

# Thermal stability of Fomblin Z and Fomblin Zdol thin films on amorphous hydrogenated carbon

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Thermal desorption spectroscopy has been used to monitor the decomposition kinetics of Fomblin Zdol and Fomblin Z lubricant films adsorbed to the amorphous carbon overcoats of hard disk media. Comparisons have been made between Fomblin Z and Zdol with vastly different molecular weights (MW = 4000 and 50 000), and films of Fomblin Z with different thickness (20 and 60 Å). Several species have been observed desorbing from the surface during heating. In all cases decomposition occurs over roughly the same temperature range of 600–750 K. This suggests that the desorption process is the result of decomposition and that the end groups of the Fomblin lubricants are not involved in determining the kinetics of this decomposition reaction. The activation barrier to the decomposition process has been estimated at  $114 \pm 6$  kJ/mol.

**KEY WORDS:** Fomblin Z; Fomblin Zdol; thin films; amorphous hydrogenated carbon; thermal stability

## 1. Introduction

The surfaces of the hard disks used for magnetic data storage are commonly protected by a thin (<100 Å) sputtered overcoat of amorphous hydrogenated or nitrogenated carbon (a-CH<sub>x</sub> or a-CN<sub>x</sub>) that is lubricated with an even thinner (<20 Å) film of lubricant. This lubricant/overcoat combination is necessary for corrosion protection and to guard against damage due to contact with the read–write head that flies at a height of <300 Å and a velocity of ~10 m/s over the disk surface [1,2]. The most commonly used class of lubricants are the Fomblins which are random co-polymers having the structure



The most common Fomblins are known as Fomblin Z and Fomblin Zdol which have fluoromethyl (X = CF<sub>3</sub>) and hydroxyl (X = CF<sub>2</sub>CH<sub>2</sub>OH) endgroups, respectively. The Fomblin fluids have very high thermal stability, low viscosity even at high molecular weights, and low vapor pressures [3–5]. As a result they serve to lubricate the disk surface for years. Nonetheless the long term stability of these lubricants on disk surfaces and the mechanisms by which they either desorb or degrade on the surface are of concern and have motivated this study and a number of studies like it [6–9].

The desorption kinetics or decomposition kinetics of adsorbed species on surfaces is most easily and commonly studied by thermal programmed desorption (TPD) spectroscopy. Although typically it requires experiments on well-defined single crystalline surfaces to allow the abstraction of good kinetic parameters from desorption data, there

have been previous studies attempting to use this method with the heterogeneous surfaces of a-CH<sub>x</sub> and a-CN<sub>x</sub> films. One set of experiments has used small compounds such as fluorinated ethers that model the Fomblin backbone in order to understand the nature of the bonding between the backbone and amorphous carbon films [10–12]. A second set of experiments has used fluorinated alcohols to model the endgroups of a particular class of lubricants, Fomblin Zdol, and has determined that its hydroxyl endgroups interact with the surfaces of amorphous carbon films through hydrogen bonding [12,13]. While these studies of the adsorption of small molecules give insight into the nature of the chemical interactions between the lubricants and the a-CH<sub>x</sub> surface they do not probe the mechanism that might lead to decomposition.

There have been TPD studies of the long chain Fomblins adsorbed on a-CH<sub>x</sub> which do give some insight into their thermal stability and the decomposition mechanism. The natural decomposition temperatures for Fomblins are high (~643 K) as concluded by Helmick and Jones [14,15]. Vurens and Mate compared the desorption of Fomblin Zdol of high molecular weight (MW = 2000) on the surfaces of graphite and a-CH<sub>x</sub> film [6]. On graphite desorption features were observed at 620 and 770 K. The same desorption features on the a-CH<sub>x</sub> films were at slightly lower temperatures (510 and 650 K, respectively). On both surfaces these desorption features were deemed to be due to decomposition processes rather than molecular desorption. The lower desorption temperature on the a-CH<sub>x</sub> films suggested that there is some feature of these surfaces that can activate the lubricant decomposition process. In another study using 2000 MW Fomblin Zdol on a hard carbon overcoat of unspecified composition, Lin et al. also observed two desorption features at 640 and 700 K [7]. The fact that these were

