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Research Article

Dependence of Pin Surface Roughness for Friction Forces of Ultrathin Perfluoropolyether Lubricant Film on Magnetic Disks by Pin-on-Disk Test

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We fabricated supersmooth probes for use in pin-on-disk sliding tests by applying gas cluster ion beam irradiation to glass convex lenses. In the fabrication process, various changes were made to the irradiation conditions; these included one-step irradiation of Ar clusters or two-step irradiation of Ar and N_2 clusters, with or without Ar cluster-assisted tough carbon deposition prior to N_2 irradiation, and the application of various ion doses onto the surface. We successfully obtained probes with a centerline averaged surface roughness that ranged widely from 1.08 to 4.30 nm. Using these probes, we measured the friction forces exerted on magnetic disks coated with a molecularly thin lubricant film. Perfluoropolyether lubricant films with different numbers of hydroxyl end groups were compared, and our results indicated that the friction force increases as the surface roughness of the pin decreases and that increases as the number of hydroxyl end groups increases.

1. Introduction

In magnetic disk storage, the head-disk spacing must be minimized to maximize the bit density. Most recently, a spacing of 4-5 nm has been attained, which is the spacing between the bottom of the head pole tip and the rigid disk surface. To further reduce this spacing, the lubricant film coated over the disk was thinned and strengthened using lubricants with increased molecular polarity. Some of the commonly used lubricants are functionalized perfluoropolyethers (PFPE) with a linear backbone and hydroxyl end groups (–OH) called PFPE polar lubricants (Solvay-Solexis). The number of –OH groups has been increased in order to ensure that the lubricant film has a relatively strong retention function [1, 2].

Recently, the dynamic flying height (DFH) has been employed during read/write operations. After being heated, the bottom of the head pole tip protrudes, forming a hemisphere-shaped protrusion with a radius of curvature of several millimeters [3]. Therefore, the evaluation of the tribological features of the protruding head and that of the disk lubricated with a molecularly thin polar lubricant film are of significant concern. Shimizu et al. studied the slider dynamics during the touchdown of sliders by using a laser Doppler vibrometer (LDV), acoustic emission (AE) measurement, and friction force measurement. They concluded that the friction force at the head disk interface (HDI) continues to increase during the touchdown sequence, even though the slider vibration and the AE signal decrease when the heater power is increased. The friction force at the HDI needs to be decreased to further reduce the HDI clearance [4]. Sonoda et al. studied the tribological properties of two types of PFPE lubricants on the basis of the component level and by performing drive level tests. Disks having a lower friction property and lower surface energy showed better HDI performances [5].

To date, many experimental studies have addressed the tribological behavior of monolayer lubricants, employing surface force apparatus (SFA), friction force microscopy (FFM), and the fiber wobbling method (FWM). Because each approach uses an elaborated probe, the direct application of the probe to an actual head-disk interface is difficult. The radius of curvature of the probe for SFA is around 10 mm [6], which is comparable to that of an actual head-disk interface subject to DFH. For FFM, the radius is around 20 nm, and for FWM, it is around 4–100 μm; the values in both cases are considerably small [7, 8]. Large differences are observed between the friction forces measured using SFA and FFM on PFPE Z and polydimethylsiloxane (PDMS) monolayer films; these differences are attributed to whether or not the probe penetrated the film [9]. Recently, friction coefficients obtained using FFM have been confirmed to be almost the same when FFM experiments were performed under a very light load [10]. These methods are based on ideal conditions without roughness (SFA) or with a very sharp tip (FFM), which are considerably different from the conditions of an actual head-disk interface. The friction force basically consists of two components: one is load-controlled and the other is adhesion-controlled [11]. The former component represents the friction linearly varying with an external normal load, known as Amonton's law, and the latter represents the nonlinear friction-versus-load characteristic, which is typically in proportion to 2/3 of the power of the loading force. In the case of multiasperity contact, load-controlled friction becomes dominant, whereas in the case of single-asperity contact, adhesion-controlled friction becomes dominant. Switching from multiasperity to single-asperity contact is induced by confining the contaminant film that intervenes between the two surfaces [12].

