Accelerated Wear Testing with a Microfabricated Surface To Evaluate the Lubrication Ability of Biomolecules on Polyethylene

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ABSTRACT: Wear of ultrahigh-molecular-weight polyethylene (UHMWPE) and wear-particle-induced osteolysis and bone resorption are the major factors causing the failure of total joint replacements. It is feasible to improve the lubrication and reduce the wear of artificial joints. We need further understanding of the lubrication mechanism of the synovial fluid. The objective of this study is to evaluate the lubricating ability of three major components in the synovial fluid: albumin, globulin, and phospholipids. An accelerated wear testing procedure in which UHMWPE is rubbed against a microfabricated surface with controlled asperities has been developed to evaluate the lubrication behavior. An analysis of the wear particle dimensions and wear amount of the tests has provided insights for comparing their lubrication performance. It is concluded that the presence of biomolecules at the articulating interface may reduce friction. A higher concentration of a biological lubricant leads to a decrease in the wear particle width. In addition, in combination with the wear results and mechanical analysis, the roles of individual biomolecules contributing to friction and wear at the articulating interface are discussed. These results can help us to identify the role of the biomolecules in the boundary lubrication of artificial joints, and further development of lubricating additives for artificial joints may be feasible. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 108: 2428–2437, 2008

Key words: biomaterials; interfaces; particle size distribution; polyethylene (PE); proteins

INTRODUCTION

The lifetime of total joint implants is mainly determined by their tribological properties. From years of research and development, an artificial joint system consisting of a Co–Cr alloy sliding on an ultrahigh-molecular-weight polyethylene (UHMWPE) surface has been proven to function well and is widely used clinically. Currently, the wear of UHMWPE and wear-particle-induced osteolysis and bone resorption are the major factors causing the failure of total joint replacements.[1] In the past several years, the wear of UHMWPE has been dramatically reduced by the use of highly crosslinked UHMWPE.[2–4]

In previous studies,[5–7] to study the effects of the size and shape of wear particles on the immunological responses, a wear testing procedure in which UHMWPE is rubbed with controlled asperities on surfaces has been developed to generate UHMWPE wear particles with controlled sizes and shapes. Figure 1 shows the microcutting process of UHMWPE particle generation. The wedge features are fabricated on the silicon surface. Through the rubbing of UHMWPE with silicon surfaces with wedge features, the mass production of the particles can be achieved, and the particle size and shape can be controlled by the dimensions of the surface features and the mechanical conditions of the rubbing process. The original purpose of this series was to generate UHMWPE wear particles for investigating particle-induced immunological responses. Furthermore, we can adopt this idea to perform accelerated wear tests of materials. The uniform surface asperities are fabricated on the material surface and applied in a linear reciprocating wear process to rub against the testing materials. The friction, wear rate, and wear particle morphology under a patterned stress situation can

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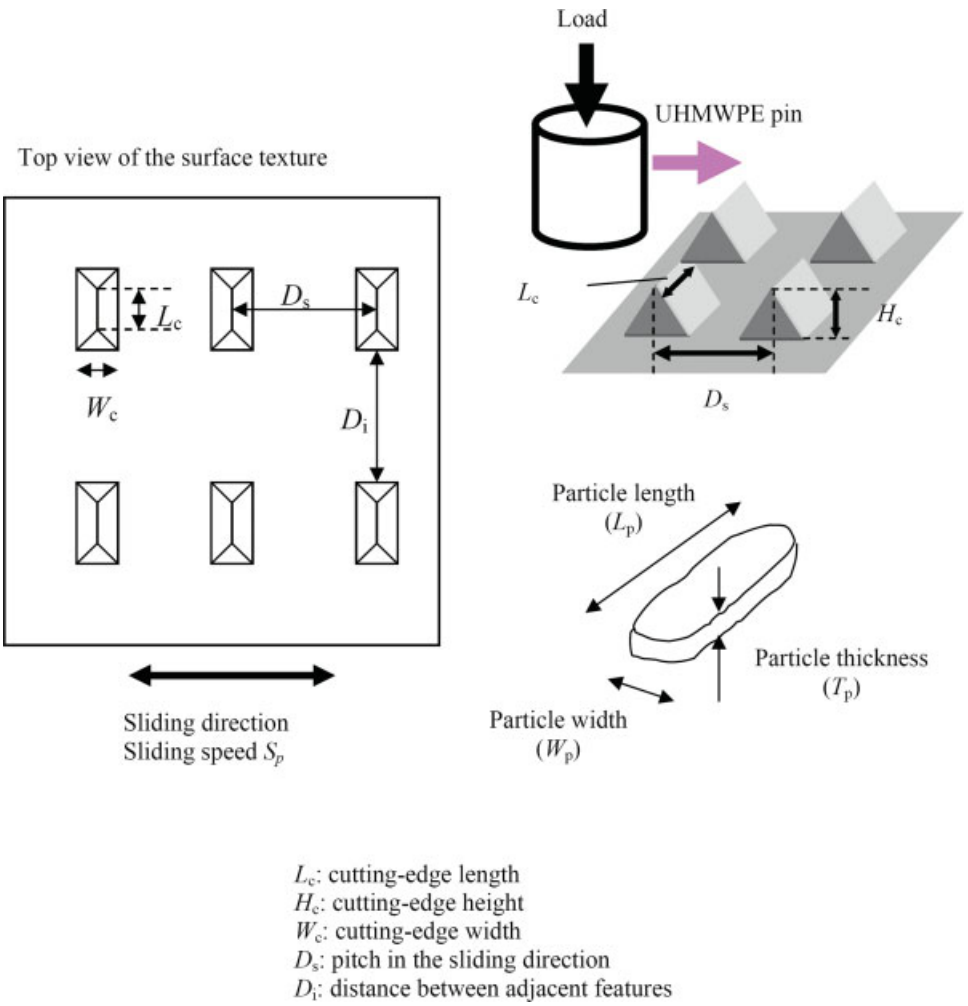

