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Wear Characteristics of Atomic Force Microscope Tip

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ABSTRACT

Atomic Force Microscope (AFM) has been widely used in micro/nano-scale studies and applications for the last few decades. In this work, wear characteristics of silicon-based AFM tip was investigated. AFM tip shape was observed using a high resolution SEM and the wear coefficient was approximately calculated based on Archard's wear equation. It was shown that the wear coefficient of Si and Si_3N_4 tips were in the range of 10^{-1}~10^{-3} and 10^{-3}~10^{-4}, respectively. Also, the effect of relative humidity and sliding distance on adhesion-induced tip wear was investigated. It was found that the tip wear has more severe for harder counter surface materials. Finally, the probable wear mechanism was analyzed from the adhesive and abrasive interaction point of view.

Key Words : Adhesion, AFM, Nano-wear, Si tip, Si_3N_4 tip,

1. Introduction

The various phenomena that occur at the micro/nano-scale have been actively researched since the advent of Atomic Force Microscope (AFM) [1]. Particularly, since AFM can be applied to probe into the tribological characteristics of materials at the nanoscopic level, it has been widely utilized to study various surface related properties [2-4]. Applications of AFM include Scanning Probe Lithography (SPL) where physical and chemical interactions between the tip and the counter surface are exploited to fabricate sub-micrometer level patterns [5-7]. AFM technology is also being used to develop ultra-high density information storage systems. The principle behind the so called probe recording technology using the AFM is based on the ability of the extremely sharp tip of the AFM to alter the media surface as to perform bit recording and subsequent reading [8].

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AFM tips are commonly fabricated by using semiconductor manufacturing processes using materials such as Si and Si_3N_4. For applications in scratching and indentation, sometimes the tips are coated with diamond to attain maximum hardness and prolonged durability. In other applications the tips may be coated with metals to attain electrical conductivity or specific magnetic properties. Such alterations of the tip are required for various surface modification processes using the AFM [9].

The geometry of the AFM tip directly affects the resolution of the measurement, and hence, sharp tips are generally preferred for nano-scale measurements. The radius of the tip is typically in the order of tens of nanometers. As the diameter of the tip decreases, the contact pressure between the tip and the surface increases for a given applied load. The pressures can be extremely high, enough to cause abrasive action to the surface as well as the tip itself. Whether by such abrasive action or other surface damage mechanisms, the issue of AFM tip wear poses a serious problem to the reliability of the AFM based systems. For example, insufficient life of AFM tip would limit the feasibility of probe recording technology [10,11].

As efforts to find means of reducing the wear of AFM tips, much research has been performed on this subject. However, most of the works are confined to observing the tip wear through experiments without adequate explanation on the mechanisms of tip wear [12]. In this work, the wear and surface damage characteristics of Si and Si_3N_4 AFM tips were investigated as they were slid against counter surfaces. The degree of wear was characterized by using Field Emission Scanning Electron Microscope (FESEM) and the adhesion force between the tip and the counter surface was measured to better understand the contribution of adhesion force to tip wear. The following sections describe the experimental work in detail.

2. Experimental Details

2.1 Specimens

In the experiments, commercial Si and Si_3N_4 AFM tips were used. The counter surfaces against which the tips were slid against were Si (100), Cu, and Diamond-Like-Carbon (DLC) films.

2.1.1 AFM tips

The Si and Si_3N_4 tips used in the experiments had conical and pyramidal geometry, respectively. The radius and stiffness for Si and Si_3N_4 tips were 10 nm, 0.26 N/m and 20 nm, 0.4 N/m, respectively. Following the contact sliding experiments, the tips and the surfaces were characterized for surface damage.

2.1.2 Counter surface

Si (100) counter surface was initially used to compare the wear behavior of Si and Si_3N_4 tips. Then, Si tip was used to compare the wear behavior of various counter surfaces including relatively ductile Cu film and hard DLC film. The Si (100) surface was cleaned with SCl solution prior to the experiments. It is expected that despite the cleaning procedure, about 1 to 2 nm of oxide film will exist on the surface. The Cu and DLC films were prepared by electron beam evaporation and DC sputtering method, respectively. The size of the counter surface specimens were 10×10 mm and the film thickness and surface roughness values are given in Table 1.

