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Nano-scale adhesion and friction on Si wafer with the tip size using AFM

R. Arvind Singh, Eui-Sung Yoon†, Hyun-Jin Oh and Hosung Kong

Tribology Research Center, Korea Institute of Science and Technology, P.O. Box 131, Cheongryang, Seoul 130-650, Korea

Abstract: Nano-scale studies on adhesion and friction were conducted in Si-wafer (100) using Atomic Force Microscopy (AFM). Glass (Borosilicate) balls of radii 0.32 μm, 1.25 μm and 2.5 μm mounted on cantilever (Contact Mode type NPS) were used as tips. Adhesion and friction between Si-wafer and glass tips were measured at ambient temperature (24 ± 1°C) and humidity (45 ± 5%). Friction was measured as a function of applied normal load in the range of 0-160 nN. Results showed that, both adhesion and friction increased with the tip radii. Also, friction increased linearly as a function of applied normal load. The effect of tip size on adhesion and friction was explained as the influence of the capillary force exerted by meniscus and that of the contact area on these parameters respectively. The coefficient of friction was estimated in two different ways, as the slope from the plot of friction force against the applied normal load and as the ratio between the friction force and the applied normal load. Both these estimates showed that the coefficient of friction increased with the tip size. Further, the influence of the adhesion force on the coefficient of friction was also discussed.

Key words: Nano, adhesion, friction, tribology, AFM

Introduction

During the last decade, studies using Atomic force microscopy (AFM) have increasingly promoted the fundamental understanding of the tribological phenomena such as adhesion, friction and wear between materials at nano/micro scale levels [1]. Recently, the adventage of micro-electro-mechanical systems (MEMS) has also popularized investigations directed towards understanding the tribology at these levels. Silicon is a widely used material in MEMS, and hence most of these investigations are directed towards understanding and enhancing its tribological performance at nano/ micro scales [2]. At these scales, the ratio of surface area to volume is high, which renders the surface forces such as adhesion and friction to play an important role in defining the tribology at the contact. It is important to understand the effects of adhesion and friction while studying tribology at nano and micro scales.

Attractive forces such as capillary, electrostatic, van der Waals and chemical bonding contribute to adhesion under different circumstances. Maboudian *et al.* and Komvopoulos [3,4] provide an excellent review on the various surface forces that cause adhesion. Experimental studies towards understanding the adhesion between solid surfaces were first conducted by Israelachvili and Tabor [5]. They measured van der Waals forces between cleavage sheets using the Surface Force Apparatus (SFA). In the recent years, such studies are now being conducted using AFM [6,7]. Investigations in tribology at nano/micro scales has brought forth various approaches towards reducing the surface forces at the contact between solids, by undertaking topographical [4] and chemical modifications [8,9] of the surfaces.

In the present work, nanoscale studies on adhesion and friction in Si-wafer (100) were conducted experimentally using AFM. Adhesion and friction were measured with tips of different radii. Friction was measured as a function of applied normal load. The effect of tip size on adhesion and friction has been discussed in terms of the capillary force and the contact area, respectively. To understand the behaviour of the coefficient of friction at nanoscale, the coefficient of friction was estimated in two different ways, namely, as the slope from the plot of the friction force versus the applied normal load and as the ratio of the friction force to the applied normal load. Further, the relationship between the adhesion force and the coefficient of friction was also discussed.

Experimental

Test apparatus

Nano-adhesion and nano-friction tests were conducted using a commercial Atomic Force Microscope (Multimode SPM, Nanoscope IIIa, Digital Instruments). A view of the Atomic Force Microscope used in the present study is shown in Fig. 1.

Adhesion measurements were conducted in Force-Displacement mode and the friction force was measured in LFM (Lateral Force Microscope) mode.

Specimens

Glass (Borosilicate) balls of radii 0.32 μm, 1.25 μm and 2.5 μm, mounted on triangular cantilever (Contact Mode type NPS; nominal spring constant 0.58 N/m) were used to study the effect of tip size on nano-adhesion and nano-friction.

