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Products: Atomic Force Microscope attoAFM I

User Manuals

Atomic Force Microscope attoAFM I/MFM/KPFM P5788

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I. Introduction

The attoAFM I is designed particularly for the use at extreme environmental conditions such as ultra-low temperature, high magnetic fields and high vacuum. Reliable functionality at these extreme conditions is provided by implementing the outstanding attocube systems nanopositioning modules as well as a force detection scheme based on an all fiber low-coherence interferometer (explained in detail in the next section).

To perform low temperature microscopy, the attoAFM I is cooled by a controlled exchange gas atmosphere in a liquid Helium bath cryostat or a variable temperature insert or a closed cycle cooling system.

The instruments may be surrounded by both liquid helium and liquid nitrogen radiation shields, separated by vacuum insulation.

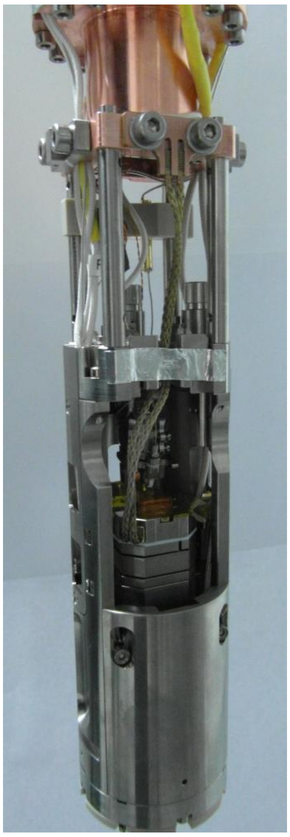


***Figure 1**: The attoAFM I standard setup*

I.1. System Overview

The attoAFM I microscope is a tool to image, characterize, analyze, and manipulate materials surfaces on a nanometer scale in low temperatures as well as high magnetic fields. This short chapter wants to give a rough overview on the concepts of the system.

The attoAFM I consists of the *measurement head* which is inside a housing suspended on the lower end of the *microscope stick*, which itself is placed in a *vacuum tube*. Before cooling down the system, this vacuum tube is evacuated and backfilled with some He exchange gas for coupling to the temperature outside of the vacuum tube. Typically, this vacuum tube is then cooled by insertion into a *He bath cryostat*, a *Variable Temperature Insert (VTI)*, a *pulse tube cooler*, or by other means.

The main part of the attoAFM I measurement head is the AFM *cantilever*, a thin bar typically made from silicon and possibly functionalized with different coatings, which is placed before the end of an *optical fiber*, hence forming an optical cavity, the length of which can be measured by a *laser/detector system*.

The sample to be examined is placed (in standard setups) on an *ANPxyz101* positioner stack for coarse motion (5 x 5 x 5 mm^3) and an *ANSxyz100* scanner (typ. 30 x 30 x 15 μm^3 at 4 K, for details see the specifications page at the end of the manual). Just underneath there is typically also a temperature sensor and a small heater, which allows controlling the temperature of the sample precisely.

When the sample is brought into contact with the cantilever, the interaction forces can be measured by recording the length of the cavity. While the sample is scanned, typically the distance between -tip and sample is kept constant by the use of a *feedback loop*. For these purposes, one uses the *electronic scan controller* ASC500, which generates all required voltages and records all relevant signals. Additionally, a *high voltage amplifier ANC300* is part of the system, amplifying the unipolar, 0 – 10 V signals to 0 – 150 V, as well as generating the signals required to move the stepper positioners.

Regarding the optics, *laser* light is guided to the fiber-cantilever cavity, and the back-reflection is then detected by a *photo-receiver*. The signal generated by the photo-receiver is analyzed and recorded with the ASC500 controller.

I.1. Safety Information

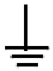
For the continuing safety of the operators of this equipment and the protection of the equipment itself, the operator should take note of the **Warnings, Cautions,** and **Notes** throughout this handbook and, where visible, on the product itself.

The following safety symbols may be used on the equipment:

Laser safety warning. Class 1M laser product.

Warning. Risk of electric shock. High voltages present.

Warning. Risk of danger. Refer to the handbook for details on this hazard.



Functional (EMC) earth/ground terminal.

The following safety symbols may be used throughout the handbook:

Warning. An instruction, which draws attention to the risk of injury or death.

Caution. An instruction, which draws attention to the risks of damage to the product, process, or surroundings.

Note. Clarification of an instruction or additional information.

I.1.a. Warnings

Note: Please read the manual for the positioners, scanners, and the controllers prior to initial operation! Mind the warnings mentioned in there!

Warning. To prevent electrical shocks do not remove the cover of the control unit. Unplug the power cord and all other electrical connections and consult qualified service personnel before servicing or cleaning. Operate only under dry conditions and at room temperature.

Warning. If this equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired. Do not operate the instrument outside its rated supply voltages or environmental range. In particular, excessive moisture may impair safety.

Warning. Any individual vacuum space must be equipped with a security overpressure valve! In case of hidden leaks air may enter into vacuum spaces and build up large amounts of condensate at cold walls and interfaces. In case such system is warmed up again such ice may evaporate quickly and easily build up extremely dangerous overpressures! If no overpressure valve is mounted on such vacuum space or the valve is blocked e.g. due to mounting to a recovery line, this overpressure may cause sudden breakdowns of the container, i.e. explosions. If in doubt, please contact attocube systems.

Laser Safety Warning. Class 1M Laser product. This device utilizes a laser which emits invisible light. There is potential for eye damage if proper caution is not taken. The laser assembly should not be modified in any way. The fiber cable should never be disconnected from the laser device for any reason. The laser's wavelength is specified at 1330 nm with a maximum power output of 1.5 mW. Please wear protective eyewear when the laser source is turned on and never look directly into to the laser port or fiber.

Warning. Plugging or unplugging modules of the electronic while connected to power line will cause damages to the electronic. For replacing modules disconnect the electronics from power and wait at least 5 minutes. Internal components need to be discharged.

Caution. Never connect any cabling to the electronics when contacts are exposed! Never connect any cabling to the electronics when the electronics is not in GND mode! The scan piezos at the heart of a positioner unit are high voltage components and can cause serious injuries. Be careful not to cause a short-cut between the contacts in the BNC or any other connectors.

Caution. For laboratory use only. This unit is intended for operation from a normal, single phase supply, in the temperature range 5° to 40°C, 20% to 80% RH. The unit must be connected only to an earthed fused supply of 100, 115 or 230 V, 50/60 Hz.

Caution. In case of failure refer to your local dealer or attocube systems. Users are cautioned not to attempt to access, open, modify, or service any part of the setup unless outlined herein or otherwise directed by the technical support staff from attocube systems. Take special care if connecting products from other manufacturers. Clean only with a dry cloth.

Caution. Because of the inherent fragility of the positioners and scanners and the extreme conditions they are operated in, no responsibility can be taken by the manufacturer for breakdowns of the ceramic piezo stacks. In case of breakage, please contact the manufacturer for details of the repair service.

II. Mode of Operation and Force Detection Scheme

The attoAFM I is an atomic force microscope built around an optical fiber based interferometer. The sensor is compatible with any commercial cantilever and measures the vertical deflection of the cantilever with picometer resolution. The microscope is designed to work both in contact and in non-contact mode.

This highly compact microscope guarantees highest resolution tip-sample positioning and an optimized sensor adjustment suitable for any environment: room or low temperature, high magnetic field or high vacuum conditions.

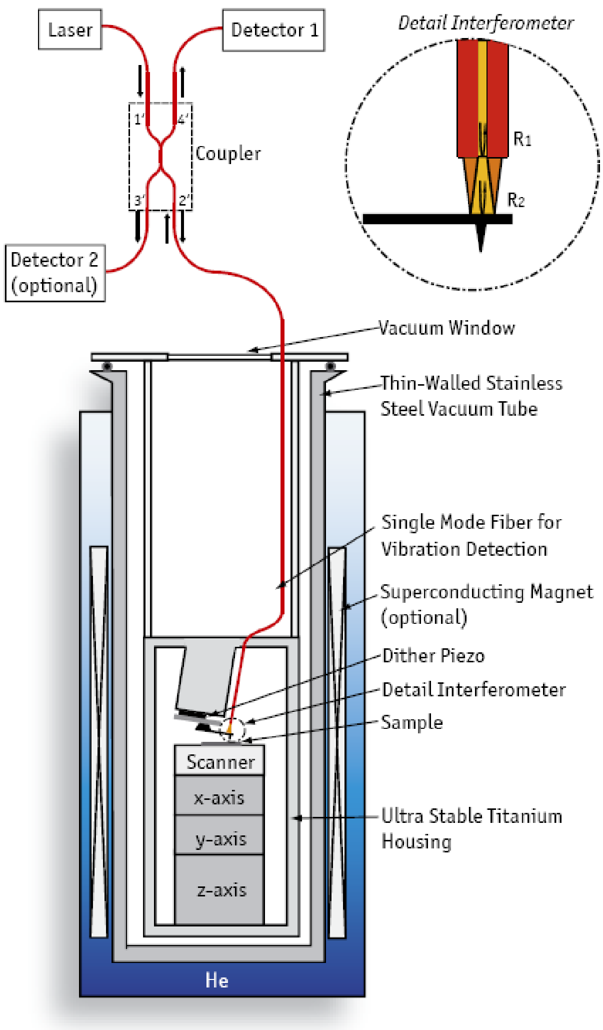


***Figure 2**: Schematic drawing of the attoAFM I system.*

The force detection scheme for the attoAFM I microscope system is based on an all fiber low-coherence interferometer (seeFigure 3).

Via a 50/50 fiber coupler, a laser beam illuminates a cavity built up from the end-face of the fiber and the back-face of the cantilever. The light reflected back from these surfaces gives rise to interferences that can be seen by a detector. Due to the low reflectivity constants these surfaces form a low-finesse Fabry-Perot cavity.

Monitoring the intensity of the interference fringes allows measuring the tip deflection. The low coherence of the laser source has the advantage of eliminating spurious interference signals resulting from other reflections in the setup (e.g. the coupler), thus leading to an increase of the signal-to-noise ratio of about 30 dB.

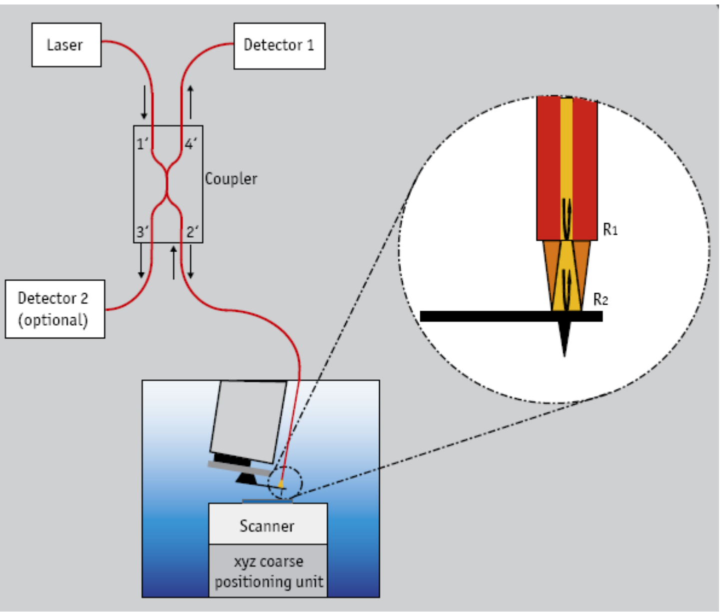


***Figure 3**: Schematic drawing of the AFM I setup.*

The periodicity of the signal corresponds to half the wavelength λ of the laser source. Hence, the system can be easily calibrated and a measured voltage difference can be translated into a height difference.

The control fiber has to be adjusted with respect to the cantilever in such a way that the interference signal is at the quadrature point, i.e. in the middle between two extrema values. In this case, the sensitivity of the sensor is highest.

There are different ways to perform topographic images. Common modes are the so-called contact- and non-contact modes.

