The Decomposition Mechanisms and Thermal Stability of ZDOL Lubricant on Hydrogenated Carbon Overcoats

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Abstract

Tribo-chemical studies 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 thermal desorption experiments in an ultra-high vacuum (UHV) tribochamber. 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. During the sliding at the carbon-coated slider/ZDOL lubricated CHx disk interface, frictional heat is the primary decomposition mechanism of ZDOL.

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

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. Their thermal stability and decomposition mechanisms have been extensively studied. Many experiments demonstrate that PFPEs, subjected to electron irradiation, are easily decomposed into smaller fragments [1]. Vurens et al. [2] used low energy electrons to bombard PFPEs and observed that the electron decomposition of PFPEs occurs at an energy below their ionization potential (about 14 eV). It is reported that the decomposition rates of PFPEs are much higher in the presence of Lewis acid forming materials, such as Fe₂O₃ [3], Al₂O₃ and AlCl₃ [4], than their intrinsic decomposition rate. Our earlier studies of the decomposition of ZDOL lubricant on nitrogenated carbon overcoats (CNx) [5] 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 due primarily to frictional actions [6]. The thermal stability of PFPE molecules has also been studied extensively. PFPE is intrinsically stable below ~570K, even in an oxygen atmosphere [7, 8]. However, Fomblin-Z dissociates in the presence of Lewis acid sites on Fe₂O₃ at 450°K [9], and on Al₂O₃ at 470°K [10]. Lin et al. [11] used temperatureprogrammed reaction/desorption (TRP/D) and electron simulated desorption (ESD) to study the roles of temperature and triboelectric charges in the decomposition of Fomblin-ZDOL lubricant molecules. They showed that the threshold temperature for dissociation of the Fomblin-ZDOL molecule is at 500-550K in accordance with the known thermal stability of the free ZDOL molecule. Vurens [12] also showed that molecules having a CF₂-CF₃ endgroup (Demnum S65) display enhanced thermal stability compared to molecules with the CF₂-CF₂-CH₂-CH₂O-phenyl endgroup (Demnum SP). Gellman [13] showed that the heat of adsorption of the hydrocarbon ether was greater than that of the corresponding fluorocarbon ether, suggesting that the ethers are bonded to the films through the donation of the electron pairs on the oxygen atom. Gellman also proposed a model in which the electropositive nature of the hydrogen in the a-CH films weakens the extent of electron donation from the ether lone pairs and, hence, weakens the bonding of the ethers to the a-CH films. Furthermore, Perry [14] used temperature-programmed desorption and scanning force microscopy to probe the interaction of ZDOL with both hydrogenated carbon overcoats and nitrogenated carbon overcoats. The data showed that the nitride surfaces are more reactive toward the ZDOL lubricant and, as a result, the thin lubricant film is more tightly bound to the overcoats. As the flying height of sliders approaches zero and near contact, the chances of contact between the head and the disk are greater. Ettles [15] used a numerical solution in two dimensions to predict the thermal effects of the asperity contact between sliders and magnetic recording disks. He showed that temperatures exceeding several hundred degrees centigrade are quite possible. Suzuki et al. [16] estimated that the contact temperature in the MnZn ferrite slider/thin film disk case may be up to 275°C. The temperature rise and signal loss were especially significant when crash of the slider occurred, resulting in large-scale wear of the disk surface. Therefore, understanding the thermal response of the materials used in the hard drive becomes imperative.

In this paper, we study the ZDOL decomposition mechanisms 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 Al₂O₃-TiC slider/CHx coated disk system. Chemical bonding theory is used to analyze the decomposition process of ZDOL. The decomposition mechanisms caused by friction, electron bombardment and catalysis are proposed. Moreover, the effects of thermal stability, tribocharge, mechanical action and Lewis acid catalysis on ZDOL decomposition are discussed.

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) equipped in a vacuum chamber with base pressure < 2 x 10⁻⁸ Torr, which was illustrated in a previous paper [17]. 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 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 Al₂O₃/TiC slider/CHx disk interface were previously investigated in the same test conditions in our laboratory, thus only the most important AMUs were monitored in this study.

