Tribochemistry of Monodispersed ZDOL Lubricants Measured by Optical Surface Analysis and Thermogravimetric Analysis

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Abstract

Tribo-chemical studies of the lubricant molecular weight effect on the tribology of the head/disk interface (HDI) were conducted using carbon disks coated with ZDOL lubricant. The studies involved drag tests with uncoated and carbon-coated Al₂O₃-TiC sliders in an ultra-high vacuum (UHV) tribochamber. The studies showed that the lubricant interaction with the carbon overcoat varies as a function of lubricant molecular weight. The friction coefficient increases as the molecular weight increases. The higher friction is due to the higher viscosity. Our studies support previous observations that catalytic reactions occurre at the endgroup functionals. The lower number of endgroup functionals for ZDOL with higher molecular weight reduces the possibility of the occurrence of catalytic reactions. The spreading diffusion coefficient of ZDOL decreases with increasing molecular weight as indicated by the optical surface analysis (OSA) data. Thermogravimetric analysis (TGA) experiments were also conducted to observe the desorption mechanisms of the PFPE lubricants.

Keywords: Tribochemistry; Hydrogenated carbon overcoat; ZDOL decomposition; Friction and wear; OSA; TGA; Molecular weight; Polydispersity; Spreading; Viscosity, Crosslinking.

I. Introduction

To reduce friction and wear magnetic recording thin-film disks have a hard overcoat lubricated with a thin layer of a liquid lubricant. Because of the superior properties, such as low vapor pressure, good viscosity, high thermal stability and chemical inertness, perflouropolyethers (PFPEs) have been extensively used as the lubricants of magnetic media in hard disk drives. The molecular structure, containing only carbon, fluorine and oxygen atoms, gives PFPEs this set of properties. The PFPE lubricant and the hard carbon overcoat on the disks provide the necessary protection of the underlying magnetic film against wear due to contact sliding at the head-disk interface. Their decomposition mechanisms associated with contact sliding have been extensively studied [1-5]. Our earlier studies of the decomposition of ZDOL lubricant on nitrogenated carbon overcoats (CNx) [6] indicate that the decomposition rate is significantly affected by the slider materials. The use of uncoated Al₂O₃-TiC sliders leads to the rapid decomposition of ZDOL due to catalytic reactions, while carbon coated sliders produce less intense decomposition which is due primarily to frictional actions [7]. The thermal stability of PFPE has also been studied extensively [8-11]. In our previous report [12], the friction and catalytic decomposition mechanisms as well as the thermal behavior of ZDOL are described, and data demonstrating the chemical reactions of the lubricant and carbon overcoat are also presented.

An effective lubricant should exhibit slow removal and loss rate, fast recovery, and minimum tribochemistry [13]. Lubricant is also lost by centrifugal flow, or spin-off, and evaporation [14]. Lower molecular weight increases the rate of recovery, and also the rate of loss through spin-off and evaporation. Hence, there is a tradeoff between lubricant loss and recovery

by spreading. Waltman and Tyndall [15] showed that the activation energy for lubricant evaporation from the carbon surface increases with increasing molecular weight. They also found that the low activation energies determined for the evaporation and bonding of ZDOL 2000 are attributed to the polydispersity in the ZDOL molecular weight [16]. In our previous work [17], it was shown that the lubricant interaction with the carbon overcoat varies as a function of lubricant thickness. Wear durability improves considerably for thicknesses greater than a monolayer. As the lubricant thickness increases, the end-group effect on the diffusion process decreases, and the diffusion coefficient increases. As a result, faster replenishment of the lubricant occurs on disks with thicker ZDOL on the protective carbon surface, enhancing the wear durability at the interface. These results reflect the importance of the mobile ZDOL layer in providing good wear durability.