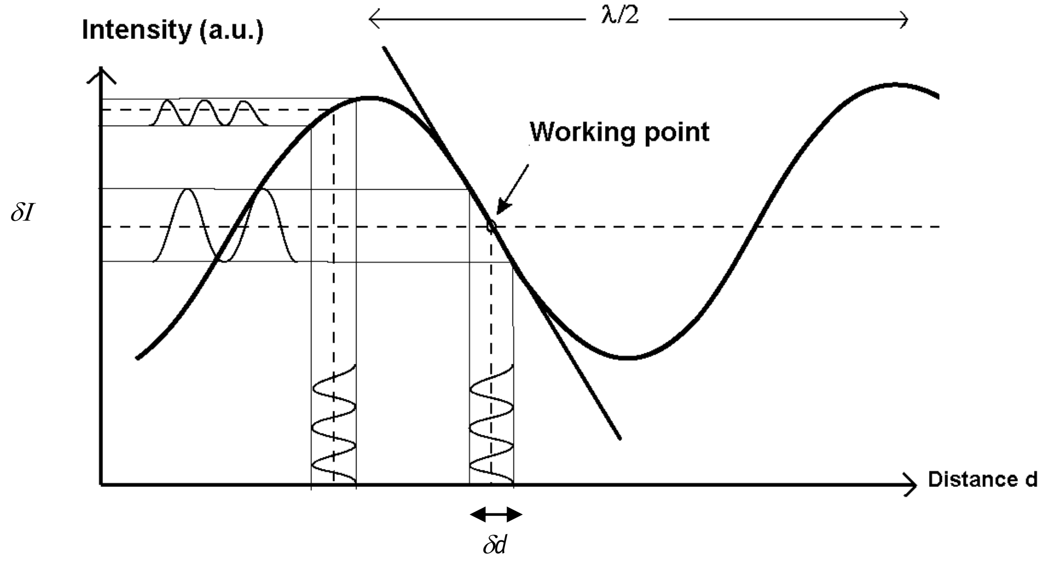


Figure 4: *Schematic drawing of the interference signal.*

Contact mode:
The tip is in direct contact with the sample, while the intensity is in the middle of the intensity range. The cantilever is not oscillating. Due to a detuning of the cantilever position before contacting the surface, a compressive force is applied onto the sample. The strength of the force corresponds to the amount of detuning times the force constant of the cantilever.

For example, the cantilever position can be adjusted off contact such that the intensity is in a minimum. Now, by engaging the feedback loop, the sample is lifted up until the tip engages with the sample surface. While still increasing the sample height, the smaple pushes the cantilever up until the intensity reaches the middle intensity value. The amount of bending corresponds in this case to a cavity length difference of $\lambda/8$, and hence the force on the tip is $F = \lambda/8*k$, with k being the force constant of the cantilever. As the feed-back loop keeps the intensity constant, the amount of bending is kept. Hence, the force on the tip is kept constant.

Non-contact mode:
For non-contact mode, no detuning is applied; the cavity length is such that the signal is in the middle between the extrema. Then, the cantilever is excited by the dither piezo at its resonance frequency. The input of the lock-in measures the AC component of the photo-detected signal, which reflects the oscillation amplitude of the cantilever. As the cantilever approaches the sample, the oscillation amplitude drops rapidly with decreasing tip-sample distance. This signal serves as the input to a feedback loop which maintains the cantilever oscillation amplitude at a so called 'set level', which corresponds to a given force between the sample and the cantilever. During the scan, the output signal of the feedback loop is recorded (z-scanner piezo voltage), providing a topographic image.

III. Components of the Microscope and Technical Specifications

III.1. Microscope Setup

The sample is mounted onto a stack consisting of an ANPxyz101 coarse positioning unit, an ANSxyz100 scanner and a copper sample plate that carries heater and temperature sensor. The attoAFM I module, consisting of the cantilever holder and the attoAFM I head together with the fiber based deflection detection system, is fixed onto a slide which is mounted in the microscope housing.

The microscope module is mounted below the 3He pot . The top of the 3He insert contains all electrical connectors and feedthroughs, the vacuum window, and the fiber feedthrough. The AFM module including the fiber- and sample positioning units is attached to the bottom of the insert, see Figure 5. The microscope housing is inserted into a inner vacuum chamber (IVC) and subsequently sled into the cryostat utilizing a log G10 tube .

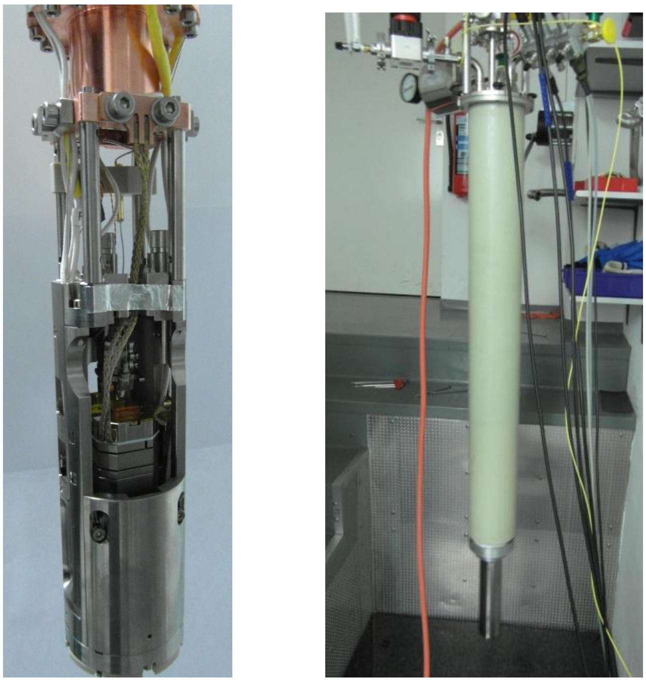


Figure 5: *Image of the attoAFM I housing*

The positioning stage ANPxyz101 is built from three individual positioners mounted on top of each other (please find the specifications in the respective manual). The ANSxyz100 scanners will be mounted on top of the positioner stack, followed by a plate with integrated heater and temperature sensor. Finally, the quik exchange sample holder is mounted to the very top of the assembly.

The stack will be delivered completely mounted. If you ever need to disassemble and consequently reassemble it, please recheck the correct orientation (which is given in the system specs) and the correct order of the different parts and screws. Wrong screws may cause severe damages to the scanners and positioners.

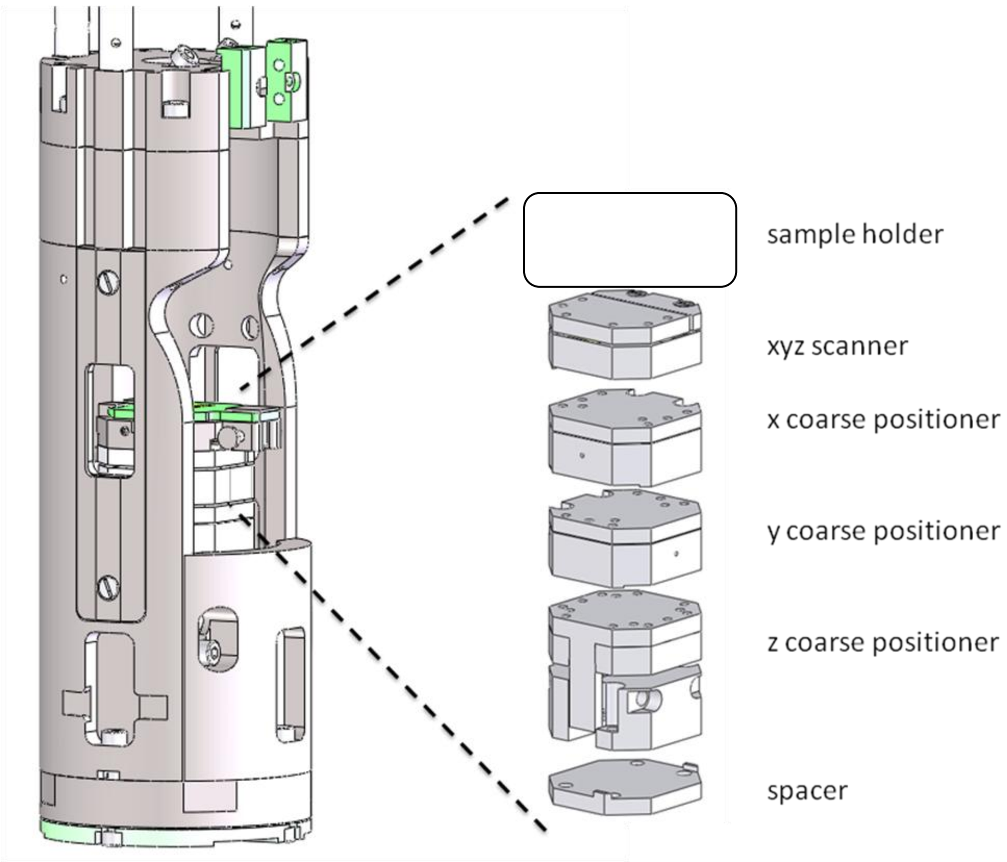


Figure 6: *Standard assembly of the stack with a lower coarse-positioning unit and an upper scanner unit. The sample holder is specially designed for the 3He system.*

III.2. Electronics

III.2.a. ANC300: High voltage amplifier

The ANC300 can be used either as 'piezo positioning controller' or as 'high voltage amplifier'.

Four axes of the ANC300 controller provide the high voltages for driving the attocube scanners for fine positioning (voltage amplifier with an amplification factor 15).The fourth axis is another high-voltage amplifier that drives the dither piezo of the attoAFM I.

For technical specifications please refer to the respective manual.

The axis number is counted from the right to the left.

For technical specifications please refer to the respective manual.

Figure 7: *ANC300 used as high voltage amplifier on the axes 1-4, and as a AC-DC coupler for the tip voltage.*

III.2.b. ACC100: LDM1300 Laser Detector module and Break-Out Panels

The ACC100 is a 19"housing that allows to attach electrical break-out panels as well as additional electronic modules, e.g. the LDM1300 laser detector module.

The electrical break-out panels convert the 12- and 8-pin Fischer connectors to easy accessible BNC connectors.

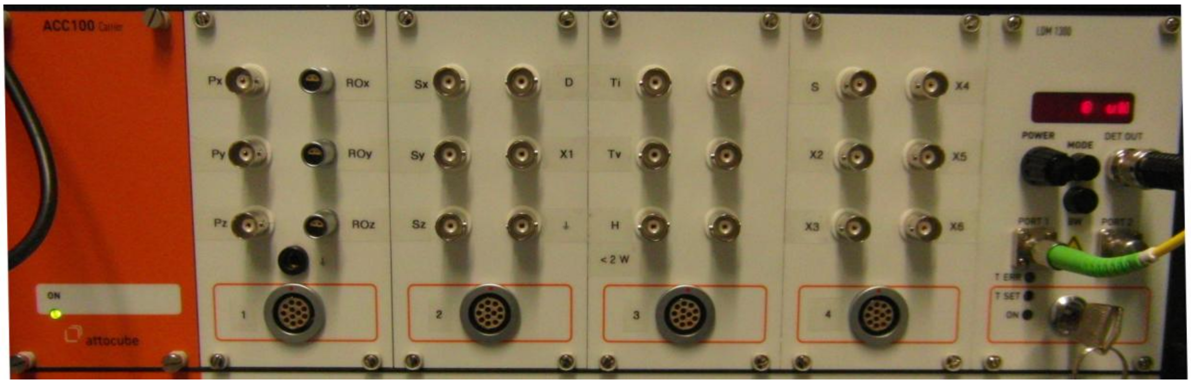


Figure 8: *The ACC100 with the Break-Out-Panels and the LDM1300.*

Four break-out panels serve as connector panels between the controllers with BNC outputs and the microscope with multi-pin Fischer cables.

For details about the labelling and electrical connections see section IV.1.

III.2.c. ANC350 Piezo controller for positioners

Piezo controller driving /RES encoded atto**cube** positioners in coarse and fine positioning mode. Please note that for the He-3-system highly resistive wires are taken for the resistive encoding. This is why you need to upload a modified RESencoder-file with the extension '_mod'. The original files may be taken without highly resistive wires.

For technical specifications please refer to the respective manual.