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coincident with the onset of hydrogen evolution from the carbon overcoat suggested that the Fomblin decomposition might be initiated by hydrogen released by the decomposition of the overcoat. More recently, there have been several detailed studies of the fragmentation pattern of the species desorbing from the  $\alpha$ -CH<sub>x</sub> surface [8,16]. These studies also observe two desorption features although at lower temperatures (352 and 429 K). Chen et al. attributed the lower desorption temperatures to the use of a heating rate of 0.2 K/s (much lower than the 5 K/s used by Vurens and Mate and 2 K/s used by Lin et al.). They used the fragmentation patterns of the desorbing species to infer that there are two different decomposition mechanisms, one referred to as a thermal decomposition mechanism and the other a catalytic decomposition mechanism [17]. These prior studies have provided several insights into the nature of the Fomblin decomposition process, however, the complex nature of the lubricants themselves and of the  $\alpha$ -CH<sub>x</sub> and  $\alpha$ -CN<sub>x</sub> overcoats leaves a great number of unknowns.

The contribution of the work described in this paper to our understanding of Fomblin decomposition is an exploration of three additional variables in the lubricant/overcoat system that might, in principle, influence the lubricant decomposition kinetics: lubricant molecular weight, lubricant endgroups, and lubricant films thickness. We find that the Fomblin decomposition kinetics on the  $\alpha$ -CH<sub>x</sub> surface is insensitive to these variables. The fact that the molecular weight of the lubricant does not influence the desorption kinetics supports the previous assumption that the process being observed is one of decomposition. The fact that the end groups do not influence the desorption kinetics implies that the mechanism is one that is rate limited by the cleavage of bonds in the fluorinated ether backbone of the molecule. This is consistent with the mechanism proposed by Kasai on the basis of studies of Fomblin decomposition on the surface of Al<sub>2</sub>O<sub>3</sub> [17–20].

## 2. Experimental

All experiments were performed in an ultrahigh vacuum (UHV) chamber with a base pressure of  $<10^{-10}$  Torr achieved through use of a cryo-pump and titanium sublimation pump. The chamber is equipped with an Ametek Dycor quadrupole mass spectrometer (QMS), which has a mass range of 1–200 amu and is capable of monitoring up to eight masses simultaneously during temperature-programmed desorption (TPD) experiments.

Seagate Technology Inc. supplied magnetic hard disk platters sputter-coated with a protective  $\alpha$ -CH<sub>x</sub> overcoat and three types of lubricant films: Fomblin Zdol of 4000 molecular weight (MW) and 20 Å thickness, Fomblin Z of 50000 MW and 20 Å thickness, and Fomblin Z of 50000 MW and 60 Å thickness. These platters were machine-punched to produce  $\sim 12.5$  mm diameter disk samples for use in the UHV chamber. The disk samples were bolted to a Ta plate with small stainless-steel nuts and bolts.

The Ta plate was in turn mounted to the UHV sample manipulator by two Ta wires spot-welded to its edge. Once mounted to the manipulator, the disk sample could be heated resistively at a constant rate to the melting point of aluminum. The sample temperature was measured using a chromel–alumel thermocouple spot-welded to the rear face of the Ta plate. Throughout the mounting procedure extreme care was taken to ensure that nothing came into contact with the lubricated surface used for the TPD experiment.

The disk samples were used for the TPD experiments as received. Normally, the chamber (and samples) used for these experiments would be subjected to a bakeout under vacuum for  $\sim 12$  h at roughly 450 K to achieve UHV conditions, however, this bakeout was omitted for these experiments to avoid any desorption of the lubricant film from the samples. Once the disk samples were mounted, the UHV chamber was subjected to approximately 12 h of pump-down at room temperature to achieve pressures in the range  $(5–10) \times 10^{-9}$  Torr. The TPD experiments were done at these pressures. The time spent in vacuum by the disk samples was minimized to avoid possible evaporation of the Fomblin film.

