# Compression-Molded, Lubricant-Treated UHMWPE Composites

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Received 9 August 2006; accepted 14 November 2006 DOI 10.1002/app.25823 Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Lubricant-treated ultra high molecular weight polyethylene (UHMWPE) composites were prepared by compression molding. Composites were made from mixtures containing up to 5.0 wt % of lubricant. Two solid lubricants, molybdenum disulfide (MoS<sub>2</sub>), and carbon black (CB), and one liquid lubricant, perfluoropolyether (PFPE), were used in the study. UHMWPE and the lubricants formed 3D networks, where the lubricant was evenly spread over the UHMWPE particles. The amounts of MoS<sub>2</sub> and CB were determined by thermogravi-

metric analyses, and the amounts of PFPE by ATR-IR spectroscopy. All the lubricant treated composites showed better friction properties than pure UHMWPE. The addition of PFPE to UHMWPE improved the hydrophobicity of the surface, whereas the addition of solid lubricant had little effect. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 1762–1768, 2007

Key words: polyethylene (PE); compression molding; additive; friction; wear

# INTRODUCTION

Ultra high molecular weight polyethylene (UHMWPE) is a material with unique self-lubricating and wearresistant characteristics. UHMWPE is used in applications where hydrophobicity, low friction, and high wear-resistance are required. Typical applications are liners and slide plates used in bulk materials handling, artificial joints, and ski bases.<sup>1-6</sup> Several methods have been investigated in an attempt to improve the tribological properties of UHMWPE. In the case of artificial joints, the wear resistivity of UHMWPE is typically improved by crosslinking by radiation or peroxide treatment.<sup>7,8</sup> Amorphous diamond coating has also been used in artificial joints.<sup>3</sup> More generally liquid crystalline polymer,<sup>9</sup> wollastonite,<sup>10,11</sup> carbon nanotubes,<sup>12</sup> and carbon fibers<sup>13</sup> have been added to improve the tribological and mechanical properties of UHMWPE. In addition to tribological improvement, carbon black (CB) has provided decreased electrical resistivity.<sup>14-16</sup> For this purpose as well as to increase the thermal conductivity, CB has been used as an additive in ski bases.<sup>5,6</sup> The tribological characteristics of molybdenum disulfide (MoS<sub>2</sub>) filled UHMWPE have also been investigated and the wear rate has been found to decrease with larger filler content.<sup>17</sup>

Journal of Applied Polymer Science, Vol. 104, 1762–1768 (2007) © 2007 Wiley Periodicals, Inc.



Molybdenum disulfide, graphite, and CB are common solid lubricants, typically used in severe conditions, such as encountered in the automotive field, the aviation industry, and metalworking. Solid lubricants may be used in free powder form, in dispersions, in greases, or as sputtered films on metal surfaces. MoS<sub>2</sub> and CB have similar hexagonal layered crystal structures. The good lubrication properties of MoS<sub>2</sub> and CB are due to the relatively easy movement of the layers against one another. Wear resistance, in turn, is enhanced by the strong interatomic bonding and packing in each layer. MoS<sub>2</sub> and CB have also been used as lubricants in polymer composites with polytetrafluoroethylene (PTFE), typically in bearing applications.<sup>18,19</sup>

Fluorine containing compounds are other additives used to modify the physical properties of polyethylene. We recently demonstrated that the hydrophobicity of polyolefins can be permanently improved by melt blending with perfluoropolyethers (PFPE) and by surface structuring.<sup>20,21</sup>

The aims of the present work were to explore the possibility of preparing lubricant treated UHMWPE composites, to study the distribution of lubricants in UHMWPE, and to compare the effects of different lubricants. The modifications were designed to produce a material with permanently improved tribological and hydrophobicity properties, which would allow a broader use of UHMWPE. Modifications were done by compression molding: lubricants were premixed with UHMWPE powder and composites were compression molded in a barrel-shaped steel mold.

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### **EXPERIMENTAL**

#### Materials and methods

The ultra high molecular weight polyethylene (UHMWPE) was Hi-Zex Million 630M from Mitsui Chemicals (Tokyo, Japan). Molecular mass was  $6 \times 10^6$  g/mol and density 0.935 g/cm<sup>3</sup>. The perfluoropolyether (PFPE) was Fomblin<sup>®</sup> Y06 from Solvay Solexis SpA (Bollate, Italy). Fomblin<sup>®</sup> Y06, which is an unfunctionalized perfluoropolyether with molecular mass of 1800 amu and density of 1.88 g/cm<sup>3</sup>. The carbon black (CB) was Pintex XE 2 B (Degussa AG, Frankfurt, Germany) with surface area of 600 m<sup>2</sup>/g and pour density of 0.13 g/cm<sup>3</sup> from Degussa AG. The molybdenum disulfide (MoS<sub>2</sub>) was from Riedel-de Haën (Seelze, Germany) with a purity minimum of 99.5%.

