Park systems NX-Hivac AFM

Version: October 28, 2024

Park NX-Hivac enables material research that requires high accuracy and high resolution measurements in a vacuum environment. High vacuum measurement offers higher accuracy, better repeatability, and less tip and sample damage than ambient or dry N_2 conditions.

Park NX-Hivac https://youtu.be/z6W1e8Uq4oQ
Park SmartScan https://youtu.be/3jzHUaOlr5U
Park SmartAnalysis https://youtu.be/BD-aAHyd5TI

NX-Hivac System Specifications

Vacuum chamber size	300×420×320 mm
Vacuum level	$<10^{-5}$ Torr
X-Y scanner	100×100 μm
Z scanner	15 μm
Optics	5M 10× lens
Single sample size	50×50 mm
Multiple sample size	10×10 mm, 4 pieces
Sample thickness	up to 20 mm

Applications

(1) Surface imaging and topography

Contact Mode https://youtu.be/4bgb5nD2JOU
Non-Contact Mode https://youtu.be/J3YB_hemZ4w
Tapping Mode https://youtu.be/En1S-HX4o-M
PinPoint Mode https://youtu.be/z5F8-18of_I
Force Modulation Mode https://youtu.be/QcJTrha8atM

(2) Electrical properties

Conductive Probe AFM (CAFM)
https://youtu.be/46_2QjqEq6w
Electrostatic Force Microscopy (EFM)
https://youtu.be/7UVSD2e2rjE
Kelvin Probe Force Microscopy (SKPM)
https://youtu.be/PTrIiKE8peI
Dynamic Contact EFM (DC-EFM)
https://youtu.be/hAe9nwOX_eg
Piezoelectric Force Microscopy (PFM)
https://youtu.be/jqpS41y4jCE

(3) Nanomechanical properties

Force Modulation Microscopy (FMM)
https://youtu.be/QcJTrha8atM
Lateral Force Microscopy (LFM)
https://youtu.be/UKK9XoeeWBA
Nanoindentation
https://youtu.be/CKHit_kzFE0
https://youtu.be/MZb8COf7Kdg
Force Distance Spectroscopy (FDS)
https://youtu.be/KKYPj3FUW5k
https://youtu.be/ns_NHCFpQdQ
Force Volume imaging
https://youtu.be/N2kC2n9d_Kg

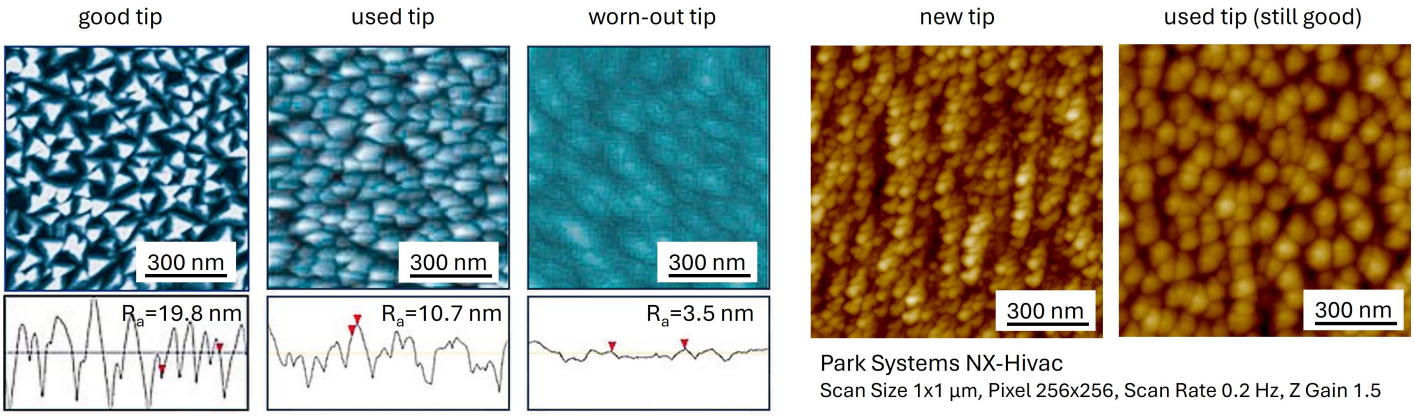
(4) Nanolithography

Achieved by mechanically deforming the sample surface (scratch the sample surface with hard tips), or by changing of the surface's chemical properties (apply a bias between the tip and the surface). Objects can be drawn (bitmap images) in the software and printed onto the sample surface.
https://youtu.be/6J_v-C7Dozw

Park AFM Innovative technology https://youtu.be/wiFCYFrXkek

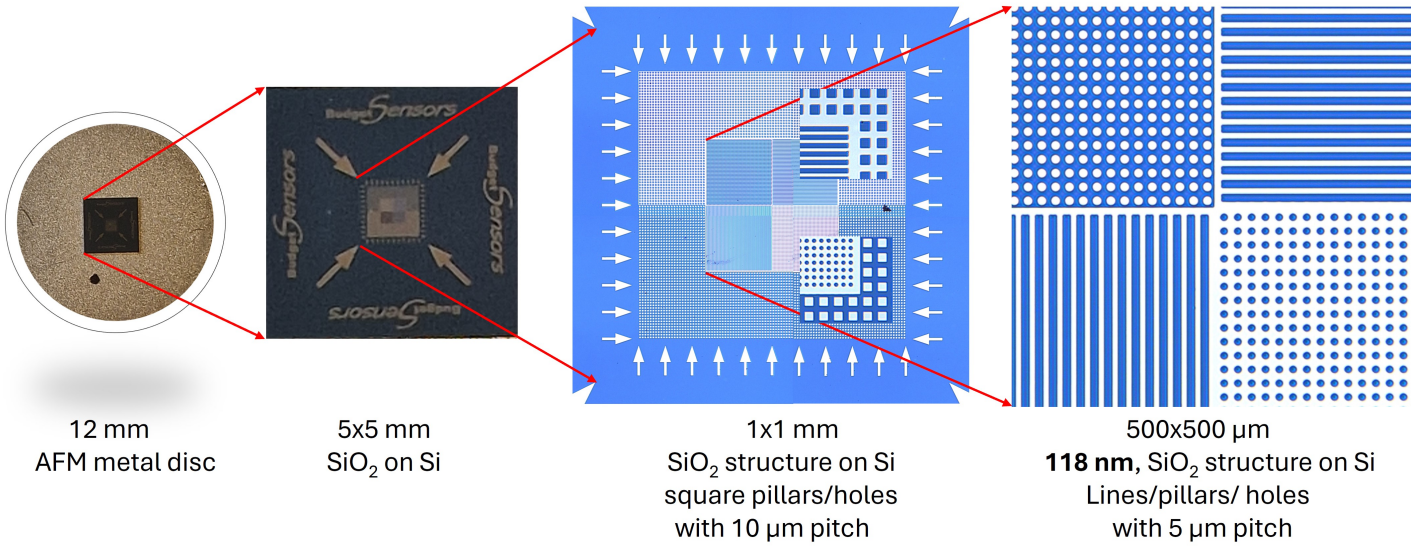
Checking the AFM tip condition "TipCheck sample"

When imaging a sample surface by AFM, it may be difficult to know whether the surface is imaged accurately or if it is affected by a blunt or broken tip. Blunt or broken tips will dramatically distort measurements like surface roughness or feature dimensions. To be certain that a proper AFM tip is used, they must be exchanged regularly (expensive) or checked by SEM imaging (time consuming). TipCheck sample is available at the lab for quick and convenient determination of the AFM tip condition, if the tip is still good, starts showing wear or is blunt or broken. A single scan is often enough to clearly show the condition of the AFM tip; with respect to tip parameters like apex, shape and sharpness.
The TipCheck sample consists of an extremely wear-resistant thin film coating deposited on a silicon chip. The thin film shows a granular, sharply peaked nanostructure which is ideal for reverse imaging of the AFM probe tip apex.
The size of the TipCheck sample is 5×5 mm mounted with electrically conductive epoxy resin on a 12 mm metal AFM disc. Product no. 34-020001, provider Micro to Nano BV.
Keep in mind that the appearance of the structure on the TipCheck depends on the specific AFM system, the specific scan parameters and the specific AFM probe. Different combinations of theses parameters will result in different visualizations of the TipChech surface.



The AFM height calibration standard sample

The **HS-100MG** is mainly a height calibration standard with a **118 nm** calibrated height. It consists of silicon dioxide structures on a 5×5 mm silicon chip mounted on a 12 mm metal AFM disc using electrically conductive epoxy resin.
The calibration area is located in the center of the silicon chip and consists of a larger square of 1×1 mm with square pillars and holes with a 10 μm pitch. In the center of this square resides a smaller square area of 500×500 μm with lines, circular pillars and holes with a 5 μm pitch.
This design also allows for X/Y-axis calibration for bigger scanners in the 10-40 μm range.

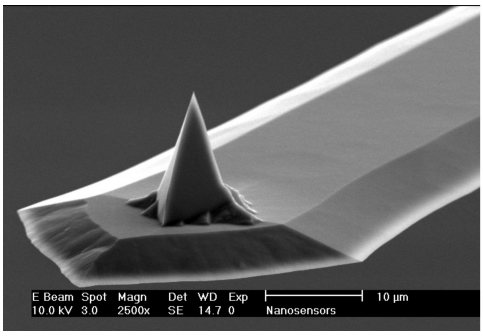


AFM Probe Selection Guide

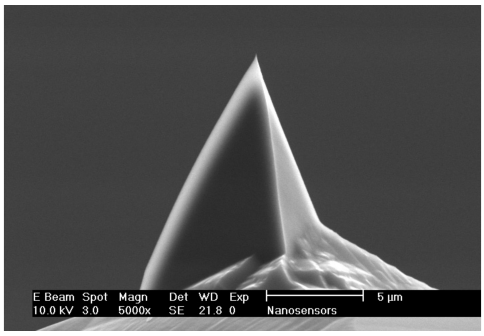
Proper selection of a probe for an atomic force microscope (AFM) is **important** to obtain good quality sample surface images. In general, an AFM probe consists of a silicon chip, a cantilever (Si or Si_3N_4) hanging from the chip, and a tip (Si or Si_3N_4) attaching at the end of the cantilever. AFM probes come in a variety of materials, shapes (geometry), **stiffnesses (spring constants)**, **resonance frequencies** and **Q-factors**. Probe selection depends on the material and application.
The probe variations come mainly from the variations of cantilever types. Typically, the backside of an AFM cantilever has a metallic coating such as gold or aluminum to increase reflectivity. Depending on the **properties of a cantilever**, it can be classified as a non-contact or a contact mode cantilever.

AFM Tips Catalog

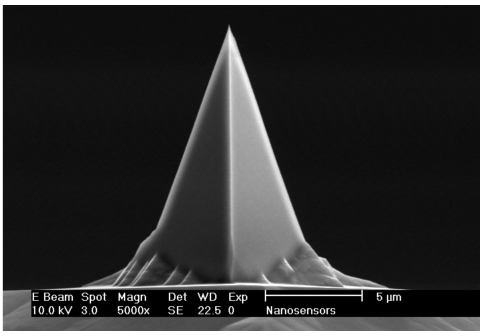
`https://www.nanosensors.com/afm-tips-catalog?gad_source=1&gclid=EAIaIQobChMImOWkmN7PiAMV306RBR14Ug9YEAMYASAAEgIzofD_BwE`



PointProbe® Plus 3D view (SEM image)



PointProbe® Plus side view (SEM image)



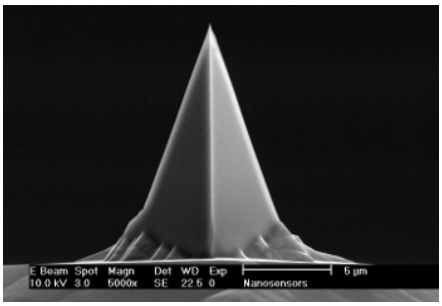
PointProbe® Plus front view (SEM image)

NON-CONTACT/TAPPING MODE

For non-contact mode, it is recommend that you use a probe with a higher resonance frequency (∼300 kHz) and higher spring constant (∼20 N/m). The reason is that when scanning in non-contact mode under ambient conditions, the tip often traps moisture, creating a contaminated layer on the sample. This happens more often when using a cantilever with a low spring constant.
Probe article number **PPP-NCHR**, standard Tapping/Non-contact mode AFM probe, Manufacturer: NANOSENSORS, Coating: 30 nm Reflective Aluminum, AFM tip shape: Standard.

Product Screencast NANOSENSORS™ PointProbe® Plus `https://youtu.be/oo-NFxKOko8`

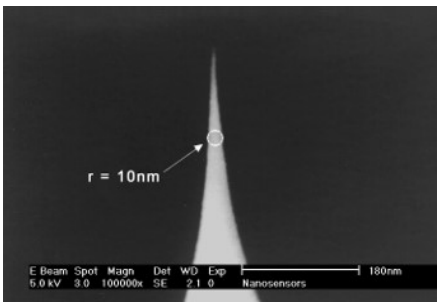
PPP-NCHR			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	125×30×4 μm	Tip height	10-15 μm
Resonance frequency	330 kHz	Tip radius	<10 nm
Spring constant	42 N/m	Material	Silicon (Si)
Material	Silicon (Si)	Price	**700 SEK**



Probe article number **SSS-NCHR**, **SuperSharp Tapping/Non-contact mode** AFM probe, Manufacturer: NANOSENSORS, Coating: 30 nm Reflective Aluminum, AFM tip shape: Standard.

Product screencast NANOSENSORS™ SuperSharpSilicon `https://youtu.be/9CsBdE31jKY`

SSS-NCHR			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	125×30×4 μm	Tip height	10-15 μm
Resonance frequency	330 kHz	Tip radius	<2 nm
Spring constant	42 N/m	Material	Silicon (Si)
Material	Silicon (Si)	Price	**1400 SEK**

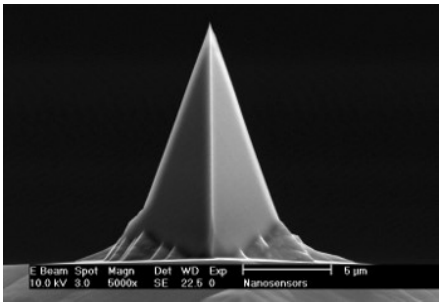


CONTACT MODE

For contact mode, a soft cantilever with low spring constant (∼1 N/m) is required to determine the small force between the sample surface and the tip. A hard or stiff cantilever will cause the tip to exert high forces to the surface, which can damage the AFM tip, the sample surface, or both.
Probe article number **PPP-CONTSCR**, Contact Mode AFM Probe with Short AFM Cantilever, Manufacturer: NANOSENSORS, Coating: 30 nm Reflective Aluminum, AFM tip shape: Standard.

Product Screencast NANOSENSORS™ PointProbe® Plus https://youtu.be/oo-NFxKOko8

PPP-CONTSCR			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	225×48×1 μm	Tip height	10 μm
Resonance frequency	25 kHz	Tip radius	<10 nm
Spring constant	0.2 N/m	Material	Silicon (Si)
Material	Silicon (Si)	Price	**700 SEK**

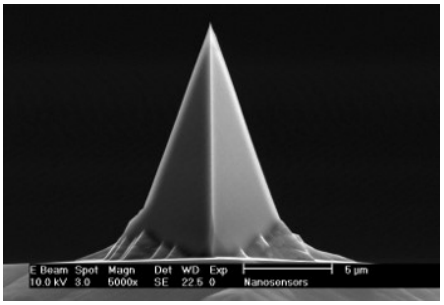


ELECTRICAL CONDUCTIVITY MODE

The electrical properties of a sample surface can be measured using the advanced mode with a conductive AFM cantilever. In order to measure electrical properties properly, the characteristics of the AFM probe such as tip shape/radius, cantilever geometry and coating materials has to be considered.
Probe article number **PPP-EFM**, Electrical Force Modulation AFM Probe, Manufacturer: NANOSENSORS, Coating: Electrically Conductive, AFM tip shape: Standard. The PtIr5 coating is an approximately 25 nm thick double layer of chromium and platinum iridium5 on both sides of the AFM cantilever.

Product Screencast NANOSENSORS™ PointProbe® Plus https://youtu.be/oo-NFxKOko8

PPP-EFM			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	225×28×3 μm	Tip height	10-15 μm
Resonance frequency	75 kHz	Tip radius	<25 nm
Spring constant	2.8 N/m	Material	Silicon (Si)
Material	Silicon (Si)	Price	**800 SEK**



PinPoint NANOMECHANICAL MODE (Force-Distance Spectroscopy)

The electrical properties of a sample surface can be measured using the advanced mode with a conductive AFM cantilever. In order to measure To measure nanomechanical properties of surfaces, Force distance (FD) spectroscopy is a straightforward and reliable technique to quantitatively study nanomechanical properties such as Young's modulus and adhesion energy, adhesion force, stiffness on a variety of samples. In FD spectroscopy, the cantilever is used as a force sensor.