These results suggest that even nanometer-scale roughness profoundly affects the tribological behavior of monolayer lubricant films, and thus, a supersmooth probe or probes with different roughnesses are required. In this study, to evaluate the tribological behavior of a thin lubricant film under similar geometric conditions of a practical head-disk interface, we fabricated supersmooth probes for use in pin-on-disk sliding tests by applying gas cluster ion beam (GCIB) irradiation [13, 14] to commercially available glass convex lenses. We successfully obtained probes with a centerline averaged surface roughness (R_a) that ranged from 1.08 to 4.30 nm. Using these probes, we measured the friction forces exerted on magnetic disks coated with a PFPE lubricant film having different numbers of –OH groups. The results indicated that the newly developed probe is useful for evaluating monolayer lubricant films on a practical scale.

2. Modification and Smoothing of Glass Sliding Pins

Considering the millimeter-sized radius of curvature of the protruded head, we selected a BK7 planoconvex lens (Sigma Koki; SLB-05-10P) with a radius of curvature of 5.19 mm and a diameter of 5 mm. To modify the surface roughness of the lens, we applied GCIB processing to its surface. Because gas cluster ions are composed of thousands of gaseous atoms, the energy distributed to each atom remains within ultralow levels, even though the GCIB energy is high. This permits surface modifications such as etching, smoothing, and deposition to be performed without strongly damaging the surface. The smoothing effects obtained using GCIB processing depend on parameters such as the type of atoms, electron bombardment energy, ion acceleration energy, and amount of dosed clusters [13]. Our aim is to optimize these parameters in order to smoothen the surface of the glass lens. Based on past research, the electron bombardment and the ion acceleration energies were fixed at 150 eV and 7 keV, respectively. Ar clusters were employed for the main processing and N_2 clusters for additional processing. Hereafter, Ar cluster irradiation is called one-step irradiation, and N_2 cluster irradiation, after Ar cluster irradiation, is called two-step irradiation. We also applied GCIB-assisted diamond-like-carbon (DLC) deposition (Nomura Plating; tough carbon) [14] to the Ar-GCIB-modified surface as successive processing from Ar irradiation by sublimating the C_{60} fullerenes into the deposition chamber. The thickness of the tough carbon was fixed at 300 nm. Finally, N_2 clusters were applied to the top surface. All of the conditions for GCIB processing are listed in Table 1. Two glass pins were prepared for each condition.

After the GCIB operations, the surface roughnesses of the top surfaces were measured by atomic force microscopy (AFM) using Nanoinstrument IIIa. Typical surface profiles of the original surface and the surface fabricated by the GCIB irradiation on glass probe surfaces without the DLC film are shown in Figure 1. Table 1 shows the GCIB conditions and the average roughness obtained after 3 sets of measurements. Figure 1(a) shows the original glass surface for pin 1 in Table 1, Figure 1(b) shows that for pin 2, and Figure 1(c) shows that for pin 3. For reference, the AFM images are also shown in Figures 3(a) and 3(c) for the original surface and the smoothest surface, which correspond to Figures 1(a) and 1(c). Although Figures 1(a) and 1(c) show noise patterns in subnanometers, these noise patterns were generated by the external vibration and the measured roughness values were not affected considerably by the noise. The roughness of the glass probe surface was reduced by the two-step GCIB irradiation, although the asperity height of the probe surface was increased significantly by the one-step GCIB irradiation. The roughness profiles of the DLC surface on the probes irradiated by the GCIB are shown in Figure 2. Figure 2(a) shows pin 4, Figure 2(b) shows pin 5, and Figure 2(c) shows pin 6. For reference, the AFM images are also shown in Figures 3(d) to 3(f), which correspond to Figures 2(a) to 2(c). After GCIB irradiation to DLC film surface, there were more asperities on the surface than the original surface. Therefore, the one-step Ar GCIB irradiation increases the asperity height on both glass and DLC surface and the two-step Ar-N_2 GCIB irradiation reduced the surface roughness.