Drag tests in the tribochamber were conducted as follows. Initially, the tribochamber was baked out at 150°F in 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 were 50% (2mm x 1.6mm) taper-flat Al₂O₃/TiC sliders with and without amorphous carbon films on the air bear surfaces. The disks were commercial 95mm smooth thin film disks with 75Å amorphous hydrogenated carbon overcoat (CHx). The hydrogen content in the CHx film was 5 at.%. The disks were lubricated with ZDOL by a dipping process. The resulting thickness of ZDOL on the disks was 9Å.

In preparation for the thermal desorption tests in the tribochamber, in addition to baking the chamber, the heater was baked at 600°F in high vacuum for 4 hours to bake out the residual lubricants left on the heater after each thermal desorption tests. A CHx/ZDOL disk was cut into square pieces of roughly 4 cm². The lubricated samples were mounted onto a heater and the temperature of the samples are measured by a thermocouple in contact with the heater near the heating sample. As with the drag tests, the chamber was pumped down to 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 1K/s starting at room temperature and stopping before 500°F. A mass spectrum was collected on a computer every two seconds (i.e., every 2K) during the heating of the samples. 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. Study of the decomposition mechanisms of ZDOL lubricant/hydrogenated carbon (CH_x) overcoats

In this section, we present and discuss the results from the UHV drag tests. Figure 1 shows the friction coefficient curves for the ZDOL lubricated CHx disks against an uncoated Al2O3/TiC slider and a carbon coated slider. Before the friction tests started, the background intensities were recorded for 80 seconds by the mass spectrometer. For the uncoated Al2O3/TiC slider, the friction coefficient increased to a peak value of 1.6 within 40 seconds and dropped to 0.4 after reaching its peak value. One wear track was observed on the disk just after the friction coefficient had dropped. This is due to three body contact due to small wear particles that were generated at the head-disk interface, which resulted in an unstable friction curve as exhibited in figure 1. For the carbon coated slider, the friction coefficient started at 0.3 and increased steadily to 0.8 within 400 seconds before wear occurred. The above results indicate that ZDOL has little lubricating action against the uncoated slider but it has good lubricating action against the carbon coated slider.

Figure 2 shows the integrated mass spectra of ZDOL fragments produced from the carbon-coated slider/CHx disk. The five primary peaks, in decreasing intensity order, belong to mass fragments F (19), CFO (47), CO₂ (44), CF₂O (66), and HF (20). This mass specturm is similar to that of ZDOL vapor as reported by Kasai et al. [4]. No H2 (2) is released in this case.

Because no contact with Lewis acid is possible in this case, the ZDOL decomposition should be caused by friction/thermal actions only, and therefore it should be directly related to the molecular bond energies. The relevant bond dissociation energies (shown in Table I) are used to judge the bond strength and possible decomposition trends [18]. Because the weakest bonds in ZDOL are C-O-C (3.5 eV), and C-C (3.8 eV), the molecule is expected to cleave preferentially at these locations. A radical mechanism proposed by Sianesi et al. [19] postulates that PFPEs dissociate by breaking the weaker C-C bonds followed by β scission to form perfluorinated aldehyde and alkene.

The following decomposition mechanism is proposed for this case: (1) the ZDOL molecule decomposes under frictional action and then (2) the gaseous by-products are further cleaved by electron bombardment in the QMS. In other words, the following processes occur:

$$-O-/CF_2-O/-CF_2-CF_2-O-/-CF_2- \rightarrow CF_2=O (66) + -CF_2-CF_2-O- (116)$$
 (1)

$$-CF_2-CF_2-O- \rightarrow -CF_2 (50) + CF_2=O (66)$$
 (2)

$$-CF_2 \rightarrow -CF(31) + F(19)$$
 (3)

$$CF_2=O \to CFO(47) + F(19)$$
 (4)