Force Distance Spectroscopy https://youtu.be/KKYPj3FUW5k
PinPoint™ Nanomechanical Mode https://youtu.be/FJXCBzs2454
https://www.parksystems.com/index.php/park-spm-modes/force-measurement/244-force-distance-spectroscopy

Recommended cantilevers depending on Young's modulus of the sample				
Young's modulus (E) of sample	**Cantilever type**	**Spring constant**	**Cantilever dimensions**	**Tip radius/hight**
1 MPa < E < 20 (ex. PDMS) , soft/flexible materials	PPP-CONTSCR	0.2 N/m	225×48×1 μm	<10 nm , 10 μm
10 MPa < E < 2000 (ex. LDPE)	PPP-FMR	2.8 N/m	225×28×3 μm	<10 nm , 10-15 μm
1000 MPa < E < 5000 (ex. PS) , hard materials	OMCL-AC160TS	26 N/m	160×40×3.7 μm	<10 nm , 14 μm

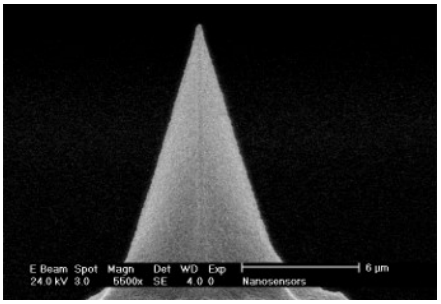
The Young's modulus (E) is a property of the material that tells us how easily it can stretch and deform. Need to consider both the spring constant of cantilever (k_c) and the stiffness of the sample (k_s) when selecting a probe.

LITHOGRAPHY MODE

Probe article number **CDT-CONTR**, Diamond Coated, Conductive Contact Mode AFM Probe, Manufacturer: NANOSENSORS, Coating: Diamond, Conductive Diamond, AFM tip shape: Standard.

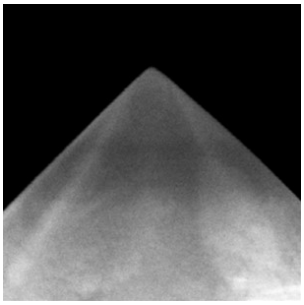
Product screencast NANOSENSORS™ Diamond Coated PointProbe Plus Silicon AFM Probes https://youtu.be/WNAlyBaomTs

CDT-CONTR			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	525×50×2 μm	Tip height	10-15 μm
Resonance frequency	20 kHz	Tip radius	<10 nm
Spring constant	0.5 N/m	Material	Silicon (Si)
Material	Silicon (Si)	Price	**2000 SEK**



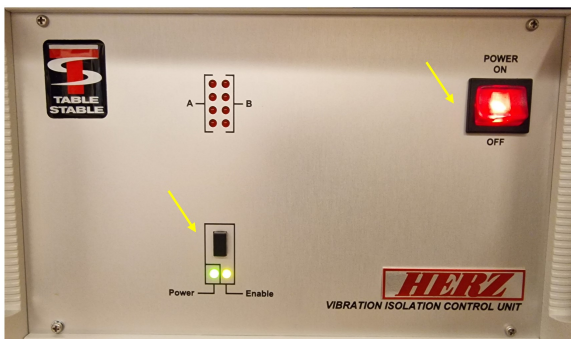
Probe article number **NM-RC**, Diamond AFM Probe, Manufacturer: BRUKER, Conductive Diamond, AFM tip shape: Standard.
This tip is specifically designed for high mechanical loads and scratch testing applications. By using wear-resistant diamond and a broad cone angle the contact size is well characterized and stays constant during repeated mechanical measurements. This probe has demonstrated highly repeatable deep (∼100 nm) indentations into materials such as fused silica and can image the indents at high resolution in-situ using the same probe. A gold reflex coating deposited on the detector side of the cantilever to enhance the reflectance of the laser beam.
Nanomechanical modes: Tomography, nanoscratching, and nanoindentation, and their combination with PeakForce QNM, FASTForce Volume, or contact resonance.

NM-RC			
Cantilever		**Tip**	
Shape	Rectangular	Shape	Tetrahedral
Length×Width×Thickness	125×30×– μm	Tip height	12.5 μm
Resonance frequency	750 kHz	Tip radius	<10-15 nm
Spring constant	350 N/m	Material	Diamond
Material	Silicon (Si)	Price	**7000 SEK**

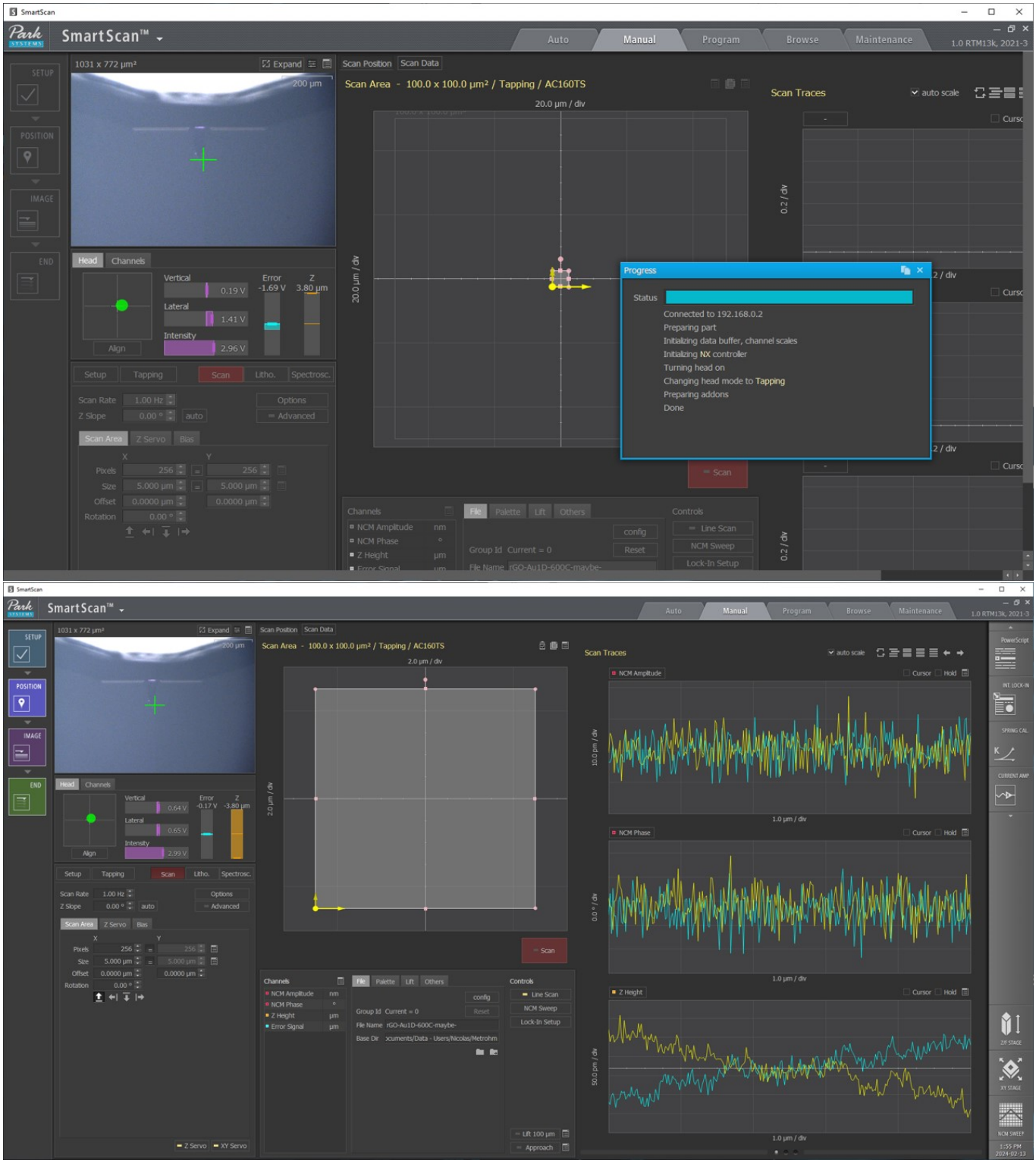


1. Startup device

1. **Start AFM** Controller.
2. **Start HERZ** vibration isolation control unit.
   HERZ is a part of the vibration isolation system that blocks the effect of floor vibration to the AFM. On the front panel of the HERZ controller, there are 8 red LEDs arranged in two groups of 4. These LEDs show the status of the system. Each 4-LED group shows the status of the associated isolation units supporting the AFM. When an LED is lighted, it means that there is external vibration and the HERZ is working to cancel it. These red LEDs are off if there are no external vibration.
   Also on the front panel, there is a yellow isolation indicator LED indicating the isolation condition. Normally when the AFM is isolated from floor vibration, this LED is on. This LED blinks when there is severe external vibration and the AFM is no longer isolated from external vibration.



3. Wait 2-5 minutes until HERZ vibration isolation is done, then start software "**SmartScan**" .



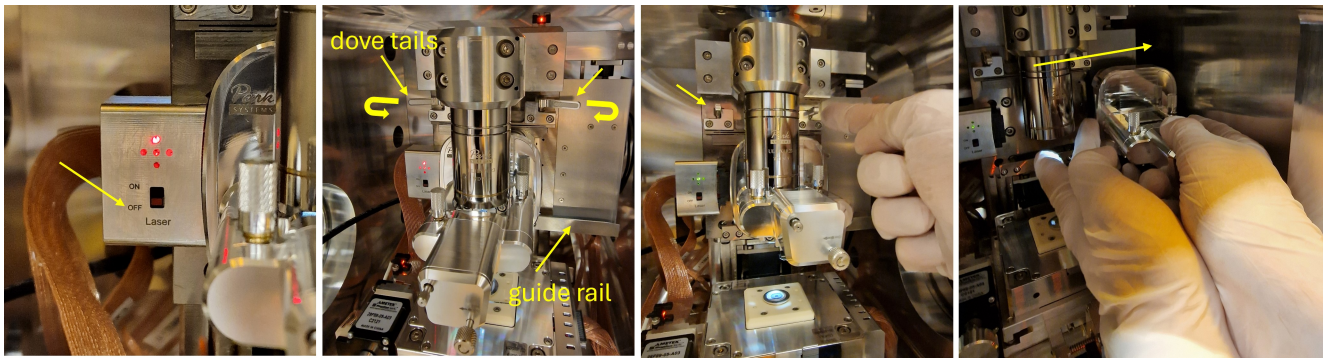
2. Probe mounting and exchange

Only need to exchange the probe when it is damaged, otherwise **go to section 3**.

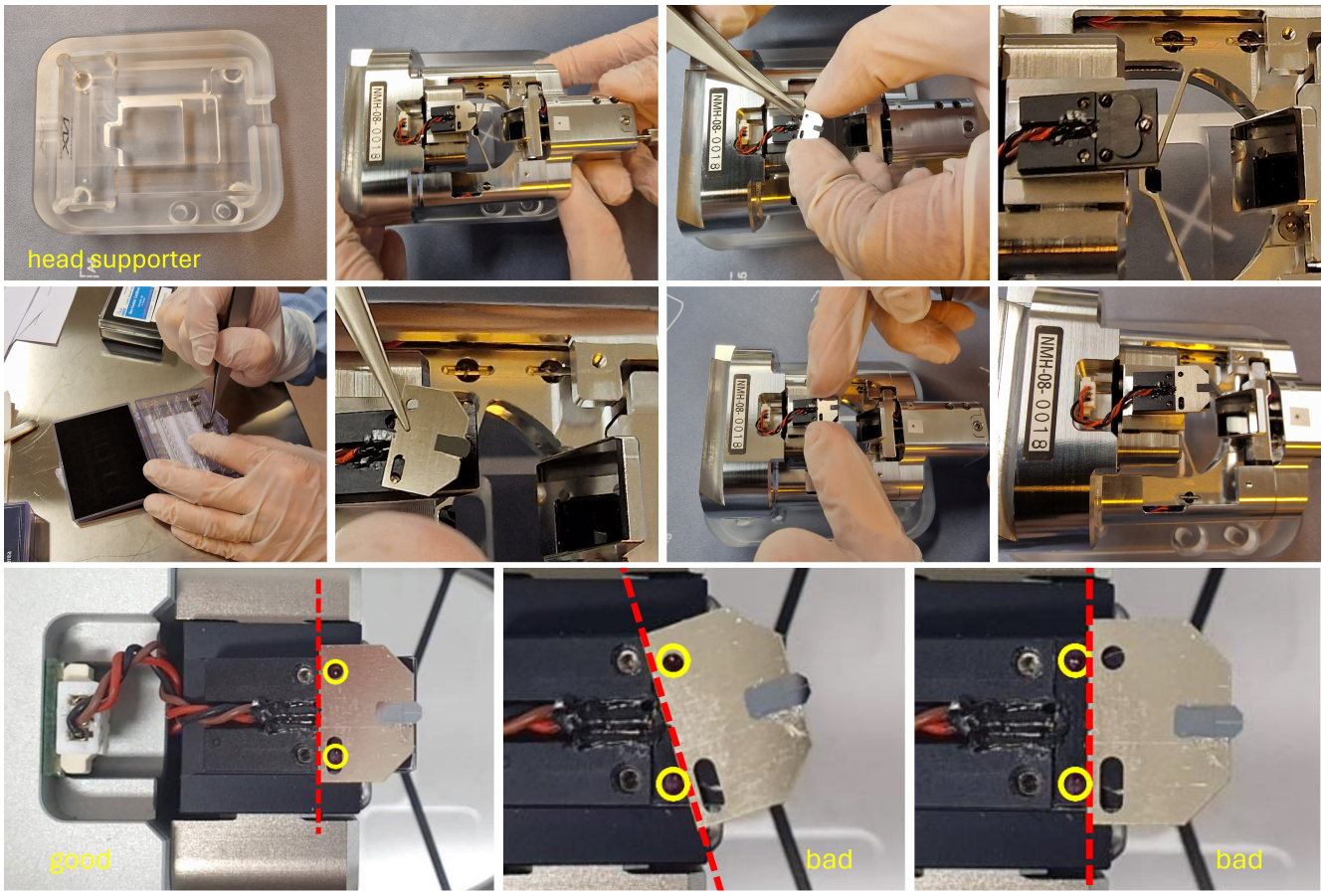
Park Systems provides various types of chip carriers (with pre-mounted and unmounted cantilever) for different measurements. For unmounted cantilever, you need a glue type chip carrier, a cantilever, and an instant adhesive for metal (Cyanoacrylate (superglue) adhesives provided with NX system are recommended). The cantilever then is attached onto the marked area on the glue type chip carrier. More details are given in official NX-Hivac USER'S MANUAL Version 1.6 pages 50-53. This kit is available in the lab.

We use the pre-mounted cantilever on the chip. Exchanging probe means removing the chip from the head and replace it by a new one as described below, where the chip will be held in place by magnets. In this case the beam position will only slightly change when exchange probs, very slight alignment will be needed.

1. Switch off laser.
2. Release the AFM head by pulling the dove tails inwards (to your direction).
3. Very gently slid the AFM head along the guide rail to left of the AFM body. Push the head to the end of the guide rail, do not use force.



4. Mount the head backside down on the head supporter.
5. Carefully take off the cantilever carrier, may use a tweezer, and replace by a new one. Fix it on position as shown in figures. The hole and slot on cantilever carrier should fit in two balls (marked yellow) on probehand.



