Experiment Nr. 42 - Atomic Force Microscopy

(Exploring the molecular scale in real space)

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0. Abstract

The Atomic Force Microscope (AFM) enables you to have a look on surfaces on a molecular level. Instead of only pictures real topography data are collected. You will learn this on a technological example, a lithographic mask, and on a scientific example, a polymer surface.

1. Introduction

Imagine a microscope that creates three-dimensional images down to the atomic scale, that works in air and in liquid as well as in vacuum, that uses a technique for which biological specimens need no staining, and that can map electronic, mechanical, and optical properties, and, moreover, that can manipulate a surface to the level of moving atoms one by one. These are the remarkable capabilities of scanning probe microscopy (SPMs), which is being used to solve problems in fields from condensed-matter physics to biology. SPM can be used to study the structure and physical properties of the specimen surface. The exact nature of these problems depends on the field of research. In semiconductor physics, SPM techniques might be applied to investigate the arrangements of atoms at the surface or their electronic states. In biology, the questions relate to the structure and interaction of molecules adsorbed to inert or biological surfaces. In manufacturing, SPM provides quantitative topography for silicon wafers, lithography, compact-disc production, and so forth.

SPMs have no lenses. Instead, a "probe" tip is brought very close to the specimen surface (few nanometer), and the interaction of the tip with the region of the specimen immediately below it is measured. The type of interaction measured defines the types of SPM: When the interaction measured is the force between atoms at the end of the tip and atoms in the specimen, the SPM technique is called atomic force microscopy; when the quantum-mechanical tunneling current is measured, the technique is called scanning tunneling microscopy. Atomic force microscopy and scanning tunneling microscopy are the parents of more than a dozen SPM techniques. Think of a physical property, and there is likely to be an SPM technique to measure it.

During this laboratory course you can learn the fundamentals for the use of an Atomic Force Microscope (AFM) and how to get information from the surface on a nanometer level.

2. Atomic Force Microscopy

The AFM was invented in 1986 by Binnig, Quate and Gerger [1]. It was the first of the SPMs, which overcame the limitation of STM (Scanning Tunnelling Microscope) in imaging thin samples on electrically conductive materials.

In Figure 1.1 a sketch of an AFM is presented. The five essential components of an AFM are: A sharp tip mounted on a soft cantilever spring; a way of sensing the cantilever's deflection; a feedback electronic system; a display system that converts the measured data into an image; a mechanical scanning system.

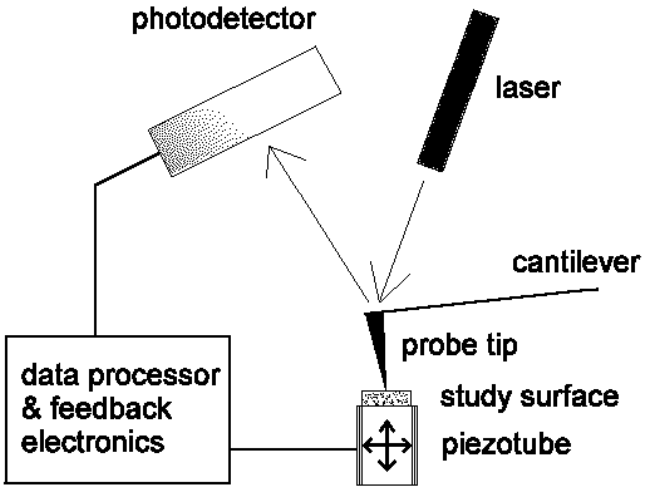


Figure 1.1: Schematic of an Atomic Force Microscope.

The tip (that part which directly interacts with the sample) is mounted on the cantilever. Forces between the tip and the sample deflect the cantilever. The cantilevers deflection is detected and converted into an electronic signal that is utilized to reconstruct an image of the surface. One of the most utilised methods to detect the cantilever deflections is the *optical method*: It consists in focusing a laser beam on the back of the cantilever and measuring the displacements of the reflected beam on a multiple segment photodiode. The corresponding signals are acquired and processed by a feedback electronic. The feedback system is used to control the cantilever deflection and to direct consequently the piezoelectric scanner movements. Usually, the sample is mounted onto a piezoelectric translator that moves the sample in the x, y and z directions underneath the tip.

When the tip translates laterally (horizontally) relative to the sample surface, one measures the sample *topography*.

When the tip moves back and forth at one fixed point of the sample surface, the forces between tip and surface deflect the cantilever. One measures the cantilever deflection Z_c and the force

acting on the cantilever is the product of the cantilever spring constant k and the cantilever deflection Z_c. This yields two curves (for the approach and the withdrawal), the so-called *force-distance curves* (Figure 1.2). Each curve is characterised by a *zero line* and a *contact line*. In the approaching region, when tip and sample are still far away from each other, the cantilever is at the equilibrium position and the detected force is zero (*zero line*). On further approach, the cantilever will be deflected by the surface forces. At a certain distance one can observe an abrupt jump of the tip onto the sample surface that corresponds to a point of discontinuity in the force-curve (*snap-in*). This occurs if the gradient of the attractive forces becomes bigger than the sum of the elastic constant and of the gradient of repulsive forces. When the distance is further decreased, the tip is pressed against the sample (*contact line*) until a user defined force value is reached. At this point, the direction of the sample motion is inverted and the tip is withdrawn from the sample. At a certain distance the tip detaches from the sample (*snap-out*) and the cantilever comes back to its equilibrium position. A zero force is acquired again (*zero line*). AFM can measure forces from pN to nN. During the approach, surface forces can be measured; the force corresponding to the *snap-out* is equal to the adhesion force between tip and sample. The slope of the *contact line* provides information on the sample stiffness.

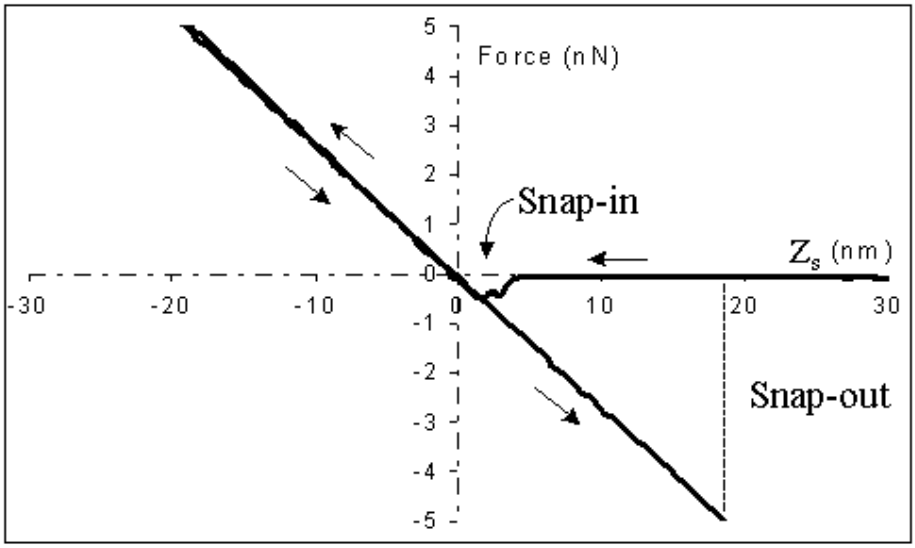


Figure 1.2: Typical force distance curves (one cycle of approach and retraction), acquired between a silicon nitride tip and a silicon wafer, in air. The force corresponding to the snap-out is always greater than the snap-in force due to sample deformations by the tip during the contact. Thus, the increased contact area increases the adhesion force. Moreover in ambient air, a water meniscus forms at the tip and acts against the tip pull- off from the surface.

3. Fundamentals of theory

3.1 Surface Forces

In the following paragraph, we are going to give a brief introduction about the intermolecular forces that can act on the tip during a scan. For an exhaustive treatment, the Israelaschvili text is highly recommended [2].

The basic principle of an AFM is that it is easy to make a cantilever with a spring constant weaker than the equivalent spring constant between atoms [1]. For example, the vibration frequencies w of atoms bound in a molecule or in a crystalline solid are typically 10^{13} Hz. Together with a mass m of the atoms in the order of 10^{-25} kg, a interatomic spring constants k (given by w^2m) of about 10 N/m is received. For comparison, the spring constant of a piece of household aluminium foil that is 4 mm long and 1 mm wide is about 1 N/m. By sensing Ångstrom-size displacements of such a soft cantilever spring, one can image atomic-scale topography. Furthermore, the applied force will not be large enough to push the atoms out of their atomic sites.

