

VEECO DIMENSION 3100 ATOMIC FORCE MICROSCOPE



USER'S MANUAL

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I – Principles of Atomic Force Microscopy (AFM)

Unlike optical or electron microscopy (SEM, TEM), Atomic Force Microscopy, and more generally Scanning Probe Microscopy, do not depend on the interactions between a photon or an electron beam and the sample. SPM techniques are based on the measure of the tunnelling current (in the case of Scanning Tunnelling Microscopy) or the local forces (in the case of AFM) between a tiny tip, which is supposed to be monoatomic, and the sample's surface.

I-1 – Contact Mode AFM

The first AFM technique to be developed was the Contact Mode AFM; it is basically what a blind person does when reading a document in Braille: scans the surface and feels the “rugosity” on the surface.

From a technical point of view, the tip is mounted on a small cantilever which is connected to the AFM head. Usually, the system is designed in a way that the curvature of the cantilever is kept constant and the changes of its height are recorded. There are several ways to monitor the cantilever's height; the most common is the optical detection. A laser beam is reflected by the back of the cantilever to a detector made of 2 or 4 photodiodes. Every move of the cantilever results in a change in the ratio of light intensity received by the different photodiodes. This set-up is schematically represented on Figure 1. A feedback loop is integrated into the system and generally adjusts the height of the cantilever in order to keep the intensity received by the photodiodes constant.

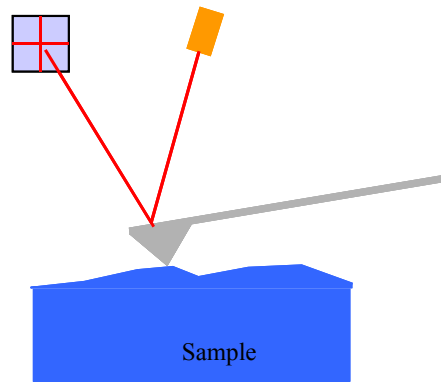


Figure 1: Schematic representation of Contact mode AFM.

Contact-mode AFM has led to great achievements such as atomic resolution imaging on insulators – it had already been achieved for metallic and semi-conducting samples using STM -, nanolithography or nano-oxidation. But it is not suitable for the study of sensitive surfaces - the tip can damage the surface- or nanostructures – the tip will remove them. For these purposes, tapping and non-contact modes have been developed.

I-2 – Tapping and Non-Contact AFM

For those techniques, the system induces vibrations of the cantilever at a frequency close to its resonant frequency; there is a piezoelectric drive in the tip-holder to control the vibrations. In tapping mode, the tip periodically touches the surface and the system tries to keep the amplitude of the vibration constant. Tapping mode is the technique we use most of the time for basic imaging.

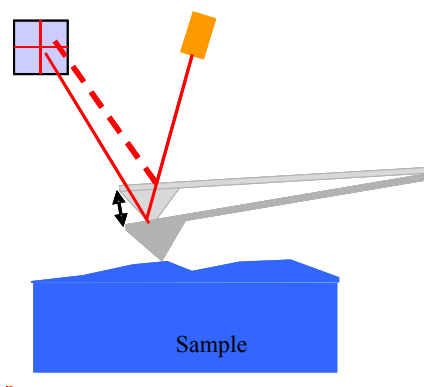


Figure 2: Schematic representation of Tapping mode AFM.

Non-contact mode is based on the same technical principles, the only difference is that the cantilever is a bit higher and the system detects the vicinity of the surface. It is also a lot more complicated to set up than tapping mode.

I-3 – The other AFM techniques

The main advantage of Atomic Force Microscopy is its versatility: to my knowledge, there are dozens of properties which can be analysed using techniques derived from the basic principles described earlier. In the ATI, a few techniques are available: Lateral Force Microscopy (LFM), Conductive-AFM (C-AFM), Scanning Capacitance Microscopy (SCM), Magnetic Force Microscopy (MFM). We also have the nanomanipulation option which allows us to do nanolithography and nano-oxidation. If you want more details, just ask Cristina or myself.

I-4 – The AFM tip

As there are a lot of different AFM techniques, a lot of different tips are available on the market. They can be made of different materials, the most popular being Silicon and Silicon Nitride. Different coatings can be used, depending on the application. For instance, a metal like chromium or platinum is used to coat C-AFM tips, a magnetic alloy based on cobalt for MFM tips. You can also find chemically functionalised tips which would be used to detect one type of molecules on a surface.

The other important factor for a tip is the aspect ratio. I show three types of tips on Figure 3. On the left, the pyramid-shaped tip is the most common and the cheapest type of tips, the one we have here for basic use. In the middle, the so-called ATEC tip is very useful if you want accurate positioning because you can see the tip from the top. On the right-hand side, I show a picture of a High Aspect Ratio tip, very useful for accurate imaging of complex structures. Unfortunately, ATEC-tips and even more High Aspect Ratio tips are very fragile and expensive. Therefore, they must be kept for very special experiments.

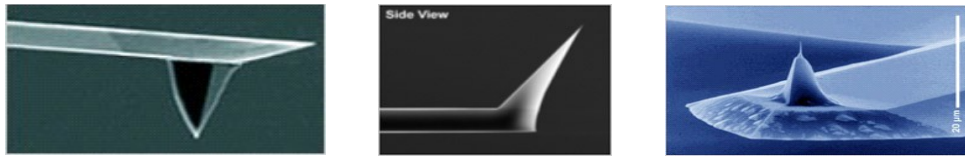


Figure 3: Examples of AFM-tips.

The last thing to mention about tips is the reflective coating of the cantilever, a metallic coating like Al increases the intensity received by the detector.

I-4 – The piezo-electric scanner

Scanning Probe Microscopy wouldn't be as good as it is without the piezo-electric scanner. Just to remind you, a piezo-electric material is a crystalline compound which size changes when a bias is applied. A diagram of a cylindrical scanner is represented on Figure 4. Basically, the horizontal (x,y) deflection is used to scan the sample's surface and the vertical (z) deflection is used to adjust the height of the cantilever.

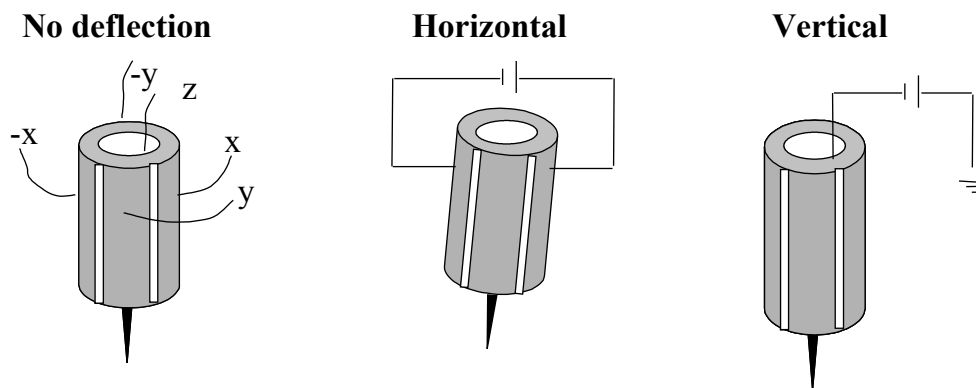


Figure 4: the piezoelectric scanner.

