Friction and Wear Behavior of Ultrahigh Molecular Weight Polyethylene as a Function of Crystallinity in the Presence of the Phospholipid Dipalmitoyl Phosphatidylcholine

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Received 14 July 2008; revised 13 April 2009; accepted 18 May 2009
Published online 22 February 2010 in Wiley InterScience (www.interscience.wiley.com). DOI: 10.1002/jbm.b.31587

Abstract: In this study, the friction and wear behavior of ultrahigh molecular weight polyethylene (UHMWPE) were evaluated as a function of polymer crystallinity in the presence of the phospholipid dipalmitoyl phosphatidylcholine (DPPC) dissolved in ethanol. Samples of UHMWPE were separately heat treated to get high and low crystallinity samples. Degree of crystallinity was evaluated using differential scanning calorimetry. Quantitative friction and wear experiments were conducted using a custom-made microtribometer with commercially available spherical Si3N4 probes in controlled and phospholipid-dissolved lubricants. The higher crystallinity sample exhibited slightly lower friction than the lower crystallinity in the control and decreased significantly when phospholipids were present. The higher crystallinity sample showed a higher wear resistance than the lower crystallinity sample during all reciprocating wear tests. DPPC acting as a lubricant had a marginal effect on the wear resistance of high crystallinity UHMWPE, whereas the low crystallinity sample became more prone to wear. Atomic force microscopy topography images and contact angle measurements of both samples before and after phospholipid exposure indicate that the higher crystallinity sample absorbed a greater density of DPPC. Increasing crystallinity is a way of escalating adsorption of surface active phospholipids onto UHMWPE to make it a more wear-resistant load-bearing material for total joint replacements.

Key Words: ultrahigh molecular weight polyethylene (UHMWPE), crystallinity, friction, wear, phospholipid

INTRODUCTION

For the past 40 years, ultrahigh molecular weight polyethylene (UHMWPE) has been the primary material used for the articulating cup of artificial hip joints.1–9 Its low friction and low wear resemble properties of cartilage lining natural human joints more than any other biocompatible material. However, it has been found that polymeric wear particles from the UHMWPE in artificial hip joints have been the major factor limiting the life of an implant, causing osteolysis and loosening of the joint,5,10 requiring revision surgery. Much attention has been on the tribological behavior of UHMWPE in an effort to reduce its wear. Many studies conducted have focused on altering the physical and chemical properties of UHMWPE to increase wear resistance, such as gamma-irradiated crosslinking11 and orientating crystalline12 structure, which have correlated with decreases in wear. Others have focused on the environment of the joint interface in vivo.

Healthy, human joints are lubricated by synovial fluid and can achieve remarkably low friction behavior, with coefficients as small as 0.002–0.006.13,14 Synovial fluid is a complex plasma solution mostly of proteins, hyaluronic acid (HA), and lipids and is sealed by the joint capsule, comprising of a membrane-lined fibrous layer of connective tissue.15 With the growing industry for total joint replacements, research to establish which constituent of the synovial fluid is responsible for the highest lubricating contribution for the materials involved (UHMWPE/ceramic) has received growing attention. Understanding this contribution of the fluid becomes important because it might be entirely different from the effect that synovial fluid would have on natural cartilages. Sawae et al.4 compared friction of UHMWPE in the presence of albumin (major protein component of synovia) versus HA and found albumin caused higher friction than HA. Gispert et al.16 also compared friction between albumin and HA, as well as a solution of albumin and HA combined. There were no consistent results as to the individual contributions to friction and lubrication, but it was clear that the combination of albumin and HA in a lubricating solution caused the lowest friction response and wear rate.

Surface active phospholipids (SAPL) have been reported to act as a boundary lubricant in human joints13,17,18 by adsorbing phosphate head groups onto the surface of the
joint, and leaving fatty acid tails to create a hydrophobic monolayer. It has been shown that in osteoarthritic joints, the synovial fluid does not lubricate as well as in healthy joints and this is often attributed to a deficiency of SAPL. It is hypothesized that the presence of phospholipids between articulating surfaces would also reduce wear by reducing interfacial contact, though further studies are needed to confirm this theory. Investigations on the tribological influence of phospholipids on UHMWPE have correlated decreasing friction and wear in the presence of phospholipids. The most abundant lipid in synovial fluid is dipalmitoyl phosphatidylcholine (DPPC), making up 45% of the total volume weight of lipids, and is one of the most common phospholipids studied on UHMWPE. DPPC dissolved in propylene glycol and ethanol on UHMWPE have shown lower friction values when compared with control lubricant without DPPC. Mazzucco et al. reported a lubricant of DPPC in phosphate buffered solution (PBS) to have the lowest coefficient of friction between polyethylene and cobalt chrome surfaces, when compared with HA and protein dissolved in PBS.