Within the UHV chamber the sample was positioned approximately 1 cm from an aperture to the QMS. A stainless-steel mesh was placed between the disk sample and the aperture to the QMS. While the ionizer of the QMS was turned on the mesh was biased at  $-300$  V in order to repel any electrons from the ionizer and eliminate the possibility of electron-induced damage to the Fomblin. Since the disks were already lubricated, the procedure for the TPD experiments was straightforward. The desorption step involved heating the sample at a constant rate of 2 K/s from room temperature ( $\sim 300$  K) to 850 K to induce the desorption or decomposition of the adsorbed Fomblin film. During heating, the QMS monitored the desorption of species from the surface.

## 3. Results and discussion

Fomblin decomposition was investigated using TPD to study lubricants having different molecular weight, different endgroups, and lubricants deposited at different film thickness. Figure 1 shows the TPD spectra obtained from a disk lubricated with a 20 Å layer of 4000 MW Fomblin Zdol. The spectra were generated by heating the sample at 2 K/s while simultaneously monitoring signals at  $m/q = 47$  (CFO<sup>+</sup>),  $m/q = 66$  (CF<sub>2</sub>O<sup>+</sup> or Zn<sup>+</sup>),  $m/q = 69$  (CF<sub>3</sub><sup>+</sup>),  $m/q = 100$  (C<sub>2</sub>F<sub>4</sub><sup>+</sup>), and  $m/q = 119$  (C<sub>2</sub>F<sub>5</sub><sup>+</sup>). Desorption features are observed at 625 and 720 K. These desorption peak temperatures match those observed by Lin et al. [7] (640 and 700 K) for 2000 MW Fomblin Zdol with the same heating rate, and by Vurens and Mate [6] (510 and 650 K) for 2000 MW Fomblin Zdol with 5 K/s heating rate. Chen et al. [8] observed much lower desorption peak temperatures (352 and 429 K) for 2000 MW Fomblin Zdol with 0.2 K/s heating rate. They attributed the lower desorption temperatures to the use of a much lower heating rate of 0.2 K/s.

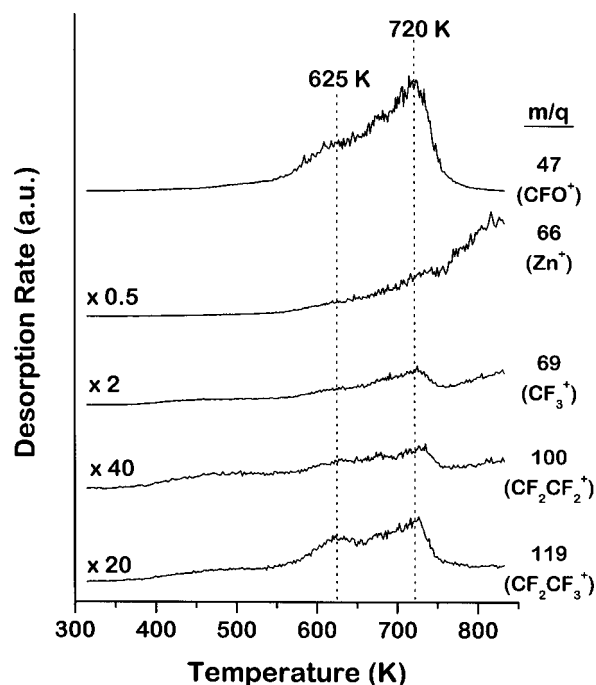


Figure 1. TPD spectra obtained from heating a disk lubricated with a 20 Å layer of Fomblin Zdol ( $\text{HOCH}_2\text{CF}_2\text{O}-(\text{CF}_2\text{CF}_2\text{O})_n-(\text{CF}_2\text{O})_m-\text{CF}_2\text{CH}_2\text{OH}$ ) with molecular weight of 4000. Two desorption peaks were observed at 625 and 720 K. The spectra were generated by simultaneously monitoring  $m/q = 47$  ( $\text{CFO}^+$ ), 66 ( $\text{Zn}^+$ ), 69 ( $\text{CF}_3^+$ ), 100 ( $\text{C}_2\text{F}_4^+$ ), and 119 ( $\text{C}_2\text{F}_5^+$ ). The heating rate was 2 K/s.

