Effect of Film Thickness on the Tribological Characteristics of Zdol Lubricant on Silicon Surface

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ABSTRACT

In precision mechanical components that experience sliding, it is important to reduce the friction to minimize surface damage. Particularly, new lubrication methods are needed to reduce friction in silicon based micro-systems applications. In this work, the tribological characteristics of PFPE (Perfluoropolyether) Zdol lubricant on silicon were investigated based on the thickness of the film. The lubricant was coated on silicon wafer specimens by the dip coating method. It was shown that the friction coefficient as well as stiction decreased as the thickness of the film increased. The results of this work may be applied to improve the tribological performance of silicon based micro-system components.

Key Words : Contact angle, Friction, Stiction, Surface energy, Zdol

1. Introduction

Silicon based micro-systems have great technical potential in various fields such as information storage and bio-MEMS.[1] The performance of micro-system components often depends on their surface durability, which in turn is affected by the tribological characteristics.[2,3] To meet the durability requirements of micro-system components, a reliable lubrication method for silicon based materials needs to be developed.

Recently, Self-Assembled Monolayer (SAM) films have been investigated for their effectiveness in lowering the friction of micro-system components. Though SAMs are found to be effective in minimizing the surface energy, they seem to show some limitation in durability.[4] Hence, further optimization is needed to be able to utilize SAMs for lubrication of silicon based materials.

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In this work, PFPE Zdol lubricant was investigated for its effectiveness in reducing friction and stiction of silicon. PFPE has been widely utilized in the hard disk drive industry to protect the disk media from surface damage incurred by friction or environmental corrosion. It is a good candidate to be used for other precision applications since the thickness of the PFPE film is less than a few nanometers. Specifically, Zdol, a family of PFPE type lubricant, was coated on silicon wafer specimens and tested for its tribological characteristics with respect to coating condition such as thickness.

The silicon wafer specimens were coated by Zdol by the dip coating method followed by a heat treatment process. The thickness of the Zdol film was measured by an ellipsometer. The surface energy of the coated film was assessed by measuring the contact angle of a water droplet placed on top of the coating. Also, a chemical analysis of the Zdol coating was performed using an X-ray Photoelectron Spectroscopy (XPS). The tribological characteristics of the Zdol film with respect to its thickness were obtained using specially built tribo-testers. Specifically, friction and stiction characteristics of the film were investigated.

2. Experiment

2.1 Specimens

Table 1 shows the physical and chemical properties of Zdol. The hydroxyl group of the Zdol molecule serves to chemical bond with silicon or DLC surface by forming Si-OH or Si-H bonds.[5,6] The bonds are known to be largely covalent in nature. However, the bonds are established by heat treating the specimen, and during this process, the bonds are divided into those that are bonded chemically and those that are adsorbed physically.[7]

Table 1 Chemical property and structure on Zdol

End group	Molecular weight (amu)	Density ($\times 10^3$ kg/m^3 at 20 ℃)	Kinematic viscosity (mm^2/s)
-OH	2000	1.81	80 @ 20℃

Formula : $HO\text{-}CH_2\text{-}CF_2\text{-}O\text{-}(CF_2\text{-}CF_2\text{-}O)_m\text{-}(CF_2\text{-}O)_n\text{-}CF_2\text{-}CH_2\text{-}OH$ $(m/n \approx 2/3)$

Zdol solution with concentration ranging between 0.05 ~ 1.2 wt% was used to obtain different coating thickness. The silicon wafer specimens were cut and ultrasonically cleaned using n-hexane, acetone, and methanol solutions[7] prior to being dipped into the lubricant solution. The desired thickness of Zdol coating could be obtained by adjusting the concentration of the solution as well as the lifting speed of the specimen from the solution bath.[8] Following the dip coating process the specimens were heat treated in an oven to stabilize the bonds.

2.2 Experimental derails

2.2.1 Experimental equipment

A dip-coater was designed and built to perform the Zdol coating process. The equipment was designed to lift the specimen from the solution bath at a desired speed. Fig. 1 shows the photograph of the dip-coater. A DC motor was used to move the specimen holder in the vertical direction. The speed of the motor could be adjusted by the voltage controller. For the experiments performed in this work a lifting speed of 1.14 mm/s was used.

