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# Micro/nano Tribological and Water Wetting Characteristics of Ion Beam Treated PTFE Surfaces

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**Abstract: Micro/nano tribological and water wetting characteristics of ion beam treated PTFE (polytetrafluoroethylene) surfaces were experimentally studied. The ion beam treatment was performed with a hollow cathode ion gun at different argon ion dose conditions in a vacuum chamber to modify the topography of PTFE surface. Micro/nano tribological characteristics, water wetting angles and roughness were measured with a micro tribo tester, SPM (scanning probe microscope), contact anglemeter and profilometer, respectively. Results showed that surface roughness increased with the argon ion dose. Water wetting angle of the ion beam treated samples increased with the ion dose, so the surface shows an ultra-hydrophobic nature. Micro-adhesion and micro-friction depend on the wetting characteristics of the PTFE samples. However, nano-tribological characteristics showed different results. The scale effect of surface topography on tribological characteristics was discussed. Also, the water wetting characteristics of modified PTFE samples were discussed in terms of the surface topographic characteristics.**

**Keywords :** Nano, micro, adhesion, friction, hydrophobic film, tribology, SPM

## Introduction

The topographical changes and chemical modifications of engineering surfaces are newly emerged as a representative research direction to solve the adhesion problems occurring at MEMS (microelectromechanical system) elements [1]. The research works on the topographical changes of surfaces, such as laser texturing and micro dimple formation, are based on the principle of the reduction in real contact area [2]. Laser-textured bumpers of HDD (hard disk drive) [3] and dimples at the comb drive surfaces [4] are the typical examples of applications.

Recently many researchers also focused on the effect of surface topography in order to achieve the mimetic characteristics of natural leaves and the tissue of animals [5]. Lotus effect [6] is one of the research works for achieving the mimetic surfaces. The Lotus effect is explained by the characteristics of microstructure pattern of unique surface roughness as well as the hydrophobic properties of the epicuticular waxes. Therefore, adhesion of water or particles onto the surface is extremely reduced. Wolter *et al.* [6] characterized the water-repellent and self-cleaning plant surfaces. Watanabe *et al.* [7,8] reported ultra-hydrophobic nature of a polymeric thin film that was prepared combining a phase separation of tetraethyl orthosilicate induced by the addition of an acrylic polymer and subsequent fluoroalkylsilane coating.

However, complex chemical process is needed to achieve such a hydrophobic nature surface. The tribological properties have not been clearly reported yet.

In this work, the effects of topography on wetting, adhesion and friction were studied experimentally. PTFE (polytetrafluoroethylene) samples which have different topography were fabricated by the bombardment of accelerated argon ion beam, and their wetting and tribological characteristics were evaluated. In order to study the scaling effect of the topography on the tribological characteristics, micro- and nano-scale tribo tests were performed with a micro-scale tribometer and a SPM (scanning probe microscope).

## Experimental Details

### Specimens

PTFE samples modified by an accelerated argon ion beam were used to study the effects of topography on wetting and tribological characteristics. Fig. 1 shows the schematic diagram of an ion beam assisted surface modification device. The main concept of PTFE surface modification using an ion beam treatment was shown in Fig. 2. PTFE molecules are known to have the partial crystalline structure [9]. The grain boundaries of a relatively low bonding strength could be etched out more rapidly than other parts by the accelerated argon ion beam. As a result, it could be possible to fabricate PTFE surfaces having different surface topography in relation with the argon ion dose, as illustrated in Fig. 2. The conditions of the surface modification of PTFE were summarized in Table 1.

### Test apparatus and methods

Nano-adhesion and nano-friction tests were performed with a

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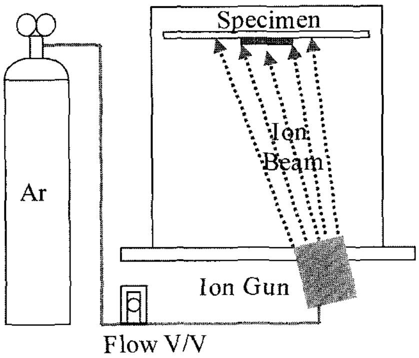



