Surface and Coatings Technology 128–129 (2000) 341–345

SURFACE & COATINGS TECHNOLOGY

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Investigation on microtribological behavior of thin films using friction force microscopy

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Abstract

A purpose-built atomic force and friction force microscope (AFM/FFM) was employed to study micro friction and wear behavior of thin films of Langmiur–Blodgett (L–B), gold (Au), polytetrafluoroethylene (PTFE), silicon nitride (Si_3N_4) and PTFE/Si_3N_4 multi-layers prepared by different deposition techniques. The results show that the L–B film has a low friction coefficient but can be worn easily under a light load less than 20 nN; Au film and silicon wafer has a higher friction coefficient than that of L–B film and can be worn under a higher load of approximately 50 nN; the PTFE/Si_3N_4 multilayer has a lower friction coefficient than that of Si_3N_4 and has a higher micro wear resistance than that of PTFE. The friction force of the PTFE/Si_3N_4 multilayer is linear with the load at the nanometer scale. The worn track is formed in PTFE film and PTFE/Si_3N_4 multilayers when the load is greater than 70 nN. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Friction force microscope; Thin films; Microtribology; Micro friction and wear

1. Introduction

In recent years, microtribology has become a foremost area in tribology. It is an important technology when developing new microdevices, and it is also an essential science in order to understand the origin of friction and wear [1,2].

Although the traditional theories of lubrication, friction and wear, which have been developed for over 100 years, formed the important basis for general engineering design and material development, there are still many problems left unresolved when the theories are applied to tribological micromechanisms and active controlling of lubrication, friction and wear. Especially, the theories of macro-tribology are no longer suitable for explaining tribological behavior and mechanisms which occur in micromachine or super precision instrument because of the ultra light load and nanometer scale. As a result, nanotribology [3,4] has emerged as an essential research area by modern science and technology.

As it is known, many kinds of contact surfaces with relative movement consist of thin films deposited on bulk materials, especially in micromachines or super precision instruments. However, the properties of thin films are often different from that of bulk materials. Therefore, it is very difficult to use the traditional experimental methods and theories to study and explain the tribological behavior of these thin films for special use.

In order to study micro friction and wear, scientists have developed friction force microscope (FFM) which serves as an excellent tool in microtribological research [1,5–7]. However, one's research results often do not have the comparability with others' because of the different experimental conditions. Hence, more re-

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0257-8972/00/$ - see front matter
PII: S0257-8972(00)00602-2

search should be done in this area. In this paper, a purpose-built FFM was used to study micro friction and wear of some typical thin films.

2. Experimental procedures

2.1. Samples preparation

Four kinds of films are prepared for the experiment:

1. A single Langmuir trough rinsed with deionized distilled water was employed in the preparation of L–B films. Two layers of L–B film consisting of triacetic acid and $CrCl_2$ were prepared on Si (100) substrate which was treated with hydrophilic solution and then thoroughly cleaned before the L–B film making.
2. Pure gold film was prepared in a vacuum sputtering apparatus. The substrate is Si (100) wafer. The film thickness is approximately 800 nm.
3. The PTFE/Si_3N_4 multilayers were prepared by Ar^+ ion beam alternative sputtering pure PTFE and Si_3N_4 ceramic target in a polyfunctional beam assisted deposition system. The Si (100) wafer is the substrate. The mutlilayer has 11 layers with an alternative PTFE and Si_3N_4 layer, the innermost and outermost layers were Si_3N_4 films. According to the deposition rate of PTFE and Si_3N_4, the sputtering time was 5 min for each PTFE layer, and 10 min for each Si_3N_4. The thickness of each PTFE layer was approximately 80 nm which was almost the same as that of the Si_3N_4 layer.
4. Pure PTFE and pure Si_3N_4 films were also prepared using the same sputtering parameters. The sputtering time was 1 h for pure PTFE film, and 2 h for Si_3N_4. The film thickness of pure PTFE and pure Si_3N_4 was almost equal to that of PTFE/Si_3N_4 multilayers. It has been shown by Wang et al. [8] that the PTFE in the two kinds of samples is in the crystalline state.

2.2. Test procedures

The micro friction and wear behaviors were carried in an atomic force microscope and friction force microscope developed by the authors' laboratory [9] under ambient conditions. The spring constant of Si_3N_4 cantilever used in this research was 0.06 N/m (A) and 0.38 N/m (B), respectively. The curvature radius of the Si_3N_4 tip was approximately 50 nm. The scanning direction of the tip is shown in Fig. 1.