Cylindrical scanners are the most popular scanner for two reasons: they are robust and allow scanning relatively quickly. But, unfortunately, their shape induces a few artefacts during imaging.

II – The Veeco – Dimension 3100

This Atomic Force Microscope is located in the nano-electronics labs (17DJ00). Apart from Contact and Tapping mode AFM, it allows doing non-contact AFM, Lateral (LFM) and Magnetic (MFM) Force Microscopy in the basic set-up. It is also equipped with two modules for Scanning Capacitance Microscopy and Conductive-AFM. As we also have a XY closed-loop scanner and the nanomanipulation option of the software, we can do nanolithography and nano-oxidation

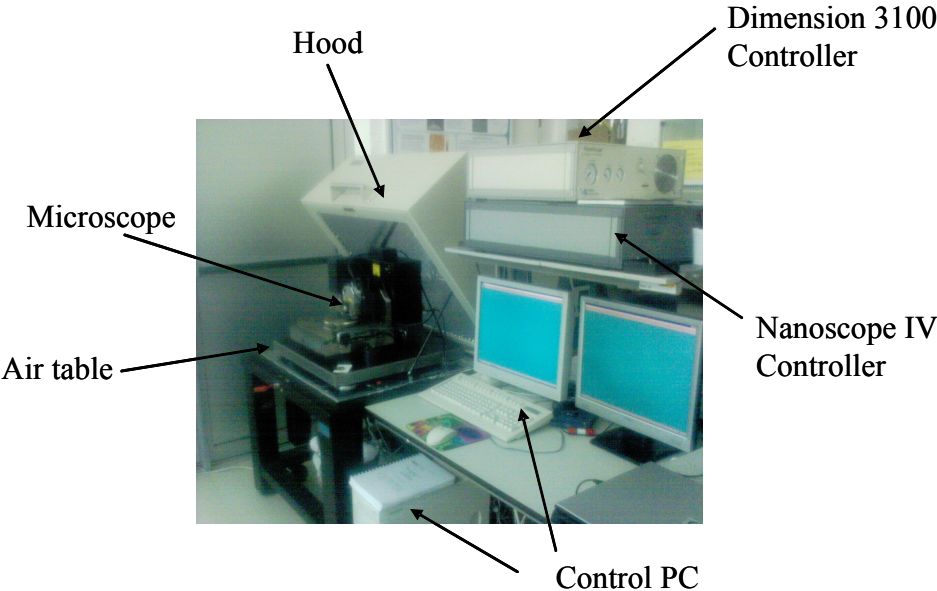


Figure 5: picture of the Dimension 3100 – general view

The microscope is installed on a marble table insulated from the ground by four air pillows, this system protects it from most vibrations generated by pumps, chillers, compressors, vehicles in the car park or by the building itself. Furthermore, it is protected by a

hood which protects it from two others sources of noise: vibrations transmitted from the atmosphere (sounds) and magnetic or electric fields (essentially for options like MFM, SCM and C-AFM). It is connected to two controllers: the Nanoscope IV controls the AFM head and communicates data to the control PC, the Dimension 3100 monitors secondary functions like the optical microscope or the sample stage. In normal condition, we can scan areas between $500 \times 500 \text{ nm}^2$ and $60 \times 60 \text{ }\mu\text{m}^2$ in the XY plane; in the Z direction, we can detect anything between 1 nm and 500 nm high. The resolution is 20 nm in XY (because of the tip convolution) and 0.1 nm in Z. Note that the use of a High-Aspect-Ratio tip will improve the XY resolution.

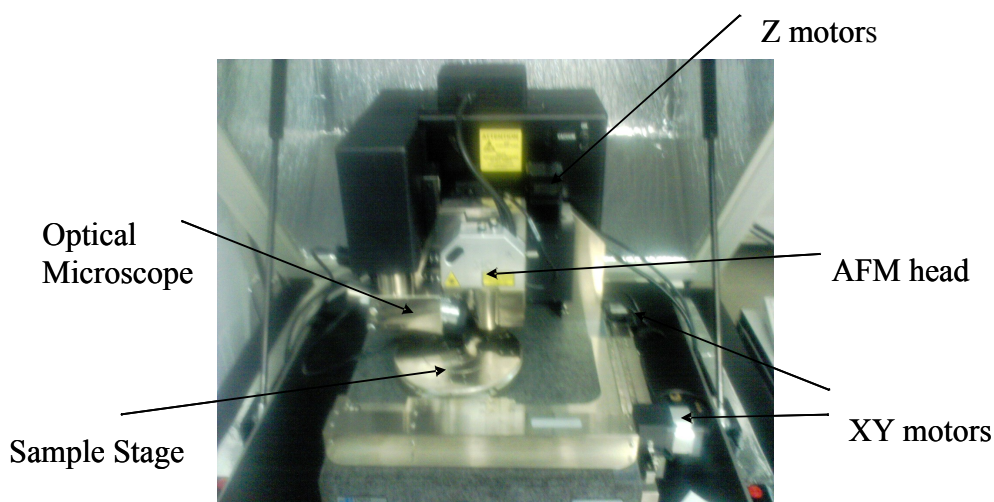


Figure 6: picture of the Dimension 3100 – detailed view

The detailed view of the microscope shows the three main parts of the microscope:

- the AFM head which position is controlled by the Z motor,
- the sample stage controlled by the XY motors
- the optical microscope which allows the user to choose a location on the sample.

The AFM head is shown on figure 7. Its main components are the laser source, the scanner and the detector. The laser source is located in the top right corner of the head, the bright red LED shows it is on. The scanner is in the metallic cylinder at the bottom. The

detector is located at the top right, the black window with a red dot give a view of what the detector records. The cubic box on the right of the scanner is the sensor for C-AFM (we also have one for SCM). Finally, there are two knobs on the top side and two others on the left-hand side of the head which are used to align the laser beam and the detector, respectively.