Thermal analyses were carried out with a Mettler Toledo TGA/SDTA851<sup>e</sup>. Differential scanning calorimetry (DSC) was used to determine the melting point, heat of fusion, and crystallinity of the composites. Thermogravimetric analyses (TGA) were made to determine the amount of lubricants in composites. Both DSC and TGA measurements were done under nitrogen gas flow. The flow rate in DSC was 80 mL/ min, and in TGA 50 mL/min. DSC measurements were done in 40 µL Al cups. TGA measurements were done in 70  $\mu$ L Al<sub>2</sub>O<sub>3</sub> cups, which were annealed at 1000°C for 5–10 min before measurements. Heating programs were done with StarE. DSC measurements were carried out between 25 and 200°C with a heating rate of 10°C/min. The heating program for DSC measurements included two heating and cooling cycles, and the results were taken from the last cycle to eliminate the thermal history of the samples. TGA measurements were done between 25 and 600°C with the same heating rate ( $10^{\circ}C/min$ ). The degree of crystallinity  $X_c$  (%) was calculated according to the following equation

$$X_c(\%) = (\Delta H_f / \Delta H_f^0) \times 100\%$$
<sup>(1)</sup>

where  $\Delta H_f^0 = 293 \text{ J/g}^{22}$  is the heat of fusion for a totally crystalline polymer and  $\Delta H_f$  is the measured heat of fusion.

The distribution of lubricants was studied with an Olympus BX51 optical microscope. Slides 20-µm thick were cut with a Leica RM2165 rotary microtome and images were recorded from these slides with a magnification of  $100 \times$ . As well, the distribution was studied with a Hitachi S4800 FE-SEM equipped with upper and lower (semi-in-lens) secondary electron detectors. Samples were mounted onto a stub with copper adhesive tape. Accelerating voltages of 2-3 kV and a general working distance of 8 mm were applied.

Infrared spectroscopic (IR) measurements with a Nicolet Avatar 320 FTIR spectrometer and attenu-

ated total reflectance (ATR) accessory were carried out to examine the PFPE treated UHMWPE composites. In the ATR technique, the IR radiation penetrates a few micrometers into the sample,<sup>23,24</sup> giving information about the composition of the sample from a relatively thick layer.

Contact angle measurements were carried out with a KSV Cam 200 contact angle meter. Static contact angle measurements were made at room temperature with ion exchanged water. A drop of water (5  $\mu$ L) was placed on the sample and the sample was photographed once a second for 30 s. The contact angle was determined mathematically by fitting a Young–Laplace curve around the drop. Values obtained between 6 and 30 s were averaged to obtain the contact angle for each measurement. Six parallel measurements of each sample were made in this way and average contact angle values were calculated for each UHMWPE composite.

Rockwell R hardness measurements were carried out with an ALPHA Duromaster 2015. Measurements were done with a metal ball of diameter 12.70 mm, and the preload was 588.4 N. Six parallel measurements were made of each composite and the average hardness values were calculated for each sample. Since the hardness measuring device was not calibrated before the measurements, the absolute values of the hardness include some uncertainty, but the comparison of different compositions is still reliable.

The frictional properties of the UHMWPE composites were measured with Zwick Z2.5/TN1S materials testing equipment. Test pieces were pulled against a flat UHMWPE sheet for 100 mm at a constant speed of 200 mm/s, and the total load of the system was 483.2 g including the test piece and the carriage. The frictional force was detected between 20 and 100 mm and the average coefficient of friction for each composite was calculated from six parallel measurements.

An abrasion wear test was adapted from the ASTM G 65 standard.<sup>25</sup> The quartz sand used as abrasive was of particle size 0.1-0.6 mm and the flow rate was about 305 g/min. Normal load was 28 N, the rubber wheel had a diameter of 235 mm, and the rotation rate was 200 rpm. Test time was 10 min and four parallel measurements were made of each UHMWPE composite.

