Effect of Lubricant Bonding on the Tribological Performance of ZDOL on Hydrogenated Overcoats

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Abstract

Tribochemical studies of the effect of lubricant bonding on the tribology of the head/disk interface (HDI) were conducted using hydrogenated (CH_x) carbon disk samples coated with perfluoropolyether ZDOL lubricant. The studies involved drag tests with uncoated and carbon-coated Al_2O_3-TiC sliders and also thermal desorption experiments in an ultra-high vacuum (UHV) tribochamber. We observed that a larger mobile lubricant portion significantly enhances the wear durability of the (head/disk interface) HDI by providing a reservoir to constantly replenish the lubricant displaced in the wear track during drag tests. In the thermal desorption tests we observed two distinct temperatures of desorption. The mobile ZDOL layer is desorbed at the lower thermal desorption temperature and the residual bonded ZDOL layer is desorbed at the higher thermal desorption temperature.

Keywords: Tribochemistry; Hydrogenated carbon overcoat; ZDOL decomposition; Friction and wear; Thermal desorption; Bonded fraction; Mobility

I.   INTRODUCTION

Perfluoropolyethers (PFPE) are employed as lubricants at the head-disk interface in magnetic recording disk drives to reduce friction and wear. 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. The lubricant decomposition mechanisms associated with contact sliding have been extensively studied [1~5]. Our earlier studies of the decomposition of the ZDOL lubricant [6, 7] indicate that the decomposition rate is significantly affected by the slider material. The use of uncoated Al_2O_3-TiC sliders leads to the rapid decomposition of ZDOL due to catalytic reactions, while carbon-coated sliders produce less intense decomposition due primarily to frictional action [8]. The thermal stability of PFPE has also been studied extensively [9-12]. In our previous report [13], the friction and catalytic decomposition mechanisms as well as the thermal behavior of ZDOL were described, and data demonstrating the chemical reactions of the lubricant and carbon overcoat were also presented.

The thickness of the PFPE lubricant layer must be carefully chosen to provide maximum wear protection over the lifetime of the drive. If the lubricant film is too thick, excessive stiction is observed upon start up of the drive. On the other hand, if the lubricant film is too thin, insufficient protection of the head-disk interface is provided, and tribological failure can occur in the early operating period. The trend in the industry in recent years has been to decrease the amount of lubricant used. Lubricant thicknesses of a single layer or less (sub-monolayer) are becoming quite common in disk drives. Therefore, the interactions between the lubricant and the

carbon surface may be more important to the tribological performance of the disk drive than the bulk properties of PFPE lubricants.

Tyndall et al. [14] used surface energy measurements to extract information about the PFPE lubricant-carbon interfacial interactions. They measured the dispersive surface energy to determine the lubricant coverage of the carbon surface and yield information on the relative orientation of the lubricant backbone with respect to the carbon surface. They found that the surface energy of PFPE lubricated disks decreases with increasing lubricant thickness. Waltman et al. [15] showed that, in the case of bonded ZDOL, much stronger interactions occur between the hydroxyl end-groups of ZDOL and the carbon surface as compared to the mobile ZDOL. Also, Karis et al. [16] showed that the polar component of the surface energy for ZDOL exhibits oscillations as a function of lubricant thickness. As the amount of lubricant applied to the surface increases, the surface energy decreases since the fraction of the high-energy carbon surface covered by the low surface energy PFPE increases. Neutralization of the surface active sites by the addition of hydroxyl terminated ZDOL results in a decrease in the measured surface energy with increasing ZDOL thickness. A local minimum in the polar surface energy results at the point where the number of lubricant end-groups matches the number of active oxide sites on the carbon surface. Matching the ZDOL end-group density with the active site density on the carbon surface results in the complete coverage of the carbon surface by ZDOL lubricant.

In our earlier work [17], we studied the ZDOL thickness effect on the tribological performance of ZDOL on hydrogenated carbon films (CHx). We observed that the lubricant interaction with the carbon overcoat varies as a function of lubricant thickness. Wear durability

of ZDOL against DLC coated sliders improves considerably when the carbon overcoat surface is fully covered with one layer of ZDOL as compared to less lubricant. This enhanced performance with thicker ZDOL can be attributed to two mechanisms: (1) full coverage of the carbon surface with ZDOL, and (2) a thicker layer of mobile ZDOL to reflow into the wear track and replenish the lube displaced during dragging. These results illustrate the importance of the mobile ZDOL layer in providing good wear durability. However, the bonded ZDOL layer also interacts strongly with the carbon surface, resulting in a lower removal rate of ZDOL molecules from the carbon surface. Thus, an optimized composition (mobile/bonded) of ZDOL lubricant should provide the best tribological performance at the head-disk interface.

In this report, we study the effect of ZDOL bonding on hydrogenated carbon films (CHx) using an ultra-high vacuum (UHV) tribochamber equipped with a mass spectrometer. The studies consist of drag tests and thermal desorption experiments in the UHV tribochamber. Two decomposition processes of ZDOL under sliding friction conditions are studied - one is with a carbon film coated slider/CHx coated disk system, and another is with an uncoated Al_2O_3-TiC slider/CHx coated disk system. Samples with a total of 12Å ZDOL lubricant but different mobile fractions are used in this study. We observed that wear durability in slow speed drag tests improves with an increase in the mobile portion.

