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A facile method to improve tribological properties of silicon surface by combining nanogrooves patterning and thin film lubrication

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ABSTRACT

Surface micro/nanohierarchical structure designing and thin film lubrication are two main methods to alleviate adhesive and frictional problems encountered by micro/nanoelectromechanical systems (MEMS/NEMS). In this study, silicon surfaces with micro/nano grooves were prepared by photolithography and further chemically modified by multiply-alkylated cyclopentane (MAC) thin films to improve the tribological behaviors of MEMS/NEMS. The effects of nanoscaled roughness (including groove pitch and groove fractional surface coverage) and chemical modification on the wetting and tribological properties of surfaces were systemically investigated. The results of contact angle measurement indicated that the surface hydrophilicity decreases with increasing of surface roughness and lowering of the surface energy (chemical treatment with MACs). Tribological study showed that with the increasing of nanoscale roughness and combined with chemical modification, the tribological properties are greatly improved, which may be affected by real area of contact and the surface chemistry.

1. Introduction

During the last decade, MEMS/NEMS have developed rapidly due to their excellent performance and low unit cost [1]. However, adhesive and frictional problems that cause the failure of MEMS/NEMS become severe as the size of devices shrinks to micro- and nanoscales [2–4]. And extensive efforts have been paid to minimize the friction and inhibit adhesion and stick-slip friction [5–10]. Traditionally, silicon has been the most widely used material for fabrication of MEMS/NEMS devices. Therefore, most of the investigations have been directed towards enhancing the tribological performances of silicon wafers [1,11,12]. Among these works, topographical modification (surface micro/nanohierarchical structures designing) [13,14] and chemical modification (use of thin lubricant films such as self-assembled monolayers (SAMs) or perfluoropolyether (PFPE) films) are two main subjects [15,16].

The surface friction forces, including adhesion and static electricity, in MEMS/NEMS are greatly dependent on the contact geometry and surface topography [17]. So, surface topographical modification is an effective way to reduce adhesion and friction of the contact interface in micro/nano systems. Moreover, as many studies have shown, the surface roughness influences adhesion and friction greatly [18–20]. An effective way to control surface roughness is to prepare textured surface and change the height and fractional surface coverage of micro/nano- hierarchical textures. Previous reports showed that, compared to non-textured surfaces, textured surfaces have significantly improved adhesion/stiction and friction performances [20].

As to thin film lubrication, alkylsilane SAMs constructed on silicon surface is one of the most popular nano film lubricants for MEMS/NEMS [8,21]. However, generally, the SAMs exhibit poor load-carrying capacity and durability due to its low molecular flexibility and mobility [22–24]. Besides, PFPE films, considered as potential substitute lubricants for MEMS/NEMS [16], are susceptible to catalytic degradation by Lewis acids and tend to cause metal fluoride corrosion on metal surfaces [25–27]. It is highly needed to find better lubricants systems with longer durability and better tribological performance for MEMS/NEMS. Multiply-alkylated cyclopentanes (MACs), which have excellent viscosity properties, mobility, thermal stability and low volatility, are presently gaining wide acceptance on actual space lubrication and is expected to be applied in MEMS/NEMS [28,29]. Our previous results indicate that MACs can improve the friction performance of silicon substrate [4].

In this article, silicon surfaces patterned with grooves of varying pitch and fractional surface coverage were fabricated by lithography. The pitch and fractional surface coverage of grooves are optimized to obtain a textured surface with superior friction behaviors. To improve the tribological properties further, the textured silicon wafer was then chemically modified with MACs lubricant films. As far as we know, tribological studies on the combination of

