Measuring Calibrated Polystyrene 0.3 micron Spheres

Author: G.H.J. Florussen
Report no.: GM 123, January 1996
Summary

This report consists of two separate parts. The first and more little part is about how to get an AFM tip quickly on a desired position. Because of the special configuration of the AutoProbe XL AFM of Park Scientific Instruments, it is easy to get the tip on any place on your sample. Besides an AFM tip there’s also a microscope with which targets on the sample can be searched. The distance between these two elements is measured many times by moving the stage and it seemed that there’s a discrepancy between the stored offset values (x & y) and the measured ones. This means that the offset values has to be adjusted frequently which is mainly caused by the stage positioning system. Still, the uncertainty in tip position, after locking your target with the microscope, is between the 5 and 15 microns even if the x- and y-offset were adjusted right before. In this part you find why and how the x- and y-offset have to be adjusted.

The second and major part of this report deals about measurements of calibrated (soft) polystyrene 0.3 micron spheres (NIST) with the PSI AutoProbe XL AFM. It seemed very hard but possible to visualise these little soft spheres but the height of the spheres was measured 22% too small. In order to find out why, the interaction force between the tip and a sample surface is estimated by means of a very sensitive force balance. According to this experiment it seemed that this interaction force, that a feedback loop tries to keep constant, is in the order of magnitude of a tenth or even of a microNewton. Because of this rather big interaction force it seemed that the tip is severely wearing the soft PS spheres, which is proven with an experiment. After four times scanning the same area on the sample, the spheres were scratched so much by the tip that it was impossible to interpret the fifth scan. A characteristic peak-valley value decreased for about 16 nm a scan!

Unfortunately it is not known how this scratching actually takes place but at least we know that it occurs. Because of the rather big interaction force you have to be aware of deformation and/or scratching. Especially when there’s a big difference in hardness between the silicon tip and the sample material. Soft samples are likely to be worn by the tip and hard samples on the other hand can lead to excessive tip wear which gives even more trouble. The tip will lose its well defined shape and get shorter which introduces errors in the obtained images.
Contents

1 Introduction ........................................... 3
  1.1 Atomic Force Microscopes ......................... 3
  1.2 Measuring calibrated polystyrene 0.3 micron spheres .......... 4
  1.3 The PSI AutoProbe XL .......................... 5

2 Getting an AFM tip quickly on a desired position ............ 7
  2.1 Measurement of the offset values ................. 8
  2.2 Analysis of results ................................ 10
  2.3 Adjustment of x- & y-offset ........................ 10

3 Measuring calibrated PS 0.3 micron spheres .................. 12
  3.1 Analysis ........................................ 12
  3.2 Measurement results .............................. 14
  3.3 Interaction forces between tip and sample ............ 17
    3.3.1 Force balance ................................ 18
    3.3.2 Approximation of interaction force ............ 20
  3.4 Sample wear ................................... 21

4 Conclusions .......................................... 23

5 Appendices ........................................... 25
Chapter 1

Introduction

1.1 Atomic Force Microscopes

An AFM microscope is one kind of a SPM (Scanning Probe Microscopes) just like STM, SEM, NSOM etc. With this kind of microscopes atomic resolution is possible. A big advantage of an AFM is the possibility of imaging non-conductive materials. This means that non-conductive samples can be researched without the necessity of putting a conductive layer on top of its surface which introduces uncertainties.

An atomic force microscope has a very small cantilever on which a tiny and very sharp (pyramidal) tip is mounted. This tip is moved in contact, relative to a sample surface in a certain pattern (successive line-scans are made to obtain a rectangular area) while a feedback-loop tries to keep the deflection of the cantilever constant. Therefore the sum of all the forces that interact between the tip and the sample surface is kept constant. The deflection of the weak cantilever (stiffness in the order of magnitude of Newtons per meter) can be measured with different techniques. The PSI AutoProbe XL uses a piezolever: piezo-resistant material on the cantilever gives a change in resistance by a change in cantilever deflection. This change in resistance is measured with a Wheatstone-bridge and this is then converted to cantilever deflection. Other deflection sensing methods are for example laserbeam deflection or tunnelling.

The scanning motion of the tip, relative to the sample surface, is made with a piezotube. This is a cylinder of piezo material with electrodes on it. When a voltage is applied to this kind of material, it will contract or extend and visa versa. Because of the cylindrical shape, the resonant frequency is very high (somewhere between 10 & 100 kHz). It is important to have the resonant frequencies that high, not only because they determine the scanning speed, but also because they determine its rigidity against vibration.

By applying voltages to this tube, it will deflect and very little motions can be made in x, y & z directions. The PSI AutoProbe XL moves the tip in all three orthogonal directions and the sample surface is fixed. By monitoring all the voltages, which are converted into displacements by a computer, that are applied to the scanning tube a topographical image of the sample surface is obtained. There are also AFM systems that move both the tip
and the sample to make a relative motion possible with each their own advantages but this is merely a choice of the designer of the machine. In the following diagram you can see how an AFM schematically works. The only difference between this scheme and the PSI AutoProbe XL is that in this scheme the sample surface is moved instead of the tip.

![Diagram of AFM scheme](image)

**Figure 1.1: scheme of an AFM**

### 1.2 Measuring calibrated polystyrene 0.3 micron spheres

At NIST, the National Institute of Standards and Technology, calibrated tiny spheres are available and sold (see appendix A). The spheres are made of polystyrene (PS) and available with a diameter of 0.3 and 0.1 microns. These spheres are put in a little bottle with a certain liquid.

Because the size of the spheres and the dimensions of the sharp tip are known, we are able to say what can be expected from the image, which should represent the topography of the sample surface. The tip-sample surface convolution can therefore be taken into account. By measuring these spheres we want to get an idea how reliable the obtained image is.

