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Technical note

A NOVEL METHOD TO PREPARE PROBES FOR ATOMIC FORCE SPECTROSCOPY

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Colloidal probe microscopy is one of the most commonly used techniques to measure interaction forces between microscopic bodies. Colloidal probes are attached to the free end of AFM cantilevers. Usually they have a diameter that ranges from a few microns up to a hundred microns. Here we describe a technique, based on material evaporation, that allows to control continuously the size of the AFM probe from few tens of nanometers to several hundreds of nanometers. This fills the gap between the unmodified commercial AFM probes and the standard colloidal probes.

Keywords: Atomic Force Microscopy, Colloidal Probes, Force Spectroscopy

1. Introduction

Since 1984, when the AFM was first introduced, its use has grown in many areas from biology to polymer science, from microelectronic industry to colloid science [1, 2, 3, 4, 5], surface characterization of coatings [6] and many others.

The AFM is used not only to measure the surface topography of the sample, but also to study the separation dependence of the force acting between the AFM probe and the sample surface (force spectroscopy). In the Force Spectroscopy Mode (FSM) the AFM scanner holds the x-y sample position while the vertical (z direction) is ramped periodically, modifying the separation between the probe and the sample surface.

This mode of operation of AFM has become a powerful technique due to the extreme versatility of AFM; by choosing the proper cantilever elastic constant, FSM can measure force between 1pN to 0.1 mN. On the other hand the fast response time of the piezo-tube allows to explore both the dynamic and static regime of many systems.

Another important aspect of FSM is the possibility of modifying the interacting probe. For a standard cantilever the probe is a nanometer sized tip, but the size and the properties of the interacting probe can be varied by attaching a colloidal particle on the cantilever.

Practically, the radius R of the colloidal particle can be chosen to be between 1 and 100 micrometers.

But the actual value of R can not be continuously varied and the reproducibility of the experimental condition can sometimes be poor. First of all, because the local radius of curvature of the colloidal particle can vary from point to point for the same particle and secondly, because the procedure involved in cleaning the probe surface is in general complicated, and the result obtained on different particles can be slightly different.

Here we present a method to continuously control the radius of curvature of the AFM probe by evaporating a thin film of dielectric material onto the standard tip of a commercial cantilever. In the interaction zone, the obtained probe can be approximated with a sphere. The radius of curvature is controlled by the amount of material evaporated and can be as big as few hundreds of nanometer.

In this note we first report some test measurements performed by the usual colloidal probe FSM and then we introduce our new method to obtain sub-micrometer sized probes for FSM.

2. Experimental results

As a test we studied the force acting between the probe of an AFM (AutoProbe CP, Veeco Instruments, CA) and a silica substrate in a ionic solution. Interaction forces were measured using the AFM in different solutions of $NaCl_{(aq)}$, in particular at concentrations 10^{-4}M, 10^{-3}M, 10^{-2}M, 10^{-1}M. Colloidal spheres of silica were attached at the free end of triangular cantilevers of nominal spring constant 30 mN/m (Veeco Instruments, CA).
In order to attach the silica sphere to the free end of the cantilever, the latter was put under a stereomicroscope. With the help of a micromanipulator the free end of the cantilever was put in contact with a small drop of glue and immediately after with the sphere that has to be attached. Due to the capillarity the sphere is picked up and, once the glue dries, it is rigidly attached to the cantilever. To verify the correct position of the sphere on the cantilever, the resolution of an optical microscope is sufficient, but in order to resolve the surface smoothness of the colloidal particle a Scanning Electron Microscope (SEM) is necessary (Fig.1a,b).

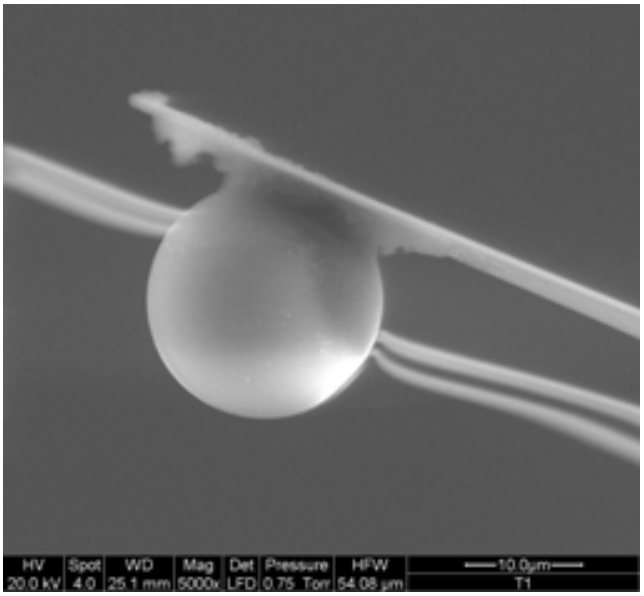


Fig. 1a. SEM pictures of attached spheres with a 20 μm diameter.