The surface roughness was measured using an AFM within a 5×5 μm^2 area and the hardness was measured using a nanoindentor. In order to avoid the effect of the substrate during the indentation measurement, the indentation depth should be kept to a minimum. The indentation depth monitored by the load-displacement curve indicated that the indentation depth was about 40 nm. The resulting hardness values from the indentation tests were found to be slightly higher than the previously reported values.

Table 1 Specification of counter surface materials

Material	Preparation	Thickness	Ra	Hardness
Si(100)	SC1 cleaning	~1 nm (chemical oxide)	0.13 nm	13.8 GPa
Cu	E-beam evaporation	180 nm	0.8 nm	3.2 GPa
DLC	DC sputter	300 nm	0.9 nm	12.4 GPa

2.2 Experimental method

A commercial AFM system was used for the experiments. The geometry of the tips before the sliding tests was recording using an SEM. The tip radius of the Si and Si_3N_4 tips were found to be 10 nm and 20 nm, respectively. The force was applied to the counter surface through contact with the tip as the tip was made to scan over a 5×5 μm^2 area. Following the scanning procedure, the same region was measured for topographical changes over a 10×10 μm^2 area at 20% of the initially applied load. 100 scans were performed to compare the wear behavior of Si and Si_3N_4 tips, and in other cases, 10 scans were performed. Each scan consisted of 256 lines, and therefore, the sliding distance of the tip per scan over the 5×5 μm^2 area in reciprocating motion was 5 μm × 2 × 256 = 2.56 mm. In the case of the Si tip, the applied force during scanning was 10 nN and 2 nN force was applied for the observation of the scanned region using the AFM mode. As for the Si_3N_4 tip, since its radius was larger than that of the Si tip, a higher load of 20 nN was applied in order to maintain a similar level of contact pressure. Then, 4 nN applied force was used in the AFM mode to observe the scanned region.

Hertzian contact analysis was performed to estimate the contact pressure between the tip and the counter surface. The maximum contact pressure was calculated to be 3.9 GPa and 4.1 GPa for Si (100) and Si_3N_4 tip, respectively. FESEM images of the tips after the scanning experiments were used to assess the wear volume of the tip region. The wear volume was then used in the Archard's wear equation to obtain the wear coefficients.

In order to verify the effect of adhesion on the tip wear behavior, different levels (20, 40, and 60%) of relative humidity were used. For Si (100), Cu and DLC coated specimens the tip was observed for wear after 10 scans over an area of 5×5 μm^2. Also, the adhesion between the tip and the surface was measured at intervals during the scanning process by obtaining the force-distance (F-D curve). In experiments other than the humidity effect tests, the relative humidity was set to approximately 20% and the scanning speed was set to 5 μm/s.

3. Experimental Result

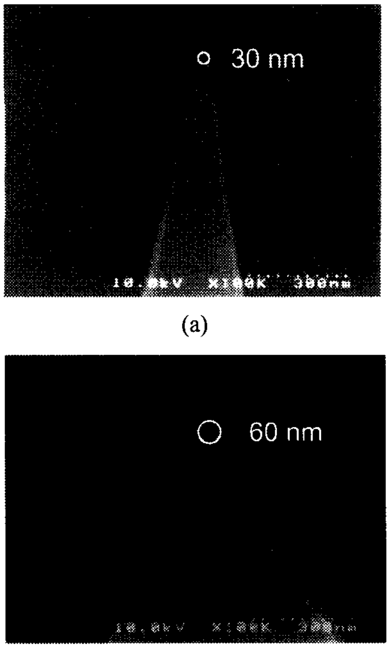
3.1 Wear of AFM tip

Wear coefficients for Si and Si_3N_4 tips were obtained and the effects of humidity and counter surface material on the wear of the Si tip were investigated.

