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Sensors and Actuators A 128 (2006) 98–108
SENSORS AND ACTUATORS A PHYSICAL
www.elsevier.com/locate/sna

Tribology of a novel UHMWPE/PFPE dual-film coated onto Si surface

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Received 1 July 2005; received in revised form 14 December 2005; accepted 17 December 2005
Available online 26 January 2006

Abstract

A novel wear resistant polymer composite layer is coated onto the Si surface. The first layer consists of ultra-high molecular weight polyethylene (UHMWPE) film and the second top layer is made of perfluoropolyether (PFPE). These two layers are coated onto Si using a simple dip-coating technique. This particular combination of dual-film has reduced the coefficient of friction by at least 6 times and increased the wear resistance of the Si surface by at least 1000 times with very minimum to no sign of wear debris formation. The tribological tests are carried out using ball-on-disk tribometer employing 4 mm diameter Si_3N_4 ball as the counterface at a nominal contact pressure of ∼370 MPa. The UHMWPE/PFPE dual-film provides hydrophobic surface with very high water contact angle (134°). Finally, this dual-film may find applications in reducing wear and stiction of micro-components (such as micro-electro-mechanical systems) made from Si.

Keywords: UHMWPE film; PFPE; Thin-film lubrication

1. Introduction

Si is the basic material from which many electronic and electromechanical components are made because of the well-matured technology of manufacturing Si components by the wet-etching process [1]. Si is generally not considered a good tribological material because it experiences high friction, adhesion and wear during sliding [2]. Hence, there is a great search for organic or inorganic coatings and lubricants for the improvement of tribological properties of Si surface [3–16]. Many of these coatings demonstrate low initial friction, but only few demonstrate high wear durability at higher normal stresses in a contact wear test [6,7,9–11,13]. The problem of wear of Si is still not fully solved and many potential coatings/lubricants are needed to be developed and tested to protect the components made out of Si so that they can run up to several millions of cycles without any interruption to the smoother operation of the device [17]. Moreover, there exists a need for an efficient and cost-effective method of applying lubricants and coatings onto the actual components (e.g. micro-electro-mechanical systems (MEMS) components) made of silicon which are three-dimensional and often of intricate shapes [18].

In the present study, a novel polymer composite dual-film, consisting of ultra-high molecular weight polyethylene (UHMWPE) as the first layer and perfluoropolyether (PFPE) as the second top layer, is coated onto the Si surface and its tribological properties such as friction and wear are investigated. This composite dual-film is coated onto Si using dip-coating technique. It has been identified (and hence selected) that the polymer coatings onto the Si surface can provide good wear durability because of their inherent properties such as low surface energy, molecular flexibility, high toughness coupled with good strength and damping characteristics and their lower shear strength [19]. One of the novelties of the present work is the establishment of a dip-coating method to apply thin (sub-micron) UHMWPE film onto Si surface. Such polymers are often hard to coat on a substrate due to their non-reactive and hydrophobic nature and often expensive thermal [20] or vapor depositional techniques [21] may be necessary.

In the bulk form, UHMWPE is highly wear resistant compared to many other polymers such as polyetheretherketone (PEEK), polyethylene (PE), polystyrene (PS), polymethyl-

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doi:10.1016/j.sna.2005.12.042

Table 1
Physical properties of the UHMWPE used in the present study

Property	Units	Value
Melt index MFR 190/15	G/10 min	1.8 ± 0.5
Bulk density	g/cm^3	0.33 ± 0.03
Average particle size d_{50}	μm	20 ± 5

methacrylate (PMMA), etc. [22]. Therefore, UHMWPE is selected and coated as a thin film onto Si surface by dip-coating technique. To further enhance tribological properties of the UHMWPE film, PFPE is coated as a second layer onto it. PFPE is selected as the top layer because of its properties such as low surface tension, chemical and thermal stability, low vapor pressure, high adhesion to the substrate and good lubricity [23]. In a previous study, we found that the PFPE overcoating onto organosilane based self-assembled monolayer (SAM) surfaces resulted in increased wear resistance and low coefficient of friction [24].