The thickness of Zdol film on silicon wafer specimen was measured by ellipsometry. Each specimen was measured at three locations and the average thickness value was used. The resulting values of Zdol thickness were between 1 to 5 nm. Also, the integrity of the coating was assessed by XPS and contact angle analyses. XPS was used for chemical analysis of the Zdol film and the contact angle was used to infer the surface energy of the film.

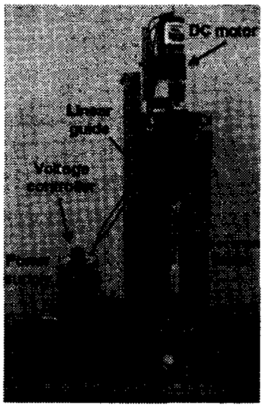


Fig. 1 Photograph of dip coater

Friction tests were performed using a pin-on-disk type tribo-tester shown in Fig. 2. The Zdol coated silicon specimens were slid against a 600 μm radius Si_3N_4 sphere under an applied load of 30 mN at 20 mm/s sliding speed. The total distance slid was set to 60 m. All experiments were conducted at room temperature at a relative humidity of about 50 %.

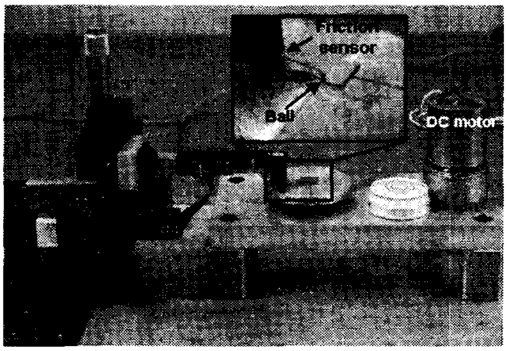


Fig. 2 Pin-on-disk tribo-tester

Stiction, which is characterized by the friction coefficient at the onset of sliding, was measured using a specially built stiction tester. The schematic of the stiction tester is shown in Fig. 3. An uncoated rectangular (3 mm x 4.5 mm) silicon specimen was placed on top the Zdol coated silicon specimen as shown in the schematic and was allowed to be at rest for 20 minutes. Then, the specimens were rotated until the top silicon specimen comes in contact with the load beam which blocks the specimen from moving together with the bottom specimen. The stiction force is measured by noting the maximum force needed to cause relative motion between the two specimens. A high data sampling rate of 20 kHz was used to capture the onset of sliding.

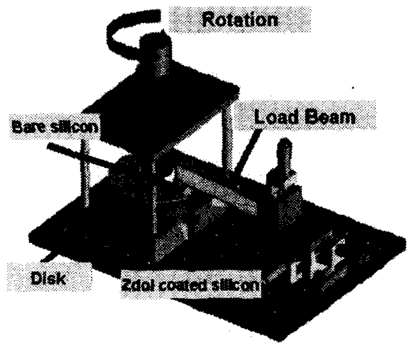


Fig. 3 Schematic of stiction tester

2.3 Experimental results

Fig. 4 shows the friction coefficient data for Zdol films of various thickness. It should be noted that the friction coefficient for uncoated silicon specimen was the highest, the value being about 0.32. Also, the friction coefficient was the lowest for the 4 nm thick Zdol film. Specifically, the friction coefficient was about 0.2 and 0.13 for 1 nm and 4 nm thick Zdol film, respectively.

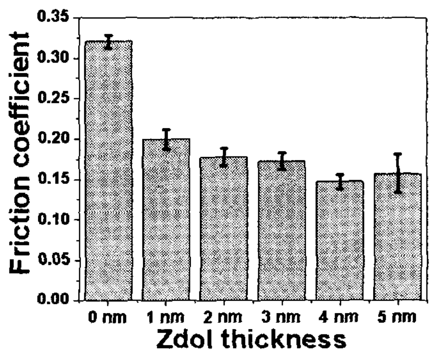


Fig. 4 Friction coefficient wrt Zdol thickness

However, in the case of 5 nm thick Zdol film, friction coefficient increased slightly compared with the 4 nm thick specimen. The reason for this outcome is the increase in surface rms roughness of the 5 nm thick Zdol coating. Considering the result of topography measurement using non-contact mode AFM, agglomeration effect of the Zdol lubricant was observed in 5 nm thick Zdol coating. Therefore, it is possible that Si_3N_4 ball which was used in the friction test experienced greater frictional resistance compared with other Zdol coated specimens.