A force microscope measures the forces between two macroscopic bodies, not between single atoms. This leads to several consequences. First, the net force is stronger than the intermolecular forces are and it acts at much larger distance. Even at 10-100 nm range, the interaction energy, which is proportional to the radius of the tip, can exceed k_BT (k_B is the Boltzmann constant, T the temperature). For example, when considering only the attractive force f in vacuum, it decays as $f \sim D^{-2}$ between a spherical tip and a flat surface (D being the tip-surface separation) compared to $f \sim r^{-7}$ for the attraction between two atoms (r being the distance between two atoms). The lower force gradient decreases the vertical resolution of the microscope. The long-range nature of the forces increases the effective interaction area and limits the lateral resolution. Secondly, the deformation of the bodies upon contact increases the contact area and results in additional contribution to the net force.

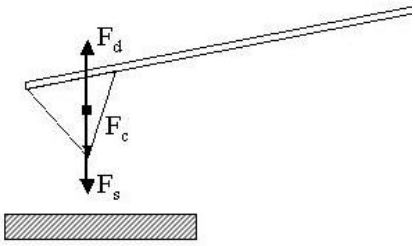


Figure 3.1: Scheme of an AFM probe: a sharp tip mounted on a cantilever. The interaction force $F_i=F_s+F_d$ is a sum of many interatomic interactions, where F_s is the surface force and the force F_d results from the sample deformation. The interaction force is balanced by F_c due to the cantilever bending.

The forces that contribute to the net force exerted on the tip can be divided in three groups (Figure 3.1): (i) surface forces, F_s, (ii) forces due to the sample

deformation, F_d, and (iii) the elastic force of the cantilever, F_c. All three forces can be of either sign.

(i) Surface forces. An elementary constituent of the interaction between a flat, rigid substrate and a sharp, rigid tip in vacuum is the pair potential between atoms at the tip and the sample. The origin of the intermolecular forces is essentially electrodynamic. At large distances (≈1-30 nm) the forces are attractive and are described by a van der Waals pair potential $w(r)=-C/r^6$, where C is the interaction constant determined by the polarizability and dipole moments of the molecules. Three different terms contribute to the van der Waals forces: the Keesom interaction (between two free rotating dipoles), the Debye interaction (between a dipole and a single charge) and London interaction (between induced dipoles). Usually the London or dispersion term is dominating. In order to relate the atomic interaction to the interaction between the macroscopic tip and the macroscopic substrate, one has to sum up all intermolecular potentials between each atom in the substrate and the tip [2]. For a sphere-surface potential, which is a good approximation for the interaction between the tip and the sample, the attractive part of the interaction energy becomes

$$W(D) = -\frac{AR}{6D} \qquad (3.1)$$

where A is the Hamaker constant, R is the radius of the spherical tip, and D is the tip-surface distance (Figure 3.1a). This gives the attractive force:

$$F_a = -\frac{dW}{dD} = -\frac{AR}{6D^2} \qquad (3.2)$$

For a typical value of the Hamaker constant in vacuum, $A=10^{-19}$ J, the attractive force emerging between a tip with an apex radius of 10 nm and a surface separated by 1 nm distance is F_a=1 nN. This value sets an approximate scale of the forces that are sensed by the atomic force microscope.

The van der Waals force depends on the medium between tip and sample because the Hamaker constant contains the dielectric constants of all the three media (Figure 3.2b). In ethanol, for instance, the attractive van der Waals force is about 5 times smaller than in water. Therefore, imaging in ethanol does less harm to the sample because the interaction between tip and sample can be reduced. Van der Waals forces can even be repulsive if the two interacting solids are not made of the same materials and if the gap in between is filled with liquid of dielectric constant smaller or higher than the others two dielectric constants. In addition, the surrounding medium can contain ions and dissolved molecules. This can change the interaction potential in a

complicated fashion, depending on the molecular composition, pH and ionic strength of the medium [2].

Under ambient conditions, the atmosphere contains water. Depending on the relative humidity, water can condense around the contact site and results in capillary forces (Figure 3.2c). The meniscus curvature varies with the relative vapour pressure and the tip shape [2]. For a spherical tip with radius *R*, when the shape of the meniscus is spherical and the radius of the meniscus is small compared to *R*, the capillary contribution to the adhesion force can be calculated as

$$F_{cap} = 4\pi R\gamma_l \cos\Theta \qquad (3.3)$$

where γ_l is the surface tension of the condensing vapour and Θ is the contact angle between the meniscus and the substrate. For water with γ_l=73 mN/m and small contact angle, the capillary force is F_{cap}=9 nN.

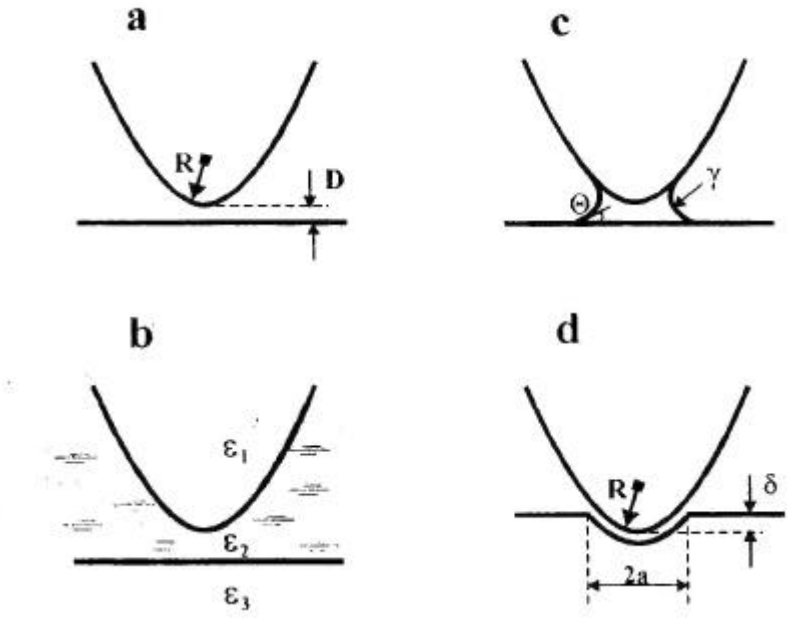


Figure 3.2: Different types of the tip-sample interaction: (a) rigid tip and rigid surface in vacuum, (b) interaction in a dielectric medium, (c) capillary condensation of water vapour in the contact area (d) deformation of a soft sample induced by a rigid tip.

At shorter distances (in the order of Å) the *repulsive forces* start to dominate. The repulsive interaction between two molecules can be described by the power-law potential $\sim 1/r^n$ (n>9) caused by overlapping of electron clouds resulting in a conflict with the Pauli exclusion principle. For a completely rigid tip and sample whose atoms interact $\sim 1/r^{12}$, the repulsion would be described by $W \sim 1/D^7$.

(ii) Forces due to the deformation of samples. So far the tip of the AFM and the sample have been assumed to be rigid. While this is often a good approximation for the tip, samples (especially organic specimen) are often significantly deformed elastically by the tip. The simplest approach to describe elastic deformation of the sample is the Hertz theory [2]. The relation between the deformation force F_d and the contact radius a is given by (Figure 3.1):

$$F_d = \frac{Ka^3}{R} \qquad (3.4)$$

where K is the elastic modulus of the tip-sample contact with $K = \frac{4}{3}\left[\frac{1-n_t^2}{E_t} + \frac{1-n_s^2}{E_s}\right]^{-1}$, E_t, n_t and E_s, n_s the Young's moduli and Poissons's ratios of the tip and sample, respectively. For a typical contact radius a=5 nm, K=l GPa and R=10 nm, the deformation force will be F_d=12.5 nN. In order to include the effect of the surface forces on contact deformation, two main models have been developed: Derjaguin, Muller and Toporov (DMT) and Johnson, Kendall, and Roberts model [2]. The choice of the model (DMT or JKR) depends on the experimental configuration in AFM force measurements. For large, soft solids, the JKR model describes the situation realistically. For small, hard solids it is appropriate to use the DMT model. In this model a "neck" is built between both solids in the contact area resulting in an enhanced contact radius (like a combination between Figure 3.2c and 3.2d). In the JKR model the contact radius a is given by the following expression:

$$a = \left(\frac{R}{K}\right)^{1/3}\left(F_d + 3pRw_a + \sqrt{6pRF_d w_a + (3pRw_a)^2}\right)^{1/3} \qquad (3.5)$$

where F_d is the deformation force (or applied load), w_a is the work of adhesion. For w_a=0 the results of Hertz are obtained. The models have two consequences in common which are not included in the Hertz model. First, they predict a finite contact area even if no external force is applied and secondly, both require an opposite external force (pull-off or adhesion force) to separate the two bodies. For the DMT model the adhesion force is related to the surface energy γ (or to the cohesion energy $w_c=2g$) of the solid surfaces in the medium used as

$$F_{adh} = 4pRg \qquad (3.6)$$

This equation assumes that the tip and the sample are of the same material. If the tip and the surface are made of different materials, the cohesion energy w_c should be replaced by the work of adhesion w_a. For a hydrocarbon polymer, where mainly dispersion forces are responsible for the tip-surface interaction, the work of adhesion can be estimated as $w_a = 2\sqrt{g_t^d g_s}$, where $g^d{}_t$ denotes the dispersion part of the tip-surface energy of the tip and g_s is the surface energy of the

sample [2]. For a silicon tip and a hydrocarbon polymer surface (g_t=100 mJ/m^2 and g_s=25 mJ/m^2, respectively) the adhesion force will be about F_{adh} = 6 nN.