Tensile tests were carried out with material testing equipment ZWICK Z010/TH2A model 2001. Calculations were done with TestXpert version 8.1 software. Tensile modulus (Young's modulus) was determined at a strain value of 0.1% with crosshead speed of 5 mm/min. Tensile strength at yield and tensile strength at break were measured with crosshead speed of 50 mm/min. The tensile specimens were cut from compression molded composites with

TABLE I Composition of the UHMWPE Composites				
Composite	UHMWPE	CB	$MoS_2$	PFPE

1	UHMWPE	100.0			
2	5% CB	95.0	5.0		
3	3.3% CB	96.7	3.3		
4	5% MoS <sub>2</sub>	95.0		5.0	
5	3.3% MoS <sub>2</sub>	96.7		3.3	
6	5% PFPE	95.0			5.0
7	2% PFPE	98.0			2.0
8	1% PFPE	99.0			1.0
9	2.5% CB + 2% PFPE	95.5	2.5		2.0

The amounts of UHMWPE and lubricant are given in weight percentage (wt %).

width 3.0–6.0 mm, thickness 4.0 mm, and gauge length 20 mm. Six tensile specimens were tested for each composite.

#### **RESULTS AND DISCUSSION**

# Processing of UHMWPE composites

Before compression molding the materials were mixed and dried. Mixing was done with an IKA A 10 mill in the case of CB and MoS<sub>2</sub> composites and in a beaker with a glass stirrer in the case of PFPE. Mixed composite materials were dried in an oven at 85°C for at least 12 h before molding. Compression molding was done in a custom-made barrel-shaped steel mold with inner diameter of 124 mm and total height of 195 mm. Nine different composites were molded each with a mass of 150 g. The compositions of the UHMWPE composites are shown in Table I.

Processing parameters were selected according to the literature.<sup>1</sup> During the heating cycle the temperature was raised from 25 to 240°C in 210 min and kept constant at 240°C for an additional 60 min. The pressure varied between 35 and 60 bar during that time. The system was then cooled during 120 min, while the pressure varied between 60 and 100 bar.

Test samples were prepared from the molded disks (diameter 124 mm and height about 15 mm) by shaving off a layer  $\sim$  1.5-mm thick from either side and cutting out pieces of dimensions 20  $\times$  60 mm. Surfaces were polished with a Struers Labo-Pol 5 grinding machine by wet grinding with silicon carbide grinding paper (grit 320, 800, 1200, and 2000), and after rinsing of the surfaces with water, samples were dried in a vacuum exsiccator.

#### Characterization of UHMWPE composites

#### Thermal analyses

Results of the thermal analyses are shown in Table II. According to the DSC analyses, pure UHMWPE had a melting point of about 138°C and the compo-

sites had melting points 1–4 degrees lower. The heat of fusion of pure UHMWPE was about 147 J/g and the crystallinity about 50%. When CB was added to UHMWPE the heat of fusion decreased by 15 J/g (5.0 wt %) and 10 J/g (3.3 wt %), indicating up to 5% decrease in crystallinity. The addition of  $MoS_2$  or PFPE to UHMWPE did not have a notable influence on the heat of fusion. TG analyses of the composites treated with solid lubricant (CB or  $MoS_2$ ) showed a clear correlation between the amount of lubricant added to the composite and the residue after TGA. In case of PFPE, the residue was about the same level as for pure UHMWPE.

#### Microscopy

According to the optical microscopy study, the particle size of the UHMWPE powder was between 50 and 300 µm. Compression molded pure UHMWPE was homogeneous and free of defects. All of the lubricant treated UHMWPE composites looked similar: lubricant was evenly spread over the UHMWPE particles, forming "a 3D-network" with lubricant thickness between a few micrometers and a few tens of micrometers. The left column of Figure 1 shows the optical microscopy images recorded from UHMWPE powder and from 20-µm-thick microtome slides of UHMWPE composites. The SEM images (center and right columns of Fig. 1) confirm the findings from optical microscopy: the pure UHMWPE is homogeneous, and the solid lubricants coat the UHMWPE particles. The distribution of PFPE in the composite is not clearly seen in the SEM images.

#### Analysis by ATR-FTIR

ATR-FTIR measurements were used to determine the relative amount of PFPE in the UHMWPE composites. Figure 2(A) shows the average spectra of the composites. The spectra are scaled so that the intensity of the C-H bending band of the CH<sub>2</sub> groups at 1460 cm<sup>-1</sup> is the same in all spectra. Absorption

TABLE II
$T_{mr} \Delta H_{fr}$ and crystallinities ( $X_C$ ) of composites according
to DSC analyses, and R of the composites after TGA

	Composite	$T_m$ (°C)	$\Delta H_f (J/g)$	$X_c$ (%)	R (%)
1	UHMWPE	138	147	50	0.7
2	5% CB	137	132	45	4.7
3	3.3% CB	137	137	47	3.4
4	5% MoS <sub>2</sub>	135	150	51	5.1
5	3.3% MoS <sub>2</sub>	137	152	52	3.7
6	5% PFPE	136	150	51	0.3
7	2% PFPE	137	149	51	1.3
9	2.5% CB + 2% PFPE	134	145	49	4.2

 $T_{m}$ , melting temperatures;  $\Delta H_{f}$ , heats of fusion;  $X_{C}$ , crystallinities; R, residues.



