

NTEGRA platform

NTEGRA Spectra

Instruction Manual

Upright Configuration with Renishaw Spectrometer

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NTEGRA Spectra Probe NanoLaboratory

Upright Configuration with Renishaw Spectrometer

Instruction Manual

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Read me First!

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







Fig. 1

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

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

Feedback

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

User's documentation set

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

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

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

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

NTEGRA Spectra Probe NanoLaboratory (Upright Configuration). Instruction Manual

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1. Overview

NTEGRA Spectra PNL instrument is equipped with high performance optics that provides scanning of the sample area under the tip with the resolution of 0.4 μ m. Its optical measuring head serves for illuminating the surface with visible light (minimum spot size 0.4 μ m) and for collecting the scattered radiation including radiation from immediate vicinity of the tip. Additional capability is confocal Raman microscopy that uses the spectrometer of the instrument.

A large value of the numerical aperture of the objective lens (NA=0.7) allows illuminating the surface at wide angle with high intensity of the normal electric component in the light spot under the probe. This enables measuring tip-enhanced optical effects, including the surface-enhanced Raman scattering.

NTEGRA Spectra PNL provides the following options:

- performing spectral measurements at a certain point and acquiring spectral characteristics of various materials when the instrument operates as a regular spectrometer;
- measuring secondary signal intensity in the selected wavelength range in the mode of layer-specific volumetric scanning of the area $100 \times 100 \times 10 \ \mu m$;
- acquiring optical images of the object when the instrument operates as a regular laser confocal microscope;
- detecting the object landscape with atomic resolution as well as collecting its electrical, magnetic and nanomechanical properties with force microscopy techniques;
- performing nanoscale measurements of different optical properties relevant to techniques of nonaperturate near-field scanning optical microscopy.

The particular feature of the instrument is its capability to detect optical properties with a number of nonlinear effects, surface-enhanced Raman scattering, and the so-called Tip-Enhanced Raman spectroscopy.

2. Design

The main parts of the NTEGRA Spectra PNL are:

- Upright measuring unit:
 - Base unit;
 - Optical ACM-head;
 - Laser radiation delivery and recording system;
- Podule measuring unit.
- Spectral unit:
 - Spectrometer;
 - Lasers.
- Detectors:
 - Renishaw CCD camera;
 - Andor CCD camera (availability depends on the delivery set).
- Optical viewing system;
- Control system:
 - SPM controller;
 - Computer.

NTEGRA Spectra PNL consists of the following main parts:

- 1. Base unit (see sec. 2.1 on p. 8). It holds the measuring head, the scanner, and the sample holder;
- 2. Optical measuring head (see sec. 2.2 on p. 11). It provides acquisition of the surface image of the investigated object. Installed on the base unit;
- 3. Laser radiation I/O module (see sec. 2.3 on p. 19). It provides acquisition of the surface image of the investigated object. Installed on the base unit;
- 4. Podule unit (see sec. <u>2.4</u> on p. <u>20</u>). It serves for linking the spectral unit Renishaw with the base unit when the microscope Leica is used;
- 5. Spectral unit (see sec. 2.5 on p. 21).
- 6. Detectors (see sec. <u>2.6</u> on p. <u>21</u>). Serve for detecting signals coming from the investigated object. The spectrometer can be supplied with the following detectors:
 - CCD camera Renishaw;
 - CCD camera Andor.

- 7. Optical viewing system (see Appendix <u>1</u> on p. <u>73</u>). Provides monitoring the sample both at normal and at small angle to the sample surface for following purposes:
 - aiming the laser beam at the cantilever;
 - selecting the scan range on the sample surface;
 - visual inspection of the probe in processes of approaching and scanning.
- 8. Control system:
 - Main controller SPM;
 - Slave controller SPM;
 - Computer;
 - Interface boards.

2.1. Base Unit

The base unit is shown in Fig. 2-1.



Fig. 2-1. Main components of the base unit 1 – exchangeable mount; 2 – stepper motor; 3 – temperature and humidity sensor; 4 - LC-display

The base unit is contains a stepper motor with an approach mechanism that serves to place a sample under the probe.

Data on ambient temperature and humidity are measured with the sensor 3 and then displayed by the LC-display 4.

Base unit electrical slots

Slots for connecting the devices installed on the base unit:

| HEAD | - two identical connectors for connecting the measuring head; |
|-------------|--|
| SCANNER | - scanner connector, used for connecting either the exchangeable scanner or the measuring head scanner; |
| SCAN+SENSOR | - connector for the scanner or for the scanner with built-in sensors, used for connecting either the exchangeable scanner or the measuring head scanner; |
| T° | -connector of a heating element, used for connecting a heating stage, heating liquid cells etc.; |
| SM | - connector of the stepper motor; |
| AFAM | - connector of the ultrasonic piezoelectric transducer; |
| BV | - bias voltage jack, used for applying bias voltage to the sample. |

Slots for connecting the bias unit to the control system:

CONTROLLER 2 – connector for connecting to the **CONTROLLER 2** connector of the SPM controller;

CONTROLLER T^o – connector for connecting to the thermocontroller.

Exchangeable mount

The exchangeable mount is installed into the base unit of the device and is fixed with three screws. Design of the PNL allows for several types of the exchangeable mount. Here, a mount of SCC01NTF model is used.

Basic parts of the exchangeable mount are shown in Fig. 2-2.



Fig. 2-2. Exchangeable mount 1, 2 – measuring head seats; 3 – mirror mounting frame; 4 – positioning device; 5 – measuring head cables holder; 6 – approach mechanism cylinder; Fig. 2-97 – grounding jack; 8 – screws for fixing to the base unit.

There are two types of sockets on the exchangeable mount (see pos. 1, 2 in Fig. 2-2). Depending on its type, a measuring head can be installed either into the slots 1 or into the slots 2.

The frame 3 is used as a support of subsidiary mirrors of the optical viewing system (see Appendix 1 on p. 73).

The positioning device 4 (Fig. 2-3) holds a device that carries the sample (sample holder, scanner etc.).



Fig. 2-3. Scanner with movement sensors mounted on the positioning device

The positioning device is placed on the movable cylinder of the approach mechanism 6. This enables to move the sample up and down.

The microscrews 1 and the spring stops 2 (Fig. 2-4) provide adjusting in the XY plane.



Fig. 2-4. Positioning device 1 – microscrews; 2 – spring stops

2.2. Optical Measuring Head

The optical measuring head is a dedicated version of the scanning probe microscope. Its probe is located under a lens of large numerical aperture and of high spatial resolution.

The optical measuring head provides an option of side illumination of the sample with laser radiation. This option needs the side illumination module to mount on the measuring head.

Optical measuring head (Fig. 2-5) (hereinafter - measuring head) consists of the following components:

- Optical cantilever deflection detection system;
- Universal lens;
- Universal lens holder;
- Probe holder.

The probe holder is exchangeable. Type of the probe depends on the measurement mode. The following holders are used for different measurement modes:

- AFM probe holder;
- STM probe holder.

The optical cantilever deflection detection system 1 is mounted on the assembly of the lens holder 2 (Fig. 2-5). The lens hovers with a system of elastically deformed arms that provides high accuracy of beam focusing on the object as well as good stability of the assembly. The unit mount 3 contains microscrews for positioning the probe.



Fig. 2-5. Optical AFM measuring head 1 – optical cantilever deflection detection system; 2 – lens holder; 3 – mount with the probe holder and the positioning device

Main components of the AFM optical measuring head are shown in Fig. 2-6.



Fig. 2-6. Measuring head 1, 2 – screws for XY probe adjusting; 3, 4 – photodiode mirror adjusting screws; 5 –screw for fine focusing of the lens; 6, 7 –screws for XY lens adjusting; 8 – lens positioning device spring stop; 9 – entrance aperture; 10, 11, 12 – screw legs; 13 – lens; 14 –spring stop of the probe holder positioning device

The measuring head serves for measurements with confocal microscopy. Besides, contact and semi-contact modes of AFM as well as STM techniques are available.

The main functional component of the measuring head is the **Mitutoyo M Plan Apo 100** lens (pos. 13 in Fig. 2-6). The numerical aperture of the lens is 0.7 and the focal length is 2 mm while the working distance is 6 mm. The lens provides the following options along with conventional AFM techniques:

- Viewing the sample with resolution limited by 0.4 μm;
- Delivery and focusing the exciting laser radiation;
- Aiming the laser beam at the probe's tip;
- Detecting the probe deflection (needs an additional semiconductor laser diode to generate radiation of 650 nm or 830 nm wavelength).

The screw for fine focusing of the lens 5 is located in front of the lens. The microscrews 1 and 2 move the probe holder relative to the lens. Moving the probe holder provides directing the laser beam of the optical cantilever deflection detection system (hereinafter referred to as detection system) to the cantilever reflective surface. The photodiode mirror is adjusted with the screws 3 and 4 so that the beam reflected from the probe could come directly to the centre of the photodiode. For adjusting the measuring head in angle and height, three screw legs 10, 11, and 12 are used. The microscrews 6 and 7 provide positioning of the lens against the probe and external optical devices.



ATTENTION! Height adjustment of the legs 10, 11, and 12 is not recommended as this can misalign the optical system.

Cantilever deflection in AFM modes is detected with the detection system operating a semiconductor laser (with $\lambda = 830$ nm or 650 nm) that supplies radiation through the lens. The system dichroic mirror selectively reflects the radiation of the semiconductor laser. It provides a wide spectral range ($400 < \lambda < 800$ nm for the laser with $\lambda = 830$ nm) thus allowing for observing the sample through the optical microscope and for optical exciting the sample surface. High sensitivity of the detection system is due to a special design of the laser formation system.

Confocal microscopy uses a special probe (Fig. 2-7) with the tip being outside the edge of the cantilever.





Fig. 2-7. Confocal microscopy probe

Fig. 2-8. AFM probe

Table 2-1. Optical measuring head specification

| Sample size | Up to \emptyset 40×10 mm |
|-------------------------|--------------------------------------|
| Scanning type | Sample |
| Scanning range | 100×100×7 μm |
| Lens numerical aperture | 0.7 |
| Optical resolution | 0.4 μm |
| Lens focal distance | 2 mm |
| Visual field | ~70 µm |
| Laser wavelength | 830 nm 650 nm |
| Working spectral range | 400÷800 nm 400÷600 nm, 700÷900 nm |
| Dimensions (L×W×H) | 150×130×130 mm |
| Weight | 1.5 kg |

2.2.1. AFM Probe Holder

AFM probe holder (see Fig. 2-9) is placed on the measuring head. It can be replaced with another holder if necessary. It serves for measurements in AFM modes.



