

Abstract

Adhesion is important to the yield and performance of MEMS (Micro-Electro-Mechanical Systems) and NEMS (Nano-Electro-Mechanical Systems). Measuring the work of adhesion, with an AFM (Atomic-Force Microscope), allows one to study surface interactions from an energy perspective as opposed to an adhesion (force) perspective. The works of adhesion were measured with different AFM tip radii on multiple types of samples. Two sample variables were examined: four die attach conditions (no attachment, silicone, polyimide silicone, and silver glass), and two surface conditions (native oxide with and without a few angstroms of vapor-deposited diphenylsiloxane). For a normal silicon cantilever, the work of adhesion seems to be slightly less for the treated surfaces than the untreated, except for the silver-glass die attach material. There were at least three orders of magnitude difference in the works of adhesion for the different AFM tip radii. Presumably, the trend is due to some combination of torque on the AFM cantilever, material properties, and interfacial roughness.

Cantilever Stiffness Calibration

An oscillator's amplitude distribution is routinely determined by recording its deflection in time and Fourier transforming the time domain data, yielding the mean-squared amplitude. The mean-squared amplitude is related to the thermal energy of the oscillator by the equipartition theorem.

$$\frac{1}{2}k_b T = \frac{1}{2}k \langle x^2 \rangle, \quad (1)$$

where k_b is Boltzmann's constant, k the spring constant of the oscillator, and $\langle x^2 \rangle$ the mean-squared amplitude of the oscillator. The spring constant can be calculated as follows [1]:

$$k = \frac{Q}{\pi} \frac{k_p T}{(x^2(v_k)) v_k}, \quad (2)$$

where Q is the quality factor, Δv the frequency resolution, and v_k is the frequency at kinetic resonance.

Tip Calibration (Sphere)

The radius of a sphere attached to a cantilever can be determined by minimizing the square root of the square error of the radius [2]. Defined as

$$R = \sqrt{(z-h)^2 + (x-k)^2}, \quad (3)$$

where R is the radius of the sphere, z is the sample height, x is a point along the fast scan direction, and h, k define the center of the circle. The square root of the square error (standard deviation) is

$$\delta R = \sqrt{\frac{N \sum R_i^2 - (\sum R_i)^2}{N(N-1)}} = 0. \quad (4)$$

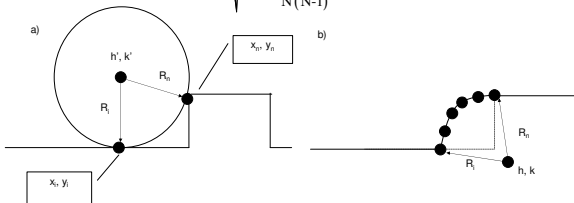


Figure 2. a) Schematic cross-sectional view of a sphere and step function. Since the topography image from an AFM is a dilation between the spherical tip and sample, the resulting cross-sectional data can be fit to a circle, where matching subscripts on $x, y,$ and R refer to a single point of the cross-sectional data. b) The expected cross-sectional data, after averaging all scan lines of the topography image, and their relation to the radius and center of a circle. Each data point of the circular-arc segment was fit to the equation for a circle. The fit parameters h and k , representing the center of the circle, were found by minimizing the standard deviation of the radius for all points in the data range that had arclike behavior. The corresponding average radius value was then the best-fit estimate for the radius of the sphere attached to the cantilever.

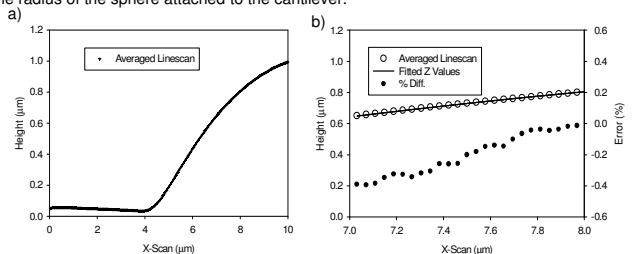


Figure 3. a) A typical graph of a 48 μm sphere with all line scans averaged from a 256 by 256 pixel topography image that was 10 by 10 μm^2 . b) The scale on the right side of the graph corresponds to the percent difference between the fit and the data. The best fit was chosen so that the percent difference (between the fit and data) was in the range of $\pm 5\%$. The scale on the left side of the graph represents a magnified portion of the total graph from part a, which illustrates that it is difficult to distinguish between the fitted data and the actual data.