Figure 9: *The ANC350 piezo controller.*

III.2.d. Laser detector module: LDM1300

Figure 9: *LDM1300.*

The attoAFM I system includes a temperature-stabilised laser-detector module, the LDM1300.

The wavelength of the laser diode is 1310 nm. The built-in photodetector may be read out via the 'DET OUT' BNC connector.

For technical specifications please refer to the respective manual.

III.2.e. ASC500 SPM Controller

The modular and flexible digital SPM controller ASC500 combines state of the art hardware with innovative software concepts to offer an unmatched diversity of scanning-probe microscopy applications to the customer. All desirable functions and high-end specifications for controlling the experiment of your choice in AFM, CFM, SNOM, and STM are available. The flexible, FPGA-based architecture allows the implementation of your particular requirements to the system.

Figure 10: *The ASC500 SPM controller*

III.3. (Dis-)Assembling the attoAFM I Module

This section will describe how to assemble and align the AFM module.

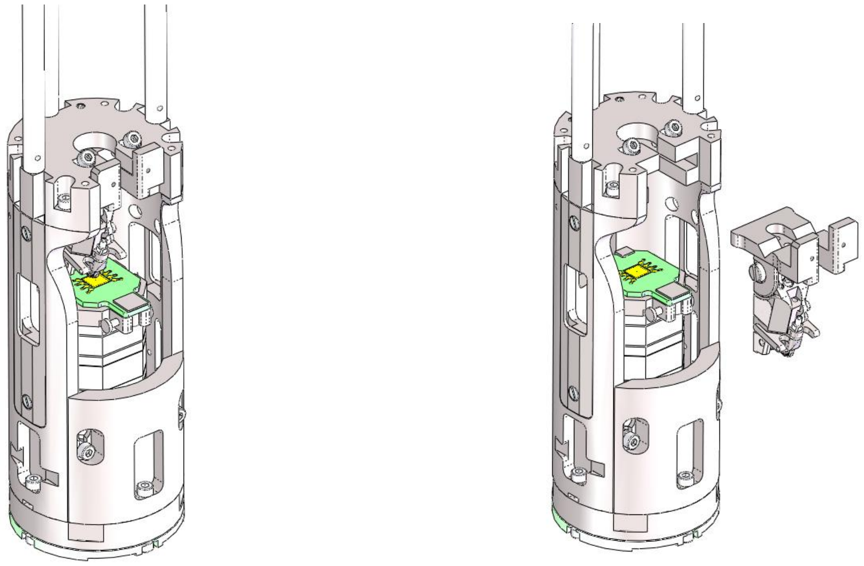
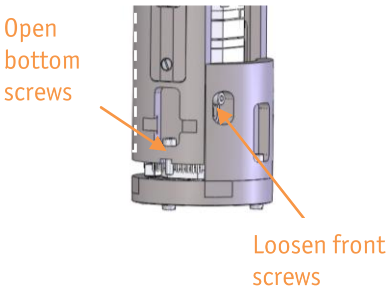


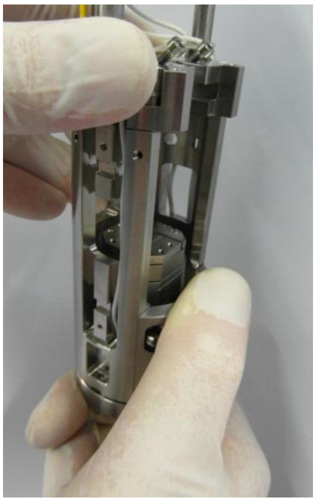
Figure 11: *The assembled attoAFM I module.*

III.3.a. Inserting (removing) the stack into (from) the microscope housing

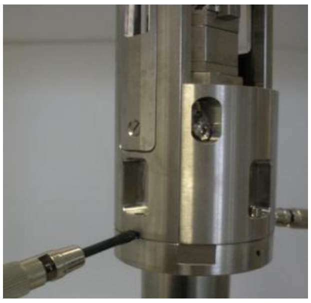


The microscope stack is fixed to the housing bottom. By loosening the two screws in the front and opening the three screws on the **left, right and back side** the whole bottom part including the positioner stack may be pushed down and electrically disconnected from the stick.

The stack and bottom plate may then be taken off towards the front of the housing.

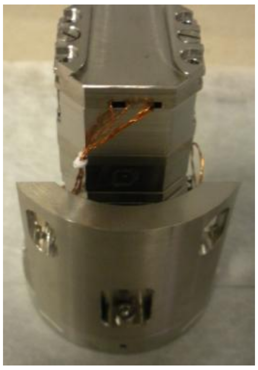


As there might be some force needed to disconnect the bottom plate, we recommend keeping the housing with one hand and using the thumb of the other hand to push the front plate down.



Noteworthy, two slits on the sides of the bottom plate may be used to smoothly lever off the plate using a screw driver.

Before mounting the stack back into the housing, inspect the cabling before pushing the bottom plate upwards. Make sure not to squeeze any wire!



'Bottom part', including the housing bottom plate and positioner stack.

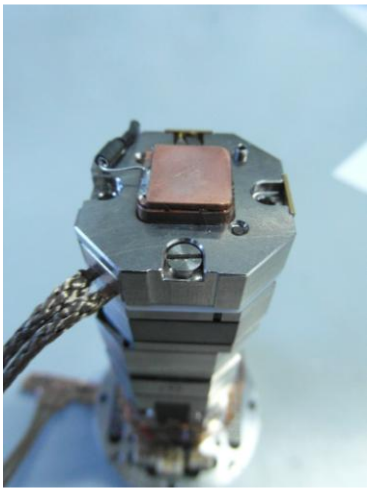
You must not disassemble the stack. If there is any problem with the stack please contact attocube AG for support.

III.3.b. Mounting the Scanner-Positioner Stack

Carefully place the stack onto the spacer plate on the bottom plate. Make sure that the stack cannot fall down. Move the stack backwards until the outer shape matches with the spacer plates.

Introduce two screws (M2.5 x 8) in the bottom plate in order to fix the stack, see picture. Once the screws are tightened a little by hand, you can use a screwdriver to tighten them further.

III.4. Sample Exchange and Thermal Contact



In order to change samples you need to take of the stack, as described above.

A copper plate is fixed on top of the stack, electrically isolated but thermally coupled, to the copper body that is directly connected/coupled to the ^{3}He-pot with copper braids.

For cleaning the sample holder acetone as well as alcohol may be used (please take care that no conductive paste gets between the sample plate and the lower copper body).

The sample holder may be electrically connected with a single pin connector to set the sample potential. In the present setup the pin 'S' is supposed to be connected to the sample holder (with a manganin wire) .

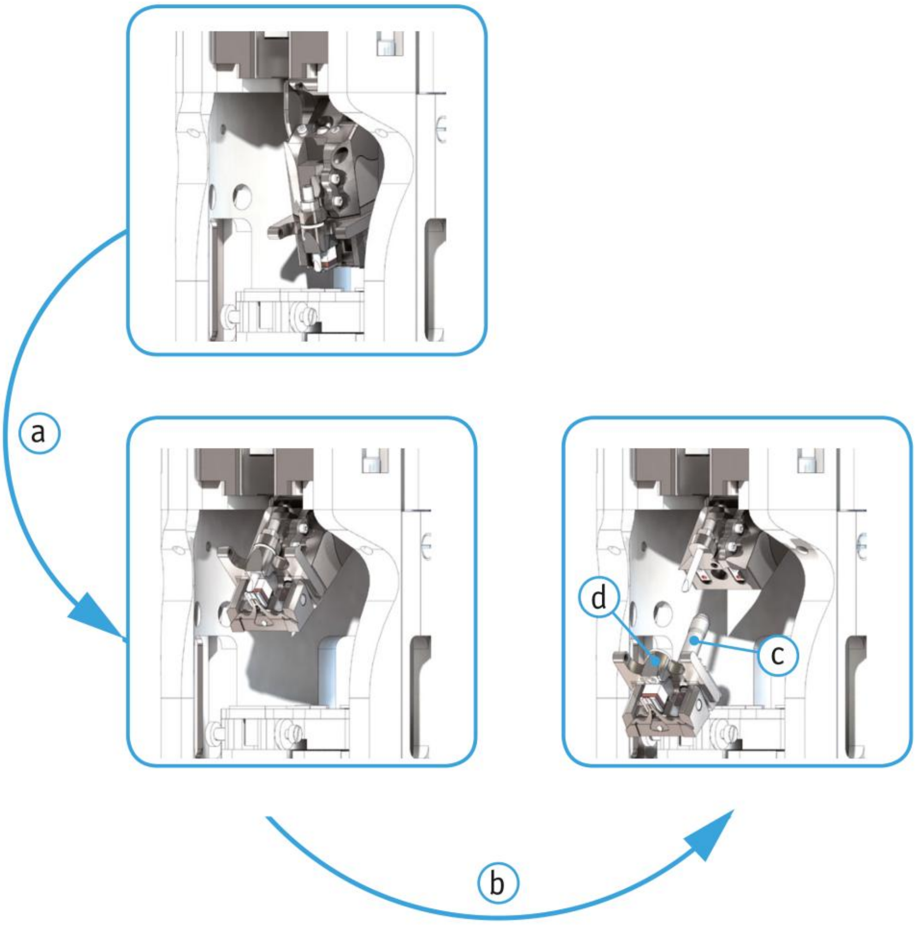
In order to fix the sample Apiezon N (available in the tool box) and/or silver paste (available in the tool box) should be used.

The temperature sensor may be fixed with a headless set screw. Note that this screw should only be fixed gently, in order to protect the sensor. Attocube also recommends to cover the smaller top plate of the sensor with Kapton tape (that can be found in the tool box).

For additional electrical connections, the pins X2...X6 on the housing bottom may be accessed on the break-out panel '4' of the electronic rack, delivered with the microscope.

After the sample has been changed, the housing can be assembled again and at last the copper rods need to be attached to the upper copper body as shown in the picture to the left with two screws. These screws should be tighly closed in order to give as good thermal conduction as possible. Attocube also recommends to polish the inner surfaces of this contact from time to time and to add a little bit of Apiezon grease (that can be found in the tool box) before closing it.

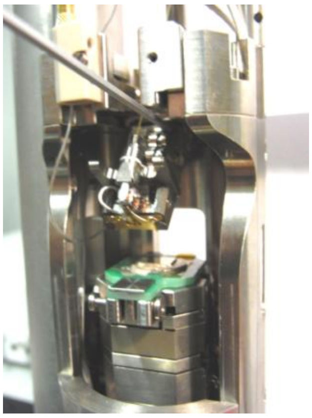
III.5. Changing the AFM Cantilever

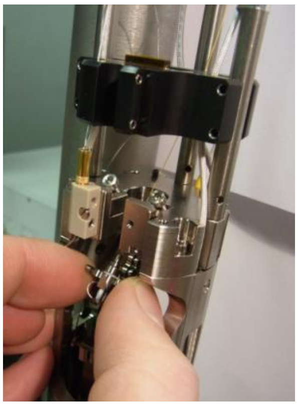


The attoAFM I (+) head includes an alignment-free cantilever holder that is compatible with all commercially available XY-auto alignment AFM tips.

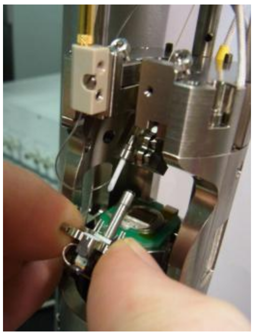
In order to change the tip the attoAFM I (+) head needs to be handled as follows::

1. Turn the AFM head upwards. Insert an Allen key in the provided hole as shown in the picture to the left (a)





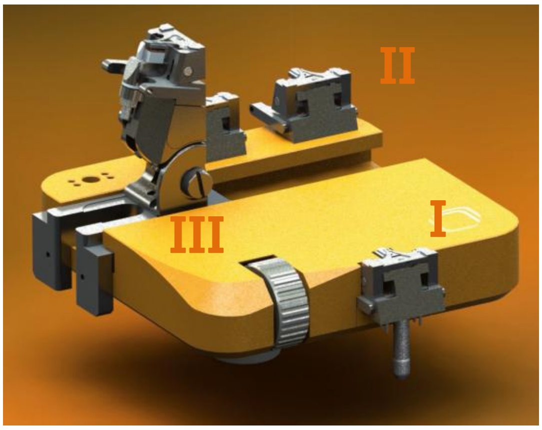
2. Grab the cantilever holder on its arms and smoothly strip it off, see picture to the left. (b)



3. Perform the AFM tip exchange (for details, see description below)
4. Once a new tip is mounted, reattach the cantilever holder:
A guiding rod (c) automatically centers the cantilever holder, while the fiber ferrule is still far away from any potentially harmful obstacle.
5. Feed the ferrule into the cantilever holder through another guiding sleeve (d). The ferrule is protected by an additional soft Teflon cap.
6. Tilt the head back into the housing – it flips conveniently and firmly into its dedicated parking position.