The tip-sample deformation and the capillary forces are the two major factors that limit the lateral resolution of the sample because they increase the effective size of the probe.

(iii) Spring force of the cantilever. The interaction forces between sample and tip are balanced by the elastic force due to the cantilever bending: $F_c = kDZ_c$, where k is the spring constant of the cantilever and DZ_c is the measured cantilever deflection. Summarizing, the deflection of the cantilever, DZ_c, results from a combination of deformation and surface force: $F_c = F_d + F_s$. For example, the total surface force between a polymer surface and an AFM tip can be estimated as $F_s \approx$ 15 nN [3]. With a cantilever of k = 0.4N/m, a net repulsive force of 0.4 nN will be measured corresponding to 1 nm deflection. Since in this case the surface and deformation forces are of opposite sign (see Figure 3.1), they result of the same order of magnitude too. Therefore, surface forces should be as small as possible to minimise damage and indentation of soft polymer samples. For example, sharp probes show a lower capillary attraction and lower adhesion forces, and therefore enable gentler probing of a soft polymer than a blunt tip. A sharp tip can also be moved in and out of the sample layer more readily than a blunt tip. This is particularly important for tapping imaging mode described in the next Section.

3.2 Operating modes

The image contrast can be achieved in many ways. The three main operating modes are distinguished on the interaction that they experience.

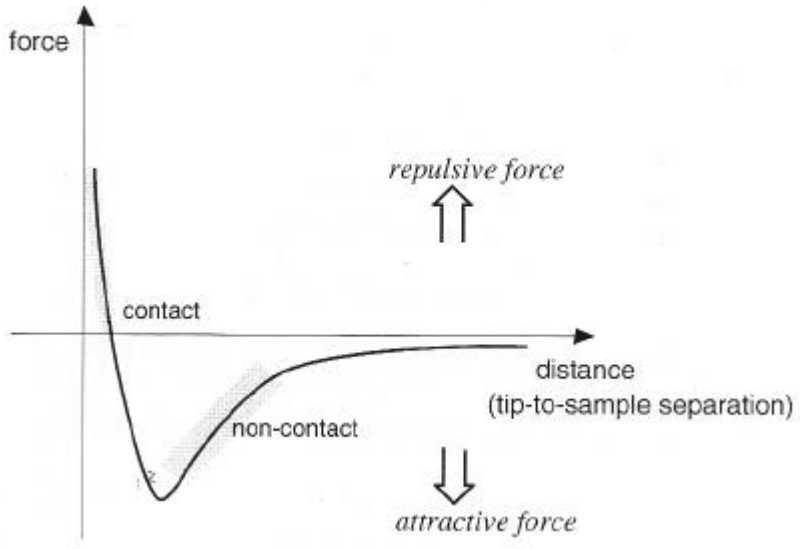


Figure 3.3: Sketch of the interaction force versus tip-sample distance. Typical values for the force and the distance are given in Figure 1.2.

a) Contact Mode (CM) As the name suggest, the tip and sample remain in close contact as the scanning proceeds. By "contact" we mean the repulsive regime of the force curve (less than few angstrom) (see Figure 3.3). While the tip scans the surface, the cantilever deflection changes due to the surface profile and a feedback loop maintains a constant cantilever deflection by changing piezo-voltages. The image is obtained displaying the piezo-voltages.

Advantages:

- High scan speed (5 Hz, the real velocity depends on the scan size)
- The only mode that can obtain "atomic resolution" images
- Rough samples with extreme changes in topography can sometimes be scanned more easily

Disadvantages:

- The high lateral (shear) and normal forces can damage soft samples (i.e. polymers or biological samples)

b) Tapping Mode (TM) The cantilever is oscillated below its resonant frequency and with a higher oscillation amplitude (20 to 100 nm). The cantilever is positioned above the surface so that it touches the surface for a very small fraction of its oscillation period. The oscillation amplitude is the signal responsible for the imaging contrast.

Advantages:

- High lateral resolution (1 nm)
- Imaging condition more stable than in NCM (see below)

Disadvantages:

- Lower scan speed than contact mode
- Higher normal forces than NCM (see below)

c) Non Contact Mode (NCM) The cantilever is oscillated above its resonant frequency with small oscillation amplitude (<10 nm). The tip does not touch the sample that means that the tip-sample distance corresponds to the attractive force regime (mainly van der Walls forces). The resonance frequency of the cantilever is decreased by the attractive forces and this in turn changes the oscillation amplitude. The image is obtained keeping the amplitude constant.

Advantages:

- Lower lateral resolution (1 to 5 nm)
- Lower lateral and normal forces and less damage to soft samples

Disadvantages:

- Lower scan speed than contact mode
- Quite instable imaging conditions

The images of this laboratory course will be taken in TM or NCM.

3.3 Introduction to polymers

A polymer is a macromolecule composed of many monomer units or segments. If all the monomer units are the same, it is called a **homopolymer**, if different, a **copolymer**. Proteins are copolymers of amino acids.

If you have only a homopolymer, in the bulk it appears homogeneous. Mixing two different homopolymers, they usually repel each other and separate into two phases. An example from the everyday life it is the mixing oil and water: the oil drops will float on the water surface.

If one chemically links two different homopolymers, one obtains so-called **diblockcopolymers**. The linkages inhibit the macroscopic phase separation but the two blocks still keep on repelling each other. Figure 3.4 shows how these systems react: the two species (let's called them A and B) still segregate but the domains have only mesoscopic dimensions corresponding to the sizes of the single blocks. In addition, as all domains have a uniform size, they can be arranged in regular manner. As a result ordered mesoscopic lattices emerge.

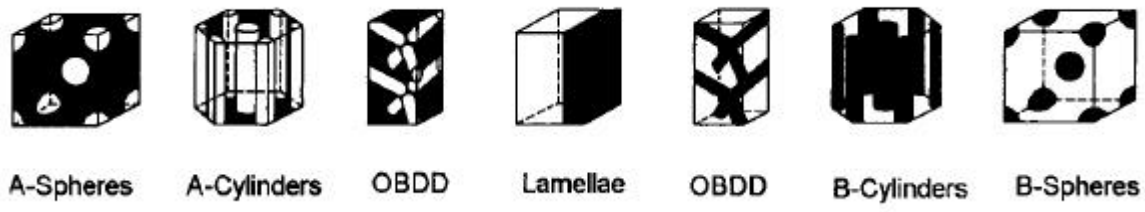


Figure 3.4: Different classes of structures in diblock-copolymers. According to the ratio between the degree of polymerisation of the A's and B's, N_A and N_B, the different classes form. For $N_A << N_B$ spherical inclusions of A in a B-matrix are formed and they set up a body-centred cubic lattice. For larger value N_A, but still $N_A < N_B$, the A-domains have a cylindrical shape and they are arranged in a hexagonal lattice. Layered lattices form under essentially symmetrical conditions, i.e. $N_A \approx N_B$. Then, for $N_A > N_B$, the phases are inverted, and the A-blocks now constitute the matrix. The 'ordered bicontinuous double diamond' (OBDD) structures exist only in a narrow range of value N_A/N_B, between the regime of the cylindrical and lamellar structures.

Switching now from the bulk to a surface, one degree of freedom is missing for the structure formation. In addition new contributions have to be taken into account.

The interactions polymer-surface, polymer-solvent and solvent-surface drive the system behaviour. In Figure 3.5 we have, initially, a solid surface of A in contact with a solution of molecules C in the solvent B. Again there are three possibilities:

1. Molecules C are attracted to A while molecules B will be repelled from it, and an adsorbed monolayer or film of C will be energetically favourable (Figure 3.5b). In this case C is said to **wet** the surface.