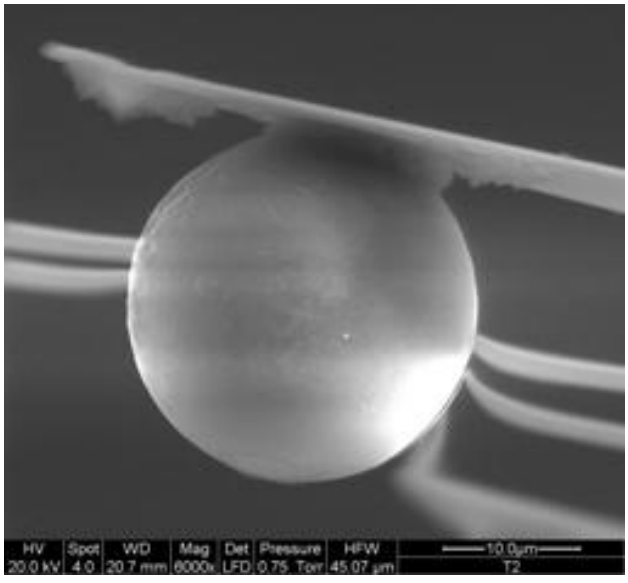


Fig. 1b. SEM pictures of attached spheres with a 25 μm diameter.

The substrate used to measure interaction forces were pieces of silicon wafers covered with a small layer of native oxide.

Using these spheres, two sets of force vs. distance curves were obtained (Fig. 2). The first set of curves (Fig. 2a) was measured using the 20 μm diameter sphere (A) with an acquisition time of 10s. The second one (Fig. 2b) was obtained using the 25 μm diameter (B) sphere, with an acquisition time of 1s. The short acquisition time has contributed to decrease the noise due to external factors (mainly mechanical noise).

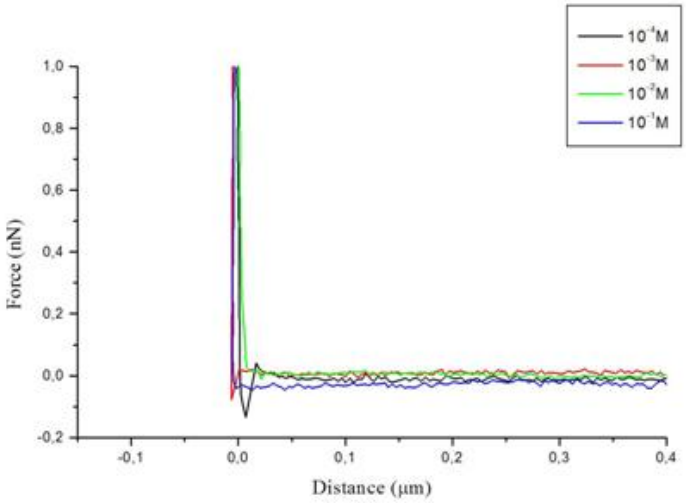


Fig. 2a. Force vs. distance curves obtained with sphere A.

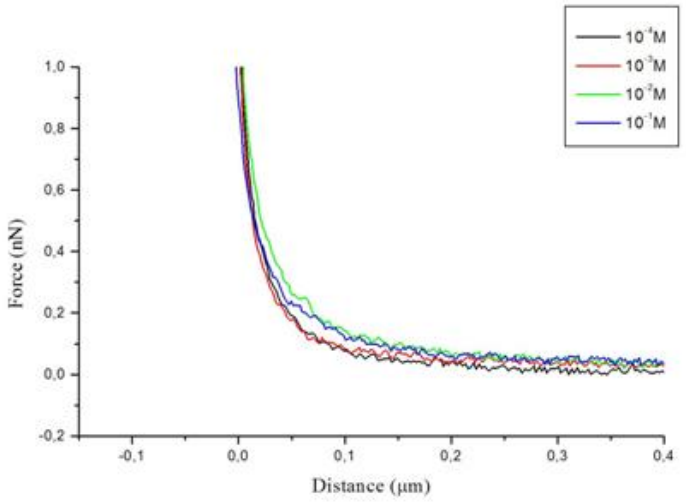


Fig. 2b. Force vs. distance curves obtained with sphere B.