Figure 1 Schematic of the particle generation process in which microfabricated wedge features are rubbed against UHMWPE. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

be observed and measured. The advantage is that the testing duration can be shortened, and a comparison of the wear rates can be made. By observation of the wear particles retrieved from the tests, the lubrication mechanism of the lubricating molecules can be evaluated. A previous study also adopted this procedure to investigate biological lubrication on the wear of UHMWPE.[8]

From an engineering viewpoint, the synovial fluid can be viewed as a lubricant containing main components such as albumin, globulin, lipids, and hyaluronic acid. Each component of the synovial fluid may reveal various frictional behaviors. Under high-load and low-sliding-speed conditions, boundary lubrication is believed to be the dominant mechanism during the articulation of artificial joints. Boundary lubricants of biomolecules in the synovial fluid may form protective layers on the sliding surfaces. These protective layers keep the two surfaces from direct collision and thus decrease the possibility of wear of materials.[9,10] Some studies have attempted to investigate the lubrication ability of these biological molecules.[11–13] It is still feasible to further improve the lubrication and reduce the wear of artificial joints *in vivo*. We need further understanding of the lubrication mechanism of the synovial fluid.

The objective of this study was to adopt accelerated wear testing procedures to evaluate the lubricating ability of three major components in the synovial fluid: albumin, globulin, and phospholipids. A combined analysis of the wear particle morphology and wear rate of the articulation process between microfabricated surfaces and polyethylene can assist us in understanding the lubrication mechanism of the biomolecules. These results can help us to identify the role of the biomolecules in the boundary lubrication of artificial joints, and further development of lubricating additives for artificial joints may be feasible.

EXPERIMENTAL

Linear reciprocating wear tests were carried out under the lubrication of albumin, globulin, and

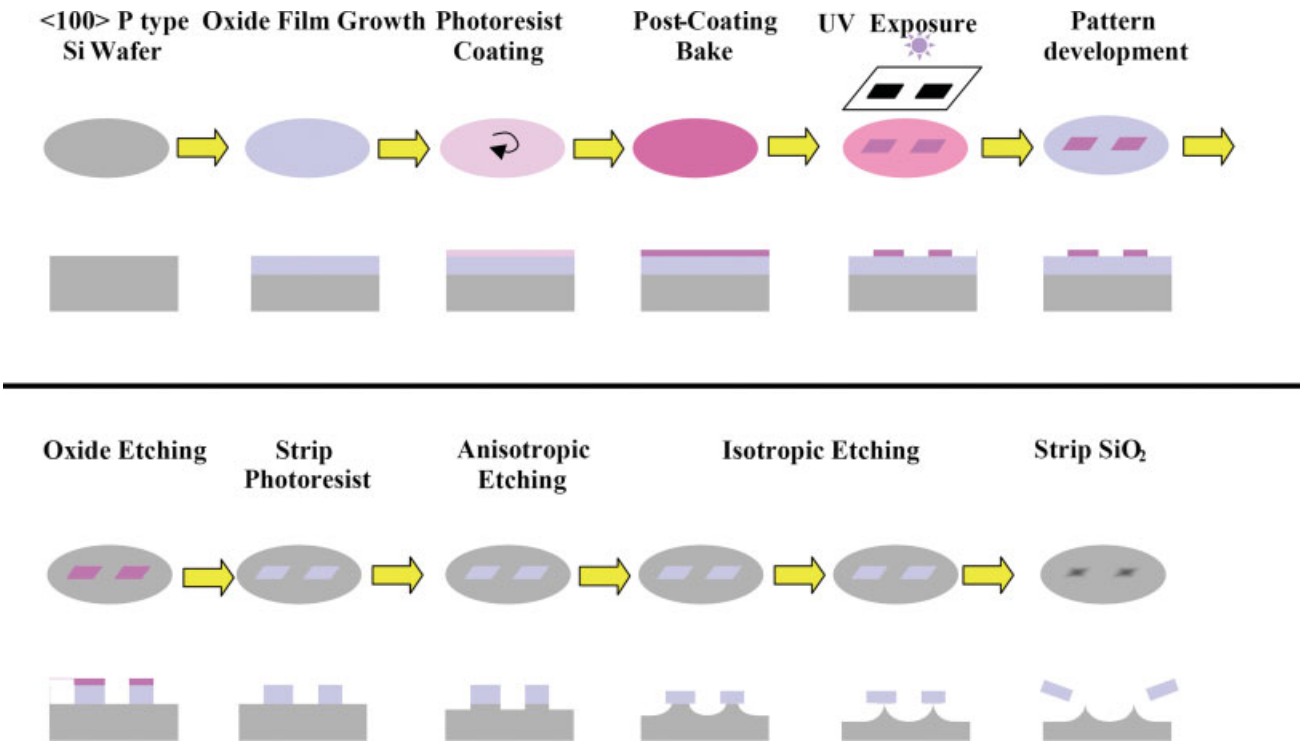

Figure 2 Schematic of the microfabrication process of surface features on the silicon wafer. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

phospholipid solutions in this study. The articulating materials were UHMWPE and the textured surface of silicon. The generated wear particles were isolated, and the dimensions of the particles were quantified. The details of the materials, wear process, particle collection, and particle characterization procedures are described next.