2. Molecules C are repelled from the surface and form clusters in the bulk solvent B (Figure 3.5c).

3. Finally, when both molecules B and C are attracted to the solid surface, no uniformly adsorbed film of B or C will form but different regions of the interface will collect macroscopic droplets of the C phase (Figure 3.5d). This is known as **dewetting**.

When the total surface energy of the whole system is minimized, the contact angle θ formed by these droplets is given by:

$$g_{AC} + g_{BC}\cos q = g_{AB} \qquad \text{(Young equation)}$$

where $\gamma_{AC,}$ $\gamma_{BC,}$ and γ_{AB} are the interfacial energies. The interfacial energy γ_{AB} of the A-B interface is the free energy change necessary to bring these two surfaces into contact.

When a polymer dewets the surface, lateral structures will appear. Also a homogeneous film can become unstable and rip into holes. For the building of holes, two different mechanisms can be responsible: a binodal (nucleation) and a spinodal (amplification of thermal fluctuation at the surface) mechanisms. One can distinguish between the two processes from the presence of a dominating length scale on the surface for the spinodal dewetting and from the dynamic of the holes growth for the nucleation.

For further reading, see the enclosure.

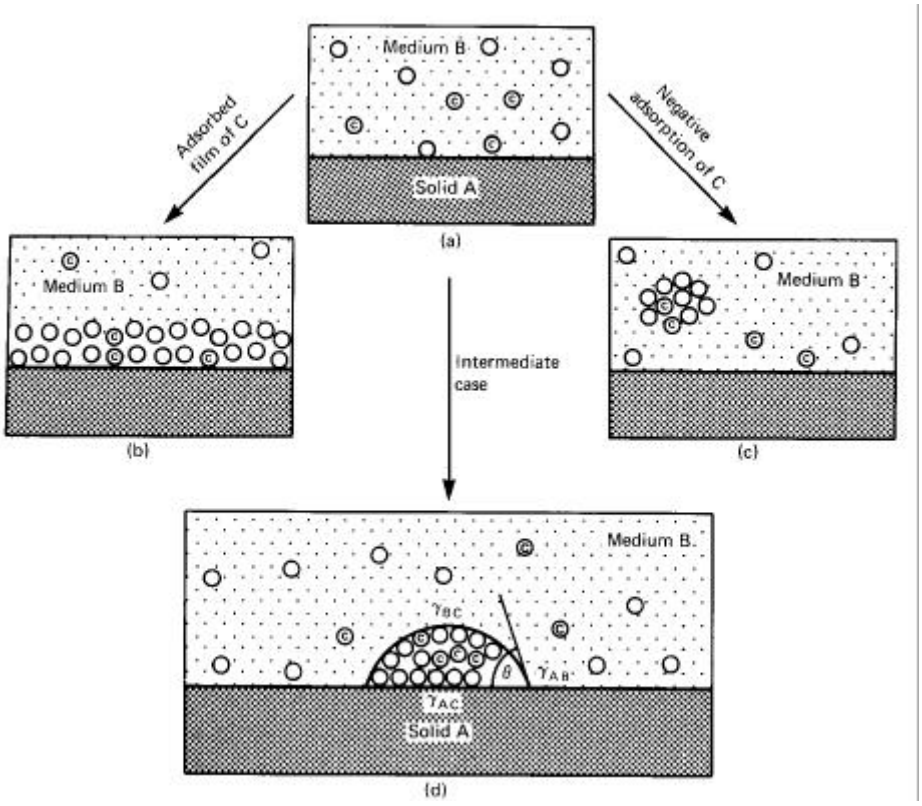


Figure 3.5: (a) Low concentration of molecules C in medium B. (b) *Wetting:* an adsorbed film of C develops and grows in thickness as the concentration of C in B increases. (c) *Unwetting*: resulting from repulsion between C and A molecules. (d) *Dewetting*: intermediate case between the two above, corresponds to the $-1<\cos\theta<1$.

4. Utilised apparatus

The system that you are going to utilise (supplied by ThermoMicroscopes-Veeco, California) consists of the four principal components illustrated in Figure 4.1:

- An AutoProbe instrument, that is the AFM itself;
- An Optic unit with video monitor, that allows to look at the cantilever and at the sample surface from the top view;
- An AutoProbe Electronics Module;
- Computer, Monitor and Keyboard.

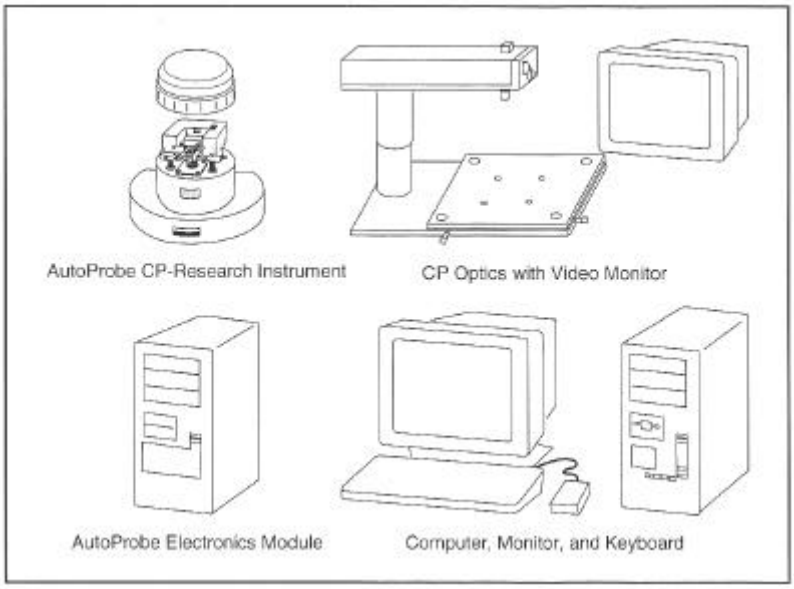


Figure 4.1: AutoProbe System Components

The Autoprobe instrument is shown in details in Figure 4.2. The primary components are:

- The probe head that includes the laser, the detector and the cartridge with the cantilever;
- A manual XY stage to move in a rough way the cantilever over the sample surface;
- A motorized Z Stage to move the cantilever towards and away from the sample for big distances (~cm);
- A sample holder where the sample is fixed;
- A scanner to translate the sample in the X, Y and Z directions. The maximum scan range is ~90 µm laterally and 7.5 µm vertically.

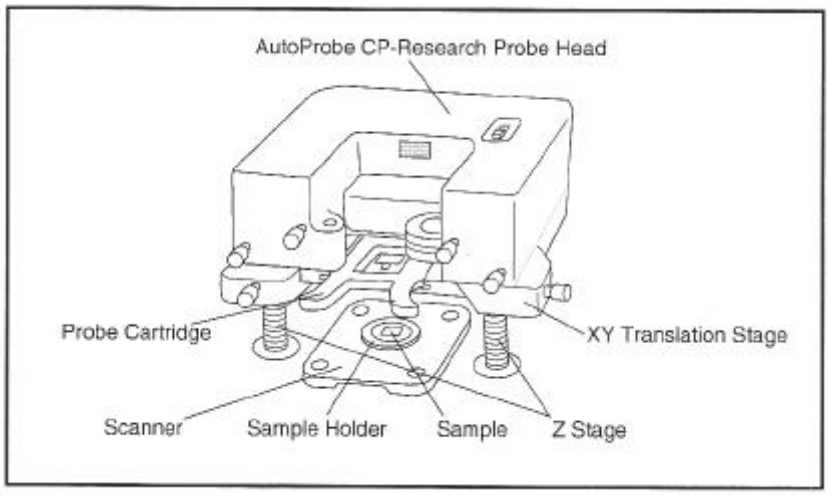


Figure 4.2: Instrument Components

The utilised tips have a conical shape with a very small apex angle (Figure 4.3) that allows a better following of the sample surface profile. The tip is placed on a triangular shaped cantilever made of silicon. The elastic constant *k* and resonance frequency *w* of the cantilever vary according to its dimensions and thickness. On the following table are listed the characteristics of the cantilevers that you can use (L is the length, W is the width):

Type	L (µm)	W (µm)	Thickness (µm)	Force Constant (N/m)	Resonance Frequency (KHz)
A	180	25	2	2.1	80
B	180	38	2	3.2	90
C	85	18	2	13	280
D	85	28	2	17	320

