Atomic Force Microscope

Practical User Guide for the DI Dimension 3000

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Version 1, English
Edition 1

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1. Preface

It is the intention of this manual to provide a brief summary for the operation of the Dimension 3000 AFM and its NanoScope IIIa controller. This version covers only the tapping mode. The following sections should provide enough information to handle the microscope appropriately and to obtain a reasonable result. Therefore, it is more detailed than a simple checklist. But to assist the operator effectively, it contains neither physical background information nor technical explanations.

However, the attendance of training is an obligation. Additionally, the operator is advised to consult a staff member in case of serious questions, especially malfunction or misoperation. Finally, reading this paper cannot substitute experience, in particular aligning the laser and setting the scanning parameters

For a deeper insight into the physics of Scanning Probe Microscopy (SPM), Atomic Force Microscopy (AFM) and modes like contact and intermittent please consider appropriate secondary literature, e.g. textbooks or scientific articles like

B. Bhushan (Edt.), *Springer Handbook of Nanotechnology*, 2004 Springer Berlin Heidelberg.
see Part B Scanning Probe Microscopy

C. Dupas, P. Houdy, M. Lahmani (Edts.), *Nanoscience - Nanotechnologies and Nanophysics*, 2007 Springer-Verlag Berlin Heidelberg.
see chapter 4 Atomic Force Microscopy

but also

Command Reference Manual, 2001 Digital Instruments Veeco Metrology Group.

SPM Training Notebook, 2003 Veeco Instruments Inc..

Dimension™ 3000 Instruction Manual, 1997 Digital Instruments Veeco Metrology Group.

Stage microscopes feature an automated X-Y stage and Z-axis capable of programmed movement. The movements of all axes are slow, but are capable of exerting high forces. A hand caught in the stage could be injured severely.

The stage contains a diode laser with an output of less than 1.0 mW at 670 nm. Don’t look into the laser beam.

2. Turn-on procedure

The following steps are required to start the system (All units are shown in Figure 1):

1. Make sure that the computer is switched on and has finished booting
   If the computer is switched off, switch it on and let it boot completely.
   It is not necessary to logon and to enter a password.

2. Switch on the controller (see back side).
3. Switch on the power supply of the stage (see back side, the turning knob on its front side is inoperable)
4. Switch on the microscope lamp
5. Switch on the vacuum pump
   There is no special order required for switching on the microscope modules mentioned in (2) to (5).

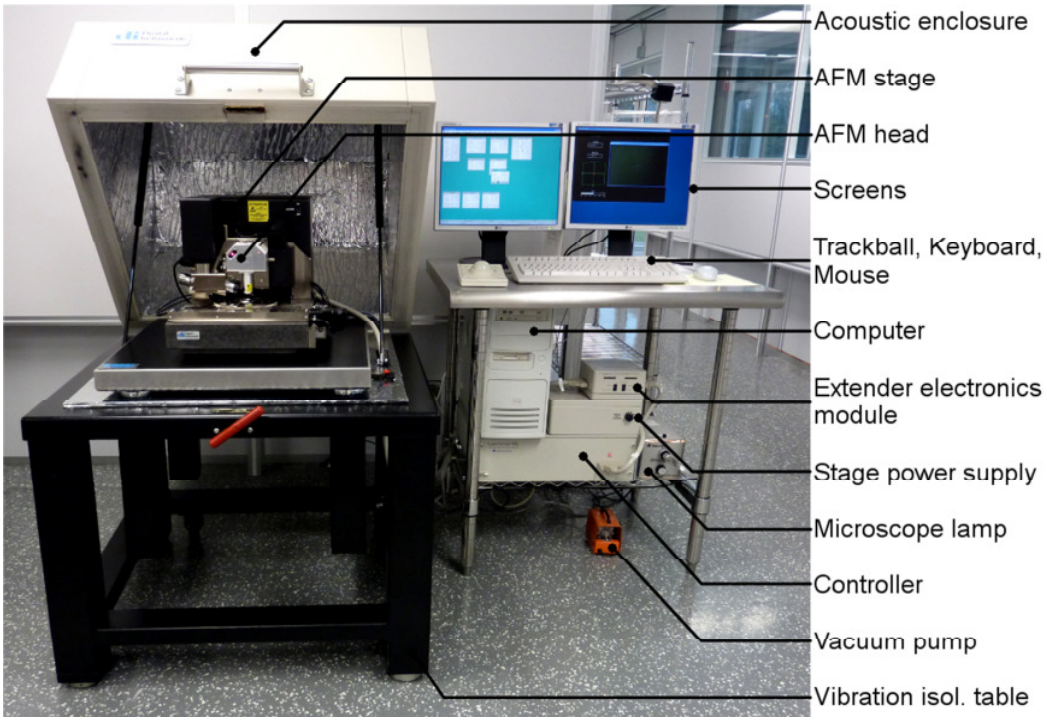


Figure 1: Complete setup of the AFM with the stage on the left and the operator station on the right.