SAPL have an affinity for hydrophilic surfaces because of their ionic phosphate heads. UHMWPE is considered a hydrophobic polymer (contact angle ~80–100 degrees), which has low wettability and the potential to resist adsorption of the lipid. Gispert et al. showed a marked change in contact angle and surface tension on UHMWPE using separate solutions of saline with HA and bovine serum albumin (BSA) compared with saline alone. The presence of HA in saline increased the contact angle (hydrophobic) whereas BSA decreased the angle making it more hydrophilic. The effect of crystalline morphology at the surface on the water contact angle of polypropylene was examined by Yui et al. and found to correlate well: higher crystallinity at the surface gave lower contact angles, confirming that crystalline regions of polypropylene contain more surface energy than amorphous regions. Molecular simulations have shown similar trend in polyethylene. One potential for increasing adsorption of SAPL onto UHMWPE is to increase the crystallinity of the material. The authors have studies the effect of crystallinity on the friction and wear behavior of UHMWPE in dry conditions and found that increased crystallinity resulted in lower friction and wear.

In the present study, friction and wear behavior of UHMWPE as a function of crystallinity in the presence of a DPPC-ethanol lubricant was investigated. Two UHMWPE samples with different crystallinities were made using specific thermal treatments. Friction and wear tests were done with a custom-built ball-on-flat microtribometer under separate lubricant of a pure ethanol control and a solution of DPPC phospholipids dissolved in ethanol.

**EXPERIMENTAL DETAILS**

**Materials**

Commercially available, ram extruded GUR 1050, (available in the form of rod-stock) medical grade UHMWPE (Poly Hi Solidur, Fort Wayne, Indiana) was cut into two 30-mm square pieces, 2-mm thick. This material was not treated to any irradiation and they are not crosslinked. Samples were soaked in methanol and held in an ultrasonic bath for over an hour to remove any residue associated with the cutting process and were then air dried with nitrogen. Differential scanning calorimetry (DSC) gave a peak melt transition temperature of 140°C for the prepared sample. Two samples with significant difference in crystallinity were desired. Separately, samples were heated in a vacuum oven to 200°C. To create a high crystallinity polymer, the first sample was held at this temperature for 5 h to ensure thorough melting; the oven temperature was then reduced to 110°C, the recrystallization temperature, where the sample remained for 60 h to allow time for lamellar folding into a crystalline state. The second sample was held for 5 h at 200°C, to give sufficient time for thorough melting and disordering of the chains. Next, the sample was rapidly cooled by a liquid nitrogen quench to freeze the mobility of polymer chains in the amorphous region and minimize recrystallization. Both samples were clamped with low pressure against a clean quartz plate while heating to impart low, consistent surface roughness. All UHMWPE samples were rinsed with methanol and air dried with nitrogen before further treatment or testing.

**Differential Scanning Calorimetry**

A Perkin-Elmer DSC (Pyris 1) was used to characterize the melting point and percentage crystallinity of the polymer samples after thermal treatment. Ten milligrams of the sample was used for all DSC runs. The samples underwent a heating cycle from 25 to 200°C at a rate of 10°C/min and also a cooling cycle from 200 to 25°C at a rate of 10°C/min. The melting point was calculated from the crossover point of the tangents drawn to the horizontal and vertical portion of the melting peak. The percentage of crystallinity was calculated from the ratio of the area under the melting peak (using Pyris software) to the enthalpy of melting of a 100% crystalline sample (291 J/g).