In this report we study the ZDOL molecular weight effect on carbon films using an ultrahigh vacuum (UHV) tribochamber equipped with a mass spectrometer. The studies consist of drag tests in the UHV tribochamber. Two decomposition processes of ZDOL under sliding friction conditions are studied, one is with a carbon film coated slider, and another is with an uncoated Al₂O₃-TiC slider. We illustrate the lubricant molecular weight effect on the tribological performance. Optical surface analysis (OSA) measurements were also used to observe the spreading behavior of PFPE lubricants. The lubricant mobility decreased with increasing molecular weight. Finally, the thermogravimetric analysis (TGA) data shows that the transition temperature of monodispersed ZDOL increased with increasing molecular weight.

II. EXPERIMENTAL PROCEDURE AND SET-UP

2.1 UHV Drag Tests

The UHV tribochamber consists of a disk spindle, a slider actuator, a substrate heater, and a high-resolution quadrupole mass spectrometer (QMS) in a vacuum chamber with base pressure $< 2 \times 10^{-8}$ Torr, which was described in details in a previous paper [18]. The QMS provides *in-situ* detection of the gaseous products generated during drag tests and thermal desorption studies. The QMS can monitor simultaneously 15 different atomic mass units (AMUs) ranging from 1 to 500 along with friction from strain gauge transducers. It is noted that all AMUs from 1 to 192 of ZDOL decomposition products generated at the Al₂O₃/TiC slider/CHx disk interface were previously investigated under the same test conditions and only the most pertinent AMUs were monitored in this study.

Drag tests in the tribochamber were conducted as follows. Initially, the tribochamber was baked out at 150°C at high vacuum for 24 hours. The chamber was then backfilled with Argon gas as the disk and slider samples were mounted inside. Next, the chamber was pumped down to a base pressure of 2 x 10^{-8} Torr and the channels of the QMS were assigned to selected AMUs. Background intensities were recorded before the drag tests were initiated with the following parameters: 0.2 m/s drag speed, a load of 25 mN, and a sliding time of 20 minutes. The sliders were 30% (1.2mm by 1mm) negative-pressure Al₂O₃/TiC sliders with and without amorphous carbon films on the air bearing surfaces. The disks were lubricated with ZDOL by a dipping

process. The resulting thickness of ZDOL on the disks in this study was 24Å. The ZDOL lubricants of different molecular weight were obtained by fractionating the commercially available ZDOL. The lubricants were fractionated by solvent evaporation [19]. The table below lists the narrowly dispersed fractions of ZDOL used in this work.

Lubricant	Number-average molecular weight Mn (g/mole)	Polydispersity (M _w /M _n)
ZDOL-1	4200	1.15
ZDOL-2	7600	1.02
ZDOL-3	8500	1.07

The number-average molecular weight M_n is defined as $M_n = W/N$. Where

W = total sample weight = $\Sigma w_x = \Sigma n_x M_x$,

N = total number of moles in the sample (of all sizes) = Σn_{x_1}

 $w_x = total weight of x-mer,$

 M_x = molecular weight of x-mer,

 $n_x =$ number of moles of x-mer.

The weight-average molecular weight M_w is defined as $M_w = \Sigma w_x M_x / \Sigma w_x$. It may be shown that $M_w \ge M_n$ (since $w_x \ge n_x$) [19]. These two averages are equal only for a perfectly monodisperse (all molecules the same size) polymer. The ratio M_w/M_n is known as the polydisperse index, and is a measure of the breadth of the molecular weight distribution. Polydisperse values range from about 1.02 for carefully fractionated polymers to over 50 for some commercial polymers. In our study the polydispersities of these monodisperse ZDOL lubricants are between 1.02 and 1.15, which are much lower than those of non-fractionated ZDOL lubricants (polydispersity above 1.4).

2.2 Optical Surface Analysis (OSA)

The optical surface analysis (OSA) system was also used to observe the lube migration behavior. The OSA is an *in situ* device, which has approximately 5 micron resolution and 2 MHz of bandwidth [20]. This device can be used to measure less than one Angstrom of carbon wear or lubricant depletion/accumulation. The OSA can also measure index of refraction changes caused by lubricant degradation or lubricant migration. The OSA is essentially a quantitative robot reflectometer microscope for imaging and analyzing the surface of a thin film disk. It operates by illuminating the surface of a carbon-coated disk with a highly stabilized laser diode beam at a wavelength of 780nm. A wavelength of 780nm is chosen since carbon is reasonably absorptive at this wavelength. Also, off-the-shelf optical components are available at this wavelength. The OSA is designed so that it allows a choice of either P or S polarized light, and the scattered or specular component of light coming from the surface can be measured. More information about the principle of operation can be found Meeks's paper [20]. Samples with two lube molecular weights were tested in the OSA measurements: one is MW 4200 and the other is MW 7600.

