Homework No. 2 (Based on week 2 lectures)

This problem is based on a Case Study (experimental) data from the literature [1].

Sample properties

Properties of three materials *in air* are relevant here:
Gelatin film- has a low and variable Young's modulus (10-100kPa).
Polystyrene wells- hydrophobic with a Youngs modulus in the range 3-3.5 GPa (much stiffer than gelatin but softer than mica).
Mica surface – stiffest of the three (Young's modulus ~ 170GPa) but hydrophilic.

In particular two samples are studied in ambient air. The first consists of thin film patches of gelatin (<5 nm high) deposited on polycarbonate wells and the second consists of thin film patches of gelatin deposited on freshly cleaved mica.

Cantilever properties and operating conditions

All images were obtained with a NanoScope IIIa MultiMode AFM (Digital Instrument) in air with tapping mode, using the E-type scanner (10 x10X 2 micrometer) and silicon tips (Model TESP, which has a rectangular shape cantilever of 125 micrmeter long with ca. 50 N/m force constant and ca. 300 kHz resonant frequency). Typically, 2 lines/s scan speed was used for scanning on a 2x2 micrometer area, with a typical setpoint A/A^{far}=0.95 (Note that the paper states 0.85 but the figures suggest 0.95, so we will stick with the experimental data they present) and A^{far}~50 nm, where A^{far} is the cantilever oscillation amplitude before interaction begins (also called free amplitude).The cantilever is driven exactly at its natural frequency and the natural frequency is determined from a tune very near the sample surface. The cantilever is excited acoustically.

Important note

In the phase images in this article, *the authors plot the natural output of this AFM system which is in fact the phase lead* (should be= -90 degrees at resonance). *However in the dynamic approach curves they convert the measured phase and plot phase lag*. This fact is subtle but very important to interpret the data.

Results

Typical images of gelatin partially covered polystyrene well surfaces are shown in Fig. 1. The phase images (see Fig. 1b) always show a two-component feature. Note that all phase images in this paper are presented in the original NanoScope IIIa fashion, i.e. in terms of phase lead. So brighter colors indicate smaller phase lag angles.

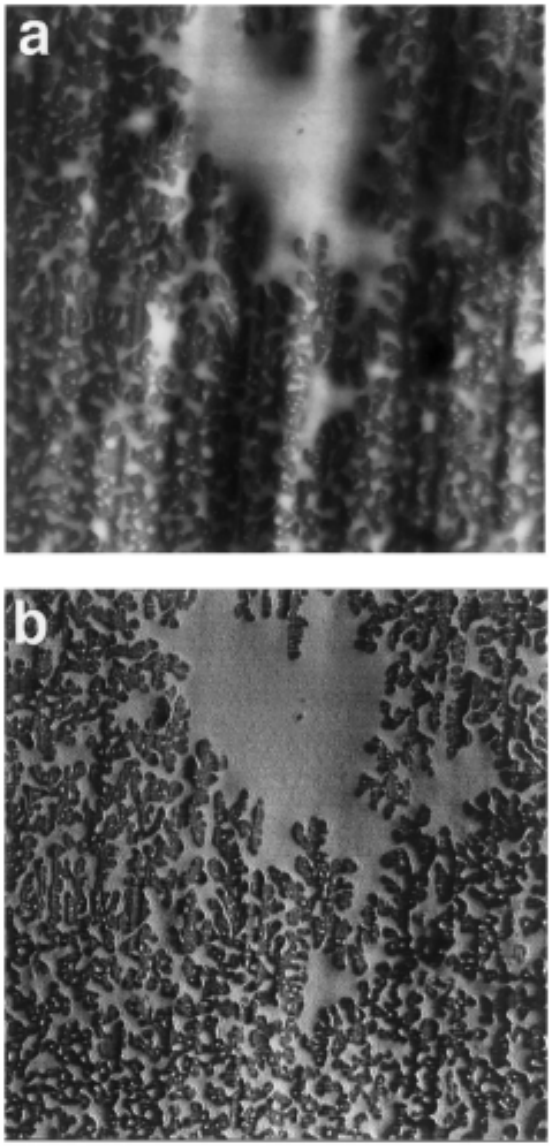

Fig. 1. Height (a) and phase lead (b) images of gelatin on a polystyrene well surface measured with Tapping Mode AFM, acquired on a 2x2 microns area with A^{far}=50 nm and A/A^{far}=0.95. The full grey scales are 45 nm (a) and 11 degs (b), respectively. The polystyrene well was incubated with gelatin solution for 45 min, followed by twice tris bu¤er rinses before naturally dried.