6. Start the microscope software *Nanoscope 5.30r3sr3*
   A corresponding shortcut is on the *Windows desktop*.

Shortcut icon of the NanoScope GUI on the Windows Desktop

7. Start the *Real time mode* of the AFM by activating the corresponding button on the left monitor

Real time mode icon of the GUI

8. The left screen should now show the *Real Time Control Monitor* (see Figure 2), and the right screen should show the *Display Monitor* (see Figure 3). The red diode on the AFM scanner head, which indicates the active laser, should glow.

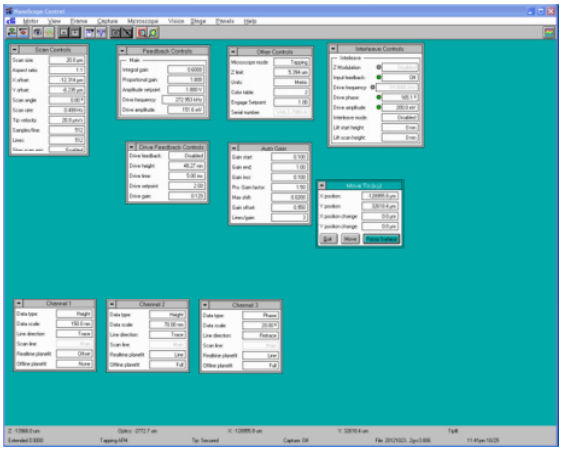


Figure 2: *Real Time Control Monitor*, which is typically on the left screen.

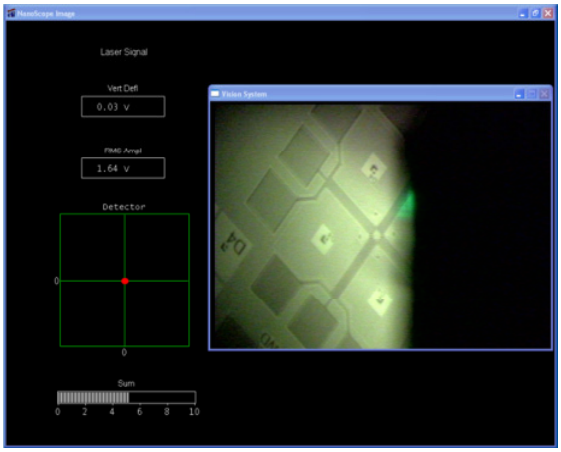


Figure 3: *Display Monitor*, which is typically on the right screen. The smaller window on the screen shows the Vision System image of the microscope.

Now, the machine is powered up.

3. Tip mounting

The following steps are required to mount a tip into the machine:

1. Mount a cantilever substrate onto an AFM tip holder
   1.1. Put a standard AFM tip holder onto the cantilever installation fixture (see Figure 4)
   1.2. Shift the clamp on the back side of the tip holder backward
   1.3. Take a cantilever substrate out of the storage box and deposit it onto the surface in front of the tip holder's pocket
   1.4. Shift the chip, or substrate into this pocket
   1.5. Fix the chip holder with thumb and pointer of the one hand, and shift the clamp over the chip with the pointer of the other hand

- **A dropped substrate is a broken tip!**
- **A substrate lying on its top side is a broken tip!**
- **Don't touch the cantilever substrate with your hands, and don't touch the short edge that holds the cantilever with tweezers!**

