Precision Attachment of (Silica) Spheres to AFM Cantilever Tips

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Over the past decade the Atomic Force Microscope has been utilised in a myriad of applications across many fields of research and industry. One area that has been of particular interest is the measurement of small scale forces. The AFM is capable of measuring electrostatic and van der Waals forces with high precision based on small deflections of the AFM cantilever.

The forces between colloidal particles dominate the behaviour of a great variety of materials. The AFM has been extensively used to measure the forces between planar surfaces and individual colloid particles. One of the key driving forces for such experiments and the corresponding theoretical framework that has ensued is the ease of use of standard AFM tips for such measurements.

The original force measurements performed with the AFM utilised the standard tip on the end of the cantilever as the point of interaction. The problem with this approach is that the pyramidal shaped tip is extremely problematic to model due to its geometry and irregularities. A number of solutions have been proposed to circumvent this problem, all having limited success.[1-3]

Hutter and Bechhoefer determined an effective radius curvature of an AFM tip.[1] Drummond and Senden[2] proposed a method that involves measuring the forces between a silicon nitride tip of unknown curvature and a 'standard' surface bearing an adsorbed surfactant. Arai and Fujihira[3] have suggested that modelling the tip apex as a spherical surface is only appropriate for tip cone angles greater than 30 degrees.

These indirect approaches introduce added degrees of error into the experiment, particularly since the Derjaguin approximation[4], which is the standard mathematical method for interpreting force-distance data, does not hold for small radii surfaces.

The solution to this problem was first presented by Ducker *et al* [5] and Butt[6], who attached individual colloidal spheres on to the end of AFM cantilevers to create probes of precisely defined shape and dimension. A good description for preparation is given in Preuss and Butt[7]. The difficulties with sticking on spheres are that you often get more than one on the cantilever at a time. They may not be placed on the edge of the cantilever or they may even be behind the pyramid or on the other side of the cantilever. In the next section we propose a new technique for the precision placement of spheres on tips using the Dimension™ 3100 AFM.

Experimental - Sticking spheres onto both cantilever tips using the Dimension™ 3100 AFM.

Here we propose a method analogous to that of Ducker *et al* [5] and Butt[6], but using the Dimension™ 3100 AFM.

STEP 1

Initially a small amount (~10^{-15} L) of glue is placed on the cantilever using a three-dimensional microtranslation stage. (This could be also completed with the Dimension™ 3100 AFM.)

Once the cantilever has a small amount of glue on the end of the two tips of the cantilever, it is placed into the Dimension™ 3100 AFM cantilever holder and set up in the AFM as for normal contact imaging.

STEP 2

A 50 micron thick cover slip is attached to a microscope slide with sticky tape (see Figure 1). The tape must be carefully positioned on three sides only as shown.

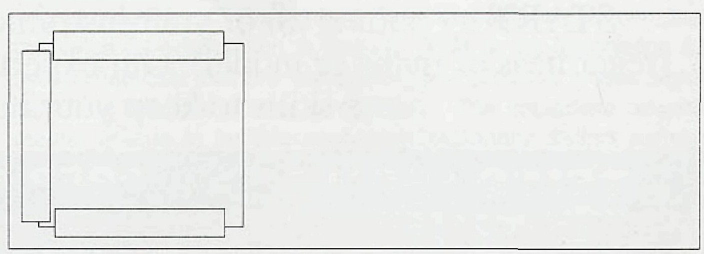

Figure 1

A small amount of silica is placed on the slide and spread out using a drop of ethanol. This is then left to dry - *i.e.*, can be used once the ethanol has been evaporated.

STEP 3

The slide is then placed on the AFM stage and positioned under the head. The system is lined up with the shorter of the two cantilevers over the cover slip, while the longer cantilever is over the glass slide (see Figure 2). This step is crucial as the longer cantilever always makes contact first and in order to place a sphere on the short cantilever there needs to be an appreciable sample height change. The cover slip placement means that the sample is 50 microns higher under the short cantilever than under the long cantilever.

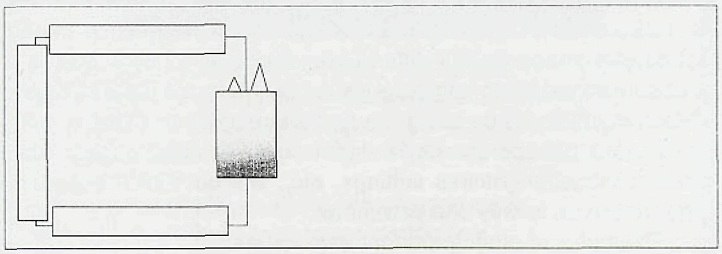

Figure 2

Now the laser is positioned on the short cantilever and the glass cover slip is brought into focus. At this point the 1-5 micron spheres should be readily visible optically.