**Phospholipids**

The phospholipids used in this study were synthetic, 99% pure L-α-Phosphatidylcholine, dipalmitoyl (DPPC), P0763 by Sigma-Aldrich Chemical Company. The DPPC was dissolved into ethyl alcohol at a concentration of 0.5 mg/mL, corresponding to the approximate concentration of DPPC found in human synovial fluid. Sodium azide was added to prevent microbial growth. The liquid was then vortex mixed for 3–4 min and ultrasonicated for 30 min at a temperature of 43°C to create a homogeneous solution. The DPPC-ethanol solution was used as a lubricant in friction and wear tests of the polymer samples; before lubricated tests, samples were covered and soaked in the solution for ~3 h before testing. Polymer samples were also tested in pure ethanol which served as the control sample for the present study. Throughout testing, surfaces were submerged in the solution.
Microscale friction was obtained using a custom-built reciprocating microtribometer in a ball-and-flat configuration. Four tests were conducted on each sample using a smooth, spherical Si$_3$N$_4$ probe (radius ~1.2 mm and RMS roughness of 4.3 ± 0.5 nm on a 20 × 20 μm scale) over a single stroke (length 15 mm) at a rate of 1 mm per second with a linear increase in the normal load (0–200 mN). The counterface roughness of the probe is comparable to that reported in retrieved joint surfaces. Reciprocating wear tests were completed on each sample using the microtribometer and a smooth, spherical Si$_3$N$_4$ probe (radius ~1.2 mm). A 140-mN constant normal load was applied to the samples for 1000 cycles of 15 mm stroke lengths at a speed of 5 mm per second. Dry-sliding friction and wear tests were complete in dry conditions, (<15 % humidity). Three sets of lubricated friction and wear tests were done at 32°C using a stage heater to approximate fluid temperatures in a joint. A profilometer was used to measure depth and width of the tracks from all wear tests. Wear depths reported are averages of six measurements on each wear track and across the three tests for a given sample.

Atomic Force Microscopy
An atomic force microscopy (AFM) was used to measure surface roughness of the polymer samples. Experiments in contact mode were carried out with a Dimension™ 3100 AFM (Nanoscope IV, Veeco Instruments, Santa Barbara, CA) in controlled low humidity (6 ± 2% relative humidity) conditions to minimize effects of adsorbed water vapor. Standard V-shaped silicon nitride probes from Veeco with a quoted normal spring constant of 0.58 N/m and tip radius of 10–40 nm were used. The normal spring constant of the cantilever used was calibrated to have an actual value of 10–40 nm. The normal spring constant of a smooth, spherical Si$_3$N$_4$ probe (radius ~1.2 mm) was 0.58 N/m and tip radius of 7.27 nm on a 20 × 20 μm scale. The counterface roughness of the probe is comparable to that reported in retrieved joint surfaces. Reciprocating wear tests were completed on each sample using the microtribometer and a smooth, spherical Si$_3$N$_4$ probe (radius ~1.2 mm). A 140-mN constant normal load was applied to the samples for 1000 cycles of 15 mm stroke lengths at a speed of 5 mm per second. Dry-sliding friction and wear tests were complete in dry conditions, (<15 % humidity). Three sets of lubricated friction and wear tests were done at 32°C using a stage heater to approximate fluid temperatures in a joint. A profilometer was used to measure depth and width of the tracks from all wear tests. Wear depths reported are averages of six measurements on each wear track and across the three tests for a given sample.

Scanning Electron Microscopy
A JEOL JSM-606LV scanning electron microscope was used to image the microtribometer wear tracks from both the reciprocating wear and the scratch tests. The sample chamber was low-vacuum and accelerating voltages of 1–2 kV at ranges of 10–200× magnification were used. These images were used to measure wear widths and scratch lengths. Scanning electron microscopy (SEM) was also used to image the Si$_3$N$_4$ probes to confirm a spherical shape (Figure 1) and to check for polymer film transfer after reciprocating wear tests. A conductive, gold layer (~200 Å thick) was sputter coated onto the probes before imaging. Low accelerating voltage, 1–2 kV, was used.

Optical Microscopy
An Olympus BX51WI (Leeds Precision Instruments) with Twin Epi-Fluorescence Optical Pathway (Prairie Technologies) microscope used to image the wear tracks and probes.