II. EXPERIMENTAL PROCEDURE AND SET-UP

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 x 10^{-8} Torr, which was described in detail in a previous paper [18]. The QMS provides 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. We also monitor friction or temperature data from strain gauge transducers or a thermocouple, respectively. It is noted that all AMUs from 1 to 192 of ZDOL decomposition products generated at the Al_2O_3/TiC slider/CHx disk interface were previously investigated under the same test conditions and only the most pertinent AMUs are 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 30 mN, and a sliding time of 20 minutes. The sliders used were 30% (1.2mm by 1mm) negative-pressure Al_2O_3/TiC sliders with and without amorphous diamond like carbon (DLC) films on the air bearing surfaces. The disks were commercial 95mm smooth thin film disks with a 75Å amorphous hydrogenated carbon overcoat (CHx). The hydrogen content in the CHx film was 30 atomic percent. The disks were lubricated with ZDOL by a dipping process. We used FTIR to measure the lubricant thickness of these disks before the UHV drag tests. These FTIR thickness measurements were confirmed by ellipsometry and X-ray reflectivity spectroscopy. After the thicknesses were measured, the disks were washed in perfluorohexane and 2,3-dihydro-perfluoropentane to remove any soluble lubricant [19], and then the thickness was remeasured. The lubricant that was retained on these

disks is defined as the "bonded" portion, while that removed by the solvent wash process is defined as the "mobile" portion. The resulting thickness of ZDOL on the disks in this study were 12Å but with four different mobile portions (0%, 33%, 67%, and 100%). The molecular weight of ZDOL is 2000 AMU. The chemical formula of ZDOL is given below:

$$HO\text{-}CH_2\text{-}CF_2O\text{-}(CF_2O)m\text{-}(CF_2CF_2O)n\text{-}CF_2\text{-}CH_2\text{-}OH, \text{ where } n/m = 2/3$$
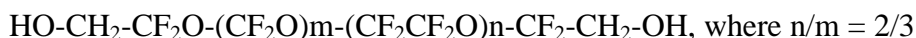
In preparation for the thermal desorption tests in the tribochamber the heater was also baked at 600K at high vacuum for 4 hours to bake out the residual lubricants left on the heater after each thermal desorption test. A CHx/ZDOL disk was cut into 2cm squares. The lubricated samples were mounted onto the heater and the temperature was measured by a thermocouple in contact with the heater near the sample. As with the drag tests, the chamber was pumped down to 2×10^{-8} Torr and the channels of the QMS were assigned to selected AMUs. A typical experiment consisted of heating a sample at a rate of 0.2 K/sec starting at room temperature and stopping before 550 K. A mass spectrum was collected on a computer every two seconds during the heating. Afterwards, the spectra were analyzed in order to obtain the thermal desorption profile for each mass as a function of sample temperature.

III.   Results and discussion

A. *Results from the UHV drag tests*

In this section, we present the results from the UHV drag tests. Before starting the friction tests, the background intensities were recorded for 80 seconds by the mass spectrometer. Figure

1(a) shows the friction coefficient curves for the 12Å of all bonded ZDOL lubricant. The friction coefficient curves for the 12Å ZDOL disks with different bonded fractions are similar to those in Fig. 1(a) and will not be shown here. For the 70Å DLC-coated Al_2O_3/TiC slider, the friction coefficient started at 1 and increased steadily to 1.2 within 40 drag cycles before wear occurred. For the uncoated Al_2O_3/TiC slider, the friction coefficient increased to a peak value of 1.8 immediately and then dropped to 0.5. A wear track was observed on the disk just after the friction coefficient dropped. The friction variation, as shown in Fig. 1(a), is most likely due to three-body contact resulting from small wear particles that were generated at the head-disk interface. The above results indicate that 12Å of all bonded ZDOL has little lubricating action against both the uncoated and the DLC-coated Al_2O_3/TiC sliders.

Figure 1 (b) shows the integrated mass spectra of ZDOL fragments produced at the head-disk interface. The four primary peaks are from mass fragments CFO (47), CF_2O (66), CF_3 (69), and C_2F_5 (119). In our previous studies of CHx carbon overcoats paired with carbon-coated sliders [13], the primary mechanism of ZDOL decomposition was due to frictional effects, and it was characterized by the generation of CFO (47), and CF_2O (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 CF_3 (69) and C_2F_5 (119). In order to eliminate the effect of the background spectrum the background level was subtracted from the integrated mass spectrum. This mass spectrum for the 70Å DLC coated Al_2O_3/TiC slider is similar to that of ZDOL vapor when it is heated to 300°C, as reported by Kasai et al. [5], where the primary decomposition peaks are masses 47 (CFO) and 66 (CF_2O) due to frictional heating [13]. Because no contact with Lewis acid is possible in this case, the ZDOL decomposition is attributed to friction/thermal actions

only, and therefore it should be directly related to the molecular bond energies. The relevant bond dissociation energy [20] is used to judge the bond strength and possible decomposition trends. Because the weakest bonds in ZDOL are C-O-C and C-C, the ZDOL molecule is expected to cleave preferentially at these locations. A mechanism of radical formation proposed by Sianesi et al. [21] postulates that PFPEs dissociate by breaking the weaker C-C bonds followed by β scission to form perfluorinated aldehyde and alkene. Therefore, masses 47 (CFO) and 66 (CF_2O) are the primary fragments due to frictional heat action.