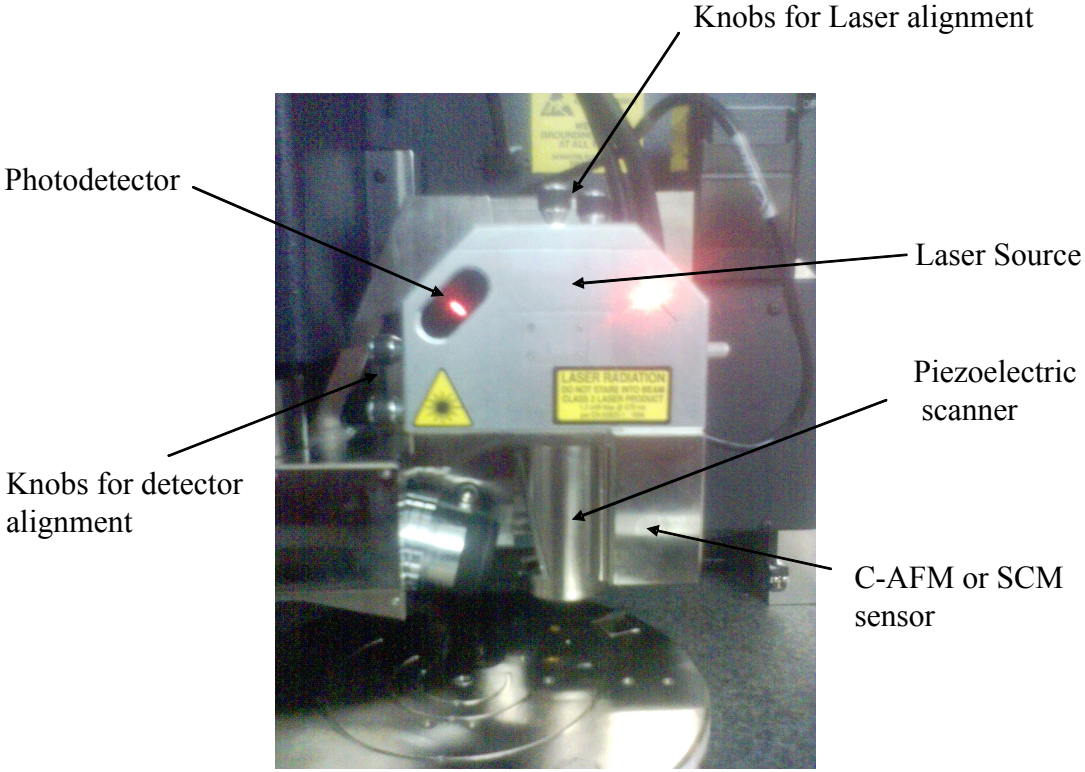


Figure 7: picture of the AFM head

III – Making an experiment

III-1 – Starting the machine

This is pretty basic, you have to start the computer and the two controllers, the switches are on the back side of the controllers (on your left when you face the desk). People at Veeco recommend starting the Nanoscope IV controller first as it gives instructions to the dimension 3100 controller. The computer runs with windows NT4.0 and has no password. Then, launch the software called Nanoscope 6.12r1 by double-clicking on the icon in the bottom-left corner of the desktop. Once the software is on, you have to turn on the communication between the PC and the controllers; you do it by clicking on the RealTime icon. It represents a yellow optical microscope (Figure 8)

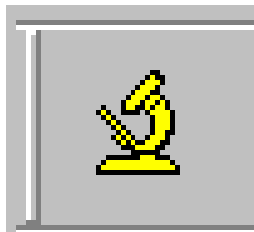


Figure 8: the RealTime icon

The system needs a few minutes to set up the communication; you can use it to prepare your sample.

III-2 – Preparing a sample

The biggest advantage of AFM is the simplicity of the sample preparation; there is no need for metal coating or complicated cleaning techniques, unless it is required for another reason. Having said that, it is always better to work with a clean sample; in the lab, we can blow dry nitrogen on the sample to remove dust or use a solvent like acetone or isopropanol on the chemical bench.

Two options are available with the dimension 3100. For small samples, less than 1 cm large, the best solution is to stick it onto a SPM specimen disc using a non-conductive sticky pad, to put it on the magnetic sample holder (see figure 9 for details) and to install it on the sample stage.

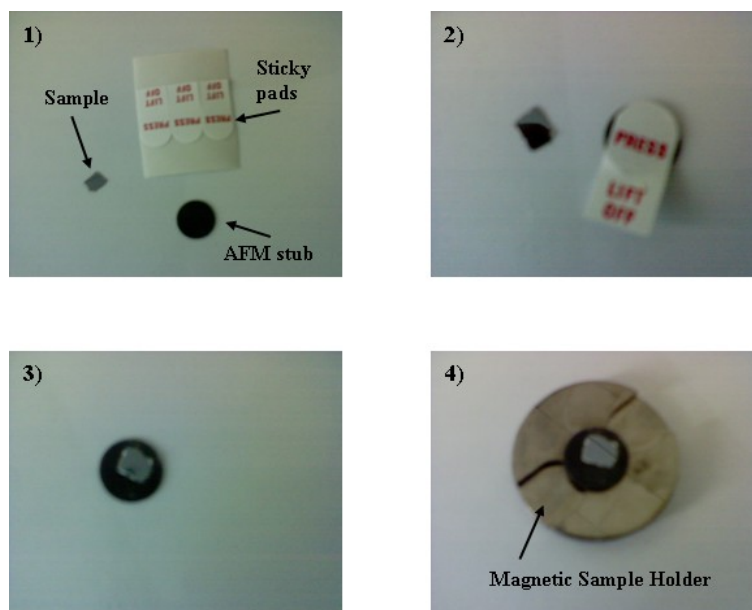


Figure 9: preparation of a small sample.

For a bigger sample (wafer, CD, glass slide...), it is even easier: just put it on the sample stage and switch on the aspiration to make sure it will not move during the experiment. You do it by turn the switch called vacuum (top right corner of the microscope) to ON. Once the sample is ready and installed on the stage, you can come back to the soft and set up the experiment.

III-3 – description of the software

The software should now be ready and a window asking you which views you want to add to RealTime should appear. Choose Select All and OK so that you have all the options available.

Before we go further, I am going to describe the different windows of the software (Figure 10).

