

Effect of the additive X-1P on the Tribological Performance and Migration Behavior of PFPE Lubricant at the Head-Disk Interface

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Abstract

In this report we study the effect of X-1P as an additive in PFPE lubricants using an ultra-high 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 Al_2O_3 -TiC slider. An optical surface analyzer (OSA) is also used to observe the lube migration behavior. We observe that X-1P passivates the head and prevents the catalytic reaction of the PFPE when used as an additive. In addition, X-1P also increases PEPE's mobility and, hence, improves the tribological performance at the head-disk interface.

Keywords: Tribochemistry; Hydrogenated carbon overcoat; Nitrogenated carbon overcoat; Friction and Wear; Lube migration; Lewis acid; Catalytic reaction, X-1P additive

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 on 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 are described, and data demonstrating the chemical reactions of the lubricant and carbon overcoat are also presented.

X-1P, a cyclic phosphazene lubricant, is a new non-polymer lubricant being developed for the disk drive industry [14]. X-1P compounds possess excellent lubricity with optimal reduction in friction and wear achieved when a combination of CF_3 and F substitution is employed. Consideration of pour point, oxidative stability, and lubricity data, as well as economical factors, led to X-1P, a derivative containing four meta- CF_3 and two para-F substituents, as the leading fluid candidate. Much of the early work with X-1P involved its use as a pure fluid coated by dip or gravity/drain coating processes, resulting in a thickness less than 1 nm. The advantage of X-1P in lubricant applications is its low vapor pressure, high thermal stability, and good solubility

in non-CFC (chlorofluorocarbon) solvents. X-1P was found to perform well on hard disks during contact start/stop (CSS) and stiction testing with a lubricant thickness of about 0.5 nm [15, 16]. During CSS tests, the friction coefficient increases substantially more for perfluorinated disks than for phosphazene disks. In addition, friction decreases with an increase of relative humidity from 40% to 80%. These results indicate that X-1P lubricant interacts with water from the environment, resulting in a lower surface energy and a lower viscosity/friction. Perettie et al. [17] also observed that X-1P significantly outperforms ZDOL in CSS testing under hot/wet (30°C/ 80 RH) conditions. One possible explanation for these results is ZDOL decomposition, specifically its lack of stability in the presence of Lewis acid surfaces such as Al₂O₃. It is speculated that X-1P maintains its structural integrity because of lower tribochemical reactivity with the surface.

However, the high viscosity of X-1P is an important issue in ambient conditions, which induces an initial transfer of the lube to the head during the initial increase in the stiction/friction [16]. Thus, it is very important to coat X-1P at less than 6Å if it is used as a single lubricant. The viscosity of X-1P is much higher than that of the AM or ZDOL lubricants at room temperature. To achieve a viscosity for the phosphazene lubricant system corresponding to the viscosity of AM or ZDOL, it may be necessary to mix the X-1P lubricant with these low viscosity PFPE lubricants. This essentially eliminates the need for the ultra thin layer required for X-1P to be used as a single lubricant but enhances the performance of the head-disk interface. There are two issues to be addressed here: (1) does X-1P perform as well in a mixture as it does “neat” and is the lubricating mechanism the same for both, and (2) when used with PFPEs, does X-1P help the performance and why?

In this report we study the effect of adding X-1P in PFPE lubricant using an ultra-high 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 $\text{Al}_2\text{O}_3\text{-TiC}$ slider. The optical surface analyzer (OSA) is also used to observe the lube migration behavior. We observe that X-1P passivates the head and prevents catalytic reaction of the PFPE when used as an additive. In addition, X-1P also increases the PEPE's mobility and, hence, improves the tribological performance at the head-disk interface because a faster lubricant replenishment rate into the test track to constantly provide lube to be decomposed.

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 $< 2 \times 10^{-8}$ 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 $\text{Al}_2\text{O}_3/\text{TiC}$ 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 2×10^{-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 $\text{Al}_2\text{O}_3/\text{TiC}$ sliders with and without amorphous diamond like carbon (DLC) films on the air bearing surfaces. The disks were commercial 95mm super smooth thin film disks. The disks were lubricated with ZDOL or AM 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. The resulting thickness of PFPE on the disks in this study was 12Å.

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 [19]. 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 780 nm. 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. Figure 1 shows a schematic diagram of the OSA, and more information about the principle of operation can be found in Meeks's paper [19].

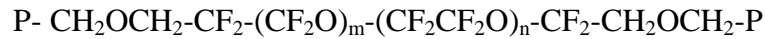
III. Results and discussion

A. Results from the AM3001/CNx samples

In this section, we present the results from the UHV drag tests and OSA measurements of the AM3001/CNx samples. The disks were commercial 95mm very smooth thin film disks with a 105Å amorphous nitrogenated carbon overcoat (CNx). The nitrogen content in the CNx film

was 14 atomic percent. The disks were lubricated with 12Å AM3001 with different X-1P additive percents. AM and X-1P lubricant are mutually soluble in CF (chlorofluorocarbon) solvents because they both have the phenyl ring in their chemical structure. The chemical formulas of AM3001 and X-1P are given below:

AM3001



where P is the piperonyl end group (-CH₂-phenyl=O₂=CH₂)

X-1P

Before starting the friction tests, the background intensities were recorded for 80 seconds by the mass spectrometer. Figure 2 shows the friction coefficient curves for the 12Å AM3001 lubricated disks without any X-1P additive. For the 70Å DLC-coated Al₂O₃/TiC slider, the friction coefficient started at 0.9 and increased steadily to 1.7 within 65 drag cycles before wear occurred. For the uncoated Al₂O₃/TiC slider, the friction coefficient increased to a peak value of 1.3 immediately and 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 Fig. 2, 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 AM3001 has little lubricating action

against both the uncoated and the DLC-coated $\text{Al}_2\text{O}_3/\text{TiC}$ sliders. The friction coefficient curves for the 12Å AM3001 lubricated disks with two different X-1P additive percentages are similar to Fig. 2, and they are shown in Figs. 3 and 4.