Material and lubricants

UHMWPE materials (GUR1050), obtained from United Orthopaedic Corp. (Taipei, Taiwan), were used in this study. UHMWPE cylinder pins were machined to be 6.35 mm in diameter and 25.4 mm in length with diamond turning on both end surfaces without polishing. The mean roughness of the UHMWPE pin end surface was 0.82 μm. Lubricants of albumin, globulin, and phospholipid solutions were prepared by the dissolution of human serum albumin (HSA) powders, human immunoglobulin G (IgG) powders, and dipalmitoyl phosphatidyl choline (DPPC) powders obtained from Sigma (St. Louis, MO) in saline solutions, respectively. Different concentrations of 0.1, 0.5, 2.5, and 12.5 mg/mL of the aforementioned lubricants were prepared. All UHMWPE pins were presoaked in the specified lubricant for at least 15 days so as to become completely saturated with the immersed lubricant in the experiment.

Microfabrication of the textured surface

Texturing of the silicon wafer surface was achieved by photolithography patterning and etching of the bulk substrate.[7] A flow chart of the fabrication process is shown in Figure 2. Two-inch-diameter polished type P silicon wafers with (100) orientation were purchased from Summit-Tech (Taipei, Taiwan) and used as the base material for the texturing process. Wet oxidation of the silicon wafers was carried out in a glass-tube oven at 1100°C for 135 min to form a silicon dioxide film with a thickness of 1 μm. A pattern of rectangles with different sizes and aspect ratios was formed on a chrome direct-writing photomask. The silicon dioxide surface was spin-coated with a Shipley 1813 positive photoresist (Philadelphia, PA). The dark-featured photomask with rectangular patterns (5 μm × 60 μm) was then placed on the photoresist surface and exposed to an ultraviolet source in a mask aligner to decompose the surrounding polymer surface, leaving a positive rectangular pattern. The decomposed photoresist polymer was then removed in a Shipley 351 developer. The resulting positive photoresist surface was then etched first with a BOE solution (Buffer Oxide Etch) (diluted buffer HF solution) to etch away the SiO_2 in a wet chemical bath. The photoresist was then removed through washing with acetone/alcohol. Subsequently, the silicon material was subjected to isotropic silicon etching (HNA etchant; HF/HNO_3/

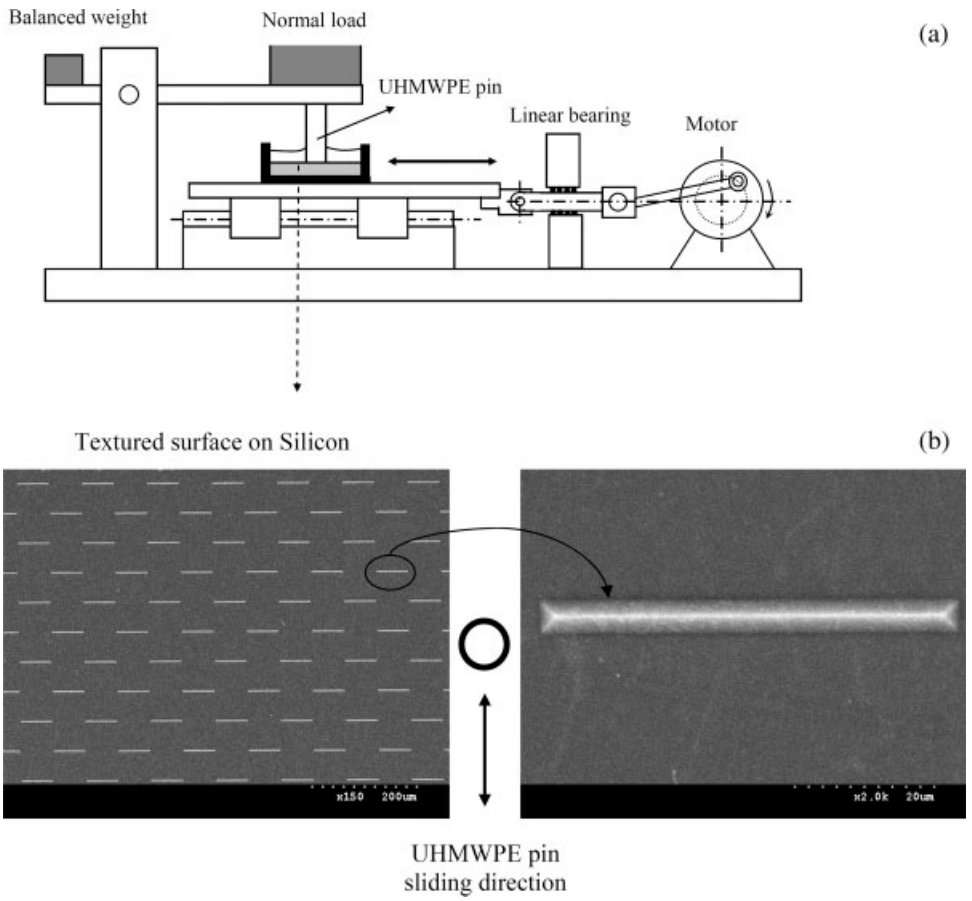

Figure 3 (a) Experimental setup of linear reciprocating wear testing and (b) SEM images of the microfabricated surface used in this study.

CH_3COOH liquid volume ratio = 8 : 75 : 17) in a wet chemical bath at room temperature. The SiO_2 layer was removed after an isotropic undercutting etching process. The resulting surface features were an array of rectangular ridges with sharp edges. Finally, a layer of a 5-nm Cr coating was evaporated onto the surface to increase the strength and wear resistance of the surface texture. The height of the surface textures were measured with a Mahr profilometer (Gottingen, Germany), and the feature length and width were measured from scanning electron microscopy (SEM) observations.