$$CFO \rightarrow CO(28) + F(19) \tag{5}$$

In reactions (4) and (5), Oxygen is not a decomposition product because the C=O bond is stronger than the C-F bond within the CF₂=O and CFO molecules. The occurrence of HF may result from the formation of F and subsequent attack of F on surface adsorbates such as hydroxyl groups (-OH) or surface CH groups [11]. In the same way, CF₂H is from the formation of CF₂ and the hydroxyl group (-OH); CO₂ is from the formation of CO and the hydroxyl group (-OH). Moreover, the intensity of CO₂ is stronger than that of CO since the CO₂ molecule (two C=O bonds) is more stable than the CO molecule (one C=O bond). The maximum degradation intensity is from the F (19) component because F (19) is the final decomposition product of ZDOL. No larger groups than CF₂O appear in the mass spectrum; the reason is that the groups larger than CF₂O may contain weaker single bonds (e.g., C-O-C and C-C), that are easily cleaved by frictional shear and electron bombardment.

For the uncoated slider case, the integrated mass spectra of ZDOL fragments are shown in figure 3. Note that the mass intensity scale is one order of magnitude higher than that in figure 2. Besides the peaks associated with the frictional decomposition of the ZDOL molecule, new peaks appear at CF_3 (69), C_2F_5 (119), and H_2 (2) which are due to the catalytic effect of the Al_2O_3 -TiC slider material. Moreover, the major five peaks in descending order are CO_2 (44), F (19), CF_3 (69), C_2F_5 (119), and CFO (47).

The following catalytic decomposition mechanism of ZDOL on an AL_2O_3 -TiC surface is proposed, based on our results and those in literature:

1. ZDOL decomposes and forms CF_2 =O from friction stimulation, while the CH_x film degrades and produces H_2 .

$$-O-/-CF_2-O-/CF_2-CF_2-O-/-CF_2- \rightarrow CF_2=O (66) + -CF_2-CF_2-O- (116)$$
 (1)

- 2. Complex chemical reactions of ZDOL decomposition products occur with H_2 and produce HF [11].
- 3. CF₂=O and HF react with Al₂O₃ to produce a strong Lewis acid, AlF₃.

$$Al_2O_3 + 6HF \rightarrow AlF_3 + 3H_2O \tag{6}$$

$$Al_2O_3 + 3CF_2O \rightarrow 2 AlF_3 + 3CO_2 \tag{7}$$

4. Finally, rapid decomposition reactions along the main ZDOL chain take place on the AlF_3 surface to form methoxy (CF₃-O-), ethoxy (CF₃CF₂-O-) compounds, and acyl fluoride (R-CF=O).

$$R\text{-}CF_2\text{-}O\text{-}CF_2\text{-}R \rightarrow R\text{-}C=O + CF_3\text{-}R$$

$$(CF_3\text{-}R: CF_3\text{-}O\text{-}, CF_3CF_2\text{-}O\text{-}) (8)$$

Subsequent electron impact fragmentation of the products from (8) then leads to the CF₃ (69) and CF₃CF₂ (119) fragments observed in the mass spectrum. It is noted that reaction (1) is demonstrated by our test results. Reaction (6) is often used to etch alumina surfaces (Al₂O₃ films are etchable in HF) [20], and reaction (7) is supported by the occurrence of significant amounts of CO₂ and the results of John et al. [21], where acyl fluoride (R-CF=O) reacts with alumina and forms AlF₃. Reaction (8) was obtained by Kasai et al. [4].

The mass spectrum in figure 3 can be explained according to the above mechanisms as well as the measurement principle of mass spectroscopy. The decomposed products of ZDOL contain both frictional products and catalytic products. The occurrence of the CF₂O (66) and CFO (47) peaks is a result of friction and electron decomposition as shown in figure 2. Meanwhile, the catalytic process may also produce these fragments. One of the catalytic products, R-CF=O, may decompose into two fragments (CF₂=O and CF=O) under electron impact as shown in reaction (9). This explains why the intesities of CF₂=O and CF=O are stronger in figure 3 (uncoated slider case) than in figure 2 (carbon coated slider case).