†Corresponding author; Tel: 82-2-958-5651, Fax: 82-2-958-5659
E-mail: esyoon@kist.re.kr

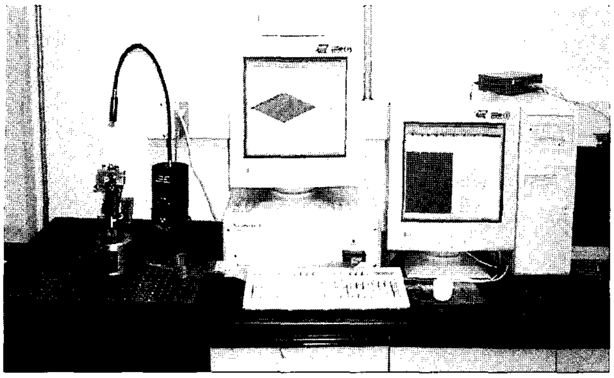

Fig. 1. A view of AFM used for the measurement of nano-adhesion and friction.

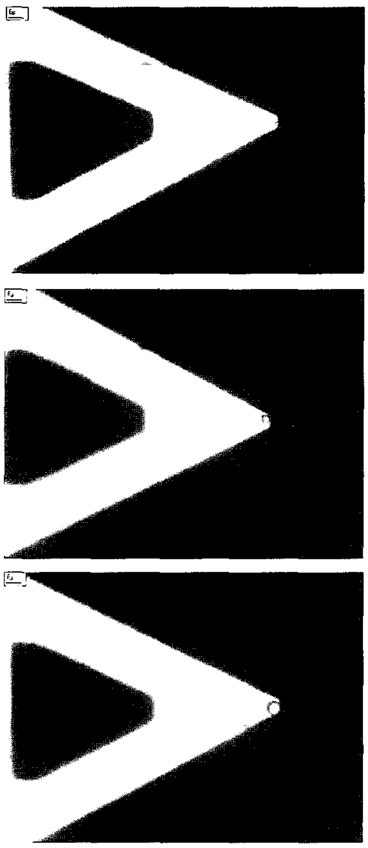

Fig. 2. Optical micrographs of borosilicate balls with radii 0.32 μm, 1.25 μm and 2.5 μm used as tips, mounted on commercial Si_3N_4 triangular cantilevers.

Fig. 2 shows the optical micrographs of the tip specimens taken at 400x magnification. All experiments were conducted on Si-wafer ((100), produced by LG Siltron) at ambient temperature (24 ± 1°C) and humidity (45±5%).

Table 1 shows the properties of the test specimens used.

Table 1. Young's modulus and Poisson's ratio of the test materials

Material	Young's modulus E (GPa)	Poisson's ratio v
Silicon Wafer	165	0.28
Borosilicate Ball	64	0.20

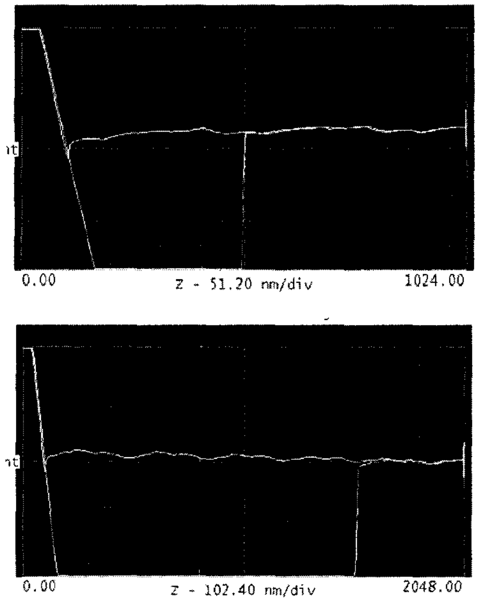

Fig. 3. Typical force-displacement curves observed for two different tip radii (0.32 μm and 1.25 μm).

Test methods

Adhesion force measurements

Adhesion force measurements were done at 40 nN normal load and the adhesion force (maximum pull-off force) was estimated from the force-displacement curves. Each test was repeated for 15 times and the average values were plotted. Fig. 3 shows two examples of such curves for two different radii, namely 0.32 μm and 1.25 μm. The horizontal axis represents the distance the piezo travels (and so the sample) and the vertical axis shows the tip deflection. The horizontal flat portion of the curve indicates the approach of the tip towards the sample. As the tip approaches closer to the surface, at point A, it snaps onto the surface due to attractive forces. The sloped portion on the left side of the point A, shows the deflection of the tip that exerts the set applied normal load onto the sample. As the piezo retracts, the tip enters into the adhesive regime after crossing over the zero deflection line (horizontal flat line). At point B it snaps free of the adhesion force. The distance between the points A and B shows the distance moved by the tip in the adhesive regime (x). The adhesion force (F_a) experienced by the tip was estimated using the values of the distance (x) and the stiffness of the cantilever (k) as $F_a = kx$.