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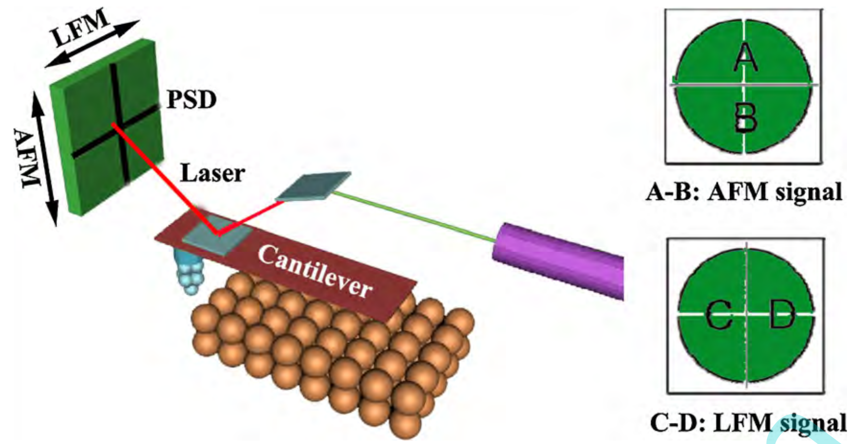

Fig. 1. Illustration of photoelectric signs of load and lateral force.

surface structure designing and MACs lubricant films have not yet been reported. Researches in this aspect will be a very meaningful thing for the practical application of MEMS lubrication.

2. Experimental

2.1. Materials and sample preparation

MACs were synthesized by the previous procedure [4,29]. N-Hexane (purity > 98%) was used as received. PFPE (formula $HOCH_2CF_2O–(CF_2–CF_2O)_m–(CF_2O)_n–CF_2CH_2OH$, m and n are integers, MW3800, with the commercial name Zdol 3800) was purchased from Aldrich Chem. Co. Ltd and used as received.

Silicon masters with groove micro/nano structures (with groove length of more than 2 cm) were designed and fabricated using photolithography techniques. Polished single-crystal silicon (1 0 0) wafers were ultrasonically cleaned sequentially in acetone, ethanol and ultra-pure water each for 5 min, and then dried under a stream of nitrogen. Then the silicon wafers were coated with photoresist and exposed through a photomask under UV light by use of a suss Ma6 mask aligner. After adequate exposure, the substrate was exposed in SF_6 and C_2H_4 for further silicon etching using ICP etcher (Corial 200IL, France). Finally, the photoresist was removed and the patterned silicon wafers were ultrasonically cleaned sequentially in acetone, ethanol and ultra-pure water each for 5 min again, and then was thoroughly washed by ultra-pure water and dried under a stream of nitrogen.

Then the silicon masters were slowly dipped into and withdrawn from a tank containing the MAC solution (0.05% (w/v)) with a uniform velocity of 50 μm/s and the dwelling time was up to 120 s in order to obtain a uniform coating. Each sample was allowed to air dry in a clean room before conducting measurements as described below.

2.2. Characterization of films

Thermogravimetric analysis was conducted in nitrogen with a Perkin–Elmer 7 series apparatus at a scanning rate of 10 °C/min to study the volatility of MACs.

The film morphologies were examined with an atomic force microscope (AFM) (CSPM4000, Being Nano-Instruments Ltd., China), using contact mode.

The static contact angles for ultrapure water on the samples were measured with a DSA100 contact-angle meter. At least five replicate measurements were carried out for each specimen, and the measurement error was below 2°.

2.3. Nano-friction behavior measurement

In order to obtain the relations between surface texture/surface chemistry and nanotribological properties, comprehensive study of the nanofriction with various nanotextures using AFM was present. And it is expected that this study could be helpful to aid the design and selection of appropriate nanopattern parameters for MEMS/NEMS.

