Tribology International 127 (2018) 138–146

Contents lists available at ScienceDirect

Tribology International

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

Simultaneous *in situ* measurements of contact behavior and friction to understand the mechanism of lubrication with nanometer-thick liquid lubricant films

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ARTICLE INFO

Keywords:
Nano-lubrication
Friction
Sliding-induced vertical displacement
Eyring thermal activation energy approach

ABSTRACT

A high-performance microscopic observation system was developed and integrated with a purpose-built pin-on-disk type tribotester. This allows for simultaneous *in situ* measurements of the vertical displacement and friction force of a pin sliding on disks lubricated with nanometer-thick liquid films at velocities up to 0.1 m/s, with accuracies of approximately 0.6 nm and 10 μN. Upward pin displacement was observed, exhibiting an exponential increase followed by a slight increase with increasing sliding velocity. The pin displacement also increased with film thickness and lubricant viscosity. We conclude that even nanometer-thick liquid lubricant films can generate upward dynamic pressure. Further insight into the shearing process was gained by analyzing the measured friction forces using the Eyring thermal activation energy approach.

1. Introduction

Miniaturization and integration of devices and systems are constantly progressing, motivated by the ever-increasing demands for innovative and versatile functions, higher performances, and increased energy efficiency. This leads to more situations where system components come into contact and move relative to each other at nanometer separation distances. To ensure reliability and durability of such advanced miniaturized systems, achieving effective lubrication with nanometer-thick liquid films (nano-lubrication) is of major concern [1–3]. For example, to achieve ultra-high recording densities in hard disk drives, the flying height of the magnetic head over the disk must be reduced to sub-1-nm regime [4–6]. This causes sliding contact between the head and the disk to occur more frequently, thereby placing increasingly stringent requirements on the lubrication performance of the 1–2-nm thick films of liquid perfluoropolyether (PFPE) coated on disk surfaces. Unlike in the case of macroscale lubrication, which is well described by continuum theories along with bulk liquid properties, in the case of nanoscale lubrication, interactions with solid surfaces become dominant, giving rise to unique liquid properties, such as increased viscosity and oscillatory density profiles [7–9]. Therefore, fundamental understanding of nano-lubrication is of great scientific significance.

The surface force apparatus (SFA) [10–14] and atomic force microscope (AFM) [14–16] are commonly used for studying nano-lubrication. The former measures normal and frictional forces of molecularly-thin liquid films confined in regions with well-defined areas and gap thicknesses, formed by applying normal loads to two atomically smooth surfaces with curvature radii of approximately 1 cm in crossed cylinder geometry. The latter uses a probe with a sharp tip with a radius of several nanometers, or an attached colloidal particle with a radius in the micrometer range to slide against sample surfaces under a constant normal load and measures frictional forces acting on the probe. However, because both methods employ piezoelectric driving mechanisms, sliding velocities are limited to a maximum of several millimeters per second. Although the SFA has been fitted to cover sliding velocities up to 10 m/s by introducing a motor-driven rotating-disk setup, large-amplitude oscillatory responses, originating from the loading or force sensing mechanisms rather than the contact sliding interfaces, became overwhelming at sliding velocities beyond approximately 1 cm/s [17].

To measure frictional properties of nanometer-thick lubricant films at high sliding velocities, we have developed a pin-on-disk type

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https://doi.org/10.1016/j.triboint.2018.05.043
Received 28 March 2018; Received in revised form 26 May 2018; Accepted 31 May 2018

tribotester where unidirectional sliding at constant speeds ranging from 0.1 to 100.0 rpm is achieved using a highly precise air-bearing spindle servo motor and a high resolution built-on incremental encoder [18,19]. By developing a sliding-pin-and-suspension assembly that minimizes the frictional moment acting on the sliding pin, we have successfully suppressed vibrations from the loading and the frictional force sensing mechanisms, thereby achieving steady contact sliding at velocities up to 0.2 m/s [20,21]. Moreover, unlike the commercially available tribotesters, ours operates under small external loads (less than several micro-Newton), thereby effectively suppressing damage to nanometer-thick films caused by contact sliding. Using our tribotester and a lateral force microscope with our original dual-axis probe, we measured the frictional properties of 1.3 nm thick PFPE films with different bulk viscosities over a wide sliding-velocity range from 1 μm/s to 0.2 m/s [22]. We found that these films displayed trends similar to the Stribeck curves: the friction coefficient initially decreased and then increased as the sliding velocity increased, while the critical sliding velocity at the inflection point decreased as the bulk lubricant viscosity increased. The initial decrease at sliding velocities below the critical value is likely attributed to the decrease in solid-solid contact area caused by the increased entrainment of lubricant molecules into the contact sliding interface. However, the mechanism underlying the frictional behavior at sliding velocities beyond the critical value is yet to be fully elucidated.

In this study, we designed and constructed a microscopic observation system and integrated it onto the top of the previously developed tribotester. This allows us to measure the friction force while observing *in situ* the contact region between the pin and disk mediated with nanometer-thick liquid lubricant films. From the observed contact regions, we are able to derive the vertical displacements of the sliding pin. Based on the results of the simultaneously measured vertical displacements and frictional forces and theoretical analyses using the Eyring thermal activation energy approach, the possible mechanism of nanolubrication at high sliding velocities is discussed.