For the uncoated Al_2O_3/TiC slider, the mass intensities of the fragments CF_3 (69) and C_2F_5 (119) increased more than 100% as compared to the DLC coated slider case. The decomposition mechanisms of ZDOL in the case of the uncoated Al_2O_3/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 (CF_3) and 119 (C_2F_5), resulting in worse tribological performance as shown in Fig. 1(a). In our study of CHx and CNx overcoats [22], we observed that the hydrogen evolution from CHx overcoats initiates the lubricant catalytic decomposition with uncoated Al_2O_3/TiC sliders, forming CF_3 (69) and C_2F_5 (119). The generation of hydrofluoric acid (HF) during thermal desorption experiments confirms the formation mechanism of Lewis acid, which is a necessary component for catalytic decomposition of ZDOL lube, as described in our earlier work [22]. On the other hand, for CNx films, lubricant catalytic decomposition was prevented due to less hydrogen evolution from the CNx overcoat.

Figure 2 shows the degradation intensity of mass 47 (CFO) during UHV drag tests on CHx disks with different ZDOL bonded fractions. The degradation intensity of the frictional fragment 47 (CFO) increased as the bonded fraction decreased for both the uncoated and coated slider cases. This suggests that an important role of the mobile ZDOL layer is to replenish the lube displaced during dragging. With a larger mobile portion, more lubricant reflows into the test track. Thus, more material is decomposed resulting in the stronger degradation intensity as shown in Fig. 2.

Figure 3 shows the wear durability of these CHx disks as a function of ZDOL bonded fraction. With DLC coated sliders, the wear durability significantly improved when the lubricant mobile fraction increased. These results demonstrate the benefits of using more mobile ZDOL - a reflow of the lubricant into the wear track provides more material to be decomposed so the carbon overcoat surface is protected for a longer duration against sliding. As the mobile portion increases, the replenishment rate also increases as suggested by O'Connor et al.[23] and Ma et al. [24,25,26] in their studies of the spreading behavior of PFPE on silica surfaces. In their papers, Ma states that the functional end-groups of ZDOL significantly retard the diffusion process due to their stronger interactions with the carbon surface. In the context of our study, as the mobile portion 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 more mobile ZDOL on the protective carbon surface which enhances the wear durability of the interface. However, there was no benefit in using more mobile lubricant for the uncoated slider cases. Strong catalytic reactions occurred with the uncoated slider during dragging, resulting in the very poor tribological performance for every case.

B. *Results from UHV thermal desorption tests*

In this section, we present and discuss the results from UHV thermal desorption experiments. Figure 4 shows the thermal desorption profiles for mass 47 (CFO) of CHx disks with different ZDOL bonded fractions at a heating rate of 0.2 K/sec. Two thermal desorption peaks were found during these experiments: one is between 350 K and 400 K, and the other is between 425 K and 500 K. In our earlier report [17], we concluded that most of the mobile lubricant is desorbed at the first thermal desorption peak, while the bonded lubricant portion increased due to the annealing effect of temperature during the first period. These results are similar to those found by Waltman et al. [15, 27]. They found that the initially applied mobile ZDOL is depleted via evaporative loss as well as bonding of the lubricant to active sites on the carbon surface at elevated temperatures above 320 K. The initially applied lubricant is thermodynamically unstable during the thermal desorption process. As a result of the intramolecular motion within the ZDOL polymer on the surface, the free hydroxyl endgroups of the mobile lubricant pass through orientations favorable for bonding. Since transitions from the mobile state to the bonded state are thermodynamically driven by the decrease in the free energy associated with this transition, the bonded lubricant present on the surface increases with increasing temperature during the first desorption period. The relative branching into the evaporation and bonding channels is molecular weight dependent with increasing molecular weight favoring bonding. We observed that the activation energy for ZDOL evaporation is highly dependent on molecular weight and decreased rapidly with decreasing molecular weight [28]. In Fig. 4, we also observed that more mobile lubricant was desorbed during the first period with an increasing mobile portion, while more bonded lubricant was desorbed during the second period as the bonded

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portion increased. In addition, for the pure mobile ZDOL case, some mobile lubricant bonded during the first period and then desorbed during the second period. For the pure bonded ZDOL case, very little was desorbed during the first period. Thus, we can conclude that only the mobile ZDOL layer is desorbed during the first thermal desorption period and the residual bonded ZDOL layer is desorbed during the second thermal desorption period. So the desorption energy of bonded ZDOL lubricant is higher than that of mobile ZDOL lubricant. Waltman et al. [15] also showed similar results, that the surface energy of the bonded lubricant is substantially lower than that of the mobile lubricant, reflecting the increased interaction strength that occurs as a result of bonding.