Fig. 2-9. AFM probe holder

Design of the AFM probe holder is shown in Fig. 2-10. The probe is placed on the sapphire bench-shaped support and then fixed with the clamping spring 2. The spring moves up and down with the handle 3.



Fig. 2-10 Design of the AFM probe holder 1 – sapphire support; 2 – clamping spring; 3 – handle

The AFM probe holder is placed in the mount of the measuring head and then secured with the spring stop 1 (see Fig. 2-11). The screws 2 and 3 serve to move the probe holder for adjusting the optical cantilever deflection detection system. The AFM probe holder is connected electrically to the slot 4 in the measuring head (see Fig. 2-11).



Fig. 2-11. AFM probe holder installed on the measuring head 1 – spring stop; 2, 3 – screws for X and Y movement of the probe; 4 – probe holder connector

2.2.2. STM Probe Holder

STM probe holder (see Fig. 2-12) is placed on the measuring head. It can be replaced with another holder if necessary. It serves for measurements in the STM mode.



Fig. 2-12. STM probe holder

Design of the STM probe holder is shown in Fig. 2-13. The probe 1 is inserted into slots of the holder 2 and then fixed with the spring 3. Protrusion of the sharpened end of the probe outside the holder should not exceed $3\div4$ mm.



Fig. 2-13 Design of the STM probe holder 1 - probe; 2 - holder; 3 - clamping spring

The STM probe holder is mounted on the measuring head quite similarly to the AFM probe holder (see Fig. 2-11). The STM probe holder is connected electrically to the **HEAD** socket in the base unit of the instrument. With the STM probe holder, the socket of the measuring head is left open.

2.2.3. Side Illumination Module

To operate with side illumination of the sample, the measuring head is equipped with the exchangeable module for side illumination (see Fig. 2-14). The side illumination module provides delivery of the exciting laser radiation at angle of 60° to normal to the sample surface as well as aiming the laser beam at the probe tip. Positioning of the laser beam on the sample is controlled with the micrometric screws 1 and 2, while focusing uses the screw 3.



Fig. 2-14. Side illumination module 1,2 – screws for X and Y movements of the laser spot; 3 – screw for fine focusing of the laser beam; 4 – positioning device; 5 – entrance aperture

The side illumination module is secured with four screws on the front side of the lens holder (Fig. 2-15).



Fig. 2-15. Side illumination module installed on the measuring head

Laser radiation comes into the side illumination module through the port 5. To focus the laser beam on the cantilever, the focusing module is used (Fig. 2-16). This module is screwed into a hole that is located inside the side illumination module. Focusing modules for different radiation wavelengths are available.



Fig. 2-16. Focusing module

2.3. Laser Radiation I/O module

The Laser radiation I/O module (Fig. 2-17) provides linking the spectral unit with the measuring head. The module is mounted to the back of the base unit and to the Optical viewing system holder (see Appendix $\underline{1}$ on p. $\underline{73}$).



Fig. 2-17. Laser radiation I/O module 1, 2 – adjusting screws of the steering mirror (M2); 3 – laser beam focusing screw (L1); 4 –scanner; 5, 6 – adjusting screws of the steering mirror M1; 7 – a turret with exchangeable mirrors (M3); 8 – entrance aperture; 9 – stopping screw; 10 – adjusting ring

Schematics of laser radiation input/output is shown in Fig. 2-18. Here, the delivered beam is shown in green while the radiation scattered by the sample (to be analyzed by the spectrometer) is colored with red.



Fig. 2-18. Schematics of laser radiation input/output M1 – mirror; L1, L2 – lenses; M2 – steering mirror; M3 – exchangeable mirror; 1, 2 – adjusting screws of the steering mirror; 3 – laser beam focusing screw; 4 –scanner

When the side illumination module is used for measurements, the laser radiation I/O module serves only for transmitting the scattered radiation to the spectrometer. In this case, the laser beam is delivered through the side illumination module.



ATTENTION! For adjusting the laser beam, use the screws 1, 2, and 3. Do not turn the screws 5 and 6 and the screws 9 and 10 located on the radiation feedthrough as this can misalign the optical system.

The exciting laser radiation comes through the entrance aperture 8 of the radiation feedthrough. For fine positioning of the radiation on the entrance aperture 8, the radiation feedthrough can move vertically (with the adjusting ring 10) for ± 10 mm and rotate around its axis. The stopping screw 9 fixes the feedthrough in the desired position.

Laser radiation I/O module a mirror M1 (Fig. 2-18) at its entrance. With the screws 5 and 6 (see Fig. 2-17), the mirror M1 is aligned so that the laser beam is directed along the lens L1 axis.

The screw 3 is used to move vertically the lens L1 that focuses the laser beam on a point located in the front focal plane of the lens L2.

The lens L2 patterns the spot (taken from the mirror M2) in the entrance pupil of the measuring head lens. Thereby, a laser beam comes from the steering mirror M2 and goes to the lens pupil under various angles, but it invariably falls into the center of the pupil.

The steering mirror M2 can be controlled either manually with the screws 1, 2 or instrumentally by the piezoelectric scanner 4. The mirror M3 is located on the turret and can be changed. The turret has four states that correspond to four types of mirrors:

- NO totally reflecting mirror. With this option, no portion of the laser beam transmits the mirror and so image on the display is unavailable.
- N1 partially transparent mirror (reflection to transmission ratio is 10/90). The display shows an image of the sample and of the laser spot.
- N2 mirror is absent and the laser radiation does not fall onto the sample. The display shows only an image of the sample.
- N3 custom mirror. In particular, this can be a mirror with a high reflection in the range of 600÷800 nm and a high transmission outside it for the laser with the wavelength of 633 nm.

2.4. Podule Measuring Unit

Podule measuring unit is optional for the delivery set. It serves for linking the spectral unit Renishaw with the base unit when the microscope Leica is used.

For details on the Podule measuring unit, please, refer to Renishaw user's manual.

2.5. Spectral unit

Renishaw spectral unit is designed for spectroscopy measurements and for imaging.

For details on the spectral unit, refer to Renishaw user's manual.

2.6. Detectors

Andor CCD

Andor CCD is used in a wide measurement range and has brilliant spectral and time characteristics. It contains a built-in thermoelectrical cooling system (Peltier element), which allows to low the temperature of the CCD-matrices down to -90 °C.

Renishaw CCD

Renishaw CCD camera is incorporated into the spectrometer. It is used in spectroscopy measurements. For details on the CCD camera, please, refer to Renishaw user's manual.

2.7. Optical Fiber Transport System

Optical fiber transport system (hereinafter – OTS) and radiation feedthrough (see Fig. 2-19) are designed for easy, safe and secure transfer the laser radiation to the NTEGRA Spectra PNL entrance port or immediately to the sample when the side illumination module is used.



Fig. 2-19. Input device and OTS

Polarization plane of the radiation is defined by the line mark on the **Output** socket of OTS (see Fig. 2-20). Rotate the socket to the desired position when installing OTS.



Fig. 2-20. **Output** socket. Line mark indicates the polarization plane

Table 2-2. OTS technical characteristics

| Parameter | Value |
|---------------------------|-----------------------|
| Fiber type | single-mode |
| Pigtail length | 2÷3 m |
| Beam diameter at entrance | 0.65 mm |
| Transmission efficiency | > 65 % |
| Output beam divergence | diffraction limit |
| Maximum input power | $\leq 100 \text{ mW}$ |

3. Basic Safety Measures

General Safety Measures

- Ground the instrument before operation!
- Do not disassemble any part of the device. Disassembling of the product is permitted only to persons certified by NT-MDT.
- Do not connect additional devices to the instrument without prior advice from an authorized person from NT-MDT.
- This instrument contains precision electro-mechanical parts. Therefore protect it from mechanical shocks.
- Protect the instrument against the influence of extreme temperature, the direct impact of sun radiation, and moisture.
- For transport, provide proper packaging for the instrument so as to avoid its damage.

Electronics

- To reduce possible influence of power line disturbances on the measurements, we recommend supplying the instrument units with a surge filter.
- Before operation, set the power switch of the SPM controller to the position corresponding to value of the local electrical power line (this is only done with the controller being off!).
- Switch the SPM controller off before connecting/disconnecting its cable connectors. Disconnecting or connecting the cable connectors during operations may cause damage to the electronic circuit and disable the instrument. A warning label is attached to the SPM controller of the instrument (Fig. 3-1).



Fig. 3-1

Laser

• Observe safety measures for operation with devices containing sources of laser radiation. Do not stare into the beam. A label warning about the presence of laser radiation is attached to the measuring head. Labels are different for different wavelength and output power of laser (Fig.).





Fig. 3-2



Scanner

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

Optical components

Optical components include the following units and elements:

- optical elements in the spectrometer unit;
- laser.

To avoid contamination of the optical elements with dust or moisture, keep the room clean and never disperse sprays or other substances near the instrument. Do not touch the optical elements. For cleaning the optical components, use special cleaning sets.



ATTENTION! Never uncover the spectrometer without extreme necessity. Only an authorized person is permitted to clean elements of the spectrometer. In the case of contamination, request for help to the manufacturer.

Radiation detectors

When storing or handling the CCD-camera, avoid ingress of contamination into the camera. Before connecting the water cooling system, turn off the computer and disconnect the camera from the computer. Prevent ingress of moisture into the camera case. Keep the vent holes on the case open when using the camera. Prevent too intensive illumination of the camera. Make sure that the camera is at the room temperature when finishing the work.

4. Operating Conditions

For normal operation of the instrument, the following operating conditions are recommended:

- Ambient temperature: $+18\div23^{\circ}^{\circ}C$;
- Temperature stability: better 1°C per hour;
- Relative humidity less 60 %;
- Atmospheric pressure: 760 ±30 mmHg;
- Supply voltage 220 V (+10%/-15%), 50/60 Hz with grounding;
- The working room must be protected from mechanical vibrations, airflows and acoustic noise, both internal and external;
- Use a massive vibration-isolating optical bench for mounting the PNL;
- RMS vibration measured in the 1/3 octave frequency band must not exceed the values given in the graph;



- The device must be protected from the direct sunbeams;
- The base unit must be located on a separate table, away from the computer and from monitors, to reduce electromechanical noise.