Samples were made by putting some drops with a pipet on glass plates. The drops then contain for about 0.5% weight concentration calibrated PS spheres. Some glass plates were given a greasy layer in order to get smaller and more convex drops. Raindance, Turtle Wax
and Rain X are the trade names of the greases. Also on a plain glass plate some drops were put. First the 0.3 micron spheres were used for sample preparation.

1.3 The PSI AutoProbe XL

On the PSI AutoProbe XL, besides a scanner (with a maximum range of 100 microns in x and y) on which a cantilever plus sharp tip is mounted, there's also a microscope, see figure 1.2. This microscope has a magnification of 300 times and zoomed in a magnification of 1250 times.

![Figure 1.2: The AutoProbe XL of Park Scientific Instruments](image)

The sample is put on the stage which can be moved over a large area: 209 mm in x and 236 mm in y direction. First you look straight down with the microscope for your target by moving the stage. This is the so called on-axis view. After you have found your target on your sample, you want to put the tip there. To do so, you just click a button on the windows monitor to switch to the oblique view and the stage moves automatically the x- & y-offset between the microscope and the tip. These values are stored in the computer. The oblique-view is always focussed on the AFM tip and under an angle of 45 degrees so the sample can only be seen when the tip is very close to the surface. Because searching for your target in the oblique-view is much more difficult then with the on-axis microscope, this is a convenient way of target searching by the ability of switching from view. Now,
after switching from view, the tip is supposed to be right above your target you were looking at.

Then you lower the tip until the sample surface is reached. When the tip is in contact you can make an image of your sample. The stage is now fixed and the scanner moves the tip across the sample surface and a topographical image of the sample surface is built up by successive line-scans. In this mode (topo) a feedback loop tries to keep the deflection of the cantilever constant by applying voltages to the scanner (piezo tube). These z voltages represent the height of the sample surface by a known conversion of voltage into height and an image is obtained.

However, there are some parameters that need to be adjusted before getting an image. The first two parameters are the x- and y-slope of the stage. With these two parameters the angles of the stage with respect to the granite table (with air-dempers for vibration isolation) can be adjusted. Then the z feedback loop parameters have to be adjusted. These are the z-gain (multiplier proportional action), the z cut-off frequency (frequencies in the feedback loop above this value are thrown out) and the force-setpoint. The feedback loop wants to keep the cantilever deflection (read force) equal to this setpoint. By adjusting the force-setpoint you change the amount of deflection of the cantilever that the z feedback loop tries to keep constant. So you’re able to scan a surface with bigger or smaller interaction forces. The z-gain and the z cut-off frequency are chosen as high as possible to let the tip follow the sample surface topography as good as possible. But if these two parameters are chosen too high the feedback loop starts oscillating, what must be avoided.

The stage is moved over a x-slide and driven by a screw connected to a stepper-motor. This x-slide moves over a y-slide that is quite similar. The step size of the stage, with airfoad to minimize friction, is 5 microns for x and y both. There is no feedback loop to position the stage.

In appendix B you find some drawings of the PSI AFM machine and some specifications of the used tips and cantilevers. There you can also see how the cantilever and tip are mounted on the scanner tube.
Chapter 2

Getting an AFM tip quickly on a desired position

As described in the introduction, it is possible to get the PSI AutoProbe XL AFM tip easily and quickly on the desired place just by switching of view. But how reliable are the offset values between the microscope and the tip that are stored in the computer? Is your tip, after switching to the oblique view, really above your target you looked at under the microscope in the on-axis view? There are three reasons why the offset values needs to be adjusted frequently:

- stage positioning system;
- kinematic mount that holds tip, cantilever and ceramic substrate (see appendix B);
- putting in a new/other tip.

The first item, the stage positioning system, will be discussed further on this chapter. It seemed that the offsets varies quite much; this is not due to the offset between the microscope and the tip: this is fixed, but is to due to the bad repeatability of the stage positioning. It is the movement of the stage that makes switching from the on-axis view to the oblique view and visa versa possible.

The second item also influences the offsets. Several experiments showed that by taking a cantilever plus tip, mounted on a ceramic substrate out and put back into the kinematic mount again also caused a change in offsets. The x-offset can be changed for about 260 and the y-offset for about 230 microns. Obviously the kinematic mount under the scanner is not capable of getting the same tip on the same position again after taking the tip out. This means that, after switching from view and the tip has been taken out and put back again, the tip is not on the same position where it was, so the stored offset values needs to be adjusted.

The last item is simply that no cantilever plus tip on a ceramic substrate is exactly of the same dimensions as another one. So if a new tip is put under the scanner, the offset values are altered and again the new offset values have to be put in the computer.
2.1 Measurement of the offset values

In order to find out what the offset values are, the following experiment is done:

- first find your target with the on-axis microscope and write down the x- & y-coordinates of the stage;
- then switch to the oblique view and find your target with the tip by making an image. Again write down the stage coordinates and also the position of your target in the obtained image;
- calculate how much the stage has moved in x and y and compensate these values for the position of your target in the obtained image. These are then the x- and y-offset values (that should be stored in the computer);
- repeat this all $8 \times 5 = 40$ times and plot the results. Every measurement sequence contains 5 offset measurements that are made right after each other. The 8 sequences were taken on several days.

As target a corner on a calibration grid is used but this is not really relevant. Just make sure that you use a target that is easy to identify.

One single tip is used for all these measurements and is not replaced, so offset changes due to the kinematic mount or different tips are not present in this experiment.

The AFM machine itself was used as measurement system: the machine just count the number of pulses that are send to the stepper motors and the coordinates of the stage are displayed on the windows monitor. The stage travel after switching from view, that is supposed to be the x- & y-offset, can not be done better than the performance of the stage positioning system itself. This makes the use of calibrated position measuring systems like laser-interferometers for example useless. Probably you know the offset values more accurate and you can store these values in the computer but you won't improve the stage travel between the two views.

