EFFECT OF COLLOIDAL PROBE RANDOM SURFACE FEATURES ON ADHESION

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ABSTRACT
We have uncovered the fact that colloidal probes often have random surface features which when not properly accounted for, can significantly affect the magnitudes of the measured adhesive forces using atomic force microscopes (AFM). Colloidal probes have been used to measure the pull-off forces between the probe and surfaces. We prepared a series of colloidal probes by attaching glass spheres (radii of 3.3 µm to 17.4 µm) to the end of AFM cantilevers. Adhesive force between the probes and a silicon wafer surface was measured using an AFM under various loads from 6 nN to 100 nN in dry air. Results showed that the values of the pull-off forces did not correlate with the radii of the probes. Direct imaging of the glass sphere surface using a sharp tip revealed substantial random surface features which altered the size of real contact areas. We extracted the load-bearing areas from the topography data and used them to normalize the adhesion data. The measured adhesive force, after being normalized, was found to be independent of the load and the sphere radii, in agreement with prevailing contact mechanics theory.

INTRODUCTION
Atomic Force Microscope (AFM) equipped with sharp probe tips is a powerful tool to measure forces at nanoscale. However the high contact pressures of the sharp tips and the lack of definitive tip shape and orientation often cause measurement artifacts. Colloidal probes, made of spheres attached to AFM cantilevers, have been developed to measure adhesion between the probe and various surfaces [1-2]. The spherical tip shape makes the data amendable to various contact mechanics models such as JKR [3] and DMT [4] for adhesive force measurements. For smooth spheres, the real area of contact is proportional to the radii, therefore, the adhesive force measured by the pull-off force between such a probe tip and a flat surface should be proportional to the radii.

The tip radius of a colloidal probe is normally measured by scanning an AFM grating samples using the colloidal probe [5]. This method however would not provide detailed surface features at the nanometer scale.

In this paper, we report our study on adhesion force between glass spheres of micrometer radii and a flat silicon surface using the colloidal probe method. Each of the glass spheres used in the adhesion measurements were imaged directly using AFM at the location where it touched the silicon surface. The AFM images provide not only a direct measurement of the tip radii but also detailed information on the surface roughness and unusual surface features. Bearing areas [6] were extracted from the measured topographic images and used to analyze the adhesion force data.

EXPERIMENTAL DETAILS AND RESULTS
Colloidal probes were prepared by gluing glass spheres of various radii from 3.3 µm and 17.4 µm onto the end of AFM cantilevers. Adhesion force between the colloidal probes and flat silicon wafer surface, placed in dry air (RH < 3%) environment, was measured using an AFM system (Veeco, Multimode NanoScope IIIa). The sensitivity of the AFM instrument, i.e., photodiode voltage vs. piezoelectric scanner displacement, and the spring constants of the cantilevers in the Z direction were calibrated. After the adhesion measurement, the spheres were imaged directly by the AFM using a two-cantilever method. Figure 1 presents the topographic images of the nine spheres used in our experiments. They show random nanometer scale surface features. The surface of silicon wafer used in the adhesion measurement was found to be much smoother (Ra=0.3 nm).

The adhesion force is defined here as the pull-off force, which was measured by operating the AFM in the force-displacement mode. The loading force varied from 6 nN to 100 nN. Value of adhesion force measured for each colloidal probe does not depend on the load, consistent with the JKR [3] and DMT [4] theories.
The measured adhesion force is plotted as a function of radius of sphere on colloidal probe in Figure 2. If the sphere surface were smooth, the adhesive force would be proportional to the sphere radius according to JKR [3] and DMT [4] models. The data presented in Figure 2 exhibit very large scatters and no clear conclusion can be drawn about the relationship between adhesion and the radius of the spheres.

In order to properly account for the surface features present on the surfaces of the spheres, the measured adhesion force was plotted as a function of roughness parameters that include \( R_a \) (average roughness), \( R_{rms} \) (root mean square roughness) and \( R_{max} \) (peak to valley roughness). Results suggested that these normal roughness parameters were inadequate to compensate for these random surface features.

To fully account for these random surface features, we need to know the material properties of these bumps and then use the full contact mechanics model to estimate the real area of contact of each spheres. Such measurements are not feasible using current instrumentation. A quick and simple way is needed. We decided to try to use the bearing area concept [6] to see whether this would sufficiently account for the observed features. We suspect that these features are caused by SiO\(_2\) particles stuck on the surface so we used the material properties of glass for this purpose. The bearing area for each sphere was computed from the topographic data measured using AFM. A plot of the bearing area as a function of sphere radius (not shown) assembles closely the plot of adhesion force versus radius. Figure 3 shows the adhesion force, with each data point normalized to the corresponding bearing area, as a function of sphere radius. Within the experimental uncertainties, the figure shows a constant adhesive force as a function of sphere radius. This result suggests that the bearing area is a viable method to account for these random surface features which are often present in colloidal probe studies. We also show that detailed features of the colloidal probes can be obtained by directly scanning the colloidal probes using another sharp tip.

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REFERENCES