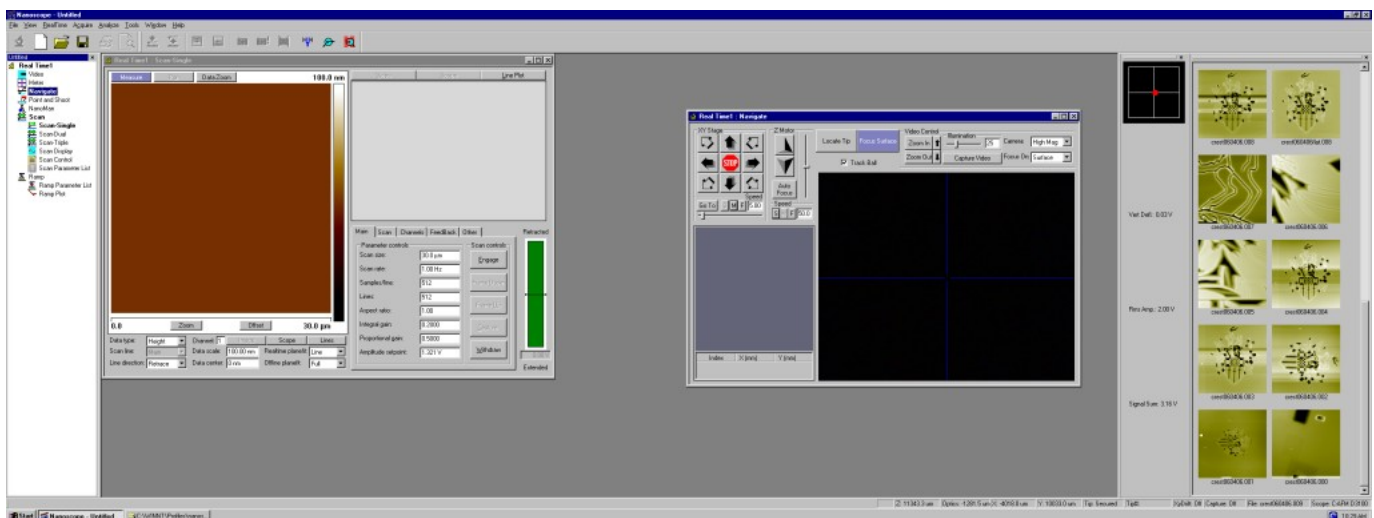


Figure 10: the Nanoscope 6.12r1 software

Like any other software in a windows environment, Nanoscope 6.12r1 has a menu and a tool bar both located in the top left corner. There is also a status bar in the bottom right corner. My advice would be to keep an eye on it, it gives useful information. The screen is then divided into four windows: from left to right, the workspace window, the client window, the RealTime status window, and the image window.

III-3-1 – The workspace window

It works like windows explorer and shows all the available options. If you want one thing which is not in that menu, you can add it from the acquire option of the menu bar. To open anything in the workspace, just double-click on its name and it will appear in the client window.

III-3-2 – The client window

The client window is the main part of the user's interface; it is composed of several windows corresponding to functions which can be called from the workspace menu:

- the "video" gives a real time view of the image received by the optical microscope (Figure 11).

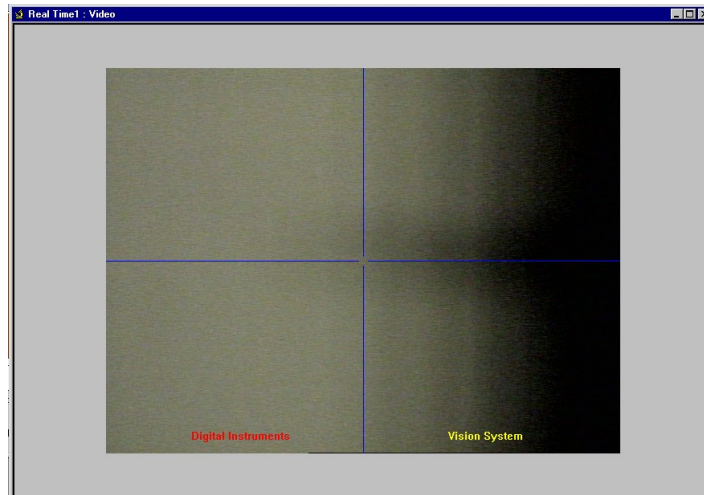


Figure 11: the video window

- the “meter” shows the status of the detector when the tip is not engaged, it is important when you start an experiment (Figure 12).

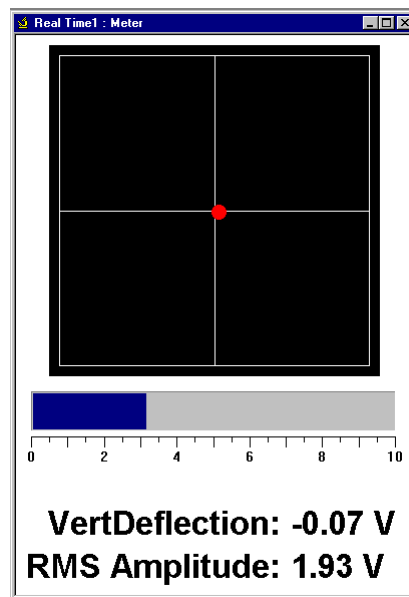


Figure 12: the meter.

- the “navigate” window is a very important part of the software, it is the interface between the microscope and the user when the tip is not engaged (Figure 13). It will be described in details later.

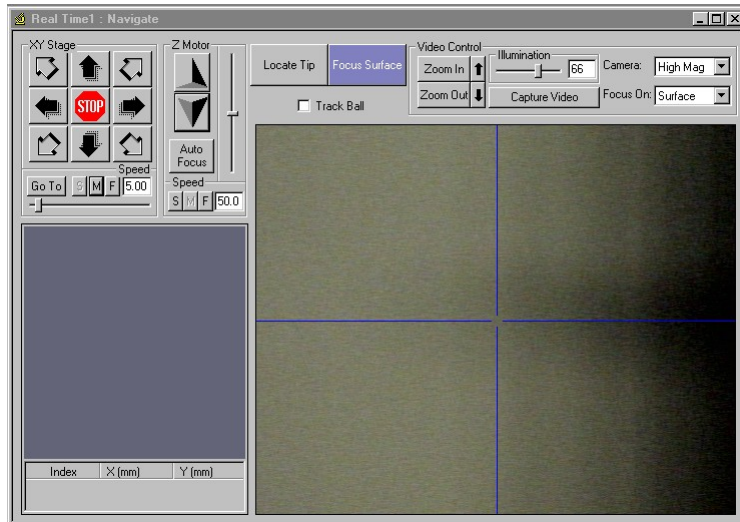


Figure 13: the navigate window.

- “Point and Shoot” and “Nanoman” are two options that are not really relevant for basic imaging. “Point and Shoot” helps you choosing points to do force spectroscopy (or $I(V)$ measurements in C-AFM). “Nanoman” is the software for nanomanipulations.

The different scan options:

- “Scan single” is designed for people who want to record one type of image, it contains a view of the image being recorded (on the left), a plotter which can show the video image, a scope of the scanning line being recorded or a plot of all the lines which have been recorded for this image, the last part is a scan control panel in the bottom right corner (Figure 14).