Using these figures, we could compare the changes in roughness produced by GCIB processing in terms of R_a units, which are defined as the centerline averaged roughness over a roughness curve, R_q, which is defined as the root mean square roughness, and $R_{p\text{-ave}}$ which is defined as the maximum peak height averaged over the three highest points in a measured area of $25 \times 25\,\mu\text{m}^2$. We chose the $R_{p\text{-ave}}$ value as a representative value for determining the distance of separation between two solid surfaces in contact with one another. For Figure 2(a), the original glass surface, sharp peaks were sparsely distributed, which can be seen in

Table 1: Conditions for GCIB processing and surface roughness.

No.	Dose, ion/cm^2		DLC	Roughness, nm		
	Ar $\times 10^{16}$	$N_2 \times 10^{16}$	nm	R_a	R_q	R_{p_ave}
1	0	0	0	1.40	3.01	10.26
2	2	0	0	4.30	6.35	34.83
3	1	2	0	1.08	1.40	4.99
4	1	0	300	1.51	2.16	17.33
5	2	0	300	1.07	1.39	7.87
6	4	0	300	1.05	1.37	13.63

Figure 2(a) as white colored spots. R_a was 1.40 nm, a value that indicated a well-finished surface for optical use, and R_{p_ave} was 10.26 nm, which was seven times higher than R_a. For Figure 2(b), one-step GCIB without DLC, the degree of roughness increased to more than that of the original surface, which indicated that the Ar clusters were considerably strong. In the case of Figure 2(c), two-step GCIB without DLC, the sparsely distributed peaks decreased to R_{p_ave} of 4.99 nm, which indicated that N_2 GCIB was effective in smoothing and that R_a improved to 1.08 nm. As a result, we obtained a wide variety of surface roughnesses ranging from 1.08 to 4.30 nm in R_a units and from 4.99 to 34.83 nm in R_{p_ave} units.

3. Friction Tests

Pin-on-disk-type friction tests were performed using GCIB-treated planoconvex lenses that were glued to a commercially available flying head slider and loaded by a slider suspension spring (with a spring constant of 19.0 N/m) onto a magnetic disk at a fixed load of 9.8 mN. Friction tests were performed five times for the same pin on the same sliding track, which was slightly shifted for each pin from the inside to the outside at intervals of 1 mm, with slightly varying disk rotation, to obtain a constant velocity of 2 mm/s. This rotational velocity was considerably slower than the actual HDI velocities. However, the friction forces measured by the pin-on-disk tester using the low rotational velocity were useful for comparing the basic tribological properties of lubricant films on magnetic disks because it was obvious that the friction forces played an important role in the interaction between the slider and the disk surface, as described in some references [15, 16]. The pin was cleaned with a solvent (DuPont; Vertrel XF) when the disk was changed, that is, before every first rotation for the different lubricant films.

The experimental disks were coated with a nitrogenated, 4 nm thick DLC film with an R_a value of 0.2 nm. The experimental lubricants were PFPE Z-dol (with a molecular weight of 2000) with one hydroxyl group (–OH) at each end, Z-Tetraol (2400) with two –OHs at each end, and Z-Tetraol-multidentate (Z-TMD; 3000) with an additional four –OHs on the main Z-Tetraol chain [1, 2], as shown in Figure 4.