No further mechanical alignment is necessary.

III.5.a. The cantilever mounting platform



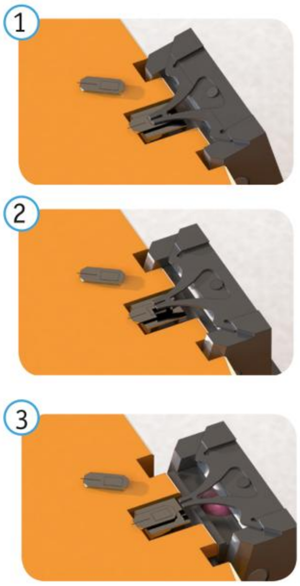
The mounting platform provides three stages for the alignment and adjustment procedures of the cantilever on the AFMI head, namely:

I – cantilever exchange stage

II – AFM head parking site

III – slider module parking site

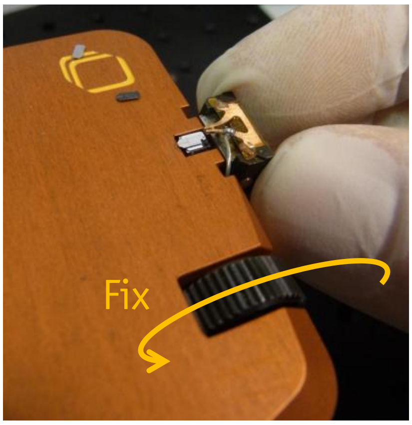
III.5.b. Cantilever exchange



In order to change the cantilever you need to:

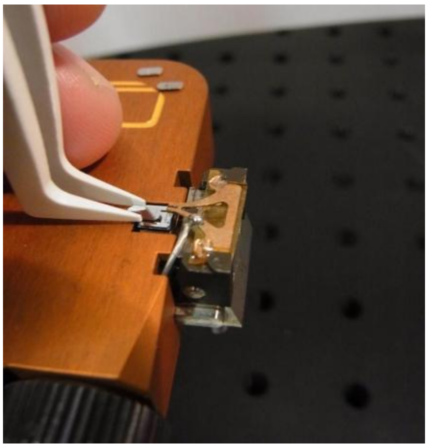
Put the cantilever holder into the cantilever exchange stage (**1**).

Slide back the spring blade, first push the back part down (spring opens) (**2**) then pull the slider towards the back(**3**).

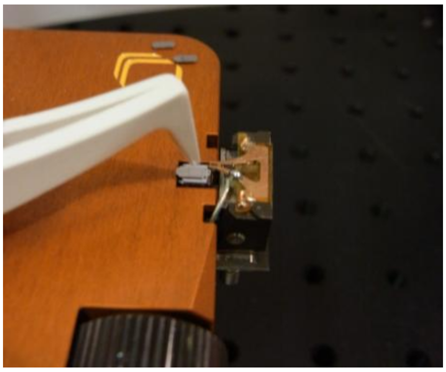


In order to fix the AFM head into cantilever exchange stage, you need to turn the black knurled **w**heel sitting left to it.

Perform cantilever exchange **4.-5.**;



Do only use the plastic tweezers for handling the cantilevers near the alignment chip. Metallic parts may easily destroy this silicon chip.



If the cantilever is not sitting correctly within the chip you may gently push it with the end of the plastic tweezers until it gets into the right place.

Close the holder. **6.-7**.

Remove holder from the exchange basis, and insert it back, see above, into the AFM head.

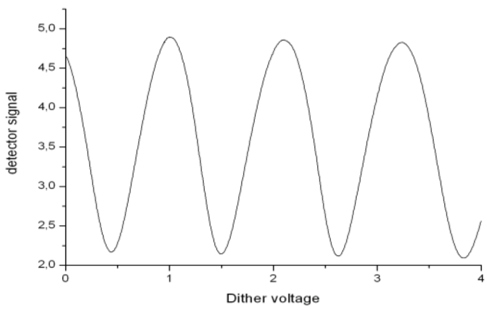
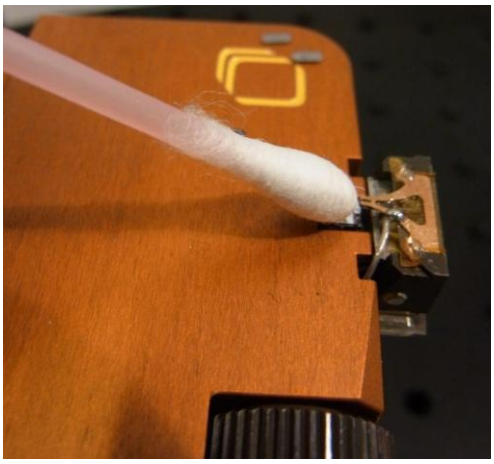


Figure 12: *Interference fringes seen in the cavity signal on changing its length (spectroscopy ADC1 vs. DAC1).*

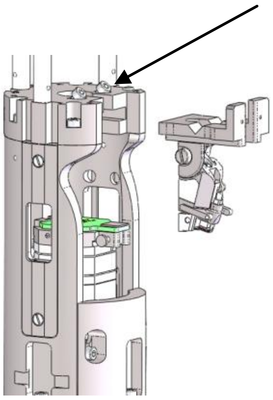
Finally, run a spectroscopy on the dither piezo (interference signal ADC1 vs. DAC1 dither voltage) in order to see whether you get a decent interferogram (in this context, "decent" is defined by the intensity of the interferometric signal and its symmetric shape)..
If the interferogram becomes comparable to the one depicted in Figure 12 with a contrast of ≈ 1 V a good measuring condition is met.

Typically, the cavity gap amounts to 10-50 µm. Be aware that the cavity will decrease when cooling the microscope to liquid Helium temperature (up to 10 µm depending on the lever in use).



Note that the silicon cantilever-alignment chip needs to be kept very clean. Hence, from time to time it needs to be gently brushed with a alcohol-soaked Q-tip.

III.5.c. Mounting the attoAFM I sensor head into the housing



Before mounting the attoAFM I head into the housing make sure that the z-positioning stage is fully retracted and that the cantilever cannot touch the sample, i.e. sits clearly above the later.
Now slide the aligned attoAFM I head into the microscope housing and tighten the two screws on top of the housing (see arrow).
This will fix the slider with the microscope head in place that also makes an electrical contact to the microscope stick (e.g. the dither piezo connection).

○

IV. Connecting the attoAFM I Module

IV.1. Electrical Connections

IV.1.a. ASC500 Controller Connections

Table 1 gives an overview of all the electrical connections of the ASC500 controller.

Controller	Port	Controller	Port	Usage
ASC500	X-Out	ANC300 (SCAN)	DC in x (1)	X scanner
	Y-Out	ANC300 (SCAN)	DC in y (2)	Y scanner
	Z-Out	ANC300 (SCAN)	DC in z (3)	Z scanner
	DAC1	ANC300 (SCAN)	DC in D (4)	dither offset (DC)
	DAC2	AC/DC coupler for KPFM	DCin	tip voltage
	DAC3	Sample S1	S	sample voltage
	ADC1	photo detector	DET out(T-piece)	interferometer signal (DC)
	HF1 in	photo detector	DET out (T-piece)	interferometer signal (AC), AFM
	HF1 OUT	ANC300 (SCAN)	Mod in D (4)	dither excitation
	HF2 in	photo detector	DET out (T-piece)	interferometer signal (AC), KPFM
	HF2 OUT	AC/DC coupler for KPFM)	ACin	KPFM excitation
	Ground (rear side)	break-out panel	⊥	grounding of the stack
	USB port	PC	USB	PC - ASC500 communication
	NSL port	ANC350 (Step)	NSL	ANC350 (STEP) communication

Table 1: *Overview of controller connections.*

IV.1.b. Break-Out Panel Connections

The break-out panels are connected to the different controllers via BNC cables. These cables are labelled for your convenience. See Table 2 for a complete list.

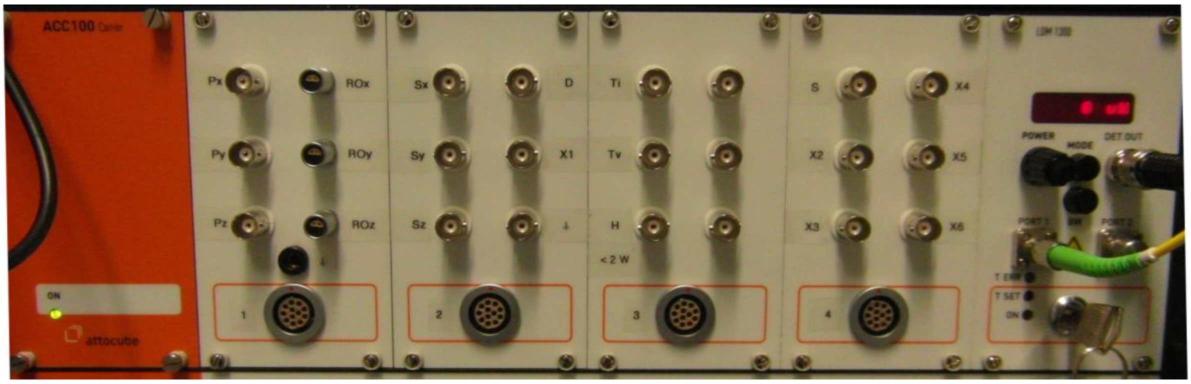


***Figure 13**: Break-Out Panels.*

Label break-out panel	Controller connection	Usage
Px	ANC350 Axis 1 (OUTPUT)	X Positioner: Px
Py	ANC350 Axis 2 (OUTPUT)	Y Positioner: Py
Pz	ANC350 Axis 3 (OUTPUT)	Z Positioner: Pz
Sx	ANC300 Axis 1 (OUTPUT)	X Scanner: Sx
Sy	ANC300 Axis 2 (OUTPUT)	Y Scanner: Sy
Sz	ANC300 Axis 3 (OUTPUT)	Z Scanner: Sz
T-I	temperature monitor Ch A	temperature sensor current
T-V		temperature sensor voltage
H	temperature monitor (optional)	
⊥	ASC500 ground (rear side)	grounding of the stack
X1	---	optional user contact (above housing)
X2	---	optional user contact
X3	---	optional user contact
X4	---	optional sample contact
X5	---	optional sample contact
X6	---	optional sample contact
D	ANC300 Axis 4 (OUTPUT)	Dither DC voltage/ AC excitation
⊥	ASC500 Ground (rear side)	grounding of the stack

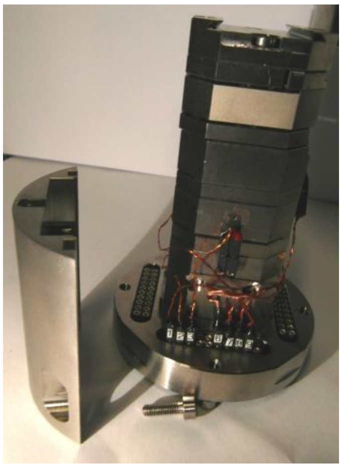
Table 2: Overview of Break-Out panel connections.

