

Photolithographic Silicon Patterns with Z-DOL (perfluoropolyether, PFPE) Coating as Tribological Surfaces for Miniaturized Devices

R. Arvind Singh, Duc-Cuong Pham and Eui-Sung Yoon[†]

Nano-Bio Research Center, Korea Institute of Science and Technology, Seoul 136-791, Korea

Abstract: Silicon micro-patterns were fabricated on Si (100) wafers using photolithography and DRIE (Deep Reactive Ion Etching) fabrication techniques. The patterned shapes included micro-pillars and micro-channels. After the fabrication of the patterns, the patterned surfaces were chemically modified by coating Z-DOL (perfluoropolyether, PFPE) thin films. The surfaces were then evaluated for their micro-friction behavior in comparison with those of bare Si (100) flat, Z-DOL coated Si (100) flat and uncoated Si patterns. Experimental results showed that the chemically treated (Z-DOL coated) patterned surfaces exhibited the lowest values of coefficient of friction when compared to the rest of the test materials. The results indicate that a combination of both the topographical and chemical modification is very effective in reducing the friction property. Combined surface treatments such as these could be useful for tribological applications in miniaturized devices such as Micro/Nano-Electro-Mechanical-Systems (MEMS/NEMS).

Keywords: silicon, micro, patterns, friction, tribology

1. Introduction

Nano/micro-scale tribology plays an important role in many emerging fields, such as magnetic recording media and MEMS/NEMS [1,2]. MEMS/NEMS devices are comprised of elements that are small in size and operate at nano/micro-scales. At these scales of size, the ratio of surface area to volume is high (~1000) and hence, the surface forces such as adhesion and friction strongly influence the tribological behaviour of their tiny elements. These forces are critical as they decrease the performance and consequently reduce the durability of the miniaturized devices [1,2].

Silicon is a popular MEMS/NEMS material; however its tribological properties are poor, which is mainly due to its high interfacial energy [3,4].

In order to improve the performance of silicon, different surface modification approaches have been undertaken that include chemical modifications and topographical modifications [4]. In this investigation, we studied the effect of the combination of topographical and chemical modifications on the micro-friction property of Si (100) surfaces.

2. Experimental

Si (100) surfaces were topographically modified into micro-patterns using photolithography and DRIE (Deep Reactive Ion

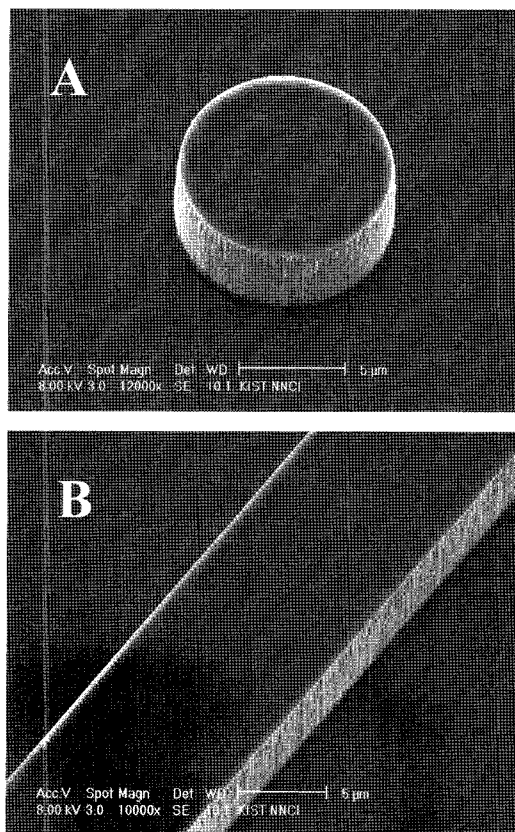


Fig. 1. Images taken using Scanning Electron Microscope showing: (A) single micro-pillar and (B) single micro-channel.