STEP 4

Focus on the tip and place the end of the tip in the middle of the cross hairs (see Figure 3). This needs to be performed at the highest magnification so that the precise position of the tip is recorded.

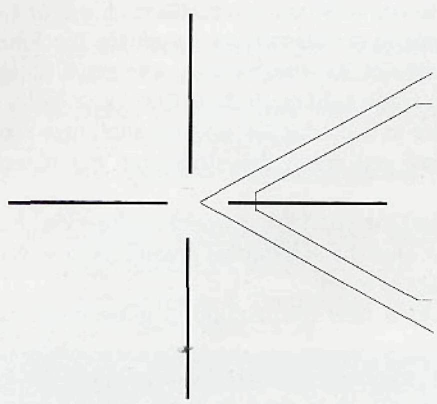

Figure 3:

STEP 5

The system is then returned to "focus surface" mode and the sample is focused and moved until a sphere is sitting directly within the cross hairs (see Figure 4).

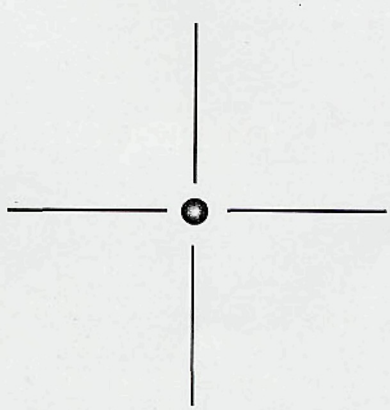

Figure 4

Set the Scan Parameters to be:
Dimension parameters:
Contact mode
Scan size = 0
X-offset = 0
Y-offset = 0
Vertical Deflection – setpoint value ~ 1.5 to 2 Volts

It is important to keep the scan size to zero as this prevents the sphere from being moved around. One of the consequences of moving the sphere around is that glue may be deposited on the interaction side of the sphere and this is highly undesirable.

STEP 6

The final stage is to engage the AFM in order to pick up the sphere. Once engaged it is recommended that the system be left to sit for some 20-30 seconds, after which time the retract button can be pushed to pull the cantilever away from the surface. At this point the system will still be focused on the surface, and as the cantilever moves upwards the position of the sphere will again be visible. If the sphere has been removed from the surface by the cantilever the surface will appear clean.

If by chance the sphere remains in its previous location, the system should be engaged again with a slightly greater force. Alternatively, the head can be manually stepped down by a few microns. If there is still no sphere on the tip, it is likely that not enough glue has been applied, in which case step 1 needs to be repeated.)

STEP 7

If spheres are required on both tips, this can be easily achieved at this point. Firstly, the head needs to be raised a good 100 microns above the surface. Once this has been done the long cantilever needs to be set up with the laser for imaging. The cover slip surface also needs to be underneath the long cantilever, thus the system needs to be laterally positioned. Refocus the cantilever and surface and repeat steps 4 – 6 in order to place a sphere on the second cantilever.

The entire AFM process should take less than 10 minutes. In addition the cantilevers will contain:

1. Perfectly positioned spheres
2. One sphere per tip
3. Spheres from 1 micron diameter upwards
4. No glue residue on the functional side of the sphere

Further details regarding the application of glue using the Dimension 3100 AFT can be acquired from <shaneth@unimelb.edu.au>. ■

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3. Arai, T. and M. Fujihira, *Effect of Tip Shape On Force Distance Curves For Afm in Aqueous Electrolytes*. Journal of Electroanalytical Chemistry, 1994. **374**(1-2): p. 269-273.
4. Derjaguin, B.V., Kolloid Zeitschrift, 1934. **69**: p. 155.
5. Ducker, W.A., T.J. Senden, and R.M. Pashley, *Direct measurement of colloidal forces using an atomic force microscope*. Nature, 1991. **353**: p. 239-41.
6. Butt, H.-J., *Measuring electrostatic, van der Waals, and hydration forces in electrolyte solutions with an atomic force microscope*. Biophysics, 1991. **60**: p. 1438.
7. Preuss, M. and H.-J. Butt, *Direct Measurement of Particle-Bubble Interactions in Aqueous Electrolyte : Dependence on Surfactant*. Langmuir, 1998. **14**(12): p. 3164-3174.