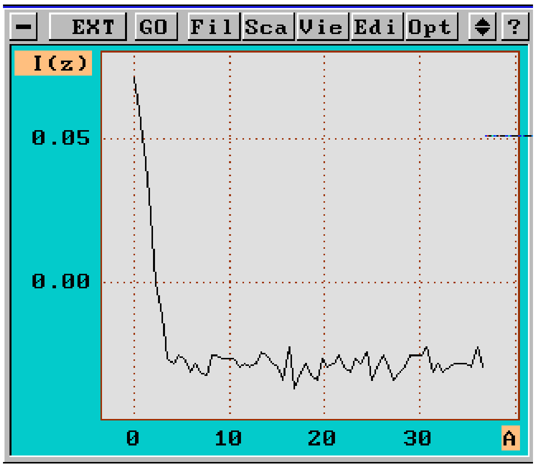


Fig. 130

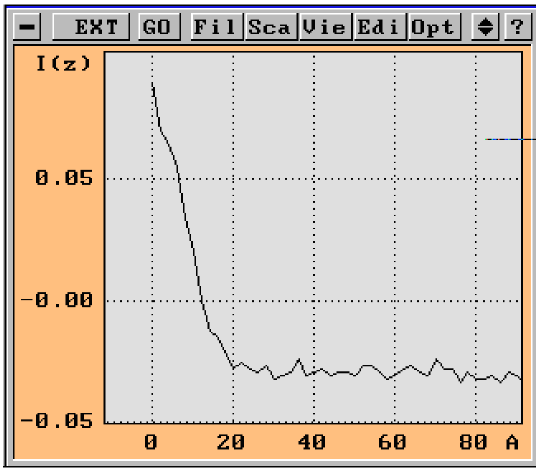


Fig. 131

3.2.7 Evaluation of the tip's stability over time

Evaluation of the tip's stability over time is done via tunneling currentcontrol 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.

3.2.8 Shutdown procedure

Open the microscope control menu - “Operation”, select the "Approach" item (Fig. 132) and press "Shift"+"Backward" (Fig. 133). Here the movement of the approach system will go on until interrupted by pressing the ESC key.