$$R-CF=O \rightarrow CF_2=O \text{ or } CF=O$$
 (9)

The CF_3 peak is due to the cleavage of the catalytic products with methoxy end groups (CF_3 -O-) as shown in reaction (10). The C_2F_5 peak is due to the cleavage of the catalytic products with ethoxy end groups (CF_3CF_2 -O-) as shown in reaction (11).

$$CF_3$$
-O-R $\rightarrow CF_3$ (10)

$$CF_3CF_2$$
-O-R $\rightarrow CF_3CF_2$ (11)

The occurrence of the HF and CF_2H fragments is due to the H_2 evolution of the worn CHx surface and subsequent complicated reactions of H_2 with ZDOL decomposition products (CFx). The formation of HF involves a basic radical reaction of the H and F radicals as shown in reaction (12). The formation of CF_2H may involve more complicated radical reactions.

$$H + F \rightarrow HF$$
 (12)

The strongest intensity peak is from CO_2 which is due to reaction (7) and is not from the formation of CO and the hydroxyl end group as mentioned before. The evidence is that the CO intensity is not very strong in figure 3 as compared to the CO_2 intensity since CO is the source of the formation of CO_2 by that mechanism.

Based on the above mass spectroscopy results, it is found that the decomposition mechanisms of ZDOL in the case of the uncoated Al₂O₃/TiC slider are much more complicated than those with the carbon coated slider. For the carbon-coated slider, ZDOL decomposition is caused by frictional actions, which include frictional heat, triboelectrons, and mechanical shear actions. For the uncoated slider, ZDOL decomposition is controlled by tribochemical and catalytic reactions. Tribochemistry involves three aspects: the first part is the tribochemical decomposition of ZDOL and the tribochemical degradation of the CHx overcoats, producing significant amounts of gaseous products such as CFO, CF₂O and H₂. The second part is the tribochemical reactions of ZDOL decomposition products with H₂, forming HF and CF₂H. The

third part is the tribochemical reactions of the Al₂O₃ surface with HF and ZDOL decomposition products, forming a strong Lewis acid AlF₃. Once the AlF₃ is formed, the catalytic decomposition reactions dominate the decomposition process of ZDOL, forming CF₃ and C₂F₅, which are responsible for the high decomposition rate of ZDOL.

B. Study of thermal desorption of ZDOL

In this section, we show and discuss the results from UHV thermal desorption experiments. Figure 4 shows thermal desorption profiles for four primary ZDOL desorbed fragments CFO (47), CF₂O (66), CF₃ (69), and C₂F₅ (119). The first two fragments are under friction/mechanical shear actions and electron bombardment as discussed before. The last two fragments are due to catalytic reactions in the presence of Al₂O₃ also as discussed before. Two desorption features peaked at 162°F and 315°F. During the first thermal desorption period (between 100°F and 250°F), mobile ZDOL lubricants were desorbed and friction/electron bombardment associated fragments (CFO and CF₂O) are observed. However, no catalytic related fragments (CF₃, and C₂F₅) are observed. These results illustrate that CFO (47) and CF₂O (66) are the primary thermal desorption fragments of mobile ZDOL in addition to the primary decomposed fragments of ZDOL under friction/mechanical shear and electron bombardment actions. These results are consistent with Lin and Kasai's results [10,11]. During the second thermal desorption period (between 250°F and 400°F), bonded ZDOL lubricants were desorbed and all four primary fragments are observed. But the friction heat/electron bombardment associated fragments (CFO and CF₂O) are much stronger than the catalytic related fragments (CF₃, and C_2F_5) which may be due to the complicated reactions between the bonded lubricants and the CHx surface as a consequence of desorption of bonded ZDOL from the CHx surface.

Figure 5 presents the desorbed mass spectra of ZDOL fragments produced from the ZDOL/CHx disk during the thermal desorption experiments at the first peak (162°F). The five primary peaks in descending order belong to mass fragments F (19), CFO (47), CO₂ (44), CF₂O (66), and HF (20) which is the same order as in figure 2. Furthermore, the ratios between these five peaks in figure 5 are close to those in figure 2. These results further prove that frictional heat is the dominant decomposition mechanism of ZDOL in the carbon-coated slider/CHx disk case.