2.3 Thermogravimetric Analysis (TGA)

The thermogravimetric analysis (TGA) is a sensitive measurement to observe the weight change of a sample as a function of temperature. Typical applications include the assessment of thermal stability and decomposition temperature in polymers [22-25]. The TGA experiments were carried out in a TGA model 2050 manufactured by TA Instruments Inc., which has a temperature range from liquid nitrogen temperatures to 650° C, and was operated at a mass resolution of 0.3 µg. A typical sample size is on the order of 8 mg. Evaporation rate data was collected during a linear temperature ramp-up, usually 10°C/min, under a dry nitrogen purge. The samples were contained in aluminum pans. Samples with lube molecular weights from 1500 to 7500 were tested in the TGA measurements.

III. Results and Discussion

3.1 Results from the UHV drag tests

In this section we present the results from the UHV drag tests. Figures 1 through 3 part (a) show the friction coefficient curves for the ZDOL lubricated CHx disks against an uncoated and a DLC coated Al₂O₃/TiC pico slider. Before starting the friction tests the background intensities were recorded for 80 seconds by the mass spectrometer. Figures 1 through 3 part (b) show the integrated mass spectra of the ZDOL fragments produced from the head-disk interface. The four primary peaks belong to mass fragments CFO (47), CF₂O (66), CF₃ (69), and C₂F₅ (119). In our previous studies [12] of CHx carbon overcoats paired with carbon-coated sliders, the primary mechanism of ZDOL decomposition was due to frictional effects, and it was characterized by the generation of CFO (47), and CF₂O (66). The absence of a carbon-coating led to a more complex catalytic decomposition mechanism of ZDOL , and it was characterized by the generation of CF₃ (119).

Figure 1(a) shows the friction coefficient curves for the 24Å monodispersed ZDOL MW4200 lubricated disk. For the 70Å DLC coated Al₂O₃/TiC slider, the friction coefficient started at 1.2 and increased steadily to 1.4 within 1990 drag cycles before wear occurred. For the uncoated Al_2O_3/TiC slider, the friction coefficient started at 1.3 and increased steadily to a peak value of 1.9 within 295 cycles, then dropped to 0.3 after reaching its peak value. A wear track was observed on the disk just after the friction coefficient dropped. The friction variation, as shown in figure 1(a), is most likely due to three body contact resulting from small wear particles that were generated at the head-disk interface. These results indicate that 24Å of monodispersed ZDOL MW4200 has good lubricating action against the DLC coated slider, but provides little protection against the uncoated slider. Figure 1(b) shows the integrated mass spectra of the major ZDOL decomposed fragments produced from the head-disk interface. This mass spectrum for the 70Å DLC coated Al₂O₃/TiC slider is similar to that of ZDOL vapor, as reported by Kasai et al. [5], where the primary decomposition peaks are at 47 (CFO) and 66 (CF₂O) due to frictional heating [12]. Because no contact with Lewis acid is possible in this case, the ZDOL decomposition is caused by friction/thermal actions only. For the uncoated Al₂O₃/TiC slider, the mass intensities of the four major ZDOL fragments are higher than those in the DLC coated slider case. The decomposition mechanisms of ZDOL in the case of the uncoated Al₂O₃/TiC slider are much more complicated than those with the DLC coated slider. For the uncoated slider, catalytic reactions control the ZDOL decomposition and the catalytic reactions lead to the generation of masses 69 (CF₃) and 119 (C₂F₅). Therefore, masses 47 (CFO) and 69 (CF₃) are the highest peaks in the uncoated slider case as shown in figure 1(b). The friction curve and mass spectra of higher molecular weight are similar to Fig. 1 and are shown in Figs. 2 & 3.

