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Influence of micro/nano-textures and chemical modification on the nanotribological property of Au surface

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ABSTRACT

Nanotribological properties play an important role in many applications, such as micro/nanoelectromechanical systems (M/NEMS) and high-density storage technologies (hard drivers). Therefore, it is important to study the nanotribological properties of these surfaces which are separated by only a couple of nanometers. Patterning feature or surface texture with micro/nano-scale dimension is of great importance for solving these problems. In this work, micro/nano-patterned Au surfaces with different topography features were fabricated via a convenient method and then chemically modified by alkanethiol SAMs for optimization of their nanotribological performance. Surface composition and morphologies of Au surfaces with different micro/nano-textures and chemical modification were evaluated by XPS, contact angle measurements, 3D non-contact optical microscopy and AFM. AFM/FFM was used to investigate the nanotribological behaviors of Au surfaces with different micro/nano-textures and corresponding chemical modification. Results obtained in this work demonstrated the feasibility of fabricating surface textures with micro/nano-scale cylindrical holes and the possibility of controlling chemical composition on surface to improve the nanotribologcal performance of Au surface. Nanotribological properties of textured Au surfaces were greatly determined by the fractional surface coverage of cylindrical holes and consequent chemical modification. Au textured surface with dense cylindrical holes and further chemically modified with self-assembly monolayer showed significantly enhanced hydrophobicity and nanotribological performance compared with Au textured surface with sparse cylindrical holes without chemical modification.

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1. Introduction

Smaller means better and unique—less expensive, more components per chip, faster response, higher performance, lower power consumption, and new types of functions. It is well known that many interesting phenomena also occur at nanometer scale [1,2]. During the past years, miniaturization has aroused great attention among worldwide scientists. Miniaturization and integration of a range of components and devices as well as the rapid development of micro/nanoelectromechanical systems (M/NEMS) have resulted in a plethora of nanotechnological applications [3].

As the size of device shrinks to micro- and nano-scales, the surface-to-volume ratio increases and consequently causes serious adhesive and frictional problems which are the major reasons for the failure of M/NEMS [4–6]. In order to solve these problems, development of new materials or design of surfaces and interfaces with hydrophobic, low adhesion and friction performance are of

great importance [7,8]. Low real area of contact and non-wetting (hydrophobicity) are two critical surface behaviors for materials or devices in micro/nano-scale applications.

Surface texturing is considered as an important method to decrease the adhesion and stiction in magnetic storage and M/NEMS devices [7–11]. The ability to generate nanostructures is of great importance for modern science and technology [10], which assures the miniaturization of functional devices, especially M/NEMS that normally have smooth surfaces and are subjected to small applied forces. The original surface topography of these devices is likely to be preserved due to these weak applied forces. Friction forces in these systems are affected mainly by surface forces, such as adhesion and electrostatic forces [4–6], which are largely dependent on the contact geometry and surface topography.

Replica molding has been developed in the past decade which is a relatively inexpensive, convenient, high efficiency, more importantly, an environmentally friendly route towards accurate patterning of different materials [12,13], Once the master is available, multiple copies of the pattern can be produced using straightforward experimental techniques. The procedure is remarkably convenient (see Fig. 1). An elastomeric PDMS stamp

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is used to transfer micro/nano-textures from the master which is fabricated by inductively coupled plasma (ICP) ehching method [14].

Self-assembled monolayers (SAMs) have been widely investigated in the past years because of their potential applications in the field of surface modification [15–17]. Ultra-thin SAMs as protective/lubrication molecular coatings are widely used in high-density storage technologies (hard drivers) and M/NEMS for controlling wetting, adhesion, friction and corrosion problems [5,15–18]. SAMs of octadecanethiol (ODT) on gold exhibit many of the features that are most attractive about self-assembling systems: ease of preparation, density of defects low enough to be useful in many applications, good stability under ambient laboratory conditions, practicality in technological applications and controllable interfacial properties of the system including physical, chemical, electrochemical, and biochemical properties [1].

In this work, various Au surface textures were created by using the micromolding technique and then chemically modified with ODT SAMs. A systematical study of the nanoscale friction and adhesion properties of various micro/nano-textures with different fractional surface coverage and chemical modification was present and the corresponding friction mechanisms were discussed. The investigation showed that surface textures with different fractional surface coverage influenced the nanotribological properties of the surfaces strongly. And SAMs also helped to significantly decrease the adhesion and friction forces of the Au surfaces. The objective of this study is to understand and aid the design and selection of appropriate micro/nano-textures for M/NEMS.