The relative magnitudes of the desorption curves obtained in the course of a TPD experiment are determined by the electron impact fragmentation pattern of the species desorbing from the surface. These can be used to identify the desorbing species in cases where they can be compared with fragmentation patterns obtained for known standards. Even a cursory inspection of the relative ratios of signals for the peaks at 625 and 720 K in figure 1 reveals that the products desorbing from the surfaces in these two temperatures regimes cannot be identical. The one fragment of figure 1 whose desorption spectrum differs significantly from all the others is that due to  $m/q = 66$ . Of the possible fragments that might be generated by the decomposition of Fomblin this would be  $\text{CF}_2\text{O}^+$ . Detection and analysis of other fragments that desorb from the surface at  $T > 750$  K reveals that this fragment is actually due to the desorption of Zn. This is easily identified since the relative intensities of the signals at  $m/q = 64, 66, 67,$  and  $68$  match the relative ratios of the naturally occurring isotopes of Zn with those masses. The source of Zn is not known but has been observed during the heating of disks from other sources. It must be present in some step of the disk manufacturing process and although it might be used only in low concentrations the fact that Zn has a relatively high vapor pressure means that it appears in desorption spectra at lower temperatures than any of the other metals or materials used in the manufacturing of the disks.

The effect of the heating rate on the peak desorption temperatures was investigated by conducting TPD experiments on the disks coated with 4000 MW Fomblin Zdol using heat-

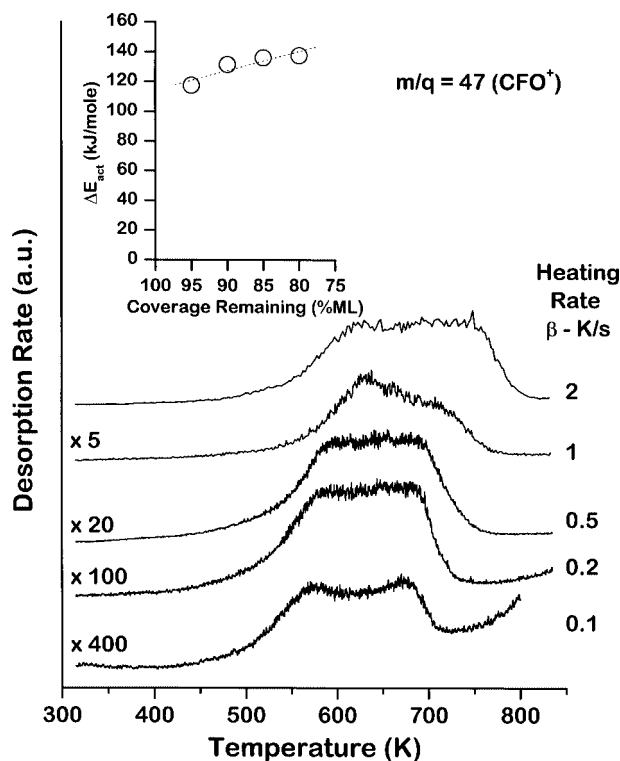


Figure 2. TPD spectra with different heating rates obtained by heating a disk lubricated with a 20 Å layer of Fomblin Zdol ( $\text{HOCH}_2\text{CF}_2\text{O}-(\text{CF}_2\text{CF}_2\text{O})_n-(\text{CF}_2\text{O})_m-\text{CF}_2\text{CH}_2\text{OH}$ ) with molecular weight of 4000. The spectra were generated by monitoring  $m/q = 47$  ( $\text{CFO}^+$ ) with heating rates from 0.1 to 2 K/s. Desorption temperatures were increased by about 60 K when the heating rate was increased from 0.1 to 2 K/s. The inset shows the coverage dependence of the activation barrier to decomposition.

