Scanning Probes Microcopies practical works
Atomic Force Microscope

Florence Marchi & Joel Chevrier

According to your level and interest in AFM, several sessions are possible. In order to determine which one corresponds to your need, please read the following sessions and discuss with the instructor.

1/ First AFM approach: imaging a grating in contact mode

In order to discover how works an AFM microscope, you are going to image *in contact mode* a test grating which is constituted by a series of design with a regular step.

a/ With the help of the instructor, set up the probe on the AFM head and adjust the laser beam at the tip end. Describe the followed steps to realize this operation and describe briefly the principle of this optical detection (draw some figure if you need). You could use the manual and documentation to help you.

b/ Observe with the optical microscope the grating : could you measure its step, its high and the width of the line ? With a classical optical microscope which is the theoretical limit?

c/ Place the grating on the sample holder and engage the tip. Please,verify that the “scan size’ is load to zero. This engagement step has to be done with the instructor.

d/ Once the tip is engaged correctly, increase slowly the scan size in order to image several lines of the grating. When you image is stable, stop scanning the whole image, scan only ***one line,*** once it is done, visualize the line in the ‘scope mode’. Now change the regulation parameters in order to identify their effects. Describe your observations.

e/ Set up the regulation parameters at their best values and record few images in order to measure the height and width of the grating line and the step grating. In this purpose, use the imaging software.

2/ Approach retract curves in contact mode : force curve

With an AFM it is possible to probe and characterize the tip-sample interaction. For this purpose, the force spectroscopy mode is used to realize force curves.

a) Once a stable image of the conductive surface is obtained, you can realize a force curve by selected force curve menu. Use the manual guide to help you and discuss with the instructor.

- Explain what is a force curve and what kind of information is possible to obtain
- Observe and describe the influence of the different adjustable parameters on the force curve.

b/ Realize force curves with different applied voltage between the tip and the sample (0, 5V ; 10V, -5V ; -10V). What do you observe? How do you explain the phenomenon?

florence.marchi@grenoble.cnrs.fr ; joel.chevrier@grenoble.cnrs.fr

3/ Nano-Lithography: Local Oxidation by AFM in contact mode

It is possible to use the AFM tip as ***a nano-pen*** in order to draw some structures on a Si surface. When a voltage is applied between a conductive AFM probe and a Si surface a local chemical reaction is induced, and more precisely an oxidation. It is then possible to create oxide lines on a specific area of the surface. These lines and structures can be used as a mask in order to obtain silicon nano-structures. This process can be also realized on conductive layer as niobium or aluminium.

a/ *Observation of the influence of the voltage value, polarity, and tip velocity on oxide lines formation*

- Open the program ‘lines’ on the nanoscript, read it and complete it.
- Run the program
- What do you observe in function of the polarity of the voltage?
- How vary the geometric dimensions of the lines with the voltage increase?
- How vary the geometric dimensions of the lines with the tip velocity?

In order to answer these questions, use the analyse software

4/ First approach of AFM in dynamic mode

If you are familiar with the use of AFM in contact mode but less in dynamic mode, more often called ‘tapping’ mode, this 4th session is for you.

A/ Imaging session

In order to discover how works an AFM microscope, you are going to image *in dynamic mode* a tested grating which is constituted by a series of lines with a regular step or Germanium nanodot deposited on silicon oxide layer by molecular epitaxy.

a/ With the help of the instructor, set up the probe on the AFM head and adjust the laser beam at the tip end. Describe the followed steps to realize this operation and describe briefly the principle of this optical detection (draw some figure if you need). You could use the manual and documentation to help you.

c/ Once the optical detection is adjusted, find the resonance frequency of the AFM probe by making a resonance curve. Once this resonance frequency is found, chose to work at a frequency just below it. Why is it required to work just before or after the resonance frequency?

d/ Engage the tip and image the sample surface. Adjust the feedback parameters in order to obtain a stable image. Once the image is stable, stop scanning the whole image, scan only one line and visualize it in the scope mode. Now change the regulation parameters in order to identify their effects. Describe your observations.

e/ Set up the regulation parameters at their best values and record few images in order to measure the height and width of the grating line, the step grating or Ge dots dimensions.; in this purpose, use the imaging software

B/ Approach-retract curves

As in force curves, approach-retract cruves can be done in dynamic mode in order to characterize the tip-sample interaction.

florence.marchi@grenoble.cnrs.fr ; joel.chevrier@grenoble.cnrs.fr

a/ Once a stable image of the surface is obtained, realize an approach-retract curve in recording amplitude and/or phase variation. Use the manual guide to help you and discuss with the instructor.

- Explain what is an approach-retract curve in dynamic mode and what kind of information is possible to obtain
- Observe and describe the influence of the different adjustable parameters on the force curve.

b/ Realize five force curves with different applied voltage between the tip and the sample (0, 5V ; 10V, -5V ; -10V). What do you observe? How do you explain the phenomenon?

5/ Injection and Detection of electric charges by EFM (Electric Force Microscopy)

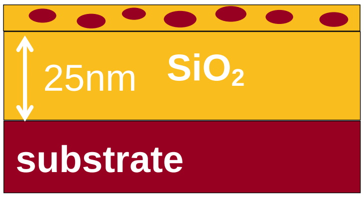
If you are familiar with dynamic mode of AFM to image surface, you may be interested by using EFM mode which is based on the dynamic mode. EFM mode allows detecting electric force created by the presence of electric charge localized on a specific part of the sample.
The presence of electric charges can be due to the nature of the probed sample or can been injected by the AFM tip. During the practical work electric charges will be injected by AFM tip on the sample[*] .

a/ Set up the sample on the AFM stage and image it on ‘tapping’ mode. Open EFM windows in order to realize an EFM image of the surface in the same time as the tapping image (2 images should be done at the same time on the image screen).

b/ Open the program ‘charge injection’ on the nanoscript software, read and complete the it (injection time, voltage injection values). Run the program

c/ Detect the electric force induced by the injected charges in EFM mode.
What do you observe: shape, dimensions of the cloud of electric charges?