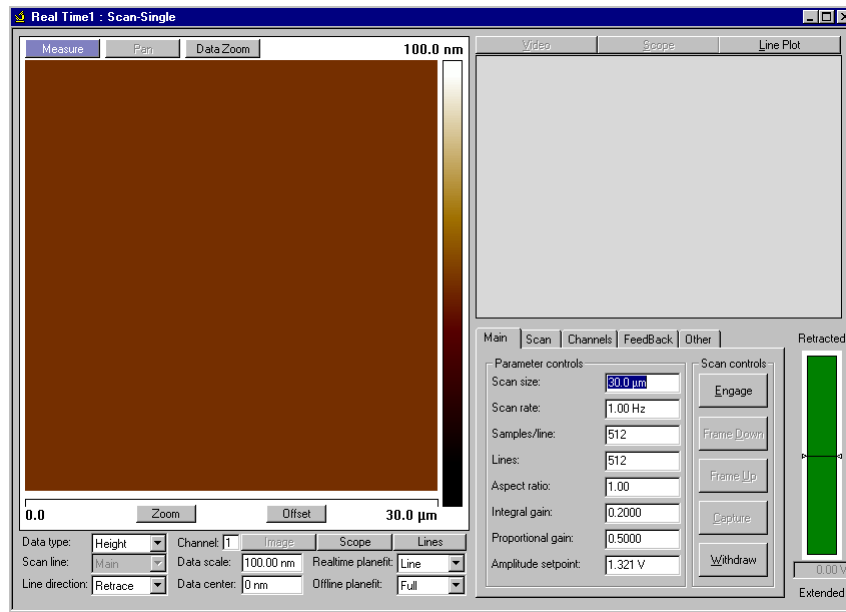


Figure 14: the Scan single display.

- “Scan Dual” shows the same kind of information for two channels, the control parameter list on the right is a bit messier than the control panel in “scan single” (Figure 15).

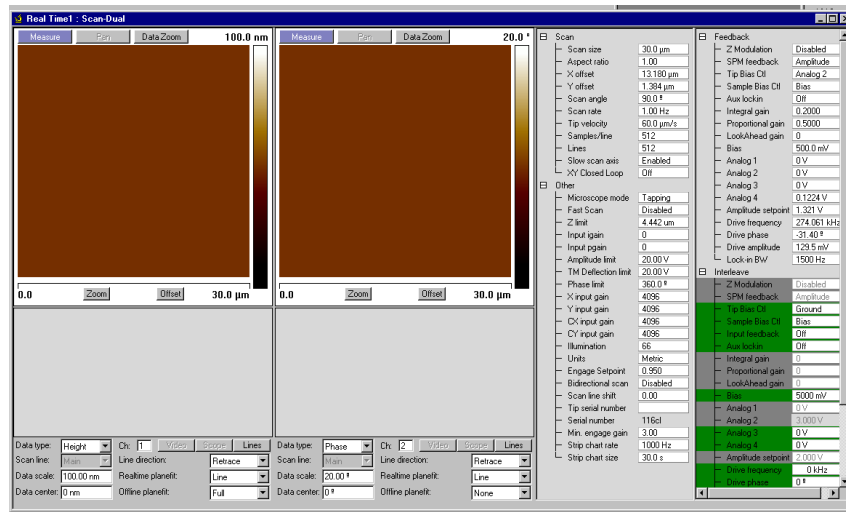


Figure 15: the scan dual display.

- “Scan Triple” is exactly the same than “scan dual” but with three channels (Figure 16).

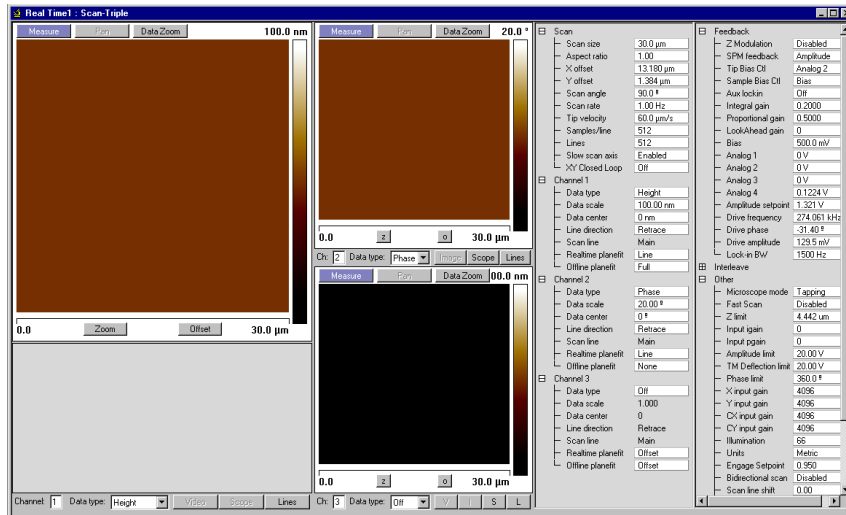


Figure 16: the scan triple window.

- “Scan display” shows the image or the scope of any channel. There is a useful trick: right-click on the bottom of the image and select “show channel parms” to have the options you see at the bottom of the scan display (Figure 17).

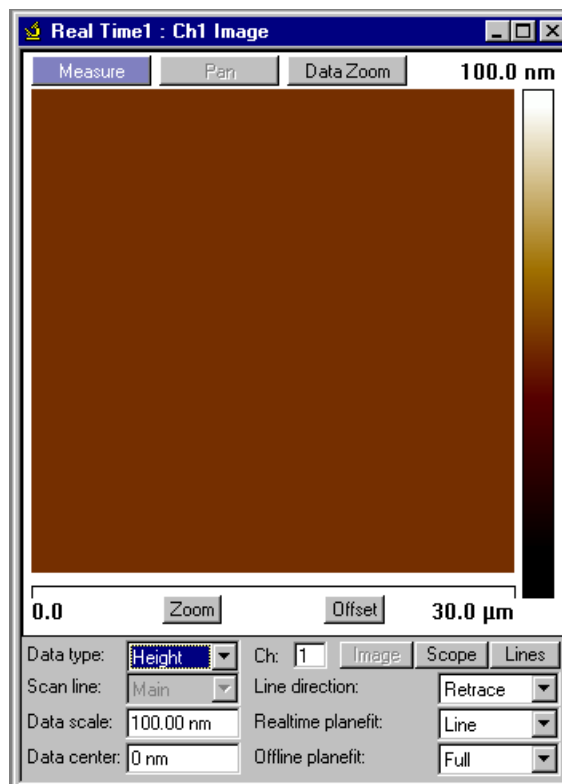


Figure 17: the scan display.

- “Scan Control” is the control panel for the scan; it is also something I will describe more accurately later.
- “Scan Parameter List” shows the same information than “scan control” but in a messier way.
- The “Ramp” options are for spectroscopy.

III-3-3 – The RealTime Status window

Located at the right of the client window, it shows the status of the detector when the tip is not engaged and the extension of the piezoelectric scanner when the tip is scanning the surface.

III-3-4 – The Image window

At the extreme right of the screen, it shows thumbnails of the images in the selected folder. If you double-click on an image, it will be opened by the analysis part of the software.

III-3-5 – Saving your workspace

Once you have a workspace you like, you can save by selecting “save workspace as” in the file menu. To prevent us from a big mess in the computer, please save your workspace in your personal folder (D:\abusers\yourname).

III-4 – Initializing an experiment in tapping mode

There are four things the user has to do before he (or she) can engage the tip safely and start scanning.