ing rates ranging from 0.1 to 2 K/s. Figure 2 shows these variable heating rate TPD spectra generated by monitoring  $m/q = 47$  ( $\text{CFO}^+$ ). Analysis of the desorption experiment predicts that the peak desorption temperature should increase with increasing heating rate [21]. This is observed in figure 2, however, the desorption peak temperatures increase by only  $\sim 60$  K over a range of heating rates from 0.1 to 2 K/s.

Since the lubricated disk samples are irreversibly damaged by heating to 850 K during TPD experiments, the reproducibility of the data are tested by repeating TPD experiments with disk samples punched from the same disk platter. Figure 1 is representative of the TPD spectra obtained from many disk samples lubricated with a 20 Å layer of 4000 MW Fomblin Zdol. In all cases, the desorption temperature range was  $\sim 550$ – $750$  K with desorption features consistent with those observed in figure 1. However, the relative intensities of the two features are not consistently observed from disk sample to disk sample. Roughly half the samples give relative intensities of the peaks at 625 and 720 K such as is observed in the spectrum of figure 1. The other half give relative intensities such as those exhibited in figure 2. Thus we cannot comment meaningfully on the causes of the relative intensities of the two desorption peaks, only on the fact that they occur for all samples and that desorption always occurs in a temperature range of 600–750 K.

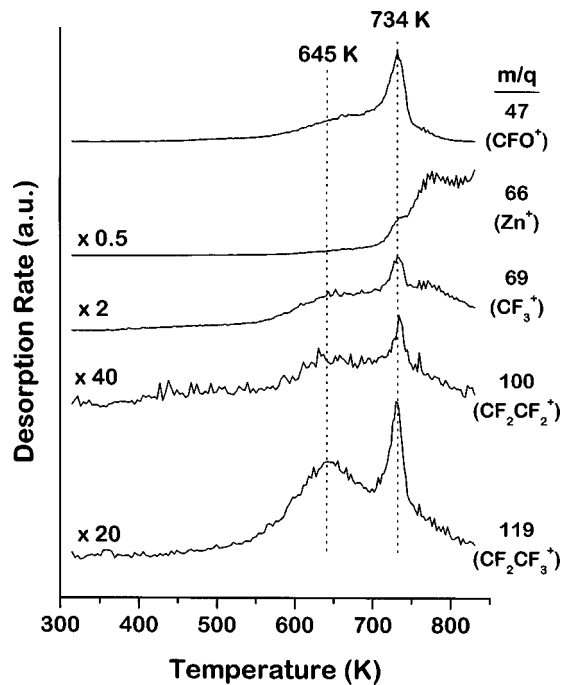


Figure 3. TPD spectra obtained from heating a disk lubricated with a 20 Å layer of Fomblin Z ( $\text{CF}_3\text{-(CF}_2\text{CF}_2\text{O)}_n\text{-(CF}_2\text{O)}_m\text{-OCF}_3$ ) with molecular weight of 50 000. Two desorption peaks were observed at 645 and 734 K. The spectra were generated by simultaneously monitoring  $m/q = 47$  ( $\text{CFO}^+$ ), 66 ( $\text{Zn}^+$ ), 69 ( $\text{CF}_3^+$ ), 100 ( $\text{C}_2\text{F}_4^+$ ), and 119 ( $\text{C}_2\text{F}_5^+$ ). The heating rate was 2 K/s.