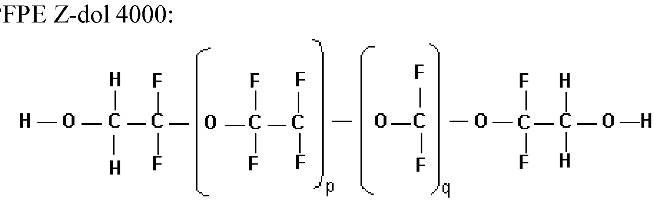
Many of the studies on the measurements of friction of ultra-thin films have been carried out using atomic force microscope/lateral force microscope (AFM/LFM). However, major limitations of AFM are their lower sliding speed (μm/s) and smaller contact areas which do not represent the actual sliding conditions of a microsystem such as MEMS. Therefore, in the present study, we have used a ball-on-disk type tribometer which has realistic sliding contact area (with track width in the range of a few hundred microns) and is capable of measuring friction and wear at higher sliding speeds (m/s). Further, long term sliding tests can be conducted on a tribometer within a reasonable time frame.

2. Experimental

2.1. Materials

Polished single crystal silicon (1 0 0) wafers were used as the substrate. These Si wafers were cut into coupons of approximately 2 cm × 2 cm size. UHMWPE polymer powder (Grade: GUR X143) was supplied by Ticona Engineering Polymers, Germany through a local Singapore supplier. The physical properties of UHMWPE, as provided by the supplier, are listed in Table 1. After several initial trials, decahydronapthalin (Decalin) was selected as the solvent to dissolve UHMWPE. PFPE (Zdol 4000, molecular weight = 4000 g/mol, monodispersed) was used to coat onto the Si/UHMWPE surface. PFPE molecules have terminal OH groups at their ends. The chemical formula of the PFPE used is as follows:

PFPE Z-dol 4000:



where the ratio p/q is 2/3. Hydrofluoropolyether solvent (H-Galden ZV) purchased from Ausimont INC was used as the solvent for PFPE. Acetone (99.8%), sulfuric acid, hydrogen peroxide and distilled water were also used for the sample preparation.

2.2. Preparation of UHMWPE/PFPE dual-film on Si surface

Si substrate was cleaned and treated with piranha solution according to the standard procedure reported in a previous publication [24].

The initial step in the preparation of the UHMWPE films was the dissolution of the UHMWPE polymer powder into Decalin. The dissolution was carried out at higher temperature of 170 °C which resulted in complete and uniform dissolution of the polymer. Magnetic stirring was used during heating in order to enhance the rate of dissolution. After ensuring the complete dissolution of the polymer powder, dip-coating was carried out almost immediately. A custom built dip-coating machine was used. The dip-coating was carried out at a constant dipping and withdrawal speed of 2.1 mm/s and an intermediate soaking time of approximately 30 s. After dip-coating, the samples were slightly dried in the air and then heated at 100 °C for approximately 20 h in clean air furnace. After thermal treatment, the samples were cooled to the room temperature at very slow cooling rate (furnace cooling). The samples were then stored in desiccator until further tribological and surface characterization.

PFPE was coated onto the UHMWPE modified Si substrates according to the procedure mentioned in our previous publication [24].

2.3. Depositional characterization and surface analysis

VCA Optima Contact Angle System (AST Products, Inc., USA) was used for the measurement of static contact angles for distilled water on the unmodified and modified Si surfaces. A water droplet of 0.5–1 μl was used for contact angle measurement. At least five to six replicate measurements, for three different samples, were carried out and an average value was taken. The variation in water contact angle values, at various locations of a sample was within ±2°. The measurement error was within ±1°.

FTIR spectrum for UHMWPE film was obtained in air on a Bio-Rad FTIR model 400 spectrophotometer using transmission mode. This spectrum was collected by accumulating 16 scans at a resolution of 4 cm^{-1}. The spectra were collected from at least five to six replicate points and they were found to be identical at all points of measurement. Bare Si, after piranha treatment, was used for background scan.

The chemical state of the bare Si, Si/UHMWPE and Si/UHMWPE/PFPE surfaces was studied by X-ray photoelectron spectroscopy (XPS). Measurements were made on a Kratos Analytical AXIS HSi spectrometer with a monochromatized Al Kα X-ray source (1486.6 eV photons) at a constant dwell time of 100 ms and pass energy of 40 eV. The core-level signals were obtained at a photoelectron take-off angle of 90° (with respect to