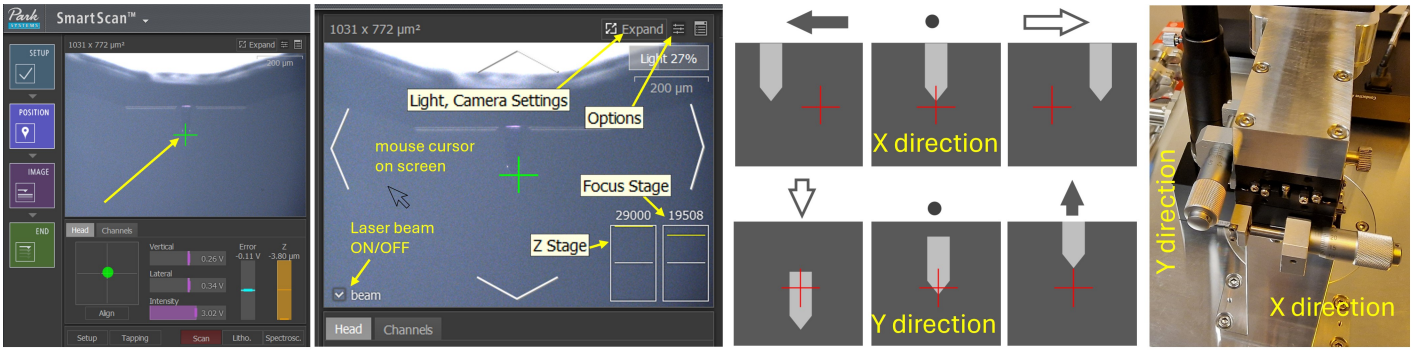
6. Before inserting the AFM head to body, choose a sufficient distance from the sample stage to avoid tip damage by crash.
7. Gently insert the AFM head along the guide rail from the right to left of the AFM body. Push the head to the end of the guide rail to connect the head to AFM body properly.
8. Secure the AFM head by tightening the dove tails backward.
9. Turn On laser.
10. Adjust the vision setting and focus on the cantilever as given in next section (section 3).

How to exchange AFM Tips - Park Systems Easy tip exchange https://youtu.be/MzGes70M8xE

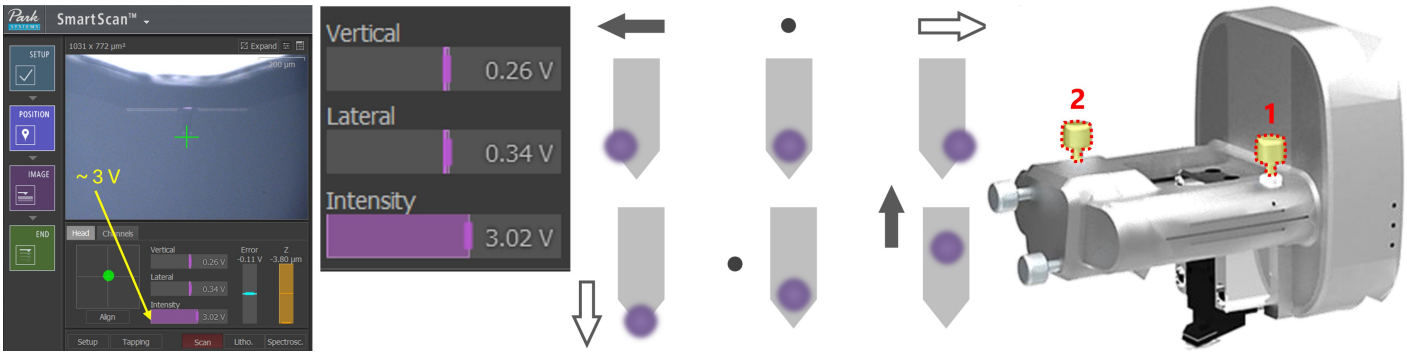
3. Cantilever focus, laser beam (SLD) and PSPD alignments

These alignments are preserved up to 99% from previous use if they are well done or no probe exchanged. Often only very little adjustment may be needed or onthing at all.

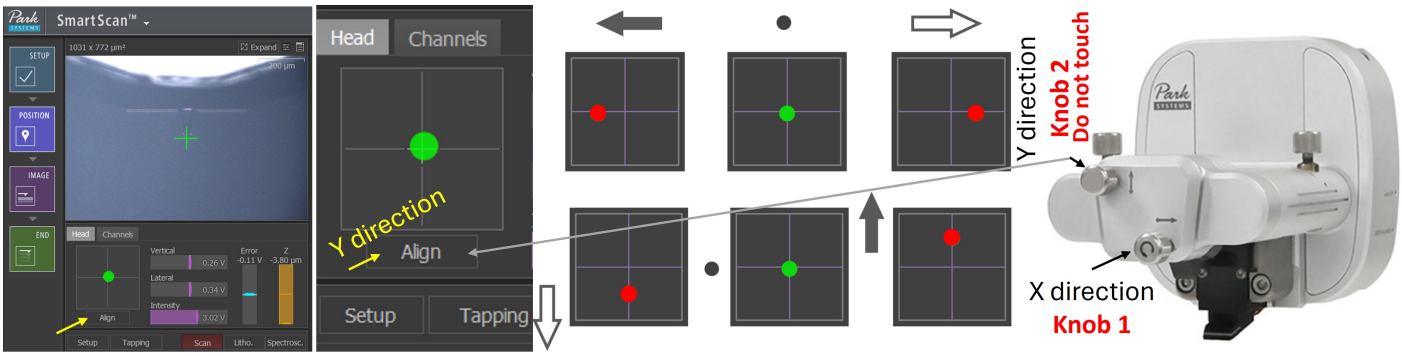
1. **Focusing the optical Microscope and cantilever position alignment (cantilever in vision window)**: This means alignment of the tip position to the green cross position. The Optical microscope is used when positioning the SLD beam onto the cantilever, the green plus in the middle of the screen should located on the sharp edge of the cantilever using X-Y knob. By having the mouse cursor on the viewing screen, arrows on all sides and **Z Stage** , **Focus Stage** will appears. This easily can be utilized for locating regions of interest on the sample surface for measurement, focus can be made on both cantiliver and on the sample surface.
   After the tip position alignment, the sample position moves to the cross position by double clicking the vision window. Thereby, it is easy to bring the tip to the sample position of interest.



2. **Laser beam (SLD beam) alignment on cantilever**: done by using knob 1 and 2 in picture below, your alignment parameter is the intensity which should be about **3.00 V** (between 2.9-3.1 V is also fine). Vertical and Lateral should be around zero or between ±0.5 V.

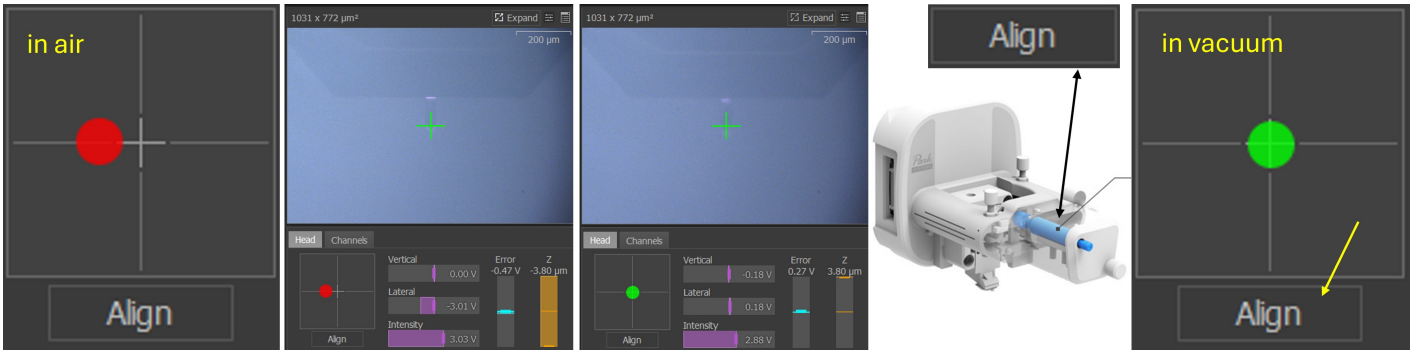


3. **PSPD alignment (center laser on PSPD)**: use Knob 1 to align the beam spot on PSPD in X direction (the spot on horizontal line). **Never touch Knob 2 (motorized knob)**, instead press the **Align** button Align on the screen to automatically align the beam on PSPD in Y direction (vertical direction).
   If the dot is red, the Z stage will not move down as a kind of protection. AFM imaging will not work.
   When no green/red dot on vision means the laser beam is OFF. Turn ON by ticking on left down corner.



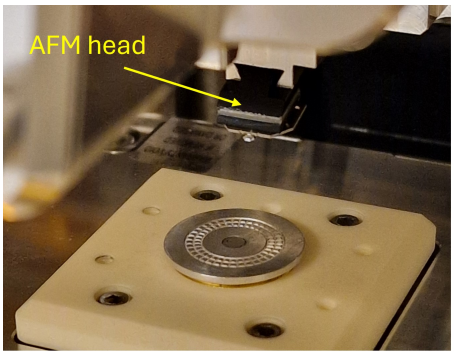
Park AFM - Laser Beam Alignment https://youtu.be/1veGzp1WLWI
Park NX20 Atomic Force Microscope https://youtu.be/PE5GL4eHKTg

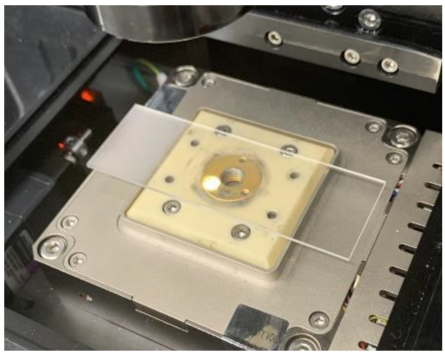
IMPORTANT: **When working in vacuum**, align the PSPD little to the left as in the picture below, it will go the center of the PSPD when operation vacuum is reached. Remember you have no access to the knobs inside the vacuum chamber. You have only access to Align button Align to aling in Y direction.
Making AFM measurement in vacuum is exactly the same as measuring in ambient air, but the vacuum is controlled by different software than the software used for AFM measurements, they are totally independent.

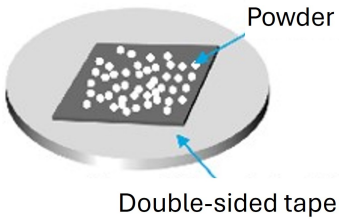


4. Sample loading

For thin films: The AFM has a magnetic sample holder on the XY scanner. Attach the sample to a metal disk and place it onto the magnetic sample holder. It is possible also to place the sample directly on the stage without any additional concern.
For dry powders: Get a double-sided tape (or carbon tape) on a metal disk then drop the sample on the top of the tape, shake the disk to remove the excess power from the surface. Cautions: the sticky surface of the double-sided tape can make it difficult to scan the surface. Besides, the powder can stick to the tip and interrupt the scan.
IMPORTANT: Never touch the AFM head during loading or unloading the sample. Be keerful and keep distance from the AFM head.



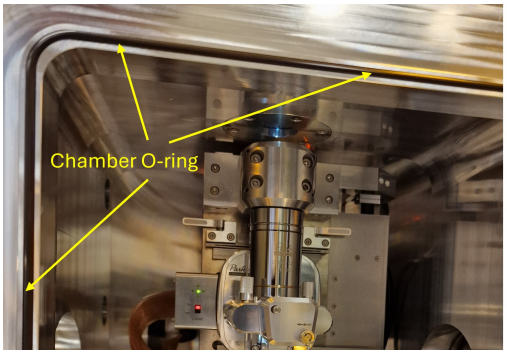


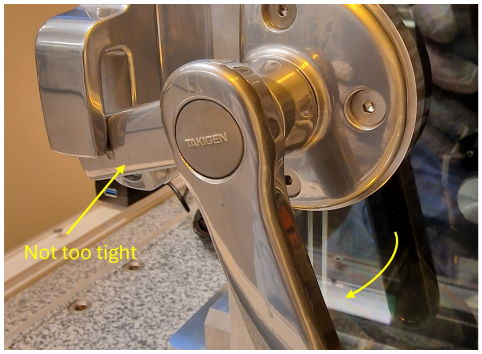


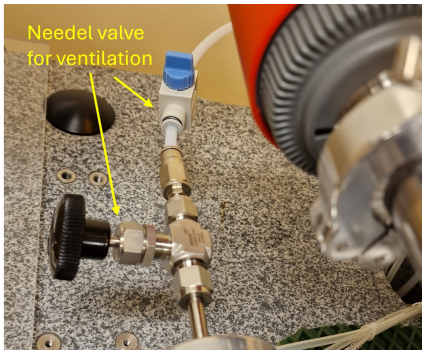
5. Vacuum control software (Vacuum AFM)

These steps are only needed when you desire AFM measurement in vacuum, otherwise go to next section.

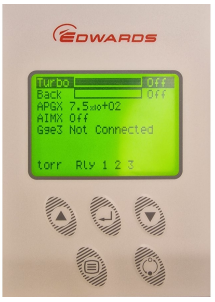
1. Place your sample in position as in section 4, and perform all the alignments described in section 3 (the case when working in vacuum).
2. Check chamber O-ring (should be clean and in position), close the chamber door not too tight, finally check both ventilation valves (needle valve), the valve should be closed.



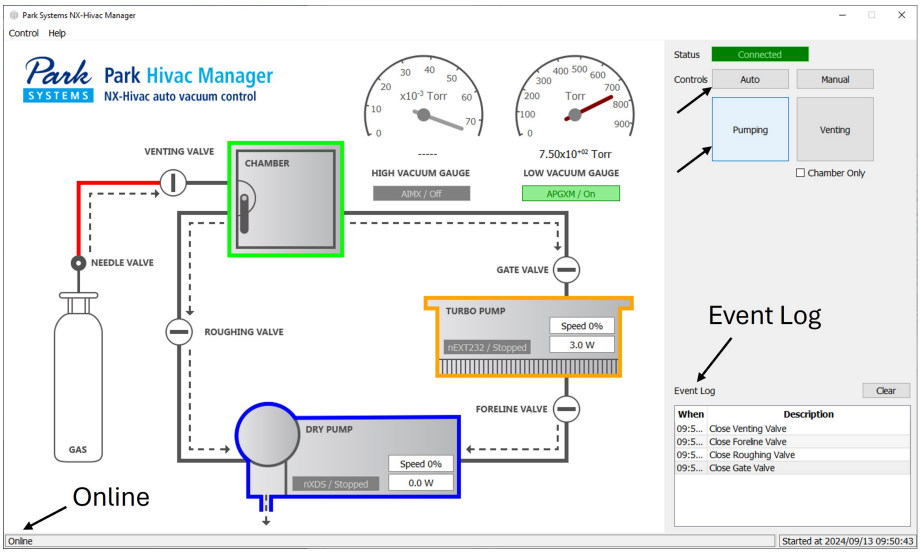


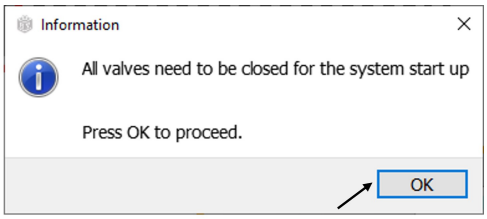


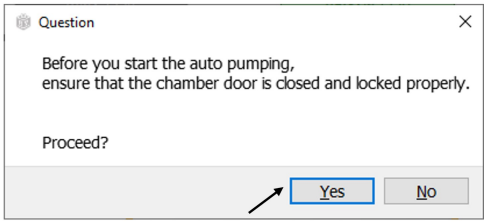
3. Start Park Systems Vacuum Controller, and Edwards vacuum sensor controller (ON/OFF button is located on back side of the device).



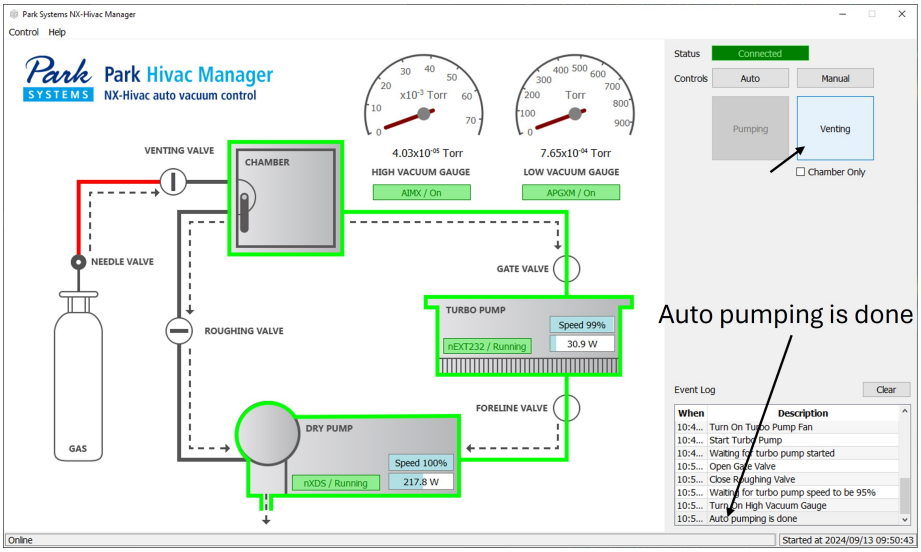
4. Start software NX-Hivac . You must see a colourful window and online on the left-down corner. If everything is grey and Offline, it means some hardware is not ON.
While there are not many parameters to control manually, always run the vacuum in Auto mode. Click on **Auto** then click on **Pumping**. Some small windows will pop-up, just confirm by **OK** and **YES**. Wait 10-15 minutes until the vacuum is about 10^{-5} Torr and you will see the message "**auto pumping is done**" in **Event Log**.

