



ELSEVIER

Contents lists available at SciVerse ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

Preparation and micro-tribological property of hydrophilic self-assembled monolayer on single crystal silicon surface

Zhixin Kang*, Qin Liu, Yinghui Liu

National Engineering Research Center of Near-Net-Shape Forming for Metallic Materials, School of Mechanical & Automotive Engineering, South China University of Technology, Guangzhou 510640, China

ARTICLE INFO

Article history:

Received 10 October 2012

Received in revised form

24 March 2013

Accepted 25 March 2013

Available online 4 April 2013

Keywords:

Sliding wear

Micro-scale abrasion

Solid lubricant coatings

Surface topography

ABSTRACT

A kind of nano-film was prepared by self-assembling 6-(3-triethoxysilylpropylamino)-1,3,5-triazine-2,4-dithiol monosodium (TES) on the surface of single crystal silicon with hydroxylated pretreatment. The features of the self-assembled monolayer (SAM) were characterized by contact angle meter, spectroscopic ellipsometer, atomic force microscope, X-ray photoelectron spectroscopy and infrared spectroscopy. The experimental results show that the contact angle of distilled water for self-assembled monolayer (SAM) was 35.5° and the film thickness was 3.7 nm, and orderly directional nano-clusters were covered on the surface by observation of the topography. Besides, the friction and wear properties of the SAM were investigated under micro-load by universal micro-tribotester. Compared with the substrate, the friction coefficient of the SAM was effectively reduced from 0.494 to 0.102, which was due to the lubricant of the SAM coating. Meanwhile, the film was not destroyed until 130 cycle times of reciprocating gliding under load of 10 g. The friction and wear tests show this hydrophilic SAM also has a lubrication effect on the surface while other SAMs often exhibited hydrophobic properties. It is expected that the hydrophilic SAM will be applied in micro electromechanical systems as the solid lubricant coatings.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Micro electromechanical systems (MEMS) and emerging nano electromechanical systems (NEMS) have influenced our lives widely [1,2]. Due to their superior performances and low cost, MEMS/NEMS are used in nanotechnology, electromechanical, electronics, information, communication, chemical and biological field [3,4]. However, when the size of devices decrease to micro or even nano scale, as a result of the large surface area to volume ratio, tribological issues such as stiction, friction and wear would be the major reasons lead to the reduction of the performances and the operating lifetimes of MEMS/NEMS devices [4–7]. MEMS/NEMS require a protective lubricant coating with low friction coefficient and high durability in order to improve the tribological performances and these account for the increased attention to the polymer thin films [8–10].

Different compounds with triazine dithiol substituent group were successfully polymerized durable functional organic nano films with low adhesion force and surface energy on metal substrates, which can be applied in anticorrosion, adhesion [11–13], and well tribological properties were displayed on magnesium alloys [14]. Self-assembled monolayer (SAM) considerably reduces friction and adhesion and has been used in various MEMS/NEMS

devices [15–17]. Silicon and Silicon compounds are the most important materials in MEMS/NEMS.

Many types of SAM revealed hydrophobic properties which were utilized to reduce the friction coefficient of MEMS for their lower surface free energy compared with hydrophilic surfaces [5,18]. However, many hydrophobic SAM solutions were poisonous, the solute such as fluoroalkyl silane, octadecyltrichlorosilane (OTS), and the solvent usually was toluene, hexane or hexadecane, which were all harmful to the body [16,18]. In our work, we developed an alkoxysilane with the function terminal group triazine dithiol which was a white innocuous solid power, and the solvent was ethanol and distilled water.

The alkoxysilane was used for self-assembled and demonstrating that surface free energy was not the necessary conditions to reduce the friction coefficient and improve the anti-wear properties. In the paper, we expect to provide a novel approach to tribological issues in MEMS, through the study of forming process mechanism and the micro-tribological behavior for this SAM film.

2. Experimental details

2.1. Materials

N-type single crystal silicon wafers {100} polished on one side were cut into pieces of approximately $1\text{ cm} \times 1\text{ cm}$ and then used

* Corresponding author. Tel.: +86 20 87113851; fax: +86 20 87112111.
E-mail address: zzkang@scut.edu.cn (Z. Kang).

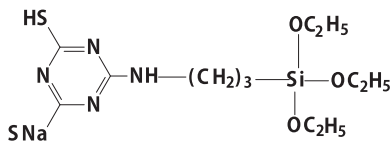


Fig. 1. Structure of the alkoxy silane TES.

for the surface modification. The triazine dithiol compound 6-(3-triethoxysilylpropylamino)-1,3,5-triazine-2,4-dithiol monosodium (TES) with alkoxy silane substitute group was specially synthesized in our lab and the structure is shown in Fig. 1. The self-assemble solution was prepared by dissolving 3 mmol TES monomer into 1 l mixture solvent (9:1 (v/v) of ethanol and distilled water), and the solution was referred to SAM solution. The samples were degreased by ultrasonic washing in acetone, following washed by ethanol and dried in cold air; then the samples were hydroxylated by immersing in piranha solution (a mixture of 7:3 (v/v) 98% H_2SO_4 and 30% H_2O_2) at 90 °C for 50 min, and immediately rinsed with distilled water, ethanol and dried in cold air; thirdly the samples were soaked in SAM solution for 15 min; afterwards, the wafers were washed sequentially with distilled water and ethanol and dried in cold air; finally, the wafers were placed in drying oven at 150 °C for 4 h. The film was short for TES-SAM. Analytical reagent grade chemicals were used throughout the present study.