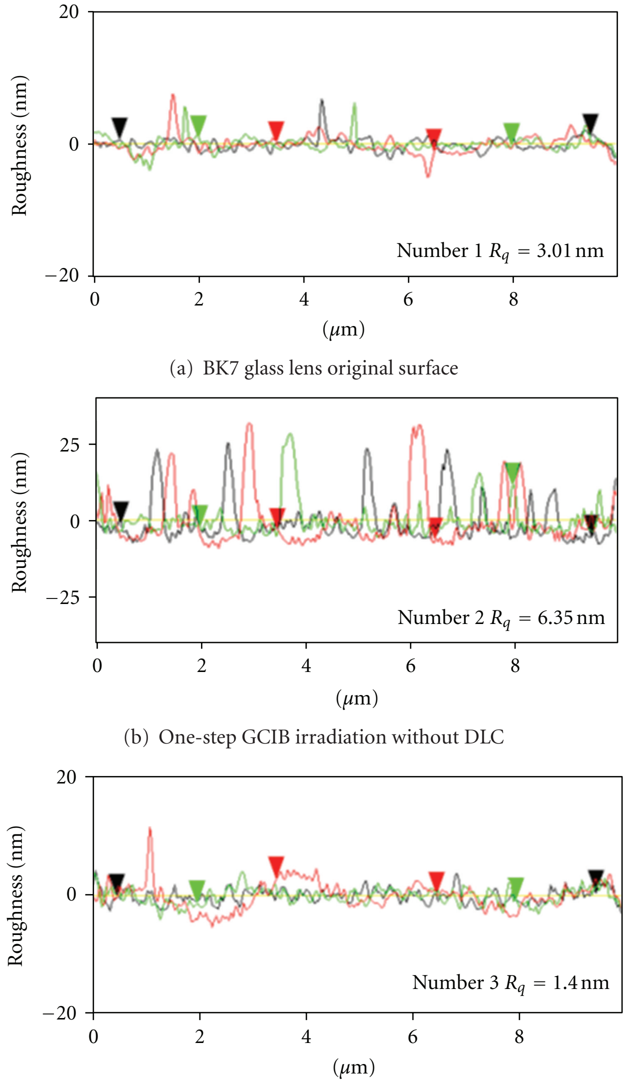
The film thicknesses, measured with an ellipsometer (MARY by Five Lab. Co.), were 1.1, 1.2, and 0.8 nm, and the bonding thickness were measured one day after the friction measurements to be 20%, 66%, and 80% for Z-dol, Z-Tetraol, and Z-TMD, respectively. Here, the bonding ratio is defined as the ratio of the coated lubricant thickness to the residual film thickness after rinsing the lubricant-coated surface with a solvent (Vertrel XF). The monolayer thickness of Z-dol2000 was approximately 1.4 nm, and that of Z-Tetraol was approximately 1.7 nm [17]. The monolayer thickness of Z-TMD was estimated to be of the same order as that of Z-dol2000 [18]. Note that the coated films in this study were either as thin as a monolayer or thinner. All experiments were performed in a clean booth in which the temperature ranged from 25° to 29° and the humidity from 55% to 65%.

4. Results and Discussions

The experimental results are plotted in Figure 5, where Figure 5(a) shows the results for Z-dol, Figure 5(b) for Z-Tetraol, and Figure 5(c) for Z-TMD. The abscissa indicates the average peak height R_{p_ave}, and the ordinate denotes the friction force F. In the figures, the symbols denote the BK7 original glass surface, the BK7 glass surface after Ar-GCIB processing, the BK7 glass surface after Ar/N_2-GCIB processing, and the DLC surface after Ar-GCIB processing. The range of variation of the five experiments conducted under the same condition is marked by short bars sandwiching the symbols. These bars corresponded to the maximum and minimum values of the measured friction forces.

The data points were fairly scattered because of the local variations in the surface features, the film thicknesses, the roughness of the disk surfaces, and the sliding-induced vibration. In the case of the Z-dol film, the friction force remained nearly constant with varying roughness. This was attributed to the fact that the bonding ratio was as low as 20%, and, thus, the mobility of unbonded molecules was large and the movable molecules with low viscosity between the probe and the disk surface reduced the friction force in the same manner as hydrodynamic lubrication, as illustrated in Figure 6.