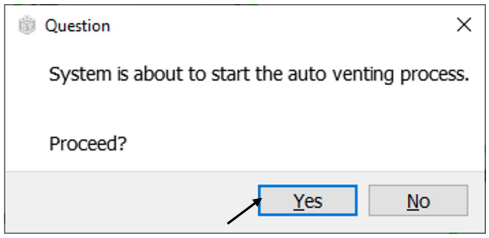


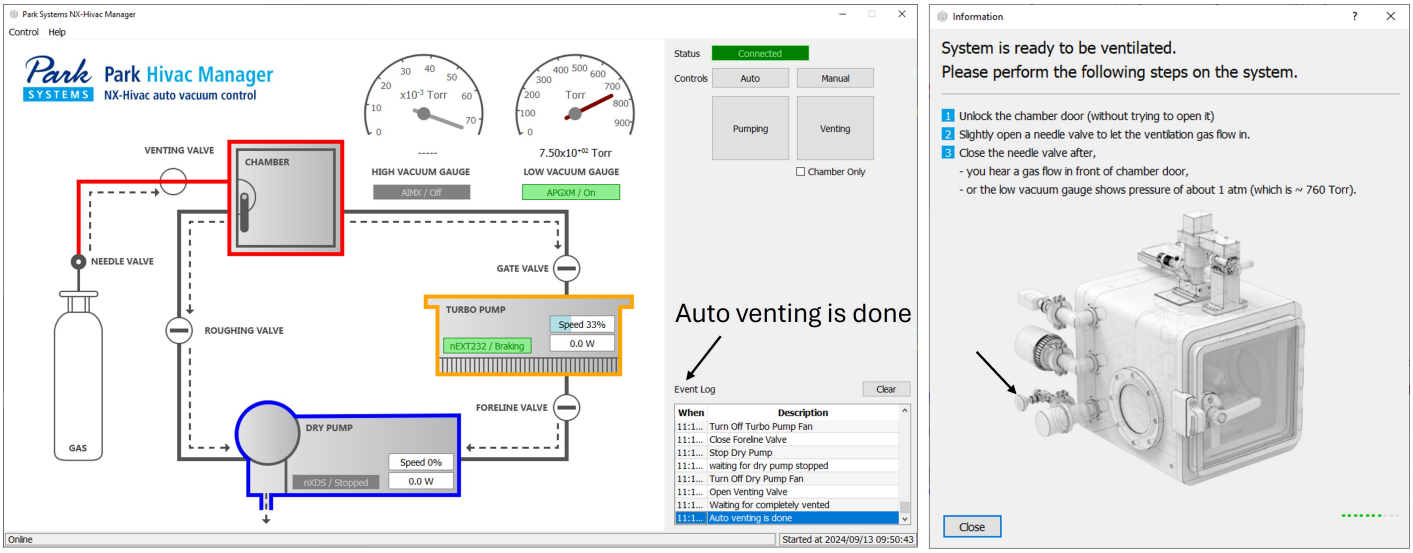




5. When AFM measurements are done, click on **Venting**, confirm by **YES**, and follow the steps in Information window. 1) Unlock the chamber door (without trying to open it), 2) Slightly open the needle valve to the ventilation gas flow in. 3) Close the vale when you hear the gas flow sound inside the chamber (the low vacuum indicator will show $7.5\times10^{+02}$ Torr.





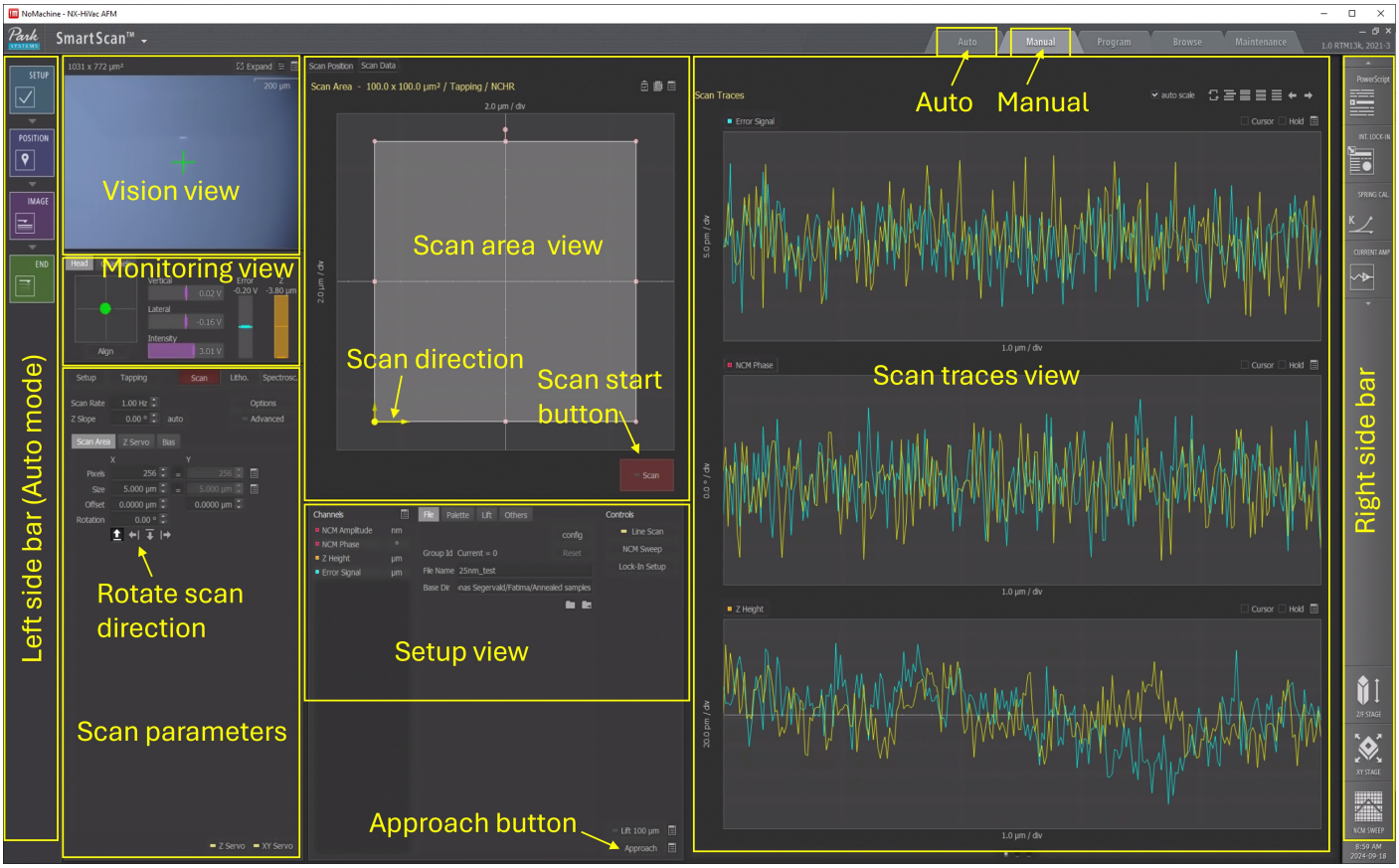


6. When done, take you sample and close NX-Hivac software. Turbo pump speed will automatically go to 0%.

7. Switch OFF all vacuum related hardware (controllers).
   You have the possibility to load the sample using a special container in a glovebox then transfer it to the AFM vacuum chamber without any exposure to air. This option is not described in this manual.

6. Performing AFM Measurement

Correct cantilever type (as described in AFM probe selection guide) should be installed on AFM head before starting the scan. Usually we use **PPP-NCHR** probs passing for **Non-contact** mode (**NCM**). AFM measurements can be done in **Auto** or **Manual** scan mode. For more accurate measurements the Manual scan mode is preferred.

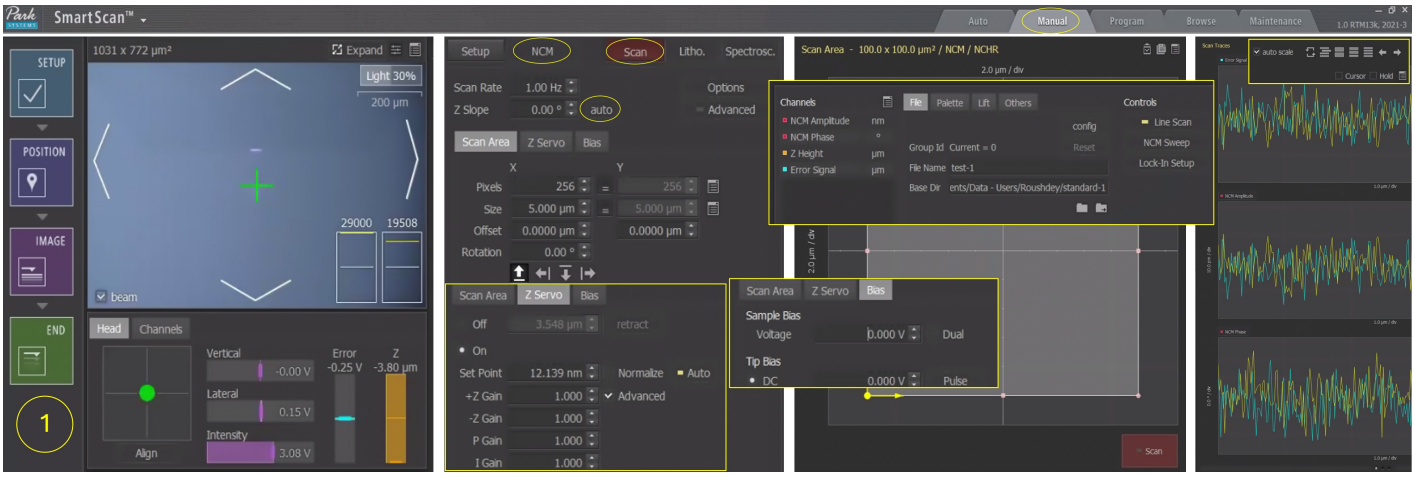


6.1 Manual scan mode

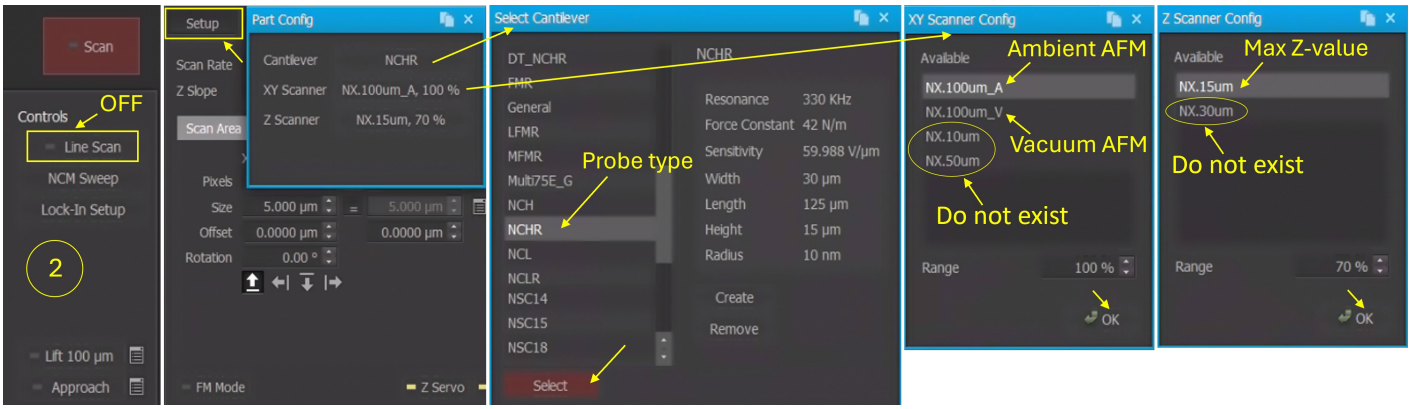
Manual In manual scan all modes are possible.

6.1.1 Non-contact mode (NCM)

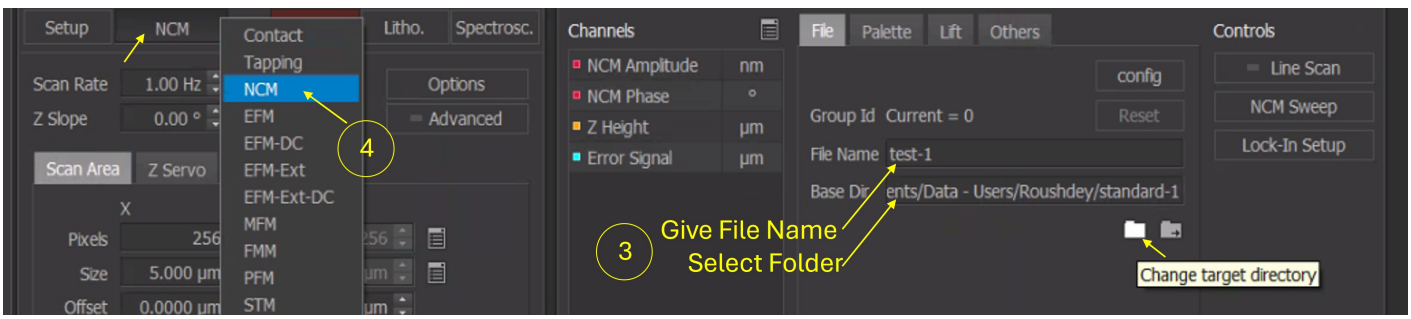
1. **Start up the AFM** system as in **section 1**, and perform all **alignments** as in **section 3**. All expected default features and values are shown below. **SmartScan** is by default on **Manual**, and some features preserved from last use. Check based on figure below.



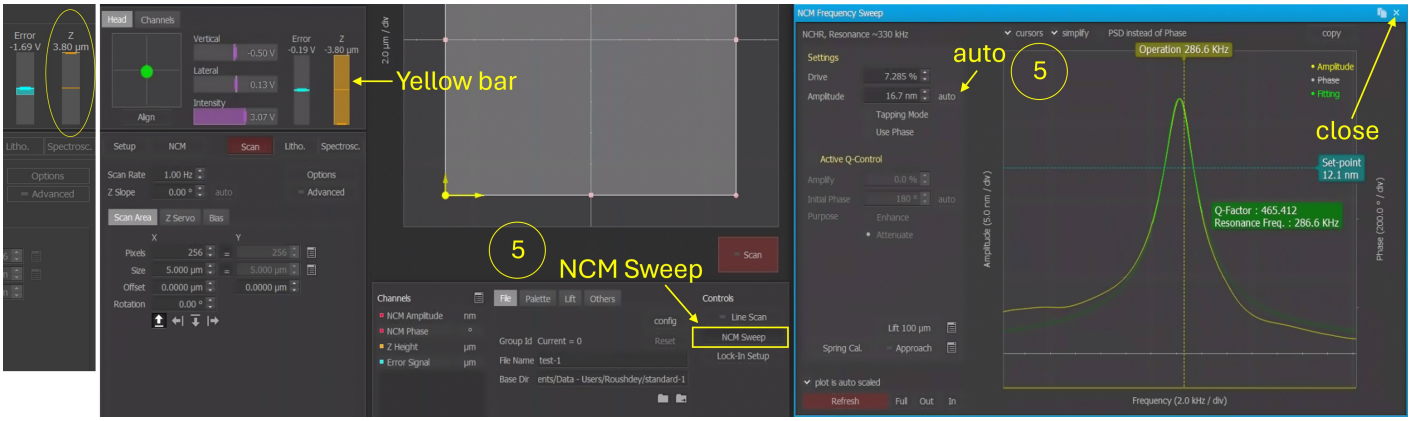
2. Go to **Setup** to select/check the configuration of **Cantilever** type, **scanning in ambient air or vacuum**, and select **Z scanner hight** (only 15 μm is available).
To have access to Setup menu you have to **turn OFF Line Scan**. For working in **vacuum** see **section 5**.