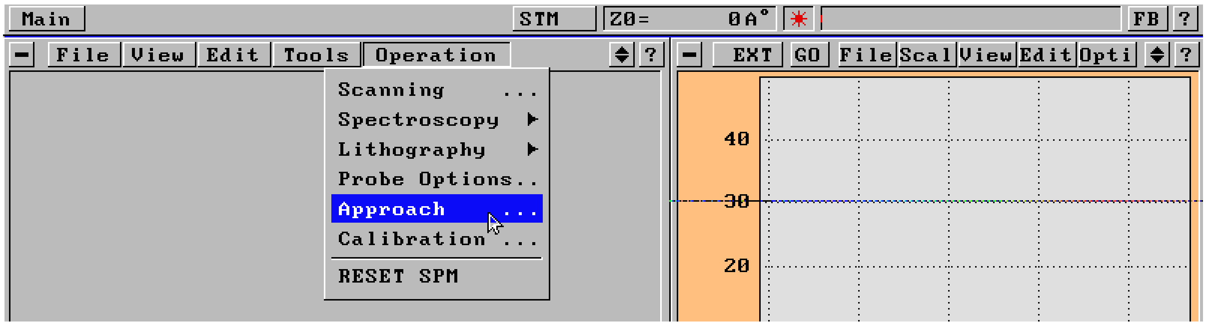


Fig. 132

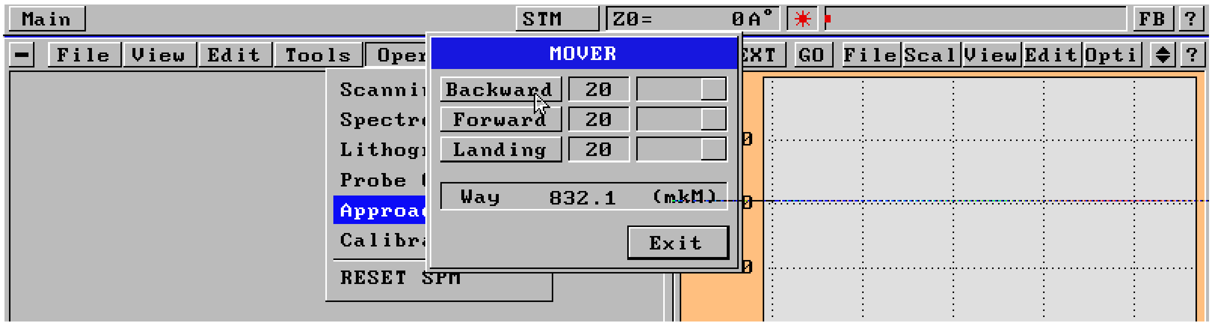


Fig. 133

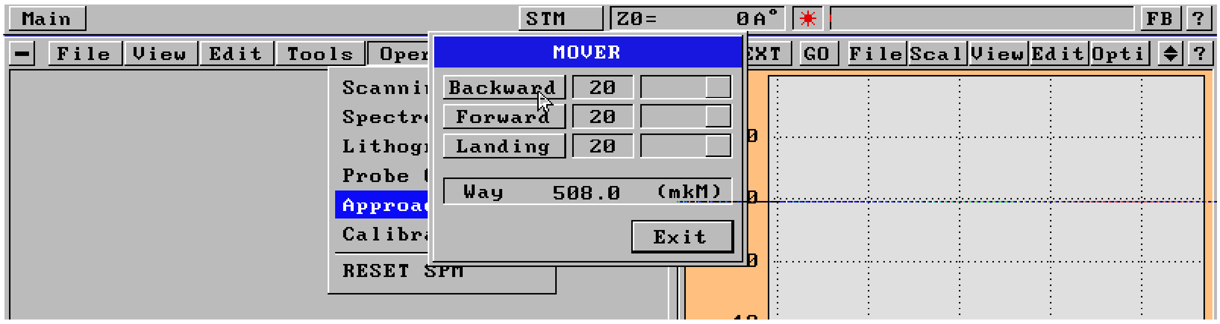
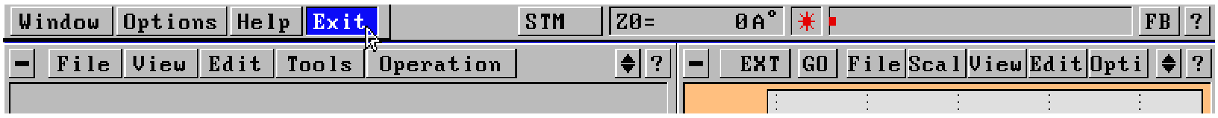


Fig. 134

WARNING:
Be especially careful when executing the "Shift" + "Forward" command. Do not do it unless required.

When the reading at the counter approaches 500 um back (Fig. 134), stop the approach system by pressing the ESC key on the keyboard.
Lift the scanner unit and place it on fixing pins. Move the sample down manually through turning the motor rotor lockwise (if viewed from bottom).
Then:

- Remove the SFM head.
- Carefully remove the sample from its fixation place at the piezoscanner
- Turn the microscope power off (button on the power supply unit).
- Exit the control program by pressing the Exit button in the Main button menu in the left corner of the screen (Fig. 135) or using the F10 functional key (after closing all the opened menus using the ESC key, otherwise the F10 is not activated).



Window
Options
Help
Exit
STM
Z0= 0A°
FB
?
File
View
Edit
Tools
Operation
?
EXT
GO
File
Scal
View
Edit
Opti
?

Fig. 135

4. Trouble-shooting

4.1 In STM mode

1	Symptoms	Possible cause	Remedy
1.	The indicator on the power supply unit is not lit.	The power supply unit is not connected to the power supply. A fuse has blown. The switch 230/110 V on the power supply unit* is not in the right position. The power supply unit is out of order.	Connect the power supply unit to the power supply. Replace the fuse. Check the position of the switch 230/110 V. Contact your dealer.
2.	The fuse blows when the power supply unit is turned on.	The switch 230/110 V on the power supply unit* is not in the right position. The power supply unit is out of order.	Check the position of the switch 230/110 V. Contact your dealer.
3.	When the power supply unit 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 power supply unit 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 power supply unit 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 power supply unit off. Check if the device is properly connected. Contact your dealer.
4.	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. The switch 230/110 V on the power supply unit* is not in the right position.	Check the voltage in the supply line. Check the position of the switch 230/110 V.
5.	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 AFM mode.
6.	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.	Use the adjusting knobs of suspension system to bring the scanner into horizontal position within ±3 degrees.