In the case of the Z-Tetraol film, the friction was almost unchanged for an R_{p_ave} of more than 15 nm. However, when the roughness became smaller than 10 nm (at which point the R_a value was roughly equivalent to the monolayer film thickness, as shown in Table 1), the scattering of the data points increased, but on average, the friction force tended to increase with decreasing roughness. Nevertheless, the bonding ratio was as high as 66%, and lubricant molecules could be removed for the relatively rough probes because of the high contact pressure; in the case of the relatively smooth probes, the contact pressure decreased because of

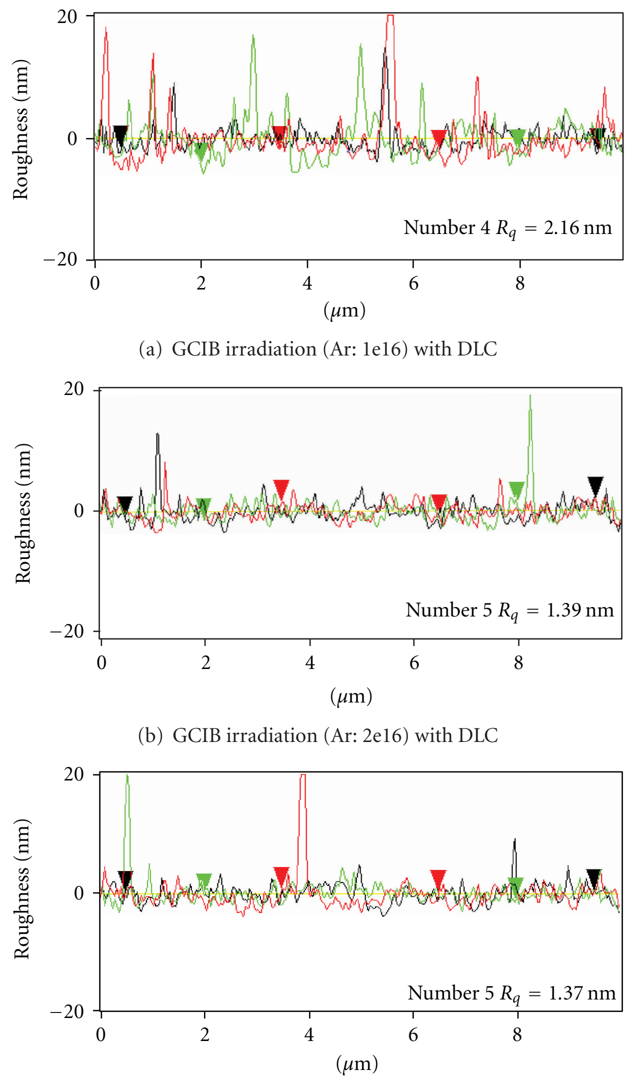


(a) BK7 glass lens original surface

(b) One-step GCIB irradiation without DLC

(c) Two-step CCIB irradiation without DLC

Figure 1: Changes in surface roughness profiles with GCIB irradiation without DLC. The abscissa indicates the scanning distance in micrometers, and the ordinate indicates the roughness amplitude in nanometers.



(a) GCIB irradiation (Ar: 1e16) with DLC

(b) GCIB irradiation (Ar: 2e16) with DLC

(c) GCIB irradiation (Ar: 4e16) with DLC

Figure 2: Changes in surface roughness profiles with GCIB irradiation with DLC. The abscissa indicates the scanning distance in micrometers, and the ordinate indicates the roughness amplitude in nanometers.

the increased contact area and many bonded molecules remained inside the contact area after being deformed, as illustrated in Figure 7, which was the confinement condition. Confined molecules exhibited a solid-like feature, and thus, the single-asperity condition became dominant [12]. This was considered a major reason for increased friction with decreasing roughness.