2. Experimental details

2.1. Apparatus and principle

Fig. 1 shows the schematic diagram of the experimental apparatus. This was an integration of a newly designed and constructed microscopic observation system and the previously developed pin-on-disk type tribotester [18–20]. We used a specially fabricated spherical plano-convex lens made of BK 7 glass as a sliding pin. The lens had an aperture diameter of 3.0 mm, edge thickness of 1.5 mm, and curvature radius of 7.5 mm for the convex surface. The arithmetic mean surface roughness Ra was 0.39 nm and the root-mean-square surface roughness Rq was 0.51 nm, as measured using an AFM (Dimension Icon, Bruker) in a 1 μm × 1 μm scan area. The sliding pin was glued into a through hole at the front end of a suspension, with the convex surface protruding 0.65 mm from the hole. The small protrusion effectively suppressed the frictional moment, thereby repressing the out-of-plane vibration of the suspension [20]. The suspension was a rectangular cantilever made of stainless steel SUS304 with 13.5, 5.0, and 0.1 mm in length, width, and thickness. The suspension was fixed to the bottom of a friction transducer with the top of the friction transducer mounted to a sensor arm. The friction transducer, fabricated using electric spark machining, consisted of two pairs of parallel leafs. We measured its spring constant to be 3.53×10^3 N/m. A rubber damper was infixed between the parallel leafs. When subjected to a frictional force, the friction transducer deflected in the horizontal direction with the deflection being detected by a capacitance type displacement sensor, which was fixed to the sensor arm, at a 2.5 nm resolution. Hence, the resolution of the frictional force measurements was estimated to be approximately 10 μN.

External loads were applied on the sliding pin by driving it downward with a piezoelectric *z*-stage. The load resolution was 8.2 μN as determined by the vertical spring constant of the suspension (82.0 N/m) and the displacement resolution of the *z*-stage (0.1 μm). Although not shown in Fig. 1, a laser sensor and another capacitance type displacement sensor were initially set above the front end of the suspension and the sensor arm to measure their respective vertical displacements [19]. From the measured vertical displacements, we can determine the deflection of the suspension, thereby obtaining the normal force exerted on the sliding pin. Similar to the force-distance curve measurements with an AFM, we can determine the adhesion force with our tribotester, by measuring the vertical displacements of the front end of the suspension and the sensor arm during the approach and withdrawal of the sliding pin with respect to either stationary or rotating disks [19,21].

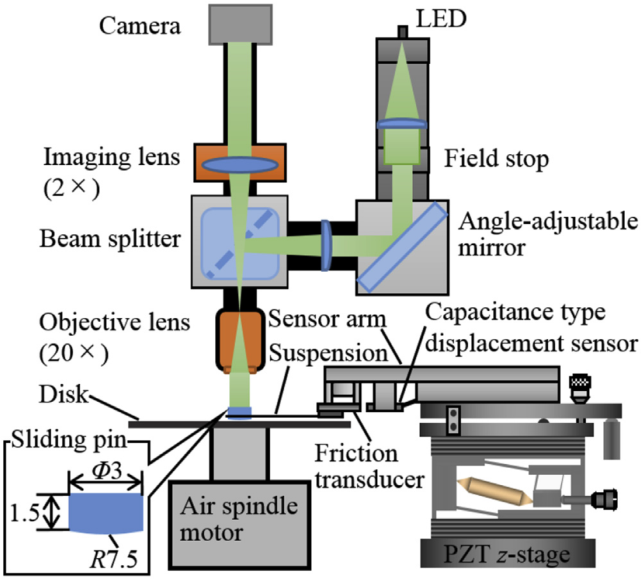

Fig. 1. Schematic diagram of experimental apparatus. This is an integration of a newly developed microscopic observation system (top part) and a previously developed pin-on-disk tribotester (bottom part). The field stop is an iris diaphragm with adjustable aperture 0–10 mm in diameter. The angle-adjustable mirror is used to ensure uniform illumination of the observation area. The values indicating the dimensions of the sliding pin are in the unit of millimeter.

Once the normal force was determined, the vertical displacement sensors were unmounted and the newly developed microscopic observation system was set above the sliding pin as illustrated in Fig. 1. We used a high-power LED light source (X-Cite XLED1, Excelitas Technologies) with a peak wavelength of 405 nm. A light beam from the light source passed through the lenses and a beam splitter and converged at the back focal point of an objective lens (M Plan Apo SL20 × , Mitutoyo). The collimated light beam from the objective lens uniformly illuminated the plane side of the sliding pin. The contact region between the convex side of the sliding pin and a disk was magnified 40 times using the objective lens and an imaging lens (MT-2, Mitutoyo). Subsequently, images of the contact region were captured with a camera. Fig. 2 shows representative images captured before and during sliding. A liquid bridge formed around the pin before sliding by the nanometer-thick lubricant film coated on the disk, whereas it became hardly discernible during sliding. We also confirmed that friction forces during steady sliding are independent of the size of liquid bridges. From the captured images, the radii of the first-order Newton's rings before and during sliding, r_0 and r_1, can also be measured. As is observed from Fig. 2 and theoretically confirmed [23,24], r_0 and r_1 were much larger than the radius of the central loaded contact area under the experimental conditions used in this study. At such radial positions, elastic deformation of the spherical pin surface is negligibly