Contact Angle Measurements
Hydrophobicity of the UHMWPE before and after exposure to DPPC was evaluated using contact angle measurements. Contact angles were measured using analysis of high magnification digital images from a CCD camera of 10 μL water droplets on each sample before and after soaking in the phospholipids solution. The images were imported into an image analysis software (Scion Image) and the contact angles were measured. Reported values are averages of three separate measurements on a given sample.

RESULTS
DSC showed that the two heat treated samples of UHMWPE had different crystallinities. Melting temperatures peaked at 134.5°C for the nitrogen quenched sample and 139.5°C for the slowly cooled sample. The peak melt transition temperature generally defines the point at which the largest lamellae of a polymer completely melt, and is characteristic of the degree of crystallinity in the bulk polymer. The crystallinity percentages of the samples were calculated from the heat of enthalpy measured normalized by that of a 100% crystalline sample, 291 J/g. Heat of enthalpy for the quenched sample was 129.7 and 162.5 J/g for the slowly cooled sample, giving crystallinities of 44.6 and 55.8%, respectively. As a comparison, conventional UHMWPE has a crystallinity of about 50%. Crystallinity measurements were performed on top surface layer (<0.5 mm), intermediate layer and the core of the sample. They showed no significant variation in the crystallinity values (Table I).

Topography maps of the two polymer samples taken by the AFM after heat treatment showed comparable surface roughness on both samples. HC-PE gave an RMS roughness of 7.27 ± 0.96 nm and LC-PE gave an RMS roughness of 5.70 ± 0.93 nm for 5 × 5 μm scans. The sample with higher crystallinity (HC-PE) displayed lamellar structure when examined with AFM (Figure 1), which is typical of recrystallization. Lamellae were not as evident in the lower crystallinity (LC-PE) sample. It is noted that friction and wear studies in general are very dependent on the materials used and their roughness values and comparisons of studies should take these into account. Roughness values after the polymer samples were exposed to the lipid solution were measured at 11.03 ± 3.33 nm and 5.81 ± 0.76 nm for the HC-PE and LC-PE samples respectively. Roughness increased 50% more for the HC-PE sample when compared with the LC-PE.

Figure 2 shows results of the contact angle measurements performed on the polymer surfaces before and after exposure to DPPC. Before exposure to phospholipids, LC-PE gave a contact angle of 88.2° and HC-PE was 59.4°. After being soaked in the DPPC solution, the contact angle of LC-PE decreased considerably to 45.4° and HC-PE increased to 76.8°.

Representative ramped-load friction responses of Si$_3$N$_4$ on UHMWPE are shown in Figure 3. The friction response of Si$_3$N$_4$ on LC-PE in both control and DPPC lubricated conditions was slightly higher than that of HC-PE. The presence
of DPPC corresponded with a 17% reduction in friction coefficient of HC-PE and a 10% increase in friction coefficient of LC-PE for ramped-load friction experiments. Friction response of reciprocating wear tests (Figure 4) after 1000 cycles (a sliding distance of 30 m) in DPPC dissolved in ethanol showed a 17% reduction in friction for HC-PE and a 22% increase for LC-PE when compared with control tests.

DISCUSSION

AFM topography images after exposure to lipids showed the presence of particulate-like structures on both samples (Figure 1). The HC-PE sample had a considerably higher density of these structures on the surface. Because both samples had been rinsed and nitrogen dried after being submerged in the DPPC-ethanol solution for friction and wear testing, it can be expected that these structures repre-

![High Crystallinity](image1)

![Low Crystallinity](image2)

FIGURE 1. Topography maps of high crystallinity and low crystallinity samples from a 5 x 5 μm scan obtained using an atomic force microscope (a) before and (b) after exposure to lipids.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Degree of Crystallinity (%)&lt;sup&gt;a&lt;/sup&gt;</th>
<th>RMS Surface Roughness (nm)&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Contact Angle (degrees)&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry HC-PE</td>
<td>55.8 ± 0.73</td>
<td>7.27 ± 0.96</td>
<td>59.4 ± 1.9</td>
</tr>
<tr>
<td>Dry LC-PE</td>
<td>44.5 ± 0.58</td>
<td>5.70 ± 0.93</td>
<td>88.2 ± 3.4</td>
</tr>
<tr>
<td>HC-PE with DPPC</td>
<td>---</td>
<td>11.03 ± 3.3</td>
<td>76.8 ± 2.8</td>
</tr>
<tr>
<td>LC-PE with DPPC</td>
<td>---</td>
<td>5.81 ± 0.76</td>
<td>45.4 ± 2.1</td>
</tr>
</tbody>
</table>

<sup>a</sup> Crystallinity percentage was calculated from enthalpy values taken from DSC normalized by 100% crystalline sample.