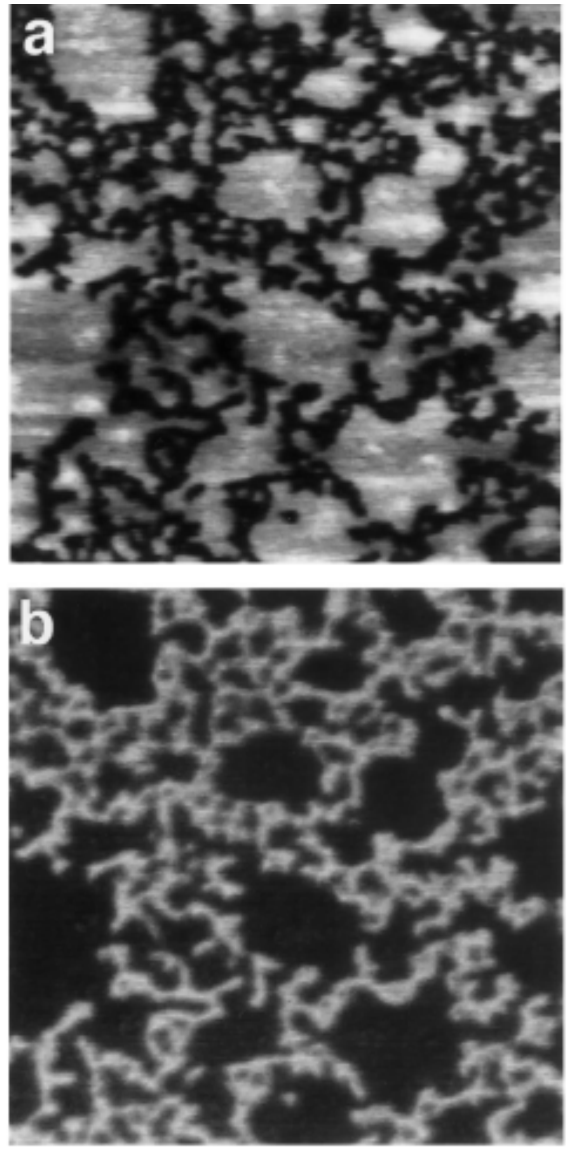

Fig. 2. Height (a) and phase lead (b) images of gelatin on a mica surface measured with Tapping Mode AFM, acquired on a 1X1 microns area with A^{far}=50 nm and A/A^{far}=0.95. The full grey scales are 4 nm (a) and 21degs (b), respectively. The mica was incubated with gelatin solution for 4 h, followed by four times of tris buffer rinse before naturally dried.

To help understand these results, the authors performed amplitude and phase vs Z curves over the three components (gelatin, polystyrene and mica) under the same conditions as the images (Fig. 3).