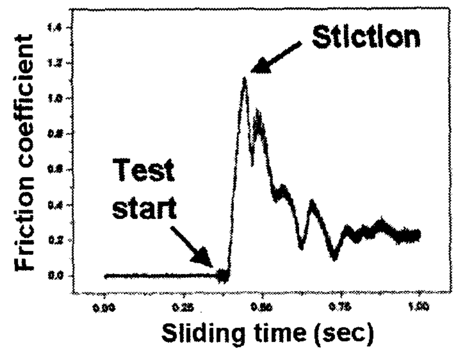


Fig. 5 Typical friction data for stiction measurement

A typical data obtained during a stiction test is shown in Fig. 5. The sharp rise in the friction coefficient at 0.35s was due to the deflection of the load beam as the top silicon specimen pushed against the load beam. Then, the friction coefficient dropped suddenly due to the slip motion. The maximum friction coefficient was noted as the stiction coefficient.

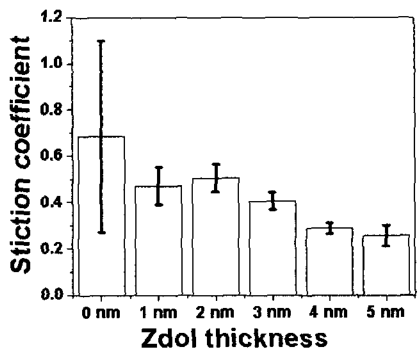


Fig. 6 Stiction coefficient wrt Zdol thickness

Fig. 6 shows the stiction data for uncoated and Zdol coated silicon specimens. As can be seen, the stiction was the highest and the most unstable for uncoated silicon surface. For 1 nm Zdol thickness, the stiction coefficient was as low as 0.4. However, as the thickness increased to 2 nm, the stiction coefficient also increased to about 0.5. From then on, the stiction coefficient decreased with increasing Zdol thickness. The reason for such a variation in the stiction coefficient with respect to the Zdol thickness is unclear. In any case, it seems that in general, the stiction is the lowest for the thickest Zdol film as in the case of the friction coefficient.

2.4 Characterization of Zdol film

The Zdol coated surface was analyzed using contact angle measurement technique as well as XPS. The primary purpose was to assess the integrity of the Zdol film. There was also a motivation to correlate the tribological behavior of the Zdol coated silicon specimens presented in the previous section to the film characteristics of Zdol. Fig. 7 shows the contact angle with respect to Zdol thickness. Without coating, bare silicon specimen resulted in the lowest contact angle. The highest contact angle resulted from the 5 nm thick Zdol specimen.

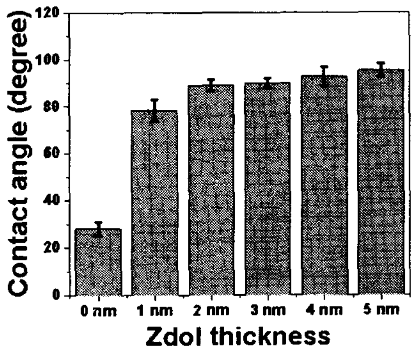


Fig. 7 Contact angle wrt Zdol thickness

Generally, contact angle is indicative of the hydrophobicity of a given solid surface. If the contact angle is greater than 90 degrees, the surface is known to be hydrophobic, and therefore, does not get readily wetted. Contact angle is also directly related to the surface energy. High contact angle suggests that the surface has relatively low surface energy.

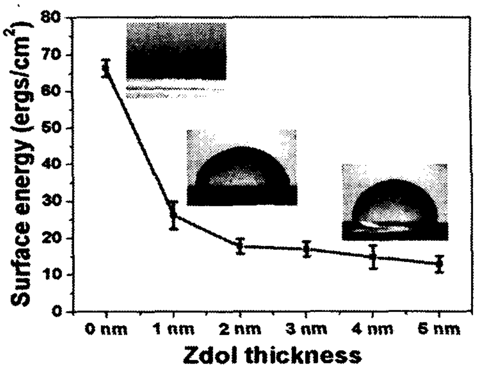
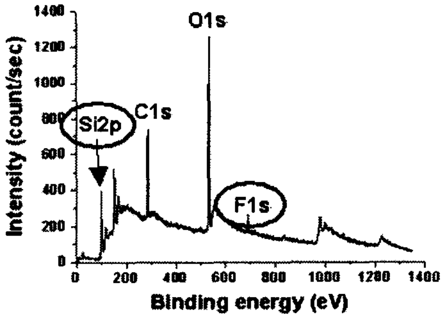
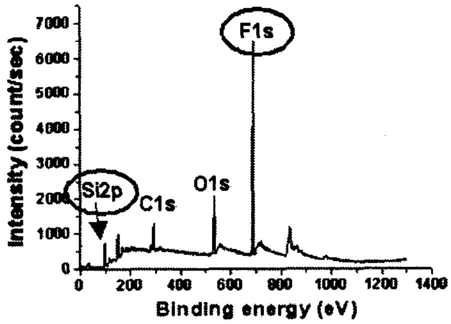