Wear process

A linear reciprocating wear test was applied to generate UHMWPE wear particles. ASTM F 732 was used as a guideline.[14] The setup of the system is shown schematically in Figure 3. A UHMWPE pin and three control pins were weighed three times. The control pins were soaked in the lubricant at the same liquid level used for the wear testing condition. The UHMWPE pin was then mounted on the tester. Linear reciprocating wear tests were run with a nominal contact pressure of 3 MPa, a stroke length of 19 mm, a frequency of 1.5 Hz, and an average sliding speed of 57.2 mm/s for 4 h. After wear testing, the four pins were weighed, and the wear loss of the testing pin was obtained after the adjustment of the weight change from the control pins.

Isolation of the wear particles

UHMWPE particles were collected by repeated water rinsing of the sample, sample holder, and parts that came into contact with particles in a sterilized beaker. To digest the biological components in the lubricant, 5 mL of a collected serum solution was added to a 20-mL NaOH solution (5*N*) in a 65°C water bath for 24 h. Three-milliliter digested solutions were added to 2-mL glucose gradient solutions (5, 10, 20, and 50%). The solution was then centrifuged at 4000 rpm and 4°C for 2 h. The upper solid layer was collected and rinsed with 30 mL of purified water. The solution was then agitated at 65°C for 1 h, and this was followed by ultrasonification for 10 min to disperse the polyethylene particles. This solution was then added to 0.90 and 0.96 g/cm^3 isopropyl alcohol solutions (2 mL), and this was followed by centrifugation at 4000 rpm and

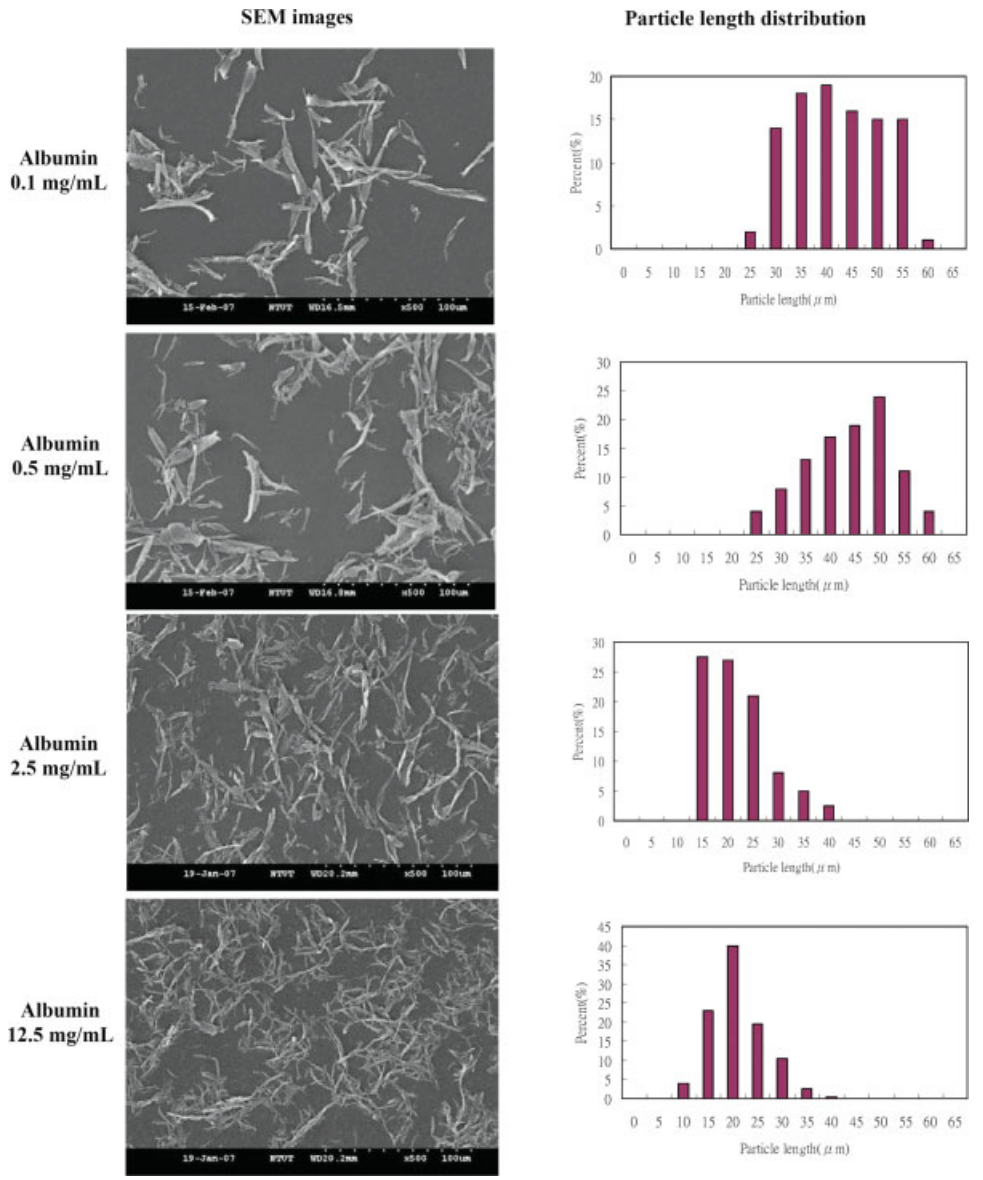

Figure 4 SEM images of the UHMWPE wear particles and particle length distributions under the lubrication of HSA solutions with concentrations of 0.1, 0.5, 2.5, and 12.5 mg/mL. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

25°C for 1 h. The particles existing in the interface between the two different isopropyl alcohol solutions were collected.

