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Scripta Materialia 57 (2007) 821–824

Scripta MATERIALIA

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# Wear detection of candidate MEMS/NEMS lubricant films using atomic force microscopy-based surface potential measurements

Manuel Palacio and Bharat Bhushan*

*Nanotribology Laboratory for Information Storage and MEMS/NEMS (NLIM), The Ohio State University, 201 W 19th Avenue, Columbus, OH 43210, USA*

Received 1 June 2007; accepted 3 July 2007
Available online 2 August 2007

The wear of a perfluoropolyether lubricant applied on silicon was investigated using atomic force microscopy-based surface potential measurements. An increase in surface potential is attributed to the change in the work function of silicon after wear and electrostatic charge build-up of debris in the lubricant. Thermally treated coatings containing a mobile lubricant fraction (i.e. partially bonded) were better able to protect silicon from wear compared with the fully bonded and untreated coatings. This enhanced protection is attributed to lubricant replenishment.

*Keywords:* Wear; Perfluoropolyethers; Lubricants; Surface potential; Atomic force microscopy

The commercialization of products based on microelectromechanical systems (MEMS) and nanoelectromechanical systems (NEMS) relies on a better understanding of the various mechanisms that control the performance and failure of these devices. Adhesion, friction and wear at the nanometer size scale become critical and can be detrimental to the efficiency, power output and reliability of MEMS/NEMS devices [1,2]. For example, adhesion is the major cause of the failure of accelerometers used in automobile air bag triggering mechanisms and in micromirror components of commercial digital light processing (DLP) equipment [3–5]. Wear has been found to compromise the performance of NEMS-based atomic force microscopy (AFM) data storage systems [6]. In order to improve tribological performance, lubricants are applied to the MEMS/NEMS device surfaces. The ideal lubricant should be molecularly thick, easily applied, able to chemically bond to the micro/nanodevice surface, insensitive to environment, and highly durable [7–9].

Perfluoropolyether (PFPE) lubricants are considered as potential candidates for MEMS/NEMS applications. PFPEs are used in the magnetic disk drive industry to reduce friction and wear of the head–disk interface and diminish disk failure [10,11]. For MEMS/NEMS devices, PFPEs are desirable because they possess the following: low surface tension and high contact angle, which allow easy spreading on the applied surface and provide hydrophobicity; chemical and thermal stability, which minimize degradation under use; low vapor pressure, which provides low out-gassing; high adhesion to substrate via organofunctional bonds; and good lubricity, which reduces the contact surface wear.

Atomic force microscopy (AFM) is well suited to characterize the changes that occur during sliding of MEMS/NEMS because it has the ability to measure material properties with high spatial resolution. An atomic force microscope, which is a typical example of a scanning probe technique, detects minute forces between the sample surface and the scanning tip attached to a cantilever [12,13]. Martin et al. [14] and Weaver and Abraham [15] demonstrated that an AFM configured as a Kelvin probe microscope can be used to create surface potential maps with lateral resolutions around 50 nm and voltage resolution in the microvolt scale. The surface potential pertains to the potential difference between two surfaces as they are brought together in electrical contact and thermal equilibrium is achieved.

Surface potential mapping is a useful tool for wear detection. DeVecchio and Bhushan [16] have shown that a surface potential change is detectable even in cases where there is little or no surface damage, making this a powerful technique for studying the initiation of wear. The technique is suitable for wear studies of MEMS/

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* Corresponding author. Tel.: +1 614 292 0651; fax: +1 614 292 0325; e-mail: Bhushan.2@osu.edu

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doi:10.1016/j.scriptamat.2007.07.005

NEMS device components because it has also been used for characterizing the wear of metals, ceramics and a conventional PFPE lubricant [17,18]. This technique has also been used to elucidate the mechanism of triboelectric charging of human hair [19,20].

In this study, AFM-based surface potential measurements before and after wear testing are presented for silicon substrates coated with the novel PFPE lubricant Z-TETRAOL (Solvay Solexis Inc.). The evolution of wear and the effect of lubricant preparation on the surface potential are investigated as well. Z-TETRAOL, a perfluoropolyether with two hydroxyl groups at each end, is a derivative of the conventional lubricant Z-DOL, which has the same backbone but contains only one hydroxyl group at each end. It has been shown that a surface coated with Z-TETRAOL is more wear resistant than one coated with Z-DOL and this enhanced durability is attributed to the endgroup composition difference [21].