IV.1.c. Pin Connections

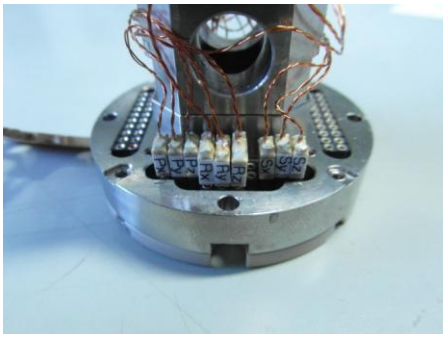
All electrical pin connections to and from the attoAFM I module are low temperature compatible pin. Most electrical connections are twisted pairs of copper wires adjusted to the correct lengths. Wires carrying positive voltages are labelled red, GND wires are marked black. All wiring from the positioner stack has to be connected to the front- and back-row pin connectors in the bottom plate of the housing. All connectors are labelled with short codes. The port numbers are counted from the left to the right (1,2,3..., see pictures below).
Table 3 describes these codes and provides details about the connections.

Port number	Usage	Connector code	Leads to Fischer/BNC connector
1 front	X Positioner	Px	1
2 front	Y Positioner	Py	1
3 front	Z Positioner	Pz	1
4 front	X Scanner	RESx	1
5 front	Y Scanner	RESy	1
6 front	Z Scanner	RESz	1
7 front	--	--	-
8 front	X Scanner	Sx	2
9front	Y Scanner	Sy	2
10 front	Z Scanner	Sz	2
1 back	temperature sensor	T-I	3
2 back	temperature sensor	T-V	3
3 back	sample heater	H	3
4 back	sample	S	4
5 back	user contact	X2	4
6 back	user contact	X3	4
7 back	user contact	X4	4
8 back	user contact	X5	4
9 back	user contact	X6	4
10 back	User contact	BNC1	BNC1
AFM head 1	Dither	D	2
AFM head 2	user contact	X1	2
Housing top	ground	⏚	2

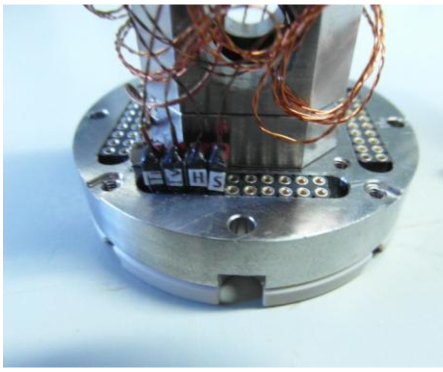
Table 3: Overview of pin connections.



In order to get access to the front-row-pin connections the front side needs to be taken off by opening one screw.



Front view of the present housing pin connections.

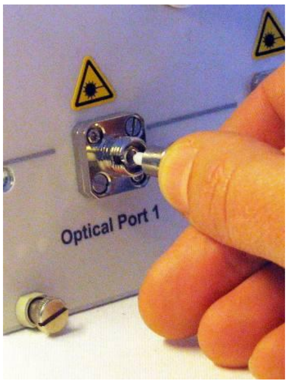


Back view of the present housing pin connections.

IV.1.d. Vacuum Feedthroughs

There are multi-pin cables used to connect the microscope to the electronics rack. The black cables are labelled '1' ,'2','3','4'.

IV.2. Fiber Connection



The optical read out system of the cantilever of the attoAFM I system requires only one optical connection which will be described in this section.

For the optical connection plug the attoAFMI fiber into the laser port1 (or port2) of the LDM1300. Please make sure to have the right orientation of the fiber groove and tongue system (see Figure 14). Do not tighten too hard as this might damage the optical connector.

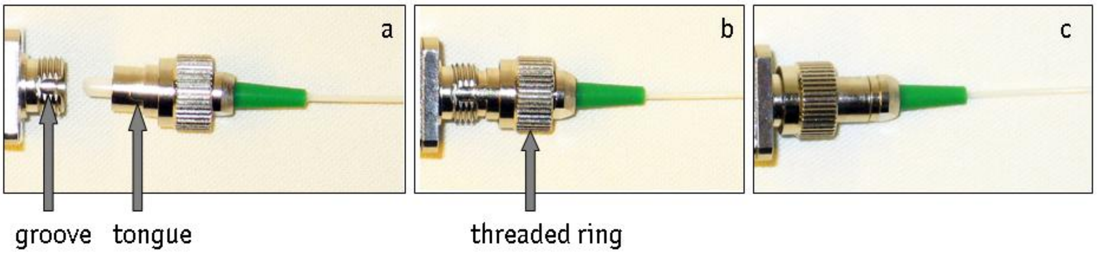


Figure 14: *Connecting the FC/APC pigtail to a connector port. Be careful to obey the correct orientation of the groove and the tongue.*

V. Measurement Procedure

This chapter will lead you through your first measurement in the different AFM modes available.

V.1. Operation Modes

Different operation modes are possible. The two fundamental ones are the Contact Mode - with and without feedback - and the Non-Contact Mode (also intermittent or modulation mode).

V.1.a. Contact Mode without Feedback (constant height)

In contact mode, the tip is brought in contact with the sample. The sample is scanned without any feedback control of the z scanner, i.e. topographic variations are directly translated into a deflection of the cantilever. The photo detector signal is acquired during scanning, which can be directly converted into a height signal by using the formula:

$$dz = \frac{\lambda}{2\pi} \cdot \frac{dV}{\Delta V} \approx 246nm \cdot \frac{dV}{\Delta V}$$

Here, ΔV is the peak-to-peak amplitude of the interferogram; dz is the change in height corresponding to a change dV in detector voltage.

Important note: This mode is only suitable for samples with a maximum height corrugation of $\lambda/4$! Otherwise, the contrast may be inverted.

The contact mode without feedback, also called const.-height mode, is the easiest and fastest way to record an AFM image, because it is not depending on z feedback. Please note that the image topography is inverted if the working point within the interference signal is set to a negative slope.

For contact mode scans, cantilevers with a small stiffness (< 5 N/m) are used in order to keep the forces between tip and sample small.

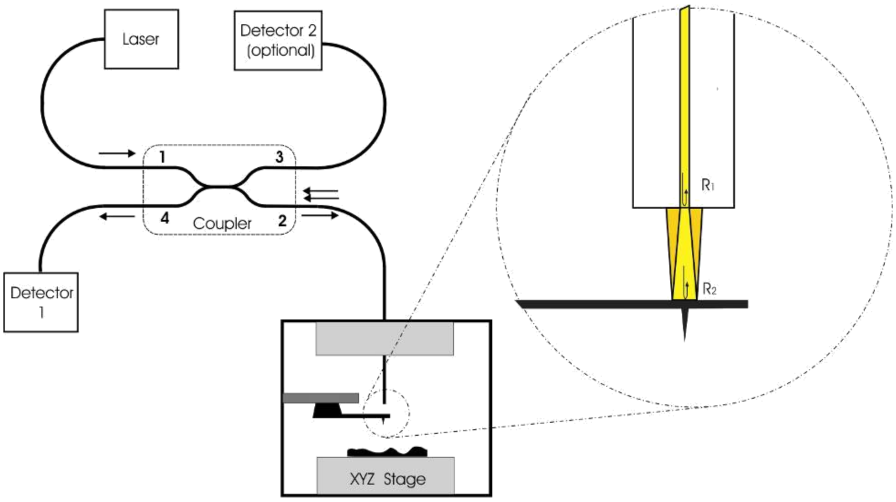


Figure 15: *Schematic drawing of the attoAFM I operated in contact mode without feedback.*

V.1.b. Contact Mode with Feedback on (constant force)

The tip is first brought into real contact with the sample surface. In this mode, however, a feedback mechanism is employed to keep the deflection signal at a constant value by varying the voltage of the z-scanner. This will maintain a constant force between tip and sample. The feedback setpoint should be set to the point of maximum slope in the interference signal (working point), i.e. to the middle between two interference extrema. The main measurement signal in this mode is the voltage applied to the z scanner, which can be translated into height data by using the z piezo calibration value (usually this is automatically done by the controller software). The error signal is the detector signal.

This mode is suggested for samples with height corrugations >λ/4!

Since a feedback always causes low pass filtering, the measurements in constant force mode are usually slower but less noisy than constant height images.

V.1.c. Non-Contact Mode

In this mode, the cantilever is excited at its resonance frequency by an AC voltage applied to the dither piezo. Correspondingly, the photo-detected AC signal at the resonance frequency reflects the oscillation amplitude of the cantilever. As the cantilever approaches the sample, this vibration amplitude decreases rapidly with diminishing tip-sample distance. Note that the oscillation amplitude is already reduced due to long-range forces (such as van der Waals- or electrostatic/magnetic forces), i.e. the tip is not necessarily physically touching the sample.
At this point, a oscillation amplitude called 'set level' is defined, corresponding to a given force between sample and cantilever. The vibration amplitude of the cantilever serves as the input to a feedback loop which maintains the cantilever oscillation at the set level by adjusting the voltage on the z-scanner (amplitude feedback). Alternatively, the amplitude and the

phase of the oscillation resonance can be kept constant (phase feedback).
During the scan, the output signal of the feedback loop is recorded (z-piezo voltage), providing the topographic information. The error signal is the oscillation amplitude of the cantilever.
For non-contact mode scans, cantilevers with a high stiffness (k > 2 N/m) are used to avoid the problem of unwanted jump-to-contact (stop of the oscillation).

Figure 16 illustrates the interference signal measured by an interferometric deflection detection system in non-contact mode. The output signal is largest if the cantilever vibrates around the point of maximum slope of the interference signal (working point). The working point is set by applying an offset DC voltage to the dither piezo.

The AC voltage detected (peak to peak) by the Lock-In amplifier can be translated into the real oscillation amplitude,

$$dz = \frac{\lambda}{2\pi} \cdot \frac{dV}{\Delta V} \approx 246nm \cdot \frac{dV}{\Delta V}$$

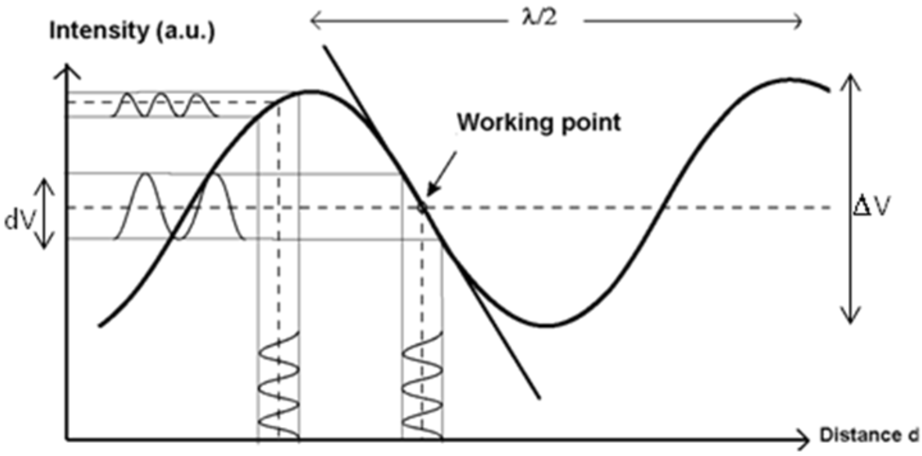


Figure 16: *Schematic drawing of the interference signal.*

Note that if the vibration amplitude of the cantilever is slightly higher than a quarter of the wavelength, the optical signal gets distorted at its extrema. This is shown in Figure 17, which illustrates the electrical signals corresponding to the dither excitation (top) and the interference signal (bottom). Sweeping the (DC) dither voltage and measuring the interference signal provides a quick calibration of the vibration amplitude of the cantilever.

