Mater. Res. Soc. Symp. Proc. Vol. 1226 © 2010 Materials Research Society 1226-II05-02

Temperature dependence of nanoscale friction investigated with thermal AFM probes

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ABSTRACT

Measurements of nanoscale friction between silicon AFM tips featuring an *in-situ* solid state heater and silicon substrates (both with native oxide) were performed. The temperature of the heater was varied between room temperature and approximately 650 °C. For these temperatures and the silicon substrate, the temperatures at the point of contact are estimated to range from room temperature to approximately 120±20 °C. Experiments were carried out in ambient atmosphere (~30% relative humidity) and under dry nitrogen. Tests under constant load revealed that in the presence of ambient, friction increased with heater temperature whereas it did not change in dry nitrogen. For experiments carried out for different tip velocities (40 to 7800 nm/s), friction decreased with velocity in ambient and did not change in dry nitrogen. Both trends can be explained by thermally-assisted formation of capillary bridges between tip and substrate and the kinetics of capillary condensation under ambient conditions.

INTRODUCTION

The temperature dependence of friction is of increasing interest as it affects energy dissipation, wear, and reliability in a multitude of systems, yet a scientific understanding is lacking (1-6). The motivation to address this problem with atomic force microscopy (AFM) is found in the complex nature of friction processes. When macroscopic objects slide against each other, the contact is not continuous but composted of many asperities. With the extremely sharp tip of an AFM, it is possible to study one of these asperities individually (7, 8). With only one asperity in contact, such experiments allow for more definitive analysis of the basic physical principles and processes governing friction. As only little is known about the temperature dependence of friction, performing tests at different temperatures is of significant scientific interest. For example, to determine the activation energies of dissipation processes in friction, measurements at different temperatures are necessary. Most studies published in this field so far (1, 2, 4, 9, 10), were performed by changing the temperature of the substrate and in ultra high vacuum (UHV), or by changing the temperature of the entire experimental chamber (11). In this contribution, the temperature dependence of single asperity friction was investigated by means of heated AFM cantilevers, thus introducing a new approach to changing the temperature in nanotribology measurements. A key advantage to these experiments is that the tip temperature can be rapidly changed *in-situ* (with a time constant of microseconds). Having much faster access to a range of temperatures not only increases throughput, but it also allows one to obtain far more reliable measurements since the temperature can be successively increased and decreased multiple times. Thus, one can rapidly and convincingly separate trends due to progressive tip wear from true thermal effects. A second advantage is that this configuration is amenable to *in-situ* tip-based nanomanufacturing. How friction and the related phenomena of wear and adhesion depend on tip temperature must be understood to enable reliable, high-throughput tip-based nanomanufacturing applications. These tips have their origin in IBM's Milliped project (12) and rely on Joule heating of differently doped parts of the cantilever (13). The typical cantilever geometry is shown in Fig. 1.

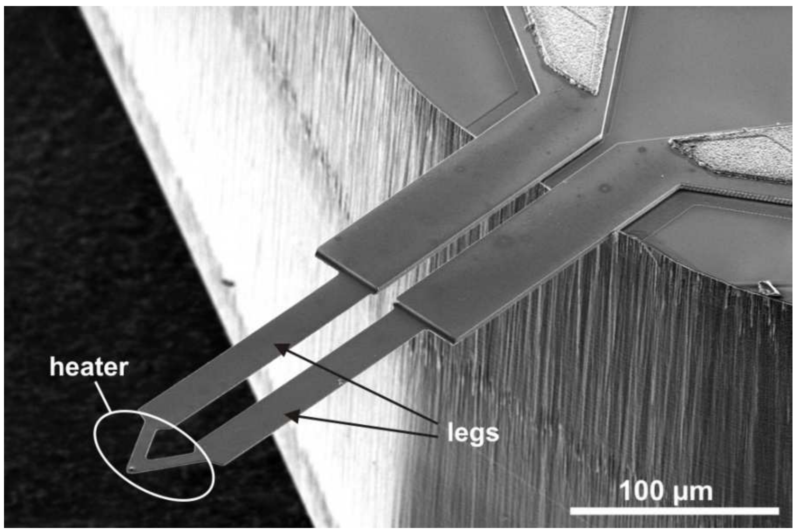


Figure 1: SEM image of a heated AFM probe. The "legs" part of the cantilever is doped differently with phosphorous than the "heater" part where the tip is located. Upon driving a current through the structure, the heater gets hot due to Joule heating. It reaches temperatures up to 650 °C within milliseconds.