**Figure 1** Images, from top to bottom, of UHMWPE powder, compression molded UHMWPE, 5.0%CB/UHMWPE composite, 5.0%MoS<sub>2</sub>/UHMWPE composite, 5.0%PFPE/UHMWPE composite, and 2.5%CB/2.0%PFPE/UHMWPE composite. In the optical microscopy images (left) the magnification is  $\times 100$ ; in the SEM images (middle and right) the magnification is  $\times 100$  and  $\times 500$ . [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



**Figure 2** (A) IR spectra of pure UHMWPE (green), 1.0%PFPE/UHMWPE (blue), 2.0%PFPE/UHMWPE (red), and 5.0%PFPE/UHMWPE (black). (B) Average peak area between 1285 and 1215 cm<sup>-1</sup> recorded from UHMWPE/PFPE composites, plotted together with earlier results for HDPE.<sup>20</sup> [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

from PFPE was integrated between 1285 and 1215  $\text{cm}^{-1}$  and the peak area was plotted together with the results for HDPE obtained in previous work,<sup>20</sup> as shown in Figure 2(B).

The results of the ATR-IR measurements confirm that UHMWPE forms composites with PFPE. The relative amount of PFPE in the bulk of the compression molded UHMWPE composites is larger than that on the surfaces of injection molded HDPE composites.

#### **Contact angle measurements**

Contact angle measurements provide an accurate method for determining the interaction energy between a liquid and a solid. The wettability of a solid surface strongly depends on both the surface energy and the surface roughness.<sup>26–32</sup> As can be seen from Figure 3, the contact angle between water and the compression molded polished UHMWPE is close to that of the injection molded HDPE. The results in Table III show that the addition of CB to



**Figure 3** Average contact angle as a function of amount of PFPE (wt %) added, plotted together with earlier results for HDPE.<sup>20</sup> [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

UHMWPE decreases the contact angle between the composite and water by a few degrees, whereas the addition of  $MoS_2$  increases it by a few degrees. The rougher surface structures of the  $MoS_2$  composites than the pure UHMWPE surface might explain the increase of the contact angle and higher standard deviation. The addition of PFPE increased the contact angle further, and it approached the value measured for the injection molded PFPE/HDPE composites (Fig. 3).<sup>20</sup>

# Tribological properties and hardness of UHMWPE composites

Table IV shows the results obtained from the measurements of Rockwell R hardness, the coefficient of friction, and abrasion wear. Lubricant treatment had little effect on the hardness of the composites. Regardless of the composition or the amount of lubricant, all composites exhibited 0–5% larger hardness values that the pure UHMWPE. A more positive

TABLE III Average Contact Angles (CA) with Standard Deviations For UHMWPE Composites

	Composite	CA
1	UHMWPE	101 ± 1
2	5% CB	97 ± 1
3	3.3% CB	98 ± 2
4	5% MoS <sub>2</sub>	107 ± 3
5	3.3% MoS <sub>2</sub>	113 ± 3
6	5% PFPE	112 ± 3
7	2% PFPE	109 ± 1
8	1% PFPE	$105 \pm 1$
9	2.5% CB + 2% PFPE	$104 \pm 1$

			1	
	Composite	Rockwell R	Coefficient of friction	Abrasion (mg)
L	UHMWPE	$77 \pm 2$	$0.39 \pm 0.01$	191 ± 1
2	5% CB	$80 \pm 3$	$0.37 \pm 0.01$	$188 \pm 9$
3	3.3% CB	$81 \pm 1$	$0.38 \pm 0.02$	$167 \pm 4$
ł	5% MoS <sub>2</sub>	$79 \pm 2$	$0.37 \pm 0.01$	$230 \pm 20$
5	3.3% MoS <sub>2</sub>	$78 \pm 2$	$0.36 \pm 0.01$	$237 \pm 7$
5	5% PFPE	$77 \pm 3$	$0.33 \pm 0.01$	$250 \pm 30$
7	2% PFPE	$80 \pm 1$	$0.36 \pm 0.01$	$220 \pm 20$
3	1% PFPE		$0.37 \pm 0.01$	
)	2.5% CB + 2% PFPE	$78 \pm 2$	$0.35 \pm 0.01$	$240~\pm~20$