Figure 4 shows the degradation intensities of (a) mass 47 (CFO), and (b) mass 69 (CF₃) during UHV drag tests on CHx disks with different lubricant molecular weights. The degradation intensities of the frictional fragments 47 (CFO) and 66 (CF₂O) decrease with both the uncoated sliders and DLC coated sliders as the molecular weight increases. Figure 4(b) shows a similar result for the catalytic fragments 69 (CF₃) and 119 (C₂F₅). However, the degradation intensities of the catalytic fragments decrease more rapidly than those of the frictional fragments as the molecular weight increases. These results indicate that less decomposition of ZDOL lubricants occurred with higher molecular weight.

Figure 5(a) shows the wear durability of these CHx disks as a function of ZDOL molecular weight. With DLC coated sliders, the wear durability was improved as lubricant molecular weight increased. This enhanced performance may be due to the slower lubricant decomposition rate. However, the wear durability, with uncoated sliders, was not monotonically improved with increasing molecular weight. More discussion will be presented on this point later. Figure 5(b) shows the friction coefficient of these CHx disks as a function of ZDOL molecular weight. The friction coefficient increased as the molecular weight increased. It has long been known that larger molecules will impede flow more than smaller ones and give a higher viscosity. Studies of the intrinsic viscosity of essentially monodisperse polymer fractions indicate a rather simple relation, known as Mark-Houwink-Sakurada (MHS) relation [26]:

 $[\eta]_x = K(M_x)^a \ , \qquad (0.5 < a < 1)$

where $[\eta]_x =$ viscosity of x-mer, while "K" and "a" are MHS constants.

The measured intrinsic viscosity of a mixture of monodisperse fractions is a weight average:

 $[\eta] = \Sigma[\eta]_x w_x / \Sigma w_x.$

The viscous component is dominant in liquids, hence their flow properties may be described by a Newtonian dashpot relation [31]:

 $\tau = \eta \, d\gamma/dt$

where τ is the applied shear stress, and $d\gamma/dt$ is the rate of strain

With higher molecular weight, the higher viscosity (η) will cause higher friction (τ) during the head-disk sliding.

Table 1 lists the normalized mass spectra of the major ZDOL decomposition fragments for DLC coated sliders and uncoated sliders with respect to different molecular weight. The normalized percentage of some fragments increased as molecular weight increased, but it decreased for some other fragments. The number of the endgroup functionals (-CF₂-CH₂-OH) dominates these phenomena. As the molecular weight increases, the number of the endgroup functionals decreases if the lubricant thickness is controlled. Therefore, the normalized degradation intensity of fragments 31 (CF), 50 (CF₂), and 51 (CF₂H) originating from the endgroup functionals decreases. These results indicate that these three decomposition fragments are mostly due to the lubricant depolymerization at the active endgroup functional, which is the second type of the chain scission. However, the normalized degradation intensity of mass 66 (CF_2O) increases as the molecular weight increases. This result indicates that the mass 66 (CF_2O) fragment is mostly from the lubricant decomposition within the main body that consists of two backbone groups 66 (CF_2O) and 116 (CF_2CF_2O) . In addition, this decomposition is due to the weak-link degradation at the lowest-energy bonds, which is the third type of the chain scission. The normalized degradation intensities of the catalytic reaction fragments 69 (CF_3) and 119 (C_2F_5) decrease as molecular weight increases. These results support our earlier observation that the catalytic reactions occur at the endgroup functionals, where the fluorine atom transfer is from the endgroup to the internal sector in the presence of the Al₂O₃ surface. The lower number of endgroup functionals with higher lubricant molecular weight reduces the possibility of the occurrence of the catalytic reactions.