Heat flow and draughts badly influence the instrument.

Do not operate the PNL if temperature or humidity is outside the recommended limits.

5. Storage and Transport Regulation

- Packaging of the equipment should be performed indoors in a ventilated room at ambient temperature of 15° C 40 °C and relative humidity at most 80 % with no corrosive agent in the atmosphere. Storage conditions should comply with the standard GOST 15150.
- The equipment should be carried by one or several modes of enclosed transport according to transport regulations applicable to the mode(s) in service.
- Only transportation of the equipment in proper package is permitted to prevent transportation damage.
- The equipment in its package should be placed and secured in a vehicle in a way that provides steady position and excludes shocks of the deliverable parts by one another or by walls of the vehicle.
- Transportation conditions should comply with requirements of the standards GOST P 52931-2008 and GOST 15150.
- Transportation and storage in regions of the Far North and equated localities should comply with requirements of the standards GOST P 52931-2008 and GOST 15846.
- Location of the equipment in a warehouse must provide easy access to it and free space for personnel.
- Devices and their parts should be stored on shelves.
- At least 100 mm distance between the stored parts and walls and ceiling of the premises should be reserved.
- Distance between the stored parts and heating facilities of the premises should be at least 0.5 m.

6. General Requirements on Installation

NTEGRA Spectra PNL is a precise optical device. Special requirements on the work room, on the power supply system, and on the optical table should be met.

NTEGRA Spectra PNL should be installed in a dark room of area of 12 m^2 (3×4 m²). The room must be equipped both with ceiling lighting and with a separate source of weak scattered light.

A system of thermal stabilization or, at least, of good ventilation should be provided. Remember that an operating laser can disperse up to 4 kW of heating power.

Keep dust concentration as low as possible.

The electric wiring of the room must meet the following requirements:

- at least 8 standard voltage sockets for mains 220 V/50 Hz with the total power of 1 kW. These are used to supply the microscope, the spectrometer, the controllers, computers and monitors;
- Other supply voltage lines are defined by the user.

The CCD-camera Andor needs water cooling for operation. Thus, the corresponding infrastructure must be provided in the room.

The PNL is mounted on an optical table of dimensions $2x1 \text{ m}^2$ or bigger with a standard net of threaded connections M5 (grid pitch 50 mm).

7. Setup and Installation

The NTEGRA Spectra PNL is put into operation by an authorized person after installation.

Connecting the Electromechanical Units



ATTENTION! Before connecting or disconnecting any parts of the instrument, power off the controller. Any connections during the instrument operation may result in damage of the electronic circuitry.

The NTEGRA Spectra PNL instrument operates under control of two controllers, main and slave. The main controller model depends on the size of the exchangeable mount – BL222NNTF (BL222RNTF) for the 50- μ m or BL227NNTF (BL227RNTF) for 100- μ m exchangeable mount. The slave controller model is BL222SS.

Commutation schematic of NTEGRA Spectra PNL is shown in Fig. 7-1. Below, the procedure on connection of electromechanical units is explained.



Fig. 7-1. Commutation schematics of the NTEGRA Spectra electromechanical units

To connect electromechanical units, perform the following steps:

1. Release the spring clips (Fig. 7-2) and install the scanner into the positioning device as it is shown in the Fig. 7-3.



Fig. 7-2. Spring clips



Fig. 7-3. Scanner installed in the positioning device

- 2. Connect the scanner slot to the SCAN+SENSOR located on the base unit.
- 3. Connect the measuring head to the HEAD slot at the base unit.
- 4. Connect the base unit to the controller by connecting the CONTROLLER1 socket of the base unit to the HEAD socket of the main controller with the first cable and the CONTROLLER2 socket of the base unit to the corresponding socket of the main controller with the second cable.
- 5. Connect the radiation delivery and recording system to the **CONTROLLER2** socket of the additional board of the controller.
- 6. Connect the CCD-cameras, and the controller to the computer.
- 7. Before operation, set the power switch of the SPM controller to the position corresponding to value of the local electrical power line (**this is only done with the controller being off!**).
- 8. Connect the controller to the power point.



ATTENTION! Controller can be connected to an electrical power supply line of $110/220 \text{ V} (60 \div 50 \text{ Hz})$ after setting the voltage selection switch to the position corresponding to this power supply line. Not following this instruction may cause damage to the electronic components.

8. Preparing for AFM Measurements

Preparation for AFM measurements includes the following operations:

- 1. Preparing and Installing the Sample (sec. 8.2 on p. 31).
- 2. Installing the Side Illumination Module (sec. 8.3 on p. 37)
- 3. Installing the AFM Probe holder (sec. 8.4 on p. 38).
- 4. Installing the AFM Probe (sec. <u>8.5</u> on p. <u>39</u>).
- 5. Installing the Measuring Head (sec. 8.6 on p. 41).
- 6. Installing the Optical Fiber Transport System (sec. 8.7 on p. 42).
- 7. Launching the Control Program (sec. 8.1 on p. 30).
- 8. Adjusting the Optical Cantilever Deflection Detection System (sec. 8.8 on p. 44).

These operations will be explained in details below.

8.1. Launching the Control Program



ATTENTION! Tighten up all connectors before turning the controller on. Disconnection of connectors during operation may cause damage to the electronic components.



ATTENTION! The controller and the spectrometers must be on before starting the control program.

- 1. Switch the spectrometer on with the button on the power supply.
- 2. Switch the controller on with the toggle switch on the front panel.
- 3. Switch on the computer.
- 4. Start the Nova Px program. The Main program window will appear on the computer monitor.

| 脑 Nova | a Px 3.1.0 Rev | / 2623 | | | | | | | | | | |
|--------------|--------------------|------------|-------------|---------------|----------------|--------------------|--------------|----------------------|-------------|---------------|---------|------|
| <u>V</u> iew | Tools <u>H</u> elp | Semicontac | t 🔹 🙀 🛛 Mag | ● 0,00 | SetPoint 5,000 | C 🕈 🕄 CH 🕈 C | àain 1,00 🗧 | | | Pass I 👻 🐴 XY | BV 0,00 | \$ ⊡ |
| [| È ⊷ Data | Aiming | Resonance | W Approach | Scanning | <u>↓</u> Curves | 🛃 Litho | O scilloscope | 亚 Scheme | Camera | | |
| | SPM Ok | SPM Init | | | | | | | | | | |
| | | | | | | | | | _ | | _ | |

Fig. 8-1. Main window of the program Nova Px

5. Operation of the Control program starts with initialization of the instrument. If initialization is successful, the **F** Initializing indicator in the Initialization panel will change its appearance to **SPM Ok** – the instrument is now ready for operation.

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

6. Click the Spectra button in the Main Operations panel. As a result the Spectra window is opened and the spectrometer control program is started.

8.2. Preparing and Installing the Sample

The instrument allows for measuring samples of $40\div50$ mm in diameter and up to 10 mm in thickness. The samples are fixed on a substrate that is installed onto the scanner sample stage.

Two types of measuring scanners are available. So, the procedure on preparing and installing the sample depends on the scanner type and should be done in one of two following ways:

- Preparing and installing the sample on the scanner with sensors;
- Preparing and installing the sample on the scanner without sensors.

These operations will be explained in details below.

Preparing and installing the sample on the scanner with sensors:

The scanner substrate with capacitive sensors is a ferromagnetic disc (see Fig. 8-2), which is installed onto a magnetic holder located in the scanner. The sample is fixed to the substrate using a double-sided adhesive tape or glue.



Fig. 8-2. Metal substrate



Fig. 8-3. Sample stage

A sample can also be mounted on a sapphire substrate of dimensions $24 \times 19 \times 0.5$ mm. This option needs the special sample stage (see Fig. 8-3) to be used.

To prepare and install the sample, perform the following steps:

- 1. Prepare a clean metal or sapphire substrate. Cut off a strip of a double-sided adhesive tape, slightly wider in size than the sample.
- 2. Stick the adhesive tape to the substrate, smooth its surface out with the back of the tweezers to remove air bubbles between the substrate and the adhesive tape.
- 3. Put the sample on the adhesive tape and carefully press it with tweezers in several places (not touching the area intended for the investigation).

NOTE. After the sample is fixed on the substrate, a noticeable vertical drift of the sample can occur within one hour (due to the slow relaxation of sticky tape). This drift should be taken into account. If the task requires the minimum drift, prepare the sample beforehand (at least, an hour before the investigation).

4. Place the sapphire substrate with the sample on the sample stage. Insert the substrate from the side of two support balls under the spring clips so that the clips secure the substrate while the substrate bottom is supported by the three support balls (see Fig. 8-4). If a metal substrate is used, skip this step and go to sec. 5.



Fig. 8-4. Sapphire substrate on the sample stage



ATTENTION! When working with a thick sample, you need to account for its height in the program. The default value of the sample is set to 0.5 mm (For more details how to set up the sample height see p. 3).

5. Place the metal substrate on the magnetic fastener of the scanner or on the sample stage with a sapphire substrate (see Fig. 8-5). The sample stage should come to the center of a magnetic holder and lay on the three support balls.



Fig. 8-5. Loading the sample into the scanner with sensors

Installing the sample on the scanner without sensors

Samples are fixed to special polycrystalline sapphire substrates, which are available in the hardware set of the instrument. Dimensions of these substrates are 24x19x0.5 mm. The sample fixes to the substrate with double-sided adhesive tape or glue.

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

The substrate SU001 is recommended for use with small size samples (less than $10\div12$ mm diameter). The substrate SU002 is recommended for use with bigger size samples (greater than $10\div15$ mm diameter).

NOTE. For studying a transparent sample, the SU002 substrate is recommended because the SU001 substrate may emit some spectral lines to the acquired spectrum thus worsening experimental results.



Fig. 8-6. Substrate SU001

Fig. 8-7. Substrate SU002

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

To prepare and install the sample, perform the following steps:

- 1. Prepare a clean sapphire substrate. Cut off a strip of a double-sided adhesive tape, slightly wider than the sample.
- 2. Stick the adhesive tape to the substrate, smooth its surface out with the back of the tweezers to remove air bubbles between the substrate and the adhesive tape.
- 3. Put the sample on the adhesive tape and carefully press it with tweezers in several places (not touching the area intended for the investigation)

NOTE. After the sample is fixed on the substrate, a noticeable vertical drift of the sample can occur within one hour (due to the slow relaxation of sticky tape). This drift should be taken into account. If the task requires the minimum drift, prepare the sample beforehand (at least, an hour before the investigation).