In the case of the Z-TMD film, the increase in friction with decreasing roughness became more pronounced. This was attributed to the bonding ratio that was as high as 80%, which was considerably higher than that in the case of Z-Tetraol, and, thus, a large majority of molecules remained inside the contact area. This strengthened the shift from the multiple- to single-asperity contact. Note that the friction forces were nearly equal for different lubricants for a relatively large roughness range, which is a basic feature of Amonton's law followed from multiple-asperity contact. On the basis of these results, we concluded that the friction forces measured by the relatively smooth probes might depend on the dynamic viscosity of the lubricant materials, because the dynamic viscosity of Z-TMD, Z-Tetraol, and Z-dol2000 was 23 Pas, 3.5 Pas, and 0.153 Pas, respectively, [19, 20]. Further studies are required to clarify the differences in the friction property of these lubricants.

With respect to the effect of DLC coating, the effect reducing the friction forces was estimated to be small, because the trend of roughness dependence for the DLC-coated pin agreed with that for the glass pin without DLC in the case of Z-Tetraol and Z-TMD. The friction measurements did not show changes at small peak heights

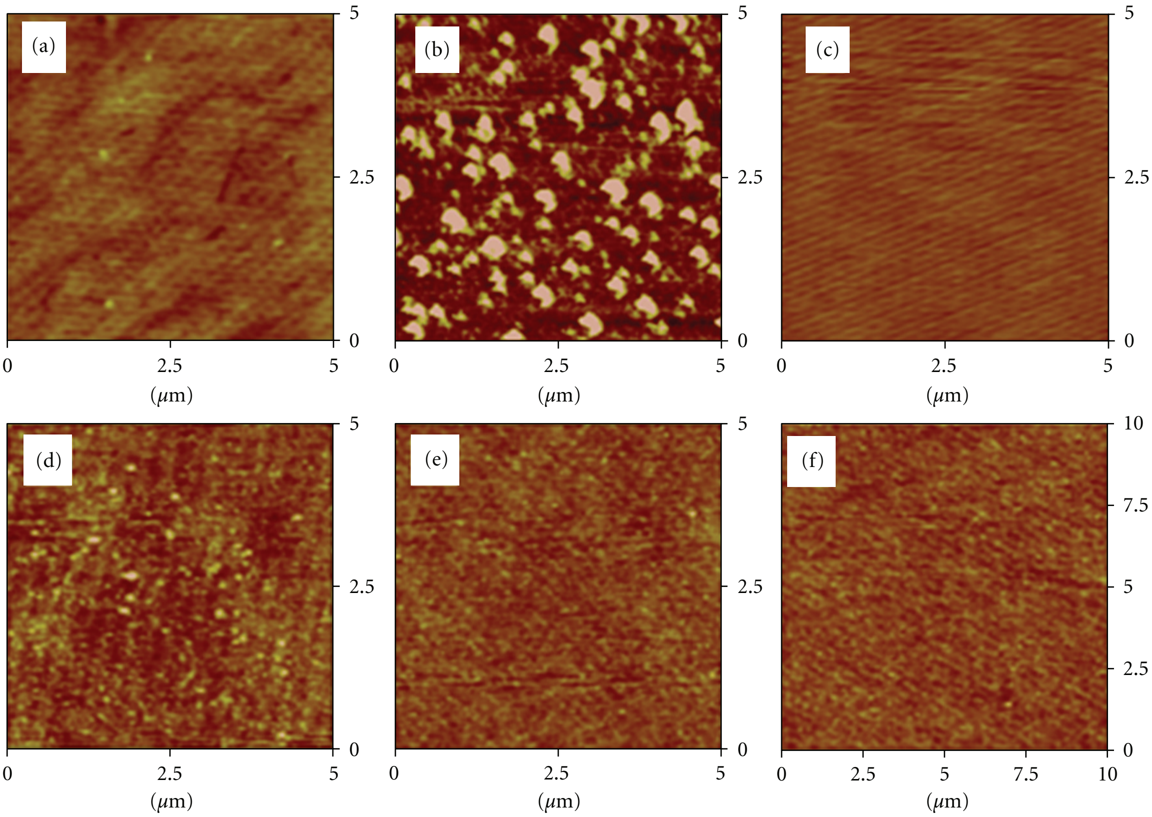