Friction force measurements

Friction measurements were done at applied normal load in the range of 0-160 nN. Measurements were done at the scanning speed of 5 μm/s (scan rate of 0.5 Hz) for the scan size of 5 μm × 5 μm. Each test was repeated for 25 times and the average values were plotted.

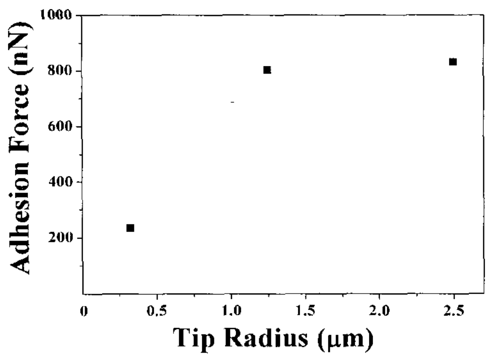

Fig. 4. Adhesion force on Si-wafer as a function of tip radii.

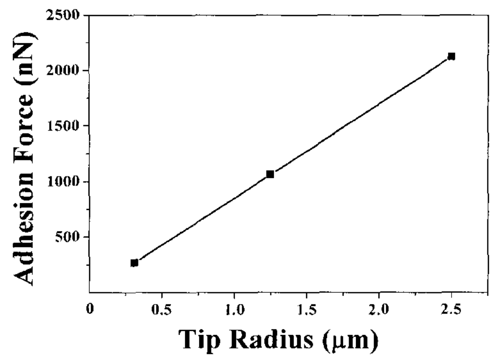

Fig. 5. Adhesion force affected by the formation of capillary for various tip radii, calculated using equation (1).

Results and Discussion

Adhesion at nanoscale

Fig. 4 shows the variation of single point adhesion force measurements as a function of tip radii. The data point for the 2.5 μm radius shows only a slight increase when compared to that of the 1.25 μm radius. This was due to the limitation of the measurement system. However, the trend shows that the adhesion force increases with the tip radii.

Adhesion at nano-scale is a combined effect of various interfacial forces such as capillary, electro-static and van der Waal forces. Among these forces, the capillary force is the strongest [3]. This capillary force is a result of water condensation from the environment that leads to the formation of the meniscus bridge. When a sphere of radius R contacts a plane, a capillary of condensed water (meniscus) is formed around the contact surface. The adhesion force generated by the capillary formation is given by [10]:

$$F_m = 4 \pi R \gamma \cos\theta \quad (1)$$

Where R is the tip radius, γ the surface tension of water and θ the contact angle of water on the mating surface. Fig. 5 shows the estimated values of adhesion force (F_m) using equation (1), as a function of tip radii, considering R = 0.32 μm, 1.25 μm and 2. μm; $\gamma = 7.2 \times 10^{-2}$ N/m (at 25°C) and $\theta = 20^{\circ}$ for Si covered with native oxide [11].

The trend of the measured adhesion force with respect to the tip radii (Fig. 4) seems to follow the equation (1), which indicates that the adhesion force is directly proportional to the tip radius (Fig. 5). Thus, tips with larger radii exhibit larger adhesion forces. Researchers in the past [6,12,13] have also observed similar relationship between the tip size and adhesion. Ando *et al.* [6], in their work, produced sub-micron size asperities of various radii by focused ion beam (FIB) method, and have measured the adhesion using AFM probe tip that had a flat square surface. Observations from their work showed that the adhesion force was directly proportional to the radius of an asperity and that the capillary force significantly influenced the adhesion force.

Friction at nanoscale

Fig. 6 (a) shows the variation of the measured friction force as a function of applied normal load, for the tips with various radii. The same plot has been redrawn as Fig. 6 (b) for the radii 0.32 μm and 1.25 μm, so that the trend could be seen more clearly. Both these plots show that the friction force increases linearly with the applied normal load. It is also evident that the friction force increases with the tip size. Insight into both these trends could be obtained by considering the fundamental law of friction given by Bowden and Tabor (1956) [14]. According to this law, the friction force is directly dependent on the real area of contact and is given by the expression:

$$F_f = \tau A_r \quad (2)$$

Where, τ is the shear strength, an interfacial property and A_r the real area of contact.