Moreover, the temperature of the first peak, which is attributed to the desorption of the mobile ZDOL layer, remains almost constant as a function of ZDOL thickness. This result indicates that the desorption energy of the mobile ZDOL layer is independent of the lubricant thickness. However, the temperature of the second peak, corresponding to the desorption of bonded ZDOL, shifts to a lower temperature with increasing ZDOL bonded portion as shown in Fig. 5. This result indicates that the desorption energy of bonded lubricant decreases with increasing bonded lubricant thickness. One possible explanation is that the surface of the amorphous carbon is populated with sites of different interaction strengths, where stronger bonding sites are associated with higher binding energy [11]. When ZDOL molecules adsorb on the surface, they first occupy the stronger sites (sites with higher desorption energy). As more ZDOL molecules (thicker lubricant) are adsorbed, weaker sites (sites of lower desorption energy) are occupied. Thus, for samples with lower bonded portions the second peak temperature is higher, and it is lower for higher bonded fractions. In our lubricant thickness study [17], we also

observed that during UHV drag tests strong lubricant decomposition occurs when the lubricant is thicker. With thicker bonded ZDOL, the thermal desorption energy of bonded lubricant decreases, so both the frictional decomposition and the catalytic reaction are enhanced for thicker ZDOL.

IV. Conclusion

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 the gaseous wear products generated at the HDI during dragging. The UHV drag tests show that the lubricant interaction with the carbon overcoat varies as a function of the fraction of bonded lubricant. The wear durability of disks with DLC-coated sliders improves with increased mobile lube fraction because the lubricant provides a reservoir to constantly replenish the ZDOL displaced in the wear track during the drag tests. However, there was no benefit found by using a larger mobile portion for the uncoated slider cases. Strong catalytic reactions occurred with the uncoated sliders and these catalytic reactions counteracted the benefits of using a larger mobile fraction.

Based on the results from the thermal desorption experiments, we conclude that only the mobile ZDOL lubricant was desorbed at the first thermal desorption peak (between 350 K and 400 K), and the residual bonded ZDOL lubricant was desorbed at the second thermal desorption peak (between 425 K and 500 K). Moreover, the temperature of the second peak, corresponding

to the desorption of the bonded ZDOL, shifts to a lower temperature with increasing bonded ZDOL thickness.

ACKNOWLEDGMENTS

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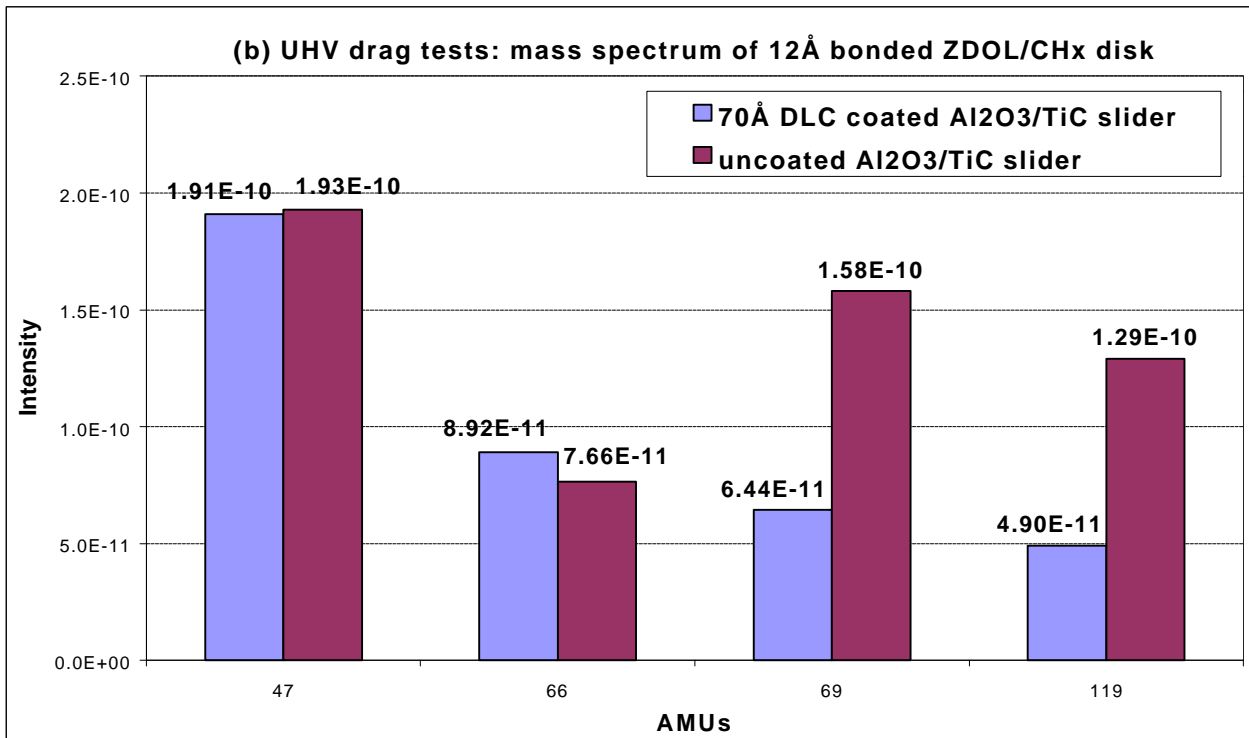
CAPTIONS