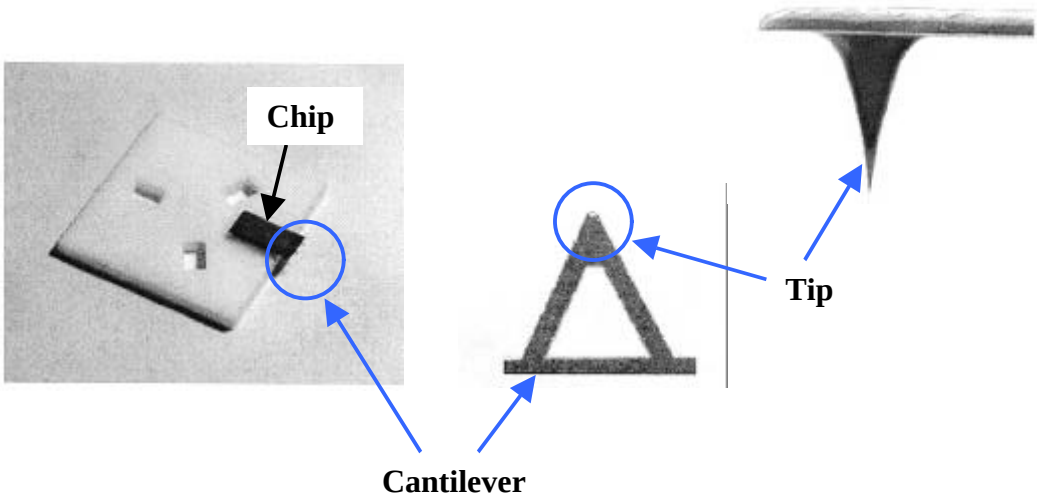


Figure 4.3: Image of the tip and cantilever utilised.

You will be told by the instructor which type of cantilever you will use in your experiments. The cantilevers are on a rectangular silicon chip that is attached on a white carrier, inserted in the cartridge. The cartridge that is fixed in the probe head (Figure 4.2).

5. Instructions for the use of an AFM AutoProbe

The software that operates the AFM is called ProScan Data Acquisition. On the screen you will visualize the following page (Figure 5.1):

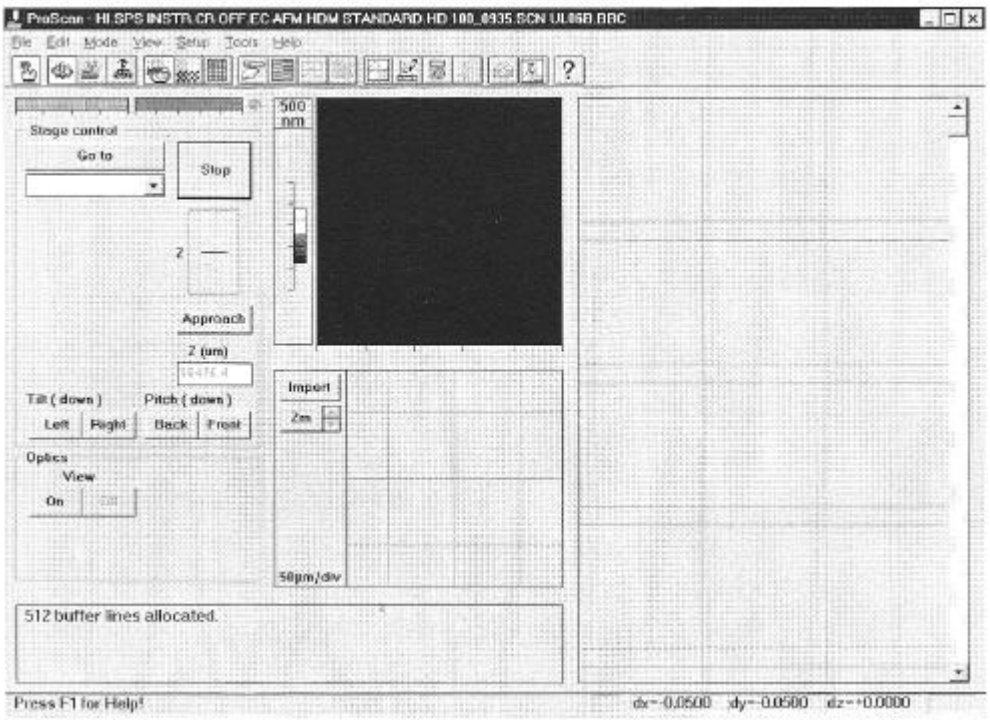


Figure 5.1: ProScan data Acquisition screen

In the following paragraph are described the steps necessary to configurate the software before a measurement, to insert sample and cantilever and to acquire an image.

Setting the Scan Parameters

Select **Setup→InputConfiguration** to open the InputConfiguration dialog box. Select the *Topography* signal in *Trace* (→), *Retrace* (←), the *Error* signal and *Phase*. These are the 4 different signals that you're going to measure. The *Topography* signal generates an image of the sample's surface using the voltage signal that is applied to the scanner in order to keep the amplitude value constant (see Figure 5.2). The *Error* signal generates an image using the error signal (i.e. difference between the probe signal and the set point) that is sent to the feedback

loop at each point in a scan. This type of image gives a measure of how well the feedback loop is tracking topography. A large error excursion indicates poor surface tracking, where edges are generally accentuated in the image. The *Phase* signal is obtained measuring the phase difference between the oscillations of the cantilever driving piezoelectric translator and the detected oscillations. This signal is very sensitive to the sign of the forces between tip and sample. It gives an impression of the mode used, TM or NCM.

Disable the AC Track command.

Select **Setup→ScanConfiguration** to open the ScanConfiguration dialog box. Choose 256×256 number of data points used to collect an image. Choose Scan Pause value to zero (it is the time the system pauses before collecting each line trace in the fast scan direction). Choose Over Scan value to 5% (the system increases the motion of the scanner to this percentage above the scan size. The data are collected and displayed over the scan area size only, eliminating edge effects due to the piezo histeresys).

Select **Setup→Approach** to open the Approach Parameters dialog box. Select the Re-Nulling box (calibrates the probe signal before an approach). Select Incremental in the Approach type window. The software moves the probe toward the sample via stepwise motion of the Z stage. The system extends the scanner and checks whether the set point has been reached after each set of steps. Click on [Done] to register the changes.

Select **Setup→ScanMaster** to open the ScanMaster Setup dialog box. Click the On button for the X and Y filter. The ScanMaster enables an xy detector to continuously adjust the position of the piezoelectric scanner tube to compensate for scanner non-linearity in the x and y directions. Choose for the Integral Gain and Ratio values of 0.5 and 1 respectively. Click on [Done] to register the changes.

Loading a sample

The sample is attached on a metallic disk via a soft adhesive sticker (by the responsible). Position the disk so that the sample is centered on the sample holder (see Figure 4.2). The magnet of the sample holder holds the disk securely in place.

Loading a cartridge

Switch off the laser (first icon from the left on the toolbar, see Figure 5.1).

Select **Setup→ConfigureParts** to open the ProScan Database Configuration dialog box. This box is used to configure the software so that it matches your instrument's hardware configuration. When you configure the software, you are selecting calibration parameters for the

hardware components that are installed on your system (for example, the scanner and the cantilever). Check that: Instrument→CP, the Head type→Standard, Scanner→100_1038, Head Mode→NCM, Beam bounce cantilever→UL20 A or UL20 B or UL20 C or UL20 D (given by the responsible), Electrochemistry→Off, Voltage Mode→High.

Insert the cartridge in the probe head.

Switch on the laser again (first icon from the left on the toolbar, see Figure 5.1).

Switch on the Optic (first button on the bottom-left, View On). Focus the cantilever on the screen.

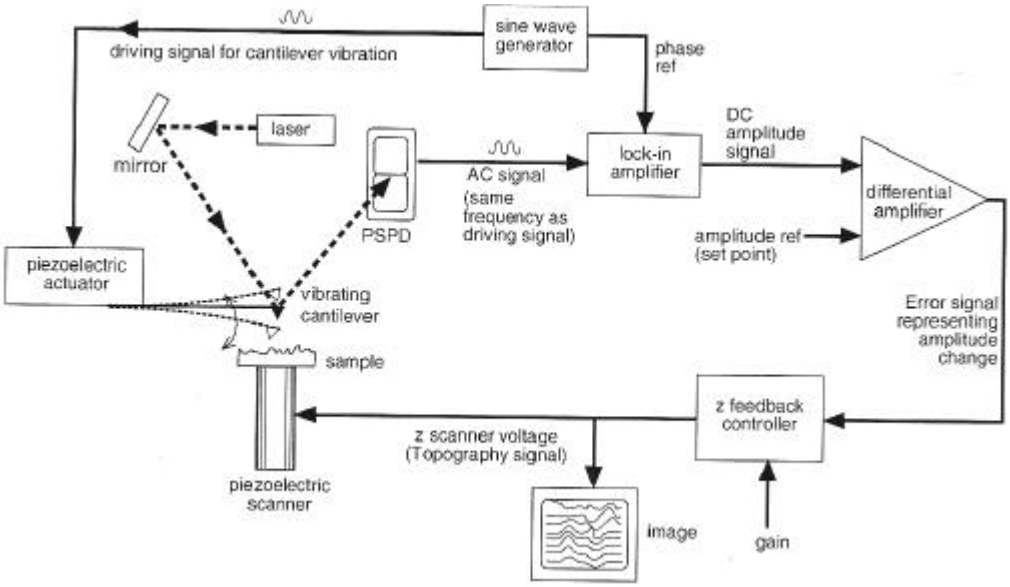


Figure 5.2: Diagram of the hardware components and signal pathways for the AutoProbe operating in NC -AFM mode.