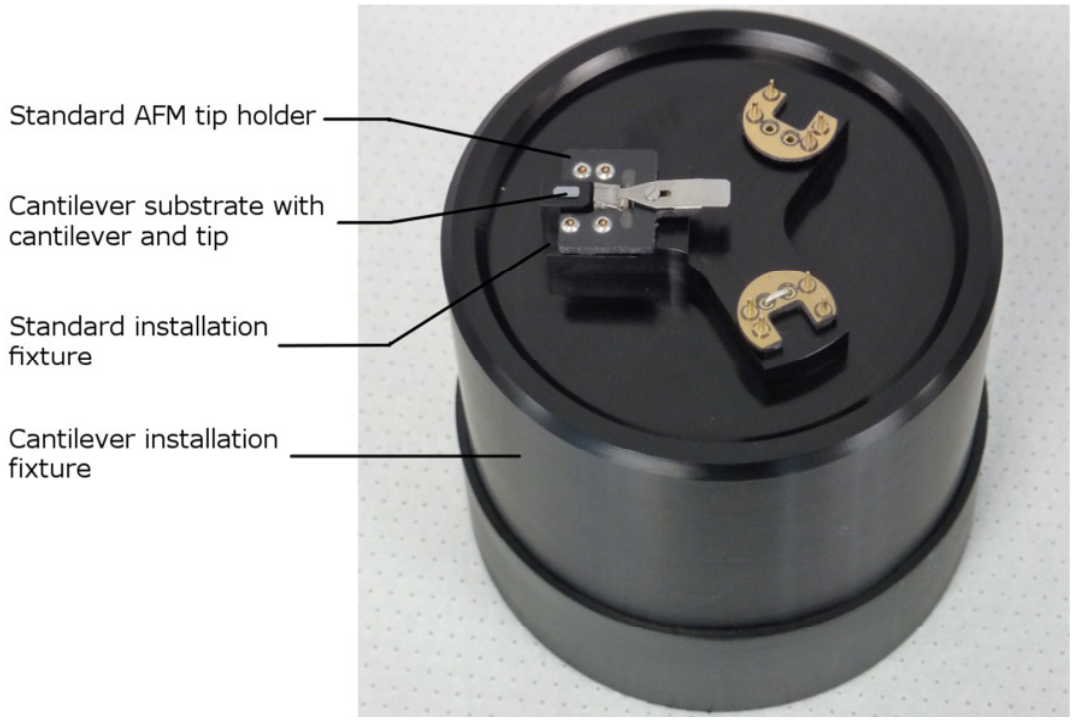


Figure 4: Cantilever installation fixture with a standard AFM tip holder in its fixture. A cantilever substrate is positioned in front of the tip holder pocket.

2. Mounting the chip holder onto the AFM head
   2.1. Open the acoustic enclosure
   2.2. Loose the AFM head (see Figure 5) by tightening the clamping screw (turn clockwise)
   2.3. Shift the AFM head upwards out of the dove tail guide rail
   2.4. Turn the AFM head upside down

- **Ensure that the laser switches off while turning the head!**
- **Neither look into the laser beam nor into any beam reflection!**
- **Take care that the connection cable between head and stage is not stressed!**

2.5. Pull the tip holder off its installation fixture and push it gently onto the four connector pins under the AFM head.
Ensure that the chip holder bears tightly on the AFM head!
2.6. Turn the AFM head in its normal direction
2.7. Move the AFM head slowly down the dove tail guide rail until it rests safely on its bedstop
2.8. Tighten the AFM head by losing the fixing screw (turn counter clockwise)