EXPERIMENT

The experiments in this study were performed with silicon AFM probes featuring an *in-situ* solid state heater (Figure 1). The heater temperature was not controlled directly. A function generator (Model 33120A, Agilent Technologies, Santa Clara, CA) in combination with a SRS scaling amplifier (Model SIM983, Stanford Research Systems, Sunnyvale, CA) were used to supply different, constant DC voltages. Calibration between heater temperature and supplied voltage (13) was performed by micro Raman experiments (14). Silicon (100) wafers with a native oxide layer served as substrate material. The wafers were provided by El-Cat (Waldwick, NJ) and cleaned with piranha solution (5 parts H_2SO_4, 1 part H_2O_2) to remove all organic contamination. After removal from piranha, the substrates were rinsed with deionized water and blown dry with dry nitrogen.

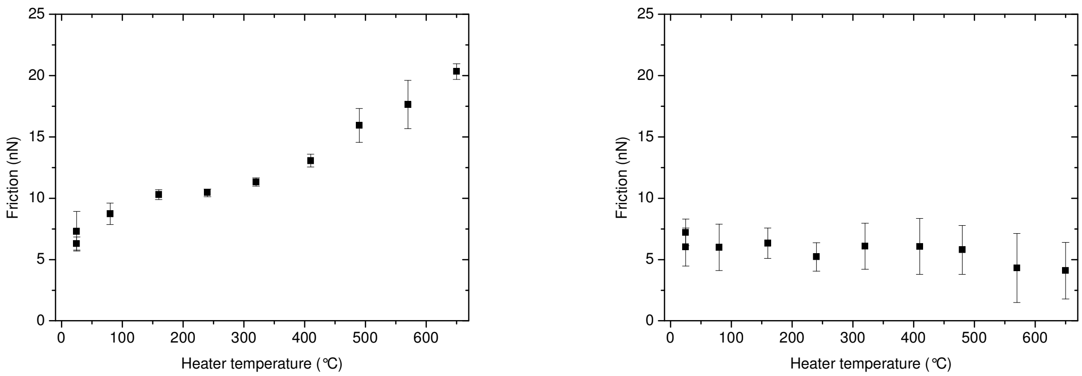
The normal force constant of each cantilever used was determined *in-situ* at all heater temperatures by applying the reference cantilever method as described by Tortenese *et al.* (15, 16). The reference cantilevers were provided by Veeco (Santa Barbara, CA). The lateral spring constant of each cantilever was determined with a diamagnetic lateral force calibrator as described by Li *et al.* (17) at all heater temperatures.

An Asylum Research (Santa Barbara, CA) MFP 3D AFM was used for the experiments. Air temperature and humidity were monitored (Humidity Meter 11-661-21, Fisher Scientific, Waltham, MA) and were approximately 21°C and 25-35% relative humidity (RH) respectively for all tests. To determine the temperature dependence of friction, experiments at a constant normal load – *i.e.*, a fixed deflection setpoint – were performed. The scan area was 100 nm by 100 nm and the scan rate was 2 Hz for 512 x 512 lines. Tests were carried out for the unheated state (no applied voltage to the cantilever) and at eight different voltages which corresponded to heater temperatures between approximately 80 and 650 °C in 80 °C increments. Any twisting and bending of the cantilever with voltage was accounted for by centering the AFM's photodiode at all heater temperatures with the tip retracted from the surface. The temperatures were varied in a randomly alternating fashion. The first experiment was carried out without applied voltage, corresponding to room temperature. The following order for the heater temperatures was chosen: 570, 80, 490, 160, 410, 240, 330 and 650 °C. The last experiment was again performed at room temperature to rule out tip wear influencing the data. Before and after each set of experiments, the shape of the tip was monitored by scans on ultra nanocrystalline diamond (UNCD) surfaces (18) and blind reconstruction. This procedure was implemented for additional confidence in the absence of tip wear. The temperatures at the point of contact between tip and substrate were not known directly. The temperature increase at the contact depends on the tip size and the thermal conductivity ratio of the tip and the sample. Using 20 nm for the tip radius and a thermal conductivity ratio of 1, the contact's temperature increase is calculated to be approximately 15 % of the heater's (19). Thus, it ranged from room temperature (RT) to approximately 120 °C. The uncertainty in the temperature rise has many sources, e.g. the exact contact pressure and radius, as well as assumptions made in deriving the theory. It is very difficult to give a precise number for the expected error in tip temperature but it might well be ±20 °C. To investigate the velocity dependence of friction, measurements at ten different scan velocities ranging from 40 nm/s to 7800 nm/s were performed. For these tests, the scan line width was 100 nm, and 512 by 128 lines were scanned. To test the effect that water vapor on our results, experiments in dry nitrogen atmosphere were carried out. The samples were prepared as described above and placed inside a BioHeater Closed Fluid Cell from Asylum Research. The assembly allowed for a closed confinement which was flushed with dry nitrogen (Airgas, Radnor, PA) for at least one hour before and during the experiments. All measured data was analyzed using MatLab (MathWorks, Natick, MA) code (20).