TABLE IV Rockwell R Hardness, Coefficient of Friction, and Abrasion with Standard Deviations for UHMWPE Composites

effect was observed in the friction measurements. In comparison with pure UHMWPE all lubricants depressed the coefficient of friction between the composite and UHMWPE sheet. With CB the decrement was about 3% and with MoS<sub>2</sub> it was about 6% regardless of the amount of solid lubricant added. With PFPE there was a clear relationship between the decrease in the coefficient of friction and the amount of lubricant added to the composite. The coefficient of friction decreased by about 4% when 1% of PFPE was added, and by up to 15% when 5% of PFPE was added (composites 8, 7, and 6). The composite with CB and PFPE (composite 9) exhibited better frictional properties than the composites with only CB or PFPE (composites 3 and 7). Composites (2 and 3) containing CB showed better abrasion wear resistance than pure UHMWPE, whereas, composites containing MoS<sub>2</sub> or PFPE (4-9) were worn more than pure UHMWPE.

#### Tensile properties of the UHMWPE composites

Results of the tensile tests are compiled in Table V. Tensile properties of pure compression molded UHMWPE are similar to the results reported earlier.<sup>1,7–10,12,14,17,33</sup> In general it is difficult to improve the tensile properties of UHMWPE.<sup>1,7,8,10,14,17</sup> Some improvements have been achieved through the addition of liquid crystalline polymer<sup>9</sup> and carbon nanotubes.<sup>12</sup> CB treatment had little effect on the tensile

properties, in agreement with earlier results,<sup>14,17</sup> while treatment with PFPE or MoS<sub>2</sub> weakened the tensile properties as compared with pure UHMWPE.

#### CONCLUSIONS

UHMWPE was modified with two solid lubricants (MoS<sub>2</sub> and CB) and a liquid lubricant (PFPE), and composites were prepared by compression molding. UHMWPE and the lubricants formed 3D networks where the lubricant was evenly spread over the UHMWPE particles. The amount of solid lubricant was determined by TGA and the amount of PFPE by ATR-IR spectroscopy. All of the lubricant treated composites showed improved friction properties as compared with pure UHMWPE. CB improved the abrasion wear resistance, while tensile properties were about the same as with the pure UHMWPE. Both MoS<sub>2</sub> and PFPE weakened the tensile strength of pure UHMWPE. The addition of solid lubricant to UHMWPE had little effect on the hydrophobicity, while the addition of PFPE increased the contact angle between water and the surface by about 10%, to about the same level as for injection molded PFPE/HDPE composites. Figure 4 shows the results of friction force, contact angle, and ATR-IR measurements for pure UHMWPE and PFPE/UHMWPE composites. As can be seen, the friction properties and hydrophobicity improve when the relative amount of PFPE increases in the composites.

	TABLE V		
Tensile Properties with	Standard Deviations	for UHMWPE	Composites

	Composite	Young's modulus (GPa)	Tensile strength at yield (MPa)	Tensile strength at break (MPa)
1	UHMWPE	$1.7 \pm 0.1$	$13.0 \pm 0.2$	$36.2 \pm 2.3$
2	5% CB	$1.4 \pm 0.2$	$13.7 \pm 2.1$	$36.3 \pm 0.9$
3	3.3% CB	$1.5 \pm 0.4$	$12.3 \pm 1.5$	$36.8 \pm 1.4$
4	5% MoS <sub>2</sub>	$1.6 \pm 0.3$	$11.2 \pm 1.2$	$21.7 \pm 0.7$
5	3.3% MoS <sub>2</sub>	$1.4 \pm 0.2$	$12.4 \pm 1.6$	$21.9 \pm 0.4$
6	5% PFPE	$1.2 \pm 0.4$	$11.8 \pm 2.3$	$34.0 \pm 2.0$
7	2% PFPE	$1.4 \pm 0.3$	$10.7 \pm 1.4$	$34.5 \pm 2.6$
8	1% PFPE	$1.2 \pm 0.2$	$12.0 \pm 2.4$	$36.4 \pm 3.1$
9	2.5% CB + 2% PFPE	$1.2 \pm 0.2$	$12.7 \pm 2.6$	$26.9 \pm 1.6$



**Figure 4** Results for compression molded PFPE/ UHMWPE composites obtained by three analytical methods. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

The authors thank Dr. Sari Suvanto for her help in SEM imaging, Mr. Panu Karttunen for his help in the friction and hardness measurements, and Mr. Olli Ylöstalo for his help in abrasion wear measurements. Mitsui Chemicals and Solvay Solexis SpA are gratefully acknowledged for their donation of materials.

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