**Fig. 1. A schematic diagram of an ion beam assisted surface modification device.**

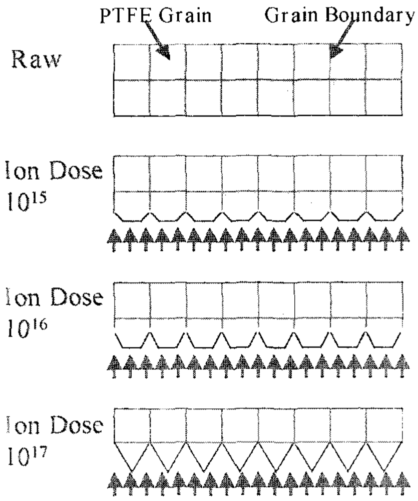



**Fig. 2. A schematic illustration on the surface roughening method of PTFE samples using the argon ion bombardment.**

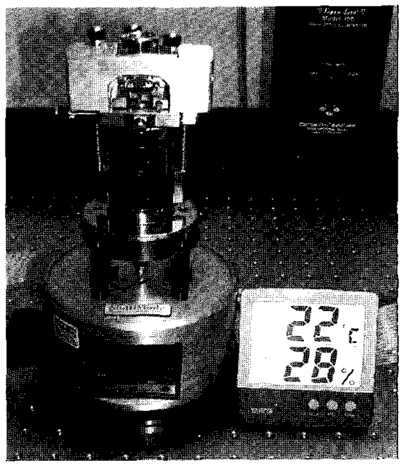


**Fig. 3. Scanning probe microscope system.**

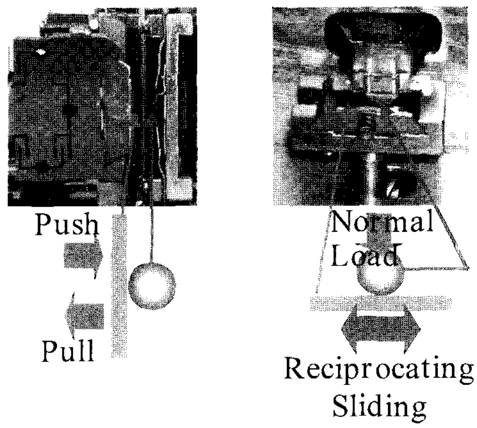



**Fig. 4. Close-up view of (a) micro-adhesiometer and (b) micro-tribometer.**

commercial SPM system (Multimode, NanoScope IIIa, Digital Instruments), as shown in Fig. 3. Oxide sharpened $Si_3N_4$ tips with the nominal radius of 15 nm mounted on a triangular $Si_3N_4$ cantilever of nominal spring constant of 0.58 N/m were used. Surface topographies were measured using tapping mode AFM (atomic force microscope). Adhesion measurements were performed using a force-displacement curve in contact mode AFM [10-12]. The measured maximum pull-off force was defined as the adhesion force in this work. Friction force was measured using LFM (lateral force microscope) [12].

**Table 1. Characteristics of samples**

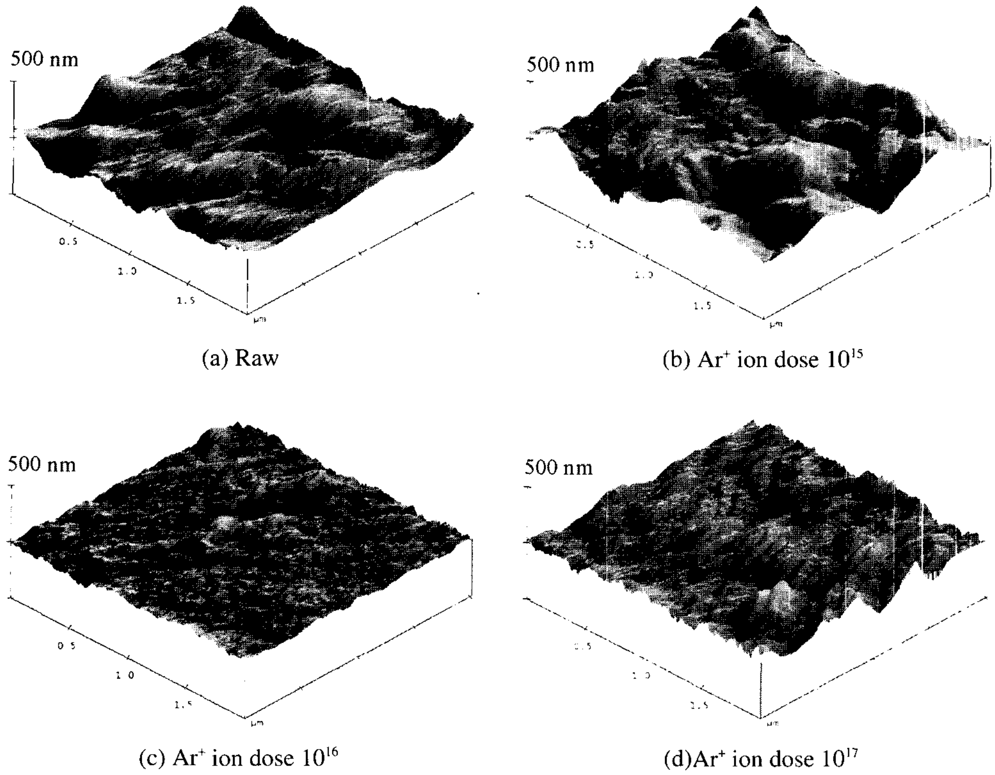
| Specimens | Surface Treatments | Wetting Angle (degree) |
|---|---|---|
| PTFE Plate | Raw | 105 |
| | $Ar^+$ ion dose $10^{15}$ | 119 |
| | $Ar^+$ ion dose $10^{16}$ | 138 |
| | $Ar^+$ ion dose $10^{17}$ | 160 |
| SPM Tip | $Si_3N_4$, Tip radius = 15 nm | |
| Ball | $Si_3N_4$, $\pi$1 mm | |