The nano-friction behavior of the films was evaluated with an AFM/FFM controlled by CSPM4000 electronics, using the contact mode. Commercially available rectangle Si_3N_4 cantilever with a normal force constant, 6 N/m and a Si_3N_4 tip with a radius of less than 10 nm (Budgetsensors Instruments Inc) was employed [4]. Details of the technique have been described elsewhere [4,30,31]. A schematic illustration of photoelectric signs of load and lateral force of the AFM/FFM is shown in Fig. 1. To obtain friction data, the tip was scanned back-and-forth perpendicular to the groove in contact with sample at typically normal loads ranged from 0 to 400 nN while the lateral deflection of the lever was measured. Friction force measurement was performed at a rate of 1 Hz along the scan axis and a scan size of 50 μm × 50 μm. For all measurements the same cantilever was used in this comparative study. Furthermore, to avoid influence of molecules which may transfer to the tip during the experiment, the tip was scanned on a cleaved mica surface to remove these physical adsorbed molecules. All the tests were conducted at room temperature and a relative humidity of 20%.

2.4. Micro-friction behavior measurement

Microtribological properties were studied on a UMT-2MT tribometer (CETR, USA), using a ball-on-plate mode. The upper ball used here were commercially available stainless steel balls (Φ = 6 mm). A load of 60 mN and a sliding rate of 30 mm/s were applied for all measurements. The friction coefficient-time plots were recorded automatically, and at least three repeated measurements were performed. The morphologies of worn surfaces were observed on a non-contact interferometric microscope (ADE Phase-Shift). All the experiments were performed at room temperature.

3. Results and discussion

3.1. Characterization of MACs

Thermal stability of MACs was examined by TGA between 20 and 600 °C, as shown in Fig. 2. Compared to commercial Zdol, MACs have higher decomposition temperature and very low vapor loss, which

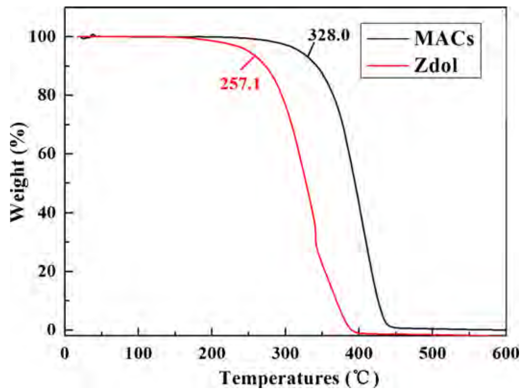

Fig. 2. TGA curves of MACs and Zdol (PFPE).

are equivalent to a commercial multiply-alkylated cyclopentane base fluid (2001A) [29]. There is little weight loss below 300 °C.

3.2. Surface morphology

The morphological characteristics of the patterns before and after the deposition of MACs are almost the same due to the micro scale groove textures and the ultra-thin depositing film with about 2.5 nm. So we only present here the 3D and line crosssection analysis AFM topographic images and the SEM images of the surfaces of patterned silicon wafers modified with MACs, as displayed in Fig. 3. The patterned surfaces have micro/nano groove features with the same depth of 80 nm and same width of 2600 nm but varying pitch and fractional surface coverage, as seen from Fig. 3. For convenience, we ascribe the patterned silicon wafers as L and the