[†]Corresponding author; esyoon@kist.re.kr
Tel: +82-2-958-5651, Fax : +82-2-958-6910

Etching) fabrication techniques. The patterned shapes included micro-pillars and micro-channels. The various steps involved in the fabrication of the Si (100) micro-patterns are described elsewhere [5]. Figure 1 shows scanning electron microscope (SEM) images of the fabricated Si patterns.

The dimensions of the micro-patterns are: micro-pillars (diameter: $10\ \mu\text{m}$) and micro-channels (width: $10\ \mu\text{m}$). The height of these patterns: $5\ \mu\text{m}$ and the pitch (distance between the centers of any two individual pillars/channels): $30\ \mu\text{m}$.

The micro-patterns were chemically treated by coating Z-DOL (perfluoropolyether, PFPE) thin film using a dip coating method. The various steps involved in coating Z-DOL are briefly described below. Before coating, the samples were cleaned using acetone and isopropyl alcohol (IPA) in an ultrasonic bath for about 5 minutes each. Later, they were rinsed in deionized water and dried using nitrogen gas. Z-DOL2000 (Solvay Solexis) diluted in hydrocarbon solvent (HT70, Solvay Solexis) at 0.1% vol. was used as the dipping solution. The cleaned silicon samples were vertically dipped into the solution for duration of 10 minutes. The samples were then pulled out of the solution at a speed of 3.5 mm/sec. After the dipping process, the samples were heated in a furnace at 150°C for 30 minutes, and subsequently they were washed in a solvent solution (HFE-7100, 3M) for about 5 minutes in order to remove the mobile fractions. The thickness of Z-DOL films on flat silicon substrates measured by Ellipsometry was about

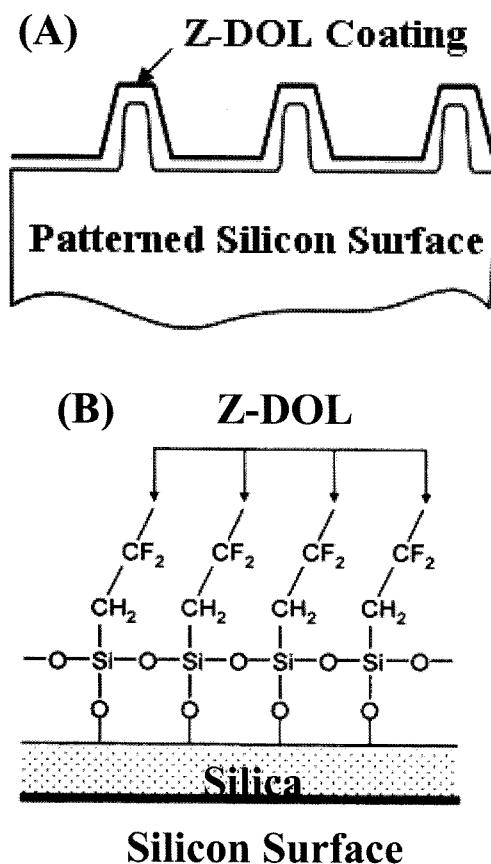


Fig. 2. Schematics of (A) combined surface modification of silicon surfaces and (B) Z-DOL coating on silicon surfaces.

2 nm. Figure 2 (a) shows the schematic of the combined surface modification of silicon surfaces i.e. topographical and chemical modifications. Figure 2 (b) shows the schematic of Z-DOL coating on silicon surfaces.