Lin et al. [11] observed that CF₂O and CFO are the main thermal desorption fragments at 640K, probably originating from desorbing CF₂O fragments. Vurens et al. [12] found that the intensity of mass fragment CFO (47) is greater than that of mass fragment CF (31) in the thermal desorption profiles of ZDOL, while for the Demnum compounds the converse is true. This indicates that the desorption products for ZDOL are substantially more oxygen rich (CFO, and CF₂O) compared to those from the Demnum compounds, which should be expected since the ZDOL backbone has an O/C ratio twice that of the Demnum backbone. These results are consistent with those in this study.

Figure 6 presents the desorbed mass spectra of ZDOL fragments produced from the ZDOL/CHx disk during the thermal desorption experiments at the second peak (313° F). The spectrum is very similar to the one in figure 5, except for fragment H₂ (2). H₂ is the highest

desorbed peak in figure 6 due to the H₂ evolution from the CHx surface as a consequence of the desorption of bonded ZDOL during this period (between 250°F and 400°F). The H₂ evolution from the CHx surface initiated more complicated desorption actions as exhibited in figure 4 (e.g., stronger desorbed fragments CF₃, and C₂F₅). During the first desorption period (between 100°F and 250°F), little desorbed H₂ fragment is observed since only mobile ZDOL is desorbed during this period, and less desorbed reactions at the ZDOL lubricants/CHx disk interface are involved. Gellman [13] found that heating a-CH films to temperatures above 500K results in dehydrogenation of the films. It is reported [17, 22] that the strength of the interaction at the head-disk interface is influenced by the hydrogen content of the CHx films. The CHx films with higher hydrogen contents exhibit better tribological properties and higher durability against contact sliding than those with lower hydrogen contents. Therefore, the H₂ evolution from the CHx surface results in poor tribological performance.

Figure 7 presents the thermal desorption profile of fragments F (19) and HF (20). The first peak of HF was found at 100°F corresponding to the drop of F at the same time. This data is the evidence of reaction (12), where the occurrence of HF is due to a basic radical reaction of H and F radicals. Lin et al. [11] observed that HF is evolved starting at 500-550K, and which is probably due to reactions with surface OH groups and surface CH groups present at the ZDOL/hard carbon interface. HF is responsible for the AlF₃ formation which is the necessary component for the occurrence of the catalytic process as shown in reactions (6) and (7).

The first thermal desorption peak of F was found at 195°F which was 33°F higher than the peak desorption temperatures of CFO and CF₂O. The delay of the F peak supports the

assertion that F is the final decomposed product of ZDOL as shown previously in reactions (3) to (5). Another evidence is that the second desorption peak of F was found at 345°F, which was 32°F higher than those of CFO and CF₂O. These thermal desorption results further support our previous bonding energy theory in reactions (1) to (5).

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. In studies of CH_x carbon overcoats paired with carbon-coated sliders, the primary decomposition mechanism of ZDOL was due to frictional effects, and it was characterized by the generation of F (19), HF (20), CO₂ (44), CFO (47), and CF₂O (66) fragments. The absence of a carbon-coating on the slider led to a more complex catalytic decomposition mechanism of ZDOL during drag tests: (1) initial friction decomposition of ZDOL and degradation of CH_x to form gaseous products such as CF₂O and H₂; (2) reaction of these products with H₂ to produce HF and CF₂H; (3) production of AlF₃ when CF₂O and HF react with Al₂O₃; and then Lewis acid catalytic reactions on the AlF₃ surface that rapidly decompose ZDOL.