The lubricants were applied on single-crystal Si(1 0 0) (phosphorus doped) with a native oxide layer on the surface using the dip coating technique. The method and the apparatus used have been described elsewhere [7,8]. Briefly, silicon substrates with dimensions of $10 \times 10 \times 0.5\ \text{mm}^3$ were cut from Si(1 0 0) wafers. The cut wafer was then vertically submerged into a beaker containing a dilute solution of 0.1% Z-TETRAOL in HFE 7100 (Methoxynonafluorobutane, 3M, St. Paul, MN) for 10 min. The silicon wafers were pulled up from solution with the aid of the motorized stage set at constant speed to obtain films of desired thickness. The lubricated sample used without post-thermal treatment is referred to as untreated. Partially bonded samples were prepared by heating at 150 °C for 30 min after dip coating, while the fully bonded samples were heated at 150 °C for 30 min and washed in the HFE 7100 solvent to remove the mobile fraction. The lubricant-coated silicon samples were then measured with an ellipsometer and the coating thickness was found to be about 1, 3 and 7 nm for the fully bonded, partially bonded and untreated samples, respectively.

With the AFM in contact mode, wear tests were conducted on the specimen surface using a diamond tip with an apex angle of 60° and a tip radius of about 100 nm attached to a stainless steel cantilever. Wear scars with dimensions of $5 \times 5\ \mu\text{m}^2$ were created and scanned for 20 cycles at a load of 10 μN on the fully bonded, partially bonded and untreated Z-TETRAOL. The 10 μN load test was also made at 1 and 5 cycles on the fully bonded coating to study wear initiation.

Surface potential measurements were taken with a Nanoscope IIIa MultiMode atomic force microscope equipped with the extender electronics module (Veeco Instruments Inc.). The extender enables the measurement of surface potential. Electrically conductive silicon tips coated with cobalt/chrome (MESP probes, Veeco Instruments Inc.) were used. Surface potential is measured using a two-pass method. The first pass is in tapping mode to measure surface height. In the second pass, the surface potential map is obtained in the so-called "lift mode" at a tip–sample separation (lift scan height) of 10 nm. In this mode, the piezo normally oscillating the tip in tapping mode is turned off and an oscillating voltage is applied directly to the conducting tip in order to generate an oscillating electrostatic force. This force amplitude is described by

$$F = (\mathrm{d}C/\mathrm{d}z)v_{\mathrm{dc}}v_{\mathrm{ac}}, \tag{1}$$

where $\mathrm{d}C/\mathrm{d}z$ is the vertical derivative of the tip–sample capacitance, $v_{\mathrm{dc}}$ is the dc voltage difference between tip and sample and $v_{\mathrm{ac}}$ is the amplitude of the oscillating voltage applied to the tip. To measure the surface potential, the dc voltage is applied to the tip until the quantity $v_{\mathrm{dc}}$ is equal to zero, giving zero oscillating force amplitude [16,17].

Figure 1 shows the surface height and surface potential images for the fully bonded and partially bonded lubricants. The corresponding images for the uncoated silicon substrate and the fully bonded Z-TETRAOL before wear testing are shown for comparison. Debris is generated around the wear region for all coatings tested. More wear debris is observed in the fully bonded samples than in the partially bonded samples. The untreated sample generated the most debris, which is expected as it contains a smaller amount of lubricant bonded to the substrate relative to the fully bonded and partially bonded coatings. The small amount of debris observed in the partially bonded sample indicates that the mobile fraction of the lubricant is involved in protecting the surface from wear. A possible explanation is lubricant replenishment, i.e. after the bonded Z-TETRAOL molecules are removed during the wear test, the mobile fraction migrates to the exposed silicon, providing further protection to the surface.

As shown in Figure 1, the change in the surface potential of the area subjected to the wear is most prominent in the fully bonded coating and the uncoated silicon substrate. The partially bonded sample exhibited a small change in surface potential, indicating less wear. Similarly, the surface potential of the tested area in the untreated sample barely changed, in spite of the large amount of debris generated due to the presence of the mobile fraction that can move to the area being worn. These findings could be correlated to the wear behavior of the uncoated silicon sample, where the increase in surface potential is well understood. The Kelvin probe method measures the surface potential difference between the tip and sample, which pertains to differences in the work functions between these two materials. For conducting and semiconducting materials, the mechanism is as follows. The surface potential is altered during physical wear because the Fermi energy level is altered. This is the energy required to remove an electron to a point just outside the material surface. Thermodynamic equilibrium is disrupted with a change in the Fermi level, and can only be restored by the flow of electrons either into or from the area subjected to wear. This mechanism does not apply to materials such as $SiO_2$ (naturally present as a thin layer) and the lubricants, which are both insulators. Physical wear on these materials would not cause a change in the surface potential because charge dissipation is poor. Therefore, a considerable surface potential change on the test area would be observed only when: (i) the lubricant has been fully removed from the substrate; (ii) the native $SiO_2$ layer has been abraded from the surface; and (iii) wear has