Besides, when you start measuring the offsets, your target is probably not in the scanning area of the tip after switching of view, so the stage position has to be adjusted manually. After that, the target must be in the scanning range of the tip. Don't make the scanning range of the tip too small (smaller than 50 microns), otherwise you will loose your target easily.
The next graphic shows the measured x-offset. Every measurement sequence consists of 5 offset measurements and this sequence is repeated 8 times.

![Graph showing measured x-offset between microscope and AFM tip.](image)

**Figure 2.1: measured x-offset**

Here are the results for the measured y-offsets.

![Graph showing measured y-offset between microscope and AFM tip (+37mm).](image)

**Figure 2.2: measured y-offset**
2.2 Analysis of results

As we can see the offset values vary quite much and is not nicely constant. We also can see that besides a difference in mean value of every sequence there's also a difference in spread around it. It seemed that the spread is bigger for a cold machine then for a warmed-up one. Both offsets fit good in a normal distribution (see appendix C). For the x-offset 95% of all the measured values are within $662 \pm 12.8\ (2\sigma)\ \mu m$ and for the y-offset within $-37422 \pm 14.0\ (2\sigma)\ \mu m$. Besides, the minimun stepsize of the stage is 5 microns.

There are many possible reasons for this awkward stage positioning. These are not further researched but to mention some possibilities:

- backlash and friction (virtual backlash) between elements in the mechanical chain;
- errors in the screws (pitch angle);
- errors in the slides (straightness);
- thermal expansion of elements in the mechanical chain (room temperature is not stabilized);
- errors in the stepsize (step angle) of the stepper motors.

You don't measure the offset on the same position in the workspace of the AFM machine all the time, because the target has been put on several positions on the stage. This explains among others why errors in the screws have to be taken into account.

Now it's quite clear why the offset values has to adjusted before making a measurement sequence. First it's the awkward stage positioning; the measured mean value of the offsets was definitely not the same for every sequence. Secondly it is the kinematic mount that holds a ceramic substrate plus cantilever with tip. By doing the same experiment but now taking the substrate out and put back again, both offsets vary over 200 microns. Thirdly it's the impossibility to make every cantilever plus tip, mounted on a ceramic substrate of identical dimensions.

2.3 Adjustment of x- & y-offset

The x- & y-offset can be adjusted, in order to get the tip immediately above your target right after switching of view, in two ways:

- toolbox of the machine (software);
- adjustment of mirror screws (x & y) in the on-axis viewing optics (hardware).

The first possibility is an option in a toolbox. First you find your target with the tip in the oblique view and then you switch back to the on-axis view and find your target with the microscope (just the former experiment in reverse). Then open the toolbox and save the new offset values (manual p307).
The second possibility is a change in the slope of a mirror. This mirror is held with flexures and pushed to a x and a y screw. By turning one of the screws, the on-axis view will translate in x or y. One revolution of the x button equals a translation of 80 microns in x. For y, one revolution causes a 160 micron shift.

When you have to adjust the offset values, it’s most convenient to begin with the first (toolbox) option because the stored offset values are likely to be far from correct. The second option is more valid for little adjustments while you’re doing measurements with the machine. For example; if you notice after a while that the tip is always a bit left of your target you just turn the x button a bit. It must be said that little experience is required.

The offset values have to be adjusted before you start doing many measurements with the AFM machine in order to save time; you don’t have to search much to get the tip on your target. For only a few measurements or inspecting large homogeneous surfaces, it might be redundant to do so.

However, this still means that the uncertainty in tip position after switching of view is somewhere between the 5 and 15 microns, even if the offset values have been adjusted right before. But for most applications (like wafer-inspection) of the PSI AutoProbe XL AFM this is good enough.
Chapter 3

Measuring calibrated PS 0.3 micron spheres

The second part of this report is about the measurement of calibrated polystyrene spheres with a diameter of 0.3 microns. They’re from the National Institute of Standards and Technology (NIST) where the spheres are made, calibrated and sold. The purpose of this experiment is to see what’s happening when you are measuring a soft material, like polystyrene (thermoplastic) with an AFM. Because we know the size of the spheres and the shape of the pyramidal silicon tip that scans the sample surfaces, we can make an analysis of what should be measured and compare this with the measurement results. In the introduction 1.2 is described how the samples with spheres are made.

3.1 Analysis

The diameter of the calibrated polystyrene spheres is 0.269 microns with an uncertainty of 0.007 µm (see appendix A). The radius of the sharp tip is smaller than 100 angstroms and the the angle of the sidewalls of the silicon tip is 55 degrees. In the following analysis the tip radius is not taken into account because this is neglectable to the size of the spheres. The roughness of the glass plates is also neglected for the same reason.

In the 2-D analysis, the tip is modelled as a triangle (a cross-section of a pyramide) with a top angle (faced straight down) of 70 degrees (see fig 3.1). Actually a 3-D model should be made to extract the tip shape out of the obtained image. The apparent top angle of the tip (180-2\(\phi\)) is in fact dependant on the scanned direction. Seen over a diagonal of the base of a pyramide, the top angle is bigger than as seen over a line perpendicular to the pyramide base (see appendix D). Because of this dependance, a sphere will look a bit square in the obtained image. The orientation of such a “squared circle” is therefore dependant on the orientation of the pyramidal tip that is mounted under the scanner. Unfortunately this 3-D convolution is not good visible in the obtained images of the spheres and therefore the model is simplified to a 2-D, instead of a more correct 3-D model.

With some geometrics, the following equations can be derived. Every dimension is
written as a function of $R$ (sphere radius) and $\phi$ (angle side wall of tip). The first analysis describes the convolution of a single sphere and a tip. The second one is about a twin sphere (two spheres next to each other).

The solid fat line is what is expected to be seen in the image of the spheres. The angle $\phi$ is the angle of the side walls of the pyramidal tip with its base but is noted between the glass plate and the tip (this is a Z angle). Because of the measurement results, it seemed not to be necessary to introduce besides a $\phi_1$ (angle front wall) a $\phi_2$ (angle back wall). Those angles were measured almost the same so only one angle $\phi$ is defined. The tip is probably mounted in the scanner with its top straight down (no significant rotation is detected) and $\phi_1$ equals $\phi_2$ quite good (silicon tip is made with a precise etching process).