2.2. Measurements

Static contact angles of distilled water on the unmodified and modified surfaces were measured with an optical contact angle meter (OCA35, Dataphysics) at ambient temperature, and a water droplet of 2 μl was used for contact angle measurement. TES-SAM film thickness was measured by spectroscopic ellipsometer (UVI-SEL-NIR-FGMS, Horiba Jobin Yvon). The surface topography was observed by atomic force microscope (AFM, Nanoscope III Scanning Probe Microscope, Veeco Metrology Group) with contact mode. X-ray photoelectron spectroscopy (XPS, AXIS Ultra, Kratos) analyses were carried out by using a monochromatic Mg $K\alpha$ X-ray source (1253.6 eV), the binding energy was calibrated using the C 1s peak energy (284.6 eV) as an energy standard. Fourier transform infrared spectroscopy (FT-IR, IR Prestige-21, Shimadzu) was used to determine the chemical functional groups. Friction and wear tests were carried out on UMT-2 (Universal Micro Tribotester, CETR) using ball-on-disk mode, and a 440c stainless steel ball of 4 mm diameter was used as the counterface. The normal load used was 10 g, which gave a contact pressure of approximately 386 MPa (calculated using the Hertzian Contact Model). Although the typical contact pressures in MEMS are in the order of few megapascals (or less than 1 MPa) [19,20] we set the contact pressure at 386 MPa to shorten the duration of the wear tests. And the friction and wear tests were conducted at room temperature about 27 °C and relative humidity about 25%~40%. The optical micrographs of wear tracks were observed by 3D super depth digital microscope (VHX-600, Keyence).

3. Results and discussion

3.1. Preparation and characterization of TES-SAM

There are significant differences of the surface features between Si substrate (after hydroxylation, similarly hereinafter) and TES-SAM, including contact angles, surface free energy and film thickness. The contact angle of TES-SAM is 35.5° while Si substrate shows a contact angle of 16.9°, and the thickness of TES-SAM is 3.7 nm. Surface free energy (SFE) was calculated by OWRK

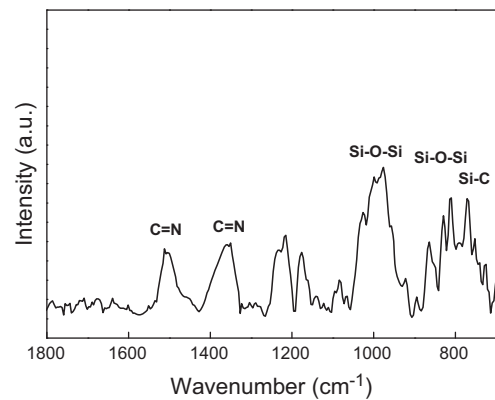


Fig. 2. FT-IR spectrum of TES-SAM on silicon surface.

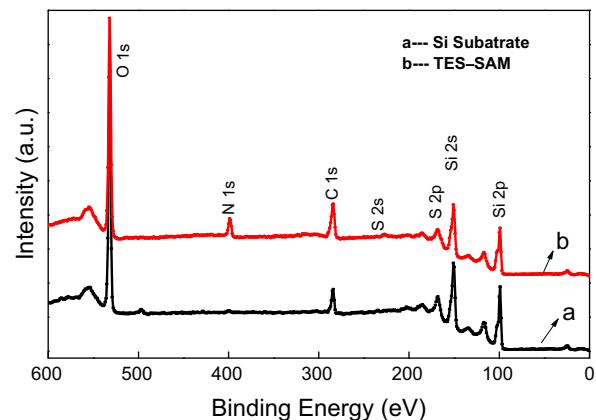


Fig. 3. XPS spectra of TES-SAM and Si substrate.

algorithm [21] shown in Eq. (1) with the contact angles of two different liquids, distilled water and diiodomethane.

$$\frac{(1 + \cos\theta)\gamma_l}{2\sqrt{\gamma_l^D}} = \sqrt{\gamma_s^D} \sqrt{\frac{\gamma_l^D}{\gamma_l^P}} + \sqrt{\gamma_s^D} \quad (1)$$

where γ_s and γ_l are the surface free energy (or surface tension) of the solid and liquid, γ^D and γ^P are the dispersion component and polar component of surface free energy (or surface tension). The SFE of TES-SAM was 61.33 mJ/m^2 . The higher SFE and hydrophilicity of TES-SAM are corresponding to the functional terminal group triazine dithiol and the feature of thiol group (-SH).

The variation of surface characteristics after modification successfully reveals the formation of TES-SAM. In order to confirm TES-SAM chemical structure and self-assembling mechanism, FT-IR spectroscopy and XPS were used to analyze surface conditions. FT-IR spectra of TES-SAM presented in Fig. 2 displays obvious absorption peaks at about 1509 cm^{-1} and 1360 cm^{-1} which are attributed to the presence of C=N bonds and confirmed to be related to the triazine ring in TES monomer [22]; the absorption peaks at about 1025 cm^{-1} and 813 cm^{-1} can be ascribed to asymmetric and symmetric stretching vibrations of Si-O-Si bond [23]. The FT-IR spectra results suggest that the TES-SAM film was successfully formed on Si substrate surface.

The formation of SAM can be also proved by XPS spectra shown in Fig. 3. Compared to Si substrate, the spectrum of TES-SAM contained N and S elements from TES monomer were detected, the presence of lesser intensity C 1s peak on the Si substrate might be due to the organic contaminants that were not removed during the piranha treatment or adsorbed from the atmosphere during

Download English Version:

<https://daneshyari.com/en/article/7004864>

Download Persian Version:

<https://daneshyari.com/article/7004864>

[Daneshyari.com](https://daneshyari.com)