4. Place the sapphire substrate with the sample on the sample stage. Insert the substrate from the side of two support balls under the spring clips so that the clips secure the substrate while the substrate bottom is supported by the three support balls (see Fig. 8-8).



ATTENTION! When working with a thick sample, you need to account for its height in the program. The default value of the sample is set to 0.5 mm (For more details how to set up the sample height see p. $\underline{3}$).



Fig. 8-8. Installing the sample on the scanner without sensors

For operation in modes requiring electrical connection between the sample and the instrument, substrates equipped with a spring contact are recommended. This spring contact is used for constant electrical bias of the sample (Fig. 8-9).



Fig. 8-9. Substrate with the spring contact

When you use special substrates an electrical connection between the sample and the device is provided by a spring contact, which is connected with a plug end to the corresponding slot located on the base unit. There are two jacks on the base (Fig. 8-10), the jack 1 to supply the desired bias voltage (BV), and the grounding jack 2.



ATTENTION! Do not use the BV slot for grounding as the Bias Voltage is applied both to the probe holder and to the BV.



Fig. 8-10 1 – bias voltage jack; 2 – grounding jack

Setting the sample height

The sample thickness influences the geometrical dimensions of the acquired image. With the same elongation of the scanner tube, a thicker sample will move to a larger distance from the probe in the XY plane (Fig. 8-11) thus increasing size of the image.

To account for this effect, the height of the sample above the scanner should be taken correctly.



Fig. 8-11. Increase of the image size with height of the sample

When defining the sample thickness, be careful not to miss thickness of the sample used for XY calibration of the scanner.
For setting the sample height in the program, perform the following steps:

1. Open the **ScanSettings** window by clicking the *Settings...* button in the Scanning window (Fig. 8-12).

| ScanForm | | |
|--|-----------------|--------|
| Image: Constant of the second seco | Settings | Quick |
| Rate 1,0102 		 Hz | Area Select | Params |
| 10: 🔐 🕎 20: 🛛 📾 🚑 🕂 🌧 🕀 🗨 🖓 👫 📕 + 🎷 ۷ | 🖌 🗆 Mode Params | |
| | MoveType Lin | e |
| | PassLine® 1 | |
| | - | - |

Fig. 8-12. Settings button

2. In the SampleHeight input field (Fig. 8-13), enter the desired composite sample height.

| 🐚 ScanSett | ings | | | | |
|--------------|--------------|-----------|------------|-------------|---------|
| Scan Driver | ScrBase | • | Name: Scan | ner 092007- | 09-019 |
| Scan Mode | Contact Topo | graphy · | • | Type: 2 | D |
| Measuremen | nts | | | | |
| Dim S | ignals | Direction | Pass | Draw | Average |
| 2D, -+ | leight | Forward | ▋₹ĹĹŢ | Show | 1 🗘 |
| | | Forward | | St | • |
| Execution | - | - | | - | |
| Vertex Dela | y 0,00 🗘 | ms | Meas. Type | Dynamic | • |
| Scan Coun | t 3 🗘 |] | Operation | Normal | - |
| 20, | Cyclic Sav | /e | | •7 | |
| 20. | Cyclic Red | draw | | NL 🕅 | Cor ON |
| FastOverPoi | ints, % 0 | \$ | PassLines | 1 | |
| Sample Heigh | nt, mm 5,00 | € | | | Ok |

Fig. 8-13. Setting the sample height

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

When mounting another sample of a different thickness, change the **SampleHeight** parameter.

8.3. Installing the Side Illumination Module

If upside illumination of the sample is used, skip this section and go to instructions of sec. 8.4 «Installing the AFM Probe holder» on p. 38.

To apply side illumination of the sample, install the side illumination module on the measuring head.

To install the side illumination module, perform the following steps:

- 1. Place the measuring head on a plane surface.
- 2. Unscrew four screws for fixing the side illumination module on the measuring head. These screws are at the front side of the lens holder (see Fig. 8-14).



Fig. 8-14. Measuring head. Arrows indicate screws for fixing the side illumination module

3. Secure the side illumination module with the four screws using a 3 mm Allen key (see Fig. 8-15).



Fig. 8-15. Side illumination module installed on the measuring head

4. Turn the measuring head upside down. Screw the focusing module into the hole inside the side illumination module (see Fig. 8-16).



Fig. 8-16. Focusing module installed on the measuring head

This completes installation of the side illumination module on the measuring head.

8.4. Installing the AFM Probe holder

To install the AFM probe holder, perform the following steps:

- 1. Place the measuring head with its bottom up on a plane surface.
- 2. Pull the clamp (Fig. 8-17).
- 3. Place the AFM probe holder so that support balls of the holder stand on sapphire seats on the base of the measuring head (Fig. 8-17). Screws of the measuring head must come to the corresponding slots with their caps.

Release the clamp.



Fig. 8-17. AFM probe holder installed. Arrow indicates handle of the clamp (here the measuring head is shown without the side illumination module)

5. Connect the AFM probe holder electrically to the measuring head (see Fig. 8-17). This completes installation of the AFM probe holder on the measuring head.

8.5. Installing the AFM Probe

To install or to replace the AFM probe, perform the following steps:

- 1. Take the measuring head, turn it over, and place it on a plane surface.
- 2. Take the probe from the container with the tweezers (Fig. 8-18). Make sure that the working side of the chip with cantilevers is directed to you during the installation. Do not turn the chip over because probes in the container are packed with their tips pointing upwards.



Fig. 8-18. Container with probes

3. Press the handle to raise the pressing spring (Fig. 8-19).



Fig. 8-19. Probe holder

4. Place the probe on the forepart of the holder and move it under the spring (Fig. 8-20).



Fig. 8-20. Installing the probe

5. Correct position of the probe with the tweezers so that it seats firmly on the forepart. Release the spring.

NOTE. The probe chips of some models may be of trapezium shape. When installing such a probe, make sure that it is not inclined to the forepart of the holder.



Fig. 8-21. Probe installed into the holder

Now, the probe is installed into the holder.

8.6. Installing the Measuring Head

To install the measuring head, perform the following steps:

1. Rotate the manual approach knob clockwise until the scanner comes to its lowest position (pos. 1 on Fig. 8-22). This will secure the probe and the sample during installation of the measuring head.



Fig. 8-22. 1 – manual approach knob; 2 – measuring head seats

2. Place the screw legs of the measuring head down to the seats 2 of the exchangeable mount so that the front legs (these are fixed with check-nuts) come into the seat with a hole and into the seat with a groove (Fig. 8-23).



Fig. 8-23. Measuring head installed into the base unit (here the measuring head is equipped with the side illumination module)



ATTENTION! Length of the measuring head legs is agjusted during the installation procedure. Do not re-adjust it as this will result in inclination of the lens and thus in misalignment of the optical detection system.

- 3. Tuck in the head cable into the cable holder. Connect the measuring head to the **HEAD** socket in the base unit of the instrument.
- 4. See from one side and rotate the manual approach knob anticlockwise to approach the sample to the probe to distance of ~ 2 mm.

This completes installation of the measuring head.

8.7. Installing the Optical Fiber Transport System

If the instrument is not equipped with the side illumination module, skip this section and go to sec. 8.1 «Launching the Control Program».

For operation with the side illumination module, another OTS is required to link the laser with the optical measuring head. This addition is necessary to deliver the laser radiation to the sample with the side illumination module installed in the instrument.

This section explains the procedure on installing the second OTS.



ATTENTION! Never look through the output aperture of the laser! Direct and scattered radiation is hazardous to eyesight.

To install and to align the optical fiber transport system, perform the following steps:

- 1. Mount the radiation feedthrough on the laser source.
- 2. Take the **Input** socket of the second OTS with your right hand while, with your left hand, press and hold the central backing-up screw of the radiation feedthrough on the laser source. Insert the **Input** socket into the slot against the stop. Make sure that the polarization line marker of the socket comes into the cavity in the radiation feedthrough (see Fig. 8-24).



Fig. 8-24. Radiation feedthrough

3. Insert the **Output** socket of the second OTS into the hole of the side illumination module against the stop (see Fig. 8-25).



Fig. 8-25. Output socket installed

4. Secure the **Output** socket by screwing two fixing screws with 1.5 mm Allen key (see Fig. 8-26). The key is supplied with the delivery set of the instrument.



Fig. 8-26. Securing the Output socket

This completes installation of the second optical fiber transportation system.

8.8. Adjusting the Optical Cantilever Deflection Detection System

Adjusting the optical cantilever deflection detection system can be performed in one of two ways:

- by the spotlight from the illuminator;
- by laser beam coming from the spectrometer.

In both cases, the adjustment procedure is the same. So, only adjustment with the illuminator is described below.

- 1. Set the Optical viewing system lens (see Appendix $\underline{1}$ on p. $\underline{73}$) above the hole for radiation delivery to the measuring head.
- 2. Switch on the illuminator, CCD-camera, and the viewing system monitor if they are off.
- 3. Watch the probe and move it with the screws 1 and 2 (see Fig. 8-27) to the position anywhere the light spot appears on the probe chip (pos. 1 in Fig. 8-28).



D)

Fig. 8-27. Adjusting elements of the detection system

2 - screws for X and Y positioning of the probe;3 - screw for fine focusing of the lens;
5 - screws for positioning of the photodiode; 6, 7 - screws for X and Y movement of the lens (here the measuring head is shown with the side illumination module)



Fig. 8-28. Aiming the light spot at the cantilever

- 4. Move the light spot until it comes in front of the probe chip by displacing the probe with the screw 1 forward to the moment the spot appears on the sample (pos. 2 in Fig. 8-28).
- 5. Move the probe with the screw 2 along the light spot until the spot comes to the cantilever (pos. 3 in Fig. 8-28). This results in displaying a distinct bright spot on the optical viewing system monitor.
- 6. Focus the objective on the end of the cantilever by turning the screw 3 (see Fig. 8-29). Monitor the focus on the image of the optical viewing system.