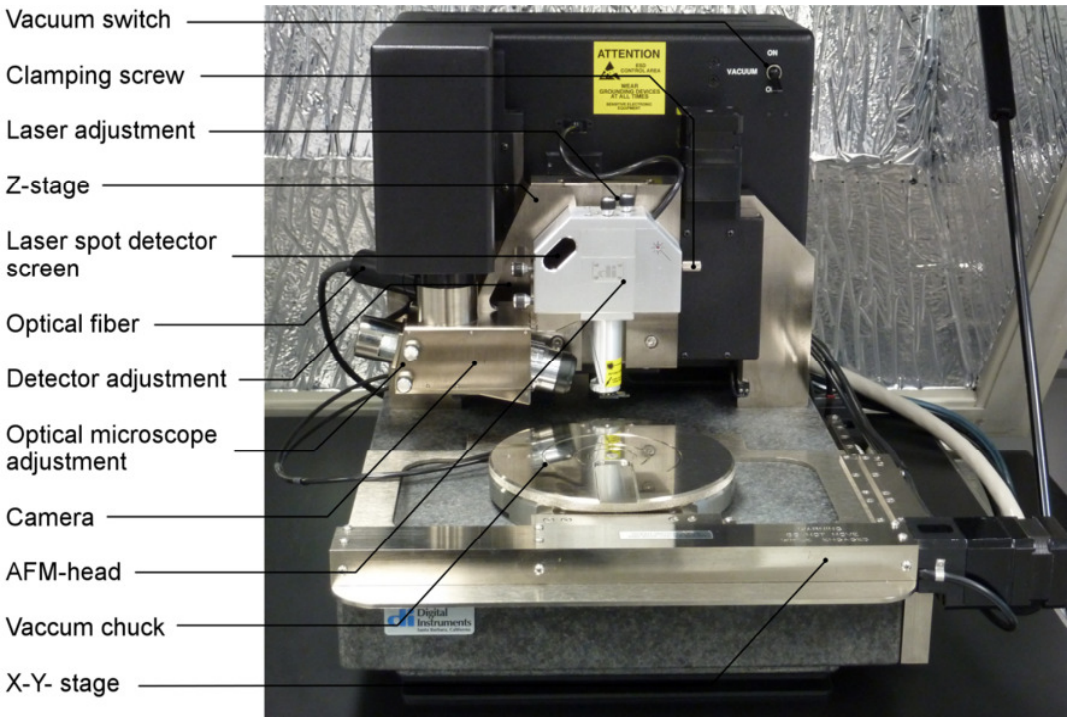


Figure 5 Dimension 3000 stage with labeling of the particular components

4. Adjusting the optical lever

This section explains how to align the laser beam on the cantilever and its reflection on the photo detector. Adjusting the optical lever is required after each AFM tip exchange. There exist three options:

A. There was no aligned and reflective sample on the chuck before tip exchange. This requires a manual laser beam alignment procedure.
B. There was and still is an aligned and reflective sample on the chuck and a manual laser beam alignment is desired.
C. There was and still is an aligned and reflective sample on the chuck and a guided tip exchange is desired.

Consider that it is not possible to move the stage in x-, y- and z-direction without a sufficient sum signal on the detector. However, this is typically zero before the laser is appropriately aligned

1. Start the *Cantilever focus* mode


Focus Tip icon of the GUI

2. Align cantilever roughly with the crosshair on the screen, see Figure 6, by turning the adjusting knobs at the camera optics

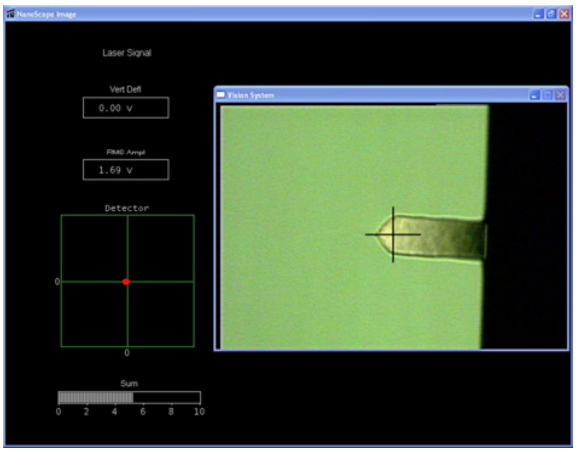


Figure 6: *Display Monitor* with a zoomed in *Vision System Image* of the cantilever. The crosshair of the optical microscope is aligned with the front of the cantilever

2.1. Bring tip into focus by using the track ball and pressing the *Focus* button
2.2. Eventually correct the tip to camera alignment
2.3. Zoom in by using the track ball and pressing the *Zoom* button
2.4. Eventually, readjust the focus of the tip

3. Align laser beam on the leading end of the cantilever by turning the adjusting screws, which are shown in Figure 7, on the AFM head

OPTION A

In the case that no sample is in focus, mostly it is not possible to align the laser spot by focusing the microscope on the tip reflection. In this case, the laser needs to be aligned by view.

3.1. Put of a piece of paper under the AFM head to follow the laser beam
Never look directly into the reflection of the laser beam on a reflective surface!
3.2. Deflect the beam in way that its complete shape becomes visible on the paper, as illustrated in Figure 8
If the beam is not visible, it is probably shadowed by the cantilever substrate, like indicated in Figure 10. So, turn the laser alignment screw for the x-axis counter clockwise until the beam appears on the paper
3.3. Find the front edge of the cantilever substrate with the laser beam, which corresponds to the schematic in Figure 9
3.4. Find the cantilever with the laser beam by traveling along the front edge, which corresponds to a movement of the y-axis
3.5. Maximize the sum signal presented by a bar called *Sum* on the *Display Monitor* with both adjusting screws. The ideal position is illustrated in Figure 11

Aligning the laser on the cantilever is not easy for beginners. It requires some patience and imagination of the setup. To ease this process, have a look at the schematics below.