Focusing the laser

Align the laser spot involves steering the laser spot onto the cantilever and then steering the reflected laser spot onto the Detector. Distinguish the picture and its mirror by the presence or absence of the typical laser speckles (why?).

Looking at the cantilever image on the television screen, walk the laser spot to the end of the cantilever, above the tip, using the cantilever alignment knobs (on the right side of the probe head, see Figure 5.3).

Turn the detector alignment knobs on the left side of the probe head to move the reflected laser spot to the centre of the Detector. The goal is to have the central green LED of the intensity indicators illuminated, with none of the red LEDs illuminated (Figure 5.3).

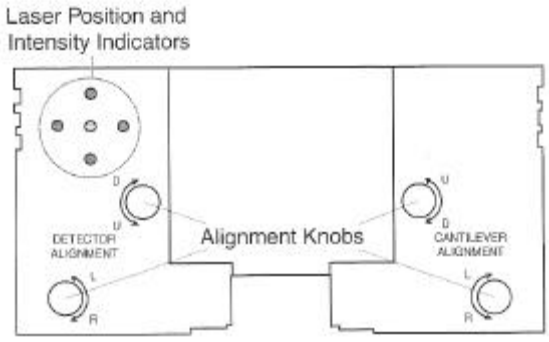


Figure 5.3: Probe head alignment controls and laser indicators

Setting NCM parameters

The scan parameters specific to NCM imaging- drive amplitude (drive%parameter), drive frequency and Imaging amplitude (set point parameter) -are set in the NCM Frequency Set dialog box (**Setup→NCMFrequency**).

Start with the drive % parameter value suggested by the system.

Click on Refresh: This prompts the system to generate a plot of the cantilever vibration amplitude as a function of drive frequency or a frequency response curve for the cantilever (Figure 5.4). The default selected sweep range covers a large portion of the total sweep range, so that the system can locate the main cantilever resonance peak.

The resonant frequency of the utilised cantilever is given in the table on page 12. Move with the cursor (+) to the right frequency region and zoom into it. Fix the drive frequency with the cursor (+) on the right side of the cantilever resonance peak.

The imaging amplitude is the amplitude of the cantilever vibration that is maintained by the system's feedback loop during a scan. The imaging amplitude is related to the value of the set point parameter. Selecting a set point value is therefore equivalent to selecting a force gradient (or tip-sample distance) that will be maintained during the scan, since the cantilver's vibration amplitude varies with the force gradient experienced by the cantilever. The value of the set point parameter is represented by the horizontal red line in the NCM dialog box (Figure 5.4). This is the default value of the set point (in this particular example is Set = -0.059). The actual value you get today will be different in number. For the moment, you can leave it unchanged.

Click on [Done] to register the parameters values.

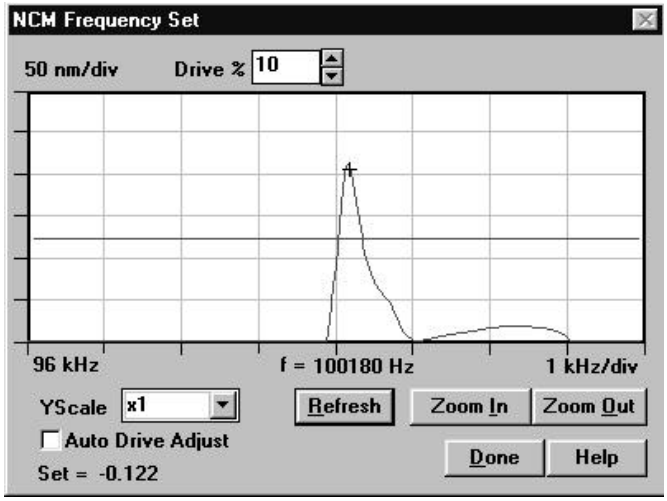


Figure 5.4: Response curve for a typical NC-AFM cantilever (the value of the resonance frequency and set point is not indicative).

Performing an approach

Focus the sample surface on the video monitor. Click with the cursor on the Z button (see Figure 5.1) until you have both sample surface and cantilever focused on the screen. The probe head moves in the z direction and approaches the cantilever to the sample. The distance from the control line determines the speed used by monitoring in z-direction. Be **very careful** at this step since you can easily crash the cantilever on the sample surface (each chip costs roughly 200 Euro). Check the actual sample surface position in the video monitor every 500 µm during your approach: note that the TRUE sample surface is on half way between the focus of the cantilever and its mirror.

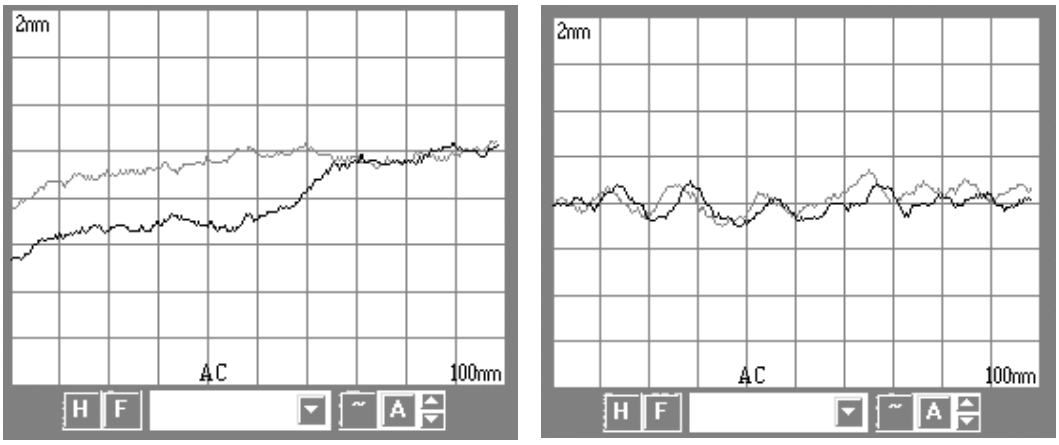
Now click on the Approach button (Figure 5.1): The cantilever will move towards the sample until the set point value is reached. The green spot directly under the toolbar blinks during the movement of the cantilever. When the blinking stops, the approaching phase is finished.

Starting a scan

Click on the third icon on the toolbar (switch on Image mode).

Put the scan size to 1 µm. On the oscilloscope window there are 2 lines corresponding to the trace and retrace signals over 1 line of the sample surface (in X direction, for example). For safety purposes, the default set point value is greater than 50% of the amplitude, Drive%. This means that the corresponding tip-sample distance will be too large for the system to detect the sample topography. Therefore the set point will need to be incrementally increased (i.e. make

the value more positive) after you perform an auto Approach. You can check when you reach the surface looking at the signals in the oscilloscope window (Figure 5.5).



(a) **(b)**

Figure 5.5: Example of a scan line when the tip is not in contact with the sample surface (a) and when is in contact (b).

When increasing the setpoint value, monitor the ZPiezobar (Figure 5.6): It is a tool for monitoring the scanner's extension and retraction in response to the feedback voltage.

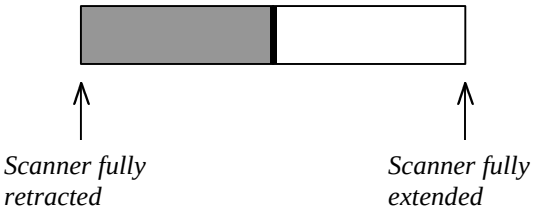


Figure 5.6: The Z Piezo bar.

As you increase the set point value, the system decreases tip-sample distance in order to achieve a greater force between tip and sample (the scanner extends). The black line in the Z Piezo bar (Figure 5.6) should be always in the middle of the bar. If the scanner is fully extended (black line to the right), the system cannot follow anymore further setpoint changes.