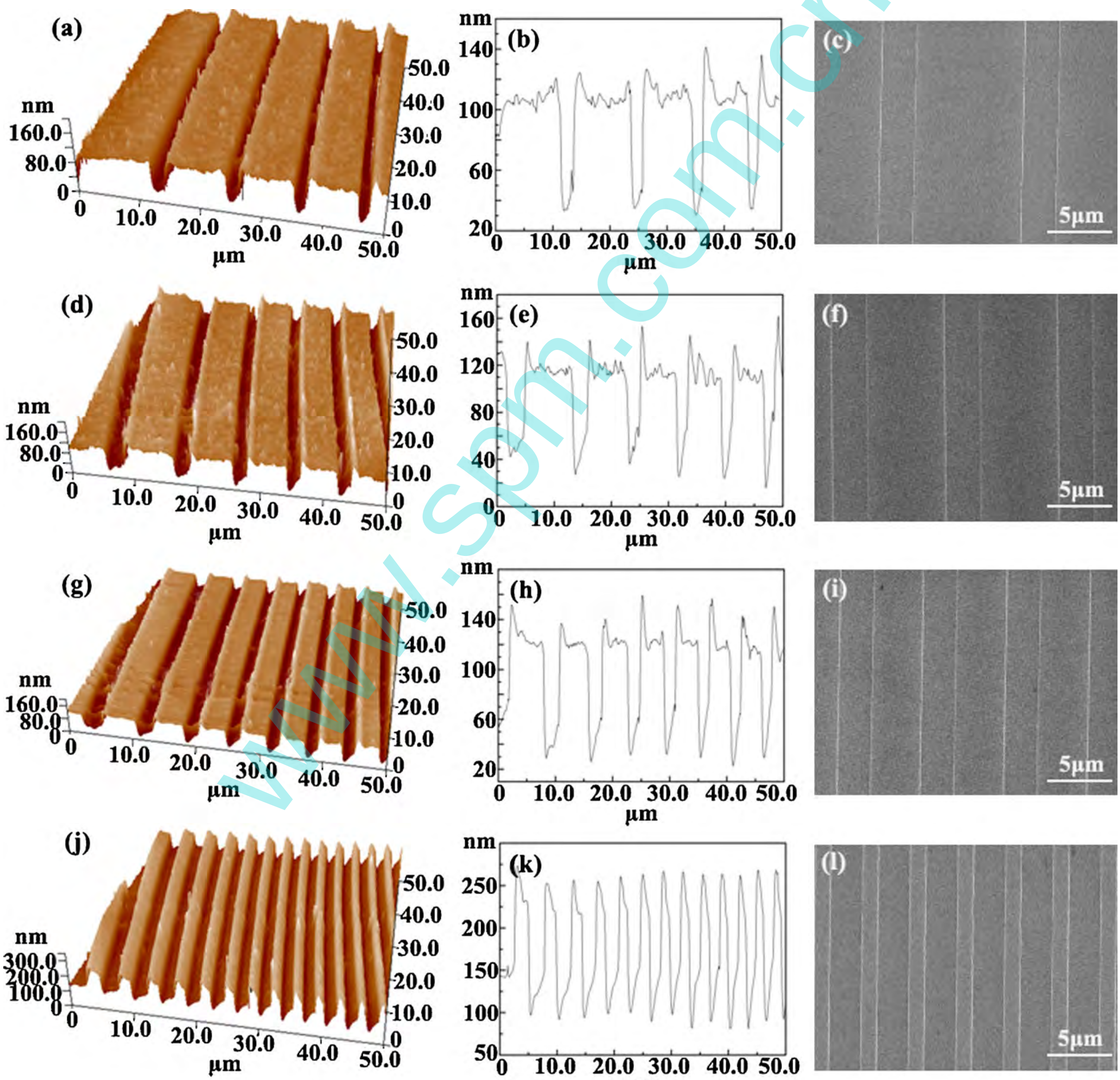

Fig. 3. 3D and line crosssection analysis AFM topographic images and SEM images of the patterned silicon surfaces modified with MACs. LM1 (a–c), LM2 (d–f), LM3 (g–i), and LM4 (j–l).

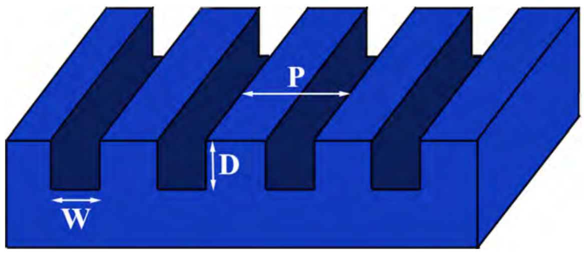

Fig. 4. Design parameters for micro/nano grooves.

Table 1
Geometrical parameters of patterned silicon surfaces with micro/nano grooves used in our experiment, groove width (*W*), groove depth (*D*), the pitch of adjacent cylinder pillars (*P*), and the groove fractional surface coverage (*R*).