2. Experimental

2.1. Materials

Octadecanethiol (ODT) was purchased from Aldrich, 98%. PDMS prepolymer (Sylgard 184 Silicone Elastomer Kit, Dow Corning, Midland, MI, USA) was purchased from Kanku Company (Shanghai, China). Both of acetone and anhydrous ethanol were analytical reagents. All reagents were used as received.

2.2. Preparation methods (replica molding)

Textured surfaces with cylindrical pillars of different fractional surface coverage were fabricated by the ICP etching technique. The fabrication process was illustrated in previous articles [19–21]. The silicon template morphology obtained by 3D-non contact optical profilemeter is shown in Fig. 1. These structures would then be duplicated into multiple copies by replica molding with organic polymers. This technique has also been adapted for the fabrication



Fig. 2. Schematic illustration of the process used to generate Au nanometer scale textures: (a) a pattern is produced by ICP etching method, (b) spin-coating of PDMS on the silicon template, (c) fabricating PDMS pattern and (d) Au surface with nanotextures.

of topologically complex, optically functional surfaces that would be difficult to fabricate with other techniques. Schematic illustration of the process used to generate nanometer-scale textures is shown in Fig. 2. A curing agent and the PDMS prepolymer were thoroughly mixed in a 1:10 weight ratio. After 10 min manual mixing the prepolymer mixture was degassed in a desiccator at room temperature for about 3 h to remove any air bubbles in the mixture and ensure complete mixing of the two parts. The micromachined silicon master was cleaned with acetone, ethanol, and distilled water in that order put in an ultrasonic bath. Then the PDMS mixture was poured onto the micromachined silicon master. The sample was left to cure for at least 24 h before peeling the polymer off from the silicon wafer. After the PDMS film solidified, the negative surface texture of the original template was transferred to the PDMS film. Then samples were sputter coated with a thin layer of gold (20 nm) and cut into small pieces for testing.

2.3. Surface chemical modification

Chemical modification of the topmost surface layers of the Au was done by further SAMs treatment. The patterned Au surfaces were immersed in 5 mmol/L anhydrous ethanol solutions of ODT and held for 24 h. ODT molecules chemisorbed spontaneously on gold surface from solution and formed uniform SAMs, sulfur atoms bonded to the gold surface bring the alkyl chains into close contact [1]. Samples were taken from the coating solution and were put in an oven maintained at 70 °C for 3 h. Then the samples were washed by sufficient anhydrous ethanol to remove the physical adsorbed molecules and dried under a flow of N_2 .



Fig. 1. 2D (a) and 3D (b) morphologies of silicon template used in this article.

2.4. XPS spectroscopy

XPS is a highly diagnostic tool for the assessment of the chemical state of elements. In this paper, a PHI-5702 multifunctional X-ray photoelectron spectrometer (Physical Electronics Inc., USA) was conducted to determine the chemical composition of the SAMs on Au surface, using Mg-K α radiation as the exciting source. The binding energies of the target elements were determined at a pass energy of 29.35 eV, with a resolution of about ± 0.3 eV. Electron binding energies were calibrated using the contaminated carbon (C1s: 285.0 eV)

2.5. Contact angle measurements

Contact angle measurements (sessile drop method) were performed using a DSA100 type contact angle meter (Kruss Company Ltd., Germany). An autopipetting system was used to deliver a fixed volume of liquid of 5 μ L for static measurements. The water droplets were imaged by a CCD camera before the included software calculated their shape and the respective contact angles. At least five points were measured for each specimen, and the measurement error was $\pm 2^{\circ}$.

2.6. Surface morphology, nanoadhesion and nanofriction measurements

AFM has become a viable tool in the characterization of friction forces at the nano-scale, because of the reduced dimensions of the contact area and of the small loads between the tip and the surface. A silicon tip that slides on a silicon surface is a suitable model system for the sliding behavior in M/NEMS [22,23]. In this work, AFM has been employed to study the morphologies of micro/nanotexture Au surfaces, because not only it has great vertical resolution but also it allows the measurement of other interesting parameters which can help us to find more value information about films, such as roughness, pattern size and surface cross-section. Adhesion and friction measurements were performed by an AFM/FFM controlled by CSPM4000 electronics (Being Nano-Instrument, China), using the contact mode. The horizontal resolution of the AFM device is 0.26 nm, and vertical resolution is 0.1 nm. The loading force was kept constant during the friction and adhesion between different scanning. Commercially available rotated monolithic silicon probe, symmetric tip shape (Budget Sensors, Multi75Al-G-10, 30 nm thick Aluminum coating) with a nominal spring constant of 3 N/m and a coated tip with a curvature radius of less than 10 nm was employed. Friction forces were obtained from friction-load line at 100 separate points on each surface with a scan velocity of 120 µm/s. The output voltages were directly used as frictional forces. No attempt was made to calibrate the torsional force constant.