Based on the above thermal desorption mass spectra, it is found that the mass spectrum of mobile ZDOL is very similar to the mass spectrum obtained from sliding at the carbon-coated slider/CHx disk interface. These results illustrate that frictional heat is the primary decomposition mechanism of ZDOL in the carbon-coated slider/CHx disk case. At higher

temperatures (above $250^{\circ}F$), bonded ZDOL is decomposed and there is H_2 evolution from the worn CHx surface. The H_2 evolution from the CHx surface initiates more complicated desorption actions, forming CF_3 and C_2F_5 . The generation of HF during the thermal desorption experiments provides the formation mechanism of HF, which is the necessary component for catalytic reactions. The dalay of the F peak in the thermal desorption profile indicates that F is the final decomposition products of ZDOL.

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CAPTIONS

- Table I: Bond Dissociation Energies
- Figure 1: The friction coefficient curves of ZDOL lubricated CHx disks on uncoated Al₂O₃/TiC slider and carbon coated slider.
- Figure 2: The integrated mass spectrum of ZDOL decomposition products generated at the carbon-coated slider/CHx disk interface.
- Figure 3: The integrated mass spectrum of ZDOL decomposition products generated at the uncoated slider/CHx disk interface.
- Figure 4: The thermal desorption profile of four primary decomposed fragments of ZDOL.
- Figure 5: The desorbed mass spectrum of mobile ZDOL fragments produced from the ZDOL/CHx disk at 162°F.
- Figure 6: The desorbed mass spectrum of bonded ZDOL fragments produced from the ZDOL/CHx disk at 313°F.
- Figure 7: The thermal desorption profile of ZDOL fragments F (19) and HF (20).

TABLE I BOND DISSOCIATION ENERGIES

Bond	[kcal/mol]	[eV]
C-F	116	5.0
C-O-C	81	3.5
C-C	88	3.8
C-H	98	4.2
C-OH	91	3.9
O-H	103	4.5
C=C	146	6.3
C=O	179	7.8

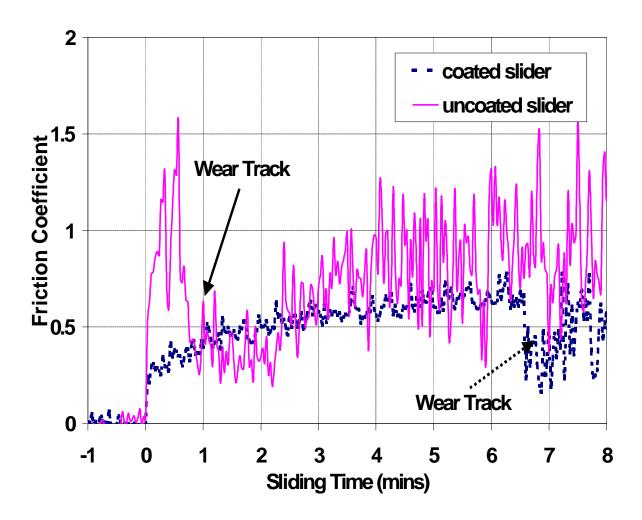


Figure 1. The friction coefficient curves of ZDOL lubricated CHx disks on uncoated Al2O3/TiC slider and carbon coated slider.

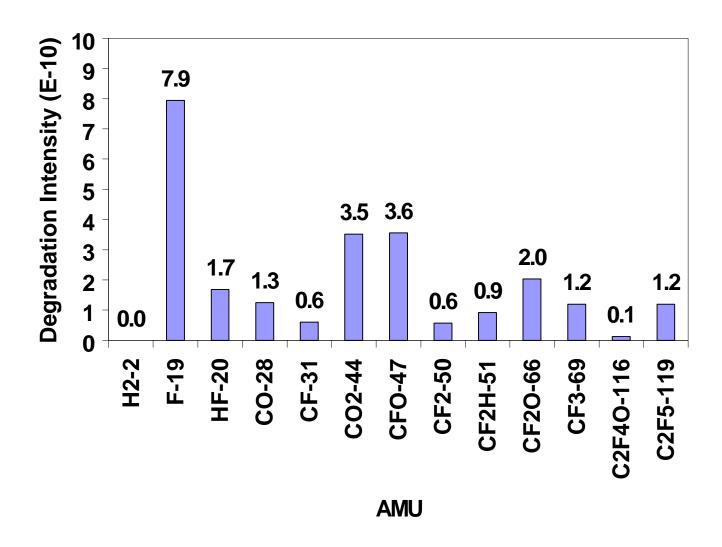


Figure 2. The integrated mass spectra of ZDOL decomposition products generated at the carbon-coated slider/CHx disk interface.