Analysis of the particles

The particles, which were dispersed well in a known volume of the solution, were collected on a 0.1-μm-pore-size membrane through a vacuum filtration process. The particles, collected on the filter paper, were examined with a scanning electron microscope. Micrographs of the particles were then analyzed with image analyzer software (Scion Image, a personal computer version of NIH Image) to measure their dimensions. Measurements were made for at least 300 particles in each case. The chemical component of the collected particles was confirmed to be polyethylene by Fourier transform infrared measurements.[6]

RESULTS

The SEM image of the microfabricated surface used in this study is shown in Figure 3. The average length, width, and height of the cutting features on the surface are 56.8 ± 0.2, 4.5 ± 0.1, and 2.1 ± 0.1 μm, respectively. Tables I–III list the length, width, and aspect ratio of the UHMWPE wear particles and wear amount under the lubrication of albumin (HSA), globulin (IgG), and phospholipids (DPPC), respectively. The SEM images of the wear particles and the distri-

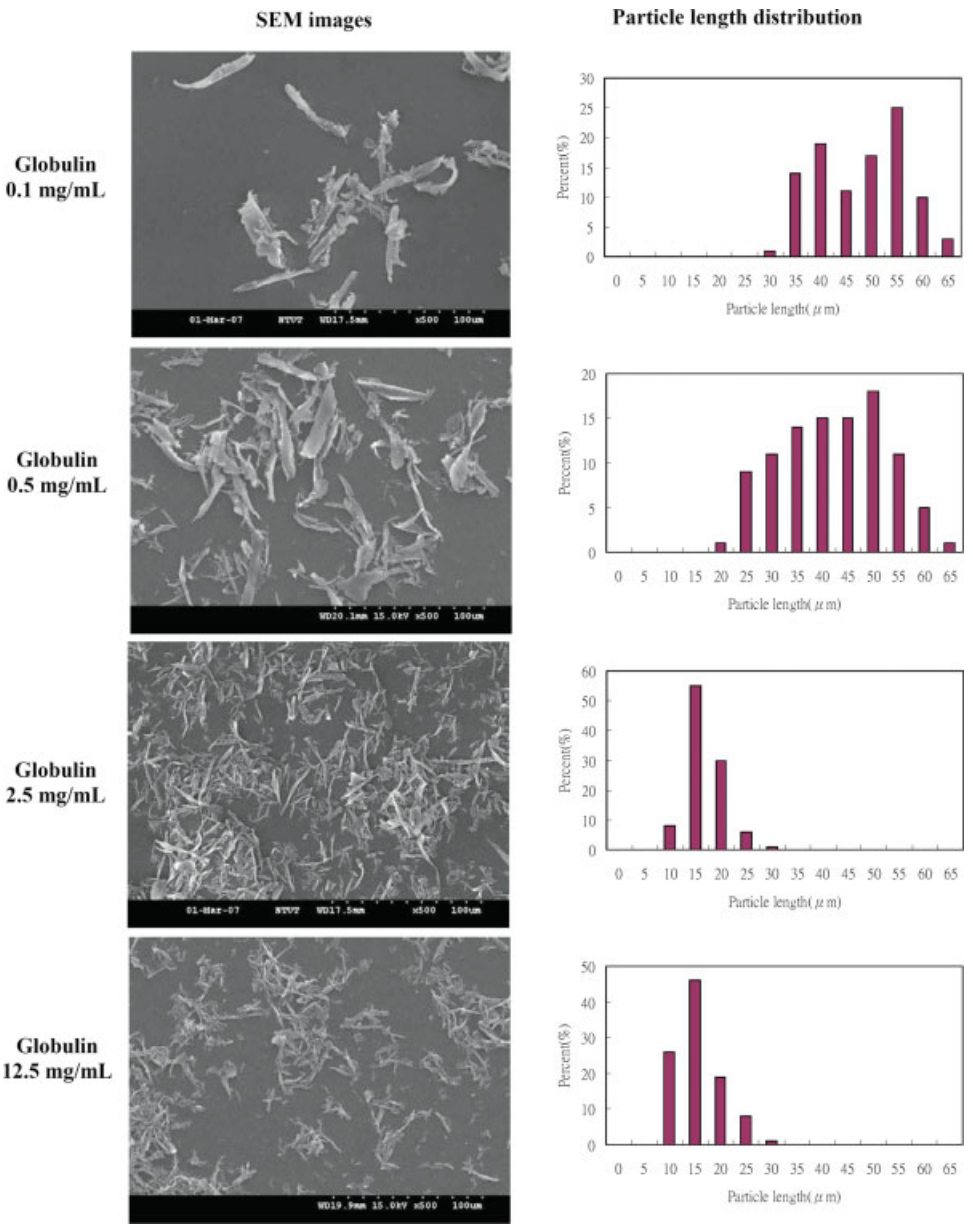

Figure 5 SEM images of the UHMWPE wear particles and particle length distributions under the lubrication of IgG solutions with concentrations of 0.1, 0.5, 2.5, and 12.5 mg/mL. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

butions of the particle length under the lubrication of HSA, IgG, and DPPC are shown in Figures 4–6.