Cantilever A was used during the micro friction test. The surface morphology as well as friction force image were obtained by scanning along the Y and −Y direction simultaneously in constant height mode. The normal force and friction signals were, respectively, recorded by upper and lower, left and right segments of the quadrant position sensitive diode. The real friction force image was calculated by the following equations [7,10]:

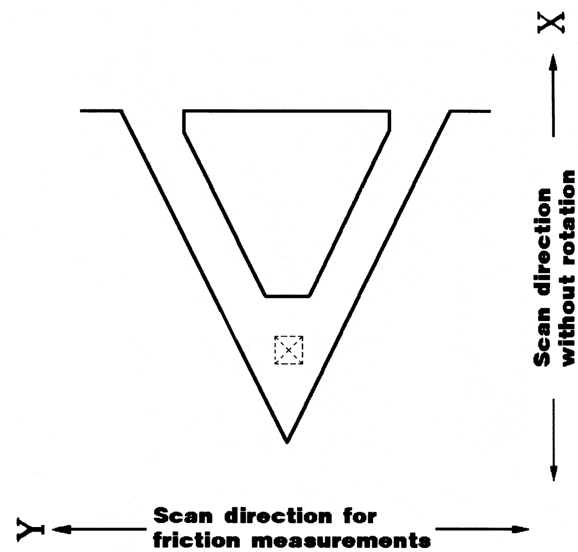

Fig. 1. A schematic diagram of the scanning direction of the probe of FFM.

Y direction,

$$F_Y(i,j) = f_0(i,j) + f(i,j) \tag{1}$$

− Y direction,

$$F_{-Y}(i,j) = f_0(i,j) - f(i,j) \tag{2}$$

Therefore,

$$f(i,j) = (F_Y(i,j) - F_{-Y}(i,j))/2 \tag{3}$$

Here, $F_Y(i,j)$, $F_{-Y}(i,j)$ is the signal of friction force obtained by scanning along the direction of Y and −Y, respectively. $f(i,j)$ is the signal of real friction force. $f_0(i,j)$ is the signal produced by other lateral forces except friction force. Hence, the average of $f(i,j)$ is

$$f = \frac{1}{N^2} \sum_{i=0,j=0}^{N-1} f(i,j) \tag{4}$$

Where N is the number of acquisition data points along the X and Y direction.

Cantilever B was used during the micro wear test. Firstly the probe scanned for a set number of times in an area along the X direction, and then the worn surface morphology was measured in a larger area. The worn depth can be calculated by measuring the difference between the worn area and the initial unworn area.

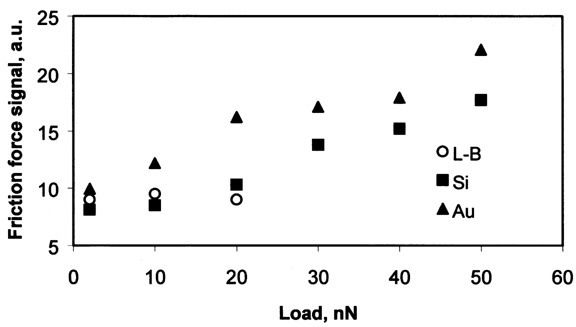

Fig. 2. Dependence of friction force signal of the L–B film, Au film and Si wafer on load.

3. Results

3.1. Characteristics of micro friction

Fig. 2 shows the dependence of the micro friction force signal of L–B film, Au film and Si wafer on load. It can be observed that the micro friction force increases with load except that of the L–B film which remains almost the same. The L–B film was worn by the tip under the load less than 20 nN because the L-B film consisted of organic molecules and was soft. Fig. 3 shows the effect of load on the micro friction force signal of pure PTFE, Si_3N_4 and PTFE/Si_3N_4 multilayers. It can be seen that the micro friction force signals of Si_3N_4 film and PTFE/Si_3N_4 multilayers vary almost lineally with the load. As for pure PTFE film, when the load is less than 70 nN, the micro friction force signal increases lineally with the load. When the load is greater than 70 nN, the micro friction force signal doesn't increase with the load, the friction force is more or less constant.