Sample	*D* (nm)	*W* (μm)	*P* (μm)	*R* (%)
L1	80	2.6	12	21.7
L2	80	2.6	10	26.0
L3	80	2.6	7	37.1
L4	80	2.6	4	65.0

patterned silicon wafers modified with MACs as LM, respectively. We abbreviate patterned silicon surface with the different groove fractional surface coverage from low to high as L1, L2, L3, and L4. Correspondingly, the patterned silicon wafers with micro/nano texture from low area density to high area density modified with MACs are ascribed to LM1, LM2, LM3, and LM4, respectively.

Fig. 4 shows the schematic process flow for geometrical parameters of silicon micro/nano grooves. The pattern is a 1D array of grooves. The critical parameters are the width of each groove (*W*), the depth of each groove (*D*) and the pitch of adjacent grooves (*P*), which were found by analysis of crosssection lines acquired from the AFM images and SEM images. Repeated measurements were done to obtain the average value for each sample. Groove fractional surface coverage *R* (%) was determined by the following equation:

$$R\,(\%) = \frac{NS_{\rm groove}}{S_{\rm scan}} = \frac{NW}{a} \qquad (1)$$

where N is the number of groove; $S_{\rm groove}$ and $S_{\rm scan}$ are the area of a groove and the patterned surface, respectively; W is the width of the groove; and a is the length of the patterned surface perpendicular to the direction of the grooves. According to the above calculation, the groove fractional surface coverage *R* (%) from low to high is 21.7, 26.0, 37.1, and 65.0%, respectively. All geometrical parameters including the width, depth, pitch, fractional surface coverage and surface roughness are summarized in Table 1, which shows that surface roughness increased as the groove fractional surface coverage increased.

Table 2
Contact angles of the patterned silicon surfaces modified without and with MACs.

Sample	1	2	3	4
L	46.78	47.08	47.22	47.70
LM	60.36	61.71	61.99	62.56

3.3. Contact angle measurements

Wettability, governed by both chemical composition and topological characteristic of the surface, is one of the most important properties of solid surfaces [32–34]. In this research, contact angles of the water liquid droplets on the samples were measured in order to obtain a quantitative understanding of the effect of micro/nanostructure and chemical modification on wettability.

Contact angles for ultrapure water on patterned silicon substrates without and with MACs are displayed in Table 2, and the relationship between the surface properties and the contact angle is evident. It can be clearly seen that the surface hydrophilicity decreases with increasing of surface roughness and lowering of the surface energy (chemical treatment with MACs). This is because with the increasing of surface roughness, air may be trapped in the contact surface, resulting in a composite solid–air–liquid interface, which leads to higher contact angle [33]. The small difference in water contact angle (WCA) among the L1 to L4 and LM1 to LM4 surfaces, may be due to the very low aspect ratio, which is only 0.03 [35,36]. And lowering of the surface energy with chemical treatment, using MACs with apolar $-(CH_2)_n-CH_3$ (hydrophobic) groups, would also result in increasing contact angle [37,38]. Suitable surface for special MEMS/NEMS application could be designed by controlling these factors.

3.4. Nanotribological study

To investigate the nanofriction properties of the patterned silicon surfaces with different surface roughness before and after modified with MAC films, we measured the nanofriction force versus normal load curves by AFM/FFM, and the results are depicted in Fig. 5. As seen from Fig. 5, nanofriction force decreased as groove area density increased, and further reduced greatly after modified with MAC films. L1 showed the largest nanofriction force and LM4 showed the smallest nanofriction force among the surfaces with micro/nanotextures investigated in this study. The nanofriction force given here is in the form of voltage signal, which is propor-