RESULTS AND DISCUSSION

Friction at constant normal load

Nanoscale friction was measured as a function of heater temperature at a fixed normal load of 112 nN and velocity of 400 nm/s in ambient and in dry nitrogen atmosphere. The results are presented in Figure 2.



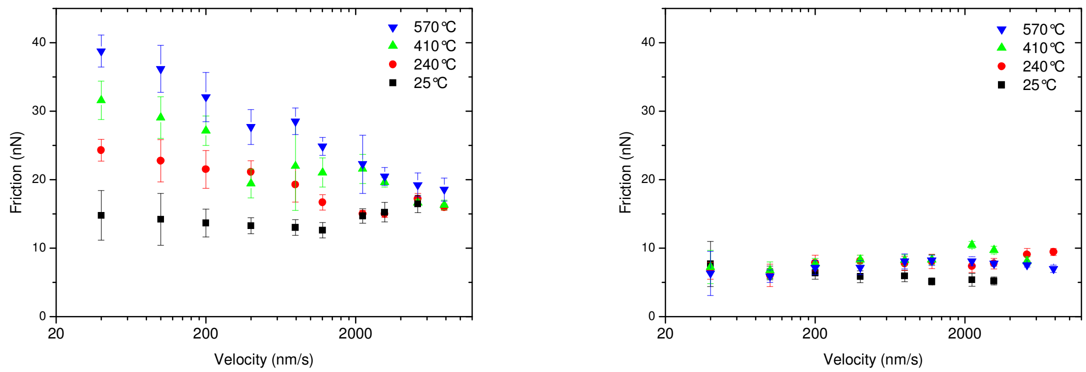
(a) (b)

Figure 2: Friction as a function of heater temperature measured at a constant normal load. The heater temperatures were chosen in an alternating order. (a) Results from measurements in ambient, (b) dry nitrogen atmosphere.

In ambient atmosphere, friction increased with temperature monotonically, increasing by a factor of nearly 4 as the heater temperature increases from room temperature to 650 °C. This result was unexpected as the most common theoretical description of dry friction is that of a thermally activated process (21, 22). In such a framework – as it is provided in the classical Prandtl-Tomlinson model – friction forces are expected to decease with temperature. In this picture, the tip sticks at metastable positions determined by local energy minima in the lateral tip-sample interfacial potential. Thermal activation assists in overcoming the barrier to the next metastable position, thus, friction reduces with temperature. However, the opposite trend is seen to be the case in our experiments. However, for the tests carried out in dry nitrogen, friction did not change over the entire range of heater temperatures. The absolute friction values were lower in dry nitrogen than in ambient. These results demonstrate that humidity strongly influences friction. How exactly water contributed will be discussed in detail below.

Velocity dependence

The dependence of friction on velocity was investigated at four different heater temperatures (RT, 240, 410 and 570 °C). The results are presented in Figure 3 at a normal load of 112 nN.



(a) (b)

Figure 3: Friction as a function of sliding velocity and temperature. Velocity was varied between 40 and 7800 nm/s. Measurements were performed for four different heater temperatures: room temperature, 240, 410 and 570 °C (estimated tip temperatures: room temperature, 56, 81 and 105 °C). (a) results from ambient, (b) from dry nitrogen atmosphere.

