Lab work N3: Atomic Force Microscope in Intermittent Contact Mode

This practical is focused on the atomic force microscope (AFM) in intermittent contact mode (also called, Tapping Mode^{TM}). It is divided into two parts: the first, concerning the AFM as a topography measurement tool and the second presenting an application of the AFM for electrical measurements (electric force microscopy, EFM).

In the topography mode, the objectives are: understanding the functioning of the Atomic Force Microscopy (AFM) in intermittent contact mode, learning how to use it and analyzing the images obtained. Nanometric objects will be scanned and the AFM given measurements of height and width are to be analyzed with respect to the tip convolution with the surface.

In the electrical mode (EFM), the objective is to charge a silicon oxide layer by the polarized tip and to detect the charging effects in lift mode.

I. Functioning principle of an AFM in intermittent contact mode

TappingMode™ AFM operates by scanning a tip attached to the end of an oscillating cantilever across the sample surface. The process consists of allowing the cantilever to reach its resonant frequency (50 000-500 000 Hz), and to bring it close to the sample until contact is made with the sample. The cantilever is oscillating with the amplitude ranging typically from 20nm to 100nm.
The tip lightly “taps” on the sample surface during scanning, contacting the surface at the bottom of its swing. The energy loss resulting from tip-sample contact naturally generates an amplitude loss, which is measured and used to identify surface features (see Figure 1). The feedback loop maintains constant oscillation amplitude by maintaining a constant RMS of the oscillation signal acquired by the split photodiode detector (see Figure 2).

The changes of the vertical position of the scanner at each (x,y) data point, in order to maintain a constant "setpoint" amplitude is stored by the computer to form the topographic image of the sample surface. By maintaining constant oscillation amplitude, a constant tip-sample interaction is maintained during imaging.
Operation can take place in ambient and liquid environments. In liquid, the oscillation doesn’t need to be at the cantilever resonance.

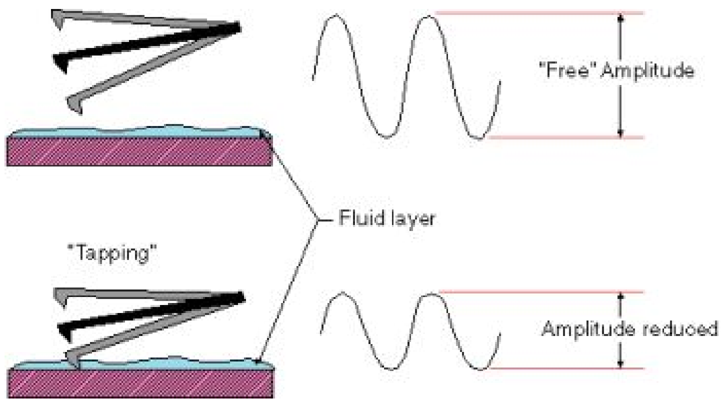

Figure 1: ***In the upper part of the image we can see the oscillation amplitude of the cantilever in air. In the lower part of the image we can observe the reduction of the oscillation amplitude due to the interaction with the surface.***

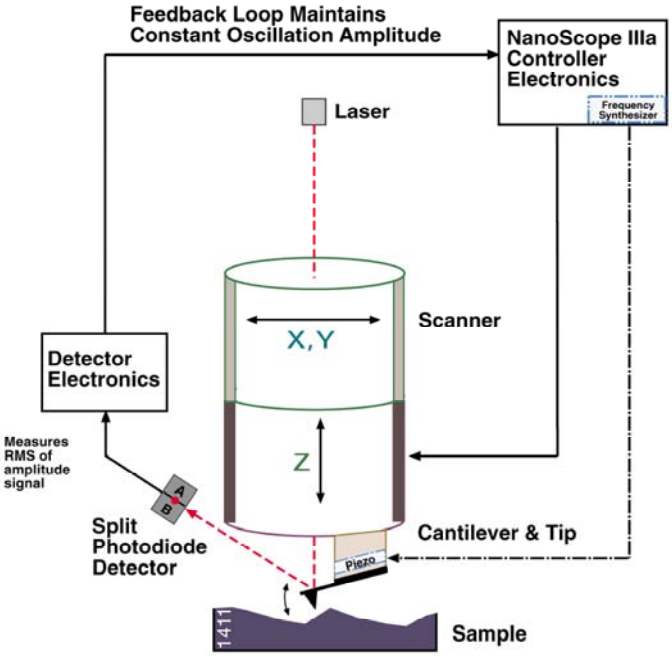

Figure 2 : ***The principle of AFM functioning in intermittent contact mode***

As the tapping probe tip comes into contact with the substrate, the high resolution of contact AFM can be achieved while drastically reducing tip or sample damage: tapping mode allows the imaging of samples that are easily damaged or otherwise difficult to image in contact mode.