**Tip Single Sphere**

![Diagram of tip and single sphere]

Figure 3.1: *model of tip and single sphere*

$$x_1 = x_2 = R \left( \frac{\cos(\phi) + 1}{\tan(\phi)} \right) \quad (3.1)$$

$$y_1 = y_2 = R \left( \cos(\phi) + 1 \right) \quad (3.2)$$

$$w = 2R \left( \frac{\cos(\phi) + 1}{\tan(\phi)} + \sin(\phi) \right) \quad (3.3)$$

$$h = 2R \quad (3.4)$$
For the twin sphere, we find the following analysis:

\[
x_1 = x_2 = R \left( \frac{\cos(\phi) + 1}{\tan(\phi)} \right) \\
y_1 = y_2 = R(\cos(\phi) + 1) \\
w_1 = w_2 = R \left( \frac{\cos(\phi) + 1}{\tan(\phi)} + \sin(\phi) + 1 \right) \\
h_1 = h_2 = 2R \\
y_3 = R \left( \frac{\sin(\phi) + \cos(\phi) - 1}{\cos(\phi)} \right)
\]

### 3.2 Measurement results

The samples were put on the stage and the scanning parameters, like the force-setpoint, the z-gain and the z cutoff frequency were optimized in order to get an image of the spheres. Only the spheres on the plain glass plate (no greasy layer) gave good results. It was not possible to get a good image of the other samples with a greasy layer. The z piezo was extending all the time while scanning such a sample. This means that the sharp tip is pushed further and further into the sample surface. Or the quality of the obtained image was too bad for interpretation or even no image at all was obtained of the samples with a greasy layer. Even for the spheres on the plain glass plate, the tip is scanning the sample surface for a while before the spheres become visible in the image. It takes some time before the optimal scanning parameters are found.

Only the obtained image of the spheres on the plain glass plate are presented (see fig 3.3 and fig 3.4).
It must be said that the quality of the image, as displayed on the windows monitor (the AFM machine) is much better than the printed image. This is due to the too limited number of pixels of the printer. On the windows monitor the spheres can be identified much more easily than on the prints. In the image a "line-scan" is made that can be compared with the analysis of the former paragraph. This linescan fits the derived geometrical relations the best for $\phi = 55$ degrees.

Only spheres that lay down in the first layer of spheres on the glass plate can be analysed. Unfortunately it seemed not possible to extract the real orientation of stacked spheres so no analysis of these spheres (by far the majority of all the measured spheres) can be made. Actually it's a pin in a hay stack to find some (single or twin) spheres that are in the first layer of spheres on the glass plate.

Still, the quality of the obtained image is not wonderfull but let's compare it with the made geometrical analysis of the single and twin sphere. In the following table, the geometrical (expected) dimensions are listed as well as the measured dimensions of the single sphere in the first one and of the twin one secondly.
As we can see the height of the spheres is measured (60 nm) 22% too small. The width of the sphere is measured much better (error is 4%). Notice that the dimensions x & y are coupled; they are determined by the slope φ of the sidewall of the silicon tip. Let’s compare the results of the twin sphere with the made analysis.
<table>
<thead>
<tr>
<th></th>
<th>geometrical</th>
<th>measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>( x_1 )</td>
<td>0.14( \mu m )</td>
<td>0.08( \mu m )</td>
</tr>
<tr>
<td>( x_2 )</td>
<td>0.14( \mu m )</td>
<td>0.06( \mu m )</td>
</tr>
<tr>
<td>( y_1 )</td>
<td>0.20( \mu m )</td>
<td>0.13( \mu m )</td>
</tr>
<tr>
<td>( y_2 )</td>
<td>0.20( \mu m )</td>
<td>0.12( \mu m )</td>
</tr>
<tr>
<td>( w_1 )</td>
<td>0.38( \mu m )</td>
<td>0.40( \mu m )</td>
</tr>
<tr>
<td>( w_2 )</td>
<td>0.38( \mu m )</td>
<td>0.35( \mu m )</td>
</tr>
<tr>
<td>( h_1 )</td>
<td>0.27( \mu m )</td>
<td>0.22( \mu m )</td>
</tr>
<tr>
<td>( h_2 )</td>
<td>0.27( \mu m )</td>
<td>0.20( \mu m )</td>
</tr>
<tr>
<td>( y_3 )</td>
<td>0.09( \mu m )</td>
<td>0.03( \mu m )</td>
</tr>
</tbody>
</table>

Again, the height of the spheres is measured (60 nm) 22\% too small. Also the \( y_3 \) value of the twin sphere is measured far too small (33\%). Now we wonder how this could happen; that especially the vertical dimensions are measured too little unlike the horizontal dimensions, that are measured much better. Obviously the voltages that are applied to the piezo scanner indicate a lower height of the spheres than should be expected. This could be explained among others by deformation and/or scratching of the spheres by the tip. To investigate if this is occurring, we would like to approximate the interaction force between the tip and the sample surface.

### 3.3 Interaction forces between tip and sample

There are many different kinds of forces that interact between an AFM tip and a sample surface. Here a list of forces is given that might interact, dependant of the used materials for tip and sample surface [1]:

- van der Waals forces. This is usually an attractive force caused by a fluctuation in the dipole moment of one atom (or molecule) inducing a dipole moment in another atom which interact with each other. This is a long range force that is usually very weak, so it is often neglectable in comparison with other forces;

- capillary forces; an attractive force arising because of the surface tension of liquids. In atmospheric conditions there's always a water layer on the sample that pulls the tip to the sample surface. This force can be strong and erratic because of strong dependance of surface energy and film thickness.

- repulsive forces. This force is the result of the overlap potential between sample and tip atoms and change very strongly by change in distance.