Fig. 8 Surface energy wrt Zdol thickness

Fig. 8 shows the surface energy with respect to Zdol thickness. The shape of the curve is inverse to that of the contact angle data. The photographs shown in the graph of Fig. 8 are the water droplets on the test specimen surface. In summary, the surface energy of the Zdol film decreased as the thickness of the film increased. This provides a good reason for the low friction coefficient obtained with the thick Zdol film specimen.



(a) XPS result of bare silicon

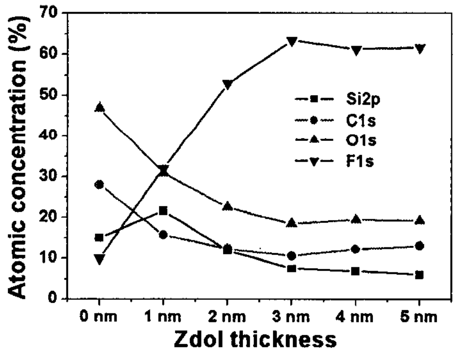


(b) XPS result of 5 nm Zdol coated specimen

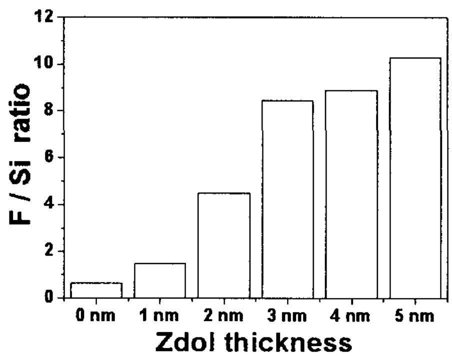
Fig. 9 XPS result of bare and Zdol coated silicon specimens

XPS analysis was performed to identify the basic difference in the chemical composition of the Zdol coated specimens with respect to their thickness. Fig. 9 shows the XPS results for bare and Zdol coated silicon specimens. As expected, there was a significant difference in the concentration of fluorine, F.

The ratio of F concentration with respect to Si in the XPS analyses was obtained for various Zdol coating thickness. Fig. 10(a) shows the atomic concentrations of several elements for the Zdol coated specimens with different thickness. The ratio of F to Si from this data is given in Fig. 10(b). It is clear that the concentration of F with respect to Si increases with coating thickness, and this is an important reason for the relatively low surface energy and friction coefficient observed with the thick Zdol coated silicon surface.



(a) Atomic concentration



(b) F / Si ratio of specimen

Fig. 10 XPS result wrt Zdol thickness

3. Conclusions

The friction and stiction characteristics of Zdol coated silicon specimens were investigated with respect to the coating thickness. The coating thickness from 1 to 5 nm could be obtained by varying the concentration of the Zdol solution. Lowest friction and stiction coefficients were obtained with 4 nm thick Zdol film. The values for the friction and stiction coefficients for the 4 nm thick Zdol specimen were 0.13 and 0.3, respectively. The decrease in friction for relatively thick Zdol film is attributed to low surface energy and high F to Si ratio.

As for the coating integrity, silicon surfaces could be uniformly coated with 2 ~ 5 nm thick Zdol film as verified by the high contact angle of water droplet and low surface energy. Also, 4 nm thick Zdol coated silicon showed the best tribological characteristics. However, in the case of 5 nm thick Zdol specimen, friction coefficient increased due to the agglomeration effect of Zdol lubricant. Therefore, it is concluded that 3 ~ 4 nm thick Zdol films on silicon are optimum for tribological applications.

Based on the results of this work, it is expected that Zdol can be utilized as an effective lubricant for silicon based materials in micro-systems applications.

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