Figure 1: (a) friction coefficient of UHV drag test on 12Å all bonded ZDOL lubricated disk; (b) mass spectrum of four major ZDOL decomposed fragments.

Figure 2: integrated degradation intensity of ZDOL fragment CFO (47) during UHV drag tests on CHx disks with different ZDOL bonded fractions.

Figure 3: wear durability of CHx disks with different ZDOL bonded fractions during UHV drag tests against coated and uncoated Al_2O_3/TiC sliders.

Figure 4: thermal desorption history profiles of mass 47 (CFO) during thermal desorption tests on CHx disks with different ZDOL bonded fractions.

Figure 5: thermal desorption peak temperatures of ZDOL for CHx disks with different ZDOL bonded fractions.

(b) UHV drag tests: mass spectrum of 12Å bonded ZDOL/CHx disk

Legend:
- 70Å DLC coated Al2O3/TiC slider
- uncoated Al2O3/TiC slider

Y-axis: Intensity
X-axis: AMUs

Data values:
- 47: 1.91E-10, 1.93E-10
- 66: 8.92E-11, 7.66E-11
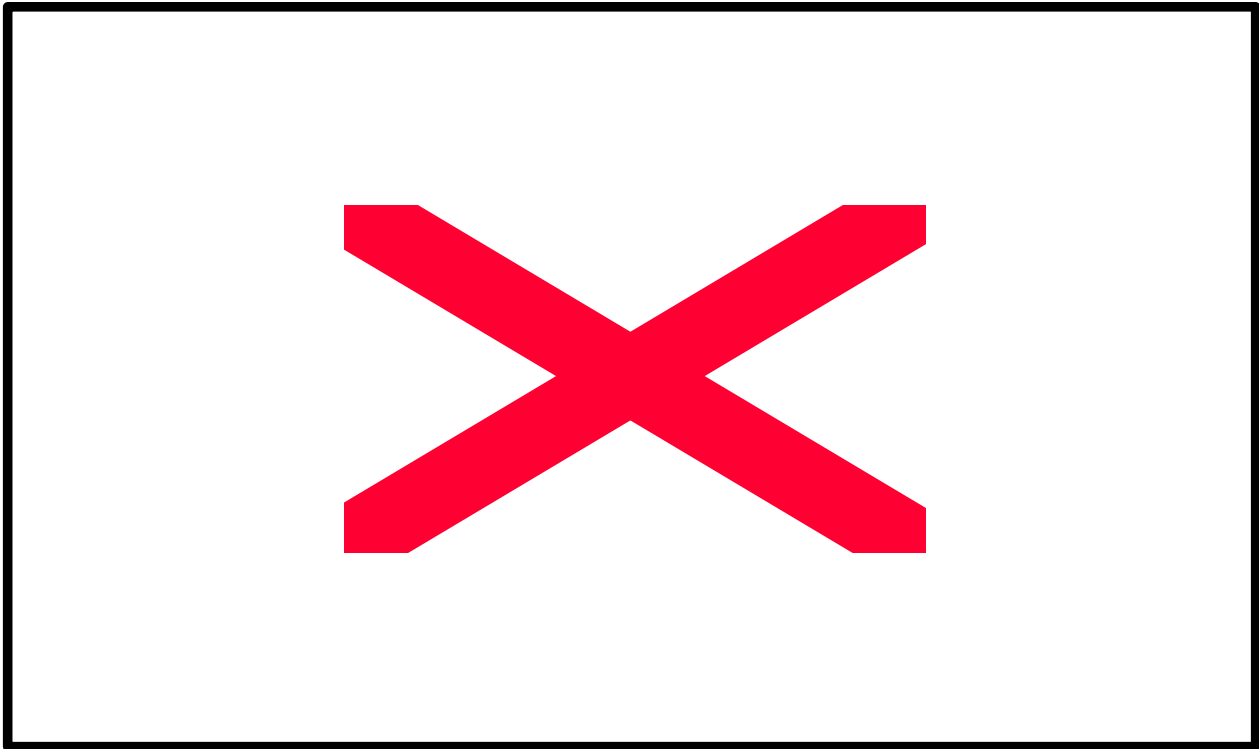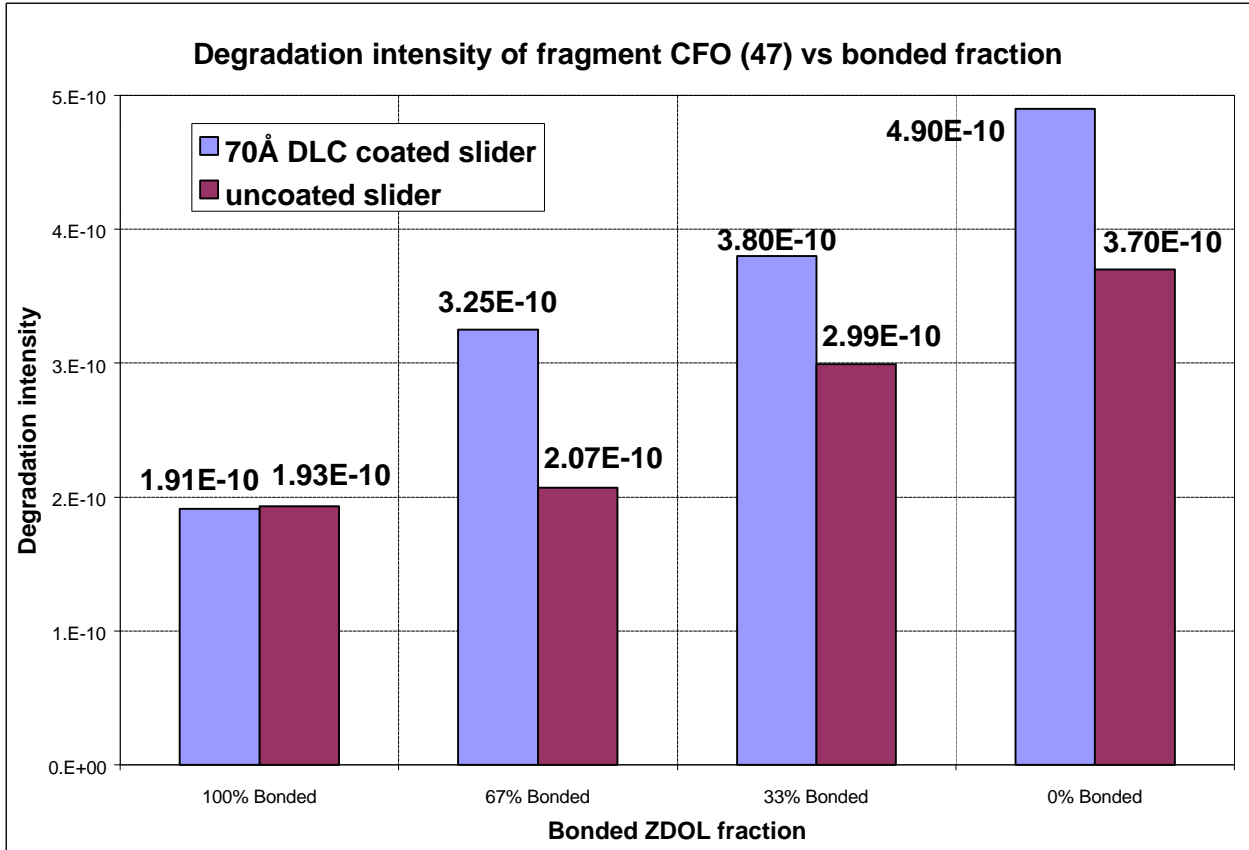- 69: 6.44E-11, 1.58E-10
- 119: 4.90E-11, 1.29E-10

