Nanocontacts: Fabrication, Characterization, and Nanotribology Studies

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Surface forces, particularly friction and adhesion, play dominant roles in the manipulation, assembly, and operation of micro- and nano-scale devices. This arises from the increased surface-to-volume ratio at small scales. Fundamental studies of friction and adhesion are required to fully understand their origins and to enable control and optimization for successful nano-device operation.

Materials with high hardness and low friction are of particular interest. Diamond-like carbon (DLC) films are a notable example, exhibiting high hardness, low friction and wear, and ease of application. We have undertaken a study of the nano-scale friction and adhesion properties of DLC-on-DLC nano-contacts.

DLC is an amorphous material with varying degrees of *sp-2* and *sp-3* carbon bonds, and has varying levels of hydrogen (up to 50 at.%), to stabilize the amorphous structure. It can be doped with elements such as fluorine to modify its surface energy and other properties. DLC is an excellent coating to reduce friction and wear in macroscopic (e.g. diesel engine components) and microscopic (e.g. computer hard disks) applications[1]. DLC deposition can be carried out at room temperature, producing smooth, conformal thin coatings. This opens the door for its application in the rapidly growing areas of micro-electromechanical systems (MEMS) and nano-devices.

The atomic force microscope (AFM) is an ideal tool with which to study contact and friction in a fundamental way[2] (Fig. 1). Here, a tip, with typically 10-100 nm radius of curvature, is attached to a compliant cantilever spring. At low applied loads, the tip can form a nanometer-scale single contact point (an “asperity”) with a variety of sample surfaces, thus providing a well-defined interface (Fig. 2). The cantilever deflections are recorded using, most commonly, a reflected optical beam. These deflections are converted to forces by using Hooke’s Law. In principle, the normal and lateral forces can be measured with sub-nanoNewton precision, with displacement accuracies in the sub-Ångstrom range. This tip is rastered using piezoelectric scanning tubes.

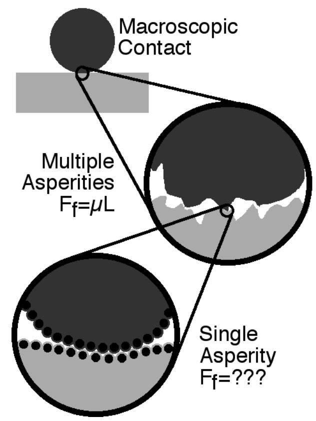

Fig. 1. The typical surface roughness of macroscopic interfaces ensures a complex multitude of contact points (asperities). A scanning force microscope provides a well-defined single asperity contact (the tip) where interaction forces can be precisely measured with nanometer/atomic resolution. Friction deviates from macroscopic behavior at this scale.

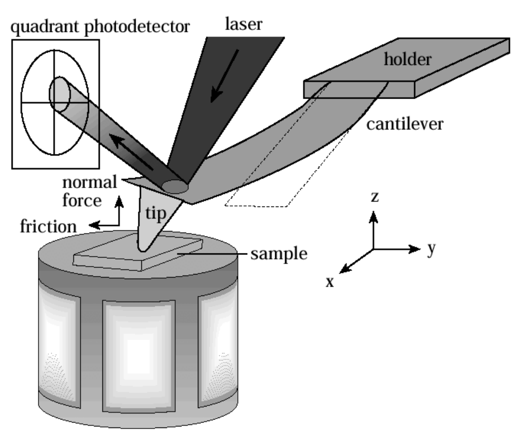

Fig. 2. Schematic of the typical AFM set-up. The tip is in contact with a sample surface. A laser beam is focused on the back of the cantilever and reflects into a four-quadrant photodetector. Normal forces deflect the cantilever up or down, lateral forces twist the cantilever left and right. These deflections are simultaneously and independently measured by monitoring the deflection of the reflected laser beam.