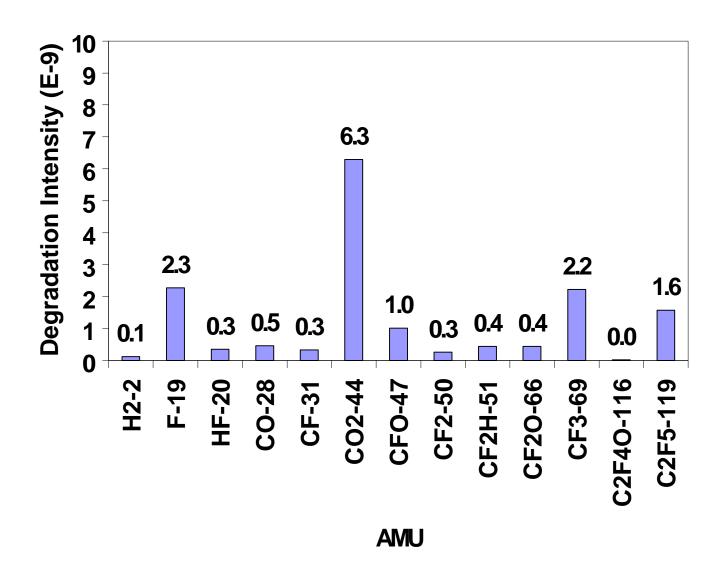


Figure 3. The integrated mass spectra of ZDOL decomposition products generated at the uncoated slider/CHx disk interface.

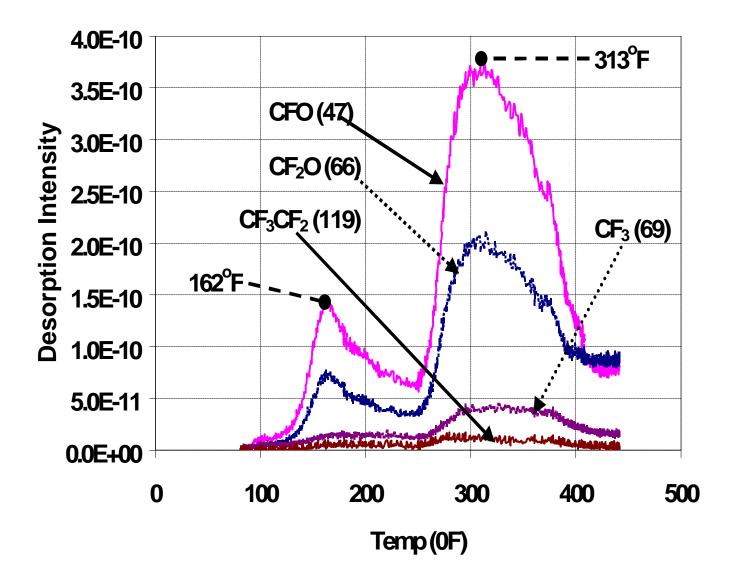


Figure 4. The thermal desorption profile of four primary decomposed fragments of ZDOL.