Before this situation occurs, click on the second icon on the toolbar (switch to Data Acquisition screen, Figure 5.1). Select "1 µm down" and press on the "Go To" button: The head probe will move down the cantilever and the scanner has to retract in order to keep the setpoint value constant.

Taking an image

Click on the third icon on the toolbar (switch on Image mode).

Click on the sixth icon on the toolbar: Select four quadrants in order to visualise the four images before selected (see under *Setting the Scan Parameters*).

Level the sample in the X and Y directions (read on pg. 20 about the slope parameters).

Click on the Image button: The system scans the sample surface in the Y direction too, creating a 2D image.

6. Tasks

6.1 *Hard sample (easy)*

Reproduce the images shown in Figure 6.1. The scan size are 5, 15, 30 and 40 µm (from the topleft image, respectively). The sample is a lithographic mask utilised in the semiconductor research. The substrate is gallium arsenid and the structures are in silicon oxide.

The goal of this first task is to get familiar with the AFM, to understand the meaning of the different parameters and how to choose them in order to get the best image.

After saving the images, analyse them and measure:

1. Value of the rms-surface roughness in the image of 5 µm scan size
2. The height and width of the structures in the image of 15, 30 and 40 µm scan size
3. If your images look different from the ones shown in Fig 6.1, explain possible reasons

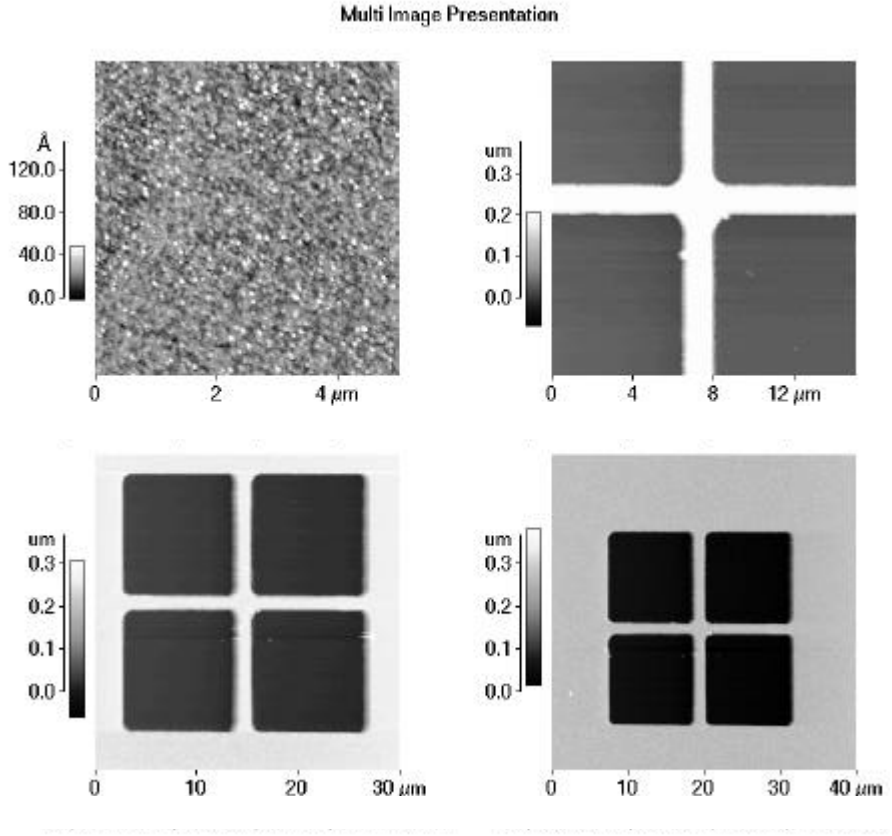


Figure 6.1: Images from a mask. The image with 5µm scan size (topleft) is made outside from the 4 squares. The squares are depression in the Silicium Oxide layer.

6.2 *Soft sample (difficult and time consuming)*

Now the sample surface is unknown to you. The sample is a polymer film deposited on a flat substrate. The polymer has created a surface structure, which has to be explored. You have to:

4. Find the parameters in order to image the real surface
5. Save images with 1, 2, 4 and 8 μm scan size
6. Calculate the value of the mean rms-surface roughness with a standard deviation from the saved images
7. Explain the physical origin of the surface structures. Why is the structure formation not in contradiction with the tendency to have a smooth film on the surface?
8. Which is the length scale of the observed structures? From this information you can distinguish if the structures arise from a single polymer chain or from the assembly of several molecules. Explain what is a self-assembly process: what is the physical meaning of this process? Why is this possible for polymers and for which system conditions? Is it possible for polymers only?
9. On the surface defects will appear. Describe the role that defects have in structure creation. Which is the driving energy that induces the structure formations?
10. Describe the imaged structures.
    Are the structures isotropic distributed over the surface? In the positive case, which are the different physical mechanisms that give rise to the structure formations? In the negative case, which physical effect creates the anisotropy?
11. Explain the differences between the observed structures, and the stationary patterns obtained in reaction-diffusion systems.

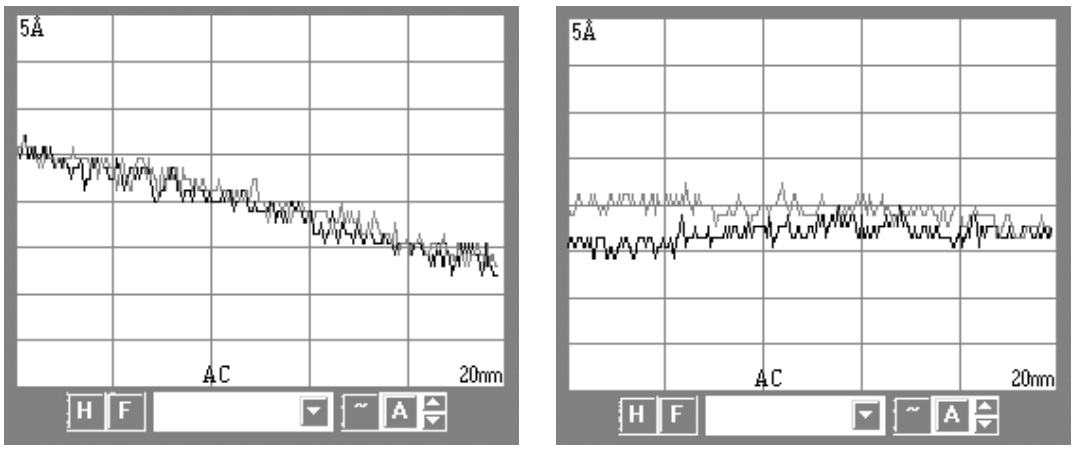
7. How to realise the tasks

Improving the image

To get the best image, you can adjust the following parameters: X and Y slope, setpoint value, Drive%, gain and scan rate.

The X, Y slope parameters values are a linear correction that is added digitally in real time to correct the scanner's x, y position. By applying corrections to the scanner, you can compensate for a slight tilt, or slope, of the sample surface. Slope occurs when the sample surface is not flat relative to the scanning plane. For instance, slope can be due to a slightly wedge-shaped sample

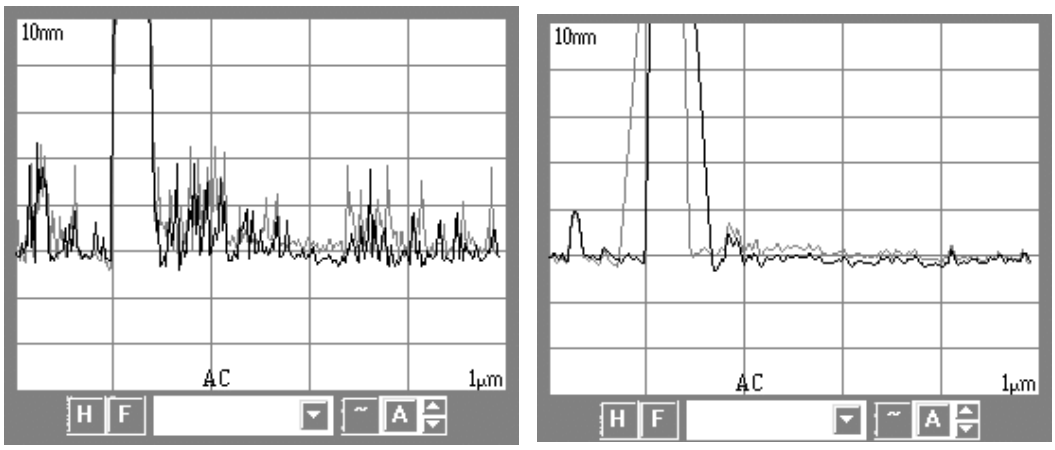
or to a wrong sample mount. You can tell if slope adjustment is needed by looking at the signal on the oscilloscope display (Figure 7.1). If the signal on the display is tilted, adjusting the slope in x and in y can often remove most of the tilt.