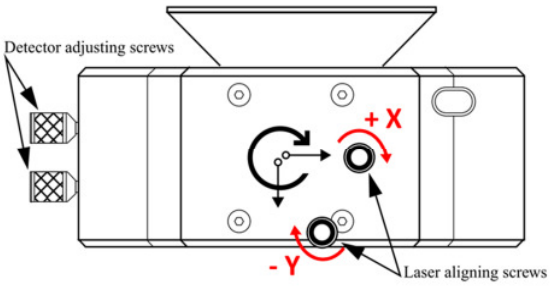


Figure 7: Top view of the AFM head with both laser aligning screws. The rear right screw deflects the laser beam in x-direction, the front left one in y-direction.

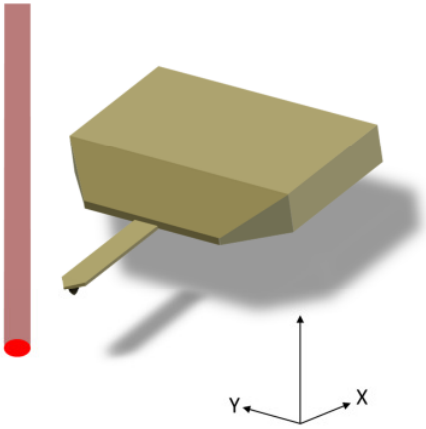


Figure 8: Laser spot is completely visible: Typically, it is in front of the cantilever substrate. Possibly it might be left or right of the substrate.

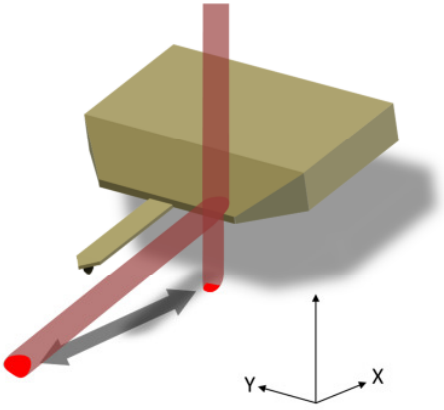


Figure 9: If the laser spot was in front of the substrate, deflect it in positive x-direction. Once it strikes the inclined front edge of the substrate, the beam splits into two, and one fraction becomes visible in a clear distance from the original position.

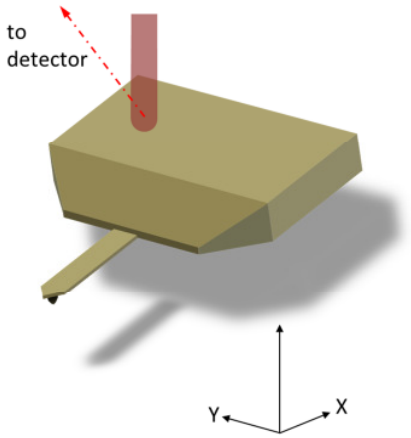


Figure 10: If the laser spot becomes completely invisible, it is typically on the substrate. Redirect it into –x-direction until it becomes visible again.
Possibly it might have left the alignment range by a huge deflection, which requires several turns of the aligning screws. Then, try to bring the beam back into the center of the circular shaped light reflection of the microscope lamp. Now, restart the alignment procedure.

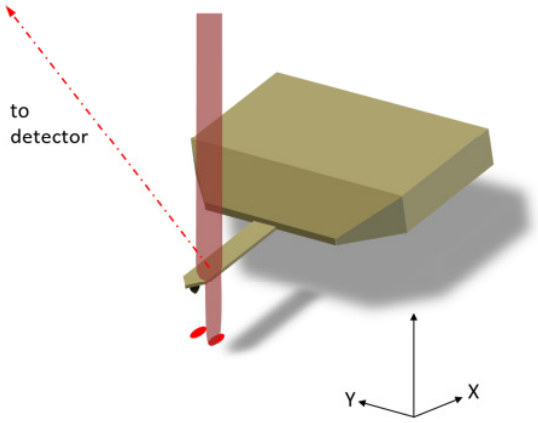


Figure 11: When letting the laser spot traveling along the front edge of the cantilever substrate, it vanishes for a short moment. At this moment it is shadowed by the cantilever and the corresponding position indicates the right deflection in y-direction. Now, maximize the sum signal by a fine adjustment in y-direction. During the second step, travel along the –x-direction. The laser will leave the cantilever at its front edge and the sum will reduce to zero. Travel back until the sum signal reaches its maximum and stop.

