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Wear

Boundary lubrication effect of organic residue left on surface after evaporation of organic cleaning solvent

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ABSTRACT

Although samples are visibly clean, organic deposits could have tremendous impacts on tribological measurements and interpretation of friction and wear behaviors. This paper discusses the boundary lubrication effects of invisible residues from organic solvents that have been widely used in tribological studies in ambient conditions. Stainless steel, soda lime glass, and copper substrates were cleaned using UV/ozone treatment or organic solvents such as alcohols where the solvent was allowed to evaporate from the surface. In ball-on-flat tribo-tests, all UV/ozone cleaned samples showed high friction and catastrophic wear immediately upon sliding due to the absence of lubricants at the sliding interface, while samples cleaned by organic solvent experienced low friction and minimal wear for hundreds of reciprocating cycles. Analyses of the surface by atomic force microscopy and vibrational spectroscopy indicated that the solvent cleaning deposits hydrocarbon residue on metal and oxide substrates. This residue is due to trace impurities with low vapor pressures inevitably present in the bulk liquid, regardless of solvent purity, which become concentrated upon vaporization of the high vapor pressure solvent. These results demonstrated that drying after cleaning with organic solvents is equivalent to a dip-coating of low vapor pressure organics onto the sample surface. This dip-coating effect can be avoided if surfaces are cleaned using UV/ozone or if the surface is immediately rinsed with DI water and blow-dried after cleaning with organic solvent. These results indicate that the friction coefficients of the samples with organic residues should not be interpreted with the shear strength model of friction.

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1. Introduction

Sample cleanliness is crucial for accurate measurements of friction and wear behaviors of sliding surfaces. Dry sliding between two surfaces that are in intimate contact can be affected by a very thin layer of lubricant. Self assembled monolayers (SAMs) have been shown to lubricate surfaces in atomic force microscopy (AFM) studies as well as decrease adhesion in microelectromechanical systems (MEMS) [\[1,2\].](#page--1-0) Many solid surfaces show a low friction coefficient near 0.2 and minimal wear with only a monolayer of adsorbed alcohol vapor [\[3,4\].](#page--1-0) This lubrication occurs not only at the nano-scale; it is also observed at the macroscale where the surface roughness is several orders of magnitude larger than the size of adsorbed molecules [\[3\]](#page--1-0). Vapor can also be used to deliver a thin molecular coating to protect surfaces from wear [\[5](#page--1-0)–[7\]](#page--1-0).

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Crucial to the proper function of these boundary lubrication methods is careful control of the chemical species on the surface. In the absence of any coating, pure metal surfaces could irreversibly adhere to one another and friction coefficients for these materials could be much higher than one $[8-10]$ $[8-10]$ $[8-10]$. Exposure to ambient air will form oxide layers on substrates with high surface energy [\[11](#page--1-0)–[13\].](#page--1-0) It can also introduce a thin layer of organic contaminants adsorbed from the gas phase. These organic molecules can react to form adventitious carbonaceous deposits on surfaces in assistance of frictional heat or electricity, which can influence the tribological properties of the system [\[14](#page--1-0)–[16\]](#page--1-0).

This paper examines the effect of cleaning tribological surfaces with commonly-used organic solvents, with specific focus on the residue left after solvent evaporation. Soda lime glass (SLG), stainless steel (SS), and copper substrates were cleaned using two methods: (i) UV/ozone and (ii) rinsing with alcohol followed by evaporation or blow dry from the sample surface. The experimental results indicated that the residue film resulting from the evaporated liquid lubricates SLG, SS, and Cu surfaces with low friction and virtually no wear for a certain period which varies depending on the residue film thickness and sliding conditions. Since SLG, SS, and Cu have very different surface chemistry and physical properties, this lubrication effect is not a material specific phenomenon. Even if only a trace amount of residues is present in ultra-high purity solvent, the evaporation of solvent results in concentration of these residues on the substrate. Thus, the solvent cleaning and evaporation could be considered or equivalent to a dip-coating of low vapor pressure organic residues which inevitably exist in liquid solvents. Based on the findings from this study, the shear strength model of friction for unlubricated solids is revisited [\[17,18\].](#page--1-0)

2. Experimental

Friction tests were conducted using a custom-built reciprocating ball-on-flat tribometer [\[19\].](#page--1-0) Substrate material was AISI 440C stainless steel (SS), oxygen-free high-conductivity (OFHC) copper (Cu), and soda lime silica slide glass (SLG). Commercially available 3 mm diameter 440C stainless steel balls were used as a countersurface for all copper and stainless steel substrate tests; 2.4 mm diameter borosilicate glass balls were used for SLG substrates. The tribometer was operated at a sliding speed of 4 mm/s with a 1 N load for each test and slide tracks were roughly 2.5 mm long. All tests were conducted in a gas environment of dry nitrogen and were repeated for at least 5 times. The nominal Hertzian contact pressure for SS ball against SS substrate, SS ball against copper substrate, and glass balls against SLG substrate are calculated to be 642 MPa, 524 MPa, and 350 MPa, respectively. Previous vapor phase lubrication studies showed that SS, Cu, and SLG substrates do not get damaged mechanically as long as the surface is covered with one monolayer of alcohol molecules [\[3,12\]](#page--1-0). Thus, friction and wear tests at these contact conditions are sensitive to chemical conditions of the sliding interface.