As it is known, during the micro friction force measurement, it is difficult to obtain the real and precise friction force due to the small size of cantilever as well as the slight difference between cantilevers. According to the principle of FFM, the friction force signal can be used as the representative of the real friction force. Therefore, a friction coefficient factor is introduced in order to compare the relative micro friction characteristics among the pure PTFE, pure Si_3N_4 and PTFE/Si_3N_4 film. Corresponding to the relationship between the friction force and friction force signal, the slope of the micro friction force signal vs. load is referred to as the friction coefficient factor, which is the equivalent of the friction coefficient, under the micro friction test [10]. From Fig. 3, it can be calculated that the friction coefficient factor of pure PTFE film is approximately 0.057 which is very small, the friction coefficient factor of PTFE/Si_3N_4 multilayers is approximately 0.115 which is located between the pure PTFE and Si_3N_4 film whose friction coefficient factor is approximately 0.171.

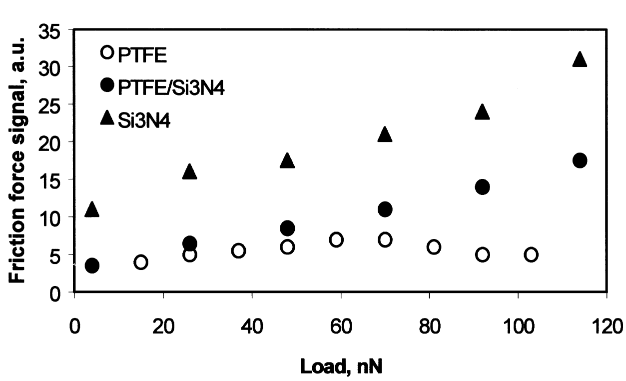

Fig. 3. Dependence of friction force of PTFE, Si_3N_4 and PTFE/Si_3N_4 film on load.

3.2. Micro wear

Fig. 4 shows micro wear scars of the L–B film. The load is light, but the wear scar is large. The depth of wear scar can be obtained according to the cross-section of the worn area. Fig. 5 shows the morphology of Au film on Si wafer after micro wear test under 50 nN. There is also a relative deep wear scar on the film. Fig. 6 shows the micro wear scar on Si wafer after micro wear test. The wear scar is shallow even under the load of 110 nN. It is obvious that the Si wafer gives good wear resistance under such experimental conditions.

For PTFE film and PTFE/Si_3N_4 multilayers, through the observation of the surface morphology after the micro wear test, an obvious worn mark and a projection in the edge of the worn marks was found at the load above 70 nN (Figs. 7 and 8). For the Si_3N_4 film, there was no worn mark observed even when the maximum load was used in the micro friction test.

The dependence of worn depth of PTFE and

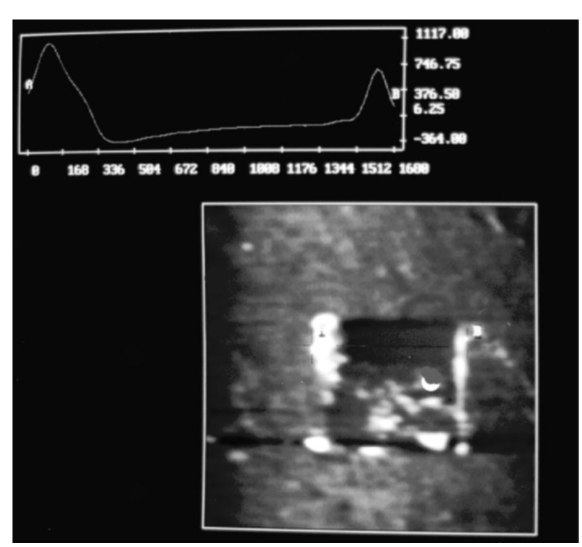

Fig. 4. Morphology of the L–B film after micro wear (20 nN, 10 cycles, scanning area 6 × 6 μm).