- frictional forces that oppose the motion of the tip relative to the sample.

- adhesion; general name for the intermolecular forces which tend to cause tip and sample to stick together. This can lead to a chemical bounding when the surfaces are brought together under compression.
- Electrostatic forces also may occur. This can be either a repulsive or an attractive force due to the build up of charge on tip and sample. This is rare since any excess charge will generally bleed to ground.

- Magnetic forces might interact, dependant on the material properties.

As we can see, there are many forces that might interact between the tip and the sample surface. The z feedback loop of the AFM tries to keep the total sum of all these different forces constant while the tip is scanning a sample surface. With an experiment (see par. 3.3.2) with a very sensitive force balance, we want to find out what the order of magnitude of all those forces together is. This is done by measuring the change in force exerted by the tip on a surface by changes in force-setpoint of the z feedback loop. After that we are able to calculate the smallest step in force that can be made by a single change in force-setpoint and then we can estimate the interaction force between tip and sample surface.

Besides, the real tip-surface interaction is not of our concern for this experiment, because we are only able to measure what the change in force is. When you change the force-setpoint, the z piezo extends/retracts (by a applied change in voltage) so that the resistance of the piezo-resistive cantilever change a certain amount, which is proportional (at least theoretically) to a change in cantilever deflection and therefore to a change in force. With this experiment the tip is put on the rod of the force balance which is of another material than the polystyrene spheres. Therefore the real interaction forces between the tip and the surface are different but the change in force by a change in force-setpoint not. To measure this change in force, a very sensitive force balance is needed and is described in the following subsection.

### 3.3.1 Force balance

The force exerted by the tip is measured with a very sensitive force balance (see appendix E). A vertical rod is mounted by two triangular springs that are only flexible in the vertical direction and stiff in all other directions. Also on the rod, a condenser plate and a very strong magnet are mounted. When a force is applied to the rod it will move downwards. Then the distance between the condenser plate on the rod and one on the frame is increased, so the capacity is decreased (and this is measured with a HP 4284A lcr meter) and the magnet moves into a coil. When a force is applied to the rod, a PI controller adjust the current through the coil in such a way that the capacity of the condenser has reached its setpoint value again as before a force was applied. So there's are proportional relation between the applied force on the rod and the change in coil current. The resolution of the balance is estimated to be smaller than 80 nN.

Before we can put the force balance under the AFM tip and start measuring, it needs to be calibrated. This is done by putting calibrated masses on and off the balance and measure the change in coil current. Then we can calculate the calibration coefficient or balance sensitivity (mA/µN). Only three calibrated masses were available: 1, 2 and 5 mg and put on the balance ten times. Of every mass, two outliers are thrown out because
errors are likely to occur since the force balance is extremely sensitive. The next plot shows the results:

![Calibration plot of force balance](image)

Figure 3.5: calibration of force balance

The balance sensitivity is 0.013 mA/µN and the three calibration points fit very good a straight line. Now we can use the balance to measure the change in force exerted by the tip on a surface by a change in force-setpoint: this is a setpoint value that the z feedback loop of the AFM tries to keep constant. The bandwidth of the AFM controller is much higher then of the force balance so both controllers won't disturb each other cq control the same.
3.3.2 Approximation of interaction force

The force balance is put on the stage of the PSI AutoProbe XL AFM and the tip is placed above the rod. Then the tip is lowered (automatically) until it is in contact with the rod of the balance. The AFM machine then sets the force-setpoint value equal to zero. Now we're going to increase and decrease the value of the force-setpoint of the PSI AutoProbe XL AFM (by clicking a button on the windows monitor), so the tip will exert an increasing or decreasing force on the balance. The change in coil current and force-setpoint are noted and plotted against each other in the next plot. This is done for four different tips and done two times. Every measurement is repeated once after a new approach of the tip on the balance.

![Plot](image)

Figure 3.6: different tips on force balance

The smallest possible change in force-setpoint is 1.6 (an arbitrary number) and the range depends on many factors (like tip shape, sample surface, water layers on surface etc.) but is between the 200 and 400 units. Because the results are quite linear, we're able to calculate the (smallest) change in force by a single (1.6) change in force-setpoint with the following equation: \( \text{force step} = \frac{\text{range in measured current}}{\text{calibration coefficient} \times \text{number of steps in force-setpoint}} \)

The results are in the next diagram:
Capital a stands for increasing force-setpoint and b for decreasing. The number between brackets is the measurement number; measurement 2 is done after a new approach of the tip on the balance.

As we can see make some cantilevers (1&4) bigger steps in force then others (2&3). Also for most cantilevers (except 4) there’s a difference in force step size for increasing and decreasing force-setpoint that is even repeatable for tip 1. Actually the AFM machine uses many conversions of quantities. The step size in force for different tips is not only dependant on the spring constant of the cantilever (K [N/m]), that converts deflection into force, but also on the “Z error cal” (µm/V) value of the cantilever that converts detector (piezo resistive element on cantilever) voltage into cantilever deflection. These two quantities are multiplied by the AFM machine and therefore influence both the step size in force of each tip. The cantilever spring constant is between the 1 and 5 N/m (manufacturer) but the “Z error cal” value is unknown. This uncertainty of a factor 5 can be seen in the different force step size of each tip, but we may not conclude that this is due to the uncertainty in spring constant only.

By experience it is known that when you have to optimize the scanning parameters before getting an image, the force-setpoint is changed over many increments. You don’t change it one single step but many more times that exceed ten single steps very easily. This means that by changing and optimizing the force-setpoint, the interaction force between the tip and the sample surface is changed in the order of magnitude of a tenth or even a microNewton. Therefore the interaction force must be at least of this order of magnitude which is much bigger than expected.