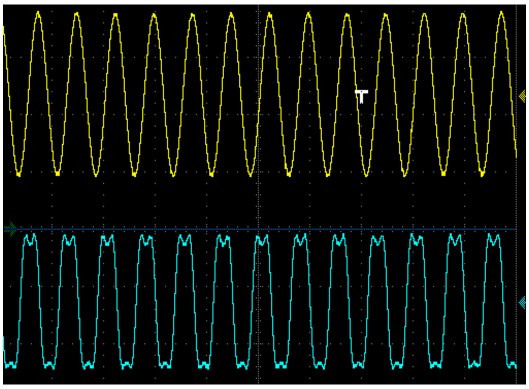


***Figure 17**: Electrical signals corresponding to the dither excitation (top) and interference signal (bottom).*

V.1.d. MFM and EFM measurements

MFM (EFM) is performed in non-contact mode utilizing a magnetic (conducting) tip, i.e. the tip of the cantilever is covered with hard-magnetic or conductive material. The tip-sample interaction leads to a shift Δf of the lever mechanical resonance frequency f_0. This interaction is directly related to the gradient ∇F of the magnetic force acting on the tip along the lever bending direction. For a given lever of spring constant K (>> ∇F), the relation between the force gradient and the frequency shift Δf (<< f_0) is given by the approximation

$$\nabla F \cong -2\,(\Delta f / f_0)\,K.$$

Thus, the magnetic (electric) information can be either extracted from the detected phase shift while measuring at a const. frequency or correspondingly from the measured frequency shift in a phase-lock loop. In order to avoid mixing of topographic and magnetic (electric) information, the magnetic signal is usually recorded at bigger tip-sample distances.

V.1.e. KPFM measurements

KPFM is performed in non-contact mode utilizing a conductive tip, i.e. the tip of the cantilever is covered with a contactable conductive material. In contrast to the MFM or EFM modes the cantilever is not mechanically excited at its resonance frequency by using the dither piezo but by using an oscillating electrical tip AC potential. The oscillation amplitude thereby depends on the tip to sample DC voltage difference. Hence, by keeping the oscillation amplitude at its minimum (i.e. HFx=0) with means of tuning the tip DC voltage a direct mapping of the sample potential is possible. The KPFM mode is performed in a constant sample to tip distance using the dual pass technique. In the first line the sample topography is recorded and in the second line the topography is retraced at a certain tip-sample distance while the KPFM signal is recorded.

V.2. Performing a Scan

The following sections describe the most important steps to be performed before a scan is started. The AFM system must be in the following state before you can go on:

- all electrical and optical connections are correct (see chapter IV).
- The tip-fiber system is well aligned.
- The step function for the positioner axes of the ANC300 axes should be activated in the hardware but disabled in the coarse menu of the software.
- For the non-contact mode, the BNC labeled *HF out* of the Lock-In amplifier (on the ASC500) should be connected to the AC IN of the AC/DC-coupler breakout panel and both the *HF in* input of the Lock-In amplifier and ADC1 are connected in series to the output of the photo detector.

With an o*pen system at ambient conditions, start to manually approach the sample* to the tip using the z coarse positioner. This should be done under eye inspection to a distance close enough for reasonable auto-approach duration but still far enough away to avoid any crash of the tip. If the system is going to be cooled down, the sample surface should be moved down 250-500 steps from the contact position to (i) avoid the tip crashing into the sample on cooling due to thermal contractions and (ii) assure a reasonable approach time at low temperatures.

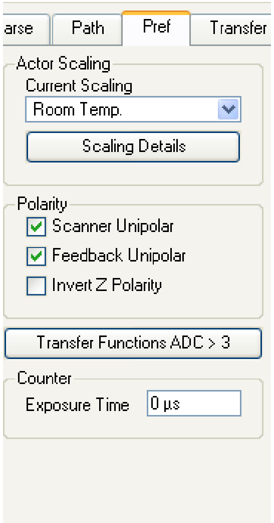
V.2.a. Set the Actor Scaling

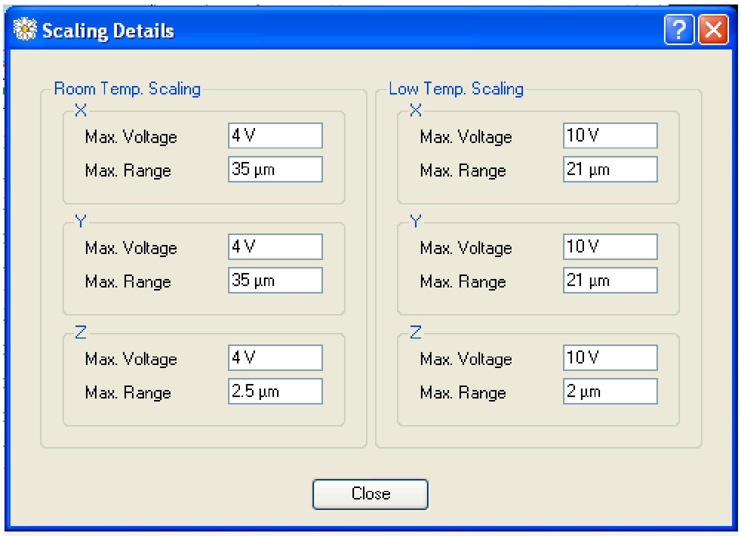
The voltage outputs of the ASC500 must be activated (check the 'output active'-button within the DAISY software)

However, before you start activating the outputs, check that the correct temperature and the according scaling details are set correctly in the 'Actor Scaling' box of the 'Pref' window.

Two sets of parameters for room temperature and low temperature may be saved. The parameters for the low temperature scaling entered here by attocube systems are the calibrated 4 K values.

Note that at room temperature 4 V is the upper limit for the ASC500 scanner outputs and at 4 K this limit increases to 10 V (this limits are valid for an amplifier with amplification factor 15, namely the ANC300). If the customer wants to measure at temperatures in between he should not exceed the linearly extrapolated voltages.





V.2.b. Non-Contact Mode

In non-contact mode the tip-fiber cavity has to be adjusted to the point of highest interference sensitivity, i.e. at which the change in the tip-fiber distance gives the biggest (linear) change in the interferometric signal. After the cavity has been adjusted to this point, the cantilever must be excited at its resonance frequency and the photo-detected AC voltage AFM Aosc will be used as measuring signal for the feedback loop. The basic steps for this procedure are sketched below.

1. Adjustment of the fiber-cantilever cavity:

Run a so-called ‘dither spectroscopy’ (spectroscopy of ADC1 vs.DAC1) to check the interferogram. To do so, choose one of the 'Spec' windows and enter the following settings:

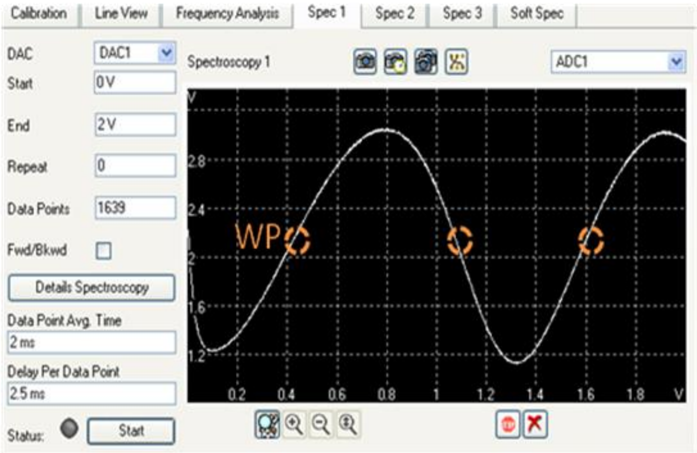
DAC:	DAC1
Start:	0V
End :	max. 4V (@RT); max. 10V (@LT)
Data Points:	>1000
Data Point Avg. Time:	2-5 ms
Delay per Data Point:	0-5 ms

Choose ADC1 as the signal to be monitored.

Start the spectroscopy.

The Spectroscopy View should look similar to the figure below. The working point WP (point of highest sensitivity where signal depends linearly on the cavity length between fiber end and cantilever) is located at the midpoint between minimum and maximum of the interference signal.

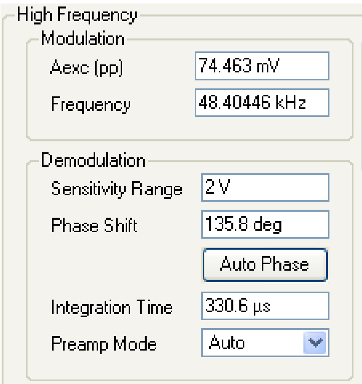
Adjust ADC1 to the WP by tuning the dither piezo voltage, i.e. DAC1, in the 'DACs' window.



2. Finding the resonance frequency f_{res} of the cantilever:

In non-contact mode, the cantilever is excited at its resonance frequency f_{res} with the an AC voltage A_{exc}. To deduce f_{res} , the excitation frequency is swept while measuring the oscillation amplitude (and the phase) of the cantilever.

The excitation amplitude and all demodulation settings are made in the 'Lock In' widow. The frequency sweep is done in the 'Calibration' window.



High Frequency box:

Amplitude (pp): 10-200 mV (@RT); 1-20 mV (@LT)
This sets the excitation amplitude

Frequency: This frequency will be deduced from the calibration

For 'Demodulation' parameters, choose:

Sensitivity Range: 1-2 V (maximum oscillation amplitude Aosc)

Phase Shift: leave 0 or set an offset phase so that the phase signal is 0 at resonance.

Integration time: set to 100 µs – 1 ms

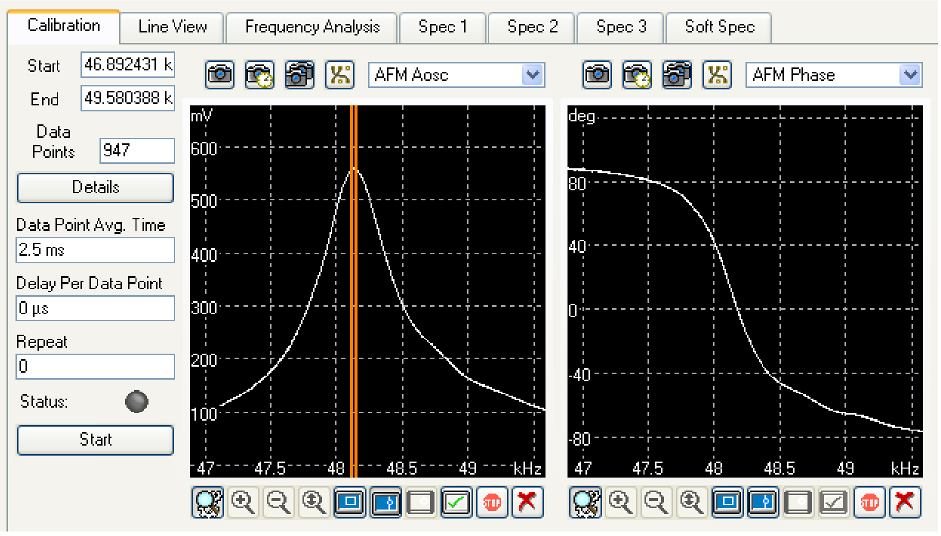
Leave the Preferences value set to 1.

Calibration:

Frequency range (Start - End):	45 kHz – 95 kHz (depending on the cantilever)
Data Points:	500-5000
Data Point. Avg. Time:	2.5ms

Delay Per Data Point: 0s

Start the calibration.



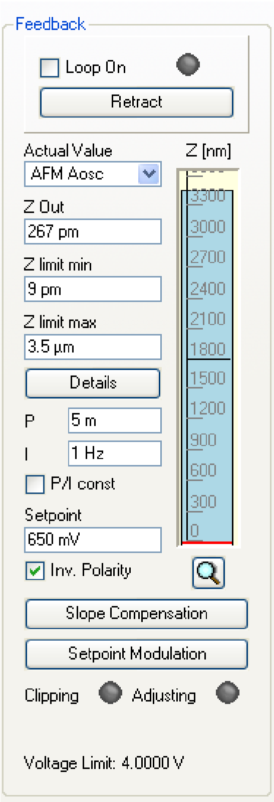
The upper figure shows the calibration window and the oscillation amplitude (AFM Aosc) of the cantilever as a function of the excitation frequency. From this resonance curve, f_{res} can be selected with the help of the *frequency selection* tool. Press the ok button (green checkmark) to set the chosen frequency as the excitation frequency. It is also possible to zoom in by using the *frequency range selection* tool.

It is possible to extract the oscillation amplitude A of the cantilever from the resonance curve with help of the calibration of the interference signal:

$A = \lambda/2\pi * V/\Delta V$ with V being the photo detector signal at resonance and ΔV being the peak-peak amplitude of the interference signal.

The amplitude A is typically chosen to be within 20 nm to 100 nm.