<sup>b</sup> Surface roughness measurements were taken on a 5 x 5 μm scan using AFM.

<sup>c</sup> Contact angle was measured using magnified digital images in Adobe Photoshop.
sent surface-adsorbed entities. Rahamim et al.\textsuperscript{30} identified phospholipids on the surface of a temporomandibular joint using electron microscopy. They found the phospholipids to be spherical, granular bodies ranging from 170 to 280 nm in diameter, and homogeneously distributed on the surface.\textsuperscript{30} Our AFM topography images showed that the adsorbed structures on the surfaces were similar in size and shape. Given that the samples were only exposed to the phospholipid solution, rinsed and nitrogen dried, it can be reasonably assumed that the particles represent DPPC adsorption to UHMWPE. The images illustrate a strong presence of globules scattered throughout the HC-PE sample, but are barely noticeable on the LC-PE sample. This indicates that the HC-PE sample adsorbed significantly more DPPC than the LC-PE sample.

As mentioned earlier, surface tension is one factor that determines wettability of a surface, and can correlate with adsorption onto a surface.\textsuperscript{31–33} Yui et al.\textsuperscript{22} compared contact angle measurements on crystalline versus amorphous regions of polypropylene and showed that higher crystalline structure correlates with lower contact angle and further, increased surface energy of a polymer. In our case, contact angle measurements were taken from magnified digital images of droplets on the surfaces. Contact angle measurements on dry UHMWPE samples before lipid testing indicated that HC-PE had higher surface energy and would be more likely to adsorb the DPPC. Hills and Crawford\textsuperscript{14}
showed that on healthy, bovine cartilage saline beads up, but on osteoarthritic cartilage deficient in SAPL, it will wet the surface. His argument was that as the polar phosphate heads adsorbed to a hydrophilic surface, its fatty acid-chained tails create a hydrophobic monolayer surface.34 The hydrophobicity of the new layer is dictated by the degree of adsorption onto the adsorbate, or its surface coverage.32,34 Contact angle measurements of the samples taken after exposure to the DPPC solution indicated that HC-PE became more hydrophobic, whereas LC-PE became more hydrophilic, agreeing with our contention of higher phospholipids adsorption onto the HC-PE sample mentioned earlier in the paper.

Wear behavior was also affected by the addition of DPPC into the tribological environment. Based on profilometer depth measurements, the HC-PE sample showed higher wear resistance than LC-PE in the control and DPPC-lubricated reciprocating tests (Table II). The increased wear resistance of HC-PE compared with LC-PE in the control can be attributed to the increased hardness associated with increased crystallinity in UHMWPE.24 The presence of DPPC in the lubricant corresponded with decrease in wear depth only for the higher crystallinity sample. After wear tests were completed, optical and SEM images of the probes were analyzed to check for film transfer. Film transfer did not occur for HC-PE in the ethanol control [Figure 5(a)]. Film

<table>
<thead>
<tr>
<th>Sample</th>
<th>Coefficient of Friction(^a)</th>
<th>Wear Depth(^b) (nm)</th>
<th>Film Transfer (^c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HC-PE with control</td>
<td>0.4348 ± 0.0012</td>
<td>165.9 ± 13.2</td>
<td>None</td>
</tr>
<tr>
<td>LC-PE with control</td>
<td>0.497 ± 0.0063</td>
<td>174.7 ± 13.8</td>
<td>None(^d)</td>
</tr>
<tr>
<td>HC-PE with DPPC</td>
<td>0.3624 ± 0.012</td>
<td>100.6 ± 32.1</td>
<td>Yes</td>
</tr>
<tr>
<td>LC-PE with DPPC</td>
<td>0.5478 ± 0.059</td>
<td>193.0 ± 38.7</td>
<td>Traces</td>
</tr>
</tbody>
</table>

\(^a\) Calculated from the friction response of a Si\(_3\)N\(_4\) probe on the microtribometer for ramped load friction tests of 0–200 mN.