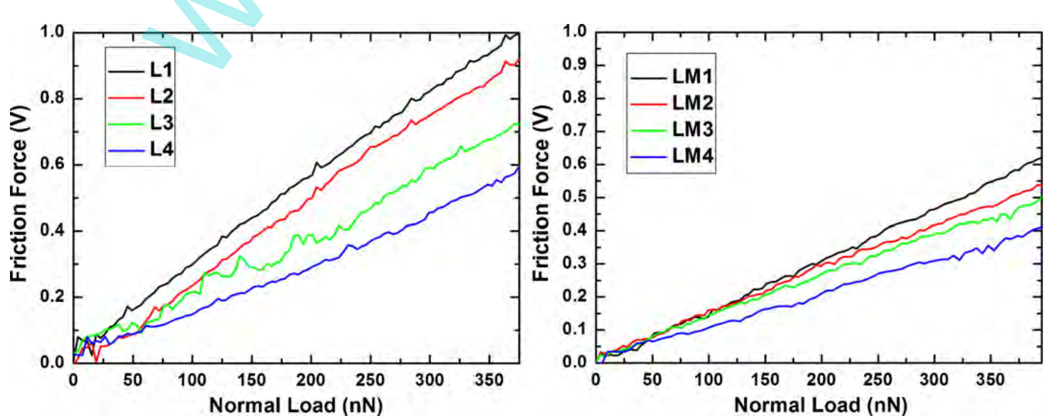

Fig. 5. The plot of friction force versus load curves for the surfaces of the patterned silicons with different fractional surface coverage before and after modified with MACs (a: L1–L4, b: LM1–LM4).

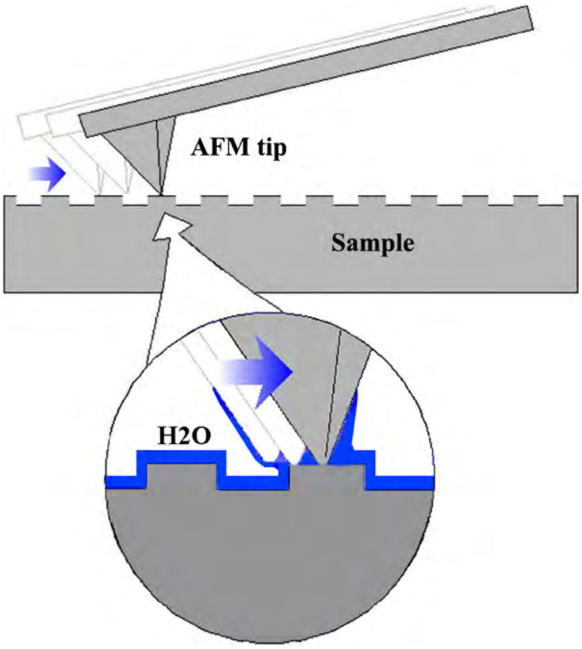

Fig. 6. Schematic of the interfacial contact region and the interaction forces acting between an AFM tip and patterned substrate in an ambient atmosphere.

tional to the real nanofriction force [39]. Therefore, comparison between the results from various surfaces could be achieved.

At nanoscale, the real area of contact and the surface chemistry affects friction in contacts strongly [40]. Firstly, for the same kind of samples, the real area of contact is mainly determined by the micro/nano-texture fractional surface coverage of material surfaces [39,41]. With increased fractional surface coverage of micro/nano grooves, the number of asperities in contact is reduced which results in the real area of contact reduced and leads to lower friction force. Secondly, if the surface is hydrophilic, it would be covered by a thin water film and easy to form capillary meniscus by the adsorbed water molecules, which would lead to large capillary force and higher friction [4,33]. In other words, if the surface is hydrophobic, the opposite result would be obtained. The schematic of the interfacial contact region between an AFM tip and patterned substrate in an ambient atmosphere is shown in Fig. 6. MACs made the surface less hydrophilic leading to lower surface energy, which also confirmed by contact angle measurement, so the friction force of LM4 is the lowest among all the surfaces.