Besides the the oscillation frequency of the resonance, the Full Width at Half Maximum (FWHM) can also be deduced from the resonance curve which allows calculating the cantilever's Q factor, $Q = f_{res}/\text{FWHM}$.

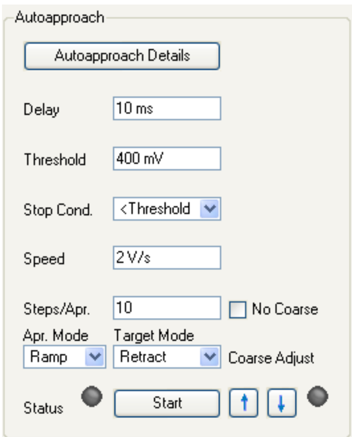


3. Starting the Auto Approach:

The auto approach procedure will expand the z-scanner while constantly checking the photo-detected Ac signal AFM_A_{osc}. If a certain threshold (*stop condition*) is not detected within one stroke, the z piezo will be retracted and a predefined number of coarse steps in z direction will be executed. Afterwards, ramping of the z-scanner will be restarted. Please note that you can affect the range of the z-piezo stroke by adjusting 'Z limit min' and 'Z limit max' within the 'Feedback' tap.

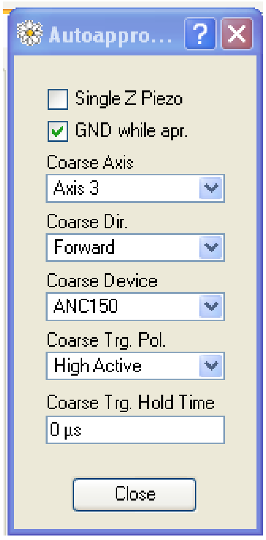
Before starting the auto approach, select the AFM_A_{osc} as 'Actual Value' in the 'Feedback' tab. Furthermore, choose the following parameters:

Z out:	0nm
Z limit min:	0nm
Z limit max:	maximum z piezo stroke. See data sheet at the end of this manual.
P:	1-5 m
I:	1-20 Hz
Inv. Polarity:	checked (signal AFM Aosc is expected to decrease with increasing Z)



In the 'Coarse' tab, one can find the 'Auto Approach' box where the following parameters should be entered:

Delay:	10 ms
Threshold:	~2/3 * of AFM_Aosc
Stop Cond.:	< Threshold
Speed:	2V/s (@RT), 3V/s (@LT)
Steps/Apr.:	see data sheet (part of this manual)
Apr. Mode:	Ramp
Target Mode:	Retract

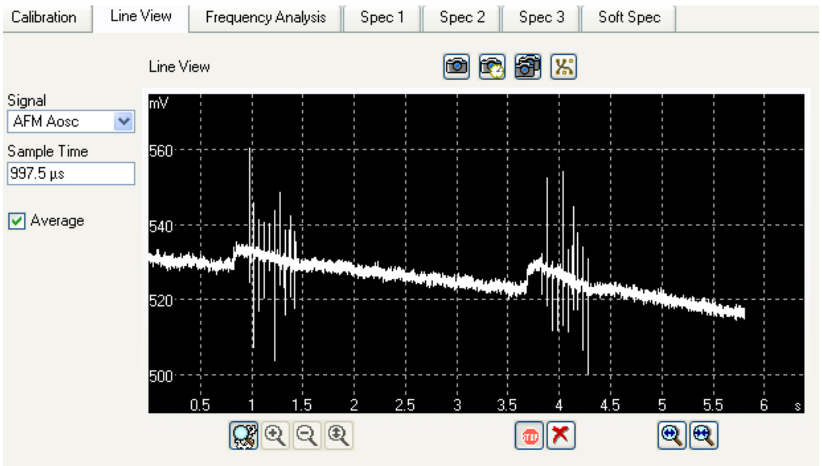


Check the 'Autoapproach Details' where the following parameters should be entered:

Coarse Axis:	Axis 3
Coarse Dir.:	Forward
Coarse Device:	ANC150
Coarse Trig. Pol.:	High Active
Coarse Trig. Hold Time:	0 µs

The auto approach should last between a few minutes and half an hour, depending on approach parameters and initial tip-sample distance. After the auto approach is finished, the sample surface is within reach of the z scanner.

The figure below shows how the auto-approach signal is typically reflected in the photo-detected AC signal AFM Aosc (at an already quite small tip-sample distance)

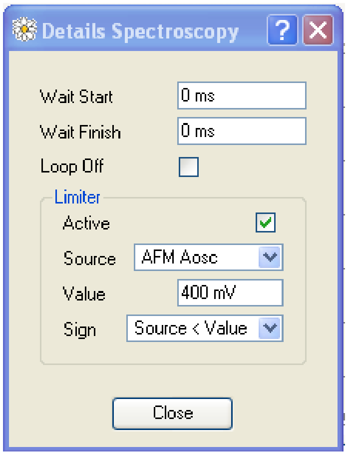


4. Finding a good setpoint:

During the measurement, the distance (force) between tip and sample will be held constant by the P/I-feedback loop that tries to keep the oscillation amplitude at a given setpoint by varying the voltage applied to the z scanner. It will now be described how a reasonable setpoint for the feedback loop can be found with help of a z-spectroscopy:

In order to perform a 'z-spectroscopy', choose on of the 'spec' windows and enter with the following parameters/values:

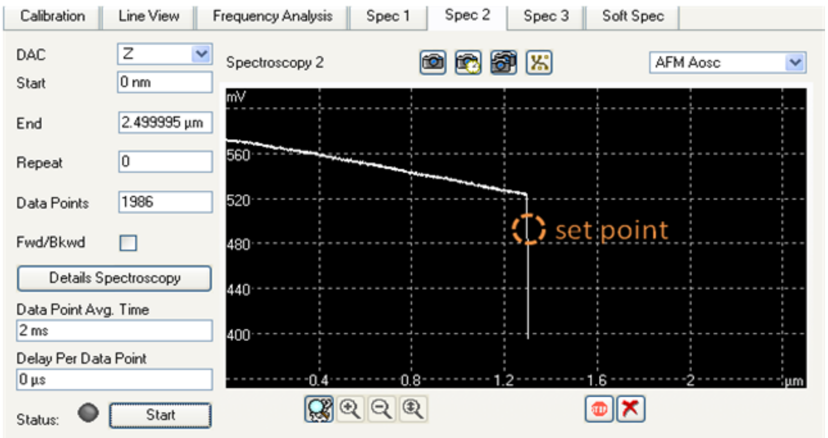
DAC: Z
monitored signal: AFM Aosc
Start: 0pm
End: maximum z piezo stroke. See data sheet at the end of this manual
Data Points: 1000
Fwd/Bkwd: not checked
Data Point Avg. Time: 2-5ms
Delay Per Data Point: 0ms



Now open the 'Details Spectroscopy' sub menu and enter the following parameters:

Limiter: (Stops the z spectroscopy before the tip crashes into the sample)

Active: checked
Source: AFM A_{osc}
Value: 0.5 * A
Limit: Source<Value



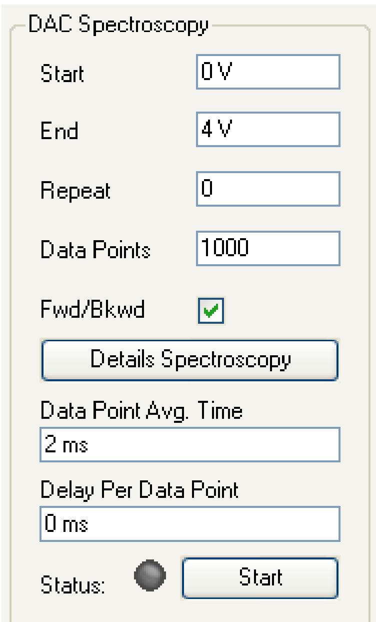
The contact point where the signal is dropping sharply should be in the middle of the z scanner range. Move manually upwards with the z positioner in a step by step fashion to adjust this position.

The set point for the AFM Aosc signal should be chosen in such a way as shown in the picture (typically, the set point value corresponds to 75% - 95% of the free oscillation amplitude).

V.2.c. Contact Mode

In Contact Mode the cantilever is not excited at its resonance frequency. Only a DC voltage is applied to the dither piezo (normally on DAC1) and correspondingly the DC voltage of the photo-detected signal (normally on ADC1) is used to detect the topography.

Contrary to the non-contact mode the tip-fiber cavity is not set to the WP by tuning DAC1 manually but by using ADC1 as the feedback input and defining the working point WP as the setpoint.



1. Adjustment of the fiber-cantilever cavity length:

Execute a dither spectroscopy (photo-detector signal (normally on ADC1) vs. DC dither voltage (normally on DAC1)) to check the interferometric signal. To do so, choose the 'Spectroscopy' and the 'Spectroscopy View' tab with the following settings:

Start: 0V
End : 4V (@RT); 10V (@LT)
Data Points: 1000
Data Point Avg. Time: 2ms
Delay per Data Point: 0ms

Start the spectroscopy.

The '*Spectroscopy View*' should look similar to the Figure 18 below.

The working point WP (point of highest sensitivity where signal depends linearly on the cavity length between fiber end and cantilever) is located at the midpoint between minimum and maximum of the interference signal.

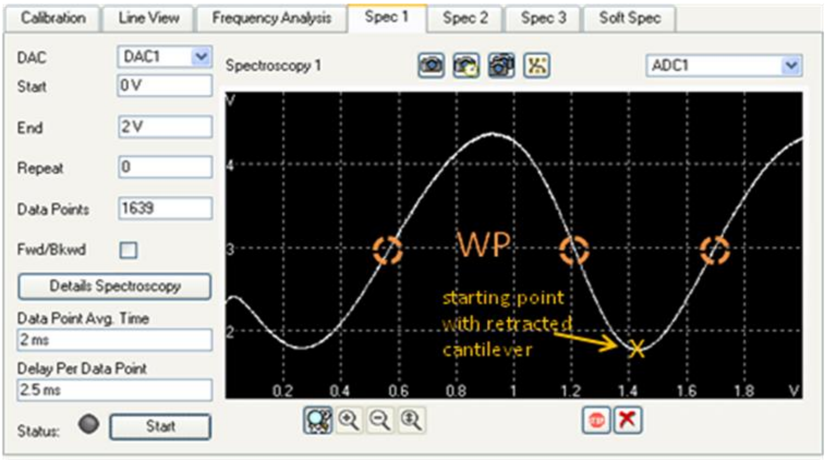


Figure 18: Working point WP.

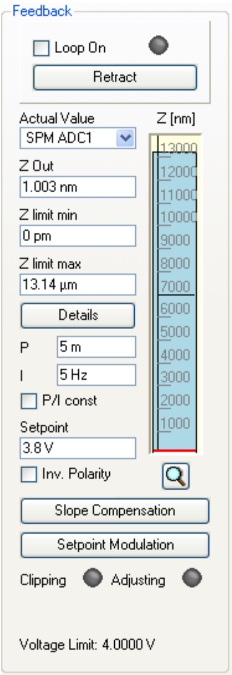
The fiber-tip cavity now must be adjusted to a minimum ($ADC1_{min}$) of the interferogram by tuning the DC voltage of the dither piezo (normally on DAC1). Hence, as soon as the sample starts pushing against the tip the cavity will decrease and the interferometric signal must increase.

2. Starting the auto approach:

The auto approach settings must be set accordingly, i.e. the voltage of the limiter must be set slightly higher than the minimum of the interferometric signal and the stop condition must be set to " > threshold". Remember that the autoapproach monitors the parameter set kin the feedback window. This parameter must be set to the DC signal of the photo-detector (ADC1).

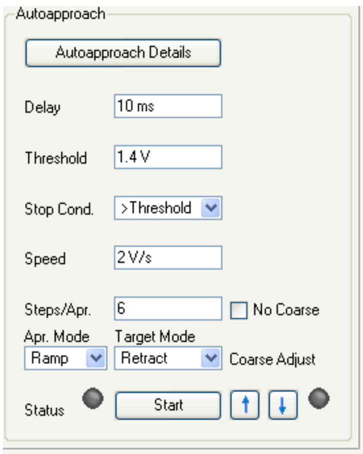
Examples for correct autoapproach settings are given in the 3 figures below.

