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Effect of pore depth on tribological behavior of anodic alumina films under nano-thin film lubrication

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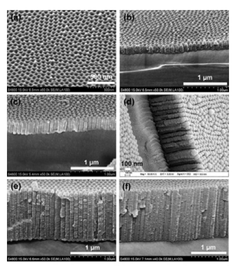
HIGHLIGHTS

- PFPE modified PAA films with high PDEP-to-PDIA ratios were prepared.
- The friction property of the PAA films improved with an increase in the PDEP value.
- The films with large PDEP value had high load-carrying capacity.
- The deep pore helped to make PFPE film has better contact with the alumina surface.
- The deep pore helped to store lubricant and trap wear debris.

GRAPHICAL ABSTRACT



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ABSTRACT

A number of parameters such as the dimple depth, dimple area density, and dimple shape, affect the tribological performance of dimpled surfaces, and a comprehensive tribological characterization of the depth-dependent nature of their material properties would be useful. However, only surfaces with dimples with depth-to-diameter ratios lower than 1 have been investigated. In this study, porous anodic alumina (PAA) films with high pore depth (PDEP)-to-pore diameter (PDIA) ratios (approximately 2.8–30) were prepared. The as-prepared films had the same PDIA value (50 nm) but different PDEP values (140 nm, 350 nm, 700 nm, 1100 nm, and 1500 nm). Perfluoropolyether was used as the lubricant to improve the tribological properties of the as-prepared films. The effects of the PDEP value on the tribological properties of the as-prepared films were investigated systematically. It was found that the antiwear performance of the PAA films was closely related to the combined effects of flexibility, mobility, and inherent lubricity of PFPE molecules and PDEP values in nanoporous structures as a reservoir for lubricants and wear particles. For PDEP-to-PDIA ratios greater than 1, under the conditions studied, the friction performance of the PAA films improved with an increase in the PDEP value. Further, after a critical PDEP value, the applied load had little effect on the friction coefficient and antiwear life of the films. The results of this study should aid the fabrication of stable films for industrial applications and act as a bridge between science and engineering on the micro/nanoscale.

1. Introduction

As the surface-to-volume ratio of the components of microelectromechanical and nanoelectromechanical devices increases, a variety of material properties, including the electronic, optical, mechanical, and tribological properties of these components,

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become strongly dependent on the size, shape, and regularity of the structure of the material used [1–6]. Thus, comprehensive micro- and nanotribological characterizations of the scale-dependent nature of the material properties would contribute to the design of reliable devices suitable for industrial applications and provide a bridge between the relevant science and technology on the micro/nanoscale.

In recent years, efforts have been made to control friction and wear by introducing surface textures [7–11]. Introducing dimple-type textures on frictional surfaces to improve their wear properties is an area of active research. The results of several theoretical and experimental studies show that a number of parameters such as the surface roughness, dimple depth, dimple area density, dimple shape, lubricant properties, and operational speed affect the tribological performances of textured surfaces [8–16]. Qiu et al. simulated the load-carrying capacity, film thickness, dimple depth, dimple density, cavitation pressure, leakage and friction force of full-film textured surfaces and the relationship between these performance parameters [12]. It was found that, under the conditions simulated, surface roughness can improve the load-carrying capacity, but its effect is limited. Ramesh et al. reported experimental and numerical investigations of the friction characteristics of microtextured surfaces under mixed and hydrodynamic lubrication [10]. The trends obtained in the experiments matched well with the simulations. However, these results are obtained under mixed and hydrodynamic lubrication and few reports are studied under the boundary lubrication, especially under the nano-thin film lubrication. Moreover, numerical parametric studies have been performed in which the dimple depth was studied for dimple-to-diameter ratios of less than 1 and as low as approximately 0.1 [17–21]. However, there are few reports on the effects of depth-to-diameter ratios greater than 1 on the tribological performance of materials under nano-thin film lubrication.

Porous anodic alumina (PAA) films with a unique honeycomb structure can be readily synthesized through a simple two-step procedure [22–25]. The pore diameter (PDIA; 10–400 nm) and pore depth (PDEP) of such films can be controlled over a narrow distribution range by choosing the proper type and concentration of the electrolyte, the applied anodization potential, and the temperature [26–29]. Highly ordered, straight nanopores in hexagonally close-packed arrays with aspect ratios as high as 1000 can be readily achieved. Such PAA films have attracted considerable attention because they are suitable for use as filters, substrates for cell cultivation, optical waveguide sensor, magnetic storage, photovoltaic solar cells, templates to fabricate nanowire or nanorod, etc. [30–36].