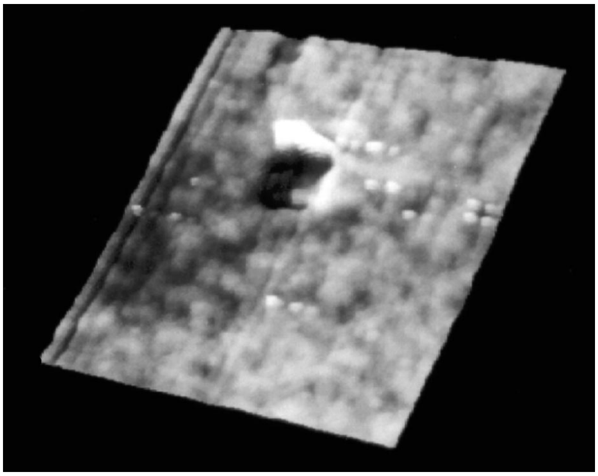

Fig. 5. Morphology of Au film on Si wafer after micro wear (50 nN, 20 cycles, scanning area 2×2 μm)

$PTFE/Si_3N_4$ films on the load is shown in Fig. 9. The worn depth of both PTFE and $PTFE/Si_3N_4$ film is in the nanometer scale. It can be seen that the worn depth increases linearly with load. However, the worn depth of $PTFE/Si_3N_4$ multilayers is approximately one-tenth of PTFE film at the same load. All these results demonstrate that the wear resistance is greatly improved by the micro-assembling of soft and hard layers.

4. Discussion

The L–B film studied in this report consists of two layers of organic molecules. The first layer of the LB film is adsorbed on silicon substrate by the polarization

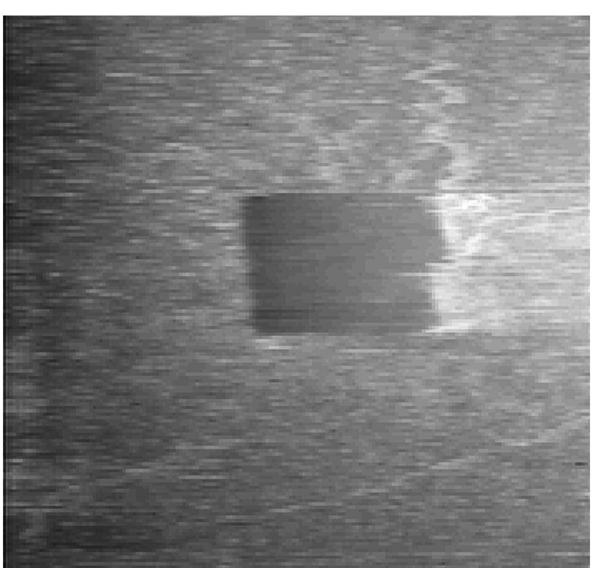

Fig. 6. Morphology of Si wafer after micro wear (110 nN, 50 cycles, scanning area 2×2 μm).

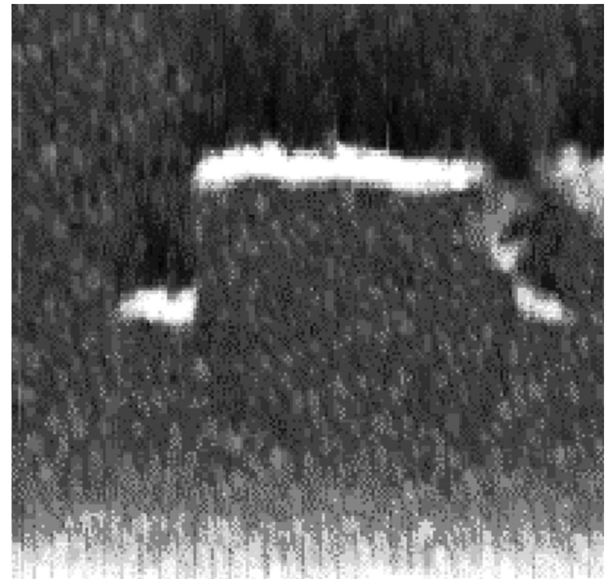

Fig. 7. Morphology of $PTFE/Si_3N_4$ film after 116 nN load and 10 cycles scanning (scanning area 2×2 μm).

terminals of the molecules. During the micro friction test, the probe contacted with the polarization terminal of the second layer. As a result, there was a special attractive force between the polarization terminal of the second layer and the probe. So, it is not strange that the L–B film does not have the function of reducing friction force under the current experimental conditions. Because the adsorption force between the terminal and the substrate was limited, the L–B film was seriously worn under a load of 20 nN.