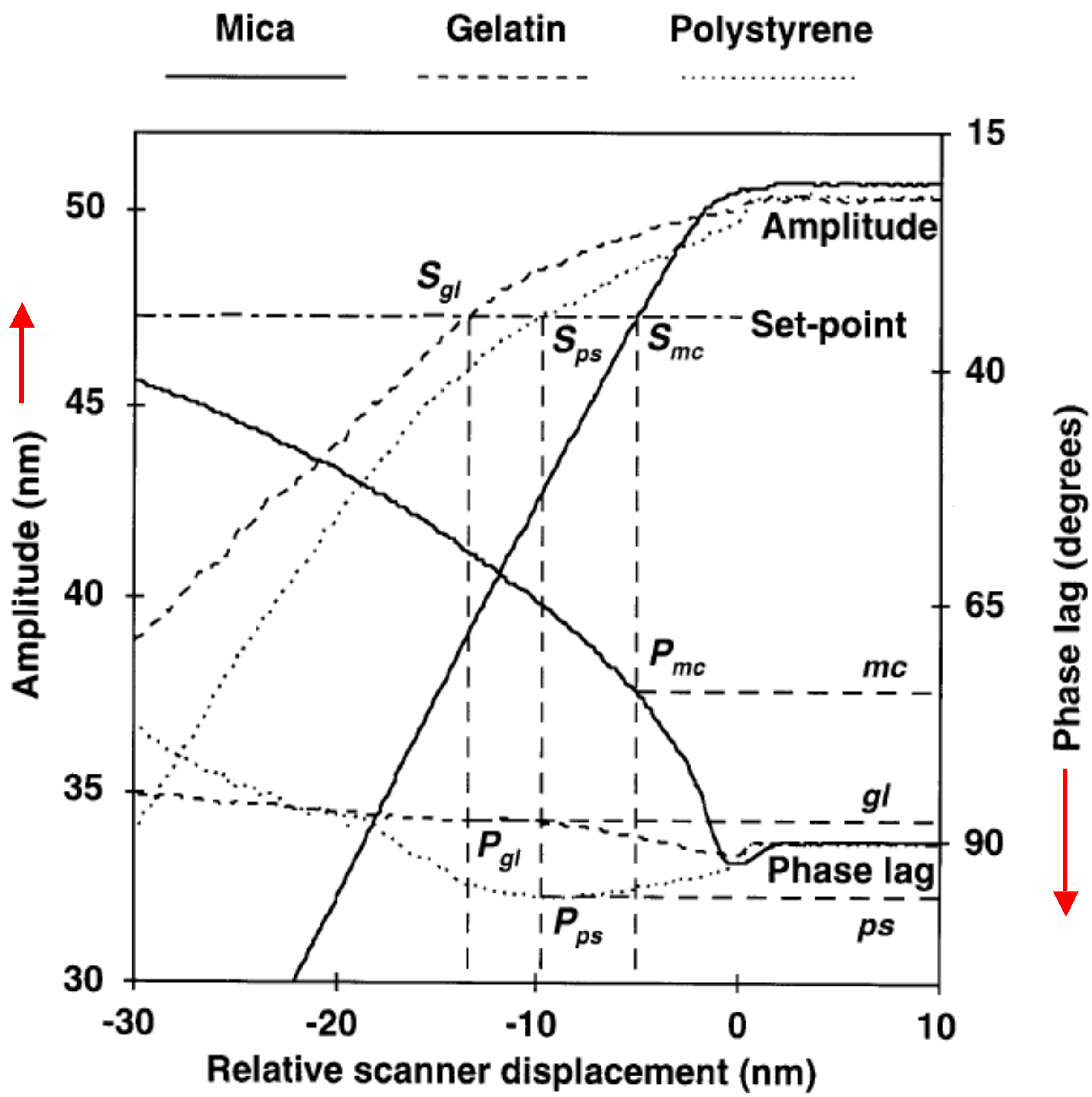

Fig. 3. Amplitude vs Z, and phase lag vs Z curves measured on mica, polystyrene and gelatin surfaces. For clarity, only approach curves are displayed. A horizontal line corresponding to the setpoint level (0.95) used in the Tapping Mode AFM imaging is drawn on the plot. This line crosses the gelatin, polystyrene and mica amplitude and phase vs Z curves at Z values S_{gl}, S_{ps} and S_{mc}, respectively which correspond respectively to the phase lag values P_{gl}, P_{ps} and P_{mc}. Note that the phase lag (right hand axis) increases downwards.

Questions:

1. From Fig. 3 when choosing to image at a setpoint of 95% (A/A^{far}=0.95) which of the following is true
   (a) Under these conditions, the cantilever is in a repulsive regime of operation on mica but in the attractive regime of oscillation on gelatin and polystyrene.
   (b) Under these conditions, the cantilever is in a repulsive regime of operation on gelatin and mica but in the attractive regime of oscillation on polystyrene.
   (c) Under these conditions, the cantilever is in the attractive regime of operation on mica but in the repulsive regime of oscillation on gelatin and polystyrene.
   (d) Under these conditions, the cantilever is in the attractive regime of operation on gelatin and mica but in the repulsive regime of oscillation on polystyrene.

