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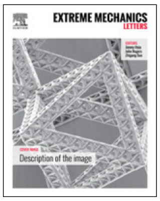

# Measurement of the strength and range of adhesion using atomic force microscopy

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ABSTRACT

Adhesion of nanoscale contacts is important in many applications, including microelectromechanical systems, fibrillar adhesives, and atomic force microscopy (AFM). Here, we quantify the properties of the adhesive traction–separation relation between ultrananocrystalline diamond (UNCD) AFM tips and polymethyl methacrylate (PMMA) surfaces using a novel AFM-based method that combines pull-off force measurements and characterization of the 3D geometry of the AFM tip. Three AFM tips with different nanoscale geometries were characterized and used to perform pull-off force measurements. Using the pull-off force data, the measured 3D tip geometries, and an assumed form of the traction–separation relation, specifically the Dugdale or 3-9 Lennard-Jones relations, the range, strength, and work of adhesion of the UNCD–PMMA contact were determined. The assumptions in the analyses were validated via finite element modeling. Both forms of the traction–separation laws result in a work of adhesion of approximately 50 mJ/m$^2$ and the peak adhesive stress in the Lennard Jones relation is found to be about 50% higher than that obtained for the Dugdale law.

## 1. Introduction

Adhesion in nanoscale contacts is a ubiquitous and well-known phenomenon that is important in many applications, including microelectromechanical systems (MEMS), fibrillar adhesives, and atomic force microscope (AFM) based metrology and manufacturing processes. Adhesion at the nanoscale is often characterized through simple AFM-based pull-off force measurements in which an AFM tip (radius of 5–100 nm) is contacted to a surface and then subsequently retracted. The force is measured via deflection of the compliant AFM cantilever and the pull-off force is defined as the peak attractive force during retraction of the tip from the surface. If the AFM tip is paraboloidal in shape, the work of adhesion is proportional to the pull-off force divided by the tip radius and can be calculated using an adhesion mechanics model, such as the Johnson–Kendall–Roberts (JKR) [1], Derjaguin–Muller–Toporov (DMT) [2], or Maugis–Dugdale [3] analyses. The tip size as well as the elastic and adhesion properties of the contact determine which adhesion model is the most appropriate [4].

AFM pull-off force measurements are commonly used to characterize adhesion at the nanoscale because of their simplicity and the widespread availability of the AFM. However, the single value of work of adhesion that is obtained from pull-off measurements does not completely define the adhesion between two surfaces. Adhesion is more fully described by a traction–separation relationship [3,5,6], such as a Lenard-Jones potential [7], that defines the adhesive stress as function of separation distance between the surfaces. While the work of adhesion is the integral of the traction–separation relation, the work of adhesion does not provide direct information on magnitude of the adhesive stresses or the adhesion range, which are critical in the design and engineering

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of nanoscale contacts. For example, the adhesion range relative to the surface roughness magnitude is critical in determining whether or not two surfaces will adhere when brought into contact under light loads (e.g., the bonding of semiconductor wafers [8]). Surface force-mediated adhesion typically has a range on the order of nanometers and the adhesive stresses increase sharply over short distances near the surface, thus measurement of the traction–separation relationship is challenging. Traditional AFM-based pull-off force measurements cannot be used to obtain information on the adhesion range as the compliant AFM cantilever snaps in and out of contact during approach and separation [9]. Specialized measurement systems, such as the interfacial force microscope [10], have been developed to avoid the snap-in/out phenomena and measure the traction–separation relation, however, these systems are difficult to use and not widely available.

While an AFM tip is often idealized as a smooth paraboloidal asperity, real AFM tips typically have nanoscale roughness and complex 3D shapes due to manufacturing variations and changes in geometry that occur during fabrication [11]. Nanoscale surface roughness, can have a profound influence on adhesion [12,13]. Jacobs et al. [14] combined molecular dynamics simulations and *in-situ* transmission electron microscope (TEM) adhesion experiments to investigate the effect of tip roughness on the adhesion of ultrananocrystalline diamond (UNCD) and diamond like carbon (DLC) AFM tips to a diamond surface. Tip roughness, which was measured in 2D through high resolution TEM imaging, was shown to have a significant impact on the work of adhesion determined from the pull-off force. The measured work of adhesion decreased by more than an order of magnitude as tip roughness increased from 0.03 to 0.5 nm. On a larger scale, Grierson et al. [15] investigated the effect of the overall geometry of AFM tip on adhesion and demonstrated that the tip evolves from a paraboloidal shape to a power law geometry due to wear with repeated sliding. The change in geometry, which was measured via TEM imaging, was exploited to extract information about the adhesion range of the AFM tip—sample contact [15]. A key limitation in both of these previous studies has been that the geometry of the tip was only characterized in 2D via TEM imaging. The lack of 3D geometry information limits the analysis that can be done to account for the effect of tip geometry when applying mechanics models to extract information about the adhesion of the contact.