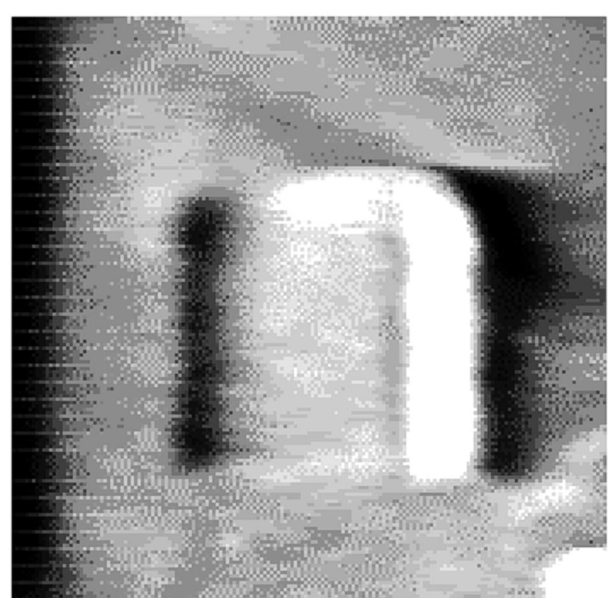

Fig. 8. Worn morphology of PTFE under 110 nN load and 50 cycles scanning (scanning area 2×2 μm).

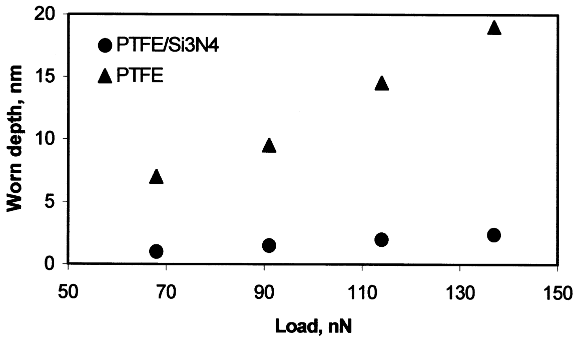

Fig. 9. Dependence of worn depth of PTFE and PTFE/Si_3N_4 film on load (20 cycles, scanning area 2 × 2 μm).

Because Au is soft and has a lower shear strength than silicon, it is normal that Au film has a higher micro friction coefficient factor than silicon, and can be easily worn under the current experimental conditions.

Because PTFE has low adhesion, high lubricity and low a friction coefficient [11], it is not difficult to understand why PTFE film has a lower friction coefficient factor and higher wear rate than PTFE/Si_3N_4 multilayers under the current experimental conditions. For PTFE/Si_3N_4, because a little PTFE was exposed on the surface of multilayers, the friction coefficient factor was smaller than Si_3N_4 but larger than PTFE, and the worn mark was found on the PTFE/Si_3N_4 multilayers.

During the friction and wear tests of PTFE film, two zones were classified according to the load. Under the load below 70 nN, the friction force which created in friction and wear tests is too small to make the PTFE film shear. Within this zone the friction force increases linearly with the load, and there are no transfer of molecules and no worn marks. The second zone is under the load above 70 nN. The friction force created in the friction and wear tests is large enough to force the PTFE molecules to slip. So there were obvious worn mark and projection in the film, and the friction force stayed almost constant with load.

Through micro-assembling of PTFE and Si_3N_4 layers, the PTFE layer in PTFE/Si_3N_4 multilayers is located between two Si_3N_4 layers. Because of the interaction between PTFE and Si_3N_4 films, the mobility of the PTFE film is limited. The PTFE molecules cannot slip easily, therefore the shear stress required to slip PTFE increases, so the multilayer film has high wear resistance.

5. Conclusions

The L–B film consisting of triacetic acid and $CrCl_2$ can be worn easily under a light load less than 20 nN, it does not have the function of reducing friction force under the current experimental conditions. Au film can be worn under a high load, but Si wafer is difficult to be worn under the experimental conditions.

There are two zones in the friction and wear tests of PTFE film. When the load was less than 70 nN, the micro friction force increased with the load. When the load was greater than 70 nN, the friction force of PTFE film was almost constant, and there was an obvious worn mark in the PTFE film.

PTFE/Si_3N_4 multilayers not only have as low a friction coefficient as PTFE but also have as high a wear resistance as Si_3N_4.

Acknowledgements

The authors would like to acknowledge the support of the National Natural Science Foundation of China (No. 59805010).

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