An increase in friction with velocity was theoretically predicted in several models (23-27). These predictions were based on the afore mentioned Prandtl-Tomlinson model. At higher velocities, less time is available at each metastable position to allow thermal activation to assist in sliding, and so friction increases with temperature. As seen in Figure 3, our data show very different behavior. For the experiments in ambient, friction decreased with velocity. The trend is more pronounced for higher heater temperatures, varying by as much as a factor of 2. For the two lower temperatures, friction values show a plateau at higher velocities or even a slight increase. In dry nitrogen, there is no trend with velocity. As in the case of constant load and velocity, the absolute friction values are lower in dry nitrogen than in ambient.

The results can be interpreted in the context of water meniscus formation at the tip-sample interface. In the ensuing discussion, we will focus on previous studies which are key to interpreting our experiments. A thorough discussion of further related literature will be part of a full journal paper on this study.

Szoszkiewicz and Riedo (11) measured friction between a silicon tip and a soda lime glass substrate in an argon atmosphere of 37% relative humidity. They encapsulated their AFM and varied the temperature of the entire setup between 26 and 59 °C. For tip velocities between 100 nm/s and 120 µm/s they reported a behavior very similar to ours: friction forces decreased with velocity. The trend was stronger for higher temperatures, and for high velocities a transition to a plateau region was seen. The transition shifted to higher velocities at higher temperatures. This is consistent with our observations with the difference that in our case, for the two highest heater temperatures the plateau region was not reached even at our fastest scan velocity possible. Szoszkiewicz and Riedo (11) argue that the behavior they observed had its origin in a capillary bridge forming between tip and substrate. They suggest that the formation of the bridge follows an Arrhenius type law for a thermally activated process. Thus, elevated temperatures helped to overcome the energy barrier for bridge formation, resulting in a larger bridge. The capillary bridge creates an attractive force between the tip and sample, and this is assumed to increase friction, either through the increased tip-sample contact area or an increase in the intrinsic frictional resistance between the tip and sample (the interfacial shear strength). Friction reduces with velocity because it becomes more difficult for the capillary to follow the sliding contact.

Using their reasoning, we can explain our results as follows: A larger capillary bridge at higher heater temperatures led to the increase in total normal force and therefore friction with temperature, as seen in Figures 2a and 3a. The suppression of a capillary bridge between tip and substrate in dry nitrogen resulted in a constant friction force in Figure 2b. The velocity dependence seen in Figure 3a can be explained in the same framework. As the tip scanned faster over the substrate, it becomes more and more difficult for the capillary bridge to follow the contact. Thus, friction forces decreased. At some point, the tip velocity becomes too high for the bridge to follow. This corresponds to the onset of the plateau region. As the formation and movement of the bridge is thermally activated, higher heater temperatures shifted this onset to higher velocities. This is what is observed in Figure 3a, and by Szoszkiewicz and Riedo (11). Consistent with this argument, we did not observe a decrease in friction with velocity (Figure 3b), and the absolute forces were lower in dry nitrogen atmosphere. The same is true for pull-off force measurements (data not shown here) where adhesion was found to be lower in dry nitrogen atmosphere than in air. The formation of capillary bridges between an AFM tip and a smooth substrate has long been assumed to occur in humid environments and to be responsible for the dependence of pull-off (adhesion) forces on humidity. It has been explicitly demonstrated by Monte Carlo simulations (28, 29) and directly observed by environmental scanning electron microscopy (30, 31). The idea of a dominance of capillary forces at ambient pressures was also proposed by Opitz *el al.* (32, 33) as well as other authors (34, 35).

CONCLUSIONS

Heated AFM cantilevers were used to measure nanoscale friction as a function of temperature. The substrate material was piranha cleaned silicon. The dependence of friction on temperature, sliding velocity, and humidity were measured. An explanation for these results, consistent with the literature, was found in the formation of a capillary bridge between tip and substrate. Due to the kinetics of capillaries, higher heater temperatures increased the rate of bridge formation, thus friction increased with temperature. The faster the tip moved over the surface, the more difficult it was for the capillary to follow the contact, resulting in a friction decrease with velocity. As the presence of a capillary bridge relied on water in the atmosphere, the behavior in dry nitrogen was different and no trend with heater temperature or scan velocity was found.

ACKNOWLEDGMENTS

This research was supported by the Nano/Bio Interface Center through the National Science Foundation NSEC DMR-0425780. C.G. acknowledges a Feodor Lynen Fellowship of the Alexander von Humboldt foundation. W.P.K. acknowledges funding from DARPA. We thank Dr. T. Das and Prof. Y. Goldman for assistance with the Asylum AFM.

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