Tests on nano-adhesion and nano-friction were performed with an applied normal load of 40 nN and the scanning speed of 4 $\mu$m/sec. Every test was repeated more than 25 times at different locations.

The adhesion forces in micro-scale were measured with a ball-on-flat type micro-adhesiometer (refer to Fig. 4(a)). Pull-off force between a $Si_3N_4$ ball (1 mm in diameter) and the PTFE samples were measured under the load of 100 $\mu$N. The same tests were repeated more than 30 times and the average pull-off force was defined as the adhesion force.

Micro-friction tests were performed with a ball-on-flat type micro-tribometer under a reciprocating motion. The close-up view of the micro-tribometer is shown in Fig. 4(b). Friction force between a $Si_3N_4$ ball (1 mm in diameter) and PTFE samples were measured under the load of 100 $\mu$N and the sliding speed of 1 mm/sec. The micro-friction tests were repeated more than three times and the average value was used as the friction force.

All of tests were performed under an ambient condition of 20~24°C and 27-33 % RH.




(a) Raw (b) $Ar^+$ ion dose $10^{15}$

(c) $Ar^+$ ion dose $10^{16}$ (d) $Ar^+$ ion dose $10^{17}$

**Fig. 5. 3D topography of the PTFE samples.**

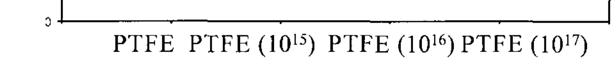



**Fig. 6. Water wetting angles of the PTFE samples.**

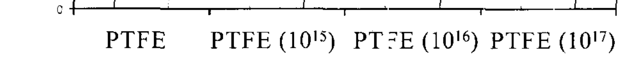



**Fig. 7. Surface roughness (Ra) vs. argon ion dose of PTFE samples.**

## Results and discussions

Surface topographies of the ion beam treated PTFE samples were shown in Fig. 5. The shapes of surface asperities were changed from dull to sharp with the increase in argon ion dose. The water wetting angles (refer to Fig. 6) increased with the argon ion dose and ultra-hydrophobic nature (wetting angle $>160^{\circ}$) was observed when the argon ion dose was over $10^{17}$.

In order to study the effect of argon ion bombardment on micro-scale surface roughness of PTFE samples, the micro-roughness of the four PTFE samples was measured with a profilometer (Talysurf, Taylor Hopson Co.) and the results were shown in Fig. 7. The micro-scale surface roughness Ra (cut-off length of 0.8 mm) increased with the ion dose and the water wetting angle increased with the ion dose (Fig. 6). It could be suggested that the changes in the surface roughness is closely related to the water wetting characteristics of ion beam modified PTFE samples.