Figure 1: (a) friction coefficient of UHV drag test with 12Å all bonded ZDOL lubricated disk; (b) mass spectrum of four major ZDOL decomposition fragments.

Figure 2: integrated degradation intensity of ZDOL fragment CFO (47) during UHV drag tests on CHx disks with different ZDOL bonded fractions.
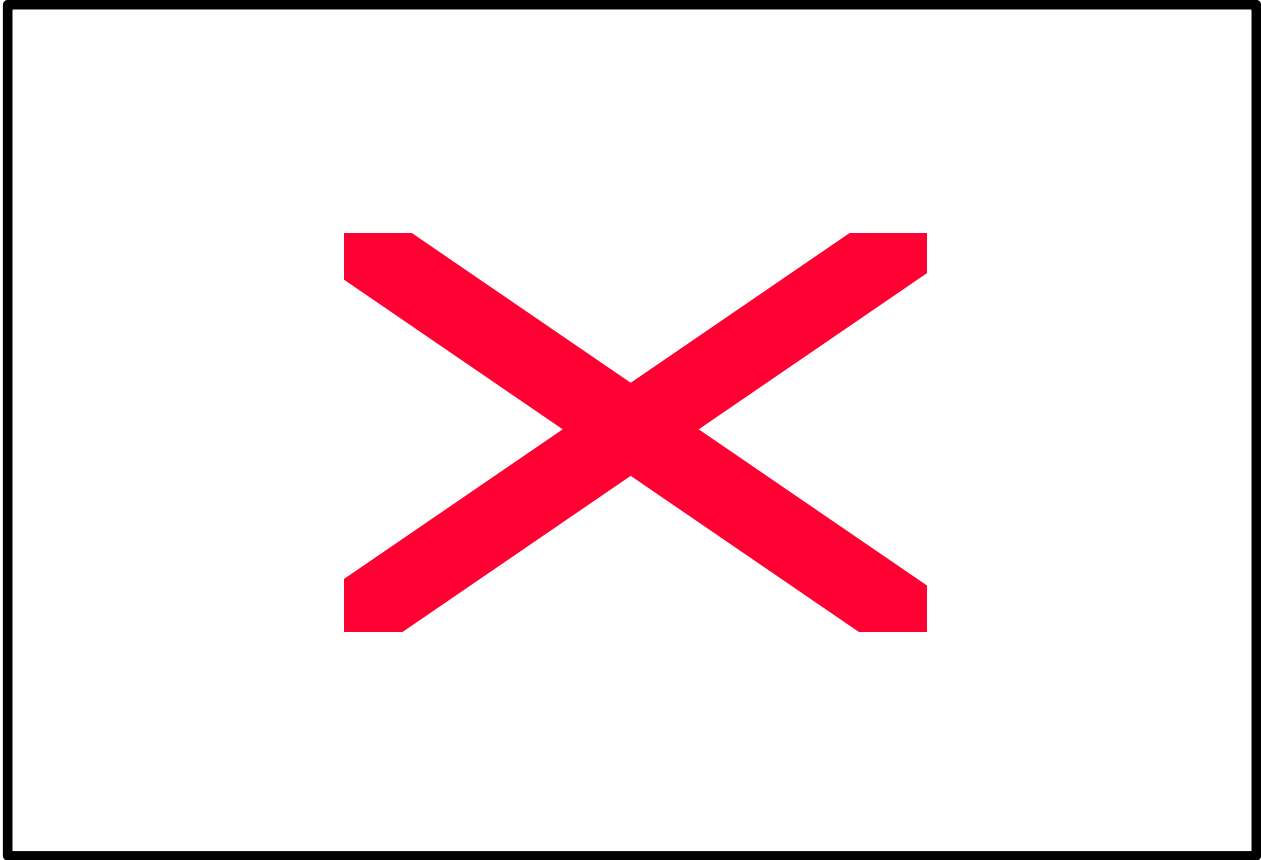
Figure 3: wear durability of CHx disks with different ZDOL bonded