3.2 Results from OSA measurements

Figure 6 shows the OSA data: a plot of lube migration as a function of molecular weight. Before the OSA measurements, lubricant was removed from a section of the disk by a solvent, HFE. This method was chosen because it was the least invasive and least likely to change the reflective properties of the carbon layer underneath the lubricant, which could affect the results. Figure 6(a) shows the lubricant thickness versus time. This lubricant thickness is defined as the difference between the region where the lubricant was removed and the untreated portion of the disk. The disk was scanned by S and P circularly polarized light every thirty minutes for thirteen hours. Figure 6(b) shows the average migration rate of ZDOL lubricant versus molecular weight. The rate is just the linear slope of the lubricant thickness versus time in Fig. 6(a). We observed that the lube mobility of ZDOL increased with higher molecular weight. From the data of the UHV drag tests, there was no such benefit in using higher molecular weight for the uncoated slider cases. Strong catalytic reactions occurred with the uncoated slider and these strong catalytic reactions speeded up the depletion of the lubricant layer. Hence, the reflow rate of mobile ZDOL in the wear track dominates the wear durability against uncoated slider. The wear durability of the ZDOL MW 4200 lubricated disk is better than those of the ZDOL MW 7600 or MW 8500 lubricated disks. The enhanced wear durability with lower molecular weight is due to its better mobility. The mobile ZDOL lubricants reflow in the wear track and improve their wear durability against uncoated sliders. For the DLC coated sliders, the depletion rate of the lubricant layer is much lower than that of the uncoated slider cases. The lubricant mobility is not as crucial as the lubricant-consuming rate. Hence, the lubricant decomposition rate dominates the wear durability against DLC coated sliders in this case.

3.3 Results from TGA measurements

Figure 7(a) shows the weight loss of ZDOL 2000 vs. temperature during the TGA experiments. The loss in weight was initially recorded in milligrams on the strip-chart recorder, and then converted to the fractional loss. To determine the transition temperature, a best-fit tangent line was drawn as indicated in Fig. 7(a), and the interception point between the tangent line and the curve represents the transition temperature. Figure 7 (b) shows the transition temperature of fractionated ZDOL vs. molecular weight. The transition temperature increased with increasing molecular weight. These results indicate that the desorption energy of lubricants increases with increasing molecular weight. It is known that the transition temperatures of polymers depend on three mechanisms: (1) the internal mobility of the chains; (2) the chain length; and (3) the cohesive energy.

From OSA data, we observed that the spreading rate of ZDOL lubricant decreases with increasing molecular weight. As the mobility (freedom to rotate about bonds) of the chains decreases the desorption energy needed to break the lubricants increases, resulting in higher transition temperatures.

The second mechanism can be demonstrated by the Gibbs free energy function as shown below:

 $\Delta G_{\rm m} = \Delta H_{\rm m} - T_{\rm m} \Delta S_{\rm m},$

where G_m is the Gibbs free energy of melting H_m is the energy needed to overcome the bonding forces S_m is the entropy or molecular order T_m is the melting temperature

At the equilibrium melting point T_m , $\Delta G_m = 0$, so $T_m = \Delta H_m / \Delta S_m$. For a given mass of polymer an increasing chain length results in more random order upon melting and a lower ΔS_m . Thus, the melting point (T_m) increases with increasing chain length (lower ΔS_m). The longer chain will also cause higher degrees of crosslinking. The crosslinking restricts the chain mobility and causes an increase in the transition temperatures.

The third mechanism is the cohesive energy. Cohesive energy is the total energy necessary to remove a molecule to a position far from its neighbors. With higher molecular weight, the total

cohesive energy per molecule becomes greater due to the higher intermolecular attractive forces (Van der Waals forces). With a longer chain, more ethers of the ZDOL lubricants are adsorbed to the carbon surface, resulting in higher cohesive energy.