In order to study the fundamental tribological properties of DLC coatings, we have successfully deposited DLC on atomic force microscope tips using Plasma Source Ion Deposition (PSID) [3]. We have also deposited DLC films on flat substrates with this method. In PSID, the target to be coated is placed in a vacuum chamber, the source gas is introduced, and a plasma of this gas is produced. High-voltage pulses are then applied to the target causing the ions in the plasma to impinge the target surface at normal incidence and deposit a film (Fig. 3). The advantage of this non-line-of-sight method is that coatings can be applied to three-dimensional objects with unusual shapes, including high aspect-ratio AFM tips.

We have deposited DLC and fluorine-doped DLC (F-DLC) films, nominally 40 nm thick, on silicon and silicon nitride AFM tips, as well as onto atomically flat silicon substrates, using acetylene and a mixture of acetylene+tetrafluoroethane as precursor gases, respectively. Depositions were carried out at target biases of 2 to 5 kV. Electron microscopy and AFM tip deconvolution experiments reveal a conformal coating terminating in a smooth single asperity <70 nm in diameter (Fig. 4), suggesting that the enhanced electromagnetic field at the tip sharpens the tip asperity. The coated substrates exhibit an astounding RMS roughness < 0.2 nm, with uniform friction and adhesion and few decohesion events.

Comparative AFM measurements were conducted by acquiring a series of adhesion and/or friction measurements on several locations of a DLC sample, immediately followed by several measurements with the same tip on the F-DLC. A new cycle of DLC and F-DLC measurements were conducted, so that we could ascertain that any contrast observed in the measurements was reproducible. This process eliminates uncertainties that may arise due to possible changes in the AFM tip. In general we observe that the initial run of measurements shows some change in friction and adhesion, but following that, the measurements become

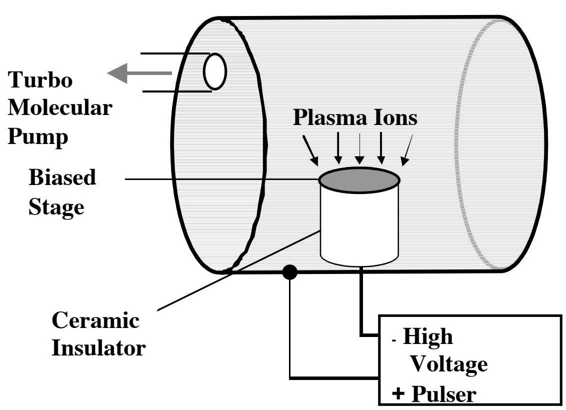

Fig. 3: The PSID technique.

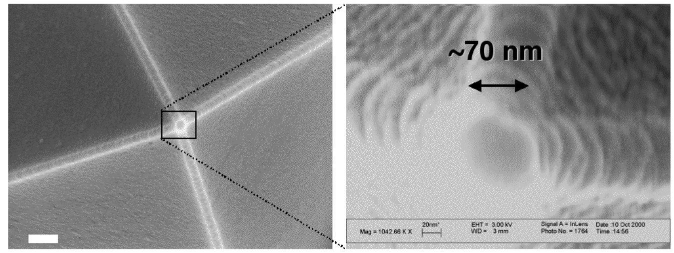

Fig. 4. SEM images of a pyramidal AFM tip coated with DLC. The apparent roughness is due to the pre-existing texture of the silicon nitride tip. At left, scale bar = 200 nm.

stable. This kind of nano-scale "run-in" with AFM tips is not uncommon. Adhesion was determined by measuring the force required to pull the tip out of contact from the surface. According to continuum mechanics, for a single asperity contact between a nominally spherical elastic body (the tip) and a flat surface, the adhesion force will be directly proportional to the product of the adhesion energy and the tip radius[4,5]. Friction was measured by determining the width of the friction loop generated by rastering the tip back and forth over a 50 nm range. The friction measurements were uniformly obtained in the absence of any externally applied load (where the tip is held in contact with the sample by the adhesion force). The measurements were obtained using a Digital Instruments Nanoscope IIIA AFM. Due to instrumental limitations, the friction force was not calibrated so it is reported as raw signal Volts. The adhesion force was calibrated using the nominal force constant of the cantilever, 0.06 N/m. This figure has a substantial uncertainty because of the cantilever manufacturing process, and so the quoted adhesion values should be considered as merely estimates.