fractions during UHV drag tests against coated and uncoated Al_2O_3/TiC sliders.
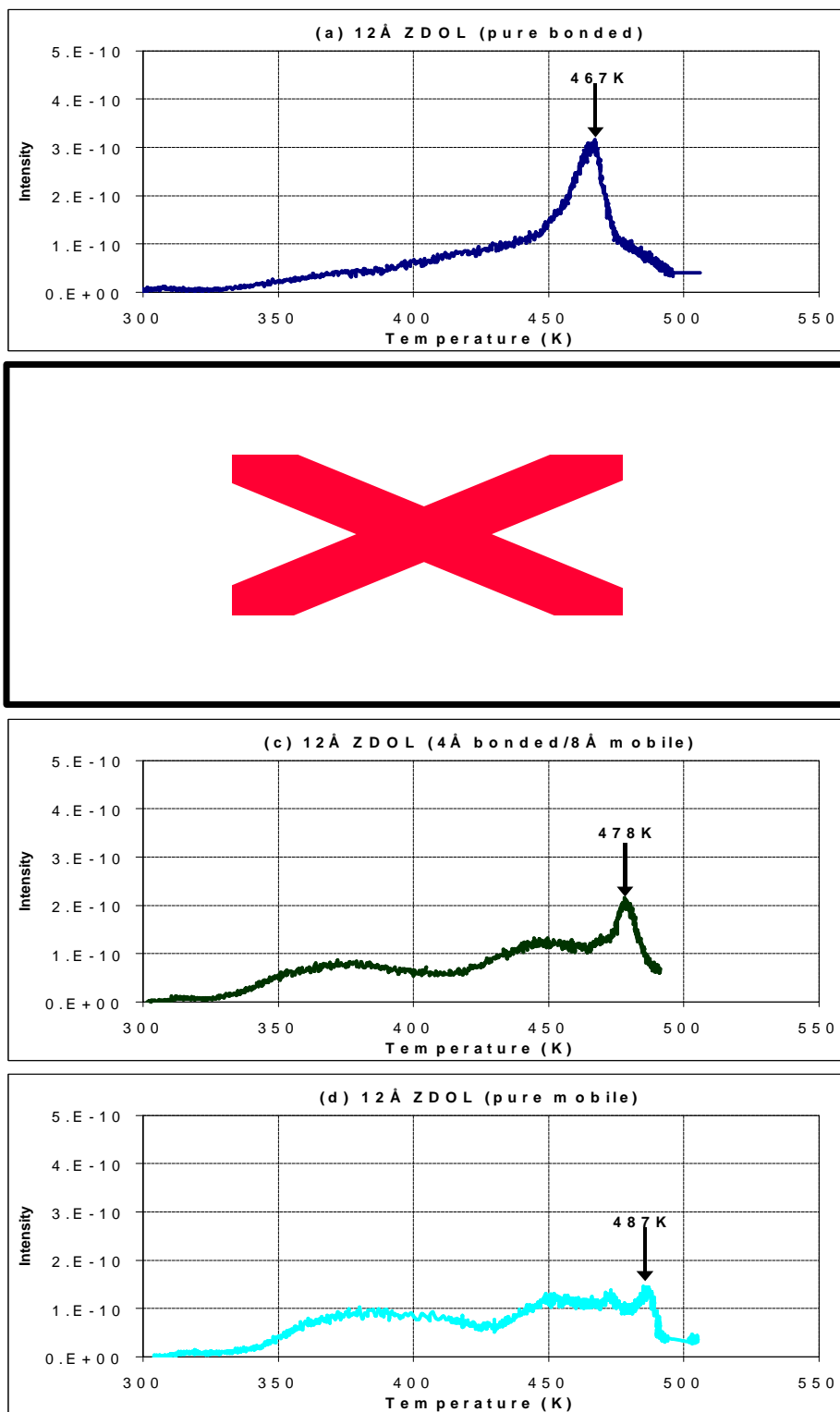
Figure 4: thermal desorption history profiles of mass 47 (CFO) during thermal desorption tests on CHx disks with different ZDOL bonded fractions.
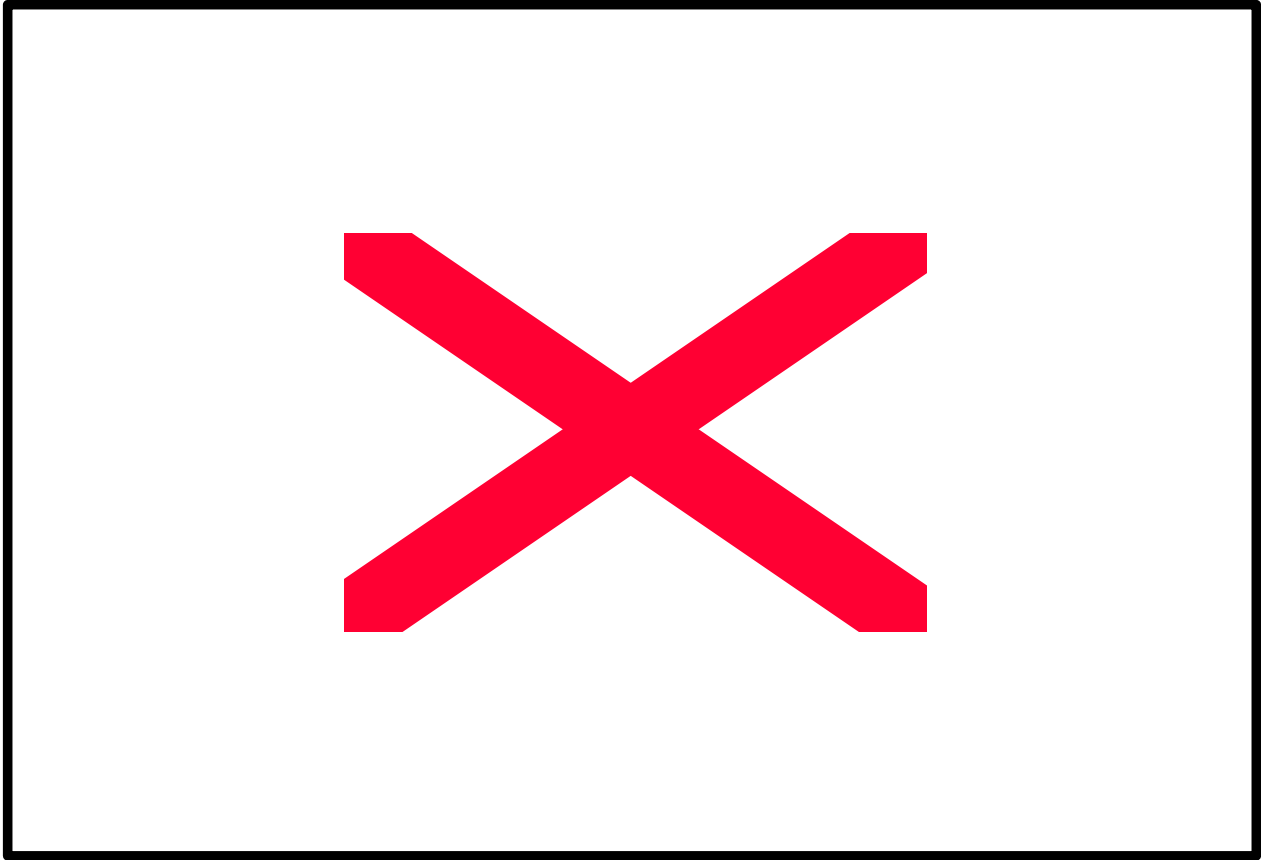
 Figure 5: thermal desorption peak temperatures of ZDOL for CHx disks with different ZDOL bonded fractions.