Fig. 8 shows nano-scale surface profiles of the PTFE samples using SPM. The nano-scale surface roughness Ra (cut-off length: 2 $\mu m$) did not reveal any close relationship with the argon ion dose. But, the nano-scale sharpness of the asperities of PTFE samples increased with the argon ion dose as shown in Fig. 8. Therefore, it was found that the water wetting angle was affected not only by the surface chemical characteristics but also by the sharpness of asperities. This result could be explained by the Lotus effect which results in the ultra-hydrophobicity by sharp surface protuberances. The effect of the asperity sharpness on water wetting characteristic was greatly noticeable and the ultra hydrophobic characteristic (wetting angle $>160^{\circ}$) was found when the argon ion dose was

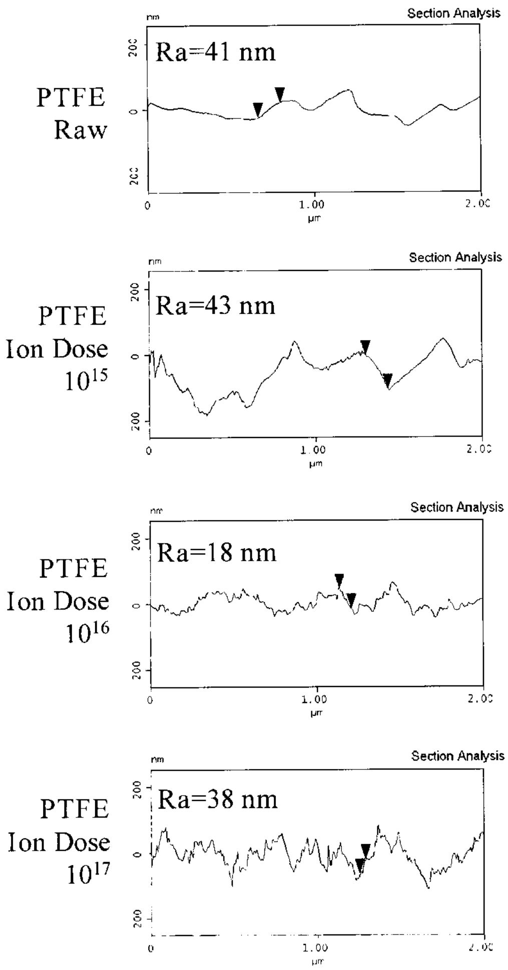
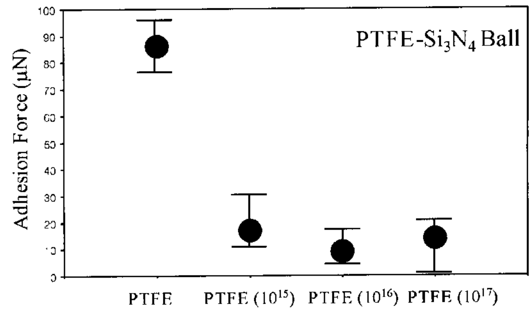



**Fig. 8. 2D topography of the PTFE samples.**

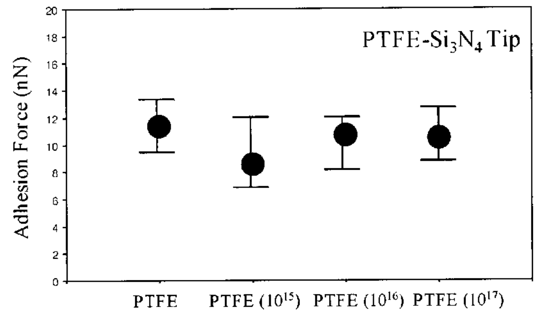
over $10^{17}$.

In order to study the scaling effect of the tribological characteristics, micro-adhesion force and nano-adhesion force were measured with a micro-adhesiometer and SPM as shown in Fig. 9. The micro-adhesion force decreased generally with the ion dose as shown in Fig. 9(a). These results with the previous result of Fig. 6 indicated that the capillary force acting between the $Si_3N_4$ ball and the ion beam modified PTFE surfaces decreases as the surface becomes hydrophobic with the argon ion dose. Therefore it could be argued that the topographical changes of ion beam modified PTFE surfaces, especially the changes in the asperity sharpness, affected the micro-scale adhesion seriously.

Nano-adhesion force showed almost no change with the argon ion dose as shown in Fig. 9(b), even though the surface wetting angle was very high (wetting angle >160°). The results was different from those of micro-adhesion. It was considered that surface topography didn't affect the nano-scale adhesion because the scale of radius of $Si_3N_4$ tip is almost the same order as the scale of asperity of the PTFE surfaces. In this case, the effect of surface topography could be negligible. Therefore, it could be suggested that the changes in asperity sharpness of the PTFE surfaces showed little effect on the nano-scale adhesion phenomena.