Figure 3: Comparison of tapping-mode AFM images of planoconvex lens before and after GCIB processing. (a) Original surface of glass lens, (b) one-step GCIB irradiation without DLC, (c) two-step GCIB irradiation without DLC, (e) one-step (Ar: 1×10^{16} dose/cm^2) GCIB irradiation on DLC, (e) one-step (Ar: 2×10^{16} dose/cm^2) GCIB irradiation on DLC, and (f) one-step (Ar: 4×10^{16} dose/cm^2) GCIB irradiation on DLC.

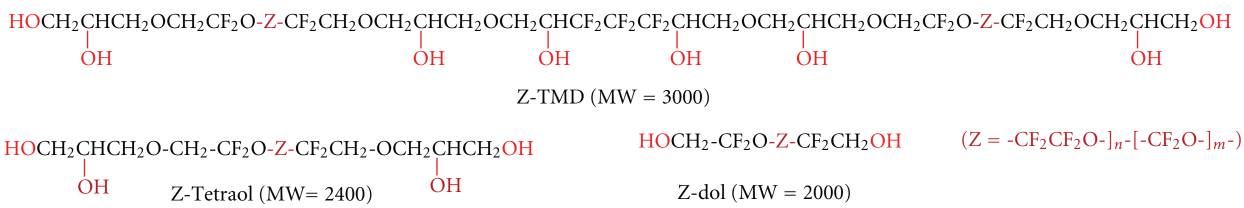


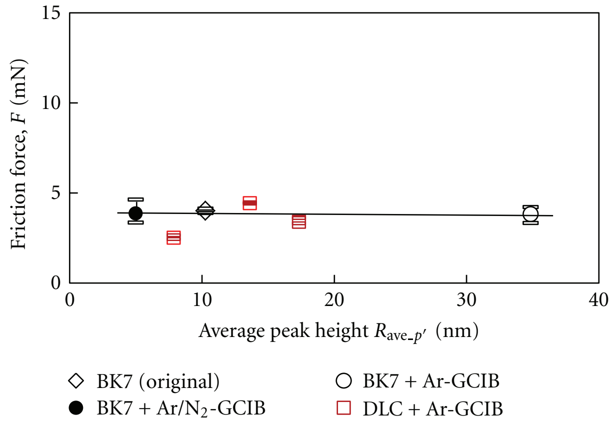
Figure 4: Chemical structures of PFPE lubricants used in this study.

for Z-dol-lubricated disks in contrast to the other lubricants evaluated.

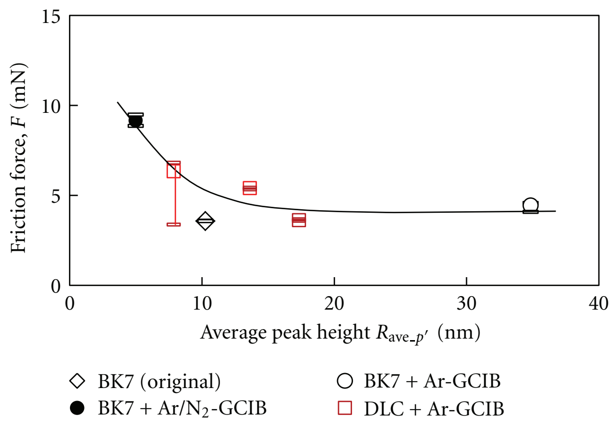
On the basis of the Previos experimental tests, we confirmed the usefulness of GCIB-fabricated probes with a wide variety of roughnesses for the evaluation of monolayer lubricant films. However, there is a possibility of a change in the chemical properties of the probe surface fabricated using the GCIB fabrication method, for example, the change in the lubricant affinity on the probe surface by argon and nitrogen implantation by GCIB irradiation [21] or DLC coating by the GCIB treatment [22]. Similarly, the DLC coating using the GCIB treatment might change the mechanical properties of the probe surface, for example, Young's modulus or hardness. The effects on these property changes brought about by GCIB fabrication need to be studied further in order to understand them precisely.