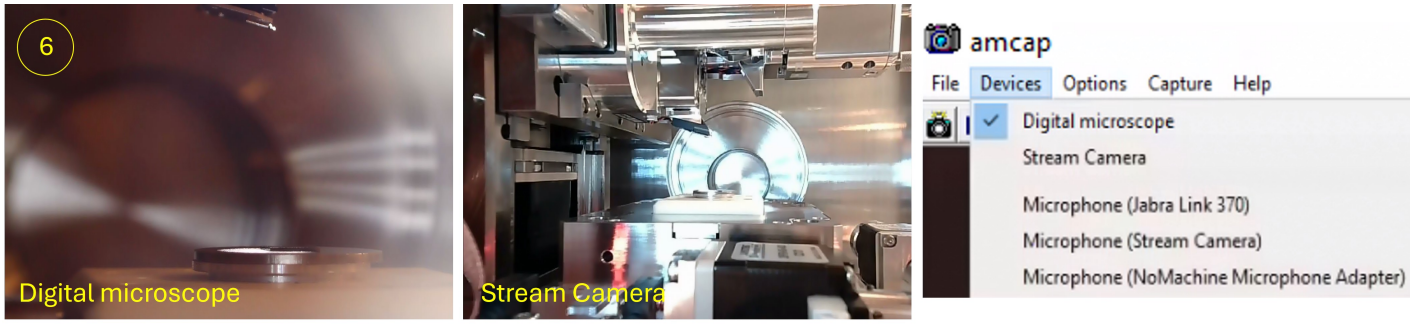
3. Select **folder** where to save your AFM files (by default is saved in the last folder selected by pervious user). Press **Change target directory** to chose folder (or create new) and then give **File Name**. The files are saved on **Documents>Data-Users>** Roushdey> standard-1.
4. When starting **SmartScan** the AFM operation mode will be on what lastly been used (Contact, Tapping, NCM, C-AFM or others).
Select the AFM operation mode by **clicking on Tapping or NCM field and select the desired AFM mode (usually NCM)**. The NCM frequency Sweep widow will pop up but will disappear when fished.



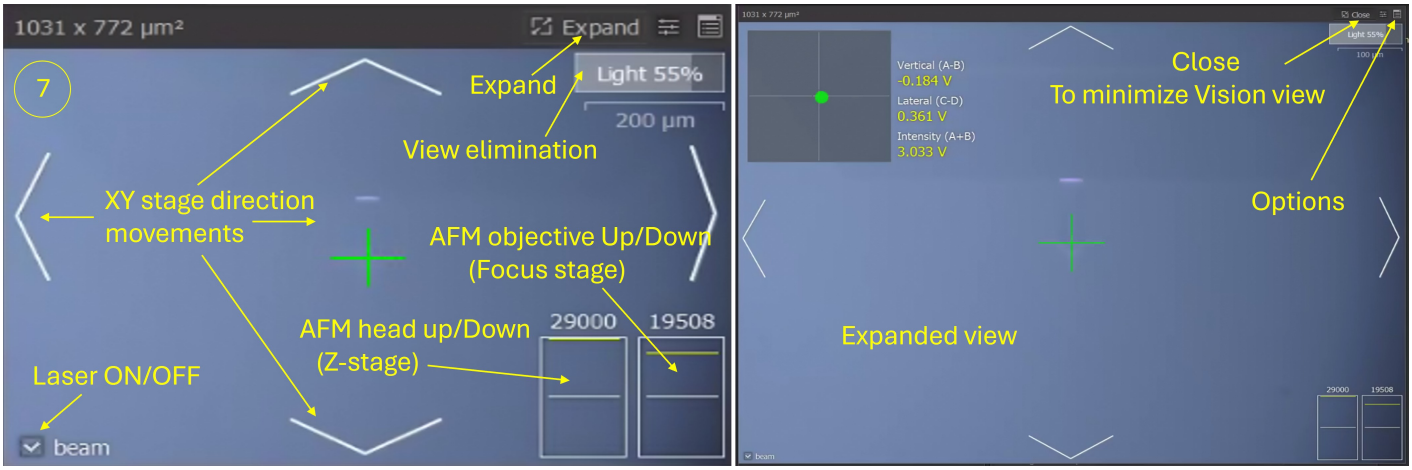
5. Sweep must be done after probe type selection or scanning mode exchange, or in case of the piezo is fully retracted (the orange bar is not fully extended). This to make sure that the probe is oscillating correctly. When probe oscillation is correct, the orange bar will be fully extended. If the orange bar is not on range (not full) means the piezo is fully retracted and Sweep must be done.
   Press **NCM Sweep** and press **auto**, the system will sweep the cantilever until it finds the resonance frequency at around 300 KHz. **Do not change any parameters manually**. Close window when finished.
   During scanning (imagining) the orange bar will fluctuate, this is normal while the probe is vibrating.



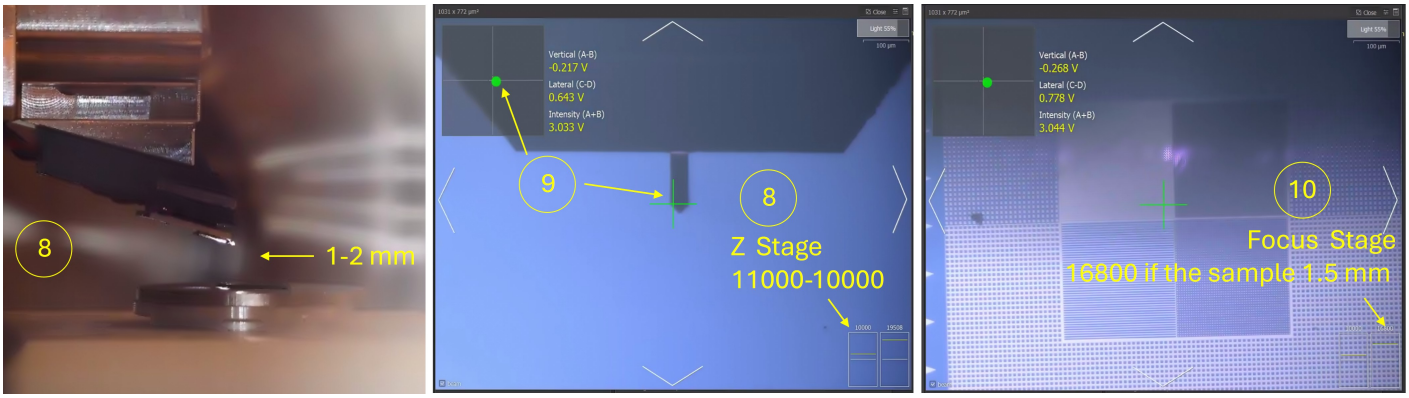
6. **Put your sample on the sample stage** (described in <u>**section 4**</u>). Connect both USB cameras (if not connected) to any USB port to be able to look/inspect sample and AFM head position inside the chamber.
   Start **AMCAP software** amcap, go to **Device** to select the wanted camera.



7. Get both AMCAP software amcap and SmartScan on one the screen, to see and control AFM head movement so you decide the distance between AFM head and sample.
   Put the mouse curser on the Vision view part, extra commands will appear. You will be able to switch ON/OFF the beam, move the sample stage in all four directions, expand view, change view elimination, focus the AFM camera (objective) on the sample surface (**Focus Stage**) and move the AFM head Up/Down (**Z Stage**). You can click on **Expand** button to enlarge the Vision view window for better control, you can go back to normal size by clicking on **Close**.
   When starting SmarScan software the system will be inistizaled and **Z Stage** will go to **29000** and **Focus Stage** will go to **19508**.

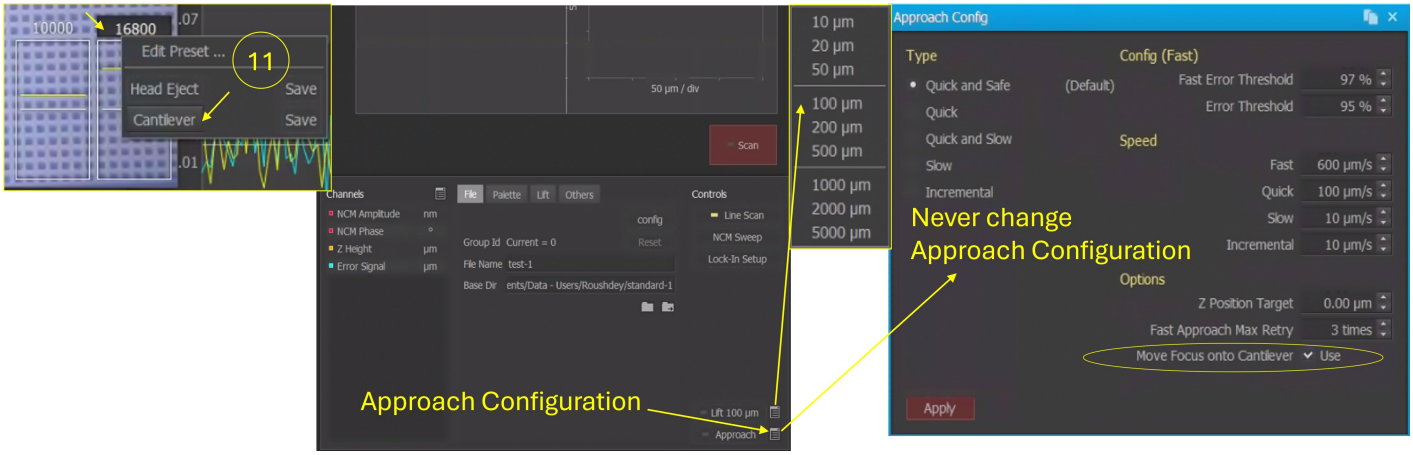


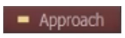
8. **The AFM head should be in near position to the surface before imaging/scanning starts**. Drive the AFM head (**Z Stage**) down close to the sample (about **1-2 mm** distance on top of the sample). To do this: Expand the vision view window and put the mouse curser on the Z Stage field (hand symbol will appear), put the hand symbol under the middle line and keep pressing left mouse button. AFM head will start to go down and you will see the **default position 29000** is decreasing, **decrease it to between 11000 - 10000**. Do this is slow steps and always inspect the distance of the AFM head and the sample on the camera.
   This example values is for a 1.5 mm sample including the sample disk, **this value may change if your sample thickness is more/less**. **Remember**: Crashing the AFM head on the sample means destroying the AFM system. Never go closer than 1-2 mm to the sample.
9. **Check the cantilever focus, SLD and PSPD alignment**. In this position, when AFM head close to the sample, the focus is better on the cantilever therefore you may find slight misalignment which can happened because Z-Stage movement or pervious not optimal alignment. Realign as described in section 3 (can not be done when working in vacuum, not very critical if not perfect, but the dot must be green).
10. **Define where to scan on the sample**. Use **Focus Stage** to focus the AFM camera (objective) on the sample, to see/find the sample surface and to locate where you want to scan. Put the mouse curser on the Focus Stage field, and moving the focus stage in the same way as moving Z stage (point 8).
    When the AFM head hight (Z Stage) at about 10000, the focus on the surface can be reached at about **16800** (by **default focus stage is set on 19508**). Look for suitable focus on the sample, extremely good focus is not important and will never lead to better AFM imaging. It is only to define where to scan on the sample. Move the sample stage by using the four side arrows or double click on wanted position, then this position will be placed in the center of the view (area where to scan).



★ **Here you can switch to Auto**  **mode if desired, (not recommended).**

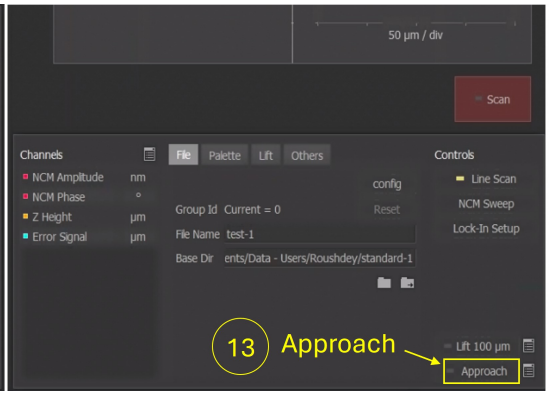
11. When the scanning position is defined, the **camera/objective focus must back to the cantilever** again, because during the measurement (or at Approach) the cantilever will be almost on the surface and when the focus is on the cantilever, the surface will be visible and in focus too.
    By default, this will be done automatically when Approach is activated.
    Possible to be done manually too (not necessary), by putting mouse curser arrow on the focus number (in this case 16800), the number will turn black, click on left mouse button, and click on **Cantilever**. Cantilever will **get in focus**.

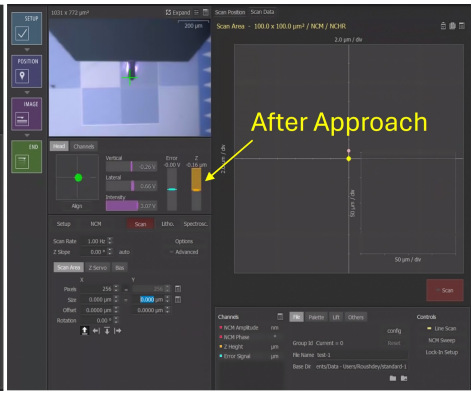


12. Set scanning **size** to **0×0 μm**, the sample stage should not be in movement during approaching to avoid probe or sample damage. In Approach the probe sample distance is only 500 nm (0.5 μm). **Never change Approach configuration**.
    Scanning size can be changed after Approach 

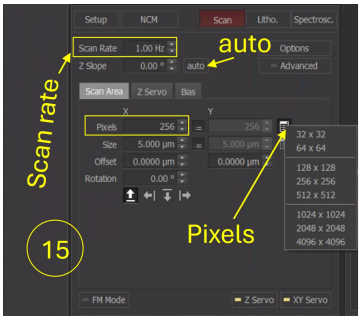
    is done, but never during Scan 

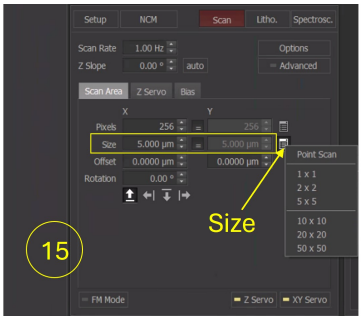
    .
13. Press **Approach** and wait until done, it can take some minutes. The cantilever will go automatically too close to the sample surface so it is feeling the forces from sample, therefore it will not be possible to change scanning position any more when Approach is activated. During Approach scan size must be **0×0 μm**.

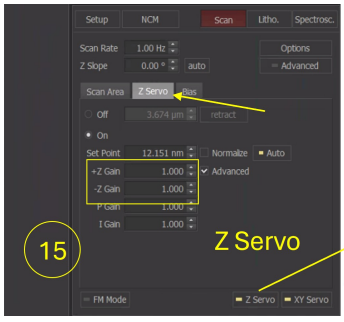


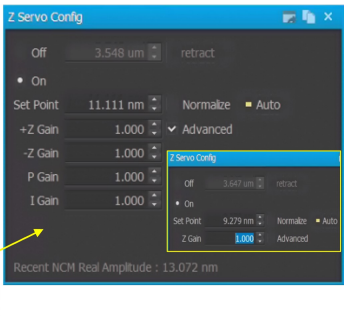


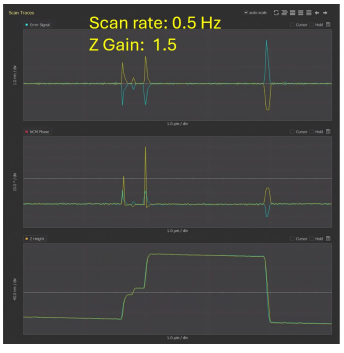
14. After Approach, set **scan size to 1×1 μm**, then it can be increased to 5 μ or more but always start from 1μ. The sample stage will start to move and the cantilever will feel the surface structures and all **Scan Trace** curves (**NCM Amplitude**, **NCM Phase**, **Z Hight**, **Error Signal**) will come on screen, before that they were just a fluctuations. The yellow curves are the forward scans and the blue curves are the back words scans. **Scanning of the surface is not started yet**.
15. **Error signal should be zero** (the yellow curve should compensate the blue cure) and **Z hight curves should be on top of each other's**.
    Theses can be achieved by changing scan parameters, mostly by **decreasing Scan Rate** and **increasing Z gain in Z Servo**. Now you are ready for scan.
    After approach you can **test/specify scan parameters**: important is **Scan rate** (lower is better, **between 0.1 and 0.5 Hz**, default value 1 Hz), **Pixels** (32, 64, 128, 256, 512, 1024, 2048, 4096, higher is better, **256 or 512** are good choices, default value 256) and the **Size** (**scanning area**, between 1 μm up tp 100 μm). These three must be well defined for better quality results and saving time. AFM imaging time is long (minimum 20-30 minutes).
    Set: **Pixel 256** or 512, **Size 1×1 μm** or 5×5 μm, increases **Z-Servo between 1-3** to make Error signal as small as possible and Z Height matching as high as possible. There are no magical values, they depend on sample, room temperature, room humidity, .. etc.