OPTION B

3.1. Focus on the sample surface by changing into focus surface mode


Focus Surface icon of the GUI

3.2. In the focus surface panel chose *Tip reflection*
3.3. Identify the tip and the laser spot (blue-green) in the *Vision Image* on the *Display Monitor*
If laser spot and tip are not visible, please proceed with *OPTION A*
3.4. Move the laser spot towards the tip or the corresponding crosshair.
3.5. Maximize the sum signal by a fine adjustment in y-direction
3.6. Travel along the –x-direction until the laser leaves the cantilever at its front edge and the sum reduces to zero
3.7. Travel back until the sum signal reaches its maximum, but don't travel further.
3.8. Finish *Focus Surface* mode.

4. Align the reflected laser beam on the photo detector by turning the detector adjusting screws on the side of the AFM head, as indicated in Figure 7
4.1. First, align the red spot on the laser screen on the AFM head in the center of the window
4.2. Then, align the red spot in the detector schematic on the *Display Monitor* of the computer screen in the center of the crosshair.
4.3. Finally, the vertical deflection, below *Vert Defl* should be adjusted to nearly 0 (smaller ±0.1)
5. Perform an Auto Tune of the cantilever to adjust the work point of the tip oscillator.
5.1. Chose the *Cantilever Auto Tune* menu


Cantilever Auto Tune icon of the GUI

5.2. Click on *Auto Tune* in the *Auto Tune Controls*
5.3. Check if the microscope adjusted the oscillator to reasonable values
5.4. Click on *Back to Image Mode*

OPTION C
Chose the *Stage* pull down menu and click on *Exchange tip*
Follow the given advices
For more information about each step, see details of *OPTION A*

The optical lever of the microscope is set up now.

5. Sample mounting

This section describes two options to load and unload a sample. For both ways, it is necessary to have a tip aligned. Otherwise all drives are blocked if the sum signal is too low or zero.

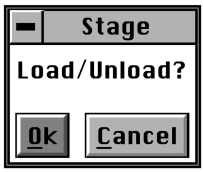
1. Check the clearance underneath the AFM tip
2. Eventually, increase the distance between chuck and tip by *Withdraw Tip*


Withdraw Tip icon of the GUI

To increase the distance even more, it is possible to use this option several times.

OPTION A

3. If the chuck is under the AFM head, use the *sample load/unload* menu in the *stage* pull down menu to move the chuck into the loading position



Load/Unload menu

4. Confirm the decision by clicking on *Ok*, and the stage will travel to its front position
5. Position the sample on the hole for the vacuum fixing
6. Switch on the chuck vacuum
7. Use the *sample load/unload* menu in the *stage* pull down menu to move the chuck back underneath the scanner of the AFM head
8. Confirm with *Ok*

OPTION B

3. Chose *Focus Surface* to enable a movement of the stage by the track ball


Focus Surface icon of the GUI

4. By using the *Focus* button and the track ball the stage travels in z-direction
   Pay attention to the distance between AFM tip and surface

5. Using the track ball without any additional option moves the stage stepwise by a dynamic control
   - fast movement of the track ball results in a fast movement of the stage
   - slow movement of the track ball allows a slow and precise movement of the stage
   - Holding a *Lock* button and rolling the track ball one time results in a continuous movement of the stage

6. Move the chuck away from the AFM head

7. Position the sample on the hole for the vacuum fixing
8. Switch on the chuck vacuum by the switch shown in Figure 5
9. Move the chuck with the sample back to the scanning position
10. Finish the *Focus Surface* mode

6. Start measurement

As the AFM covers about 5 orders of magnitude, each in x-, y- and z-direction, topographies of different kind can be measured. However, micrometer large grains require different scanning parameters compared to small nanostructures. As a result, the following section presents only a starting point for AFM measurements.

1. Align the sample under the AFM tip
   1.1. Select *Stage→ Focus Surface* or chose the Focus Surface button


Focus surface icon of the GUI

   1.2. Use the track ball and *Zoom* function to move the area of interest under the cross hair
   1.3. Click on *Ok* to end the *Focus Surface* menu.

2. Close the acoustic enclosure
3. Press the engage button for the tip sample approach


Engage button of the GUI

During surface approach, the tip should be in focus.
- Otherwise, press *Abort*!

4. Wait until the *Vision System* image of the optical microscope is replaced by the scanning NanoScope Image.
5. Adjust *Scan size* and *Tip velocity* to appropriate values
   5.1. The scanning range should cover some features, which provide a meaningful profile for an adjustment
   5.2. The tip velocity should not exceed 10 µm/s
   5.3. The slower the tip velocity, the better the image quality
6. Change into the scope mode

7. Adjust the data scale of the channels, so that the profile covers 50 – 70% of the image height of the scope mode.
8. Adjust the scan parameters
   8.1. Check if trace and retrace profiles are comparable – same look, not the same position
      8.1.1. If they are not the same, as indicated in Figure 12, decrease the setpoint stepwise for 0.025 V
      8.1.2. If they are the same: increase the setpoint stepwise for 0.025 V until their shape diverges. Then, decrease the setpoint, e.g. for about 0.050 V. Now, both traces should show a comparable profile with ideal sample tip interaction.