* - only for certain type of power supply units

		The substrate with the sample is not properly inserted into the holder.	Re-insert the substrate into the holder.
7.	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.
8.	When the I-V - 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 power supply unit is not lit.	The power supply unit is not connected to the power supply. A fuse has blown. The switch 230/110 V on the power supply unit* is not in the right position. The power supply unit is out of order.	Connect the power supply unit to the power supply. Replace the fuse. Check the position of the switch 230/110 V. Contact your dealer.
2.	The fuse blows when the power supply unit is turned on.	The switch 230/110 V on the power supply unit* is not in the right position. The power supply unit is out of order.	Check the position of the switch 230/110 V. Contact your dealer.
3.	When the power supply unit 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 power supply unit 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 power supply unit 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 power supply unit off. Check if the device is properly connected. Contact your dealer.
4.	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. The switch 230/110 V on the power supply unit* is not in the right position.	Check the voltage in the supply line. Check the position of the switch 230/110 V.
5.	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.
5à.	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.

6.	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.
7.	The signal changes smoothly up to the set-point 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.
8.	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.
9.	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.

5. Frequently Asked Questions (FAQ)

Question: *Resolution on all three axes*

Answer: Resolution on X-Y axes is 18-bit. Z-axis resolution can be changed from 15-bit up to 19-bit for any portion of Z range by program switching.

Question: *Software*

Answer: SPM imaging software and image analysis software are present. Data acquisition, image processing and analysis are in one program. The latest working version of the software can be downloaded from NT-MDT WWW web site.

Question: *AFM/STM base*

Answer: Automatic approach

Question: *XY stage*

Answer: About 10x10 mm. Manual positioning (by hands)

Question: *Please specify the differences between NT's "Resonant Mode" and your competitor's equivalent. Please be detailed in explanation.*

Answer: Here are the characteristics of our unit for ResonantMode:

- Working range of frequencies of cantilevervibration:

  For feedback maintenance 60 kHz - 1.3 MHz
- Range of frequencies at measurement of the characteristics of cantilevers 10 kHz - 2.0 MHz
- Band of a detected signal (Switched) 30 kHz; 1.5 kHz
- Frequency of longitudinal modulation of piezotube(dI/dZ in STM mode measurement) 22 kHz
- Frequency of bias voltage modulation (dI/dU in STM mode measurement) 22 kHz
- Amplitude of a voltage, applied to piezoelement of cantilevermodulation 0 - 10 V
- Amplitude of longitudinal vibration of piezotube0 ✍ 5 nm (0.3 % of z range)
- Amplitude of bias voltage modulation 0 - 10 V
- Range of the additional phase shift at detecting 0 - 360 degrees

Answer: In all modes the measured signals and signals, on which a feedbackis possible to be supported, are:

- Amplitude of the response signal
- Product of amplitude by the sine of phase shift
- Product of amplitude by the cosine of phase shift

Answer: Modulation of a cantilever can be made at one frequency, and registration of response at another frequency (at some harmonic).

If you compare the characteristics of our unit for ResonantMode with the characteristics of our competitor's equivalent, our device is better (see range of frequencies).

Question: *To achieve the atomic resolution, which scanner should be used, 7x7 um or 14x14 um?*

Answer: To achieve atomic resolution, the 7x7 um scanner should be used.

In addition, the Scan Size Transformer [P/N SS001] system will give you twice the X-Y resolution with two times less of X-Y scan size. The resolution along Z axis and vertical (Z) size of the scanner remains fixed.

For example, the 14x14 um scanner[min step along X-Y axis 0.4 A, vertical scanner 2 um] is connected to the SS001. The result will be: you obtain 7x7 um scanner with min step (resolution) along X-Y axis 0.2 A, vertical scanner 2 um.

The Scan Size Transformer (PN: SS001) can be used with any of our scanners.

Question: *Are those three scanners (3x3 um, 7x7 um and 14x14 um) interchangeable?*

Answer: Yes, they are.

Question: *If a 50x50 um optional scanner is needed, does 50x50 um scanner use the same base with 14x14 um scanner or it needs separate base? How to change the scanner base?*

Answer: The 50x50 um scanner as well as the other scanners will use their own separate base. Scanner unit that comprises scanner and approach system can be easily changed to another scanner unit according to the instructions in User's Guide. It takes a few minetes.

Question: *The scanner with sample was moved back from the tip and then reverted to the same position. What is the lateral precision of reverting to the same position?*

Answer: The tip will be reverted to initial position with the accuracy better than 1μm.

Question: *Is lithography the standard function of Solver P7-SPM-MDT?*

Answer: Yes it is.