[*] Two samples can be used during the practical works: (1) a SiO$_2$ layer deposited on Si substrate or (2) Si clusters embedded in silicon oxide layer on top of a silicon oxide layer deposited on a silicon substrate.



Sample 2

Sample 1

Some 'tricky' questions……
……………………..that you could try to answer if you have time and interested.

1-Looking at such picture, it is sometime said that Contact mode can achieve atomic resolution. This is not correct. Comments?

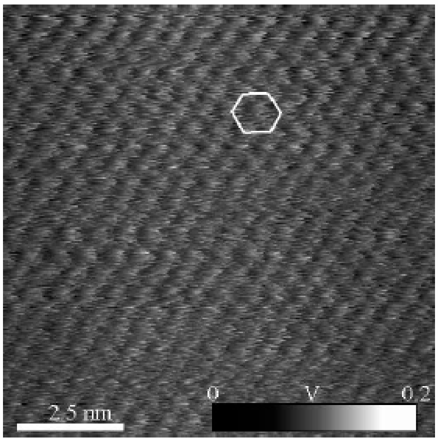


*Figure caption: AFM image in contact mode (lateral force image 10*10 nm^2, cantilever spring constant : 0.06N.m^{-1}) of a mica surface.*

2-Atoms or molecules can be displaced by the AFM tip. Can you estimate the force taking place ? Usual cantilever spring constants are close to 1N/m ? Any good reason for that ?

3-It is hardly possible to image carbon nanotubes on a flat surface in contact mode . It is easily done in dynamical mode. Comments ?

4-**lateral resolution :**

a) What are the differences between these two images ? What happens ?

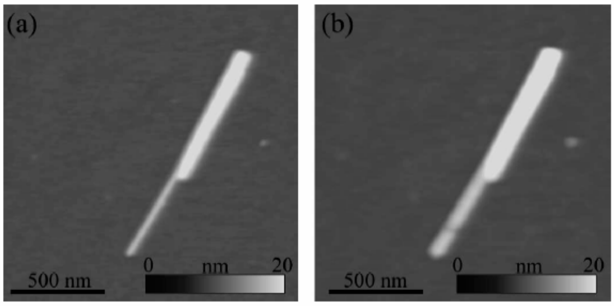


Figure 1: AFM image in contact mode (cantilever spring constant 0.012N.m-1) of an organic whiskers deposited on a Si substrate. (a) reference image and (b) image obtained after tip damage. The tip has been damage by scanning with a normal force around 100nN.

b) What is the correct measurement : the lateral one or the height one ? Please justify your answer.

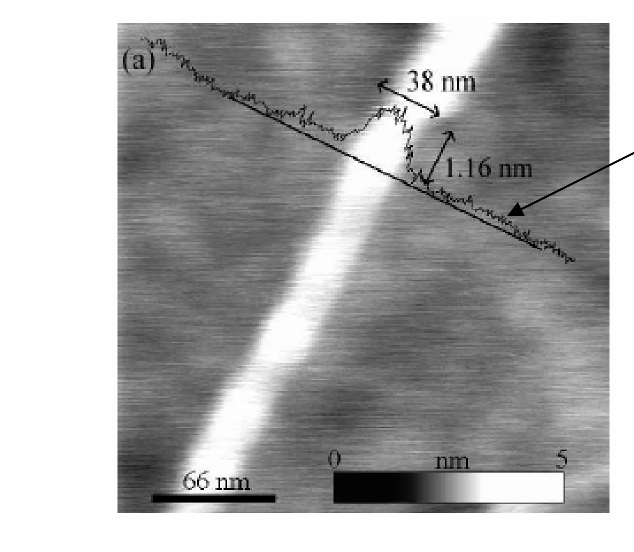


Figure 2 : AFM image of a carbon nanotube on Si sample. The heigh signal included in the image has been measured alomg the straight black line.

5- Smallest cantilever vibration measured : the Brownian motion could be easily detected in the photodiode signal you are looking at ? Do you believe it ? What is the magnitude of this effect ?

6-How many forces can you think about that are important in this AFM Practical ?

7-Charge detection : what is the force order of magnitude acting on a single electron in Millikan experiment ? How does it compare with AFM ? Surprise.. ?

8-Tapping or Non Contact AFM ? What is the difference ?

9-Can you think about an experimental method to determine the cantilever spring constant ?

10- Can you think about an experimental method to determine the oscillating amplitude ?

11-Soft (<<1N/m) or hard (>>1/Nm) cantilever ? What do you prefer ?

12-energy dissipated during cantilever oscillation ? The smallest force that can be measured ?

13- it is often said that EFM have a more

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EFM mode :

13- If some electric charges are trapped in an oxide layer, an apparent height is detected, why ? Use figure below to answer.

14- In EFM, what is the nature (attractive or repulsive or both) of the force induced by trapped charge ?

15- In EFM, a conductive AFM probe is used, what is the resolution in this mode ? Why the EFM resolution is better than the Kelvin probe mode ?