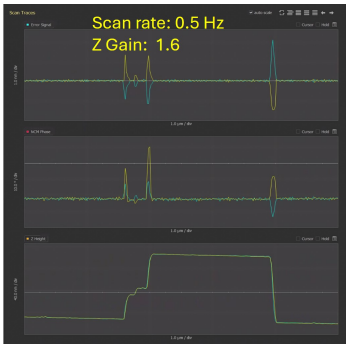




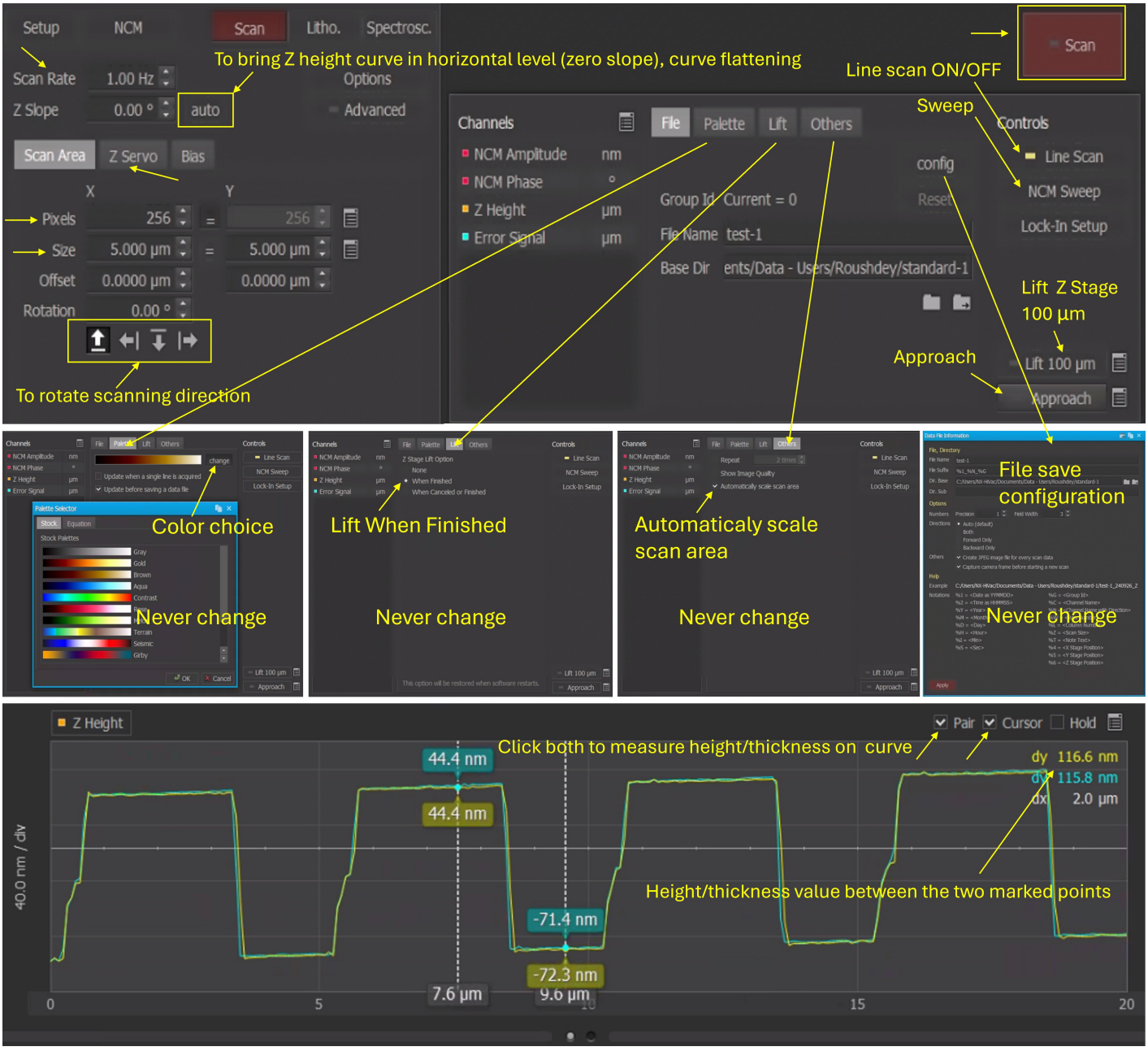








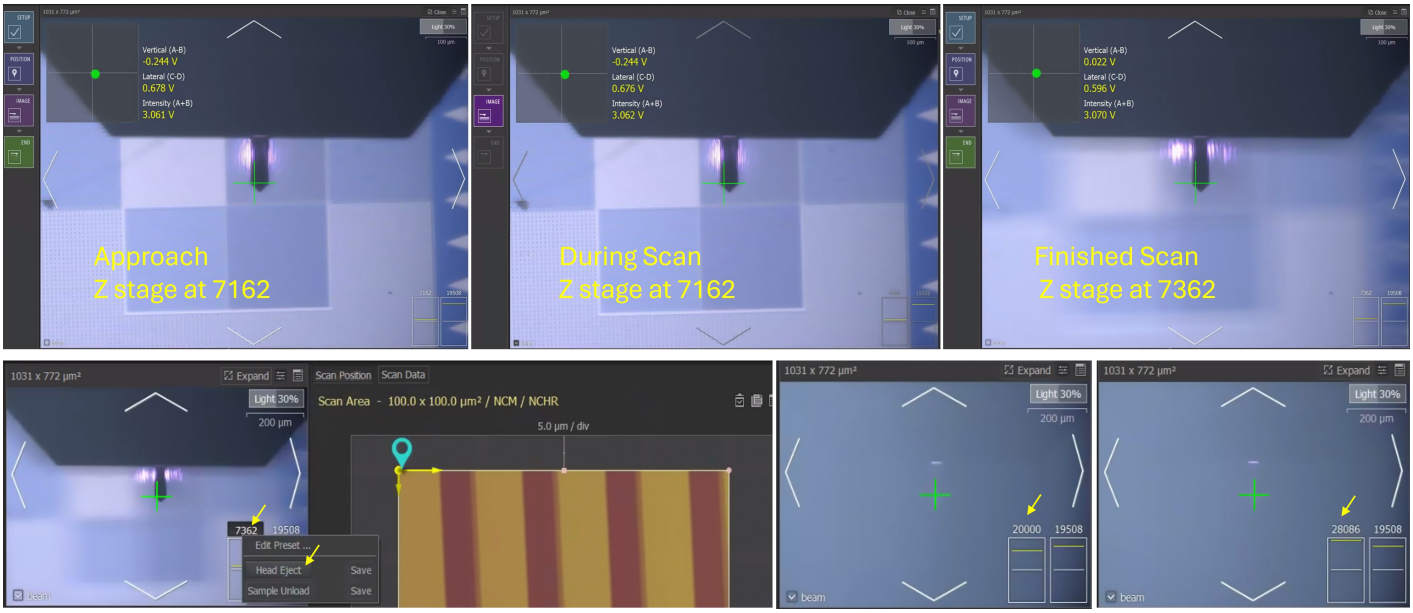
16. Good to know below marked features in software SmartScan before starting the scan. See picture below.



17. When all mentioned points are considered? **Press Scan** Scan, the button will turn to cancel Cancel and scanning will start. You will see the imaging required time under the produced AFM image. Wait until scanning is done. During this time only **IMAGE** is active on the left side bar. Nothing can be changed during this time, unless you Cancel the scan and lift Z stage.

18. **When first scanning is finished?** All items SETUP POSITION IMAGE END on left side bar will be active again, the Z Stage (AFM head) will lift automatically (but still too close the sample surface) and the **Cancel** Cancel button will turn to **Scan** Scan. In this case the cantilever is slightly over the sample and scanning position can be moved to another.
For a second scan on other position on the sample? Repeat points 10 to 17.
Never move or touch the sample physically unless you raise the AFM head to home position (25000-29000).

19. **When you want to exchange sample?** Put mouse curser arrow on the Z Stage number (in this case it is at about 7300), the number will turn black, click left mouse button, and click on **Head Eject**. The AFM Head will raise to 20000. Better if you raise upp the AFM head even more up to between 25000-29000. Exchange sample and repeat from point 8.

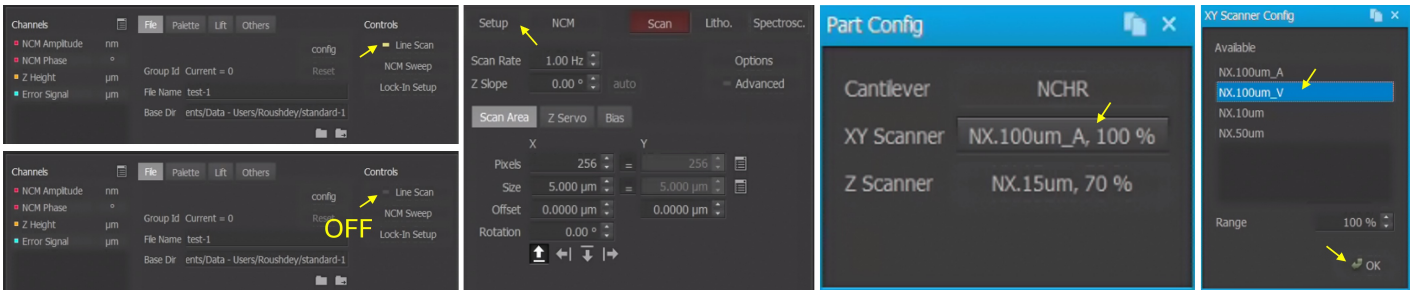


20. **When all scans/samples are finished?** Raise AFM head to home position between 25000 - 29000 as described in step before and remove sample. Shut down software, Switch off AFM Controller, Switch off HERZ vibration controller.

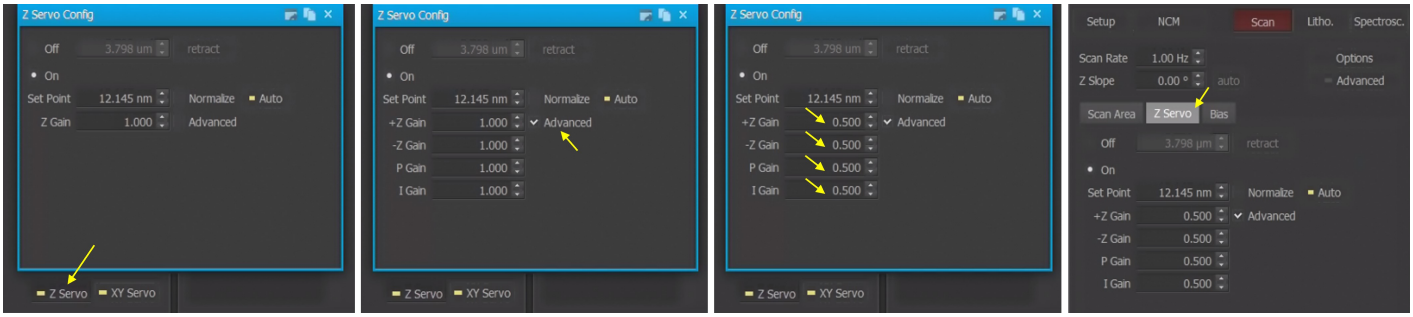
21. **For Data analysis se section —-**. All AFM images are saved in the selected folder. Copy folder and treat images in special software for analysis, corrections and 3D viewing.

When you want to scan in Non-contact mode(NCM) in vacuum?

- As in step 2. Turn **OFF Line Scan** >**Setup** >**XY Scanner** >select **NX.100um_V, 100**% >**OK**, after that turn **ON Line Scan**.



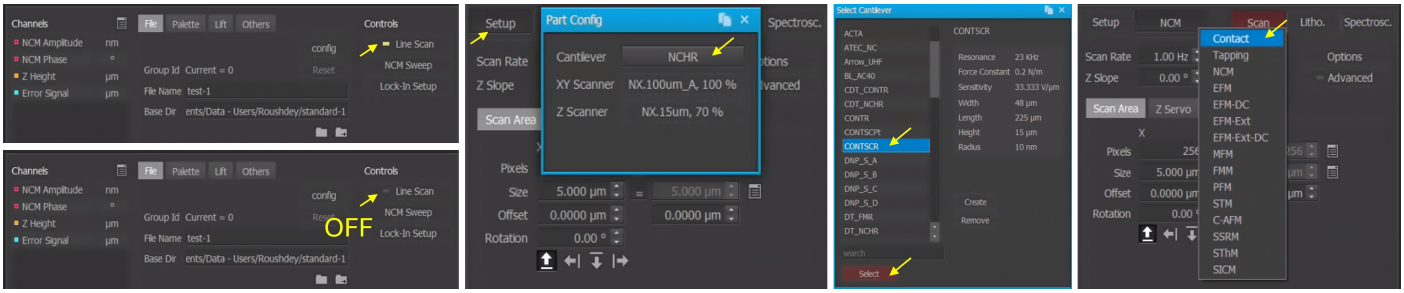
- Go to **Z Servo** >**Advanced** >**set all parameters** (+Z Gain, -Z Gain, P Gain, I Gain) **to 0.5**. You can reach Z servo in the software from the scan parameters part too.



- Start vacuum software as described in **section 5**.
- Run AFM scan as described in section 6.6.1 for Non-contact mode (NCM). Remember to align the PSPD for vacuum as described in section 3.

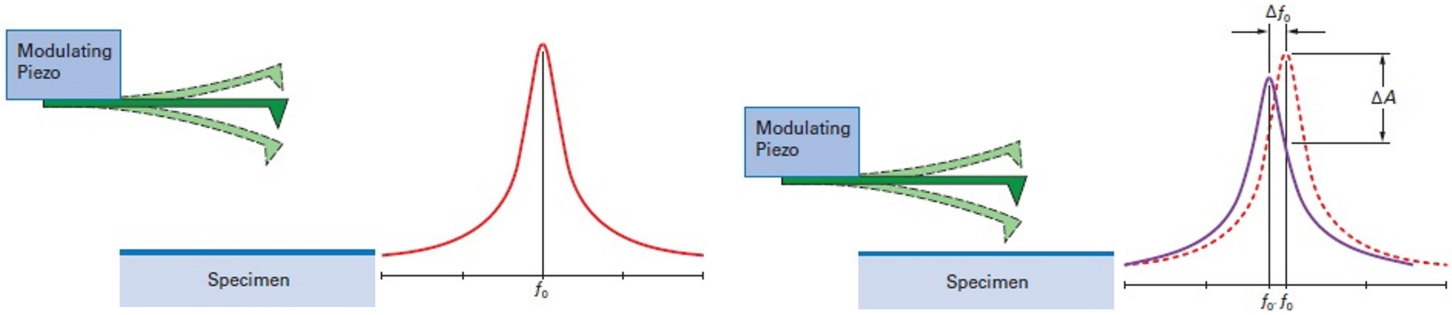
6.6.2 Contact mode

The same as in NCM mode (section 6.6.1), but you have to exchange the probe to correct type (PPP-CONTSCR), change Cantiliver sonfiguration in Setup, change operation mode to **Contact**. It may lead to more accurate images but this mode is not suitable for soft samples.
Turn **OFF Line Scan** >**Setup** >**Cantiliver** >select **CONTSCR** >**Select**, after that turn **ON Line Scan**. Finaly change scaning mode to **Contact**.

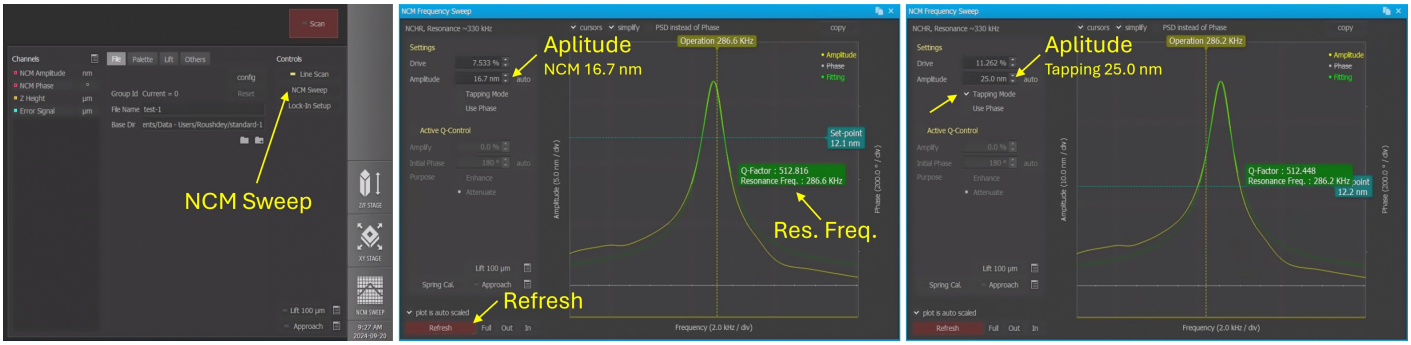


6.6.3 Tapping mode

This is the most common mode for acquiring surface topography. Lateral interaction between the cantilever and the surface can produce problems and reduces resolution of the technique, this is avoided by letting the tip touch the surface for a very short time. This mode, referred to as Tapping Mode AFM, or AC mode, the cantilever is oscillated at its resonance frequency while is dragged across the surface. Typical Tapping Mode operation is carried out using amplitude modulation detection with a lock-in amplifier. A typical response curve of a cantilever is shown in the figure.