Fig. 8-29. Objective focused on the end of the cantilever

7. Turn the screw for fine focusing of the lens counterclockwise until the specific image of the tip apex (bright point) appears. This shifts the objective focus from the cantilever to the tip apex (see Fig. 8-30).



Fig. 8-30. Objective focused on the tip apex bright point displays the tip apex

NOTE. Focusing the objective on the tip apex is available only in configurations with special probes for confocal scanning.

8. Further adjustments are performed in the **Aiming** window of the Control program (available through the Aiming button).

The **Aiming** window (see Fig. 8-31) contains a graphical panel to view the laser spot on the photodiode and a table of the current photodiode signal values.

The signals refer to:

- **DFL** the differential signal between the top and the bottom photodiode halves.
- **LF** the differential signal between the left and the right photodiode halves.
- **Laser** the total signal coming from all four sections of the photodiode. It is proportional to the laser radiation intensity, reflected from the cantilever.



Fig. 8-31. Aiming window

9. Adjust the **Laser** signal (see Fig. 8-32) in the **Aiming** window to its maximum by moving the probe with the screws 1 and 2. The **Laser** signal should be in the range 5÷30 nA.



Fig. 8-32. Laser signal achieved its maximum

10. Adjust the laser spot at the center of the photodiode (see Fig. 8-33) by moving the photodiode with the screws 4 and 5. For higher precision, find a position where the signals **DFL** and **LF** are close to zero, with the total signal **Laser** being sufficiently high.



Fig. 8-33. Laser spot adjusted at the center of the photodiode

This completes preparation of the instrument for measurements with the AFM probe.

9. Preparing for STM Measurements

Preparation for STM measurements includes the following operations:

- 1. Preparing and Installing the Sample (sec. <u>9.1</u> on p. <u>49</u>).
- 2. Installing the Side Illumination Module (sec. <u>9.2</u> on p. <u>50</u>).
- 3. Installing the STM Tip Holder (sec. 9.3 on p. 51).
- 4. Manufacturing the STM Tip (sec. 9.4 on p. 52).
- 5. Installing the STM Tip (sec. 9.5 on p. 53).
- 6. Installing the Measuring Head (sec. <u>9.6</u> on p. <u>54</u>).
- 7. Installing the Optical Fiber Transport System (sec. <u>9.7</u> on p. <u>54</u>).
- 8. Launching the Control Program (sec. <u>9.8</u> on p. <u>54</u>).

These operations will be explained in details below.

9.1. Preparing and Installing the Sample

To fix samples during the STM investigations, special substrates with an electric contact to apply bias voltage to the sample are available (see Fig. 9-1).



Fig. 9-1. Substrate with a spring contact

To prepare and install the sample, perform the following steps:

- 1. Prepare a clean substrate. Cut off a strip of a double-sided adhesive tape, slightly wider than the sample.
- 2. Stick the adhesive tape to the substrate, smooth its surface out with the back of the tweezers to remove air bubbles between the substrate and the adhesive tape.
- 3. Put the sample on the adhesive tape and carefully press it with tweezers in several places (Fig. 9-2). Do not touch the area intended for the investigation.

NOTE. After the sample is fixed on the substrate, a noticeable vertical drift of the sample can occur within one hour (due to the slow relaxation of sticky tape). This drift should be taken into account. If the task requires the minimum drift, prepare the sample beforehand (at least, an hour before the investigation).



Fig. 9-2. Prepared substrate with a sample installed

4. Turn the contact spring with the tweezers to apply the tunneling voltage to the sample and fix it somewhere close to the boundary of the sample. The central part of the sample should be freely accessible (Fig. 9-2).



ATTENTION! When working with a thick sample, you need to account for its height in the program. The default value of the sample is set to 0.5 mm (For more details how to set up the sample height see p. 35).

- 5. Install the substrate with the sample on the sample stage, sliding it in sideways under the fixing clips. The spring contact should be seen during insertion (Fig. 9-3). Make sure that the spring contact does not touch any metal parts of the tip holder.
- 6. Insert the voltage terminal into **BV** connector on the base unit (see Fig. 9-3).



Fig. 9-3. Installing a sample

9.2. Installing the Side Illumination Module

Confocal microscopy with the STM probe can be performed either with or without use of the side illumination module.

If the side illumination module will not be used, skip this section and go to sec. <u>9.3</u> «Installing the STM Tip Holder» on p. <u>51</u>.

Otherwise, install the module on the measuring head. For detail on installation, refer to sec. <u>8.3</u> «Installing the Side Illumination Module» on p. <u>37</u>.

9.3. Installing the STM Tip Holder

To install the STM tip holder, perform the following steps:

- 1. Place the measuring head with its bottom up on a plane surface.
- 2. Pull the clamp (see Fig. 9-4).



Fig. 9-4. Measuring head (here the measuring head is shown without the side illumination module) Arrow indicates handle of the clamp

- 3. Place the STM tip holder so that support balls of the holder stand on sapphire seats on the base of the measuring head (Fig. 9-5). Screws of the measuring head must come to the corresponding slots with their caps.
- 4. Release the clamp (see Fig. 9-5).



Fig. 9-5. STM tip holder installed

5. Connect the STM probe holder electrically to the **HEAD** socket in the base unit of the instrument. Leave the socket of the measuring head open.

This completes installation of the STM tip holder on the measuring head.

9.4. Manufacturing the STM Tip

The tip is the sharpened end of a platinum-iridium (PtIr), or platinum-rhodium (PtRo) (with platinum content of about 80 %), or tungsten (W) wire, $8\div10$ mm long with a diameter of $0.25\div0.5$ mm.

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

There are two techniques of manufacturing an STM tip:

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

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

Sharp-edged scissors and tweezers with kinks on the interior surface (available in the toolkit supplied with the microscope) are used to cut the wire.



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



Fig. 9-6

To prepare the apex, perform the following steps:

- 1. Clamp the wire with the tweezers so that it projects beyond its edge for 2÷3 mm (Fig. 9-6).
- 2. Cut the wire at an angle of 10÷15 degrees as close to its apex as possible and simultaneously pull the scissors along the wire axis to separate the part being cut off. This is done to avoid the contact between the cutting edges of the scissors and the tip apex. This procedure implies rather a tearing off the wire in the last moment than truncating it. This provides a sharp apex, formed at the wire's end (Fig. 9-7).



Fig. 9-7. Typical shape of a wire cutoff (apex of the tip)

3. Check the cutoff shape using the optical microscope (magnification 200x is recommended). Repeat the cutting process, if necessary.



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

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

After cutting the wire, annealing the apex in the flame of an alcohol lamp for $1\div 2$ seconds is recommended to remove organic substances. Check the apex of the tip using the optical microscope. The cutoff section should be bright, with no traces of black or dust.

9.5. Installing the STM Tip

To install or to replace the STM probe, perform the following steps:

- 1. Turn the measuring head over and place it on a plane surface.
- 2. Take the wide tweezers in one hand. By the other hand, take the tip with the narrow tweezers.



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

- 3. Squeeze the clamping spring using the wide tweezers
- 4. Insert the probe into slots of the holder with the blunt end down. Protrusion of the sharpened end of the probe outside the holder should not exceed 3÷4 mm (see Fig. 9-8).



Fig. 9-8. Tip installed into the holder

5. Release the spring. The tip should be fixed firmly in the holder.

NOTE. The quality of the tip sharpness and firmness of the tip fixation in the clamp are the primary factors that determine the quality of results obtained with STM.

9.6. Installing the Measuring Head

For details on installation of the measuring head on the base unit refer to sec. <u>8.6</u> «Installing the Measuring Head» on p. <u>41</u>.

The socket of the measuring head is free for STM measurements because the STM probe holder is connected to the **HEAD** socket of the base unit.

9.7. Installing the Optical Fiber Transport System

If upside illumination of the sample is used, skip this section and go to instructions of sec. 9.8 «Launching the Control Program».

For operation with the side illumination module, an optical fiber transport system (hereafter OTS) is required to link the laser with the optical detection system. It serves to deliver the laser radiation to the sample.

For details on installation of the OTS, refer to sec. <u>8.7</u> «Installing the Optical Fiber Transport System» on p. <u>42</u>.

9.8. Launching the Control Program

For details on launching the Control program, refer to sec. <u>8.1</u> «Launching the Control Program» on p. <u>30</u>.

10. Performing Measurements

AFM measurements

For details on performing measurements in contact and semi-contact AFM modes, refer to the manual *PNL NTEGRA*. *Performing Measurements*, Appendix.

STM measurements

For details on performing STM measurements, refer to the manual *PNL NTEGRA*. *Performing Measurements*, Chapter 4.

Measuring with the Confocal Microscopy Mode

Confocal microscopy can be performed both with AFM and STM probes. Preparatory procedures for confocal microscopy depend on the type of the probe used.

Before measuring with the AFM probe prepare for operation according to instructions of sec. <u>8</u> «Preparing for AFM Measurements» on p. <u>30</u>.

Before measuring with the STM tip prepare for operation according to instructions of sec. 9 «Preparing for STM Measurements» on p. 49.

For performing measurements with confocal microscopy, the user should be aware of principles of contact and semi-contact AFM modes as well as of STM modes.

The confocal scanning includes the following main operations:

- 1. Configuring the instrument (page 56).
- 2. Approaching the Sample and Focusing the Laser Beam:
 - case of the AFM probe (sec. <u>10.2.1</u> on p. <u>56</u>);
 - case of the STM tip (sec. <u>10.2.2</u> on p. <u>59</u>);
 - case of operation without probe sensor (sec. 10.2.3 on p. 61).
- 3. Adjusting Channels of Exciting and Detection (sec. 10.3 on p. 62).
- 4. Adjusting Scan Parameters (sec. <u>10.4</u> on p. <u>67</u>).
- 5. Scanning (sec. <u>10.5</u> on p. <u>71</u>).
- 6. Finishing the Work (sec. 10.6 on p. 72).

These operations will be explained in details below.

10.1. Configuring the Instrument

To configure the instrument for a Confocal Microscopy, perform the following steps:

- 1. Open the **Spectra** window by clicking the Spectra button.
- 2. Switch to the **Renishaw CCD** tab by selecting the corresponding title at the left top of the **Spectra** window.
- 3. Select the **Override podule position** option (Fig. 10-1).

| 🗹 Override podule positi | on Shutter State |
|--------------------------|------------------------|
| Laser power | C Closed |
| 100 | Indeterminated |
| Full Spectrum Recording | 🕺 Measurement Settings |

Fig. 10-1. Selecting the Override podule position option

10.2. Approaching the Sample and Focusing the Laser Beam

Confocal microscopy requires that the sample is located in the lens focal plane. Depending on the measuring technique, the probe is located in one of the following ways:

- close to the sample surface;
- far from the sample surface or even absent in the measurement layout.