### 3.4 Sample wear

As described in paragraph 3.2, the polystyrene spheres were measured for about 60 nm (22%) too small in height and we also know that the tip sample interaction force for the PSI AutoProbe XL AFM is probably quite big. Because the silicon tip is much harder than the PS spheres and very sharp (radius is smaller than 100 Angstroms but unknown) it is likely the the tip scratches the soft spheres. To find out if this is occuring, the next experiment is done: scan with a tip several times the same area on the sample surface with spheres, without changing one of the scanning parameters (in particular the force-setpoint of course). Then compare the peak-valley value of a specific, characteristic place in the obtained succesive images that is easy to identify.

---

1 there's no hardness measurement setup that is valid for both silicon and PS. Silicon has a mohs hardness of 7.0 and polystyrene a Rockwell M hardness between M65 and M85 [2].
In the next table a characteristic peak-valley value of the successive scans is listed. The scans can be found in appendix F.

<table>
<thead>
<tr>
<th>scan</th>
<th>height</th>
<th>width</th>
<th>Δ height</th>
</tr>
</thead>
<tbody>
<tr>
<td>scan1</td>
<td>1816Å</td>
<td>5428Å</td>
<td>-</td>
</tr>
<tr>
<td>scan2</td>
<td>1659Å</td>
<td>5465Å</td>
<td>157Å</td>
</tr>
<tr>
<td>scan3</td>
<td>1478Å</td>
<td>5460Å</td>
<td>181Å</td>
</tr>
<tr>
<td>scan4</td>
<td>1325Å</td>
<td>5391Å</td>
<td>153Å</td>
</tr>
<tr>
<td>scan5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

As we can see, the tip is really damaging the sample. Every scan the tip scratches some polystyrene from the spheres (for about 16 nm a scan). The sample wear is even that severe that the fifth scan can not be used because this image is too unstable (unsmooth, erratic line-scans) to make an interpretation useful. This sample wear however can not be compared with the former made measurements of the PS spheres because there are some uncertainties. We don't know if the interaction force, while measuring the spheres in paragraph 3.2 is the same as with this experiment (probably smaller). We also don't know how many times the tip had scanned the sample surface, before this (measurement) image was obtained or the optimal scanning parameters were found. Other uncertainties among others are the used tip radius, the differences between the PS spheres mutually (material properties) etc. etc.

This sample wear can not only explain partly why the height of the spheres was measured too small but also why it is so hard to obtain just an (good) image of the spheres. Especially with the last experiment; to get successive images without a single change in the scanning parameters. Striking is very likely to occur; there's something on the tip (dirt or PS) that is dragged over the surface and decrease the image quality quickly. Therefore the scanning parameters needs to be adjusted all the time what is quite time consuming.

If we make an analysis with the Hertzian contact deformation theory, we find that the order of magnitude of the deformation is for about the same as for the sample wear. If a load of 0,1 µN is applied we find a deformation of 3 nm and for a load of 1,0 µN 16 nm. However there are some weak points in this analysis. Uncertainties are the tip radius in particular, that is according to the manufacturer smaller than 100 angstroms (10 nm is used in the computation), the applied (constant?) load, the Young’s moduli and the Poisson’s ratios (especially of PS). Besides, this deformation theory is only valid for elastic deformation and can therefore never explain a height discrepancy of 22 % which should also contain plastic deformation. But because we know that the interaction force between the PSI AutoProbe XL tip and a sample surface is quite big (estimated range 0,1-1,0 µN) and since the tips are very sharp, it is likely to occur, especially when some soft materials (like PS for example) are involved.
Chapter 4

Conclusions

About the first part of this report the following conclusions can be drawn:

- to get a tip quickly on a desired position, the x- & y-offset between the two views need to be adjusted before doing a measurement sequence. There are three reasons mentioned why this is necessary. By storing the correct offset values in the computer, you will save time while doing a measurement sequence. However, for some applications (like scanning large homogenous areas for example) it is not necessary to do this.

- still, after adjustment of the offset values, the uncertainty in tip positioning is between the 5 and 15 microns. This uncertainty is caused by the positioning system of the stage, that has no feedback loop. Some possible reasons for this rather awkward performance are mentioned in paragraph 2.2.

The second and main part of this report is about measurements of calibrated polystyrene 0.3 micron spheres. The next conclusions can be stated:

- By an experiment with a very sensitive force balance, the interaction force between the PSI AutoProbe XL AFM tip and a sample surface is estimated to be in the order of magnitude of a tenth or even in the order of a microNewton. It seemed that by modifying the force setpoint of the z feedback loop, rather big steps in force are made. However, it is assumed that the (slow) lateral scanning motion of the tip did not disturb the force balance.

- Because of the fact that the interaction force between tip and sample surface is quite big (bigger than expected) and the tip is very sharp, the contact pressure between tip and sample surface may exceed an acceptable value. Therefore you have to be aware of wear and/or deformation when there’s a big difference in hardness/stiffness between the tip and sample. It seemed that the height of the soft PS spheres was measured much too small (22%). The tip was severely scratching the PS spheres and the sample was worn pretty fast. This sample wear also explains why it is so hard to
obtain just an image of the PS spheres and why you have to be careful in interpreting (especially quantitatively) the obtained image.

- On the other hand, when you want to scan a very hard surface like a diamond cutting tool for example, what now happened to the PS spheres will then happen to the sharp AFM tip. The tip will wear fast and the z piezo has to extend while it is scanning. The AFM machine “thinks” that the tip is going down while this is not the case and errors are introduced in the image. In this case there’s even one more thing that has to be taken into account: the tip will loose it’s well defined shape and therefore image interpreting becomes even more difficult. The area of the tip that interacts with the sample surface becomes bigger, the water layer on the sample will attract the tip differently (stronger), the tip/sample convolution becomes unknown etc. etc.