3.1.1 Wear coefficient of AFM tip

Fig. 1 shows the FESEM images of the Si and Si_3N_4 tips before the experiment. The previously mentioned radius for each tip can be verified from the images. Fig. 2 shows the tips after experiencing 100 scans against an Si (100) counter surface over a 5×5 μm^2 area. The total sliding distance of the tip during this scan was 256 mm. It is evident that the tips have been flattened due to the contact sliding interaction and the degree of damage of the Si tip was more severe than the Si_3N_4 tip. The wear coefficients for the Si and Si_3N_4 tips were calculated to be in the range of 10^{-1}~10^{-3} and 10^{-3}~10^{-4}, respectively. Hence, the wear resistance of Si_3N_4 tip was found to be superior than the Si tip under similar contact pressure conditions. The magnitude of the wear coefficient is quite high, even compared with the macroscopic scale, and this corresponds to a severe wear situation.

Fig. 3 shows the top image of the tip shown in Fig. 2(a). It can be seen that for both tips, wear debris could be found along the flattened tip region. However, the degree of wear debris amount is less for the Si_3N_4 tip compared with the Si tip. This observation is attributed to the difference in the adhesive interaction between the produced debris and the respective tips due to the difference in their surface energies. As the particle size gets smaller, the tendency for them to adhere and agglomerate will increase due to increasing surface force effects. The wear debris distribution behavior is known to have a significant effect on the wear characteristics of contact sliding systems.



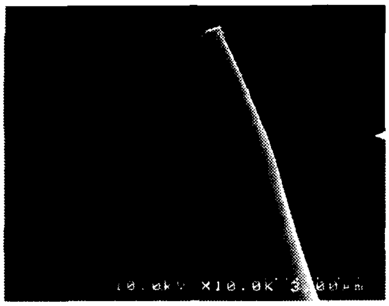
(a)

(b)

Fig. 1 FESEM images of (a) Si and (b) Si_3N_4 AFM tip before experiment (side view).

3.1.2 Effect of humidity on tip wear

The variation in the adhesion force with respect to the relative humidity (RH) was obtained by the F-D curve measurements and the degree of tip wear for various humidity levels is shown in Fig. 4. The counter surface for this experiment was Si (100) and the total sliding distance was 25.6 mm. As expected, the adhesion and wear increased with increasing humidity. As the humidity increases, the probability of water adsorption on the surface increases, and therefore, adhesion due to the meniscus effect can be profound. For RH of 40 % or more, the adhesion force is about 15 nN, which is higher than the applied normal force of 10 nN. Especially for 60 % RH, the deviation in the adhesion force increased significantly.



(a)

(b)

Fig. 2 FESEM images of (a) Si (under 10 nN) and (b) Si_3N_4 (under 20 nN) AFM tip after 256 mm sliding (side view) at 20 % RH.

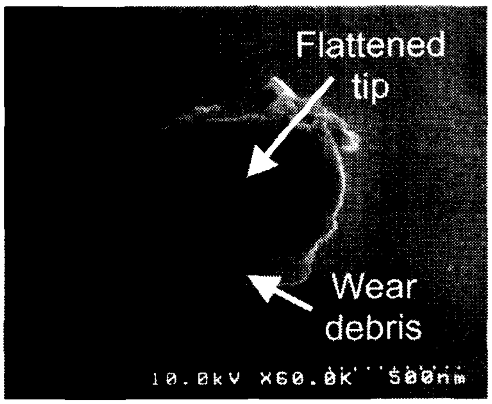


Fig. 3 FESEM image of worn Si tip under 10 nN after 256 mm sliding (top view) at 20 % RH.

DMT and JKR models were applied in calculating the effective maximum contact pressures for 40% and 60% RHs. The resulting value was 5.5 GPa, which is about 40% higher than the value estimated by the Hertzian equation. Therefore, it should be noted that the adhesion between the AFM tip and the counter surface can increase by more than a factor of two depending on the RH level. In general, it seems reasonable to state that tip wear increases with increasing humidity.

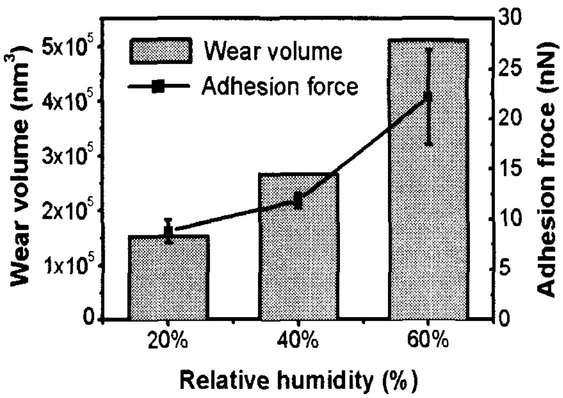


Fig. 4 Wear volume and adhesion force of Si tip w.r.t. relative humidity.