The auto approach will stop when the tip touches the sample. The cantilever will be bent and the interference signal will sharply increase, see figure to the left



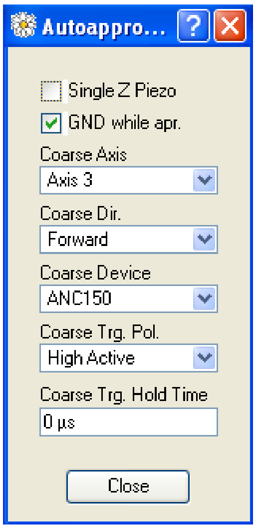
Before starting the auto approach select the ADC1 as 'Actual Value' in the 'Feedback' box. Furthermore choose the following parameters :

Actual Value: SPM ADC1
Z out: 0nm
Z limit min: 0nm
Z limit max: maximum z scanner stroke (see data sheet at the end of the manual).
P: 1-5 m
I: 10-50 Hz
Setpoint: Working point WP as indicated in Figure 18.
Inv. Polarity: not checked (signal ADC1 is expected to increase with increasing Z)



In the 'Coarse' tab one can find the 'Auto Approach' box where the following parameters should be entered:

Delay: 10ms
Threshold: a value bigger than $ADC1_{min}$
Stop Cond.: > Threshold
Speed: 2V/s (@RT), 3V/s (@LT)
Steps/Apr.: See Data Sheet at the end of this section
Apr. Mode.: When the stop condition is met, you can either tell the ASC 500 to remain within the feedback mode or to immediately retract. To choose 'Retract' is of course the safer procedure.
Target Mode: Retract

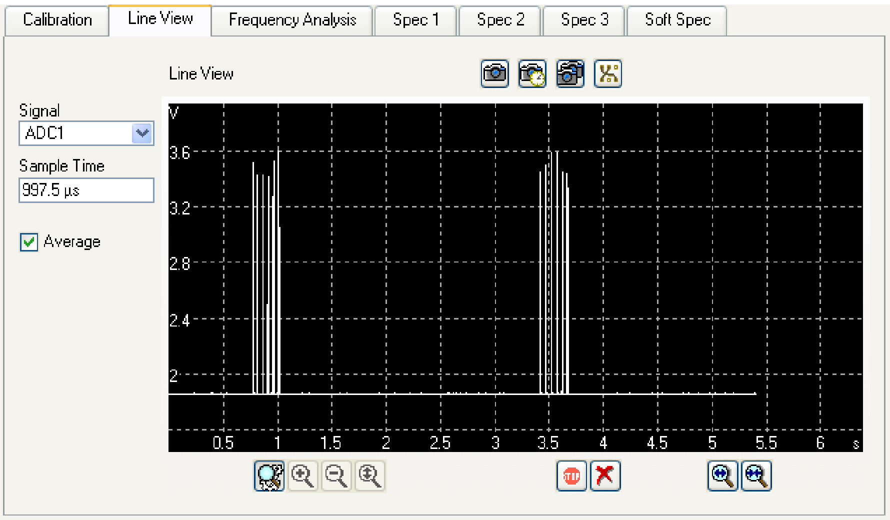


Press the 'Autoapproach Details' where the following parameters should be entered:

Coarse Axis:	Axis 3
Coarse Dir.:	Forward
Coarse Device:	ANC150
Coarse Trig. Pol.:	High Active
Coarse Trig. Hold Time:	1ms

Check the 'Axis 3' settings to have reasonable values for the coarse positioning, for example:

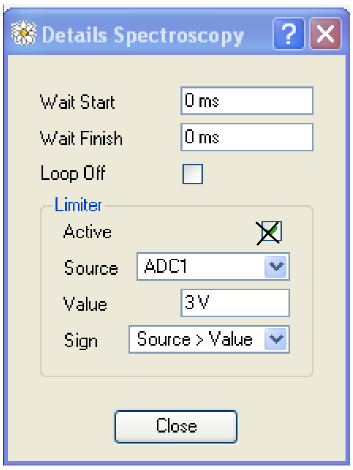
Frequency:	200-1000Hz
Amplitude:	30V (@RT), 50V(@LT)



3. Z Spectroscopy:

After the 'Auto Approach', run a so-called 'z Spectroscopy'. This is a spectroscopy where the feedback parameter is monitored while the z scanner moves the sample surface towards the tip until some breakdown condition is reached. To perform this spectroscopy, we recommend to enter the following values/ranges into one of the three 'Spec' windows (Spec1,Spec2 or Spec3):

Start: 0pm
End: maximum z scanner stroke. See data sheet at the end of this section.
Data Points: 1000
Fwd/Bkwd: not checked
Data Point Avg. Time: 2-10 ms
Delay Per Data Point: 0 s

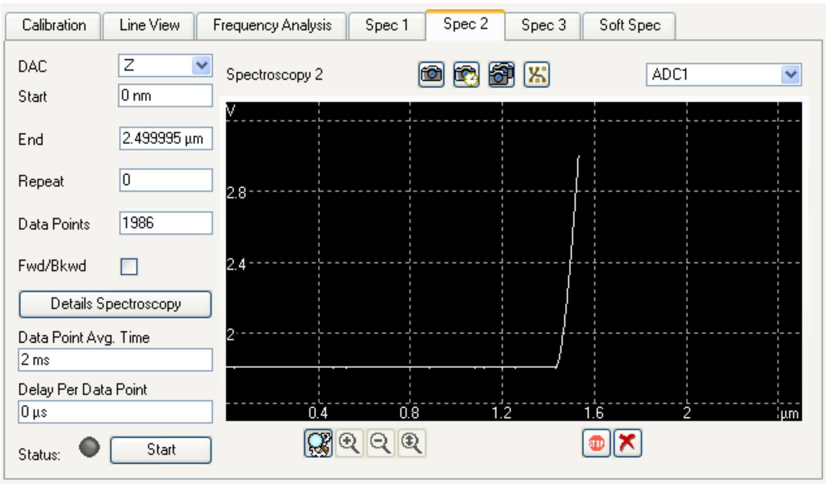


Now open the 'Details Spectroscopy' sub menu and enter the following parameters:

Limiter: (Stops the z spectroscopy before the a certain force onto the tip is exceeded)

Active: checked
Source: ADC1
Value: higher than the interference minimum
Sign: Source > Value

Start the z Spectroscopy

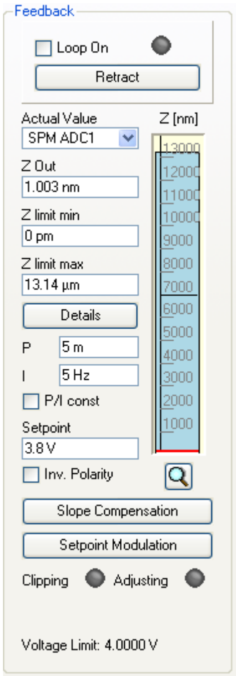


The 'jump-to-contact (JTC)', will now be seen in a rather sharp 'jump-like' increase of the interference signal (ADC1).

Note, that it is suggestive (for the validity of the scale calibration) to keep the feedback in the middle of the z scanner range. Move manually upwards (or downwards) with the z positioner step by step to adjust this position.

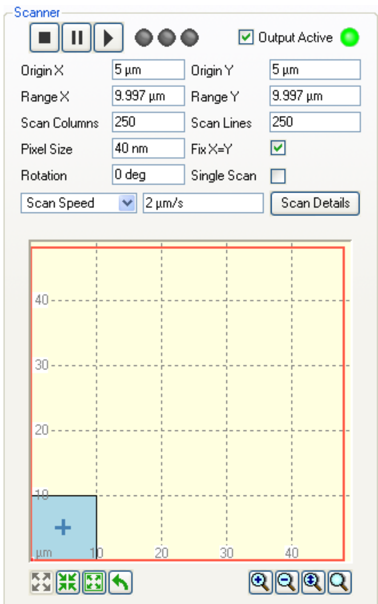
During the contact-mode measurement, the fiber-tip distance is tried to be kept constant. This means that the deflection of the cantilever and therefore the force between tip and sample is kept constant by the P/I-feedback loop.

V.2.d. Starting a scan



To finally start the scan check the following parameters in the feedback box as already shown before:

Actual Value: SPM ADC1
Z limit min: 0nm
Z limit max: maximum z scanner stroke (see data sheet at the end of the manual).
P: 1-5 m
I: 10-50 Hz
Setpoint: Working point WP as described in the two subsections above
Inv. Polarity: not checked for contact mode(signal ADC1 is expected to increase with increasing Z)
checked for non-contact mode(signal ADC1 is expected to decrease with increasing Z)



In the 'Scanner' box select the scan area (blue shaded area) of interest and choose a slow 'Scan Speed' (< 3 µm/s) to start the scan.

Now the feedback can be activated by checking the 'Loop On' box. The z piezo will expand and the tip will go into contact.

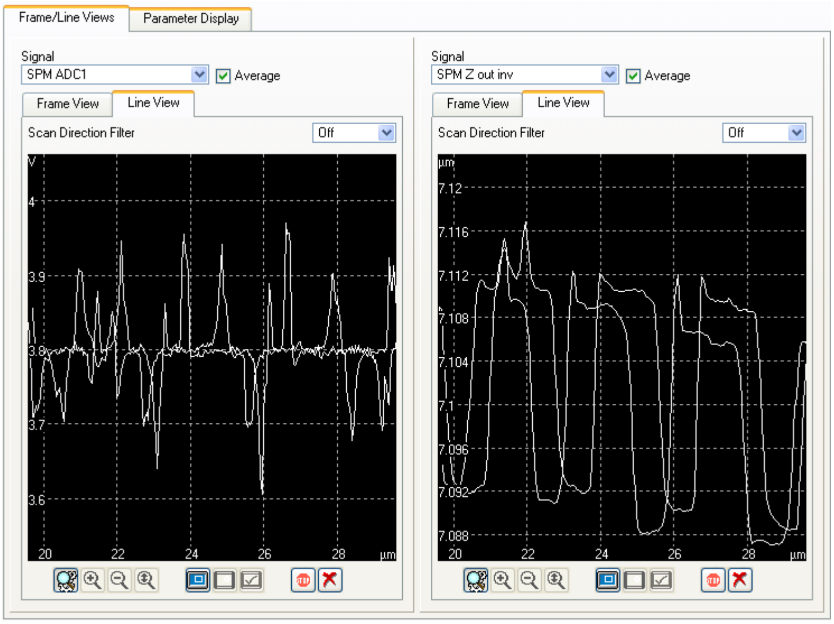
Before you start the scan, select the appropriate signals in the 'Frame/Line View' tab:

Normally, in contact mode 'ADC1' (error signal) and the 'SPM Z out inv' (topography signal) is recorded.

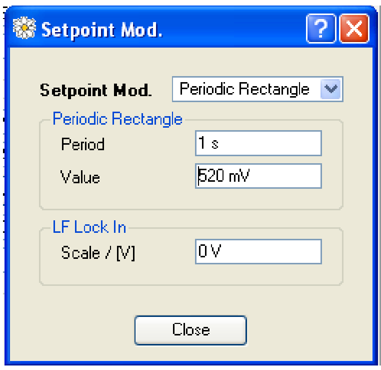
In non-contact mode 'AFM Aosc' (error signal) and the 'SPM Z out inv'

(topography signal) is recorded.

During scanning, the P and I parameters of the feedback loop have to be adjusted according to the chosen scan speed and the surface roughness. At the same scan speed, for example, a rougher surface will need a faster feedback loop compared to a smooth surface.



In addition, the sample tilt can be compensated by setting a proper slope compensation value for x and y. This feature can also be found in the 'Feedback' box.



Choosing P, I parameters:

To find reasonable start values for the P, I parameters, a 'Setpoint Modulation' can be used. This feature is found in the 'Feedback' box. As parameters choose:

Setpoint Mod.:	Periodic Rectangle
Period:	1s
Value:	95% or 105% of current setpoint

Start the feedback. The setpoint modulation will simulate a step-like pattern on the sample surface. You can choose P and I for minimum response time without overshoot (increase P and/or I) or lower noise.

Now, a scan may be started.

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