In order to achieve the AFM modes known as tapping modes, the probe is oscillated intermittently, bringing it in contact with the sample. There are two ways to oscillate the cantilever: 1) through a shaker piezo 2) through photothermal actuation (CleanDrive). The shaker piezo provides the ability to oscillate the probe at a range of frequencies (typically 100 Hz to 2 MHz). Photothermal excitation provides a cleaner and more stable actuation at an even wider range of frequencies (100 Hz to 8 MHz).



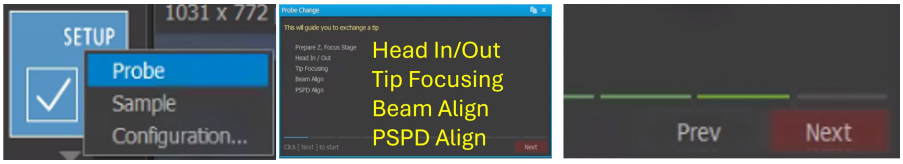
Sweep curve supposed to be checked if images are done in NCM or Tapping. Sweep window can be activated from NCM Sweep button in main window or from the right side bar, often pressing Refresh button can be enough to solve any problem.

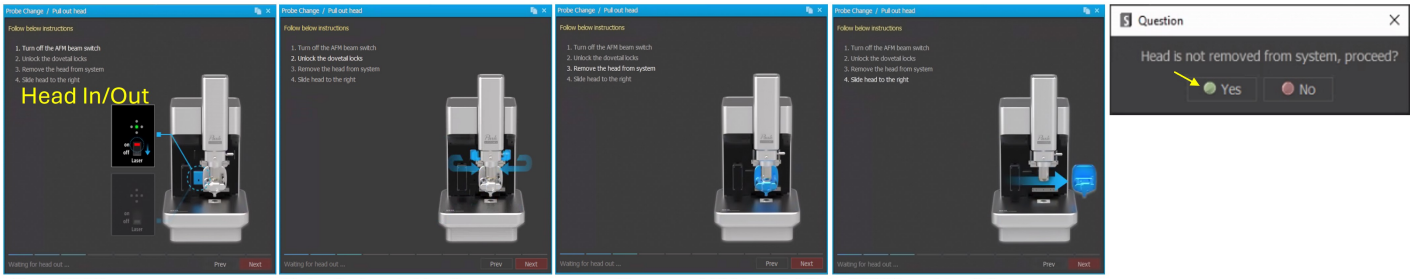
6.2 Auto scan mode

Auto In Auto scan it is only possible to measure in Non-contact **NCM** and **Tapping** modes. Here all parameters automatically controlled, not many parameters to adjust.

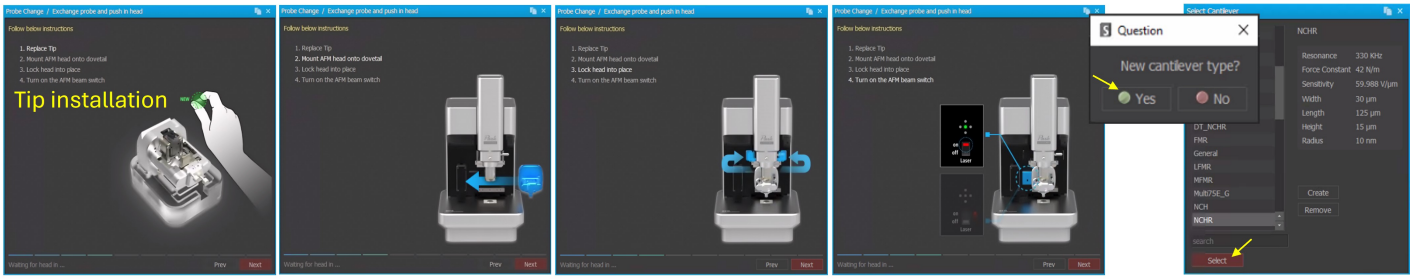
1. Do as described in points 1 to 10 in manual scan mode, before turning to Auto mode.
2. Turn scanning mode to Auto Auto. The screen will change to as seen, less objects on the screen.
3. Specify **Pixel**, **Scan Size** and **Quality/Speed**. Give file name and in which folder to save.

4. Left cklick on **SETUP >Probe** will guide you how to get out/in the AFM head, installing a new Tip, focusing the optics, Laser beam (SLD) alignment on cantilever, also PSPD alignment, continue by **>Next** and end by **>Close**.

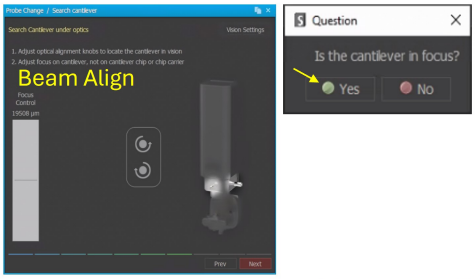




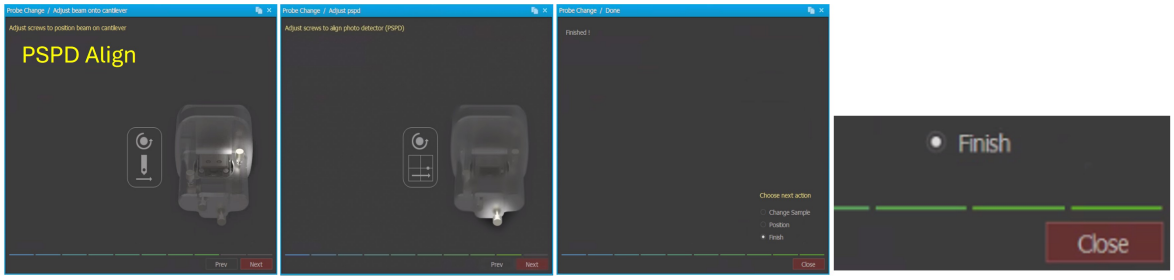
Turn OFF laser | Unlock dovetal locks | Remove AFM head / Side AFM head to right



Replace AFM tip | Return head to dovetail | Lock head in place | Turn ON laser

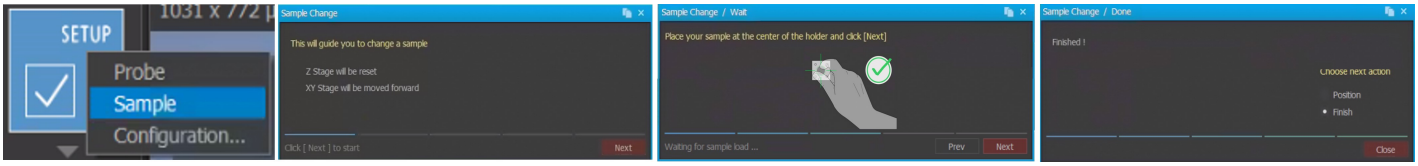


Manually adjust optical alignment (cross on cantilever). As described in section 3.

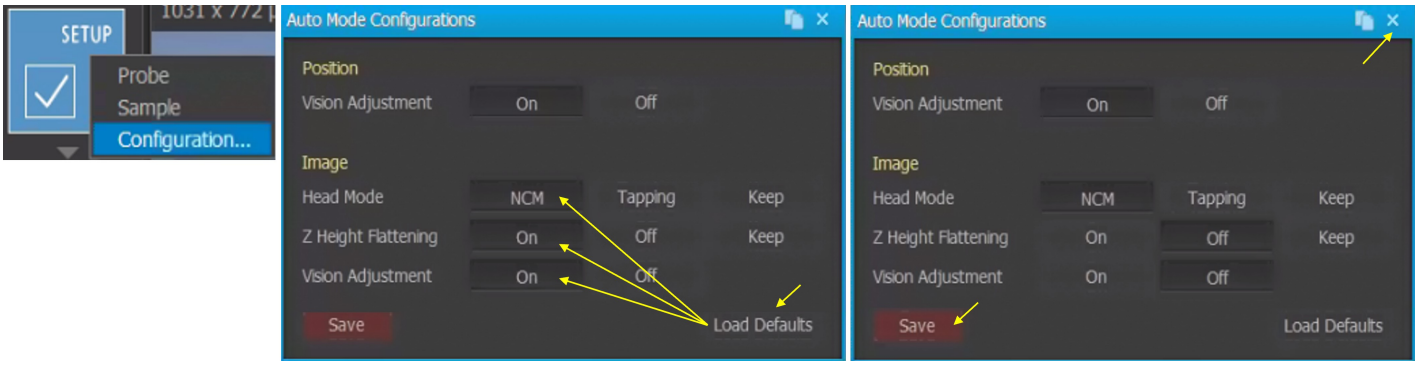


Manually align the beam on cantilever (about 3.0 V), after that align PSPD (green dot) on X-direction and press Align button for Y direction alignment.

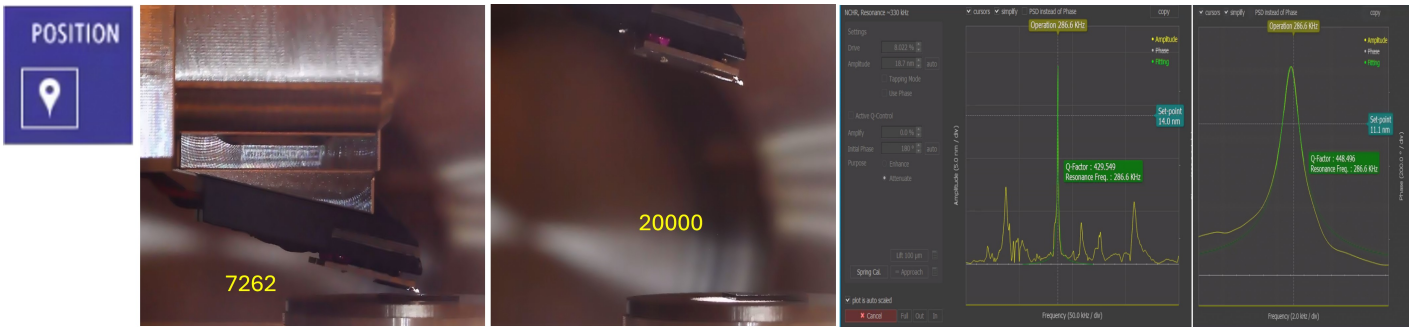
5. Left cklick on **SETUP >Sample >Next** , sample stage will come forwards to put the sample **>Next** sample stage will go back under the AFM head agin, Finished **>Close**.



6. Left cklick on **SETUP >Configuration**, to chose between NCM or Tapping, or press **Load Defaults** (NCM, Z Hight Flattening ON, Vision Adjustment ON). Close window by .

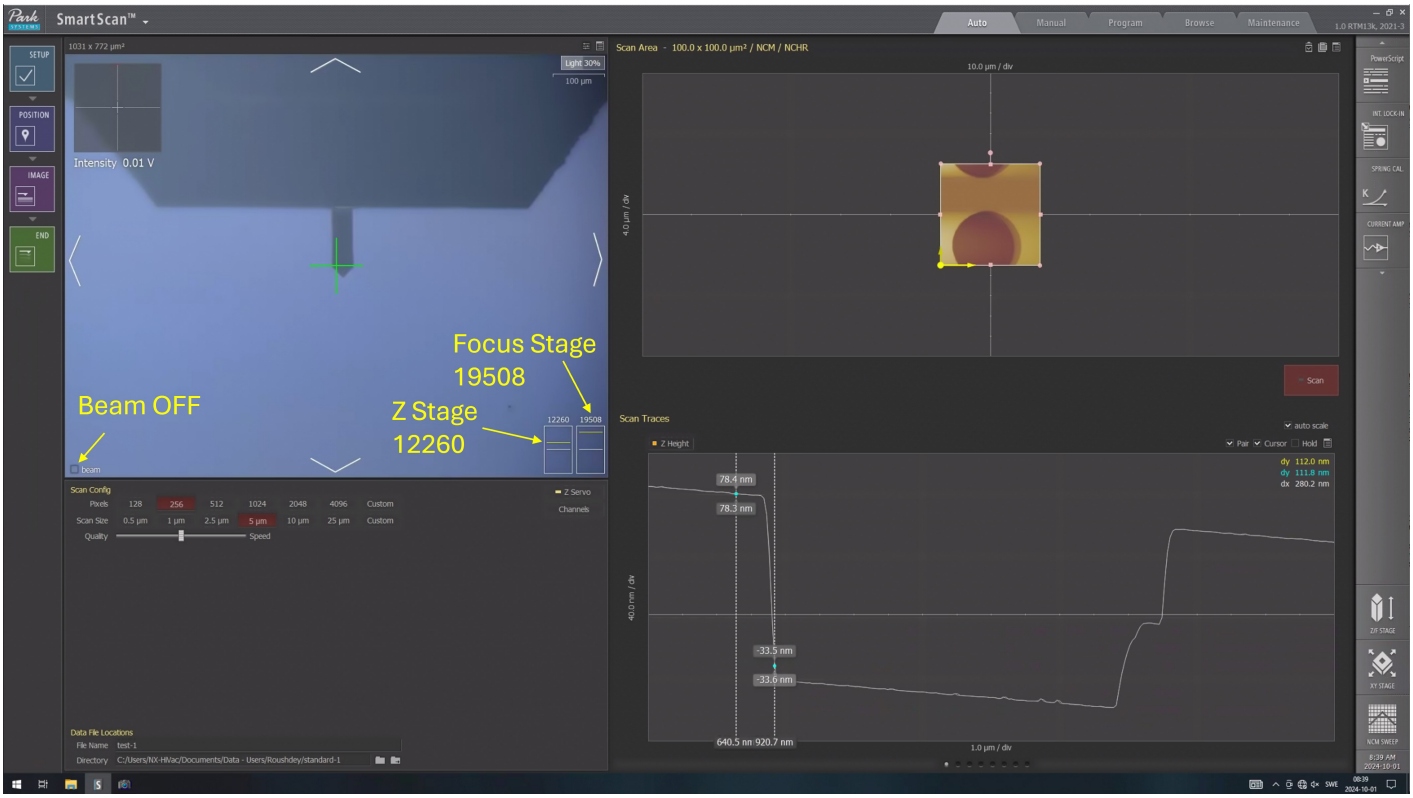


7. Left cklick on **POSITION**  , NCM frequency Sweep fitting will be automatically performed, and the **Z stage will taken down to 7262** (too close to the sample surface) from 10000-11000. Wait until all four buttons on left bar are active. This process is similar to Approach in manual mode, you can not change scan position any more on the sample unless you raise the AFM head to save height.
Remember: puting mouse curser on the Z Stage number (in this case about 7262), the number will turn black, click left mouse button, and click on **Head Eject**. The AFM Head **will reis to 20000**. Better if you raise upp the AFM head even more up to between 25000 - 29000 if you want to exchange sample.
The AFM head should be in near position of the surface before imaging/scanning starts.



8. Left cklick on **IMAGE**  , Will approach the sample and Scan button will turn to Cancel, and imaging will start, you will see the required time for scanning under the produced images. Wait until finished and all four left bar buttons are active. Make sure if the Scan Size and Pixel are the same on both manual and Auto modes. The software may take the parameters set in Manual mode in consideration although they are set to different in Auto mode.
Remember : you have to raise (Head Eject) the AFM head inforder to move to other position on the sample.

9. **END** button will take up the Z Stage to 12260, Focus Stage to 19508 and switch OFF the laser beam, therefore the green dot will disappear and the intensity will show zero.



7 C-AFM (Conductive Atomic Force Microscopy)

Conductive atomic force microscopy (C-AFM) or current sensing atomic force microscopy (CS-AFM) is a mode in atomic force microscopy (AFM) that simultaneously measures the topography of a material and the electric current flow at the contact point of the tip with the surface of the sample.
During C-AFM imaging, a **conductive tip** scans the sample surface in **Contact mode** while simultaneously measuring the current flow at each pixel for a given sample bias with a current-to-voltage preamplifier. Since the magnitude of the electric current can range from a few pico-amperes to milli-amperes depending on the sample, the current amplifier should be chosen to match the conductivity of the respective sample. Park Systems offers three different options for the current amplifier to fit the requirements of a wide application range. The first option uses an internal amplifier, the second option includes an external variable gain, low noise current amplifier (VECA), and the third option is an external ultra-low noise current amplifier (ULCA). The measurable current ranges and specifications of each amplifier are given in the table below.