Adhesive force (*F*) which is also called pull–off force were got from force-distance curve, and was calculated by, $F = K_c Z_p$ [24,25], where K_c is the force constant of cantilever and Z_p is the vertical displacement of the piezotube. For all measurements, the same tip was used in this study. All the measurements were performed at room temperature and with a relatively humidity of 30–40%. Repeated measurements were within 5% of the average value for each sample.

3. Results and discussion

3.1. Parameters of nano-texture Au surfaces

2D, 3D and line section analysis AFM topographic images of various patterned Au surfaces were depicted in Fig. 3. As seen from Fig. 3, the Au textures were created successfully which

have the same depth (50 nm) and diameter (2 μ m) but different fractional surface coverage. Table 1 lists the parameters of micro/nano-texture Au surfaces generated in this investigation. For convenience, we ascribe the micro/nano-texture Au surface as HAu and micro/nano-texture Au surface chemically modified by ODT SAMs as HS, respectively. And also we abbreviate micro/nano-texture surfaces with different cylindrical hole fractional surface coverage from low to high as HAu1, HAu2, HAu3, and HAu4. Correspondingly, Au surfaces with micro/nano-textures from low fractional surface coverage chemically modified by ODT SAMs are ascribed to HS1, HS2, HS3, and HS4.

Hole fractional surface coverage r (%) was determined by the following equation:

$$r(\%) = \frac{NS_{\text{hole}}}{S_{\text{scan}}} = \frac{N\pi R^2}{S_{\text{scan}}}$$

where N is the number of hole, R is the radius of hole and S_{scan} is the AFM scan area.

According to above calculation, the hole fractional surface coverage r (%) from low to high is 2.5%, 4.5%, 9%, 25.2%, respectively. As seen from Table 1, surface roughness increased as hole fractional surface coverage increased.

3.2. XPS spectroscopy: formation of octadecanethiol SAMs

XPS is a powerful tool to clarify the chemical states of elements within boundary film on surface. Fig. 4a gives XPS spectra of PDMS, Au on PDMS surface and Au surface chemically modified with ODT SAMs. It could be seen that the XPS peak of Au appears at 87.31 eV, which was assigned to metal Au. Thus it can be concluded that Au was deposited on the PDMS surface to form Au texture surface, which had regular cylindrical holes with different spacing interval. XPS peak of S only appeared at HS surface which implied that ODT SAMs was successfully formed on Au texture surface. Fig. 4b shows the high-resolution XPS spectrum of S2p region of the ODT SAMs film. The S2p spectrum of the ODT positioned at 162.53 eV was assigned to the bound S atoms [26]. S2p spectrum did not have additional shoulder which suggested that a uniform monolayer did form on Au texture surface and all of the molecules were chemisorbed on the Au surface [27].

3.3. Contact angle measurements

Contact angle measurements were made to further characterize the surface wetting/dewetting properties and the results were depicted in Fig. 5. It was observed that contact angles increased with increased hole fractional surface coverage with the same surface chemistry, such as the contact angle of HAu4 was larger than HAu1, and the same phenomenon was found on Au surfaces chemically modified with HS SAMs. In other words, the surfaces with smaller spacing between the cylindrical holes had even higher contact angles. And also the contact angles of HS surfaces were larger than HAu surfaces.

It is well known that the contact angle is determined by two factors, such as the surface topography and the surface chemistry [28]. For surface topography, decreasing the spacing between two adjacent micro/nano-cylindrical holes will increase surface roughness which results in higher contact angles. The surface composition of the texture surfaces was modified by ODT SAMs treatment. After chemical treatment the contact angles of all the Au surfaces with micro/nano-textures increased nearly 40°. This is probably due to ODT SAMs has terminal groups ($-CH_3$), which are hydrophobic, would lead to higher contact angle [5,6,29]. By controlling these factors and varying them independently, we can design suitable



Fig. 3. AFM topographic images of patterned Au surface: (a, d, g and j) 2D AFM images and (b, e, h and k) line section analysis of (a, d, g, j). (c, f, i and l) 3D AFM images.