- Despite the fact that the height of the PS spheres was measured too small, we can not conclude that this is fully to due to deformation and/or sample wear (however this is still possible). But at least we do know that it occurs however we don’t know exactly how. It was not possible to quantify this because too many things are still unknown (like tip radius, number of scans before image was obtained, influence of water layer (thickness) on sample wear etc. etc.).
Appendix A

Standard Reference Material 1691
Nominal 0.3 Micrometer Polystyrene Spheres

The NBS Office of Standard Reference Materials announces the availability of a new SRM for use as a primary particle size standard. SRM 1691 is intended for use with particle size measuring instruments including electron microscopes. The SRM is a suspension of polystyrene spheres in water at a weight concentration of about 0.5%.

The average diameter (~0.27 μm) of the spheres was determined by transmission electron microscopy (TEM) using SRM 1690 (nominal one-μm polystyrene spheres) to set the dimensional scale.

The size distribution of the polystyrene spheres, as determined by TEM, is narrow with a standard deviation less than 2% excluding outliers (particles with diameters not on the main peak). The number of small outliers is less than 1% and the number of large outliers is less than 0.5%.

The certification of this SRM was performed with the support of the ASTM-NBS Research Associate Program.

This SRM may be purchased for $299.00 per 5 mL vial from:

Office of Standard Reference Materials
Room 2311, Chemistry Building
National Bureau of Standards
Geithersburg, MD 20899
Telephone: 301/921-2045
National Bureau of Standards

Certificate

Standard Reference Material 1691

Nominal 0.3 μm Diameter Polystyrene Spheres

(In Cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is intended for use as a primary particle-size reference standard for the calibration of particle size measuring instruments including electron microscopes. The SRM is a suspension of polystyrene spheres in water at a weight concentration of about 0.25%.

The number average particle diameter was determined by transmission electron microscopy (TEM) using SRM 1690 (nominal one-μm polystyrene spheres) to set the dimensional scale. The value reported is the mean of five independent data sets each consisting of over 100 measurements of 1-μm standard spheres and over 30 measurements of nominal 0.3-μm spheres.

<table>
<thead>
<tr>
<th>Number Average Diameter, μm</th>
<th>Uncertainty, μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.269</td>
<td>0.007</td>
</tr>
</tbody>
</table>

The uncertainty consists of both random and systematic errors, and includes sample-to-sample variability.

The value certified for the number average diameter was confirmed by one additional technique, centrifugal limit scattering (QELS). In this technique, the average lifetimes of the Brownian motion of the particles suspended in water is measured as a function of scattering angle. This gives a diffusion coefficient which can be used with the Stokes-Einstein relationship to yield the hydrodynamic particle diameter. The result from QELS was 0.276 ± 0.007 μm.

The size distribution of the polystyrene spheres, as determined by TEM, is narrow with a standard deviation less than 2%. Excluding outliers (particles with diameters not on the main peak), the number of small outliers is less than 1% and the number of large outliers is less than 0.5%.

The material is expected to have at least a four-year shelf life when stored at room temperature provided the cap on the vial is not removed. Caps should be exercised once each year before the material was packaged.

Before sampling, manually shake and/or expose SRM to ultrasonics until the spheres are uniformly distributed. Take a sample by squeezing a drop from the vial. Use filtered (0.1-μm pore size filter) distilled water for dilution. When electrolytes are used for electrical testing, use a countermeasure that dilutes the sample with water to prevent agglomeration.

The technical direction and physical measurements leading to certification were provided by T. Lister, O. Hembret, D. Gliatin, and E. Marx of the Mechanical Production Metrology Division.

The overall coordination of the measurements by the cooperating laboratories was performed under the direction of R. Oebink, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by L.J. Kieffer.

May 1, 1984
Washington, DC 20234

Stanley D. Reshberry, Chief
Office of Standard Reference Materials
Cooperative determinations were performed in the following laboratories:

Brookhaven Instruments Corp., Ronkonkoma, New York, D. Weiser
Eastman Kodak Co., Rochester, New York, D. E. DeCann
Malvern Instruments, Malvern, England, P. McNeil-Watson
G. D. Scarle and Co., Skokie, Illinois, M. Groves

The following results are given for information only:

<table>
<thead>
<tr>
<th>Method</th>
<th>Laboratory</th>
<th>Number Average Diameter (µm)</th>
<th>Standard Deviation of Distribution (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electron Microscopy</td>
<td>Kodak</td>
<td>0.273</td>
<td>0.0026</td>
</tr>
<tr>
<td>Light Scattering</td>
<td>Kodak</td>
<td>0.272</td>
<td>0.0023</td>
</tr>
<tr>
<td>Polarization Ratio</td>
<td>Kodak</td>
<td>0.273</td>
<td>0.0023</td>
</tr>
<tr>
<td>Quasielastic</td>
<td>Kodak</td>
<td>0.272</td>
<td>0.0023</td>
</tr>
<tr>
<td>Quasielastic</td>
<td>Brookhaven</td>
<td>0.273</td>
<td>0.0022</td>
</tr>
<tr>
<td>Quasielastic</td>
<td>Sheffield</td>
<td>0.273</td>
<td>0.0022</td>
</tr>
<tr>
<td>Quasielastic</td>
<td>Malvern</td>
<td>0.273</td>
<td>0.0027</td>
</tr>
<tr>
<td>Disc Centrifuge</td>
<td>Kodak</td>
<td>0.23</td>
<td>0.0027</td>
</tr>
<tr>
<td>Ultracentrifuge</td>
<td>Kodak</td>
<td>0.23</td>
<td>0.0027</td>
</tr>
</tbody>
</table>
Appendix B
Sample AFM and oblique view
X-Y Stage
On-axis objective
Oblique optics
AFM Scanner
On-axis viewing centre
AFM and oblique view centre
PIEZO LEVER - SPECIFICATIONS
PRELIMINARY TECHNICAL DATA

Dimensions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Piezolever 2 µm</th>
<th>Piezolever 4 µm</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (t)</td>
<td>Min  1.5</td>
<td>Typ  2.0</td>
<td>Max  3.0</td>
</tr>
<tr>
<td>Resistance</td>
<td>Min  1.5</td>
<td>Typ  2.0</td>
<td>Max  2.5</td>
</tr>
<tr>
<td>Spring constant</td>
<td>Min  1.0</td>
<td>Typ  2.5</td>
<td>Max  5.0</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>Min  0.2</td>
<td>Typ  0.3</td>
<td>Max  0.4</td>
</tr>
</tbody>
</table>

This information applies to a product under development. Its characteristics and specifications are subject to change without notice. Park Scientific Instruments assume no obligation regarding future delivery unless otherwise agreed to in writing.