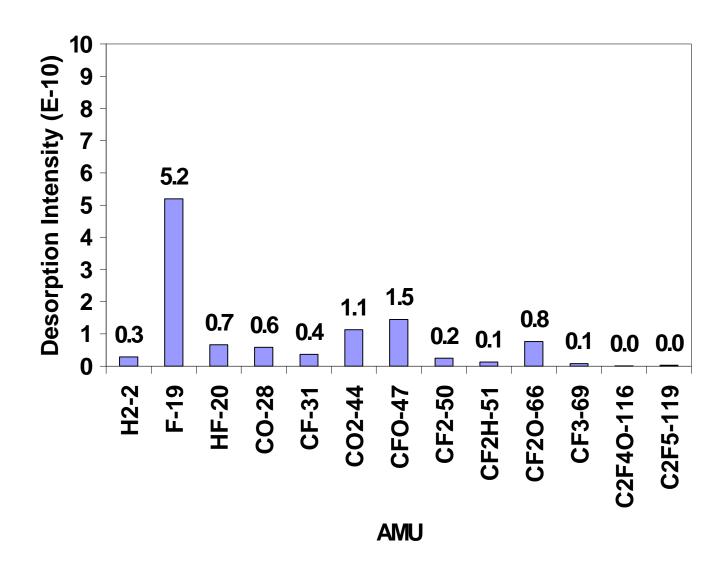


Figure 5. The desorbed mass spectrum of **mobile ZDOL** fragments produced from the ZDOL/CHx disk at $162^{\circ}F$.

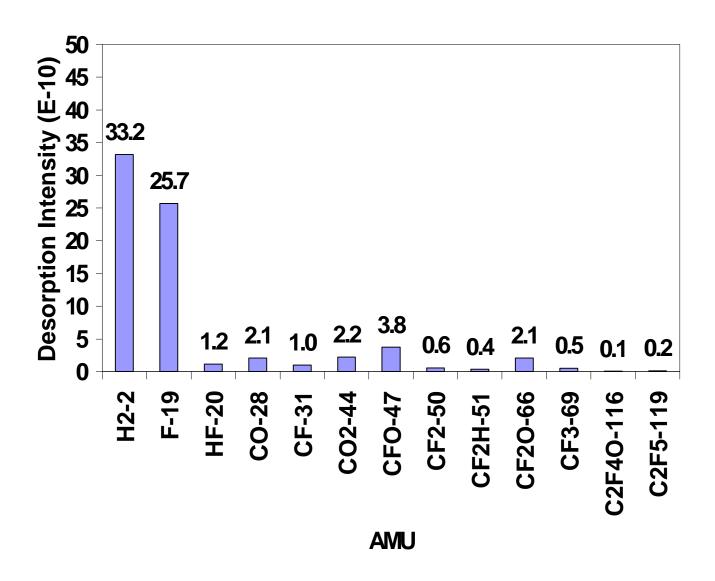


Figure 6. The desorption mass spectra of **bonded ZDOL** fragments produced from the ZDOL/CHx disk at $313^{\circ}F$.

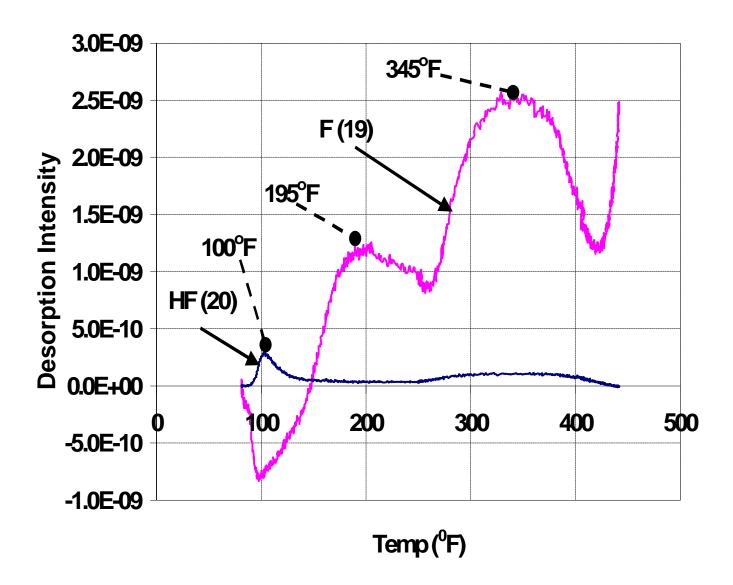


Figure 7. The thermal desorption profile of ZDOL fragments F (19) and HF (20).