Table 1

Name	Hole depth, <i>d</i> (nm)	Hole interval, <i>i</i> (µm)	Surface roughness rms (nm)	Hole fractional surface coverage, $r(\%)$
HAu1	50	9	29.2	3
HAu2	50	8	35.2	5
HAu3	50	6	47.4	8
HAu4	50	3.5	72.1	26



Fig. 4. XPS spectra: (a) PDMS, Au and ODT SAMs and (b) S2p region of ODT SAMs on Au patterned surface.



Fig. 5. Static contact angle measurements: (a) native Au patterned surfaces and (b) Au patterned surfaces with SAMs chemical modification.

surface for special M/NEMS application.

3.4. Nanofriction properties

Nano-tribological properties of the Au surface with different micro/nano-textures and chemically modified with ODT SAMs were examined by AFM/FFM, and the results are depicted in Fig. 6. The friction force was given here in the form of voltage signal, which should be proportional to the real friction force [15]. Therefore, the results from various surfaces could be compared with each other. As seen from Fig. 6, friction force decreased as cylindrical hole frac-



Fig. 7. Plots of adhesive forces between AFM tip and surfaces of bare Au nanotextures and SAMs chemical modification Au nano-textures with different surface coverage, at a relative humidity of 30–40%.

tional surface coverage increased, and also reduced greatly after chemical modification. HAu1 showed the largest friction force and HS4 showed the smallest friction force among the surfaces investigated in this work.

In general, two important factors which control friction at nanoscale in dry/wet contacts are the real area of contact and surface chemistry [7,30]. Firstly, the real area of contact is dependent upon the micro/nano-texture fractional surface coverage and mechanical properties of material surfaces [8–10]. With increased fractional surface coverage, 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 energy is higher, it is easily to form meniscus by them or the adsorbed water molecules, which would lead to larger shearing strength and higher friction. In other words, if the surface energy is lower it would get opposite result. ODT SAMs made the surface more hydrophobic leading to lower surface energy, which also confirmed by contact angle measurement, so the friction force of HS4 is the lowest among all the surfaces.

3.5. Nano-adhesion properties

The adhesive forces between the AFM tip and micro/nanotexture surfaces are summarized in Fig. 7. As shown in Fig. 7, HS4 showed the lowest adhesion force but HAu1 showed the largest adhesion force. Both surface textures and chemical modification had strong influence on the adhesion force.



Fig. 6. Plots of friction forces between AFM tip and patterned surfaces with different surface coverage, at a relative humidity of 30–40%: (a) bare Au nano-textures and (b) Au nano-textures with SAMs chemical modification.

Adhesive forces come from two sources: contact interfacial forces and noncontact forces such as Van der Waals, electrostatic forces and capillary/meniscus forces [8,30]. Firstly, the contact area between the tip and micro/nano-texture surfaces becomes small due to the significantly increased cylindrical hole fractional surface coverage, the reduced contact area resulting in more significant reduction of adhesion force [8-10]. Secondly, when the solid surfaces are hydrophilic, they can easily form meniscus by the adsorbed water molecules, thus they show higher adhesive force. However, when the surfaces are hydrophobic, they would show low adhesion [5,6,29,31]. The presence of ODT SAMs increases the hydrophobicity of micro/nano-texture Au surfaces, which also leads to the reduction of adhesion force in a humid environment. Therefore, at the micro/nano-scale when adhesive forces are significant, surface textures and chemical modification were commonly used to help lower adhesion by reducing the real area of contact and increasing the surface hydrophobicity.

4. Conclusions

Development of novel, simple, and reproducible patterning strategies to fabricate micro/nano-structures in a great variety of materials like polymers, metals, and semiconductors has been paid growing interest in recent years. In this work, the effect of surface textures and chemical modification on the nanotribological properties of Au surface has been investigated using an AFM/FFM. The results showed that patterned surfaces with higher fractional surface coverage and surface roughness, exhibited lower adhesion and friction forces. And adhesion force and friction force reduced greatly after chemical modification with ODT SAMs. Therefore, creating micro/nano-textured surfaces with suitable chemical treatment by SAMs have greater potential to reduce catastrophic failures in MEMS/NEMS devices involving ultra smooth contacting surfaces.

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