The dependence of the lubricant decomposition kinetics on lubricant thickness was investigated by using TPD to study disks lubricated with 50 000 MW Fomblin Z of 20 and 60 Å thickness, respectively. Figures 3 and 4 show the TPD spectra from both disks obtained by simultaneously monitoring  $m/q = 47$  ( $\text{CFO}^+$ ), 66 ( $\text{Zn}^+$ ), 69 ( $\text{CF}_3^+$ ), 100 ( $\text{C}_2\text{F}_4^+$ ), and 119 ( $\text{C}_2\text{F}_5^+$ ), with a heating rate of 2 K/s. Two desorption peaks were observed at (645 and 734 K) and (650 and 740 K) for the 20 and 60 Å disks, respectively. The desorption peak temperatures are nearly identical for disks with different lubricant thickness. The implication of this observation is that the decomposition kinetics are fairly independent of the lubricant film thickness. There are differences in the relative intensities of signals at the different  $m/q$  ratios in the two desorption features obtained for the two lubricant thicknesses. Again the signal at  $m/q = 66$  is substantially different from the others and this is due to the fact that it originates from Zn desorption rather than a fragment of Fomblin Z decomposition. The intensity of the low temperature desorption features ( $\sim 650$  K) tends to increase relative to that of the high temperature features ( $\sim 740$  K) as the thickness of the Fomblin Z is increased. This can be rationalized by thinking of the lower temperature desorption feature as arising from the decomposition of the mobile “non-bonded” lubricant while the higher temperature desorption feature arises from decomposition of the “bonded” lubricant [22]. Since bonding to the  $\alpha\text{-CH}_x$  surface can only occur in the first layer of adsorbed lubricant, there is more mobile lubricant in the 60 Å lubricant film than the 20 Å film and thus more material

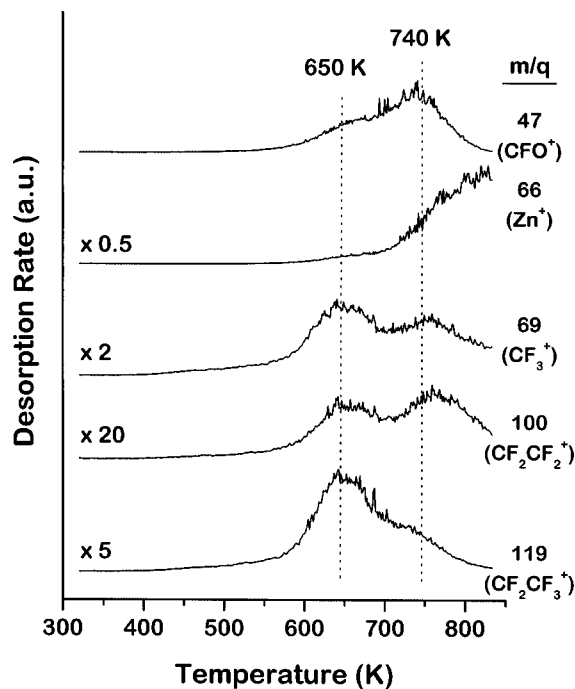


Figure 4. TPD spectra obtained from heating a disk lubricated with a 60 Å layer of Fomblin Z ( $\text{CF}_3\text{-(CF}_2\text{CF}_2\text{O)}_n\text{-(CF}_2\text{O)}_m\text{-OCF}_3$ ) with molecular weight of 50 000. Two desorption peaks were observed at 650 and 740 K. The spectra were generated by simultaneously monitoring  $m/q = 47$  ( $\text{CFO}^+$ ), 66 ( $\text{Zn}^+$ ), 69 ( $\text{CF}_3^+$ ), 100 ( $\text{C}_2\text{F}_4^+$ ), and 119 ( $\text{C}_2\text{F}_5^+$ ). The heating rate was 2 K/s.

desorbing in the 650 K feature than in the 740 K desorption feature.