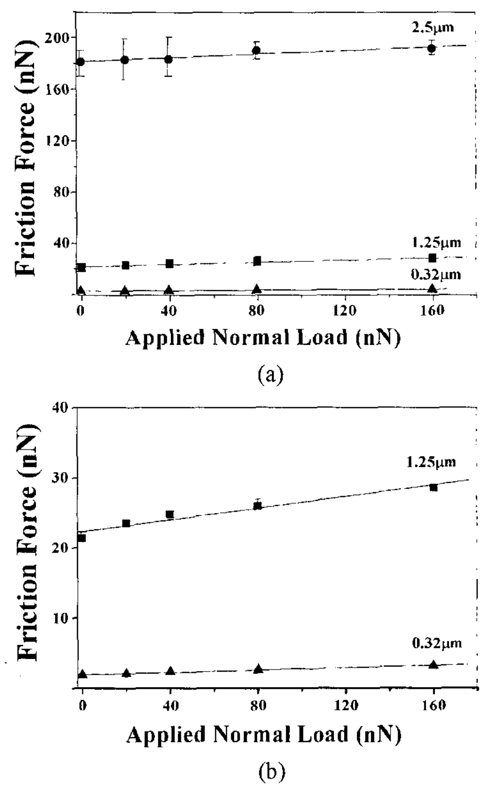

Fig. 6. Friction force measured using AFM as a function of applied normal load. (a) Tip radii 0.32 μm, 1.25 μm and 2.5 μm. (b) Tip radii 0.32 μm and 1.25 μm.

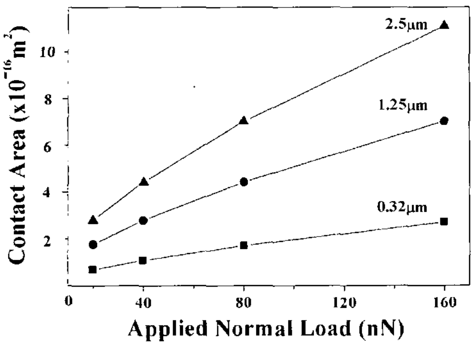

Fig. 7. Theoretical contact area estimated under Hertzian contact, for the tip sizes that were used in the present investigation.

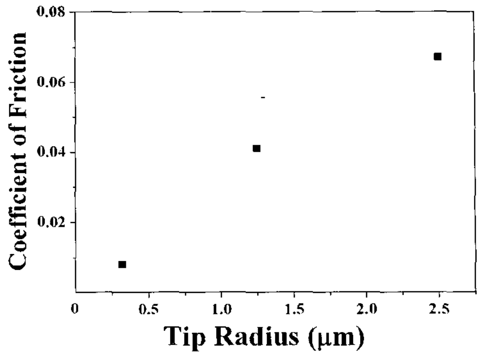

Fig. 8. Coefficient of friction calculated as the slope from Fig. 6 plotted against tip size.

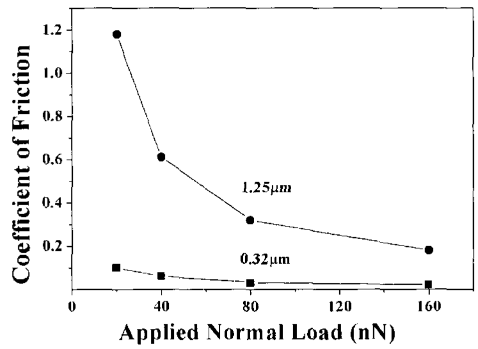

Fig. 9 Coefficient of friction calculated as the ratio between the friction force and applied normal load, as a function of applied normal load for tip radii 0.32 μm and 1.25 μm.

In order to understand the influence of the real area of contact on the friction force, we assume that the contact at the nanoscale to be a pseudo-single asperity contact and estimate the theoretical contact area under Hertzian contact [15]. The properties of materials used for these calculations are given in Table 1. Fig. 7 shows the estimated contact area as a function of applied normal load for various tip radii. From this figure it could be seen that the contact area increases with the applied load or/and the asperity tip radius.