III-4-1 – Checking the laser is aligned

As you know, the laser beam is reflected by the beam to the 4-photodiodes detector. To be sure you are not wasting your time, it is better to check the laser beam, the cantilever and the detector are aligned. Normally, we only need to align the laser with the cantilever when we change the tip but it is usually necessary to re-align the detector with the reflected laser beam. To check it, just open the meter (Figure 18).

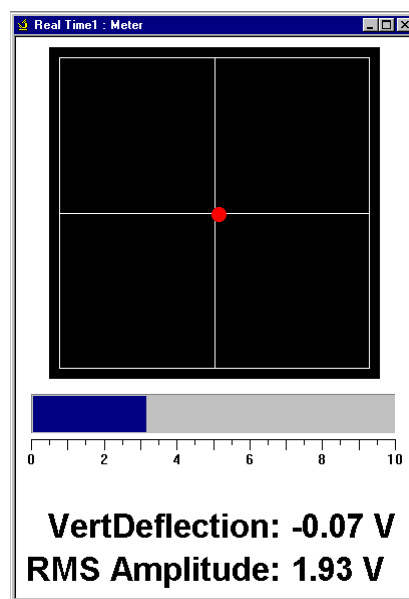


Figure 18: the meter

It shows a diagram of the four photodiodes with a red dot which represents the place where the laser reaches the detector. If everything is aligned, the red dot should be in the

centre of the diagram. The blue bar corresponds to the intensity received, it should be around three with the tips we use. “VertDeflection” corresponds to the distance between the horizontal line and the red dot, it should ideally be zero. “RMS” amplitude is the amplitude of the cantilever’s vibration in tapping mode. If the detector is not aligned, you have to move the detector with the two knob located on the left-hand side of the AFM head (see figure 7). For any other problem, please ask Cristina or me.

III-4-2 – Locating the tip

To be able to engage safely, you must tell the system where the tip is. To do so, open the navigate window in the Workspace and click on the locate tip option, the window shows a top-view of the cantilever (figure 19).

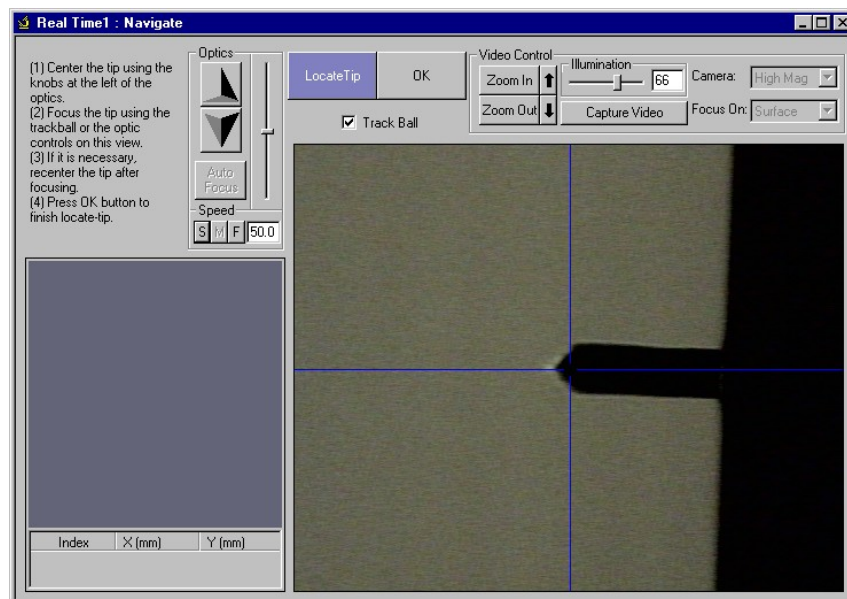


Figure 19: the “Locate Tip” window

You must then focus on the cantilever using the arrows labelled “optics” until you get a sharp image. They control the optical microscope. To help, you can change the speed of the optics motion using the slider on the right of the arrows or the presets (S for slow, M for

Medium, F for fast). You can also change the intensity of light with the slider called “Illumination” or use the Zoom (arrows labelled “Zoom In” and “Zoom Out”). If the cantilever is not in the centre of the image, you can move the optical microscope using the two knobs located on the camera (extreme left of the microscope).

Once you are happy with the focus of the tip, click on OK and come back to the navigate window to focus on the surface and choose the location you want to analyse.

III-4-3 – Focusing on the surface

The “Focus Surface” part of the navigate window is a bit more complicated than the “Locate Tip” window. The series of eight arrows in the top-left corner control the XY-stage’s motion and the two arrows on their left controls the Z-motor (see chapter II). Both controls have a slider for the speed. You will also recognize the illumination and zoom options. On the right, two sliding menus are now available. The top one allows to choose between High and Low Magnification for the optical microscope but we just have High Mag (pretty useless, isn’t it). The second one gives the choice between two focuses: on the surface or on the tip’s reflection. When it is possible, I recommend focusing on the tip’s reflection and checking the image of the surface is nice. The distance between the tip and the surface should be close to 1.5 mm.

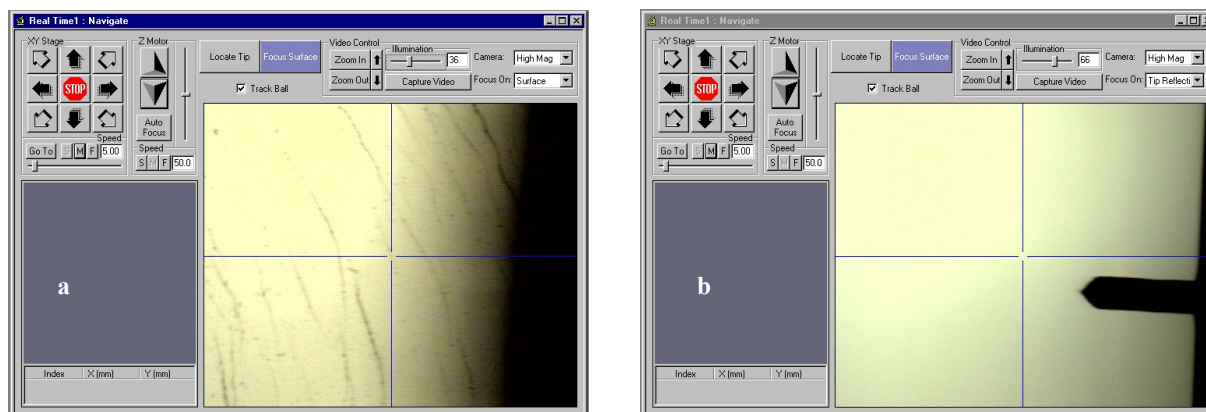



Figure 20: the “Focus Surface” window with Focus on the surface (a) and the tip’s reflection (b).

Once the microscope is focused on the surface, you can use the eight arrows to move the sample and choose the area you want to image. The stage is a bit tricky to control; you will need time to get used to it. Note that you can also use the navigate window to bring the sample under the scanner before you focus but, please, make sure the scanner is high enough to do this.