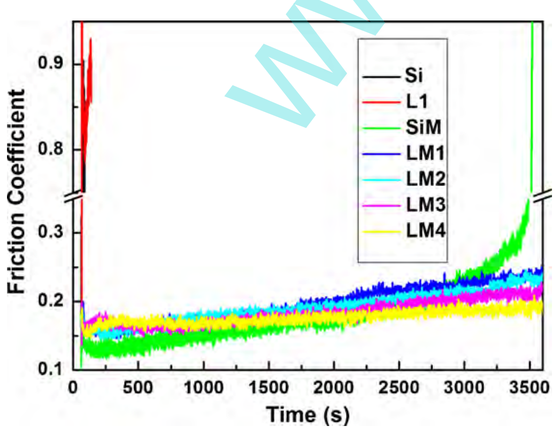

Fig. 7. Friction coefficients as functions of sliding time for the patterned silicon surfaces with different fractional surface coverage modified with MACs sliding against steel ball at normal load of 60 mN and sliding velocity of 30 mm/s, compared with L1, Si and SiM.

3.5. Microtribological study

Fig. 7 shows microfriction coefficient as functions of sliding time for the patterned silicon surfaces with different fractional surface coverage modified with MACs sliding against steel ball at normal

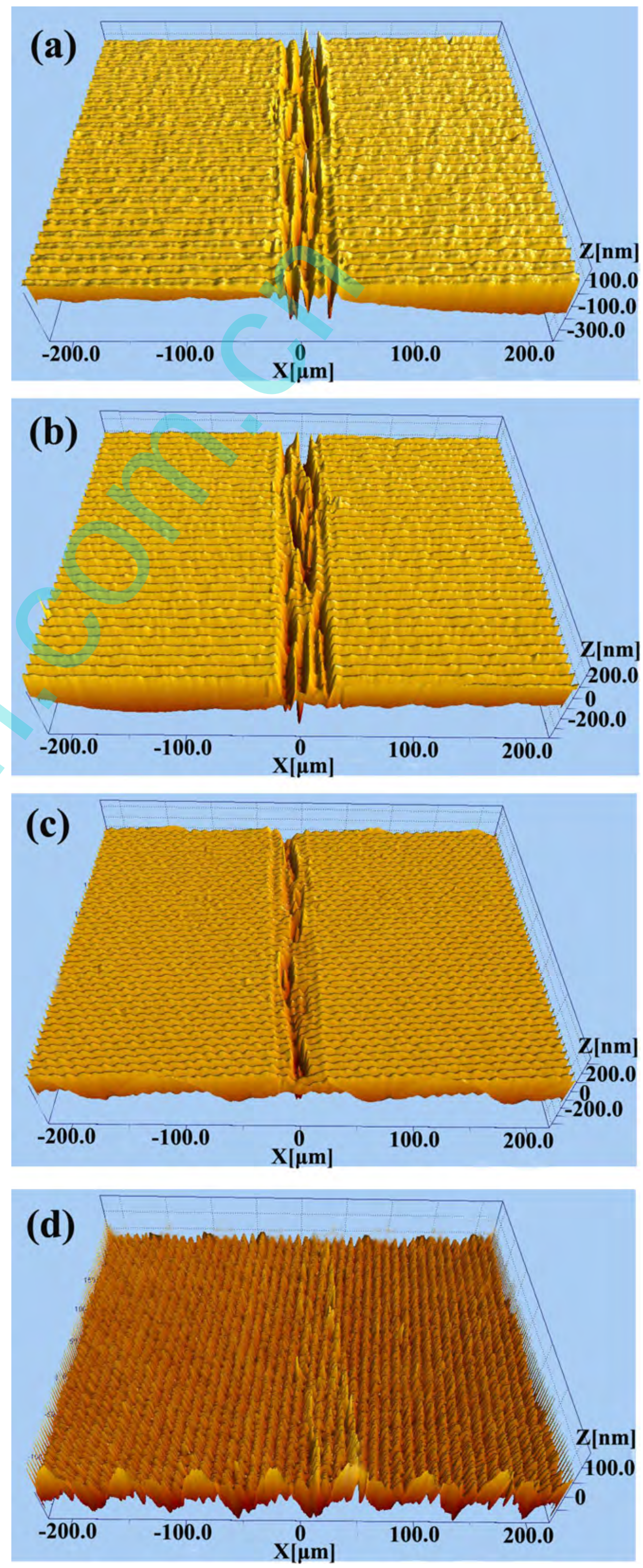

Fig. 8. 3-D images of the worn surfaces of LM1 (a), LM2 (b), LM3 (c) and LM4 (d). All tests were run against ball at load of 60 mN and a sliding rate of 30 mm/s for 1 h (442 µm × 128 µm).

load of 60 mN and a sliding velocity of 30 mm/s, compared with L1, smooth silicon and MACs covered smooth silicon (ascribed as SiM). As seen from Fig. 7, L1 and smooth silicon were worn out as soon as the counterpart ball began to slide on it, the microfriction coefficient continuously ascended to an average value of about 0.8. SiM was worn out before 3500 s as the counterpart ball slid on them, exhibiting poor wear resistance too. However, the antiwear abilities of the patterned silicon surfaces modified with MACs were much better. They can remain as an effective lubricant layer for more than 3600 s at a load of 60 mN and a sliding rate of 30 mm/s. And the microfriction coefficient decreased as groove area density increased.

The surface chemistry and real area of contact affect friction [33,39,42]. At first, the mobile hydrocarbon MAC lubricant was trapped and maintained among the groove network. Even though the mobile MAC molecules are mechanically disrupted or displaced by the contact, they can replenish to the lubricant-depleted area, which contribute to the improved antiwear abilities [43]. Then the real area of contact is dependent upon the area density [33,39,42]. With the same ball scan velocity, increasing of groove fractional surface coverage, the real area of contact is reduced greatly which lead to low friction coefficient.