The DLC substrates were first probed using a silicon nitride AFM tip. The SiN:DLC and SiN:F-DLC contacts are of interest as such interfaces may occur in MEMS applications that integrate DLC coatings. We observe that the fluorine-doped DLC films, surprisingly, exhibit higher adhesion and friction than the undoped DLC films (Fig. 5), with the exception of the first friction measurement. We believe that the first measurement is indicative of a transient increase in the tip size that occurred initially. Each friction data point in Figure 5 represents the average of 10 sequential measurements. Indeed, the 10 individual friction measurements for the anomalous first run show a progressive increase in the friction force, while the individual measurements for the remaining runs showed no such increase, indicating a stable tip configuration. Disregarding the first transient measurement for both adhesion and friction, the adhesion force was approximately 13% higher on the F-DLC, and the friction force was 33% higher. The substantially larger friction contrast indicates that factors other than just the slightly increased adhesion energy are contributing to the friction force. The friction force will be higher if the F-

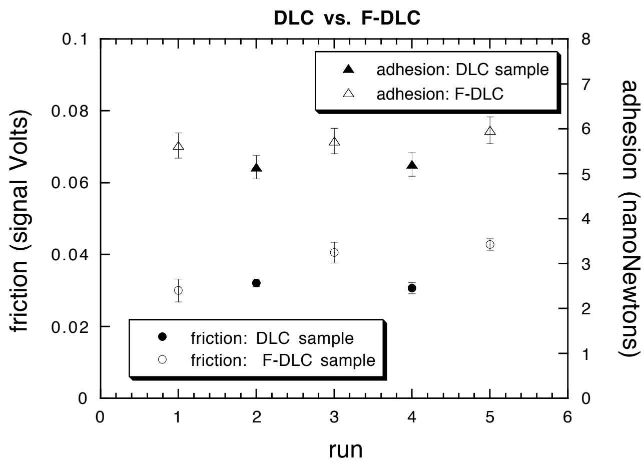

Fig. 5. Friction force (circles - uncalibrated units) and adhesion (triangles - nN of force) for DLC (solid) and F-DLC (open) samples, measured with a silicon nitride tip. The error in the friction data represents the standard deviation of 10 successive measurements. The error in the adhesion data represents the uncertainty in the adhesion force measurement due to non-linearity of the piezo scanner.

DLC has a lower near-surface elastic modulus than the undoped DLC, as that would increase the area of contact given the same load. Furthermore, the interfacial shear strength (friction force per unit contact area) may also be increased due to the enhanced interfacial interaction indicated by the adhesion energy. This explanation is consistent with the observation that increasing the fluorine content of DLC generally results in softer films with lower compressive stresses, however this dependence is influenced strongly by deposition parameters such as voltage bias and pressure [6]. Nano-scale measurements of the elasticity, as well as friction measurements as a function of load, are needed to resolve the factors that produce the substantially higher friction for F-DLC.

AFM measurements were then carried out to determine adhesion between DLC-on-DLC nanocontacts. We used AFM tips coated with F-DLC for these measurements. Tips were imaged after the measurements in an electron microscope to verify that no damage to the coating had occurred. We found that F-DLC tips exhibit 60% higher adhesion forces with fluorinated DLC samples than with undoped DLC samples. This contrast is substantially greater than the adhesion contrast measured with the silicon nitride tips. Detailed measurements of the corresponding dependence of friction upon doping are underway.

These initial results indicate that doping levels can have a substantial impact on the tribological properties of DLC at the nanometer scale. The doping modifies the surface energy, but the resulting change in the friction properties is even more substantial. This indicates that fluorination may have multiple effects by altering the surface energy, elasticity, and interfacial shear strength of DLC. Further AFM measurements will allow us to separate these contributions.

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