2. Recall that it is only possible to compare material properties from a phase contrast image in tapping mode AFM if the entire image is taken in a single regime (i.e. in the repulsive regime everywhere on the sample or in the attractive regime everywhere on the sample). By comparing Figs 1 and 3, which of the following statements must be true abut Fig. 1 and why?
   (a) Phase contrast in Fig. 1a between gelatin and polystyrene cannot be related to material property contrast because the cantilever must be in the repulsive regime on polystyrene and in the attractive regime on the gelatin.
   (b) Phase contrast in Fig. 1a between gelatin and polystyrene can be related to material property contrast because the cantilever must be in the repulsive regime on both the polystyrene and on the gelatin.
   (c) Phase contrast in Fig. 1a between gelatin and polystyrene cannot be related to material property contrast because the cantilever must be in the attractive regime on polystyrene and in the repulsive regime on the gelatin.
   (d) Phase contrast in Fig. 1a between gelatin and polystyrene can be related to material property contrast because the cantilever must be in the attractive regime on both the polystyrene and on the gelatin.

3. By comparing Figs 2 and 3, which of the following statements must be true abut Fig. 2 ?
   (a) Energy dissipation and Virial on the gelatin is greater than on mica.
   (b) Energy dissipation on the gelatin is greater than on mica and the Virial on the gelatin is less than on mica.
   (c) Energy dissipation and Virial on the gelatin is less than on mica.
   (d) Energy dissipation on the gelatin is less than on mica and the Virial on the gelatin is greater than on mica.
4. Using the information provided inFig.3, which of the following is true about the topography images in Figs. 1 and 2?
   (a) The apparent height of the gelatin film on polystyrene is ~3 nm more than its actual height relative to polystyrene and the apparent height of the gelatin film on mica is ~9 nm less than its actual height relative to mica.
   (b) The apparent height of the gelatin film on polystyrene is ~3 nm more than its actual height relative to polystyrene and the apparent height of the gelatin film on mica is ~9 nm more than its actual height relative to mica.

(c) The apparent height of the gelatin film on polystyrene is ~3 nm less than its actual height relative to polystyrene and the apparent height of the gelatin film on mica is ~9 nm more than its actual height relative to mica.
(d) The apparent height of the gelatin film on polystyrene is ~3 nm less than its actual height relative to polystyrene and the apparent height of the gelatin film on mica is ~9 nm less than its actual height relative to mica.

5. What can one say about the tip-sample contact time and peak force exerted by the oscillating tip in Fig.2?
    (a) Peak force on mica is ~30-60 times larger than on gelatin and the contact time is correspondingly smaller.
    (b) Peak force on mica is ~30-60 times smaller than on gelatin and the contact time is correspondingly larger.
    (c) Peak force on mica is ~1700-17000 times larger than on gelatin and the contact time is correspondingly smaller.
    (d) Peak force on mica is ~1700-17000 times smaller than on gelatin and the contact time is correspondingly larger.
6. If the sample shown in Fig. 2 were to be imaged using Frequency Modulation AFM (using the implementation described in class), which of the following statements would be likely true?
    (a) If imaging the sample at a positive frequency shift, the excitation (dissipation) map would be smaller on the gelatin compared to mica and the frequency shift would be identical on both gelatin and mica.
    (b) If imaging the sample at a positive frequency shift, the excitation (dissipation) and the frequency shift map would be smaller on the gelatin compared to mica.
    (c) If imaging the sample at a positive frequency shift, the excitation (dissipation) map would be larger on the gelatin compared to mica and the frequency shift would be the same on both mica and gelatin.
    (d) If imaging the sample at a positive frequency shift, the excitation (dissipation) and the frequency shift map would be larger on the gelatin compared to mica.

[1] "Interpretation of tapping mode atomic force microscopy data using amplitude-phase-distance measurements", by X. Chen, M.C. Davies,C.J. Roberts, S.J.B. Tendler, P.M. Williams, J. Davies, A.C. Dawkes, J.C. Edwards, *Ultramicroscopy*, **75**, 171, 1998.