Two molecular properties of the lubricants that might, in principle, influence the decomposition kinetics are the molecular weight and the type of endgroup. This has been investigated by TPD of lubricants with  $\text{-CF}_2\text{CH}_2\text{OH}$  (Fomblin Zdol) and  $\text{-CF}_3$  (Fomblin Z) endgroups having molecular weights of 4000 and 50 000, respectively. Fomblin Zdol and Fomblin Z have the same random co-polymer structural backbone, but different endgroups. Comparing figure 1 to figures 3 and 4 one can see that the desorption features for Fomblin Zdol ( $\text{-CF}_2\text{CH}_2\text{OH}$ ) and Fomblin Z ( $\text{-CF}_3$ ) occur over the same temperature range of 600–750 K. Since their desorption peak temperatures are very similar, we conclude that the different endgroups have influence on the decomposition kinetics. The implication is that the decomposition mechanism is rate limited by the cleavage of bonds in the fluorinated ether backbone of the Fomblin molecule. This is consistent with the mechanism proposed by Kasai [17–20]. Based on his studies of perfluoropolyether decomposition on the surface of  $\text{Al}_2\text{O}_3$ , Kasai concluded that Fomblin decomposition occurs by an intramolecular disproportionation reaction in which a Lewis acid site on the  $\text{Al}_2\text{O}_3$  interacts with two oxygen atoms flanking a monomer  $\text{-CF}_2\text{-}$  unit within the Fomblin backbone. Furthermore, endgroups aside, the primary difference between the Fomblin Zdol examined in figure 1 and Fomblin Z in figures 3 and 4 is their chain length. The Fomblin Zdol has a MW of 4000 while the Fomblin Z has a much larger MW of 50 000. Since the de-

sorption features of these vastly different molecular weight Fomblins lie within the same temperature range, we can also conclude that the Fomblin decomposition kinetics are independent of chain length. This result is consistent with the observation of Helmick and Jones that the decomposition temperature of Fomblins is not affected by backbone chain length [14,15]. The implication of these results is that although molecular weight may be used to control lubricant viscosity and mobility on a disk surface it will not influence the decomposition lifetime.

The important parameter determining the kinetics of Fomblin decomposition is the activation barrier. Assuming that the initial coverages of Fomblin Zdol were the same on the disks, one can extract the activation barrier for decomposition,  $\Delta E_{\text{act}}$ , by examining TPD spectra obtained at different heating rates ( $\beta$ ). For a first-order process, the Redhead equation [21] can be written as

$$\ln\left(\frac{\beta}{T_m^2}\right) = \ln\left(\frac{\nu R}{\Delta E_{\text{act}}}\right) - \frac{\Delta E_{\text{act}}}{RT_m},$$

where  $R$  is the gas constant,  $\nu$  is the pre-exponent in the rate constant for decomposition, and  $T_m$  is the temperature at which the maximum decomposition or desorption rate is observed. It is quite apparent that the broad peaks observed in figures 1, 3, and 4 are not due to simple first-order processes with a single rate constant. In reality the surfaces of the a-CH<sub>x</sub> films are quite heterogeneous and there are many different types of surface sites at which decomposition can be occurring, each of which has a different  $\Delta E_{\text{act}}$ . Assuming that  $\Delta E_{\text{act}}$  is a function of the coverage,  $\theta$ , the value of  $\Delta E_{\text{act}}(\theta)$  can be determined by plotting  $\ln(\beta/T_\theta^2)$  versus  $1/T_\theta$ , where  $T_\theta$  is the temperature at which a specific fraction of the adsorbed lubricant remains on the surface. This plot yields a straight line with slope equal to  $-\Delta E_{\text{act}}(\theta)/R$ . The procedure used to calculate the activation barrier for onset of the Fomblin Zdol decomposition,  $\Delta E_{\text{act}}^0$ , is as follows. At each of the heating rates used we determined the temperatures at which 95, 90, 85, and 80% of the lubricant remained undecomposed on the surface. The amount of undecomposed lubricant at a given temperature was determined from the area under the desorption curve at higher temperatures. Equation (1) was used to calculate  $\Delta E_{\text{act}}(\theta)$  for each of these four coverages. These values were then extrapolated to 100% coverage as shown in the inset to figure 2 in order to determine the value of the activation barrier for the onset of the decomposition reaction,  $\Delta E_{\text{act}}^0 = 114 \pm 6$  kJ/mol.