Figure 4 indicates that a higher concentration of the albumin solution leads to a decrease in the wear particle length. Table I shows that the decrease in the particle width and aspect ratio is associated with an increasing albumin concentration. A similar effect of the solution concentration on the particle length was also observed with globulin and phospholipid solutions, as shown in Figures 5 and 6. As indicated in Table II, an increase in the globulin concentration results in a decrease in the particle width. A larger aspect ratio of the wear particle generated in globulin was observed with a higher concentration of globulin. A reduction of the particle width comes along with an increase in the phospholipid concentration, as shown in Table III. However, the aspect ratios of the wear particles remain about the same at different phospholipid concentrations. We further compare the concentration effect on the wear of UHMWPE in various solutions. It is indicated that a higher albumin concentration leads to an increase of wear [Table I and Fig. 7(a)]. In contrast, the wear amount of UHMWPE is reduced with an increasing globulin concentration [Table II and Fig. 7(b)]. Under lubrication in phospholipid solutions, an increase in the phospholipid concentration results in an increase in wear of UHMWPE [Table III and Fig. 7(c)].

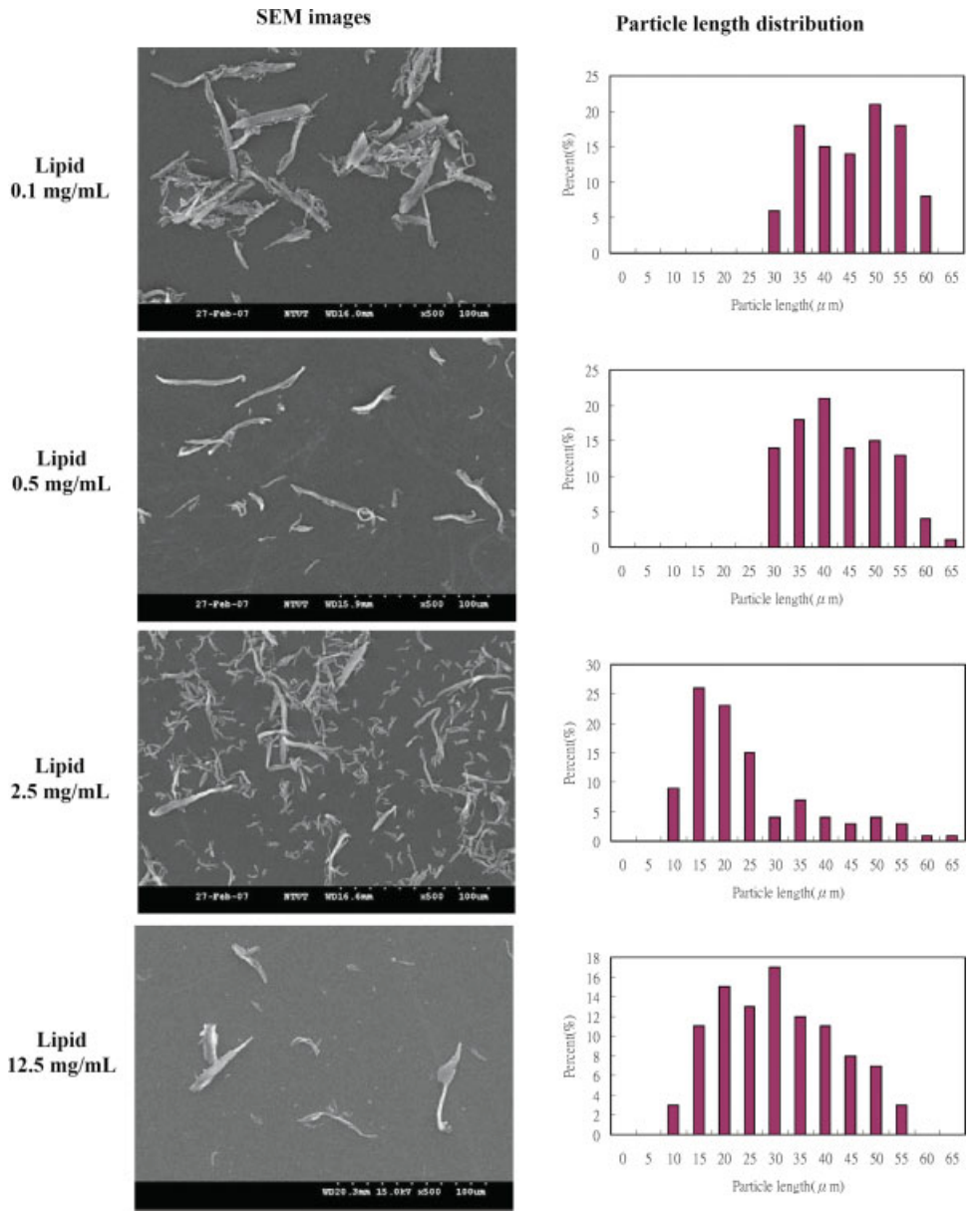

Figure 6 SEM images of the UHMWPE wear particles and particle length distributions under the lubrication of DPPC solutions with concentrations of 0.1, 0.5, 2.5, and 12.5 mg/mL. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

DISCUSSION

The aforementioned results present the effects of different molecules on the wear and wear particle morphology under an accelerated wear testing procedure in which UHMWPE is rubbed against a microfabricated surface. Various outcomes were observed with different lubricants (albumin, globulin, and phospholipid solutions). Because of the uniform surface asperity rubbing against UHMWPE, it is

TABLE I
Morphology and Wear of UHMWPE Particles in HSA Solutions

	0.1 mg/mL	0.5 mg/mL	2.5 mg/mL	12.5 mg/mL
Particle length (μm)	44.67 ± 8.67	46.27 ± 8.52	28.33 ± 7.00	23.41 ± 5.43
Particle width (μm)	5.52 ± 0.93	6.08 ± 1.32	4.13 ± 1.03	3.34 ± 0.27
Aspect ratio	8.23 ± 1.77	7.86 ± 1.80	7.10 ± 1.85	7.19 ± 1.70
Wear amount (mg)	7.6	27.8	18.57	29.63