\(^b\) Measured using a profilometer on wear tracks from a Si\(_3\)N\(_4\) probe on the microtribometer for 1000 reciprocating, 20 mm cycles at an applied load of 125 mN.

\(^c\) Observed on Si\(_3\)N\(_4\) counterface using scanning electron microscopy.

\(^d\) Some evidence of polymeric particles was observed.

FIGURE 5. SEM images of the polymer contact regions on probes after reciprocating wear tests on high and low crystallinity UHMWPE in the (a) ethanol control (b) and DPPC-ethanol solution.
transfer appeared in the case of HC-PE lubricated with DPPC in ethanol [Figure 5(b)], indicating an adhesive wear mechanism. Optical images of the probe used in LC-PE tests on ethanol showed evidence of particles in the contact vicinity. SEM images of the probe used in phospholipid lubricant tests on LC-PE [Figure 5(b)] showed a slight presence of material on the surface of the probe which could not be seen with the optical microscope, therefore, it can be said that film transfer was negligible.

Friction results correspond well with trends found in literature that shows SAPL such as DPPC to reduce friction behavior.\textsuperscript{13,17,21,35–37} Ozturk et al.\textsuperscript{17} showed a greater reduction in friction coefficient with an increase in DPPC concentration, and suggested that mixed or elastohydrodynamic lubrication occurred. Friction tests show that articulating surfaces containing the phospholipid DPPC act as a releasing agent, much like Teflon.\textsuperscript{6,14} Hills and Thomas\textsuperscript{35} further explain that the nature of surfactants cause nonpolar moieties to point outward, imparting hydrophobic surfaces less conducive to adhesion. DPPC also acts as an effective boundary lubricant because of its ideal structure for monolayer cohesion,\textsuperscript{35} a boundary lubricant is most successful when there is a strong cohesion within the bilayer, preventing asperity penetration from the counterface.\textsuperscript{13} Bell et al.\textsuperscript{38} speculated that a bilayer created from SAPL acted as a boundary lubricant, illustrating that reduction of wear increased with SAPL concentration. Saikko and Ahrroos\textsuperscript{39} also showed that DPPC as a boundary lubricant was highly effective in reducing wear volume.

The current study had results consistent with earlier findings that wear volume is reduced in the presence of phospholipids, particularly for the HC-PE sample. Based on the DPPC adsorption coverage seen on the sample, the lowered friction and increased wear resistance, it is possible that DPPC operated as an effective boundary lubricant for the higher crystallinity sample. This is perhaps attributed to the higher adsorption of the DPPC onto the higher crystallinity sample. The same cannot necessarily be concluded for the low crystallinity sample which showed only a small reduction in friction and no increase in wear resistance, likely a result of the poor adsorption as evidenced in Figure 1. Saikko and Ahrroos\textsuperscript{39} went on to note that the phospholipids did not prevent film transfer, which can be characteristic of adhesive wear, and they concluded that phospholipids are effective in reducing wear particle generation, but not transfer of the polymer. Because we did observe a transfer film in the case of HC-PE in the presence of DPPC, our results seem to agree with this observation.

CONCLUSIONS

The effect of polymer crystallinity and adsorption of phospholipid DPPC on the resulting friction and (short term) wear behavior of UHMWPE was determined. Higher crystallinity leads to increased wear resistance (in the absence of DPPC) as compared with the low crystallinity sample because of better surface mechanical properties. The difference in friction response however was very little. Contact angles measurements and topography maps of the two UHMWPE samples confirmed that higher crystallinity UHMWPE is more likely to adsorb SAPL than low crystallinity UHMWPE. The higher crystallinity sample showed significant improvement in friction and wear resistance in the presence of DPPC. On the other hand, the low crystallinity sample showed increased friction and decreased wear resistance. The increased adsorption of DPPC in the higher crystallinity sample promotes its boundary lubricative capabilities. Although long term tests need to be conducted, the results of this study suggest that, increasing UHMWPE crystallization can serve as an effective mode of escalating adsorption of SAPL to UHMWPE, which in turn can lead to increased wear resistance in joint replacement applications.

REFERENCES