The latter means that we have three preparatory procedures:

- Approaching and Focusing for Modes with the AFM Probe (sec. <u>10.2.1</u> on p. <u>56</u>);
- Approaching and Focusing for Modes with the STM tip (sec. <u>10.2.2</u> on p. <u>58</u>);
- Approaching and Focusing for Modes without the Probe (sec. <u>10.2.3</u> on p. <u>61</u>).

10.2.1. Approaching and Focusing for Modes with the AFM Probe

Approaching the sample to the probe

This procedure serves to approach the sample to the probe. It depends on the selected mode (contact, semi-contact). For detailed description, refer to the manual *Performing Measurements*, Part 3. Here, only procedural sequences are presented.

Contact AFM:

- Selecting the controller configuration;
- Setting initial level of the DFL signal;
- Approaching the sample to the probe;
- Adjusting the working level of the feedback amplification ratio.

Semi-contact AFM:

- Selecting the controller configuration;
- Adjusting the piezodriver working frequency;
- Setting initial level of the Mag signal;
- Approaching the sample to the probe;
- Adjusting the working level of the feedback amplification ratio.

Fine focusing

With proper adjustment of the optical detection system, the sample surface should be at the lens focus. If the focus appears shifted from the surface, fine focusing is required.

To obtain fine focusing, perform the following steps:

1. Fix position of the Z-scanner by clicking the arrow to the right from the feedback enabling button at the upper bar of the Main Program window. In the drop-down list that will appear, select **Save** (Fig. 10-2).



Fig. 10-2. Selecting the Save item

NOTE. Be aware that leaving the probe in the **Save** state for long time is risky because the probe can be damaged due to the scanner drift.



a)

b)

Fig. 10-3. Measuring head installed (here the measuring head is shown with the side illumination module) 1, 2 – screws for X and Y positioning of the probe;3 – screw for fine focusing of the lens; 4, 5 – screws for positioning of the photodiode; 6, 7 – screws for X and Y movement of the lens 8 – manual approach knob; 9 – screw for fine focusing of the laser; 10,11 – screws for X and Y movement of the laser beam

2. Focus the lens on the sample surface by rotating the screw 3 (Fig. 10-3).

Adjusting the Photodiode Position

To adjust the photodiode position with the fine focus, perform the following steps:

- 1. Open the feedback circuit. (the button is The Off).
- 2. Open the **Laser Aiming** window by clicking the Aiming button.
- 3. Rotate the photodiode positioning screws 4, 5 (Fig. 10-3) to move the laser spot in the position indicator to the center of the photodiode (Fig. 10-4).



Fig. 10-4. Laser spot adjusted at the center of the photodiode

4. Close the feedback circuit (

10.2.2. Approaching and Focusing for Modes with the STM tip

Approaching the sample to the probe

Before approaching the sample to the probe, make sure that the probe is in the viewing field of the optical viewing system. If necessary, move the probe under the lens of the optical viewing system (Fig. 10-5) with the screws 5 and 6 (Fig. 10-3). Watch movement of the probe either immediately or on the screen of the optical viewing system.



Fig. 10-5. Probe in the viewing field of the optical viewing system

Now, approach the sample to the probe. It depends on the selected mode (contact, semicontact). For detailed description, refer to the manual *Performing Measurements*, Part 3. Here, only the procedural sequence is presented.

- 1. Selecting the controller configuration;
- 2. Approaching the sample to the probe;
- 3. Adjusting the working level of the feedback amplification ratio.

As the approach completes, the sample surface appears out of the lens focus (see Fig. 10-6).



Fig. 10-6. Sample surface is out of the lens focus



Focus the lens on the sample surface by rotating the screw 3 (see Fig. 10-3).

Fig. 10-7. Sample surface is in the objective focus

Focusing the lens on the tip apex

- 1. Switch off the illuminator that is located on the positioning holder of the optical viewing system. For details on the optical viewing system, refer to the manual *PNL NTEGRA. Performing Measurements*, Appendix.
- 2. Open the feedback loop (The Off).
- 3. Watch the probe from a side and on the screen of the optical viewing system and aim the laser beam at the tip apex (Fig. 10-8). Move the laser beam by rotating the screws 10 and 11 (see Fig. 10-3).



Fig. 10-8. Laser beam aimed at the tip apex

4. Rotate the screw 9 (see Fig. 10-3) to focus the laser beam on the tip apex (Fig. 10-9).



Fig. 10-9. Laser beam focused on the tip apex

5. Switch on the illuminator. This will produce the screen image that shows the sample surface with the laser spot focused on the tip apex (see Fig. 10-10).



Fig. 10-10. Both sample surface and laser beam in the objective focus

10.2.3. Approaching and Focusing for Modes without the Probe

For operation without the probe, it is either removed from the instrument or retracted far away from the sample. In both cases, approaching should provide focusing the sample in the lens focal plane.

If the probe remains installed, the lens is preliminarily moved away from the probe so that the probe locates at a secure distance from the sample surface, that is, $1\div 2$ mm above the lens focal plane.

If the probe is removed, go to step 4 of the procedure below.

To approach and to focus without the probe, perform the following steps:

- 1. Rotate the screw 3 for fine focusing of the lens (Fig. 10-3) to focus the lens on the cantilever.
- 2. Rotate the screw 3 for fine focusing counterclockwise until the typical image of the cantilever tip (bright point) appears. The cantilever will be out of the focus, but the lens will be focused on the probe tip (see Fig. 2-7). With the STM probe, the probe will be out of the focus while the objective will be focused on the tip apex.
- 3. Do 3-4 revolutions of the screw 3 to move the lens down. This will place the probe to the safe distance $(1 \div 2 \text{ mm above the lens focal plane})$.

NOTE. To remove the cantilever from the lens viewing field, use the screws 1, 2 (Fig. 10-3).

- 4. Use the spectrometer to attenuate the laser beam to its minimum (see the manual on the spectrometer).
- 5. Approach the sample to the lens slowly and very carefully by rotating the manual approach knob and observing the sample on the display of the viewing system. Control the lens focus by contrast of the sample image or by focusing the laser beam on the sample (if the spectrometer laser is on).



ATTENTION! Be careful when approaching the sample. If the sampleprobe distance is less than the one, the probe can contact the sample surface thus being damaged.

10.3. Adjusting Channels of Exciting and Detection

For recording the signal from the sample surface, the following detectors can be employed:

- built-in Renishaw CCD camera;
- Andor CCD camera.

A specific procedure on adjusting channels of exciting and detection depends on the module used to detect the signal. It is performed with one of the following procedures:

1. Adjusting signal detection with the Andor CCD camera (sec. <u>10.3.1</u> on p. <u>63</u>);

2. Adjusting signal detection with the Renishaw CCD camera (sec. 10.3.2 on p. 65);

These operations will be explained in details below.

10.3.1. Adjusting signal detection with the Andor CCD camera

- 1. Open the **Spectra** window by clicking spectra button.
- 2. Go to the Andor CCD tab by clicking the corresponding title in the left top of the **Spectra** window.
- 3. If your instrument is equipped with several detectors to detect the signal, select the CCD camera Andor in the **Select for scan** list at the right top of the **Spectra** window.

If your instrument is equipped with one detector, that detector will be selected automatically and the **Select for scan** list will be unavailable.



Fig. 10-11. Selecting the detector to detect the signal

4. Define cooling temperature of the CCD-matrix (in the range of $-40 \div -50$ °C) in the **Set T(°C)** input field available in the Toolbar (Fig. 10-12).

| Image Track V. bin Cen | ter: 72 🗘 Width: 31 🗘 🗖 Crop Mode Cou | nt: 1 🔶 Acquisition | Mode Single Scan 🔻 |
|-------------------------------|---|---------------------|--------------------|
| Acquisition time = 0,012260 s | Exposure Time 0,012260 s Min = 0,000010 s | Set T (°C) -42 💲 | Cooler= 12,1 |
| Live Mode Single Scan Pixels | | | |
| | | | |

Fig. 10-12. Setting the cooling temperature

- 5. Start cooling the CCD-matrix by pressing the **Cooler off** button placed to the right of the input field. The cooling process will begin and the text caption of the pressed-in button will indicate the current temperature of the matrix.
- 6. Adjust parameters of detecting the spectrum by the CCD camera through the following steps:
 - a. Enter the exposure time in the range of $0.2 \div 1$ s in the **Exposure time** input field.
 - b. Define the horizontal speed of spectrum recording by selecting the appropriate item in the **H** list of the **Shift Speed** field (Fig. 10-13):
 - 1 corresponds to the maximum scanning speed;
 - 32 corresponds to the minimum scanning speed that provides better signal/noise ratio.

| Save As Background | Subtract Background |
|-------------------------|-----------------------|
| Full Spectrum Recording | CONTROL |
| Shift Speed V: 16 | · H: 1 (A-D №0) ▼ |
| Laser: 633,000 Save | Central Pix: 511 Save |

Fig. 10-13

- c. To acquire a spectrum from the whole CCD-matrix, click the V. bin button.
- d. Collect the spectrum (Fig. 10-14) by clicking the Single Scan button.



Fig. 10-14. Spectrum collected from the whole CCD-matrix

NOTE. To activate the option of scan-time updating the spectrum image, click the Live Mode button.

NOTE. A spectrum to be saved should be appended to the current data file as a separate frame using the 1 Toolbar button.

To improve the signal/noise ratio at low levels of the signal, the spectrum is collected only from a limited region of the CCD-matrix that collects the major portion of the radiation:

- 1. To receive the image from the CCD-matrix, press the Image button.
- 2. Acquire the scan by clicking the Single Scan button. The collected image displays where the signal is concentrated (Fig. 10-15).



Fig. 10-15. Image collected with the CCD-matrix

NOTE. To activate the option of scan-time updating of the image, click the Live Mode button.