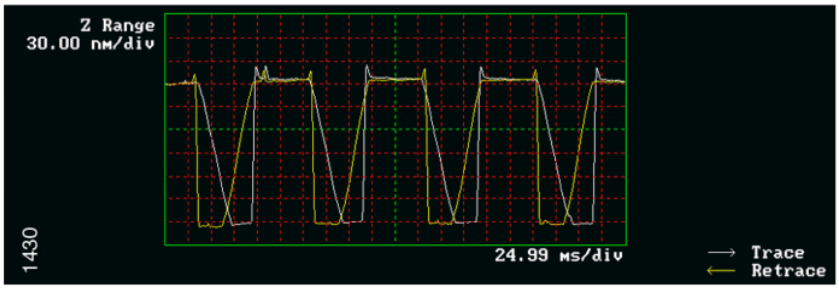


Figure 12: Setpoint too high

8.2. Adjust the integral gain

8.2.1. If the profile is noisy (see Figure 13), the integral gain is too high.

8.2.2. If the profile appears edgeless where edges are expected (see Figure 14), the integral gain is too low.

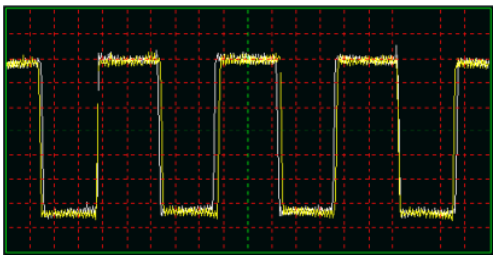


Figure 13: Integral gain is too high

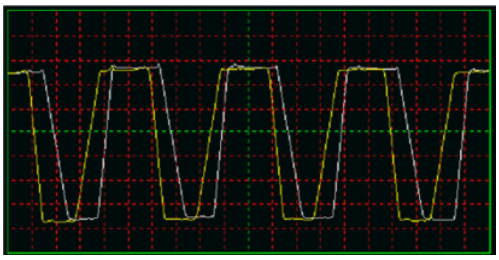


Figure 14: Integral gain is too low

8.3. Eventually, readjust the tip velocity

Remember: $Tip\ Velocity = 2 * Scan\ Rate * Scan\ Size$

The Tip Velocity is two times the scan size because one line consists of trace + retrace!

8.4. Adjusting the scan parameters like tip velocity, set point and integral gain is typically an iterative process and needs to be performed frequently whenever the scan size and scanning position is changed.

9. Detailed search for the area of interest

9.1. Don't move or touch the stage while scanning

9.2. The AFM head piezo can cover a range of 100 µm in x- and y-direction

9.3. Increase the scan area size to a reasonable value
Consider:
Large areas give a good overview over the scanning position
However, scanning large areas takes a long time

9.4. Decrease the scan resolution (*samples/line* in the *Scan Controls* menu) if possible to increase scan speed

9.5. Move the scanning area by setting offsets in x- and y-direction, either directly by inserting values into the *X offset* and *Y offset* of the *Scan Controls* menu, or by selecting *Offset* in the taskbar of the NanoScope Image. A mouse pointer in form of a green crosshair will appear on the scan images. This can be positioned by the mouse and fixed by a left click. Afterwards, press *Execute* to shift the scan area and *Clear* to remove the green crosshair.

9.6. Once the area of interest is in the center of the scanning image, adjust the scan size, either by inserting a value into the *Scan Size* box of the *Scan Controls* or by the *Zoom In* and *Zoom Out* function in the task bar of the *NanoScope Image*.
- Move the selection square with the mouse
- Change between moving and zooming by the left mouse button
- Confirm your selection by the right mouse button.

9.7. Increase the scan resolution (*samples/line* in the *Scan Controls* menu) to an appropriate value

7. Recording images

The purpose of SPMs, more precisely AFMs, is the recording of images. The NanoScope starts to take, means to record and save images on instruction. It is not possible to save an image of an already performed scan. Therefore, it is necessary to activate the capturing process, once all parameters are set and the area of interest is scanned.