As the cantilever bounces up and down, it is resistant to the adhesion and adsorbed meniscus sticking problems that can adversely affect contact mode measurements: when imaging in air, the typical amplitude of the oscillation allows the tip to contact the surface through the adsorbed fluid layer without getting stuck.

AFM Tips

The cantilevers used in intermittent contact mode are more rigid than the cantilever used in contact mode: the range is from 10 to 100 N/m. The resonance frequencies can vary from 50 KHz to 400 KHz. For the topography measurements, we will use a Si tip with a resonance frequency near 300 KHz. The shape of the tip and its radius of curvature are very important: in our case, the announced radius of curvature is <20nm.

Advantages:

- Higher lateral resolution on most samples (1 nm to 5 nm).
- Lower forces and less damage to soft samples imaged in air.
- Lateral forces are virtually eliminated, so there is no scraping.
- Adsorbed fluid layer don't affect the measurements.

Disadvantages:

- Slightly slower scan speed than contact mode AFM.

II. Experimental work

II.1 Resonance frequency research

☞ After fixing the tip on the AFM, trace the resonance curve of the cantilever. Estimate the Q factor of the system cantilever-tip from the frequency vs. amplitude of oscillation plot.

☞ In the "**Panels**" menu select "**Seek Q**". You can verify that the Q factor that you have calculated is right.

☞ Why is it important to know the Q factor of the tip? Is it possible that the Q factor of a system cantilever–tip change? In which conditions?

II.2 Adjustment of scan parameters

☞ Place the sample with the nanowires on the AFM, approach the tip and scan the sample. (A teacher **MUST** assist you for this part.)

☞ Adapt the **Scan size** slowly and centre the image by using **X** and **Y offset** (or click on **Offset** on the other monitor and centre the target on the interesting region) in order to see a nanowire in the scanning window.

☞ Add **Channel 3** and display the **Amplitude** signal in this channel. What do you expect the amplitude signal to be? How can you explain the difference between your expectations and the real image?

II.3 Tip shape artefacts

☞ Image the smallest silicon nanowire on this sample.

☞ Compare the width of the line measured by AFM with the SEM image of Figure 3.

☞ Using the section tool give an estimation of the angles of the tip. Comment on the shape of the tip and estimate the tip diameter.

☞ Disengage the tip from the sample and take off the sample with the nano-objects from the AFM chuck.

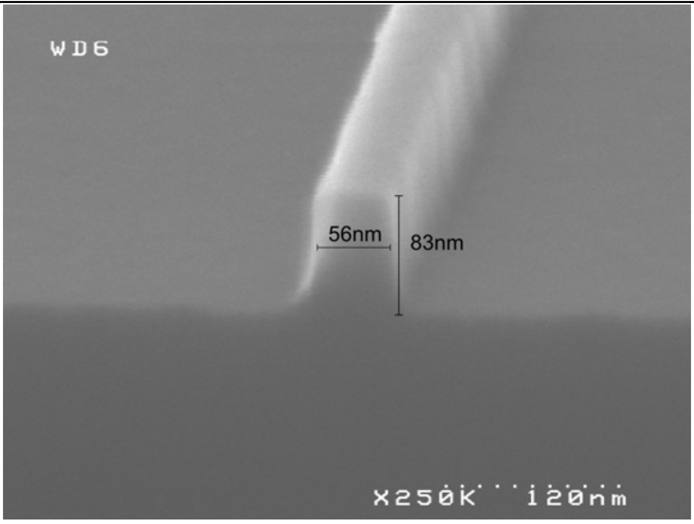

Figure 3 : ***The SEM image of the profile of the smallest silicon line.***

II.4 Approach-retract curves

These curves will be used as preliminary measurements for next section (electric force microscopy). Therefore, the tip must be changed with a conductive one.

- ☞ Place the sample for EFM measurement, engage the tip and image the surface in order to obtain a stable image. (A teacher **MUST** assist you for this part.)
- ☞ Realize and explain an approach – retract curve in tapping mode.

II.5 Electric Force Microscopy (EFM)

- ☞ On the same sample, go back to the image mode. The AFM that you are using allows scanning lines in lift mode and measuring the phase values during the lift scan. Explain the functioning of the lift mode.
- ☞ Go back to the force curve mode and check the effect of a bias voltage of 5V on the tip (for about 20s).
- ☞ Return to the scan mode and scan the same area in lift mode, with a polarisation on the tip (for example, -2V, at a lift height of 50nm) How could we explain the changes in the phase in lift mode? Save the image and measure the size of the spot.
- ☞ After applying an offset, go back to the force curve mode and check the effect of a bias voltage of -5V (for 20s).
- ☞ Return to the image mode and scan the whole area in lift with a bias voltage on the tip (for example, -2V) at a lift height of 50nm.
- ☞ Compare the size of the spots. How can you explain the enlargement of the first spot?