To further clarify the wear resistance properties, the patterned silicon surfaces modified with MACs were all running against steel ball at the same condition of the applied load of 60 mN and sliding speed of 30 mm/s for 1 h. The worn surfaces were observed on a 3D non-contact profilometer, displayed in Fig. 8. As seen clearly in Fig. 8, there was severe scratch on the LM1 surface. The scratch was inhibited to some degree as groove area density increased and the wear on the LM4 surface was significantly reduced, and the scratch was nearly indistinguishable. This is consistent with the microfriction test that microfriction coefficient decreased as groove area density increased. The worn surface morphologies agree well with the corresponding microtribological properties that LM4 exhibited the best wear resistance during the samples in this study.

The introduction of specific textures on a sliding surface is an effective way to obtain improved tribological properties [44]. One function of the textures is to trap wear debris from the interface, reducing the ploughing and deformation components of friction [45]. Another important effect of the surface textures is to act as reservoirs for lubricants. Then they can feed the lubricant directly between the two contacting surfaces [46]. The nice performance of LM4 may be due to the increased groove fractional surface coverage and a better supply of lubricant within the contact zone. For LM1, LM2, and LM3, the distances between the grooves were probably too long to constantly provide this good supply within the contact zone.

4. Conclusion

In summary, MAC films on silicon surfaces with micro/nano grooves were prepared. The effect of nanoscale roughness (groove fractional surface coverage) and chemical treatment (MAC lubricant films) on the wetting properties and tribological performance of surfaces were investigated systematically. The surface hydrophilicity decreases with increasing of surface roughness and lowering of the surface energy (chemical treatment). The results also show that tribological performance could be largely improved by designing suitable surface topography combined with MAC lubricant films. With the increasing of nanoscale roughness and combined with chemical modification, the tribological and microtribological properties are both improved greatly, which could be affected by real area of contact and the surface chemistry. It is expected that this research could provide additional insight into the interesting combination effects of surface micro/nanohierarchical structures design and thin film lubricant, the principle of which could be applied to improve tribological properties of MEMS/NEMS.

Acknowledgements

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