- 3. Using the collected image, select a band of the CCD-matrix that receives the spectral signal from the sample. This band will bound an area of the CCD-matrix in measuring the desired signal. To define the band, perform the following steps:
 - a. Click the \bowtie button in the Toolbar.
 - b. Place the mouse pointer in the line of the image where the signal is maximum and click the primary mouse button. This results in highlighting the selected line (Fig. 10-16) and in displaying its number in the **Center** input field of the Toolbar.
 - c. In the **Width** input field on the Toolbar, define width of the desired band so that it covers the major portion of the image (Fig. 10-17).



Fig. 10-16. Central line defined





- 4. To acquire a spectrum with the defined band of the CCD-matrix, press the **Track** button.
- 5. Start collecting the spectrum by clicking the Single Scan button. The spectrum (Fig. 10-18) collected over the limited band of the CCD-matrix is of better signal/noise ratio than the spectrum collected over the whole CCD-matrix.



Fig. 10-18. Spectrum collected over a limited band of the CCD-matrix

10.3.2. Adjusting signal detection with the Renishaw CCD camera

- 1. Open the **Spectra** window by clicking the Spectra button.
- 2. Go to the **Renishaw CCD** tab by clicking the corresponding title in the left top of the **Spectra** window.
- 3. Select the Use Renishaw CCD for scanning option in the Instrument Parameters Adjustment panel.

4. If your instrument is equipped with several detectors to detect the signal, select the CCD camera Andor in the **Select for scan** list (Fig. 10-17), at the right top of the **Spectra** window.

If your instrument is equipped with one detector, that detector will be selected automatically and the **Select for scan** list will be unavailable.

| Select for scan : | Renishaw CCD 📃 💌 |
|-------------------|------------------|
| | Renishaw CCD |
| | Andor could |

Fig. 10-19. Selecting the detector to detect the signal

- 5. Select the **Use trigger mode** option.
- 6. Adjust parameters of spectrum detection by the CCD camera through the following steps:
 - a. Open the **Spectral acquisition setup** window (Fig. 10-20) with the **Measurement Settings** button.

| ipectral ac | quisition setup | ? 🛛 |
|---|---|--|
| <u>R</u> ange Acg | guisition File Timing Temperature FocusTrack Advanced | |
| <u>E</u> xposure ti <u>A</u> ccumulati | ime /s 1.00 ♀ Dbjective 50 ions 1 ♀ Laser power / % 100 | Cosmic ray removal |
| - Li <u>v</u> e imag | ging | Restore instrument state on completion |
| (live ima | age map name) New | Close laser shutter on completion Minimize laser exposure on sample |
| <u>T</u> itle | Single scan measurement | |
| A single sc | can measurement generated by the WiRE2 spectral acquisition wizar | d. |
| | ОКО | тмена При <u>м</u> енить Справка |

Fig. 10-20. Acquisition tab of the Spectral acquisition setup window

- b. Go to the Acquisition tab by clicking the corresponding title.
- c. Enter the exposure time in the range of $0.3 \div 1$ s in the **Exposure time** input field.
- d. Define number of measurements at every scan point in the **Accumulations** input field.
- e. Click the **OK** button to save modifications and to close the **Spectral acquisition setup** window.

- 7. Open the laser shutter by selecting the **Opened** option in the **Shutter state** panel (Fig. 10-21).
- 8. Select the desired laser power in the Laser power drop-down list (Fig. 10-21).



Fig. 10-21

9. Collect the spectrum by clicking the Single Scan button (Fig. 10-22).



Fig. 10-22. Spectrum collected from the whole CCD-matrix

NOTE. To activate the option of scan-time updating of the spectrum image, click the Live Mode button.

10.4. Adjusting Scan Parameters

Scanning can be performed by the sample (with the scanner of the exchangeable mount) or by the laser (with the steering mirror of the Laser Radiation delivery and recording system). The scanning-by-sample mode provides an additional option to detect optical signals. The scanning-by-laser mode is capable to collect 3D images. For the latter purpose, the probe tip should be homed or retracted to a safe distance.

The procedure on adjusting the scan parameters consists of the two following stages:

- Adjusting the optical signals detection parameters performed in the Spectra window.
- Adjusting the scan parameters (selecting the mode and the scan area) performed in the **Scan** window.

Adjusting the optical signals detection parameters

Any of available CCD cameras (Andor and/or Renishaw) can serve as the detector of the scan signal. The scanning procedures with the Andor and Renishaw are much the same but they use different tabs for selecting the desired spectral regions and for assigning functions to those regions. Use of the Andor camera needs the **Andor CCD** tab while the Renishaw CCD camera operates through the **Renishaw CCD** tab. Select a proper tab according to your task.

- 1. Go to the Andor CCD (Renishaw CCD) tab by clicking the corresponding title at the left top of the Spectra window.
- 2. Click the spectrum region selection button M in the Toolbar.
- 3. Press the <Ctrb key. Holding the key pressed, highlight with the mouse the desired regions around peaks in the collected spectrum (Fig. 10-23) (the procedures on spectrum acquisition with the Andor and Renishaw cameras are explained in sec. 10.3.1 on p. 63 and in sec. 10.3.2 on p. 65, respectively).



Fig. 10-23. Peaked regions are selected in the spectrum

The highlighted regions will be displayed in the **Regions** table (Fig. 10-24).

| | Regions | | | | | | | | |
|---|---------|-------------|---------|---------|----|--|--|--|--|
| | | | | | | | | | |
| ∳ | N≗ | Units | Left | Right | QF | | | | |
| ▶ | 1 | Raman, 1/cm | -383,82 | -323,89 | 0 | | | | |
| | 2 | Raman, 1/cm | -147,94 | -89,94 | 0 | | | | |
| | 3 | Raman, 1/cm | 414,69 | 476,56 | 0 | | | | |
| | 4 | Raman, 1/cm | 1101,1 | 1199,7 | 1 | | | | |
| | | | | | | | | | |

Fig. 10-24. Table of highlighted regions

 For every selected region, define functions to apply for processing spectral data in the course of scanning. To this purpose, select the desired options in the **Functions** panel (Fig. 10-25).

| Functions | |
|-------------------------|-----------------------|
| 📝 Area | |
| Pixels nm 1/ | cm Mass Centre |
| Pixels nm 1/ | cm Width |
| Peak Value | |
| Pixels nm 1/ | m Peak Position |
| Skewness Value | |
| Subtract | |
| | |
| | |
| Save As Background | Subtract Background |
| Full Spectrum Recording | OPTIONS |
| Shift Speed V: | ▼ H: ▼ |
| Laser: 472,800 Save | Central Pix: 511 Save |
| Manual Calibration | Central WL: 575.000 |

Fig. 10-25. Functions panel

5. To record the full spectrum at every scan point, press the **Full Spectrum Recording** button.

Selecting the scan mode and the scan area

- 1. Open the Scanning window by clicking the scanning button (Fig. 10-26).
- 2. Select the scanning type using the **Driver** list:
 - **Scanner** scanning will be performed by sample with the laser beam being at a fixed position;

Þ

• Mirror – scanning will be performed by moving the laser beam. This scanning type may not provide SPM data.

| | 🕑 Restart | Driver | Scanner 💌 | Mode | Spectra | ✓ Settings |
|-------|-----------|--------|-------------------|------|-------------|-------------|
| F Hun | © | Rate | Scanner Mirror | • | Direction 🗸 | Area Select |

Fig. 10-26. Scanning-by-sample selected

- 3. Select the desired measurement mode in the **Mode** list. Depending on the actual configuration of the instrument, the following modes can be available:
 - **Spectra** scanning over the sample surface. Only signals form the detectors will be collected. For this mode, the probe tip should be homed or retracted sufficiently far from the sample surface.
 - Lift 3D 3D scanning. If this mode is selected, the Control panel will be extended with special controls to define the origin and the direction of scanning.

Standard SPM modes – scanning over the sample surface. In these modes, along with the common SPM signals the signals from the detectors (CCD cameras) can be collected. Detection channels for the CCD cameras can be adjusted in the Spectra window (see i. <u>10.3</u> on p. <u>62</u>). Selection/unselection of this option can be done in the Spectra Signals checkbox of the ScanSettings dialog (accessible through clicking the Settings... button).

| 🛐 ScanSettin | igs | | | - | | |
|----------------|-------------|------------|-----------|---------|----------|---------|
| Scan Driver | Scanner | • | Name: | Scanner | 000-XXX | |
| Scan Mode | Spectra | · · | • | | Type: 2D | |
| Measurements | s | | | | | |
| Dim Sig | inals | Direction | Pa | SS | Draw | Average |
| 2D PM | T_hf ♥ | Forward | J₽□I | | Show | 1 🗘 |
| 2D PMT | Γ_Ext ▼ | Forward |] – [| | Show | 1 🗘 |
| 2D , | | Forward |] – [| | Show | 1 🗘 |
| 2D _ | | Forward |] – [| | Show | 1 🗘 |
| 2D _ | | Forward |] – [| Ð | Show | 1 🗘 |
| 2D _ | | Forward |] – [| Ð | Show | 1 🗘 |
| 2D _ | | Forward | • | Ð | Show | 1 🗘 |
| 2D _ | | Forward |] – [| | Show | 1 🗘 |
| Spectra Signal | ls inals | | | | | |
| Vertex Delay | 0,00 🗘 | ms | Meas. T | ype St | able | - |
| Scan Count | 1 🗘 |] | Opera | tion No | ormal | • |
| | Cyclic Sav | ve draw | | | X NLC | or ON |
| PassLines | 0 \$ | Sample | Height, I | mm 0, | 50 🗘 | |
| | | | | | | Ok |

Fig. 10-27. Enabling detection of optical signals

4. Select desired signals to detect. Selection of a particular measurement mode is followed by setting the set of detected signals to be the same as it was at the previous invoke of that mode. The set of the detected signals can be viewed and edited in the

ScanSettings dialog (accessible through clicking the settings... button).