TABLE II
Morphology and Wear of UHMWPE Particles in IgG Solutions

	0.1 mg/mL	0.5 mg/mL	2.5 mg/mL	12.5 mg/mL
Particle length (μm)	50.47 ± 8.93	44.32 ± 10.59	19.61 ± 3.50	17.96 ± 4.47
Particle width (μm)	8.98 ± 2.22	7.06 ± 1.80	2.93 ± 0.48	2.70 ± 0.57
Aspect ratio	5.89 ± 1.54	6.49 ± 1.67	6.79 ± 1.29	6.81 ± 1.73
Wear amount (mg)	66.7	48.1	19.37	18.3

thought that a mechanical analysis of the squeezed biomolecular film on the interface of the wedge feature may provide explanations of the results. To understand the microcutting mechanism of UHMWPE particle generation in water, a scaled-up wedge tip scratching test was carried out in a previous study.[15] By the design of sliding experiments with a constant normal load and constant penetration depth, single wedge sliding tests with scaled-up tips have enabled the direct observation of the kinematics of the microcutting process and the measurement of the strain hardening of UHMWPE.[15] In addition, soft X-ray absorption spectroscopy has been used[16] to measure the molecular orientation at UHMWPE surfaces subjected to different shear motions by the rubbing of a UHMWPE pin against a polished Co–Cr disk. The results show that a higher degree of chain alignment is induced by unidirectional rubbing of the UHMWPE surface. It can be concluded that the molecular chains tend to align and orient in a common direction when the surface is subjected to a directional force such as shear stress from sliding. On the basis of these experimental findings, the mechanism of particle generation in water with microfabricated surfaces can be proposed as follows[15]: (1) deformation of the material occurs because of the penetration and sliding processes of the surface features; (2) strain hardening occurs under a surface-feature sliding process; (3) UHMWPE molecules align in the direction in which the shear stress is applied, and the surface layer of the material becomes more brittle; and (4) detachment of the particle occurs at the tip edge/material interface according to the fracture of UHWMPE. We have also established a contact mechanics model based on the calculation of the penetration depth and the displaced volume of UHMWPE iteratively on a force–balance basis.[17] Therefore, a calculation procedure has been developed to predict the UHMWPE particle volume with a given surface-feature geometry and applied loads.

According to the aforementioned findings, we think that the sliding distance during the process may determine the wear particle width. Therefore, the existing biomolecules at the interface are critical for the friction between the surface feature and UHMWPE. The results of effects of biomolecular lubricants on the particle morphology may provide information regarding the lubrication ability of the biomolecules. A schematic of the force analysis at the interface is shown in Figure 8. The sliding process of the surface feature over UHMWPE results in a resisting force on UHMWPE. The vertical direction of this resisting force pushes up the UHMWPE while lateral movement continues. The interfacial friction appears as an encounter force to prevent the UHMWPE from being pushed away. As shown in Figure 8, an increase in the interfacial friction leads to a larger downward force. Therefore, the surface feature shall slide longer to complete a microcutting process, and this will result in a larger particle width. In addition, the accumulated molecular layer at the interface may reduce the actual penetration depth of the cutting edge into UHMWPE and further shorten the lateral sliding distance. It is thought that higher concentrations of the protein and lipid solutions will reduce the interfacial friction. The relative motion of the cutting edge on UHMWPE materials becomes more slippery. This slippery phenomenon has two effects on the microcutting process of the particle generation. With a low frictional resistance, it maintains a higher net force pushing the cutting edge away from the material surface. The time needed to complete the generation of a single particle is shortened. Besides, a higher concentration

TABLE III
Morphology and Wear of UHMWPE Particles in DPPC Solutions

	0.1 mg/mL	0.5 mg/mL	2.5 mg/mL	12.5 mg/mL
Particle length (μm)	48.09 ± 8.81	45.31 ± 9.15	27.21 ± 12.71	33.24 ± 11.53
Particle width (μm)	7.06 ± 1.44	5.93 ± 1.35	4.30 ± 1.23	5.44 ± 1.79
Aspect ratio	6.97 ± 1.38	7.93 ± 2.10	6.22 ± 1.92	6.29 ± 1.74
Wear amount (mg)	37.96	40.5	68.6	58.3