3.1.3 Effect of counter surface material on tip wear

The effect of counter surface material on the wear behavior of tip was investigated using Si (100), Cu and DLC coated specimens. The AFM tips were scanned for 10 times over an area of 5×5 μm^2. The tips were then observed using an FESEM as shown in Fig. 5. Compared with the case of Cu coated specimen, the tips experienced greater amount of wear when they were slid against Si (100) and DLC coated specimens. Relating to the data given in Table 1, it can be seen that the wear of the tip increased as the hardness of the counter surface increased.

3.2 Surface damage of the counter surface

Attempts to assess the degree of surface damage incurred to the counter surface as a result of scanning with the AFM tip was not quite satisfactory when observed with the AFM. However, Lateral Force Microscopy (LFM) revealed more information in this regard. LFM images were thus obtained over an area of 10×10 μm^2 which includes the 5×5 μm^2 scanned region. Fig. 6 shows the LFM image of the Si (100) surface after being scanned with an Si tip over a 5×5 μm^2 area. As can be seen, there is a clear distinction in the LFM signal within and outside of the scanned region. However, the degree of surface damage could not be identified using the AFM mode since the wear amount was within the dimensions of the surface roughness.

4. Discussions

According to previous researches, the wear mechanisms of AFM tip are adhesive wear, abrasive wear, and low cycle fatigue wear [13]. Based on the experimental findings of this work, the adhesive and abrasive AFM tip wear mechanisms were further analyzed.

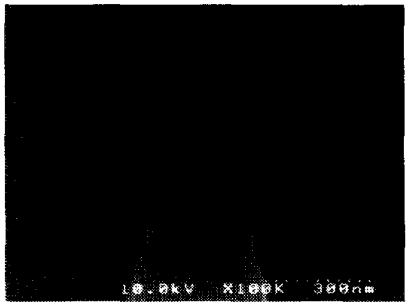
4.1 Effect of adhesive wear

In order to assess the degree of adhesion force contribution to the tip wear, the pull-off force was investigated for various material combinations. The pull-off force obtained from the F-D curve for Si and Si_3N_4 tips contacting against the Si (100) surface was found to be 8.8 ± 1.5 nN and 12 ± 1.8 nN, respectively. Since the tip radius of the Si_3N_4 tip was twice that of the Si tip, it was expected that the adhesion force of the Si_3N_4 tip would be greater. It turned out that the adhesion force was about 1.3 times greater than that of the Si tip, which does not scale with the difference in the contact area of the two tips. This is because the surface energy of Si_3N_4 is less than that of Si.

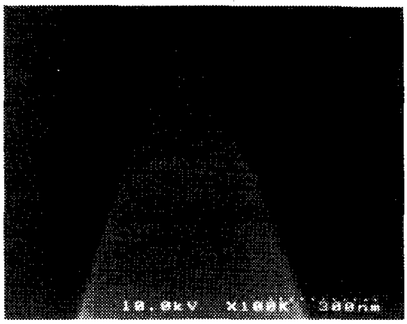
Fig. 7 shows the adhesion force between the Si tip and Si (100) surface obtained using the F-D curve at intervals during the 10 repeated scans. As the sliding distance increased, the adhesion force increased by about two folds. This increase is probably due to the increase in the probability of asperity interaction as the tip gets flattens and allows more asperities to interact against each other.

Fig. 8 shows the adhesive force before the sliding experiment. It shows that the adhesion of Cu is the highest, its value being 10 nN. However, the wear of AFM tips slid against Si (100) and DLC film was higher than when they were slid against the Cu film. Thus, it may be deduced that the wear is affected more by the hardness of the counter surface rather than the adhesion force between the tip and the surface.