(a) Micro-scale adhesion force




(b) Nano-scale adhesion force

**Fig. 9. Adhesion vs. argon ion dose of PTFE samples (a) Micro-scale adhesion force (b) Nano-scale adhesion force.**

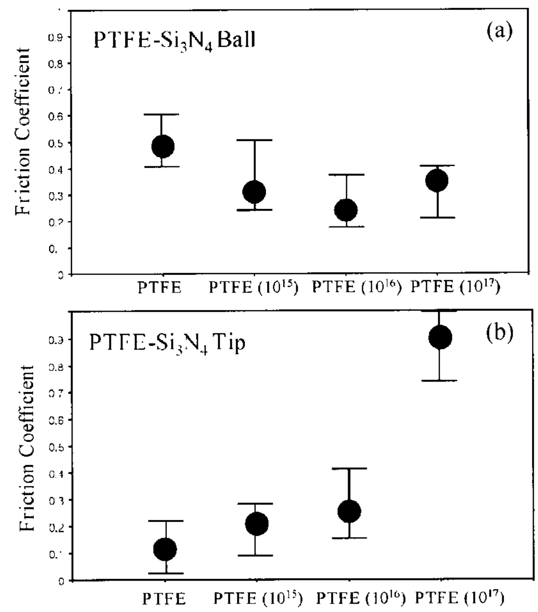



**Fig. 10. Friction coefficient vs. argon ion dose of PTFE samples (a) Micro-scale friction force (b) Nano-scale friction force.**

Fig. 10 shows the micro-friction force and nano-friction force with the ion dose. The micro-friction force decreased with the ion dose as shown in Fig. 10(a). As the surfaces become rough with the ion dose, the real contact area between the $Si_3N_4$ ball and the ion beam modified PTFE surfaces decrease, which results in the decrease of micro-friction force.

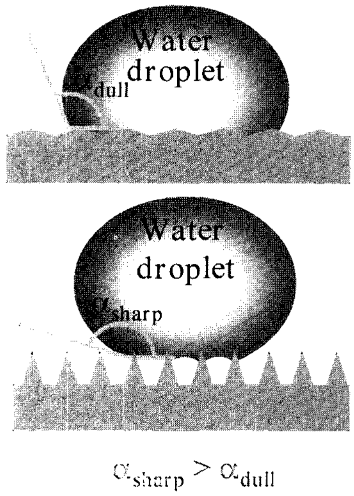



**Fig. 11. Effect of asperity sharpness on the water wetting characteristic of PTFE.**

As shown in Fig. 10(b), nano-friction force increased with the ion dose. This result is different from that of micro-friction. Therefore, it is considered that the increase in the nano-friction was resulted from the local increase of the friction force by ratcheting mechanism proposed by Bhushan [2].

Micro-scale surface roughness (cut-off length 0.8 mm) and water wetting angle of ion beam modified PTFE increased with the argon ion dose, which resulted in the decrease of both micro-adhesion and friction. But, nano-scale surface roughness (cut-off length 2 $\mu$m) didn't have any correlation with the argon ion dose and water contact angle. The asperity sharpness only increased with the argon ion dose. The nano-adhesion was not affected by the asperity sharpness but maintained almost the same. It indicates that no severe chemical change occurred at the PTFE surfaces. Therefore, it is considered that the surface asperities shape of ion beam modified PTFE surface governed the water wetting characteristics. Furthermore, it could be found that the sharpness of surface asperity affects very seriously the apparent water wetting angles. Fig. 11 illustrates the effect of asperity sharpness on apparent water wetting angle. Considering the effect of surface roughness on the adhesion and friction, it was also worth nothing that cut-off length and scale of ball and surface asperity should be considered.

## Conclusions

Micro/nano tribological and water wetting characteristics of ion beam treated PTFE surfaces were experimentally studied at a micro and nano scale. The experimental results can be summarized as follows:

1. The water wetting angles of the ion beam modified PTFE surfaces increased with the ion dose. Ultra-hydrophobic nature was found when the PTFE surface was modified under ion dose of $10^{17}$.

2. The wetting characteristics was strongly dependent on the micro-roughness and the sharpness of asperity.

3. Micro-adhesion and micro-friction were dependent on the wetting characteristics of PTFE. However, nano-tribological characteristics showed little dependency on the wetting characteristics.

4. Considering the effect of surface roughness on the adhesion and friction, cut-off length and scale of ball and surface asperity should be considered.

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