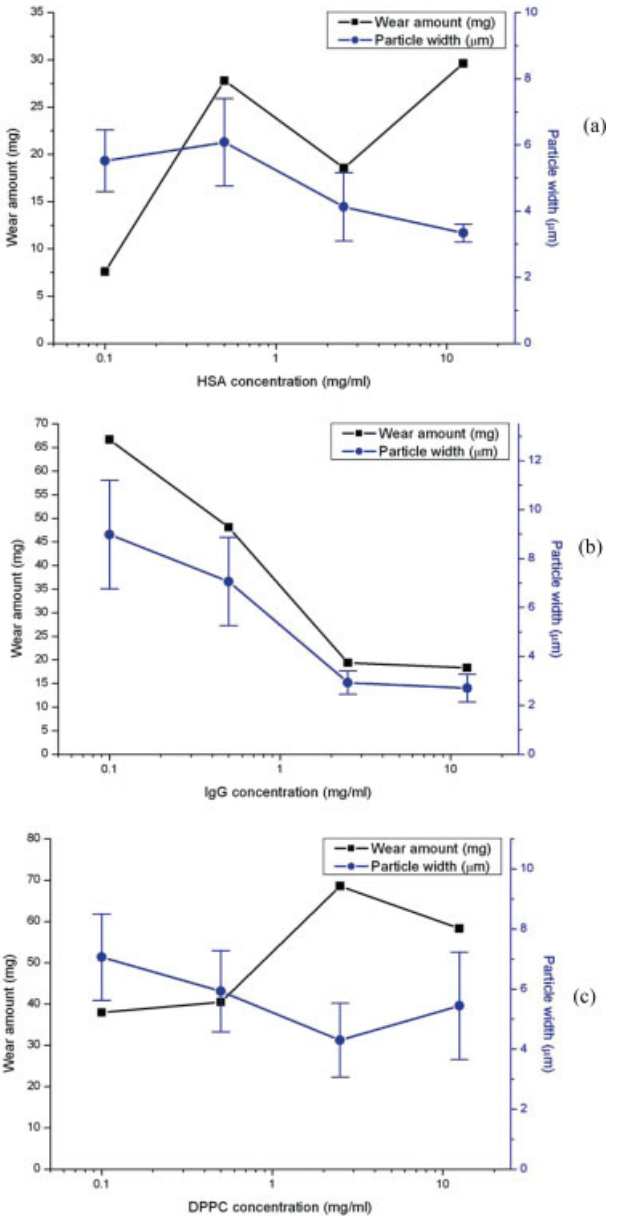

Figure 7 Plots of the wear amount and wear particle width versus the concentrations of solutions of (a) HSA, (b) IgG, and (c) DPPC. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

of the lubricant may contribute to thicker boundary lubricating layers at the interface. The experimental results in this research show that a higher concentration of lubricants leads to a smaller particle width, and this is consistent with our mechanical analysis.

In comparison with the UHMWPE wear particles generated with the same microfabricated surface in water,[8] the particle widths generated in biological lubricants are smaller. This indicates the lubrication effect of the biomolecules on the microcutting process. However, it can be seen in Tables I–III and Figure 7 that the particle width decreases with increasing lubricant concentrations for albumin, globulin, and lipid solutions. The results imply that further addition of the biological lubricant cannot reduce the friction further. Instead, the higher concentration of biomolecules may adhere to and accumulate on the sliding surfaces during the articulation process. Hence, the penetration depth of the cutting edge into UHMWPE may be reduced, and this may further lead to a decrease in the particle width.[17] Besides, it was observed that only with globulin solutions was the wear of UHMWPE also reduced with increasing lubricant concentration. With a shortened wear particle width, we observed an increase in the wear amount with increasing concentrations of albumin and phospholipid solutions. Wong et al.[18] indicated that the microcutting phenomenon of particle generation is an abrasion process related to low-cycle fatigue. One explanation of this result is that the particle may be generated after a different number of cycles for the wedge sliding over UHMWPE with different biological lubricants.

We think that interfacial biomolecules during the articulation of materials may play two functional roles. One is to reduce the interfacial friction by adhering to the articulating surface. Moreover, the biomolecules act as a bridge to transfer the frictional work into the materials during the tribological process. The accumulated energy in the material may finally contribute to the breakdown of the molecular bonds and lead to the wear of UHMWPE. Currently, the effects of the physical and chemical properties of interfacial biomolecules on the friction and wear remain a mystery. Biomolecular structure information, such as the molecular size, three-dimensional

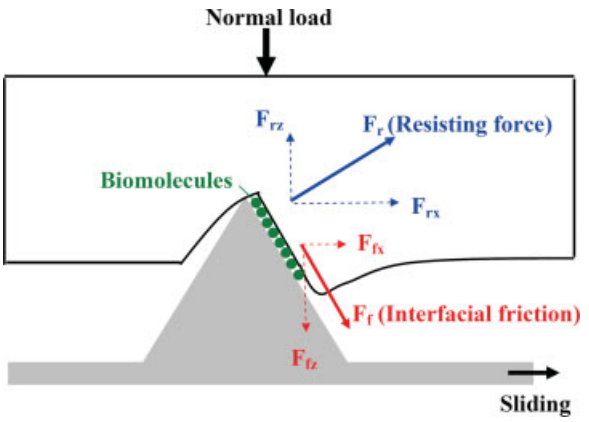

Figure 8 Mechanical analysis of the resisting force (F_r) and interfacial friction (F_f) for the process of the wedge feature sliding over UHMWPE (F_{rz} = upward direction of the resisting force; F_{rx} = horizontal direction of the resisting force; F_{fz} = downward direction of the interfacial friction; F_{fx} = horizontal direction of the interfacial friction). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

structure, and hydrophilicity properties, are factors affecting the molecular adhesion mechanism and friction. On the other hand, the density and composition of the biomolecules may be influential on the frictional energy transfer process. Hence, further studies of molecular dynamics simulations and biomolecular adsorption experiments shall be carried out to provide more fundamental insights.

CONCLUSIONS

Through the rubbing of a microfabricated surface against UHMWPE, an accelerated wear testing procedure has been successfully established to evaluate the lubrication behaviors of albumin, globulin, and phospholipids in this study. The morphology of the wear particles has been characterized, and the measurements of wear particle widths have been applied to evaluate the interfacial friction. In addition, the results for the wear amount can help us to discuss the antiwear ability of the biomolecules. A smaller wear particle width has been observed in albumin, globulin, and lipid solutions than that for particles generated in water. This indicates the better lubrication of the three biological lubricants during the microcutting process. However, no conclusive trend for the wear rate has been obtained from the three biological lubricants. It is proposed that the different biomolecular structures may contribute to the variation of the antiwear ability of the lubricants. The novelty of this new tribological testing protocol enables the fast screening of the friction and wear properties of lubricants at the same time. The newly accelerated wear tests and the results revealed in this study have assisted us in identifying the role of the biomolecules in the boundary lubrication of artificial joints. This testing platform may be beneficial to further developing lubricating additives for artificial joints in the future.

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