#### 4. Conclusion

The thermal stability of Fomblin Z and Fomblin Zdol adsorbed on the amorphous carbon overcoats of hard disk me-

dia was investigated using Fomblin Z and Zdol with vastly different molecular weights (MW = 4000 and 50 000), and films of Fomblin Z with different thickness (20 and 60 Å). In all cases, Fomblin decomposition was found to occur over the temperature range between 600 and 750 K. The Fomblin decomposition kinetics on the a-CH<sub>x</sub> surface were insensitive to lubricant endgroup, lubricant molecular weight, and lubricant thickness. The activation barrier to the onset of the decomposition process was estimated to be  $\Delta E_{\text{act}}^0 = 114 \pm 6$  kJ/mol.

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#### References

- [1] C.M. Mate and A.M. Homola, in: *Micro/Nanotribology and its Applications*, ed. B. Bhushan (Kluwer Academic, Dordrecht, 1997) p. 647.
- [2] C.M. Mate, *Tribol. Lett.* 4 (1998) 119.
- [3] W.H. Gumprecht, *ASLE Transact.* 9 (1966) 24.
- [4] D. Sianesi, V. Zamboni, R. Fontanel and M. Binaghi, *Wear* 18 (1971) 85.
- [5] G. Marchionni, G. Ajroldi and G. Pezzin, *Eur. Polym. J.* 24 (1988) 1211.
- [6] G.H. Vurens and C.M. Mate, *Appl. Surf. Sci.* 59 (1992) 281.
- [7] J.-L. Lin, C.S. Bhatia and J.J.T. Yates, *J. Vac. Sci. Technol. A* 13 (1995) 163.
- [8] C.-Y. Chen, J. Wei, W. Fong, D.B. Bogy and C.S. Bhatia, *J. Tribol.* 122 (2000) 458.
- [9] J. Wei, W. Fong, D.B. Bogy and C.S. Bhatia, *Tribol. Lett.* 5 (1998) 203.
- [10] L. Cornaglia and A.J. Gellman, *J. Vac. Sci. Technol. A* 15 (1997) 2755.
- [11] L. Cornaglia, A.J. Gellman, S. Howe and S. Nadimpalli, in: *ASME/STLE Proceedings*, eds. S. Bhatia and A.K. Menon (1996) p. 38.
- [12] N. Shukla, A.J. Gellman and J. Gui, *Langmuir* 16 (2000) 6562.
- [13] K. Paserba, N. Shukla, A.J. Gellman, J. Gui and B. Marchon, *Langmuir* 15 (1999) 1709.
- [14] L.S. Helmick and W.R. Jones, Jr., *NASA Tech. Memor.* (1990) 102.
- [15] L.S. Helmick and W.R. Jones, Jr., *Lubr. Eng.* 50 (1994) 449.
- [16] C.-Y. Chen, D.B. Bogy and C.S. Bhatia, *J. Vac. Sci. Technol. A* 18 (2000) 1809.
- [17] P.H. Kasai, *Adv. Inform. Storage Systems* 4 (1992) 291.
- [18] P.H. Kasai, W.T. Tang and P. Wheeler, *Appl. Surf. Sci.* 51 (1991) 201.
- [19] P.H. Kasai and P. Wheeler, *App. Surf. Sci.* 52 (1991) 91.
- [20] P.H. Kasai, *Macromolecules* 25 (1992) 6791.
- [21] P.A. Redhead, *Vacuum* 12 (1962) 203.
- [22] C.-Y. Chen, W. Fong, D.B. Bogy and C.S. Bhatia, *Tribol. Lett.* 7 (1999) 1.