

# Fundamentals of Atomic Force Microscopy

## ME59700/PHYS57000

### Homework 4: AM-AFM scans and basics of FM-AFM

Due Date: Dec. 7, 2010

#### **Problem 1. Setting the feedback parameters in AM-AFM scanning**

Experimentally setting the proportional and integral gains while performing dAFM measurements often requires more patience than understanding. This problem is designed to provide some insights that might help you better adjust these two gains in the lab.

After logging on to VEDA, under the application tab "Pick a tool here", select "Amplitude modulated scanning (basic)". When this application opens, you will find four application tabs that are reasonably self-explanatory.

Under the "Operating Conditions and Cantilever properties" tab, select acoustic excitation with an unconstrained amplitude of about 30 nm, a cantilever spring constant of around 40 N/m, a cantilever resonance and drive frequency of about 350 kHz, and an amplitude set point ratio of 0.9. Set the scan lines per second to 33 and select a signal to noise ratio to 30 dB.

Set the proportional gain to 0.002. Set the integral gain to 0.001.

Under "Tip-sample Interaction properties: substrate" tab, select a tip radius of 10 nm, a tip modulus of about 130 GPa and a Poisson ratio of 0.33. Select an adhesive force of about 3.5 nN and a Hamaker constant of about  $3 \times 10^{-19}$  J. Set the modulus of the substrate to 10 GPa.

Now create a "feature" that you will scan. Click on the "Feature Properties" tab. Select a geometric *step* feature that is 1 nm high and 10 nm long. Click on the "Specify materials properties" box and set the modulus, Hamaker constant and adhesive force equal to the substrate. Make sure the "Include visco-elastic forces" box is set to "no".

The scan size found under the "Simulation parameters" tab should be set to about 30 nm.

After specifying these input parameters, press simulate and allow VEDA to perform a line scan over the feature you have specified. You can view the tip position by selecting "Measure topography" under the Result tab. Perform about four simulations to illustrate the effect of adjusting the integral gain from 0.0005 to 0.001.

Make a composite plot showing the specified feature and the results of each line scan. Write a paragraph that describes the essential features that you see in the plot. To better understand the anomalous features introduced by a poor choice of feedback gains, it might be useful to include an error map and phase plot associated with each simulation. What is the best value of the integral gain?

## Problem 2. Factors Affecting Contrast in dAFM Topographic and Phase Images

A dAFM study is performed on a newly prepared polymer blend material that contains two distinct polymeric materials. Since the material is new, the results are published in the literature. Unfortunately, the authors did not take the ME59700/PHYS57000 AFM course, so their understanding of the AFM study is a bit uncertain.

Figure 1 reproduces three topographic (left) and three phase (right) images of this new polymer film deposited on a glass substrate. The authors state each image is  $1.8 \mu\text{m} \times 1.8 \mu\text{m}$ . The authors state the polymeric films are several microns thick. The authors state that when obtaining these images, the unconstrained cantilever amplitude is  $A_0=75 \text{ nm}$ . The set point ratio ( $r_{sp} = A/A_0$ ) used to acquire each image is specified. The authors also state the height contrast covers height variations in the 5 nm range for Figs. 1(a) and 1(b), while height variations in the 10 nm range are found when acquiring the image in Fig. 1(c). The authors state the force constant of the cantilevers used in the study were in the range of 13-70 N/m.

There seems to be a clear contrast observed both in topography and phase, allowing the authors to specify two distinct regions, P and Q, as marked in Fig. 1(c). But the authors are confused. They decide to perform additional experiments. A static force distance study is performed. The authors state that cantilevers with force constant in the range of 0.02 – 0.7 N/m are used. The results are plotted in Fig. 2.

Finally, the authors performed measurements of the phase shift as a function of the set point ratio,  $r_{sp}$ . The results are plotted in Fig. 3 for the two regions marked P and Q in Fig. 1(c).

With just this information, can you answer the following questions:

- a) Surprisingly, the authors do NOT state the resonance frequency of the dAFM cantilevers that were used. What is your best estimate for this frequency? State your assumptions and justify your answer.
- b) In Figs 1(b) and 1(c), there seems to be clear contrast in both topography and phase, providing evidence that the sample is indeed comprised of two distinct polymer phases. Why is the contrast greatly diminished in Fig. 1(a)? Justify your answer.
- c) The authors are confused by the images presented in Fig. 1. Why do you think they are confused?

- d) Assuming the authors correctly measured the height variations, why do you think the height variation in Fig. 1(c) is about a factor of 2 greater than the height variation in Figs. 1(a) and 1(b)? Justify your answer.
- e) Which region is stiffer, P or Q? Justify your answer.
- f) Which region has the greater adhesion, P or Q? Justify your answer.
- g) What is the approximate phase difference between the dark and bright regions in Fig. 1(c)? Explain how you arrived at your answer.
- h) Does the phase variation with set-point plotted in Fig. 3 make sense? Run a VEDA simulation to justify your answer to this question. Specify the parameters that you use when running this simulation.
- i) On the basis of your simulation, what set point value would you choose to obtain maximum phase contrast?