(a)

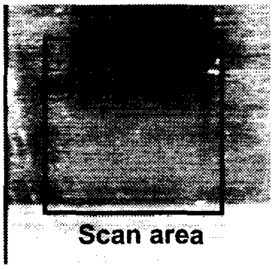


(b)



(c)

Fig. 5 FESEM images of AFM tip after 25.6mm sliding on (a) bare Si(100), (b) Cu, and (c) DLC surface at 20 % RH.



(a) (b)

Fig. 6 (a) Left to right and (b) right to left LFM images of Si (100) surface after 256 mm contact sliding with Si tip under 10 nN at 20 % RH.

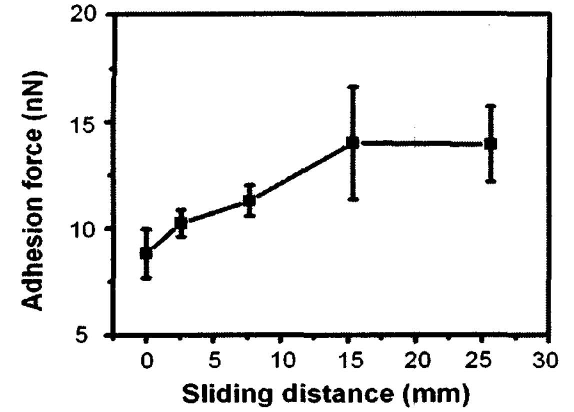


Fig. 7 Adhesion force change between Si tip and Si(100) surface w.r.t sliding distance.

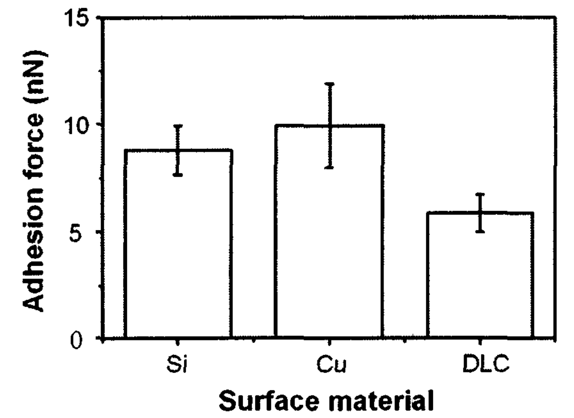


Fig. 8 Adhesion force between Si tip and Si (100)/Cu/ DLC surface at 20 % RH.

4.2 Effect of abrasive wear

From the abrasion point of view, where hardness plays a critical role, the Si_3N_4 tip is expected to wear less than the Si tip since its hardness is about twice that of Si. Indeed, it was found that the wear coefficient of Si_3N_4 tip was 1% to 10% that of the Si tip. The evidence of abrasive interaction between the tip and the counter surface suggests that plastic deformation occurs due to the sliding action. Based on the Hertzian analysis, the contact pressure was about 4 GPa and the hardness of Si and Si_3N_4 is about 10 GPa and 20 GPa, respectively [14]. It is generally known that plastic deformation in the surface region occurs when the maximum contact pressure reaches about 60% of the hardness [15]. Therefore, simply based on the Hertzian pressure, it is difficult to predict plastic deformation of the tip as well as the surface. A plausible explanation for this outcome is that due to the surface asperities of the tip and the counter surface, the local contact pressures can be much higher than the value predicted by the Hertzian contact for a smooth surface.

As for the Cu film, despite its high adhesion force, the resulting wear was less than the other two surfaces. This is indicative of the fact that relatively low hardness of the Cu film was responsible for the high wear. This supports the view that abrasive wear mechanism is dominant over the adhesive wear mechanism for AFM tip/surface sliding interaction.

5. Conclusions

The wear behavior of Si and Si_3N_4 AFM tips sliding against Si (100), Cu and DLC films under tens of `nN applied force was investigated. Based on the experimental findings, the following conclusions may be drawn:

1). The range of wear coefficients of Si and Si_3N_4 tip is 10^{-1}~10^{-3} and 10^{-3}~10^{-4}, respectively.

2). The AFM tip damage increases as the relative humidity increases to 60 %. The adhesion force increases by three times at this humidity level.

3). The hardness of the counter surface has more effect on the AFM tip wear compared with the adhesion force between the tip and the surface.

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