Here, we present a novel approach for measuring the properties of the traction–separation relationship of nanoscale contacts with AFM by combining pull-off force measurements with high-resolution measurements of the 3D geometry of the tip. Specifically, we show that the properties of the traction–separation relationship, namely the work of adhesion, adhesion range, and peak adhesive stress, can be extracted from pull-off force measurements of multiple tips with known (i.e., measured) complex geometries. These measurements are accomplished in the presence of snap-in/out by exploiting the sensitivity of the pull-off force to the nanoscale geometry of the tip.

The technique is demonstrated through adhesion measurements between ultrananocrystalline diamond (UNCD) tips and polymethyl methacrylate (PMMA) surfaces. The UNCD–PMMA interface is technologically relevant as UNCD AFM probes are used in tip-based nanometrology and nanomanufacturing processes and PMMA is a common polymer used in nanofabrication. UNCD is a polycrystalline diamond material made by chemical vapor deposition that has high hardness and wear resistance [16,17]. Thin films of PMMA are used as resists in e-beam lithography [18,19] and tip-based nanolithography [20–22]. More generally, PMMA is an amorphous thermoplastic with good transparency, chemical resistance, and high dimensional stability. As such, PMMA has a range of applications outside of nanofabrication, including as a component in nanocomposites [23,24], bone cements [25] and MEMS/NEMS [26,27].

## 2. Experimental methods

Pull-off forces between three different UNCD AFM tips (cantilever type: CTCT2, CTCT1 and SSCRL from Advanced Diamond Technologies, Inc.) and a PMMA surface were obtained using a standard AFM (Bruker Dimension Icon®). The PMMA surface was fabricated by spin coating PMMA photoresist (PMMA-A4-495 from MicroChem®) at 5000 rpm for 50 s on a silicon wafer and then heating on a hotplate at 180 °C for 10 min. The film thickness is approximately 100 nm, which is much larger ($>40\times$) than the maximum indentation depths in the AFM adhesion tests. The spring constants of the AFM cantilevers were determined via the thermal tune method [28]. Pull-off force measurements were performed by displacing the tip into contact with a PMMA sample until a specified maximum normal load was reached and then retracting the cantilever from the surface. The pull-off forces were recorded as the maximum adhesive force observed in the force–displacement curves during retraction. The approach and retraction speeds were fixed at 500 nm/s in all tests. The maximum applied loads were varied from $\sim$3.5 to 100 nN for each tip. The tests at different loads were performed in an arbitrary order. A minimum of 20 measurements were taken at each load for each tip. Each measurement was done at a new location on the PMMA sample.

The 3D geometry of each AFM tip used was measured via inverse imaging. The inverse images were collected by scanning the AFM tips over a structured silicon sample containing multiple high-aspect ratio spikes (TGT-01 from NT-MDT®). The scans were done under contact mode with a low load ($\sim$3–5 nN) to avoid significant deformation of the spike and the image size varied from $100 \times 100$ nm$^2$ to $1 \times 1$ $\mu$m$^2$ in order to obtain measurements of the detailed features near the apex as well as the overall tip geometry. The spikes are spaced $\sim$2.2 $\mu$m apart and have a sharp radius ($<10$ nm) at the end. When the radius of spike is much smaller than the radius of the AFM tip, scanning over a single spike results in an image of the 3D geometry of the AFM tip [29]. This 3D imaging technique has previously been used to measure various types of AFM tips, including spherical $SiO_2$ and Cu tips [30]. For determination of tip geometry in the subsequent adhesion analysis, we used AFM images of apex of the tip with a