Question: *We are interested in using an SPM for lithography on microchip structures. Related to this, I would like to know: can we metallize your AFM cantilevers such that they still work, or do you provide such levers already? Related to this, can we, in the AFM mode, apply tip-sample voltages in the range between 0 and 20 V (DC)?*

Answer: We produce highly doped silicon cantilevers, thus they are conductive.

So, it is not necessary to metallize them for this purpose.

You can apply tip-sample voltage in the range from -13 to +13 DC.

Question: *We actually have to sit next to the SPM while operating it. How good is the performance of the P4 SPM with someone sitting next to it? Can we achieve atomic resolution easily in that case?*

Answer: Due antivibration system you can obtain an atomic resolution sitting near the device. (As we do always).

We guarantee an atomic resolution in your laboratory as the test of microscope performance. (Of course, you should order the device with an atomic resolution possibility, i.e. with the ADS and the Scanner that permit atomic resolution).

Question: *Is it possible to investigate an assembled microchip, i.e. a piece of semiconductor, mounted and wire-bonded in a chip-carrier, in the AFM mode? The problem here might be that the sample sits about 500 microns up to 1 um BELOW the top of the chip carrier, so that it may be difficult to reach the sample surface with the cantilever.*

Answer: 500 microns will be OK, but it could be a problem with 1 um. To be sure we should see your detailed drawing. Anyway, we could change a little bit the design of cantilever holder to bring in correspondence with your demands.

Question: *What are typical features of your lithography software, like, for example, does the software correct crystal hysteresis?*

Answer: Software non-linearity and creep correction is done for X,Y scans (the residual nonlinearity is less than 1%). The correction for Z and for arbitrary tip motion is in progress now (Oct. 1996).

Question: *1) I would like to ask you about the zoom possibilities of your microscope. In some systems, the operator has only some predefined scan areas for which the nonlinearity of the peizotube is compensated. In other systems, the operator can zoom in continuously onto chosen point. What is the situation with Solver?*

1a) The "sliding cylinder bearing piezotube" is what is called in other terms an "inchworm motor"?

Answer: 1) The problem of piezo nonlinearity correction for any scan size is perfectly solved (residual nonlinearity is less than 1%) in SOLVER system. In our system zoom should be done continuously.

1a) Our piezotube scanner is fixed on the sliding cylinder that is moved by a step motor.

Question: *I would like to know if you use the same kind of tips for both resonant modes of Noncontact type and Intermittent contact type, too? Or you use end A for one and B for the other?*

Answer: The same cantilevers can be used for both noncontact and semicontact (ResonantMode) modes. The only requirement is to have sufficiently high resonant frequency (about 100 kHz or more).

Question: *What cantilevers do you use in Contact mode?*

Answer: As to contact mode, any commercial cantileverfor contact measurement can be used (with spring constant typically less then 1 nN). For example, one of the standard cantilevers with f=120 kHz and k=0.5 N/m is good enough for all modes, so you can study the same portion of the sample in all modes.

Question: *Is the image of the UltraSharp tip on your WWW page more or less a unique result, or is this how the average tip looks like?*

Answer: For UltraSharp tips' curvature radius of about 10 nm is standard and that of 5 nm is rare. But silicon tips are more fragile than Si_3N_4 tips, so it is better to use Si tips in semicontact mode rather than contact.

Question: *The gratings on your Web Page are really attractive. How do you check their dimensions in the nanometer range? I mean you have an etching process reproduced within a specified accuracy, or you MEASURE by some means each grating before delivery?*

Answer: The etching process insures sufficient accuracy. We do not measure each grating before delivery.

The SPM system configuration can be easly changed according to your d emands.

- Model P4-SPM-18, Scanning Probe Microscope (Combined SFM/LFM/STM)
  - SF002 - SFM/LFM head with resonant SFM system
  - AU001 - Adjustable insert for SFM
  - AU004 - Adjustable insert for SFM and ResonantMode SFM system
  - ST002 - STM head with precise preamplifier (3pA-1nA)
  - ST001 - STM head with preamplifier (10pA-10nA)
  - SC007 - Scanner unit with 7um x 7um x 1,5um scanner, min step 0,18A
  - SC014 - Scanner unit with 14um x 14um x 2um scanner, min step 0,4A
  - SC040 - Scanner unit with 40um x 40um x 4um scanner, min step 1,5A
  - SS001 - Scan size transformer (connector)
  - BL018 - SPM feedback and scanning control module (18 bit DAC)
  - IN001 - Interface board
  - SWD01 - Data acquisition and image processing software for workstation
  - BS002 - Power supply unit 220/110V
- Model P4-SPM-18UNI accessories include:
  - WS1212 - Pentium 100 (15 - inch color monitor SVGA (resolution 1024 x 768, 256 colors), 1 Gbyte Hard Disk, 16 Mbyte RAM)
  - BT001 - Unit for ResonantMode and STM spectroscopy
  - LC001 - Liquid sell for atomic force microscopy
  - CT001 - Cantilevers toolkit
  - WT001 - Working toolkit
  - TGS02 - Microscope grating set

The offered configuration gives you the following opportunities in your investigations.