III-4-4 – Tuning the cantilever


This function is specific to Tapping-mode AFM; the system will basically find the resonant frequency of the cantilever and adjust the frequency of the piezoelectric drive. To do

so, click on the “Tune” icon  and a window showing two graphs and a lot of parameters appears. All the parameters are set up for basic imaging, you just have to click on “Auto Tune” in the bottom-left corner and wait a bit. The system will determine the resonant frequency of the cantilever, adjust the maximum amplitude to a preset value (corresponding to 2V for the detector) and set the drive’s frequency to a value which is 5% below the resonant

frequency. We usually do not scan at the resonance frequency because the cantilever can start resonating and break.

III-5 – Engaging the tip and imaging

Once you have checked the alignment, located the tip, focused on the surface and tuned the cantilever, you can engage the tip and start imaging. You can use the scan control

display or the “engage” icon in the tool bar . The AFM head will move quickly to a position which is 100 μm higher than the surface and then go step by step until the tip “touches” the surface.

During a scan, all the parameters we need to control are available in the scan control display (Figure 21) which appears in the “Scan single” window or on its own.

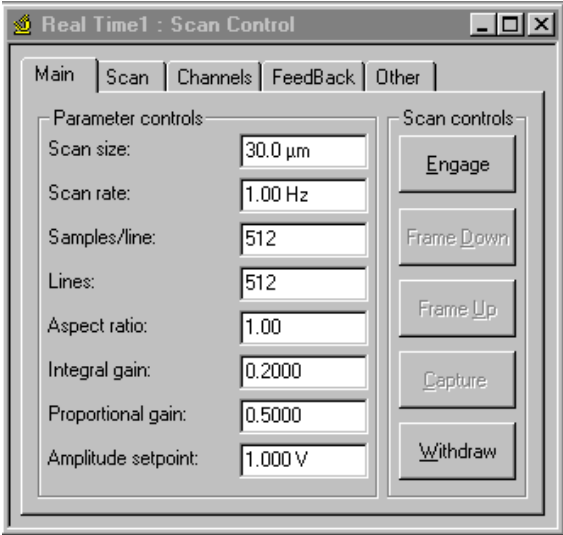


Figure 21: the “Scan control” display

The control display is divided into five tabs dedicated to different parts of the system.

“Main” shows two windows. The parameters controls shows different parameters you can change, it can be customized. The default parameters are Scan size, Scan rate, Samples/line and Line correspond to the number of pixels, Aspect ratio decides whether you scan a square or a rectangle, Integral and Proportional gains define the sensitivity of the feedback loop, Amplitude setpoint corresponds to an amplitude the system tries to keep constant. “The scan controls” box contains five basic orders: Engage or withdraw the tip, Frame Down and Frame Up to restart the image from top or bottom and Capture to save the image.

“Scan” shows different parameters associated with the scan. Scan size, aspect ratio and scan angle don’t need more comments. X and Y offsets correspond to an offset of the centre of the image with regards to the position of the scanner if no XY bias is applied. Scan rate corresponds to the inverse of the time required to scan one line and come back. Tip velocity is the actual tip’s speed; it corresponds to “2 x Scan size x Scan rate” because the tip scans each from left to right (“trace”) and right to left (“retrace”). The resolution (Samples/line and Line) is usually set at 512 x 512 pixels. Slow scan axis has to be enabled otherwise you will always scan the same line. Finally, the XY closed loop has to be off.

The third tab corresponds to the three channels of the system, the three types of data you can record for the same image. This tab is divided in three similar menus corresponding to channel 1, channel 2 and channel 3. Data type allows you to choose between eleven options including height, phase, amplitude and off. Data scale corresponds the colour scale bar; it has to be adjusted with regard to what you are imaging. “Data centre” allows you to offset the colour scale if necessary. Line direction gives you the choice between two options: trace (left to right) or retrace (right to left) for the line which will be recorded. “RealTime plane fit” and “offline plane fit” are the background corrections for the image you see and the one which

will be captured in the end. Usually, we use channel 1 to record the height image and channel 2 for the phase image.

“Feedback” contains a lot of parameters controlling the feedback loop. A normal user like you just needs to change the integral and proportional gains and the amplitude setpoint which are available in the Main tab.

“Other” contains all the settings which do not fit to the first tabs. All you need to do is to check if the microscope mode is tapping.

The scan parameter list can also be used to control the scan but it is not as user-friendly.

There are a few tricks to remember in order to have a good image. During a scan, it is always useful to keep an eye on the scope associated with the height image. It shows a blue and a red line corresponding to “trace” (left to right) and “retrace” (right to left), they should overlap each other but not perfectly. If they don’t (Figure 22), it means the feedback has problems to adjust the tip’s height and you will have to change some of the parameters.

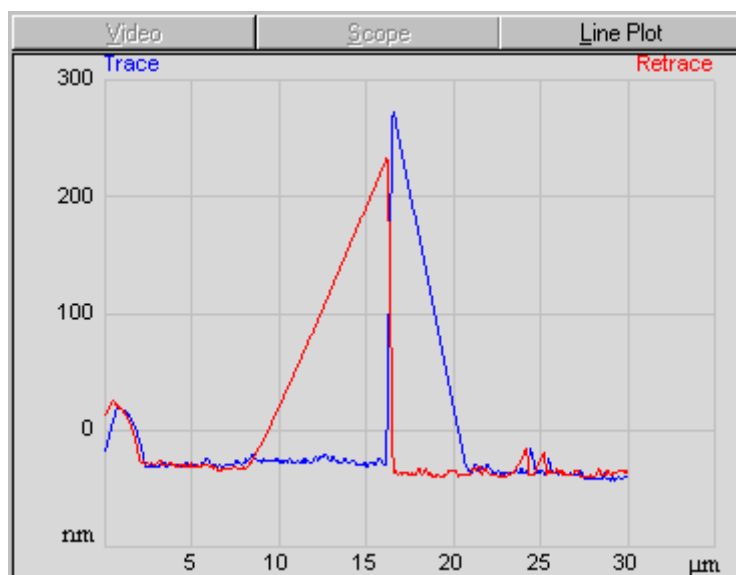


Figure 22: example of a bad scope

The main parameters to play with are the scan rate, the amplitude setpoint and the integral and proportional gains. The first thing I do is to decrease the amplitude setpoint but not below 1.00 V. If it is not enough, you can change the gains. Good gains for the kind of tip we have is 0.2 for the integral gain and 0.6 for the proportional gain, try to keep the ratio roughly equal to three when you change them. You can also scan at a lower rate. In good conditions, the scope should look like the example on figure 23.