The samples that were tested for their micro-friction behavior include the bare Si (100) flat surface, the Z-DOL coated Si (100) flat surface, the uncoated Si patterns and the Z-DOL coated Si patterns. All these surfaces were tested for their micro-friction property against soda lime balls (diameter: 1mm) at micro-scale using a ball-on-flat type micro-tribotester. In the friction tests, the sliding speed and the normal load were kept constant at 1 mm/sec and $3000\ \mu\text{N}$, respectively. The scan length was kept constant at 3 mm. Each test was conducted for about 15 minutes. Friction was estimated as an average of the steady state friction values from more than five test repeats. All experiments were conducted in a clean room, at controlled temperature of $24\pm 1^\circ\text{C}$ and relative humidity of $45\pm 5\%$. In the case of micro-channels (uncoated and coated), tests were conducted in two different directions, namely, along the length of the ridges (parallel scan) and across the length of the ridges (orthogonal scan).

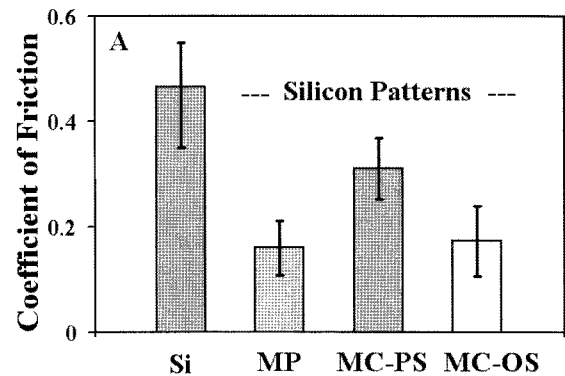


Fig. 3. Coefficient of friction values of bare Si (100) flat surface, micro-pillars (MP), micro-channels parallel scan (MC-PS) and micro-channels orthogonal scan (MC-OS).

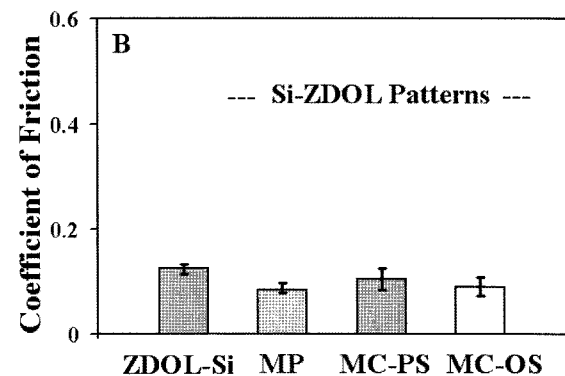


Fig. 4. Coefficient of friction values of Z-DOL coated Si (100) flat surface (ZDOL-Si), Z-DOL coated micro-patterned surfaces: micro-pillars (MP), micro-channels parallel scan (MC-PS) and micro-channels orthogonal scan (MC-OS).

3. Results and Discussion

Figure 3 shows the results of the micro-friction tests of the bare Si (100) flat surface and those of the uncoated Si patterns (MP: micro-pillars, MC-PS: micro-channels parallel scan, MC-OS: micro-channels orthogonal scan). Figure 4 shows the results of the micro-friction tests of the Z-DOL coated Si (100) flat surface (ZDOL-Si) and those of the Z-DOL coated Si patterns (MP: micro-pillars, MC-PS: micro-channels parallel scan, MC-OS: micro-channels orthogonal scan). Comparing Figures 3 and 4, it could be seen that the bare Si (100) flat surface exhibits the highest value of coefficient of friction (CoF), when compared to those of the rest of the test specimens. Further, it has been observed that the bare Si (100) flat surface exhibits wear [3,4]. Si (100) is a poor tribological material and the main reason for which is its higher interfacial energy (hydrophilic nature) [3,4]. By coating Si (100) flat surfaces with Z-DOL, the values of the friction coefficient of Si (100) flat surface reduce significantly (Figure 4). This is due to the excellent lubrication property of Z-DOL [6,7]. Interestingly, Z-DOL can be chemically bonded to silicon surfaces, which make them less prone to wear (removal of Z-DOL molecules from silicon surface during sliding). Z-DOL thin films also exhibit lower surface energies, which further reduces adhesion induced friction, at small-scales [6,7].