	Internal amplifier	VECA	ULCA
Maximun measurable current range	10 μA (at 10^6 V/A gain)	10 mA (at 10^3 V/A gain)	10 pA (at 10^{11} V/A gain)
Gain range	2 gains (1×10^6, 10^9 V/A)	7 gains ($1\times10^3 \sim 10^9$ V/A)	Fixed gain (1×10^{11} V/A)
Noise level	<0.4 pA	<0.3 pA	<0.1 pA
Bias range	-10 V ~ 10 V	-10 V ~ 10 V	-10 V ~ 10 V

In C-AFM experiments, the sample is usually fixed on the sample conductive holder using a conductive tape or paste (silver paints).

Conductive AFM `https://www.parksystems.com/en/products/research-afm/AFM-modes/Electrical-Modes/conductive-afm--c-afm-`

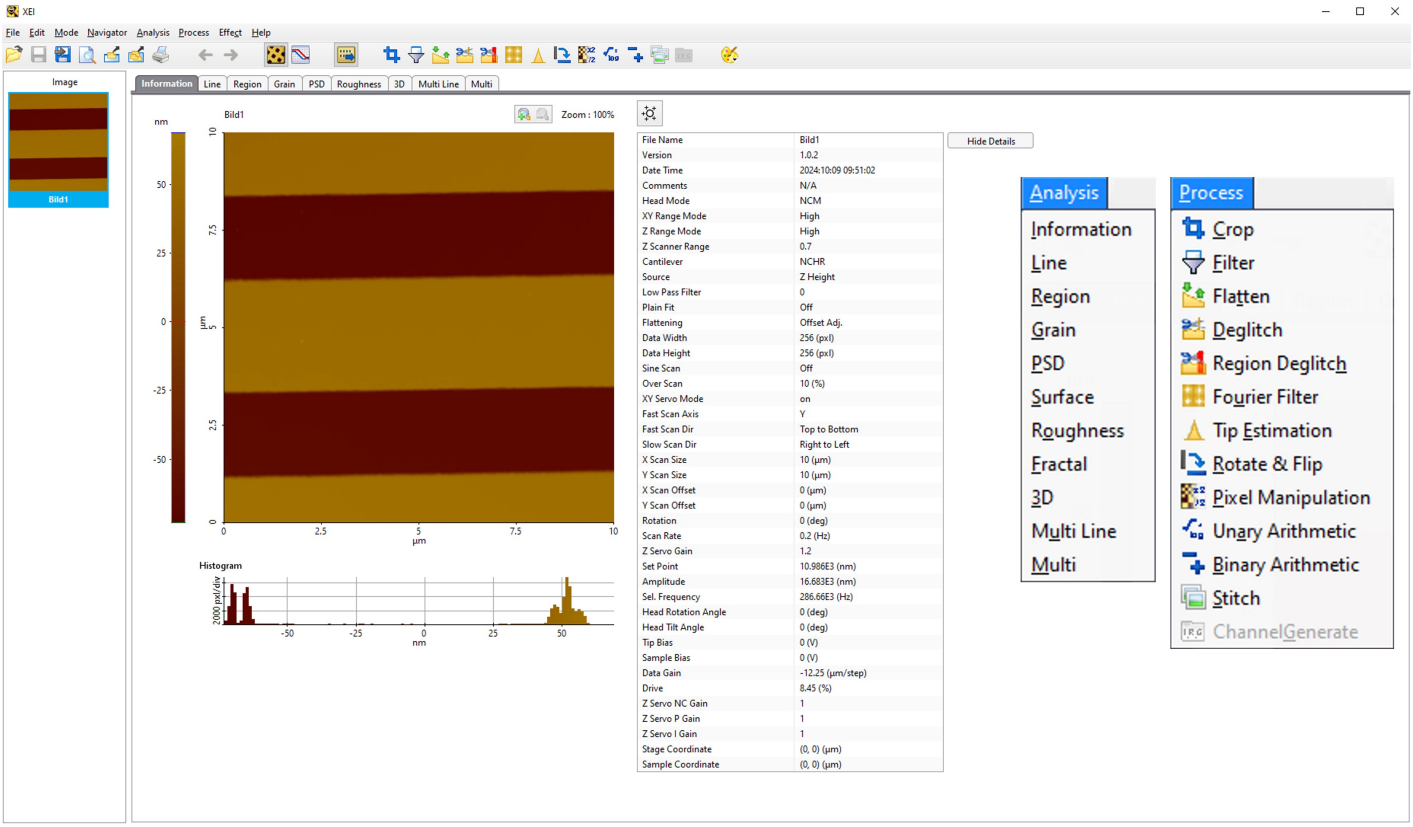
8 AFM Lithography

9 Data/Image analysis

How to interpret or analyze AFM images?

There are several ways to analyze different parts of AFM images, including line profiles, surface roughness calculations, height analysis, particle analysis, and grain analysis. This project focusses primarily on surface roughness, height analysis, and grain analysis.

XEI is a software program that provides a dynamic tools for image processing, quantitative analysis and statistics, and export and printing of processed images and measurement results. Double click the XEI icon, then open the wanted image (tiff format).



What you mostly need to know about in XEI software:

Information Information | Line | Region | Grain | PSD | Roughness | 3D | Multi Line | Multi displays the original image in 2D with the scan information associated with the image data. Show Details will show extra scan details (original scan parameters and conditions).

Line Information | Line | Region | Grain | PSD | Roughness | 3D | Multi Line | Multi gives information about the cross section or height profile of the surface in the selected line direction. You can create up to three lines for line profile analysis on the image. vertical, horizontal and/or arbitrarily sloped lines are possible. Each line you create is indicated by a different color in the Image display panel as well as in the analysis plots. Creating an Average line is the same as creating a single line. The selected average area will display in the Line Profile panel as a single Line Profile.

To measure an exact height difference and distance between two data points in the Line profile, you can insert a cursor pair on the Line Profile by selecting '**Insert a Cursor Pair**' command in the context menu. You can insert up to three cursor pairs per profile. When the cursor pair is inserted, two triangular shaped cursors appear on two arbitrary points on the Line profile and the corresponding points on the image in the image display panel as well. You can adjust the location of the cursors by dragging and dropping individual triangular cursors either from the line profile or from the image in the image display panel. You can also move the cursors with pixel precision by clicking on a cursor, then pressing the → and ← keys on your keyboard.

You can also select '**Leveling**', you will see two white bars in the line profile. You can use these bars to easily subtract the background slope. Point your mouse at each bar and the cursor will change to a left-right arrow. Drag each bar to two different points you believe to be at the same height. The slope of the line profile will change accordingly so that the two different points are brought to the same level. After leveling, the white bars can be

disappeared by deselecting 'Show leveling bars'.

In line view roughness statistics are to be obtained by drawing a line on the interest area.

Statistics

Line	Min(nm)	Max(nm)	Mid(nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
Red	51.576	53.184	52.380	52.047	1.608	0.245	0.188	1.275	1.122	5.532

Min (nm) minimum, or smallest, value in the line profile.
Max (nm) maximum, or largest, value in the line profile.
Mid (nm) arithmetic average between the minimum and maximum values. That is, Mid=(Max+Min)/2.
Mean (nm) arithmetic mean value of the line profile. It is the sum of the height of each point divided by the number of points.
Rpv (nm) peak-to-valley of the line. It is the difference between minimum and maximum, that is, (Max–Min).
Rq (nm) root-mean-squared roughness.
Ra (nm) roughness average. The average roughness is the area between the roughness profile and its mean line.
Rz (nm) ten point average roughness. It is the arithmetic average of the five highest peaks and five lowest valleys in the line.
Rsk skewness of the line.
Rku kurtosis of the line. It indicates the "spikiness" of the sample surface along that line.

Region Information | Line | Region | Grain | PSD | Roughness | 3D | Multi Line | Multi gives information about a region of the sample surface in the selected area such as the maximum and minimum height value, mean height and RMS roughness.

Roughness Information | Line | Region | Grain | PSD | Roughness | 3D | Multi Line | Multi Roughness analysis, Region View and usage are the same. Selecting an entire area or part of the image via the Toolbar will update the Roughness on the right side of the screen.
Sq (μm) Root mean square height.
Sku Kurtosis of height distribution.
Sp (μm) Maximum height of peaks.
Sv (μm) Maximum height of valleys.
Sz (μm) Sum of the maximum peak height value and the maximum pit height value within a definition area.
Sa (μm) Arithmetic mean height.
Sdq (rad) Root mean square of the surface gradient within the definition area of a scale-limited surface.
Sdr (%) Developed area ratio.
Sk (μm) Core height.
Smr1 (%) Ratio of the increment of the interfacial area of the scale-limited surface within the definition area over the definition area.
Smr2 (%) Ratio of the increment of the interfacial area of the scale-limited surface within the definition area over the definition area.
Spk (μm) Reduced peak height.
Svk (μm) Reduced dale height.
Sxp (μm) Peak extreme height.
Vvv (ml/m^2) Dale void volume of the scale-limited surface.
Vvc (ml/m^2) Core void volume of the scale-limited surface.
Vmp (ml/m^2) Peak material volume of the scale-limited surface.
Vmc (ml/m^2) Core material volume of the scale-limited surface.

3D Information | Line | Region | Grain | PSD | Roughness | 3D | Multi Line | Multi generate 3D images and helps you to see the features of the image and the relationships between those features more clearly.

Crop : you can crop a part of an image which is a region of interest.

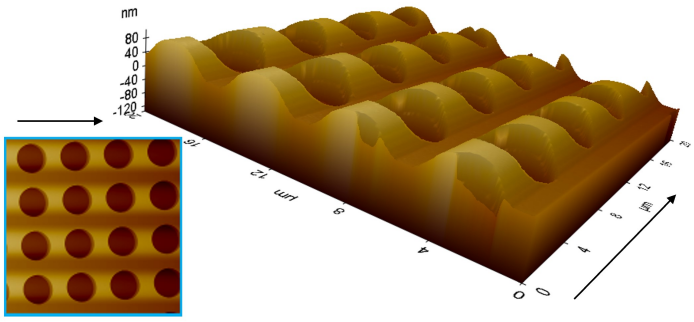
Flatten : remove curvatures and slopes from your image data.

Deglitch : remove small glitches or vertical and horizontal streaks in an image.

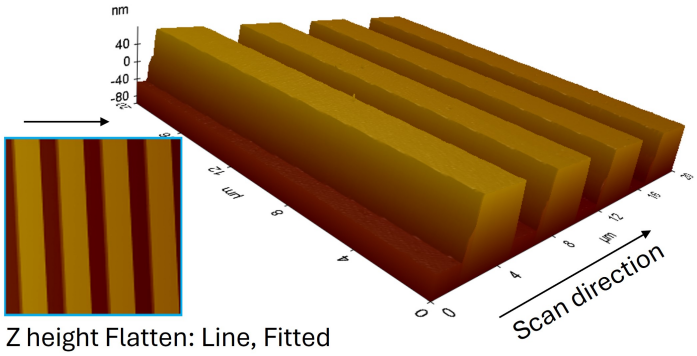
Region Deglitch : remove small artifact in an image that does not represent the true surface topography to obtain more accurate image.

The first thing you may need to do? is to flatten the image. Only with this you will get surfaces in level and define zero in Z-axis.
Flattening me be done automatically during scanning too: In the **channel** overview: set **Z Height** leveling to **Line** and Data Saving to **Fitted**.

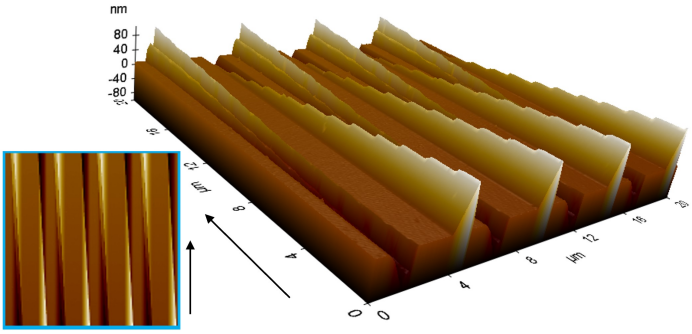
In the **channel** overview: set **Z Height** leveling to **None** and the fitting to **Raw**.



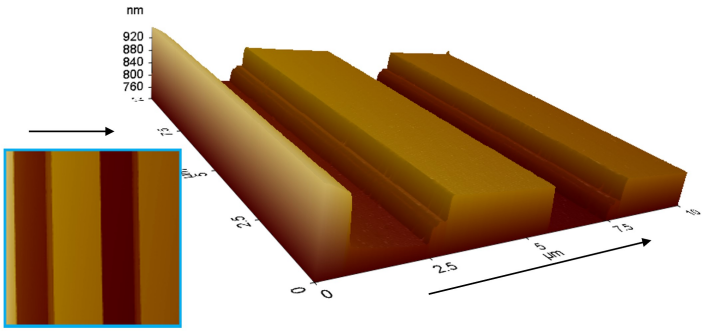
Z height Flatten: Line, Fitted



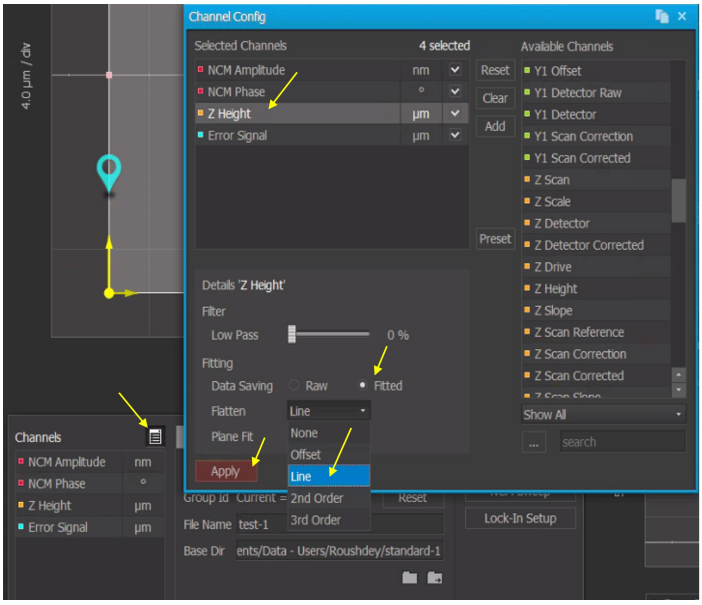
Z height Flatten: Line, Fitted

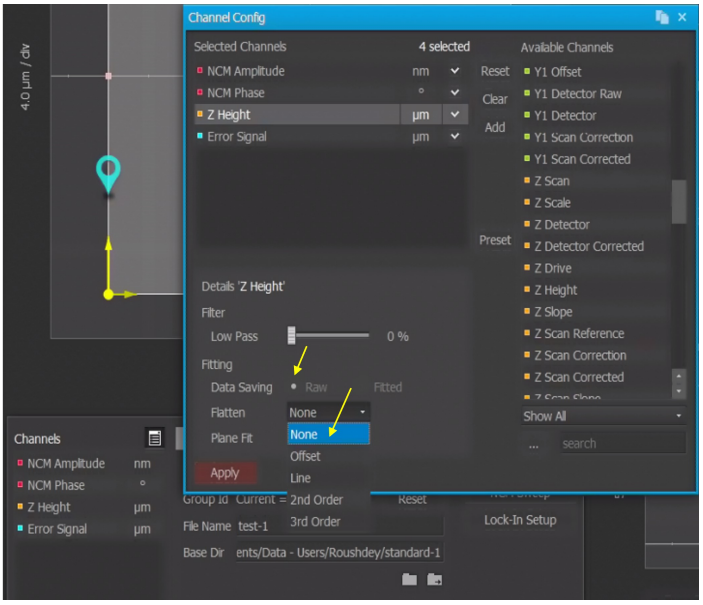


Z height Flatten: Line, Fitted



Z height Flatten: none, Row





What is the phase image in AFM?

Phase images can be used to identify which regions possess different properties, such as adhesive, viscoelastic, stiffness or frictional, which might be hidden in the topography image. Changes in phase lag often indicate changes in the properties of the sample surface. Images of topography and material properties can be collected simultaneously.

How to operate AFM (Park Systems, NX-10) [KAIST MSE] https://youtu.be/De3Uag4Ix7w