IV. Conclusions

The experiments presented here illustrate the complex chemical relationships that occur at the head/disk interface during drag tests. The UHV tribochamber was used to monitor *in situ* the gaseous wear products generated at the HDI during dragging. The ZDOL lubricants of different molecular weight were obtained by fractionating commercially available ZDOL. The UHV drag tests show that less lubricant decomposition occurs when the molecular weight is higher. The corresponding wear durability of ZDOL against the DLC coated sliders improves with increasing molecular weight. This enhanced performance with heavier ZDOL may be due to the slower lubricant decomposition rate. However, there was no such benefit in using higher molecular weight for the uncoated slider cases. Strong catalytic reactions occurred with the uncoated slider and these strong catalytic reactions speeded up the depletion of the lubricant layer. Hence, the reflow rate of mobile ZDOL in the wear track dominates the wear durability against uncoated sliders. From the OSA data, we observed that the mobility of the PFPE lubricants decreased with increasing molecular weight. These results demonstrate the better wear performance of ZDOL with lower molecular weight in the uncoated slider case.

In addition, the friction coefficient increased as the molecular weight increased. The larger molecules impede flow more than smaller ones and give a higher viscosity. Hence, with higher molecular weight the higher viscosity causes higher friction during the drag tests. From the TGA data, we observed that the transition temperature increased with increasing molecular weight. The transition temperatures of polymers is believed to depend on three mechanisms: (1) the internal mobility of the chains; (2) the chain length; and (3) the cohesive energy.

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CAPTIONS

Figure 1: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW4200 lubricated disk; (b) mass spectrum of the major ZDOL decomposed fragments.

Figure 2: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW7600 lubricated disk; (b) mass spectrum of the major ZDOL decomposed fragments.

Figure 3: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW8500 lubricated disk; (b) mass spectrum of the major ZDOL decomposed fragments.

Figure 4: integrated degradation intensity of ZDOL during UHV drag tests on CHx disks with different ZDOL molecular weight. (a) mass 47 (CFO) under frictional action, and mass 69 (CF₃) under catalytic reaction.

Figure 5: (a) wear durability and (b) friction coefficient of CHx disks with different ZDOL molecular weight during UHV drag tests against coated or uncoated Al₂O₃/TiC sliders.

Figure 6: (a) the OSA data on the lubricant thickness versus time; (b) the ZDOL lube migration rate versus molecular weight.

Figure 7: (a) the TGA data on the lube weight loss versus temperature; (b) the TGA data on the transition temperature versus molecular weight.

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	31-	47-	50-	51-	66-	69-	119-
	CF	CFO	CF2	CF2H	CF2O	CF3	C2F5
ZDOL 4200/coated slider	24%	100%	9%	17%	38%	5%	12%
ZDOL 4200/uncoated slider	42%	100%	34%	38%	67%	89%	58%
ZDOL 7600/coated slider	23%	100%	4%	10%	43%	4%	14%
ZDOL 7600/uncoated slider	22%	100%	14%	12%	74%	73%	42%
ZDOL 8500/coated slider	13%	100%	4%	11%	53%	5%	14%
ZDOL 8500/uncoated slider	19%	100%	3%	5%	78%	59%	37%

Table 1: Normalized mass spectra of the major ZDOL decomposition fragments during the UHV

drag tests with respect to different ZDOL molecular weight.





Figure 1: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW4200 lubricated disk; (b) mass spectrum of the major ZDOL decomposition fragments.





Figure 2: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW7600 lubricated disk; (b) mass spectrum of the major ZDOL decomposition fragments.





Figure 3: (a) friction coefficient of UHV drag test on 24Å mono-dispersed ZDOL MW8500 lubricated disk; (b) mass spectrum of the major ZDOL decomposition fragments.





Figure 4: integrated degradation intensity of ZDOL during UHV drag tests on CHx disks with different ZDOL molecular weight. (a) mass 47 (CFO) under frictional action, and (b) mass 69 (CF3) under catalytic reaction.





Figure 5: (a) wear durability and (b) friction coefficient of CHx disks with different ZDOL molecular weight during UHV drag tests with DLC coated or uncoated Al_2O_3/TiC sliders.





Figure 6: (a) the OSA data on the lubricant thickness versus time; (b) the ZDOL lube migration rate versus molecular weight.



Figure 7: (a) the TGA data on the lube weight loss versus temperature; (b) the TGA data on the transition temperature versus molecular weight.