All substrate samples were cleaned by rinsing with ethanol, followed by exposure to UV/ozone cleaning for at least 20 min. Tribotests investigating this surface were conducted immediately following UV exposure. For tests investigating the effect of cleaning with liquid solvent, the sample was taken from the UV/ozone chamber and then rinsed by the liquid in question, with liquid left on the sample to evaporate in air. Liquids investigated were ethanol, methanol, isopropanol, and decane, and each liquid had purity greater than 99%. After drying, these samples were tested using the tribometer.

Infrared spectroscopy analysis was carried out using a Thermo-Nicolet Nexus 670 system equipped with a MCT detector. Attenuated total reflection (ATR) tests were conducted for a liquid sample using a multi-bounce silicon crystal [\[20\].](#page--1-0) Polarizationmodulation reflection-absorption infrared spectroscopy (PM-RAIRS) was used to analyze organic species remaining on metal surfaces; detailed description of the setup is available elsewhere [\[19\].](#page--1-0) For chemical analysis of organic species on SLG surfaces, vibrational sum frequency generation (SFG) spectroscopy was used [\[21\].](#page--1-0) Briefly, the SFG signal was generated at the substrate/air interface where two laser pulses $-$ 532 nm visible pulses with 60 $^{\circ}$ incident angle from an EKSPLA Nd:YAG laser and tunable IR pulses with 56° incident angle generated with an optical parameter generator and amplifier – were spatially and temporally overlapped. The SFG signal was collected in a reflection geometry and normalized with the input intensities of visible and IR pulses. The polarization of light was s for SFG signal, s for visible beam, and p for IR beam (ssp).

Topography of the wear tracks were analyzed with a Zygo NewView 7300 profilometer in ambient environment. The surface topography of samples with different cleaning methods were investigated by using atomic force microscopy (AFM) with a Digital Instrument MultiMode scanning probe microscope. Topographic images of the sample surface were collected with a contact-mode scanning. The AFM image size was 5 μ m \times 5 μ m.

3. Results and discussion

The lubrication of evaporated solvent residue was tested on glass, stainless steel, and copper. Fig. 1 compares the friction behavior for stainless steel (SS), soda lime glass (SLG), and copper cleaned by either UV–ozone or by liquid ethanol. All tests were conducted in dry nitrogen. [Fig. 2](#page--1-0) shows the surface profile of each material after friction tests. The severely worn surfaces with wear depth deeper than several microns [\(Fig. 2](#page--1-0)a, c, and e) were observed for the samples cleaned with UV/ozone. When sliding was stopped before friction increased abruptly, the ethanolcleaned surfaces showed only plastic deformation and no significant wear ([Fig. 2](#page--1-0)b, d, and f).

Both friction and wear behaviors show that there is a drastic difference between the surface cleaned by UV–ozone and the surface cleaned by ethanol. All materials cleaned by UV–ozone experience substantial wear and high friction immediately at the start of the tribotest. The friction value near 0.3 is typical of SLG substrate wear, which occurs through scratching/abrasion and creates piles of particulate debris, which is evident at the far edges of the optical profilometry image in [Fig. 2a](#page--1-0). The friction value for SS is higher than 0.5 and the surface is severely damaged due to adhesive wear, as seen in [Fig. 2](#page--1-0)c. Similar behavior is seen for Cu in [Fig. 2e](#page--1-0), with a friction coefficient higher than 0.6. The worn tracks from the UV–ozone cleaned surface measure several microns deep for each material.

These friction and wear patterns are in contrast to the negligible wear and low friction observed for the ethanol-cleaned samples. Friction for SS and SLG begins near 0.1 and slowly rises towards 0.2 as sliding continues, while friction for Cu is constant near 0.18 until wear occurs. Wear, or evidence of material removal by rubbing, is nearly imperceptible on the materials during the low friction period. The sliding track on SLG [\(Fig. 2](#page--1-0)b) shows no height difference from the surrounding surface and is discernible mainly due to the minute pile-up of material at the periphery of the sliding track, whose height measures only tens of nanometers. The SS sliding track is virtually wearless ([Fig. 2](#page--1-0)d); only a slight depression due to plastic deformation in the bulk can be recognized. The Cu track ([Fig. 2](#page--1-0)f) is somewhat easier to see due to larger plastic deformation of the softer copper substrate.

Fig. 1. Friction coefficient for various substrates cleaned by UV/ozone or liquid ethanol. All tests conducted in dry nitrogen environment.

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