NOTE. If no optical signal (defined in the **Spectra** window) is selected for detection, the **Spectra Signals** panel of the **ScanSettings** dialog will be unavailable (Fig. 10-28). In this case, you have to return to the Spectra window and to enable the desired signals to detect.

| 1 | Measurer | ments | | | | | | | |
|---|-----------|-----------|---------|-----------|------|---------|----|---------|---------|
| | Dim | Signals | | Direction | | Pass | | Draw | Average |
| | 2D _ | BV | - | Forward | - | I | • | Show | 1 🗘 |
| | 2D _ | HV_X | - | Forward | - | I | • | Show | 1 🗘 |
| | 2D _ | | - | Forward | - | Ι | - | Show | 1 🗘 |
| | | | | Forward | 1 | | J | 51 | |
| | 2D . | _ | | | 41 | | | .4 | L |
| | 2D _ | | - | Formard | - | Ι | Ŀ | Show | 1 - |
| | 2D _ | | - | Forward | - | Ι | • | Show | 1 🗘 |
| (| Spectra S | õignals | | | | | | | |
| | Execution | n | | | | | | | |
| | Vertex D | elay 0,00 | ¢ | ms | Mea | as. Typ | ре | Dynamic | • |
| | Scan C | ount 1 | \$ | | Q | peratio | n | Normal | • |
| | | Cyr | lic Sa | ve | | | | | |
| | | Cy | clic Re | draw | | | | NL | Cor ON |
| | PassLin | ies 0 | ¢ | Sample | Heig | ght, m | m | 0,50 🗘 | |

Fig. 10-28. View of the **Spectra Signals** panel when no optical signal is selected for detection

5. Click the **Area Select** button. The Scan area selection panel will appear at the right part of the window. Select the desired scan area and define numbers of scan points for both scan directions.

10.5. Scanning



ATTENTION! Do not open any other window of the program until scanning completes because they may interfere with the running process.

Launch scanning with the **Pun** button.

Clicking the **Bun** button results in analyzing the selected spectrum regions at every scan point with the functions selected in the **Functions** panel of the **Andor CCD** (**Renishaw CCD**) tab. Outputs of the functions will be displayed in separate 2D viewers.

Results on the full spectrum will be saved in the DATA subfolder of the program installation folder as a file of the MDT format and a folder of the same name.
10.6. Finishing the Work

1. Regarding the detector in use, perform the following steps:

Andor CCD

Enter 20 °C for heating temperature of the CCD-matrix in the **Set** $T(^{\circ}C)$ input field in the Control panel of the **Andor CCD** tab. Wait until the CCD-matrix is heated to room temperature.



ATTENTION! Do not close the Control program if temperature of the CCD-matrix is lower 0 °C.

Renishaw CCD

The Renishaw CCD camera stops operation automatically after the spectrometer is switched off.

- 2. If the probe was in use, open the feedback loop (
- 3. Retract the sample from the probe. as follows:
 - a. Go to the **Approach** window with the <u>Approach</u> button (Fig. 10-29). Retract the sample from the probe tip by clicking the <u>Move</u> button several times.

| 🕨 Landing | Stop | Remove Step Move Way 0,0000 mm |
|-----------|------|-----------------------------------|
| | | Approach 📑 Step 📑 Move 🚀 Settings |

Fig. 10-29. Control panel of the Approach window

- 4. Switch off the laser.
- 5. Switch off the SPM controller.
- 6. Switch off the spectrometer.
- 7. Close the Control program.

Appendix

1. Optical Viewing System

The Optical Viewing System of NTEGRA Spectra Probe NanoLaboratory (Upright configuration) provides focusing at different distances from the sample surface thus enabling the following functions:

- pointing a laser beam onto the cantilever;
- selection of the sample surface research area;
- approaching and scanning control.

PNL NTEGRA Spectra (Upright configuration) is delivered with the optical viewing system of one of the following models:

- CCD07 model (see i. <u>1.1</u> on p. <u>73</u>);
- CCD27 model (see i. <u>1.2</u> on p. <u>74</u>).

These models are described in details below.

1.1. CCD07 Optical Viewing System

CCD07 Optical Viewing System serves to operate with an optical measuring head.

Assembly and adjustment of the optical viewing system are performed by personnel of NT-MDT Co. when installing the equipment.

Fig. 1-1 shows a general view of the NTEGRA Spectra system with the optical viewing system in place.

Basic parts of the optical viewing system (Fig. 1-1):

- positioning stand 4;
- videomicroscope holder 5;
- videomicroscope positioner 6;
- videomicroscope:
 - CCD camera 7;
 - Tube 8;
 - Beam splitter 9;
 - Illuminator 10.



Fig. 1-1. General view of NTEGRA Spectra with the CCD07 optical viewing system in place
1 – base unit; 2 – optical measuring head; 3 – laser radiation I/O module;
4– positioning stand; 5 – videomicroscope holder; 6 – videomicroscope positioner;
7 – CCD camera; 8 – tube; 9 – beam splitter; 10 – illuminator

1.2. CCD27 Optical Viewing System

1.2.1. Basic Units

Fig. 1-2 shows a general view of the NTEGRA Spectra system with the CCD27 optical viewing system in place.

Basic parts of the optical viewing system (Fig. 1-2):

- Positioning stand 4;
- Stand extender 5;
- Videomicroscope holder 6;
- Videomicroscope positioner 7;
- Videomicroscope 8.



Fig. 1-2. General view of NTEGRA Spectra with the CCD27 optical viewing system in place
1 – base unit; 2 –measuring head; 3 – Laser radiation I/O module;
4 – positioning stand; 5 – stand extender; 6 – videomicroscope holder;
7 – videomicroscope positioner; 8 – videomicroscope

The CCD27 optical viewing system can operate both with the optical measuring head and with the universal one.

Assembly and adjustment of the optical viewing system are performed by personnel of NT-MDT Co. when installing the equipment. Assembly of the videomicroscope depends on the type of the measuring head to be used.

On exchanging the measuring head, the customer reassemble the videomicroscope on his own. Fig. 1-3 shows design of the videomicroscope (pos. 8 in Fig. 1-2) for different types of the measuring head.





a) Videomicroscope for optical measuring head



Fig. 1-3. Videomicroscope 1 – CCD camera; 2 – tube; 3 – beam splitter; 4 – illuminator; 5 – illuminator start/stop button; 6 – telescope; 7 – lens; 8 – adapter

Procedures on assembling the videomicroscope are the following:

- Assembling Videomicroscope for Optical Measuring Head (see i. <u>1.2.2</u> on p. <u>77</u>);
- Assembling Videomicroscope for Universal Measuring Head (see i. <u>1.2.3</u> on p. <u>76</u>).

1.2.2. Assembling Videomicroscope for Optical Measuring Head

NOTE. If the instrument uses the CCD27 optical viewing system and it has been adjusted to operate with the universal measuring head, disassemble the videomicroscope of the optical viewing system first through steps given in i. <u>1.2.3</u> on p. <u>76</u> in reverse order. Then proceed to assembling the videomicroscope.

Assembling videomicroscope

1. Insert the adapter of the videomicroscope (see Fig. 1-3) into the lower end of the tube and secure it with three screws (Fig. 1-4).



Fig. 1-4. Mounting the adapter. Arrow indicates the adapter

2. Insert the beam splitter with the illuminator into the lower end of the adapter and secure it with three screws (Fig. 1-5).



Fig. 1-5. Mounting the beam splitter. Arrow indicates the beam splitter

This completes assembling the videomicroscope.

Preparing optical viewing system for operation

With the videomicroscope assembled, prepare the optical viewing system for operation by performing the following steps:

1. Turn on the illuminator of the videomicroscope with the button 7 (Fig. 1-6).



Fig. 1-6. General view of NTEGRA Spectra with the CCD27 optical viewing system in place 1 – latch; 2 – coarse adjustment knob; 3 – centering screw; 4 – stand angular position fixing screw; 5 – positioning screws of the videomicroscope; 6 – fixing screws of the videomicroscope; 7 – illuminator start/stop button

2. Inspect the image in the monitor of the optical viewing system to verify that radiation from the illuminator of the videomicroscope comes to the center of the lens of the optical measuring head (the object under study should be irradiated uniformly). Achieve uniform irradiation of the sample by repositioning the videomicroscope with the positioning screws 5.

Now, the optical viewing system is ready for operation.

1.2.3. Assembling Videomicroscope for Universal Measuring Head

NOTE. If the instrument uses the CCD27 optical viewing system and it has been adjusted to operate with the universal measuring head, disassemble the videomicroscope of the optical viewing system first through steps given in i. <u>1.2.2</u> on p. <u>77</u> in reverse order. Then proceed to assembling the videomicroscope.

Assembling videomicroscope

1. Insert the telescope (see Fig. 1-3) into the lower end of the tube and secure it with three screws (Fig. 1-7).



Fig. 1-7. Mounting the telescope. Arrow indicates the telescope

2. Insert the beam splitter with the illuminator into the lower end of the telescope and secure it with three screws (Fig. 1-8).



Fig. 1-8. Mounting the beam splitter. Arrow indicates the beam splitter

3. Screw the lens supplied with the optical viewing system into the lower end of the beam splitter (Fig. 1-9).



Fig. 1-9. Mounting the lens. Arrow indicates the lens

This completes assembling the videomicroscope.

Preparing optical viewing system for operation

With the videomicroscope assembled, prepare the optical viewing system for operation by performing the following steps:

1. Remove the mirror from the beam path by turning the turnet of the laser radiation delivery and detection unit with the knob 8 to position M2 (see i. 2.3 "Laser Radiation I/O module" on p. 19).



Fig. 1-10. General view of NTEGRA Spectra with the CCD27 optical viewing system in place

- 1 latch; 2 coarse adjustment knob; 3 centering screw; 4 stand angular position fixing screw;
 5 positioning screws of the videomicroscope; 6 fixing screws of the videomicroscope;
 7 illuminator start/stop button; 8 turret rotation knob
- 2. Using the coarse adjustment knob 2, move the videomicroscope holder to its midposition.
- 3. Turn on the illuminator of the with the button 3.
- 4. Loosen the stand angular position fixing screw 4.
- 5. Using the centering screw 3, adjust angular position of the videomicroscope to view the target object.

- 6. Fix the stand with the screw 4.
- 7. Verify correct positioning of the videomicroscope by viewing the cantilever (or sample) on the monitor of the optical viewing system (the image should be free of distortions). If necessary, loosen the fixing screws 6 and reposition the videomicroscope. Adjust position of the videomicroscope observing the cantilever (or sample) on the monitor of the optical viewing system. Fix the videomicroscope with the screws 6.
- 8. Using the coarse focusing knob 2, focus the videomicroscope on the cantilever (sample). Adjust the videomicroscope with the adjustment screws 5 to place the center of the field of view on the object to observe.

Now, the optical viewing system is ready for operation.