Alignment is the same as for other cantilevers.
## Synoptic chart of cantilever products

<table>
<thead>
<tr>
<th>Microlevers</th>
<th>Sharpened Microlevers</th>
<th>Ultralevers</th>
<th>Piezolevers</th>
<th>MFM Cantilevers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicone nitride cantilever</td>
<td>Silicon nitride cantilever</td>
<td>0.6 µm Silicon nitride cantilever</td>
<td>2 µm Piezoresistive cantilever</td>
<td>4 µm Piezoresistive cantilever</td>
</tr>
<tr>
<td>Silicon nitride tip</td>
<td>Silicon nitride tip</td>
<td>Silicon tip</td>
<td>Silicon tip</td>
<td>Silicon tip</td>
</tr>
<tr>
<td>Radius = 500 Å</td>
<td>Radius = 100 Å</td>
<td>Radius = 100 Å</td>
<td>Radius = 100 Å</td>
<td>Radius = 100 Å</td>
</tr>
<tr>
<td>General purpose Contact</td>
<td>High resolution Contact</td>
<td>High resolution Contact</td>
<td>Integrated detector</td>
<td>Non-contact</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Full Wafer (600 chips)</th>
<th>FWML-06AU $2,000*</th>
<th>FWMS-06AU $2,000*</th>
<th>IJWU-06AU $2,500*</th>
<th>STML-06MF $500</th>
</tr>
</thead>
<tbody>
<tr>
<td>Half Wafer (300 chips)</td>
<td>HWML-06AU $1,200*</td>
<td>HWMS-06AU $1,200*</td>
<td>STUL-06AU $400</td>
<td>STUL-20AU† $500</td>
</tr>
<tr>
<td>Strip (15 chips)</td>
<td>STMI-06AU $300</td>
<td>STMS-06AU $300</td>
<td>STUL-06AU $400</td>
<td>STML-06MF $500</td>
</tr>
<tr>
<td>Mounted (50 cartridges)</td>
<td>APML-06AU $1,000</td>
<td>APMS-06AU $1,000</td>
<td>APUL-06AU $1,500</td>
<td>APUL-20AU† $2,000*</td>
</tr>
<tr>
<td>Mounted (15 cartridges)</td>
<td>APML-06MF $600</td>
<td>APML-06MF $600</td>
<td>APML-40MF $800</td>
<td>APML-40MF $800</td>
</tr>
</tbody>
</table>

* Not to be discounted
† For owners of a PSI instrument only
‡ For owners of PSI Autoprobe XL only
§ For owners of PSI Autoprobe VP only
Appendix C

**X-offset between A.F.M. tip and microscope in microns**

![Graph of X-offset between A.F.M. tip and microscope in microns]

**Y-offset between A.F.M. tip and microscope in microns (+37mm)**

![Graph of Y-offset between A.F.M. tip and microscope in microns]

**Normal Probability Plot**

- **X-offset**
  - Number of measurement sequence: 1 to 8
  - Data range: 640 to 680
  - Sigma: 6.4

- **Y-offset**
  - Number of measurement sequence: 1 to 8
  - Data range: -440 to -340
  - Sigma: 7.0

36
Appendix D

The top angle of a cross section through the middle of a pyramid is a function of the orientation of this pyramid. For a section over a diagonal, the top angle is bigger than for a section perpendicular to the pyramids base. Let’s define an angle $\theta$ for the rotation of a cross section relative to the pyramid and the top angle $\alpha$ therefore is a function of this rotation. The value of angle $\phi$ is 55 degrees in the cross section AA which is given by the manufacturer. N.b. the measured height of the spheres is independent of $R$ and $\phi$.

![Silicon Tip Cross Section AA](image)

Figure 5.1: pyramid with cross section

$$
0 \leq \theta \leq 45 \quad \phi = \arctan(\tan(55) \cdot \cos(\theta)) \quad (5.1)
$$

$$
\alpha = 180 - 2 \cdot \phi \quad (5.2)
$$

The next plot shows top angle ($\alpha$) versus rotation ($\theta$)

![Top Angle as a Function of Pyramid Rotation](image)

Figure 5.2: $\alpha$ as a function of $\theta$
Appendix E
ELECTRICAL TECHNIQUES

Frequency = 17 kHz (approx.)
Actuator is a solenoid with a permanent magnet core.
Bridge used is a Transformer Ratio Bridge.
C (ref) = 40 pF
Appendix F
Oct 31 95 a18
0.3 mu PS spheres
scan 2

0.3
0.2
0.1
0.0

Height Profile

Power Spectrum

Histogram

Uncaring Ratio

Trace Statistics:
Height: 1659 Å
Spacing: 565 Å
Angle: 16.9°
Oct 31 95 s19
0.3 μm PS spheres
scan 3

Histogram

Height Profile

Power Spectrum

Trace Statistics:
Height: 1478 Å
Spacing: 5460 Å
Angle: 15.1°
Oct 31 95 s20
0.3 μm PS spheres
scan 4

Height Profile

Power Spectrum

Histogram

Bearing Ratio

Trace Statistics:
Height: 1325 Å
Spacing: 5391 Å
Angle: 13.8°
Oct 31 95 s21
0.3 µm PS spheres
scan 5

Distance: 4235 Å
Spatial period: 0.00 Å

Height: 1346 Å
Spatial frequency (1/p): 0.41 Å
Spatial period: 63 Å

Trace Statistics:
Height: 1346 Å
Spacing: 4235 Å
Angle: 17.6°
Bibliography