1) STM, AFM, LFM modes with atomic resolution (with scanner SC007 the X,Y resolution 0.18A and with SS001 you can obtain on SC007 scanner the X,Y resolution 0.09A on the maximum X,Y scan 3,5x3,5 um.
2) Scanners SC014 and SC040 provide a large scan size (of course you can order only one of these scanners and thereby reduce the price).
3) SSOO1 Scan Size Transformer allows reduction of the scan size by one half thus increasing the resolution twice.
4) For STM investigations we offer two measuring heads. ST002 - STM head with precise preamplifier (3pA-1nA) - this head is very useful for instance for investigation of with biological objects, that can be easily damaged.

ST001 - STM head with preamplifier (10pA-10nA) - standard STM head for most usual investigations

5) BL018 - electronic board for control of all analog signals (to reduce the price we can suggest BL016, but this will reduce the maximum resolution of your device).

6) IN001 - interface board between the device and control PC, this board installs onto a standard PC mother board.

7) WS1212 - the device can be controlled with any PC computerfrom a 386DX to Pentium models. To reduce the price you can use your PC computer according to our instructions and recommendations.

8) BT001 - Unit for ResonantMode and Spectroscopy. ResonantMode - This is unit with four types feedback control (amplitude, phase, amplitude*sin(fi), amplitude*cos(fi)), which allows you to work in different modes.

   Spectroscopy - in STM mode you can scan dI/dZ (I=const) and dI/dU (I=const, U=const), so with these dependancies you can investigate some chemical properties of the samples.

9) LC001 - Liquid Cell allows to work in liquids.

10) CT001 - Convenient tool for handling cantilevers.

11) WT001 - the set of substrates and other things.

12) TGS02 - the set of four calibration gratings for the calibration and piezoceremic control.

The maximum sample size - 40x40x10 mm.

Overall SPM dimensions - 420x420x420 mm.

Question: *What kind of software do you use to operate the microscope and for image analysis? Is the image format compatible with other programs used by your competitors?*

Answer: Software:
We use the same program to control the microscope and for image analysis. Our program will operate with different data format. The demo version of our program can freely be download from our Internet Home Page **http://www.ntmdt.ru**. Remember that our program should be run under DOS.

Question: *How are the rough and the fine approach accomplished to the sample, both in STM and AFM operation? Do you have any kind of optical system showing the scanned area before and/or during scanning?*

Answer: Rough and fine approach.

The approach is the same for all modes, the sliding cylinder bearing piezotube with the sample on its top is moved toward the probe by step motor. Rough approach can be done manually (up to 1.0....0.5 mm sample-tip separation) and then automatically.

Sample-to-tip approach usually takes less then 1 min.

Repeatability of lateral sample-versus-tip position after successive approach is about 0.1um or less.

We do not include an optical system. This allows the buyer to select and purchase a unit more suited to his application and budget. FYI: We usually use an external long-working-distance optical microscope to observe scanning area of the sample before scanning.

Question: *How is your Resonance Mode? How does it compare with your competitors?*

Answer: ResonantMode

BT001 - Unit for ResonantMode and Spectroscopy.

Our ResonantMode unit enables to study surface in practically all existing modes:

- in noncontact mode (with tip-sample interaction due to Van-der-Waals, electric or magnetic forces);
- in semicontact mode (similar to Tapping or Intermittent Contact);
- to get maps of local elasticity distribution in SFM contact mode;
- to get maps of local barrier height (local work-function) in STM mode (dI/dZ);
- to get maps of local electron spectral density-of-states in STM mode (dI/dU).

Feedback loop can maintain one of the following signals constant:

amplitude, phase, amplitude*sin(phase), amplitude*cos(phase).

Frequency range for measurements is from 2kHz to 2MHz.

ResonantMode unit also enables to measure and maintain constant different harmonics of the main frequency (2xf....10xf).

Index