AFM ($\mu\text{m}; \pm 9\%$)	Optical Micro. ($\mu\text{m}; \pm 5\%$)
1.6	NA
21.9	20.5
30.1	27.5
37.8	34.0
51.2	47.5
60.3	53.0

Table 1. The results compared for the diameter of an attached sphere obtained by fitting data from the AFM to a circle and by optical inspection using a Nikon Measure Scope MM-11™. The uncertainty corresponding to the AFM data was $\pm 9\%$, and to the optical microscope it was $\pm 5\%$. The measurement performed with the AFM can be done quickly and for a wide range of sphere diameters, including those not measurable with an optical microscope.

Work of adhesion

The work of adhesion is proportional to the pull-off force between an AFM cantilever tip and a sample surface. Stiction or static friction is the pull-off force and the work of adhesion can be calculated from the Derjaguin approximation for the case of a sphere-flat interface.

$$\omega = \frac{-F_{\text{pull-off}}}{2\pi R_{\text{tip}}}. \quad (5)$$

Using the stiffness and radius calibrations for a cantilever, the work of adhesion for the tip-sample interface can be calculated to within 15%.

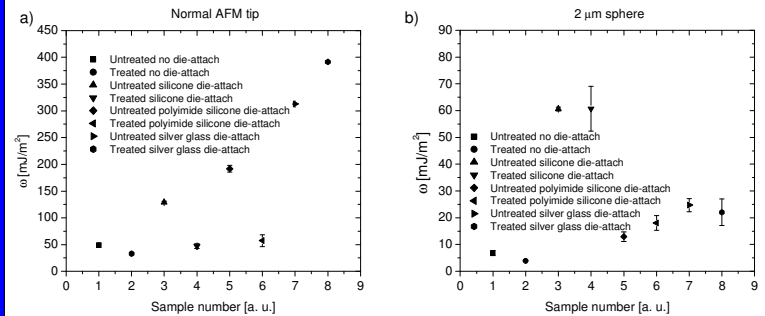


Figure 4. a) Values for the work of adhesion between a normal AFM tip and unpatterned silicon die. b) Values for the work of adhesion between a two-micron diameter bead (SiO_2) AFM tip and unpatterned silicon die. The treated surfaces are silicon oxide substrates coated with a few angstroms of diphenylsiloxane. The untreated surfaces are silicon oxide substrates.

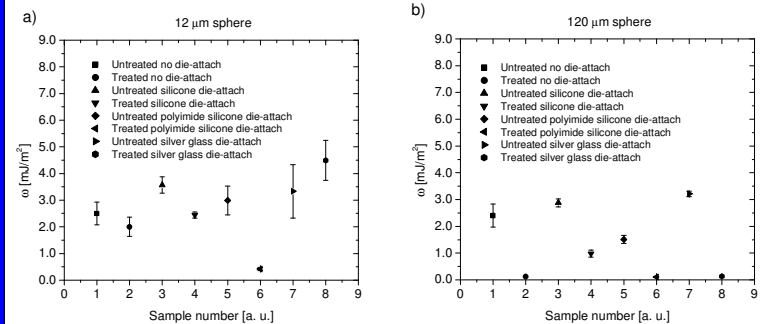


Figure 5. a) Values for the work of adhesion between a twelve-micron diameter bead AFM tip and unpatterned silicon die. b) Values for the work of adhesion between a 120 micron diameter bead (SiO_2) and unpatterned silicon die. The treated surfaces are silicon oxide substrates coated with a few angstroms of diphenylsiloxane. The untreated surfaces are silicon oxide substrates.

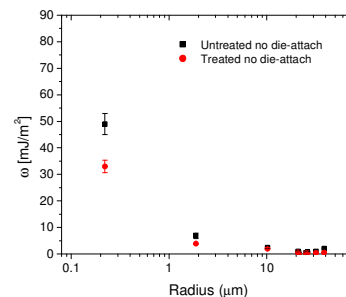


Figure 6. Values for the work of adhesion between different tip radii and untreated and treated no die-attach. The treated surfaces are silicon oxide substrates coated with a few angstroms of diphenylsiloxane. The untreated surfaces are silicon oxide substrates.

What are the possible causes for the lowered works of adhesion for increasing radius?

- Torque: Linear in $R!$
- Material properties: Increases scatter in the data.
- Roughness: Higher roughnesses seem to show lower works of adhesion. Rougher surface causes more asperity contacts, increases tip-sample separation, causes smaller attraction between tip and sample?
- Temperature, Humidity, Physisorbed Molecules, Chemisorbed Molecules?

References

[1] N.A. Burnham et al. *Nanotechnology* **14**, 1 (2003).
 [2] E. J. Thoreson and N. A. Burnham, *Review of Scientific Instruments* **75** (5), 1359-1362 (2004).