Editor's Note: For a closer look at Xin's poster abstract, be sure to check out his short video presentation in the November digital version of TLT (available at **www.stle.org**)

Nanoscale Friction and Adhesion Behavior for Few-Layer Graphene

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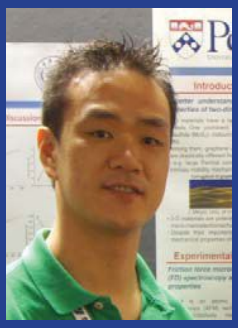

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INTRODUCTION

Two-dimensional (2-D) materials, including graphene, have drawn much attention because of their notable electronic, thermal, chemical, and mechanical properties, making them outstanding candidates for future electronic devices. For example, the electronic properties of graphene make it suitable for use in ultrahigh frequency transistors for fast nanoscale electronics,[1] or quantum dot-sized transistors,[2] as well as for micro- and nano-electromechanical systems (MEMS/NEMS), ultrahigh frequency resonators,[3,4] and high accuracy gas and mass detectors.[5,6] To date, the tribomechanical properties of 2-D materials have received less attention compared to their electronic and thermal properties, and tribomechanical properties remain poorly understood despite their importance in determining the applicability of 2-D materials to various devices. The study of the tribomechanical properties of 2-D materials is scientifically relevant since, as other studies have shown, materials will behave very differently from their 3-D counterparts due to dimensionality effects.[7,8] Therefore, before 2-D materials can be considered in real next-generation applications, the mechanical and nanotribological properties of these materials must be better understood. In this study, our goal is to characterize the nanoscale friction and adhesion properties of graphene.

METHODOLOGY

For friction experiments, samples were deposited under ambient conditions onto SiO2/Si substrates, a method that is described in,[9] and they were used for all experiments without further treatment. Friction experiments were performed in contact-mode by atomic force microscopy (AFM), as depicted in Figure 1 using a Park Systems XE-100 AFM in ambient conditions (25%-50% relative humidity, room temperature), and a RHK UHV350 AFM, where the sample chamber was purged by dry nitrogen gas (1%-2% relative humidity, room temperature). Contact-mode silicon AFM probes were used for all the experiments. The number of layers for all sample regions probed was determined based on the topographic AFM images. In several cases, Raman spectroscopy was also used independently for verifying the thicknesses.

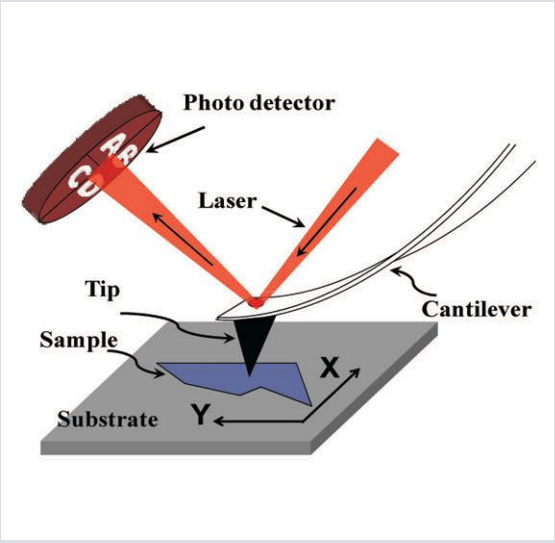

Figure 1 | **Schematic setup of an AFM that was used for both friction and adhesion measurements in this study.**

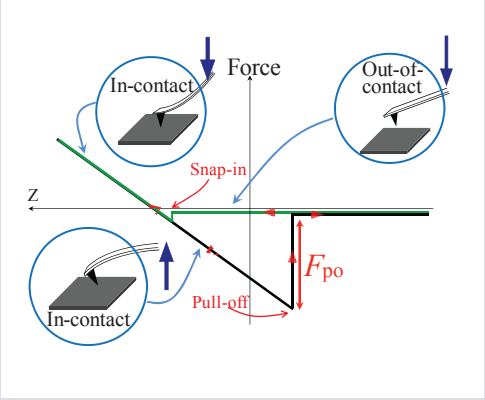

Figure 2 | **Schematic working principle of force-distance (FD) spectroscopy. A typical FD curve, with the position of the AFM tip and cantilever with respect to the sample surface shown at different stages. The adhesion force can be extracted from the pull-off force (F_{po}) of an FD curve.**

For adhesion experiments, samples of graphene were prepared in the same way as those for friction experiments. The tests were performed in the same RHK AFM system (1%-2% relative humidity, room temperature) using the regular force-distance (FD) spectroscopy, as illustrated in Figure 2.