From equation (2) it is clear that an increase in the contact area (affected by the applied normal load or/and the tip radius) would lead to an increase in the friction force. In the present work, the trend of the measured friction force seen in Fig. 6 is consistent with the friction law given in equation 2. This explains the increase in the friction force with the applied normal load or/and the asperity radius, which is due to the increase in the real area of contact. Such a relationship between the friction force and the contact area has also been reported by others [16-18]. In their work, the contact area was measured through the contact conductance experiments, which provided a direct way of determining the contact area. Although, the contact area under the influence of the adhesion force could be calculated theoretically using the JKR model [19], in the present work, we have calculated the contact area under Hertzian contact, as it would suffice for the understanding of the underlying relationship between the friction force and the contact area.

In Fig. 6 it could also be observed that the adhesion force influences the friction force. The presence of friction force at zero applied normal load is an evidence of the influence of the intrinsic adhesion force. An earlier work [20] has shown that the adhesion force is constant regardless of the applied normal load. Thus, it could be expected that the contribution of adhesion force to the friction force would also be constant even though the applied normal load increased, for the same tip size.

Coefficient of friction at nanoscale

Coefficient of friction was calculated as the slope, from the plot of friction force data versus the applied normal load (0-160 nN, Fig. 6). Estimating the coefficient of friction in such a manner eliminates the contribution of the adhesion force. Fig. 8 shows the variation of the coefficient of friction with the tip size. From this figure it is seen that the coefficient of friction increases with the tip size. As discussed earlier in section 3.2, the friction force is directly influenced by the tip size (via the contact area). Thus, the increase in the coefficient of friction with the tip size is mainly due to the increase in the friction force affected by the increase in the contact area. Bhushan *et al.* [21] have made similar observations of such a relationship between the coefficient of friction and the tip size.

Relationship between adhesion force and coefficient of friction

Coefficient of friction was also calculated as the ratio of the friction force to the applied normal load. Such a calculation would include the contribution of adhesion force. Fig. 9 shows the values of coefficient of friction (calculated as the above mentioned ratio) plotted against the applied normal load, for two different radii.

It is shown that the coefficient of friction decreases with an increase in the applied normal load. This could be explained as follows. As referred previously in section 3.2, the adhesion force inherently contributes to the friction force, but the contribution remains almost constant with the increase in the applied normal load [20]. The adhesion force acts as an additional normal load and increases the friction force. This in turn results at an increase in the coefficient of friction. Such an

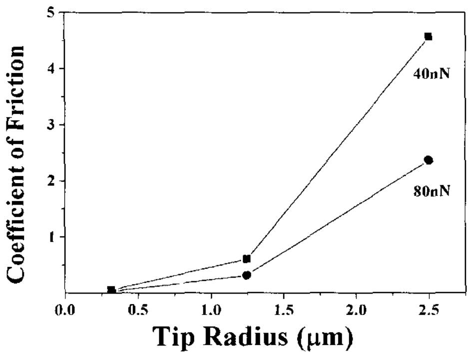

Fig. 10. Coefficient of friction estimated as the ratio between the friction force and applied normal load plotted against various tip radii (applied normal load 40 nN and 80 nN).

effect of the adhesion force is significant especially at lower applied normal loads. At higher applied normal loads, the influence of the adhesion force to the friction force becomes comparatively less than that by the applied normal load. This is seen in Fig. 9. Skinner and Gane [22] also reported such an influence of the adhesion force to the friction force. They measured the friction force and the attraction forces between the surfaces simultaneously, and showed that the friction force is proportional to the sum of the adhesion force and the normal load.

The coefficient of friction (calculated as the ratio) increased with the increase of the tip size. This is evident from Fig. 10, which shows the variation of the coefficient of friction as a function of various tip radii, for two different applied normal loads. As discussed previously in sections 3.1 and 3.2, the increase in the tip size increases the friction force due to the increase in the inherent adhesion force (Fig. 4) and the contact area (Fig. 7). This increase in the friction force in turn increases the coefficient of friction. Thus, it is seen that the there exists a direct relationship between the coefficient of friction and the tip size.

Conclusions

Adhesion and friction at nano-scale were studied with tips of various radii against Si-wafers using AFM. The test results are summarized below:

(1) Adhesion force increases with the tip size. The capillary force has a significant influence on adhesion.
(2) The contact area increases with the tip size, which results in the increase of the friction force.
(3) The coefficient of friction increases with the increase in the tip size.
(4) Adhesion and friction are both affected directly by the tip size.

Acknowledgments

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