It has been found that PAA films exhibit high hardness, good wear resistance, and excellent corrosion resistance [37,38]. However, the friction coefficient of PAA films is still too high to meet the requirements for practical use. In situ deposition of the films or the synthesis of lubricants in the pores or on the surfaces of the films is an easy way of reducing their friction coefficient. It has also been found that incorporating MoS_2, polytetrafluoroethylene, mineral oil, carbon nanofibers, nickel, or iodine compounds within the pores of PAA films helps reduce the friction coefficient and improves their wear resistance properties [39–43]. Similarly, studies have also shown that a nanoporous structure can act as a lubricant reservoir and improve the lubrication properties [44,45]. Moreover, we had previously found that perfluoropolyether (PFPE) and octadecyltrichlorosilane (OTS) could be used to improve the tribological properties of the surfaces of PAA films and that PAA modified with PFPE exhibited a lower friction coefficient and longer wear life than did PAA modified with OTS [46]. Furthermore, due to the good viscosity characteristics, low surface tension, low volatility and good chemical and thermal stability, good re-flow properties and excellent lubricity, PFPE has been used as lubricant for application in microelectromechanical and nanoelectromechanical system [47]. Therefore, in this study, PFPE was selected to fabricate nano-thin lubricant films to improve the tribological characteristics of PAA.

Kim et al. and Ge et al. have reported that the pore density of PAA films affects their frictional properties [48,49]. However, there are few reports on the effect of pore depth on the characteristics of PAA films. In this study, PAA films with the same PDIA value but different PDEP values (and PDEP-to-PDIA ratios of more than 1) were fabricated, and the effect of the PDEP value on the tribological behavior of the PAA films was systematically investigated.

2. Experimental

2.1. Materials

High-purity aluminum foil (99.999%, thickness of approximately 150 μm); PFPE ($HOCH_2CF_2O—(CF_2—CF_2O)_m—(CF_2O)_n—CF_2CH_2OH$, where m and n are integers; molecular weight = 3800; commercial name Zdol-3800) was provided by Aldrich Chem. Co. Ltd. All the other chemicals used were of analytical grade and used as received. Ultrapure water was used in all the experiments.

2.2. Fabrication of PAA films with the same PDIA value but different PDEP values

PAA films with the same PDEP value (4500 nm) but different PDIAs values (10 nm, 50 nm, 200 nm, and 400 nm) were fabricated by a two-step anodization method to select the optimum PDIA, as shown in Fig. S1 (Supplementary material). Using PFPE as the lubricant, the effect of PDIA on the tribological properties of the as-obtained templates was investigated systematically. The detail analysis is described in Supplementary material. It was found that when the PDIA was 50 nm and 200 nm, the friction coefficients of the PAA films were relatively stable and low even when the load was 300 mN and the scratches on the PAA films were nearly indistinguishable (Supplementary material, Figs. S2 and S3). So in this study, 50 nm was selected as the PDIA.

PAA films with the same PDIA value but different PDEP values were fabricated by a similar two-step anodization method [50,51]. Prior to being anodized, Al foil samples with a diameter of 28 mm were sequentially cleaned by ultrasonication in acetone, ethanol, and ultrapure water for 5 min each to remove any surface contaminants. The cleaned Al wafers were then electropolished in a mixture of perchloric acid and alcohol ($HClO_4/C_2H_5OH$ = 1:4 by volume) for 5 min to smoothen any surface irregularities. The electropolished Al wafers were first anodized in a 0.3 M oxalic acid solution under a constant voltage of 40 V and at a temperature of 3–5 °C. The oxide layer was then removed by dipping the wafers in a mixture of 1.8 wt% chromic acid and 6.0 wt% phosphoric acid at 60 °C for 1 h.

Table 1
The preparation conditions of PAA with different PDEPs.

	Bath solution	Voltage (V)	Time for first anodization	Time for second anodization (min)
1	0.3 M oxalic acid solution	40	40 min	2
2	0.3 M oxalic acid solution	40	1 h	4
3	0.3 M oxalic acid solution	40	1 h	9
4	0.3 M oxalic acid solution	40	1 h	11
5	0.3 M oxalic acid solution	40	1 h	16