1. Give in a name for your next image or a series of images by selecting the *Capture* pull down menu and *Capture Filename*
   The rules for naming a file are as follows:
   - The specified filename must be DOS compatible
   - The filename may be up to 32 characters in length
   - A three-digit extension will be appended to the file name. The extension is sequentially numbered after each image is captured. If an extension is specified as part of the file name, the labeling will begin with the specified extension number.
   - Some software, like Gwyddion, can open images with an arbitrary extension, also with indexing numbers

2. Capturing an image can be performed either by the *Capture Image* icon or by activating *Caption* in the *Caption* pull down menu, or press ***CTRL + C***.

    Capture Image icon of the GUI

3. The current event is shown in the *Status Bar* at the lower edge of the *Real Time Control Monitor.*

   The status reflects the following options:
   - **On** - Indicates that the Capture mode is in progress
   - **Off** - Indicates that the Capture has stopped
   - **Next** - Indicates that the Capture mode is waiting for the next frame to begin capturing
   - **Done** - Displays when the Capture finishes
   - **Movie** - Displays when the Continuous Capture mode begins
   - **Forced** - Indicates a Forced capture. This is done by clicking the Capture command a second time. The advantage is that the capture begins or resumes without waiting for the start of a new frame

4. If any scanning parameter is changed while the machine is capturing, the present scan is rejected and the status in the *Status Bar* changes to *Next* indicating that the next image will be captured
5. If the capturing process should be performed on the present scan and not the following one, repeat the capture activation (2) until *Forced* is stated in the *Status Bar*
6. To stop the recording of an image chose the *Capture* pull down menu and *Abort* or use the *Abort* icon, or press ***CTRL + A***

    Abort Capture Image icon of the GUI

7. All captured images can be seen in the *Offline Mode*

    Offline mode button

8. All captures images can be found in the capture folder on the system drive
   A corresponding shortcut can be found on the *Desktop*

8. Shutting down the AFM

After finishing the last measurement, the following steps are required to transfer the AFM into its common stand-by mode.

1. Click on withdraw to stop the scanner and to retract the tip from the sample surface


Withdraw icon of the GUI

2. Reset all parameters to normal values:
   Panel *Scan Controls*
   - Scan size: 1 µm
   - Aspect ratio: 1:1
   - X-offset: 0
   - Y-offset: 0
   - Scan angle: 0
   - Scan rate: 1 Hz
   - Tip velocity: 2 µm/s
   - Sample/line: 128
   - Lines: 128
   - Slow scan axis: Enable

   This prevents an accidental load on the tip and sample surface after the next approach.

3. Change into the offline mode

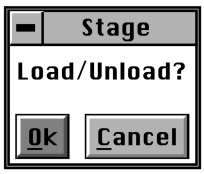

Offline Mode icon of the GUI

4. Check each of your images by double clicking on the filename in the list
   This closes the image file and allows an editing with external software, e.g. Gwyddion.

5. Change back into real time mode


Real Time Mode icon of the GUI

6. Click on the **Stage → Load New Sample** option



Load/Unload menu

   Click on *Ok*

The AFM head will be raised (z-axis) to a safe position.
The stage will move the chuck with the sample to the front center position.

7. Switch of the chuck vacuum and remove the sample with plastic tweezers

8. Click on the *Stage → Load New Sample* option
   Click on *Ok*
   The chuck is moved back into its original position

9. Released the clamp of the AFM head by tightening the knurled screw on the right of the AFM head mount groove

10. Pull the AFM microscope head gently out of the Z-stage AFM mounting groove

Handle the AFM head with care!
Hold it safe in your hand
Never let it drop down or knock against the stage!

11. Turn the AFM head around (upside down) and release the AFM tip holder

Don't look into the laser beam or its reflection before it switches off automatically!
Don't stretch the connection cable between AFM head and stage!

12. Put the AFM tip holder on the cantilever installation fixture

13. Install the AFM microscope head without tip holder by carefully sliding its dovetail into the Z-stage's mounting groove
    Push it to the end of the groove and lock it by releasing the knurled head clamp screw, located at the upper right of the Z-stage, until the thread is just loosened plus approximately 1-1.5 turns

14. Remove your tip from the tip holder

15. Leave the tip holder in the cantilever installation fixture

16. Close the acoustic shielding box

17. Switch off the AFM if no user booked it afterwards:
    - Controller
    - Stage power supply
    - Microscope lamp
    - Vacuum pump

18. Shut down the AFM controller software, but leave the computer on
19. Copy your files from the "captions" folder to your destination folder or device