(a) **(b)**

Figure 7.1: Surface profile imaged with strong tilting (a) and without (b).

The gain parameter controls how much the Error signal (i.e. the difference between the probe signal and the set point value) is amplified before the signal is sent to the scanner. Higher gain values mean that the feedback loop is more sensitive to changes in the Error signal (i.e. the probe signal, since the Error signal is the difference between the probe signal and the set point). When higher values are used, surface features can be tracked more closely. However, if the gain is set too high, the Error signal will fluctuate too strongly in response to small changes. As a result, the system will oscillate. You will be able to see these oscillations in the signal trace displayed on the Oscilloscope Display (Figure 7.2).



(a) **(b)**

Figure 7.2: Surface profile imaged with too high gain value (a) and with a good one (b).

Finally, the scan rate is the frequency of the back and forth rastering of the scanner. Usually, X is the scan direction. Along the X direction, the computer collects a line of data. Movement in the Y scan direction positions the tip for the next line of data. Slower scan rate gives the system more time to respond to changes in surface topography. A good range of scan rate to use is from about 0.5 Hz for more difficult samples (with large variation in topography) to about 2 Hz for flat samples.

After parameter changes, it is possible to measure one complete image and save it (click the fifth icon on the toolbar).

For every saved image, take notes of the following values: File Name, Scan Size, Scan Rate, Set Point, Gain and Drive% (see following table). The same parameters values are valid for 4 different images: *Topography* signal in Trace (→), Retrace (←), the *Error* signal and *Phase.*

File Name	ScanSize (µm)	ScanRate (Hz)	Set Point	Gain	Drive%
06110001→03	1	1	-0.0202	0.2	5

Image Processing

With the AutoProbe Image software you can open the files, process them or extract important information like lateral distances between features, height of protuberances, roughness of the surface, Fourier Transformation for periodical lateral array and so on. Figure 7.3 shows the Auto Probe Image window:

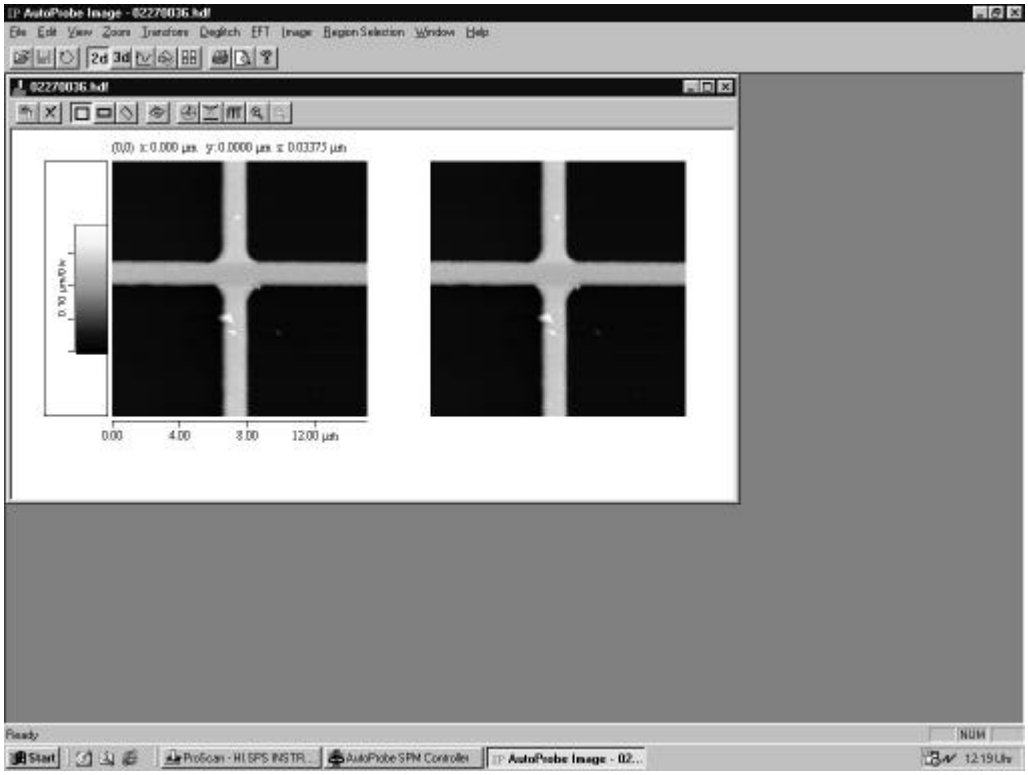


Figure 7.3: Auto Probe Image window

Some of these processes are:

Flattening: It corrects the image from possible tilting of the sample subtracting a certain surface (plane, sphere, 3rd order,) to the acquired data. For example, to actually mount the sample perfectly level is usually not a realistic goal because of the extremely small scale involved in the AFM (100 nm or less). The software allows to compensate for the sample tilt and then to measure the real height in the image. Click on the 8th icon in the second icons line: this window pops up (Figure 7.4).

If scanned in X-direction:

Click on Direction → Vertical. The profile of the surface in the direction perpendicular to the fast scan direction appears. Select Order 1 and click on Whole Image: the software subtracts a plane of the all image. Click on Update image.

Now switch to Direction → Horizontal. Subtract again an Order 1 of the Whole Image. Then, always in the Horizontal direction, select Order 2 and click Line by Line. Now the software fits to each line a 2nd order function and makes the subtraction.

Save the corrected image under another name. Do not overwrite the true data!!

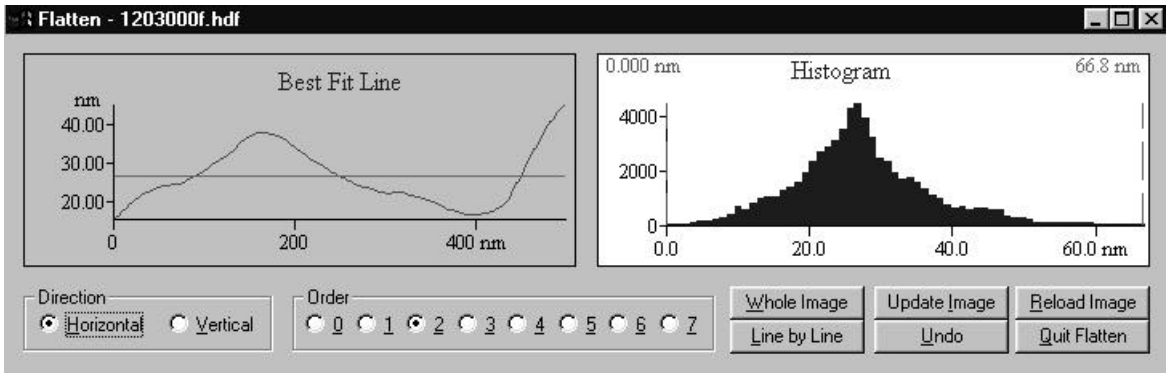


Figure 7.4: Window of the flattening command.

Line Analysis: Click on the 6th icon in the first icons line. You can fix a line over the surface and the software shows the cut through this line. You can measure the height (|) or distance (—) along it.

Region Analysis: Click on the 7th icon in the first icons line. You can select a region on your image and read the value for the roughness relative to the region. The rms roughness, for example, is the arithmetic average of the absolute values of the measured profile height deviations.

Remark about task 6.1

After saving the image of 5µm scan size, increase the scan size stepwise (10 µm and then 15 µm). When the tip reaches the depression, the height of the feature on the sample increases from few tens nm to some 100 nm. In order to improve the profile of the feature, increase the gain value to 2 or 3.

Remark about task 6.2

Since the sample is softer than the one utilised for the task 6.1, you have to bring the vibrating cantilever as close as possible to the sample surface without touching it (this is the NCM). Start with decreasing the Drive % value until the signal of the sample surface in the oscilloscope window "disappears". Then increase the set point value to reapproach to the surface until the signal appears again. If the tip gets too close to the surface during a scan, the strong attractive force can damp the vibrations. When the tip is either hitting the surface or is too close to it, glitches can occur in the signal on the oscilloscope display and the signal trace become unstable. If the tip is too close to the surface, you can decrease the value of the set point to increase the tip-sample distance. A typical gain for polymeric surface is smaller than 0.5.

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