From Figure 3, it could be seen that the silicon patterns exhibited CoF values lower than those of the bare Si (100) flat surface. This result could be understood by considering the fundamental law of friction given by Bowden and Tabor [8]. According to the law, friction force is directly proportional to the real area of contact. In the present case, the patterned surfaces exhibit lower friction values when compared to the bare Si (100) flat surface due to the fact that they project reduced real area of contact. Patterning of surfaces causes a reduction in the real area of contact when the size of the asperities (pillars/channels) is considerably smaller than that of the counterface slider (glass ball) [4].

From Figure 3, it is also seen that the friction behavior of the micro-channels is dependent on the sliding direction. As seen from the figure, the CoF value along the length of the ridges (parallel scan, MC-PS) is higher than that across the ridges (orthogonal scan, MC-OS). During sliding, when the counterface ball moves in the parallel direction the contact with the ridges is a continuous one. On the other hand, when the ball slides orthogonally the contact is discontinuous. Hence, when the ball slides in the parallel direction, the contact area is larger when compared to the other sliding direction [5]. This results in higher CoF value for the parallel scan (MC-PS) when compared to the orthogonal scan (MC-OS).

Further, from Figure 4, it could be seen that the Z-DOL coated Si (100) patterns showed considerable reduction in the CoF values when compared to the Z-DOL coated Si (100) flat

surface. In addition, the coated patterns did not exhibit any wear. Comparing the CoF values of the coated Si (100) patterns with those of the bare Si (100) flat surface, Z-DOL coated Si (100) flat surface and uncoated silicon patterns, it could be seen that the surfaces with both the topographical and chemical modifications have the most enhanced tribological behavior. In case of Z-DOL coated micro-channels, it was found that the friction behavior was strongly dependent on the sliding direction, due to the variation in the real area of contact.

4. Conclusions

Topographical modification of a surface renders a reduction in its friction property through the reduction in the real area of contact, whereas chemical modification reduces friction by lowering the interfacial energy. From the present investigation it could be concluded that a combination of these two kinds of modifications is most effective in reducing the friction and wear of surfaces. This combined approach could prove as a promising tribological solution for miniaturized devices.

Acknowledgement

This research was supported by KIST Institutional Program and the Intelligent Microsystem Center (IMC; <http://www.microsystem.re.kr>), which carries out one of the 21st century's Frontier R&D Projects sponsored by the Korea Ministry Of Commerce, Industry and Energy.

References

1. Bhushan, B., "Nanoscale tribophysics and tribomechanics", *Wear* 225, pp.465-492, 1999.
2. Bhushan, B., "Springer Handbook of Nanotechnology", Springer-Verlag, Berlin, Heidelberg, p.983, 2004.
3. Yoon, Eui-Sung, Singh, R. Arvind, Oh, H. J, Kong, Hosung., "The effect of contact area on nano/micro-scale friction", *Wear* 259, pp.1424-1431, 2005.
4. Singh, R. Arvind and Yoon, Eui-Sung., "Friction of chemically and topographically modified Si (100) surfaces", *Wear* 263, pp.912-919, 2007.
5. Pham, Duc-Cuong, Singh, R. Arvind, Yoon, Eui-Sung., "Dual surface modifications of silicon surfaces for tribological application in MEMS", *KSTLE Intl. Journal* 8 (2), pp.26-28, 2007.
6. Liu, H, Bhushan, B., "Nanotribological characterization of molecularly thick lubricant films for applications to MEMS/ NEMS by AFM", *Ultramicroscopy* 97, pp.321-340, 2003.
7. Tao, Z, Bhushan, B., "Degradation Mechanisms and Environmental Effects on Perfluoropolyether, Self-Assembled Monolayers, and Diamondlike Carbon Films", *Langmuir* 21, pp.2391-2399, 2005.
8. Bowden, F. P, Tabor, D., "The friction and lubrication of solids", Clarendon Press, Oxford, pp.90-121, 1950.