5. Conclusions

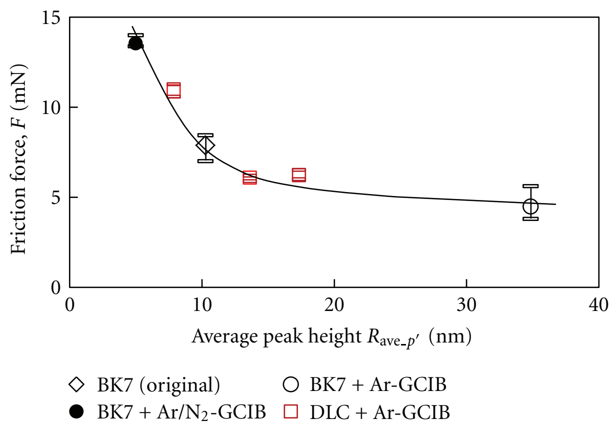
We fabricated supersmooth probes for pin-on-disk sliding tests by applying gas cluster ion beam (GCIB) irradiation to glass convex lenses. Under varying GCIB processing conditions, we obtained a wide range of surface roughnesses of the probes, with values that ranged from 1.08 to 4.30 nm in R_a units. Using these probes, we compared PFPE monolayer lubricant films with different numbers of hydroxyl end groups (Z-dol, Z-Tetraol, and Z-TMD). In the case of Z-dol, the friction force remained nearly constant over the



(a) Zdol

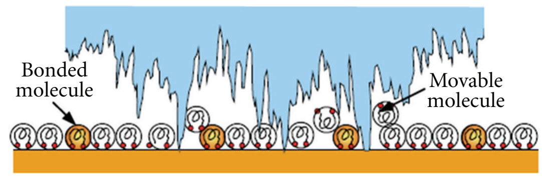


(b) Z-tetraol

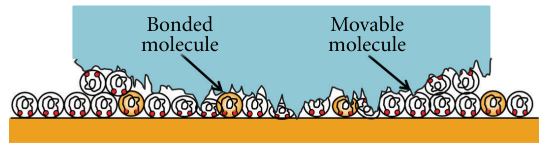


(c) Z-TMD

Figure 5: Comparison of friction forces among three types of lubricants with different numbers of hydroxyl end groups.

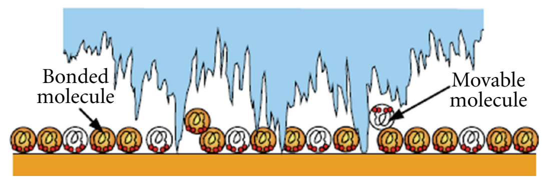


(a) Rougher probes

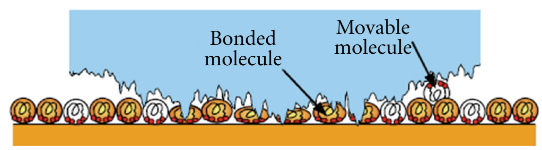


(b) Smoother probes

Figure 6: Multiple-asperity contact model that holds both for relatively rough and relatively smooth probes for a movable molecule dominant film.



(a) Rougher probes



(b) Smoother probes

Figure 7: Transition model from multiple- to single-asperity contact that occurs with decreasing surface roughness for a bonded molecule dominant film.

experimental roughness range. In the case of Z-Tetraol and Z-TMD, the friction force increased with decreasing roughness.

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