ILLUSTRATIVE RESULTS

From the friction measurements, we observed a trend that the friction force—starting from one single layer—decreases monotonically with increasing number of layers of graphene. Moreover, the friction force on samples of about four layers is approximately the same as on the bulk materials. Those results are shown in Figure 3. A similar trend has been predicted using finite element modeling (FEM) simulations. A puckering effect of the thin sheet has been proposed to cause this phenomenon. In that effect, thinner sheets are more susceptible to out-of-plane elastic deformation than thicker sheets, provided they are not strongly bound to the substrate.[10,11]

In contrast to the friction results, the adhesion between an AFM tip and graphene, measured using the regular FD spectroscopy, does not exhibit a significant dependence on the number of layers. This insensitivity to the number of layers is consistent with the results from FEM simulations. The experimental results are plotted in Figure 4. The variation in adhesion for different numbers is within 10%, whereas the friction measured between the tip and one monolayer of graphene is 50% greater than that for four layers or more.

We also obtained a second set of adhesion tests using a modified form of FD spectroscopy. In that test, adhesion is measured immediately after sliding the AFM tip over the same area for a sufficient distance without breaking the tip-graphene contact until the pull-off itself. Our preliminary results suggest that the adhesion between graphene and the tip has a sliding-history dependence. The adhesion is enhanced by sliding the AFM tip over the same area. This suggests that the enhanced contact area between the graphene and the tip requires a sufficient amount of sliding. This may be emblematic of the fluctuating nature of the graphene sheet, whose geometric configuration is strongly affected by the sliding action.

SUMMARY

In this work, we studied the friction and adhesion characteristics between exfoliated 2-D materials and nanoscale single asperity tips using AFM. We observed layer-dependent friction for graphene. This observation coupled with finite element modeling (FEM) suggests that the trend is caused by the puckering effect of the thin graphene sheets. Regular FD spectroscopy also

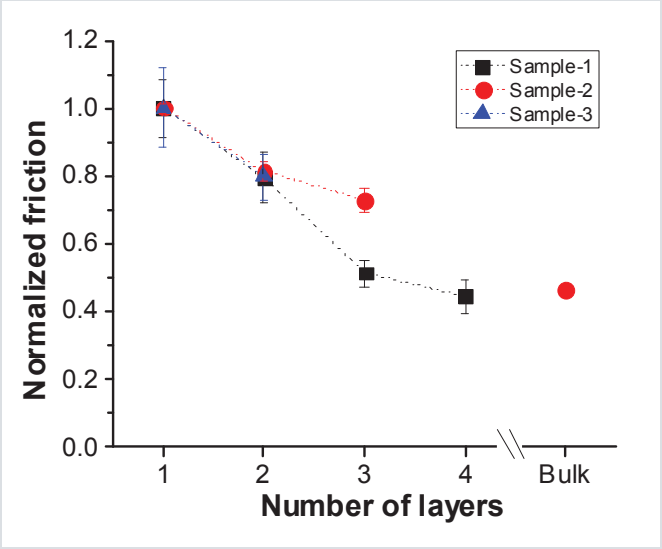

Figure 3 | **Normalized friction force on areas with different number of layers. Friction forces were normalized to the value obtained on the thinnest layer.**

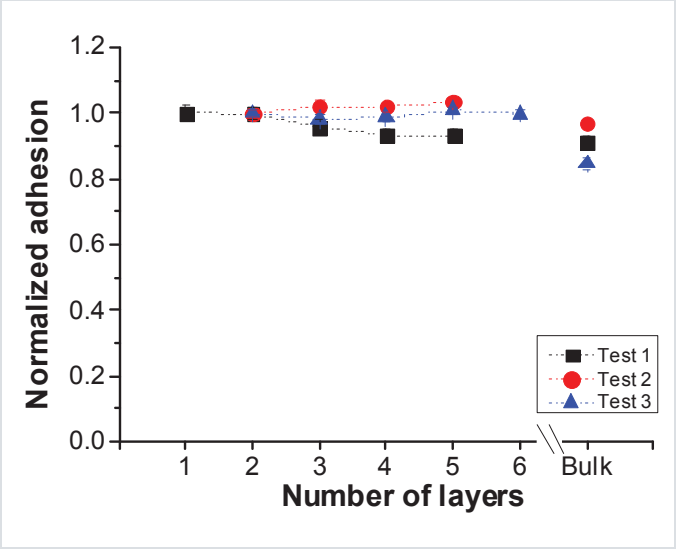

Figure 4 | **Normalized adhesion force versus graphene layer number in dry N_2 for a silicon tip on three different samples.**

showed that the pull-off force between graphene and the AFM tip did not have an appreciable dependence on the number of layers, which is consistent with FEM predictions. However, our preliminary results obtained using a modified form of FD spectroscopy suggest that the graphene adhesion has a sliding-history dependence.

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