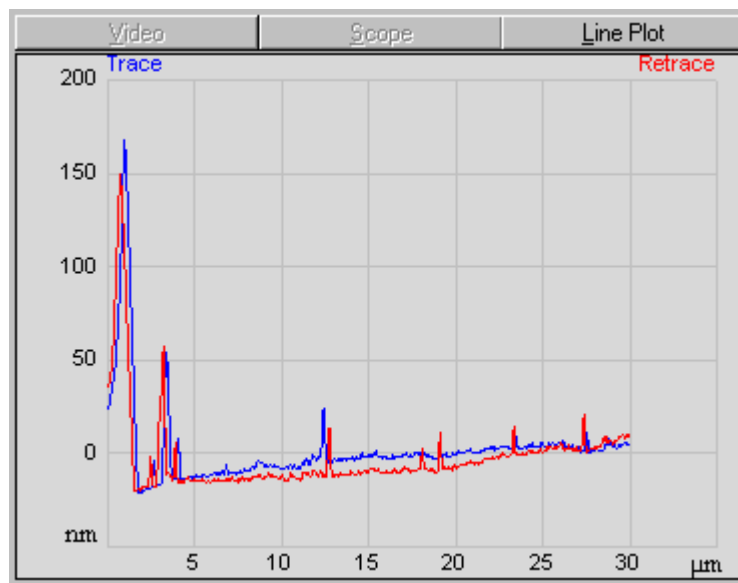


Figure 23: example of a good scope

For the study of nanostructures, it is useful to be able to zoom or offset the centre of the image. The easiest way to do it is the zoom (or offset) option in the Scan single or scan display windows. Click on the zoom (or offset) button at the bottom of the image, choose the location you want to zoom in (or the new centre of the image) and click on execute.

In terms of safety, you have to keep an eye on the RealTime Status window on the right of the screen. Now, it shows a green vertical bar representing the extension of the piezoelectric scanner. A black line represents the position of the scanner on this bar. If the

scanner is retracted, the line is close to the top; if it is extended, the line is near the bottom. There is danger if the scanner is fully retracted or fully extended. When it is fully retracted, there is a big risk of breaking the tip or the scanner. When it is fully extended, the tip will lose contact with the surface. Luckily, there is an alarm, the bar turns yellow when it starts being dangerous and then red when it is very dangerous. You can try to finish the image and withdraw if it is yellow but you have to keep an eye on it. When it turns red, please withdraw and then engage the tip again. One other dangerous moment is when you see the scanner's extension wobbling; it also means you should withdraw the tip.

III-6 – Capturing data.

Of course, if you do not save the images you obtained, all you have done is useless. To capture an image, you have to click on the capture icon which represents a camera (figure 24). The other icons are capture now, which will save the bit of the image that has been recorded, and abort capture.

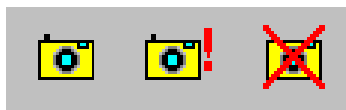


Figure 24: the capture icons


Another way to save is the capture button in the scan control display. To specify the folder and the name of the image, you must go to the menu RealTime and choose “Capture Filename”. All the folders for the different users must be located in the directory “D:\abusers\” and the filename must be terminated by a counter (e.g. image.000) the system will increment automatically. Note that if you don't change the capture filename, your image will be saved under the last name entered. You can check if the capture is on in the status bar.

Once you have captured an image and if your folder is selected in the image window, it should appear on the right of the screen.

I must emphasize the fact that the system is designed to save image which have been collected with constant parameter. If you played with a parameter, it will want to save the next image. You can save a bit of time by restarting the image. You can also force the capture by clicking a second time on the icon but I don't recommend it as it is not good to present images which have been recorded with changing conditions.

If you need to leave urgently (meeting with your supervisor or coffee break), there is a cool option: the capture and withdraw in the RealTime menu. It will save the image and retract the tip.

III-7 – Withdrawing the tip.

When your experiment is finished, you have to withdraw the tip by clicking on withdraw in the scan control display or on the withdraw icon . Then, you must come back to the navigate window and move the head up using the z-motor control. Once the bottom of the scanner is two to three centimetre higher than the sample stage, you can safely take your sample back.

III-8 – Switching off the machine.

It is pretty simple, switch off the soft, the communication between the system and the PC will stop. Then, turn off the controllers (first the Dimension 3100, then the Nanoscope IV) and the computer. Note that the computer does not shut down automatically when windows NT is off; you will have to wait and turn it off manually.

IV – Analyzing and transferring data

IV-1 – Analyzing images.

Fancy images are not everything in science, it is also important to extract data. You can use Nanoscope 6.12r1 to analyze your images. It is very simple and quick to start the offline part of the software, just double-click on a thumbnail in the image window at the right of the screen. The image will then be opened in the client window and new icons appear in the tool bar (Figure 25).



Figure 25: the analysis tool bar.

Form left to right, the first icon (representing a star) is the image icon, and it just brings the image back to the top of the client window. It can be very useful when a lot of windows are open. The second icon correspond to the depth analysis, it gives a histogram of the height distribution of the sample. The third icon represents a razor blade; it is the section (or profile) analysis. It allows the user to measure things like distance (XY or Z) between different features on the surface. The following icon corresponds to the roughness analysis, for people interested in that aspect, the RMS roughness usually reported in publications is

called “Image Rq” (q stands for quadratic). The next icon is the 3D plot, it is sometimes useful if you want to highlight some features like nanostructures deposited on the surface, monoatomic steps or structures you obtained by lithography. PSD means power spectral density; it is useful for the analysis of periodical structures. The icon representing a magnifying glass is the zoom. The following icon corresponds to the XY drift; it compensates the scanner’s thermal drift if there is some. In normal conditions, the scanner we have doesn’t drift. The icon representing a sort of roll is “flatten”; it is basically a background correction, like the next icon which is plane fit. The next icon (a small square going into a bigger square) is the median filter, it is the smoothing function. The icon representing a funnel is the low pass filter; it will remove some high-frequency noise if there is some on the image. The last function is erase lines.

I have to warn you about a small bug of the software: when you want to apply the same function to two channels of the same image (flatten for instance), the software will allow you to move to the second channel only if you delete this function from the workspace window on the left of the screen.

One last advice, always keep the original image somewhere, work on copies. I personally recommend transferring your data to your PC in the office and analyzing the images there, it will be an automatic back-up. There is a good freeware for the analysis: WsXM available for download from <http://www.nanotec.es>.

IV-2 – Transferring data.

Unfortunately, USB flash-disks are not compatible with the control computer as the Operating system is windows NT. We installed two options to back-up your data:

- a CD-burner, the software is called “NTI CD-Maker 2000 plus” and there is an icon on the desktop;
- and a Zip-drive.

Also note that I make a back-up copy of the directory D:\abusers every six month.

V – Concluding remarks

I hope this document will help people to use the Atomic Force Microscope. This manual doesn't describe all the experiments one can make with this machine, as I concentrated on basic imaging in tapping-mode. If you need training on other AFM techniques, please feel free to ask Cristina or myself for help. You can also read the 600 pages-long manual provided by Veeco. I would like to finish by asking you to tell me if you find a part of this manual unclear or if something is missing, I will be happy to update it.