# Homework 4: Fundamentals of Atomic Force Microscopy ME59700/PHYS57000

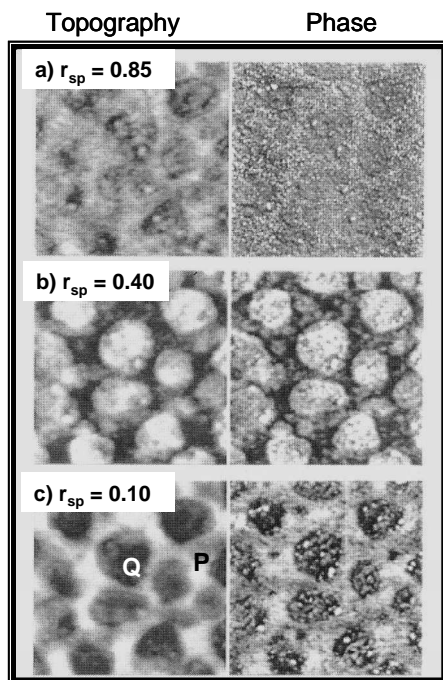


Figure 1:  $A_0=75$  nm;  $1.8 \mu\text{m} \times 1.8 \mu\text{m}$

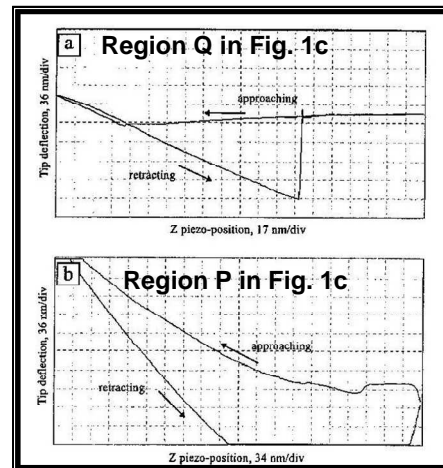


Figure 2

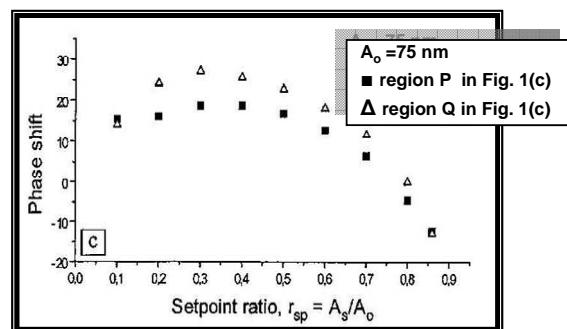


Figure 3

### Problem 3. FM-AFM

According to the theory of FM-AFM discussed in lecture, the fractional frequency shift of an AFM tip (attached to a cantilever with spring constant  $k_c$ ) as it approaches a substrate should be related to the tip-substrate force gradient by

$$\frac{\Delta f(z)}{f_o} = -\frac{1}{2k_c} \frac{dF_{ts}}{dz}$$

Using the FM-AFM tool in VEDA with the tip-sample force ( $F_{ts}(z)$ ) determined by a DMT contact model, determine what amplitude of the cantilever motion is required before this equation valid.

After logging on to VEDA, under the application tab "Pick a tool here", select "Frequency Modulated Approach Curve". When this application opens, you will find four application tabs that are reasonably self-explanatory.

A useful set of input parameters might be: acoustic excitation with one eigenmode, a first order lock-in filter,  $R_{tip} = 5$  nm,  $E_{tip} = 130$  GPa,  $E_{sub} = 1$  GPa, Poisson ratio of tip and substrate = 0.33,  $H = 1.9 \times 10^{-19}$  J,  $F_{adh} = 1.5$  nN,  $k_c = 40$  N/m,  $Q = 600$ ,  $f_o = 150$  kHz,  $f_{drive} = 150$  kHz, PLL proportional gain = 0.01, PLL integral gain = 0.001, amplitude proportional gain = 0.01, and amplitude integral gain = 0.001. Use a first order tip approach speed of about 2 nm/s and a sampling frequency of about 5MHz.

Under the "Simulation" tab, be aware that the "Choose X-axis variable" input allows you to plot the calculated variables vs. either z-distance or minimum gap. Make sure you know how to relate z-distance to minimum gap.

You can plot  $F_{ts}(z)$  by selecting "Tip-sample force viewer" from the main application tab in VEDA.

Perform frequency shift simulations for four (4) unconstrained amplitudes of  $A_o = 1.0$  nm, 0.5 nm, 0.25 nm and 0.1 nm.

To analyze the VEDA simulations, make four(4) two-axis graphs from the VEDA outputs. Each graph should contain the results of one of the four (4) unconstrained amplitudes specified above.

The graph might include  $F_{ts}(z)$  [obtained from the Tip-sample force viewer] plotted on the left ordinate. The *fractional* frequency shift must be calculated from the information provided under the "FM Feedback (delta f and drive amplitude)" [accessed under the Result tab in the FM Approach Curve

application]. Plot the fractional frequency on the right ordinate. Calculate the derivative of  $F_{ts}$  (and divide it by  $2k_c$ ). Plot this quantity on top of the *fractional* frequency shift. If your plot is done properly, you should be able to view  $F_{ts}(z)$ , the *fractional* frequency shift, and the derivative of  $F_{ts}$  (divided by  $2k_c$ ) all on one plot.

When does the fractional frequency shift match the derivative of  $F_{ts}$ ?

From these plots, write a thoughtful paragraph that explains which unconstrained amplitude(s) are required before the equation given above is valid.

#### **Problem 4. A dAFM “pocket guide”**

During the course of the semester, various parameters and equations have been discussed to help you better understand the fundamentals of dynamic atomic force microscopy (dAFM). Before you forget what you have learned, design a **ONE** page “dAFM pocket guide” that you can post next to your AFM. The “pocket guide” should contain only a few (maybe only 5-10) key results that will remind you of important findings that you have learned this past semester.

Be aware there is no single correct answer to this problem, and the sheet that each of you develop may well be based on your scanning experience. The goal of this exercise is to demonstrate awareness of relevant dAFM scanning parameters discussed throughout the semester.

Make sure your “dAFM pocket guide” is neatly formatted and labeled. We’d like to see it posted by your AFM if we visit your lab in the future!