From the first set, a sharp tip-like behaviour is evident, particularly for lower electrolyte concentrations, whereas for higher concentrations van der Waals forces almost disappear to electrostatic repulsive forces advantage. In the second set of curves, van der Waals attraction forces completely disappear to repulsive forces advantage. Particularly electrostatic forces become stronger at higher concentrations, as we have already noticed in the first set of curves. Electrostatic forces become appreciable at a distance of 0,2 μm. As it can be noticed, the two sets of curves are quite different. This could not be attributed to the little difference in the spheres dimensions. We think our problems of non reproducibility are mainly related with the spheres gluing and with a not correct procedure of cleaning the probes.

The technique used to glue the spheres was not the suitable one for our experiments, in this procedure the part of the spheres that will interact with sample is the one that stays attached on the glass slide, so probably the most dirty one. Moreover sometimes spheres that look perfect under the stereomicroscope have a strange shape (Fig.3).

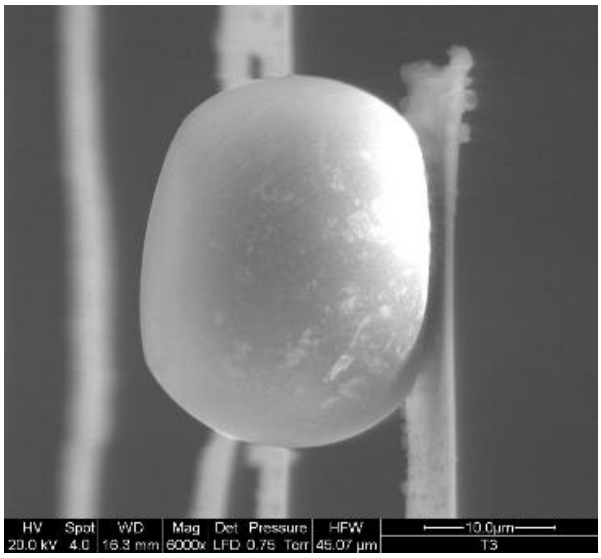
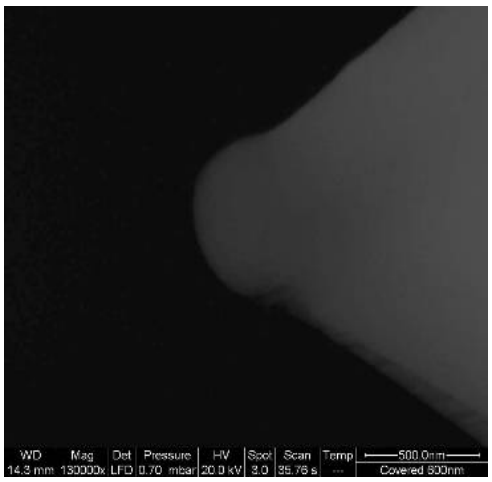


Fig. 3. A deformed sphere.

For our future investigations we also would like to use spheres of about 1-2 µm in diameter to analyse forces between a plane and a smaller spherical surface.

Here we propose an alternative solution to obtain a curved surface of hundreds of nanometers, based on the idea to cover a commercial tip with a thin layer of dielectric material. The process of evaporation enables to obtain a spherical like surface which covers the pyramidal tip providing at the same time cleanness since the process is performed in vacuum (about 10^{-6}mbar). Therefore 600nm of SiO_x have been deposited, through physical vapour deposition, on a pyramidal tip at the end of a triangular cantilever in order to obtain an approximately spherical surface, the outcome is shown in Fig.4. The evaporation rate was slow in order to get a layer as compact as possible. The samples were placed at about 30 cm away from the evaporation source.



a

b

Fig. 4a,b. Commercial AFM tip with a 600 nm of evaporated silicon oxide.

A curvature radius of about 335 nm was obtained and the interaction surface can be approximated to that of a sphere with a 670 nm diameter. This is an encouraging result, but indeed the process of evaporation should be further investigated to get the evaporation parameters suitable for everyone experimental needs. The main advantage is that this process allows to control accurately the thickness of the evaporated material. But it is also necessary to notice that a new measure of the spring constant of the cantilever should be done, because the thickness of the evaporated material is comparable with the cantilever one, usually units of microns. Finally, once the evaporation is performed it is necessary to check every time the tip shape through electron microscopy. Nevertheless this technique appears very promising in order to get clean spherical surfaces.

3. Conclusions

In this note we have described the possibility to create probes suitable for force spectroscopy measurements using physical vapour deposition techniques. Probes are prepared starting from commercial ones. The process is performed in vacuum and allows to get tips with a radius of curvature of hundreds of nanometres. The tip shape has been investigated through Scanning Electron Microscopy. Further studies have to be performed to investigate the influence of the evaporation parameters on the tip shape, on the compactness of the deposited layers, and finally on the variation of the elastic constant of the cantilevers.

Acknowledgements

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