Figure 5 shows the OSA data: a plot of lube migration as a function of X-1P percentage. 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 5(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 5(b) shows the average migration rate of AM3001 lubricant versus X-1P percent. The rate is just the linear slope of the lubricant thickness versus time in Fig. 5(a). We observed that the lube mobility of AM3001 increases with increasing X-1P percent.

Figure 6 shows the wear durability of these CNx disks as a function of X-1P percentage. For both the 70Å DLC coated and uncoated $\text{Al}_2\text{O}_3/\text{TiC}$ sliders, the wear durability improved when the X-1P additive percentage increased. These results demonstrate the benefits of using X-1P additive to increase the mobility of AM3001 lubricant - a faster lubricant replenishment rate into the wear track provides more material to be decomposed so the carbon overcoat surface is protected for a longer duration against sliding.

Figure 7 shows the integrated mass spectra of AM3001 fragments produced at the head-disk interface. The four primary peaks are from mass fragments CFO (47), CF₂O (66), CF₃ (69), and C₂F₅ (119). In our previous studies of CH_x carbon overcoats paired with carbon-coated sliders [13], the primary decomposition mechanism of PFPEs 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 PFPEs, and it was characterized by the generation of CF₃ (69) and C₂F₅ (119). In order to eliminate the background effect spectrum the background level was subtracted from the integrated mass spectrum. This mass spectrum for the 70Å DLC coated Al₂O₃/TiC slider is similar to that of PFPE vapor when heated to 300°C as reported by Kasai et al. [5], where the primary decomposition peaks are masses 47 (CFO) and 66 (CF₂O) due to frictional heating [13]. Because no contact with Lewis acid is possible in this case, the AM3001 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 AM3001 are C-O-C and C-C, the AM molecule is expected to cleave preferentially at these locations. A radical mechanism 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 (CF₂O) are the primary fragments due to frictional heat action.

The decomposition mechanisms of PFPEs 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 PFPE 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. 5. In our study of CH_x and CN_x overcoats [22], we observed that the hydrogen evolution from CH_x overcoats initiates the lubricant catalytic decomposition with uncoated $\text{Al}_2\text{O}_3/\text{TiC}$ 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 acids described in our earlier work [22], which is a necessary component for catalytic decomposition of PFPE lube. On the other hand, in our study of CN_x films, lubricant catalytic decomposition was prevented due to less hydrogen evolution from the CN_x overcoat.

Figure 7 shows the degradation intensities of four major AM3001 fragments during UHV drag tests on CN_x disks with different percentages of X-1P. The degradation intensities of the major AM3001 fragments increased as the X-1P percent increased for both the uncoated and coated slider cases. We believe the role of the mobile AM3001 layer is to replenish the lube displaced during dragging. With a larger X-1P percent more AM lubricant reflows into the test track due to its faster migration rate. Thus, more material is decomposed, resulting in the stronger degradation intensity as shown in Fig. 6.

B. Results from the ZDOL2000/ CH_x samples

In this section, we present the results from the UHV drag tests of the ZDOL2000/ CH_x samples. The disks were commercial 95mm very smooth thin film disks with a 105\AA amorphous hydrogenated carbon overcoat (CH_x). The hydrogen content in the CH_x film was 30 atomic percent. The disks were lubricated with 12\AA ZDOL by a dipping process with/without 6% X-1P

additive. X-1P and ZDOL are mutually insoluble, requiring tight processing controls to optimize performance. The chemical formula for ZDOL is given below:

ZDOL

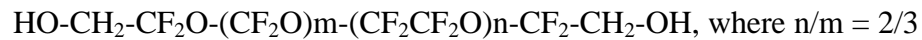


Figure 8 shows the wear durability of these CHx disks as a function of X-1P additive percentage. For both the DLC coated and uncoated sliders, the wear durability was significantly improved by adding X-1P into ZDOL. The enhanced wear durability may be attributed to two mechanisms: (1) X-1P improves PFPE's mobility as described in section A; (2) X-1P passivates the catalytic decomposition of PFPE. We will address the second mechanism here. Figure 9 shows the degradation intensities of four major ZDOL fragments during UHV drag tests of CHx disks with different X-1P percents. The degradation intensities of the major ZDOL fragments decreased with the addition of X-1P into ZDOL for both the uncoated and coated slider cases, most notably, the catalytic reaction associated with the uncoated slider has been prevented by using the X-1P additive. Kasai [23] also observed that X-1P passivates the catalytic activity of Al_2O_3 – the activity centers of Lewis acids Al_2O_3 are passivated by the nucleophilic (Lewis base) attack of the X-1P phosphazene ring. The usefulness of X-1P as an additive results from its ability to cover the catalytically active Al_2O_3 surface on the slider, thus preventing the disproportionation reaction [24].

Waltman et al. [25] also used a computer code to identify the reactive sites on the X-1P molecule. Their theoretical studies indicate that the strongest binding between X-1P and AlF_3

occurs with the endocyclic nitrogen and aluminum. The reason for the strong bonding between AlF_3 and the phenyl ring nitrogen atom originates from the polar character of the endocyclic P-N bond, which polarizes the nitrogen atom negatively. The binding energy of the P-N bond is of the order of -55 kcal/mol. The addition of X-1P into ZDOL lubricant significantly retards the degradation of ZDOL. Since the binding energies for the perfluorinated ethers are of the order of only -10 kcal/mol, the X-1P imparts protection to ZDOL by preferentially interacting, and therefore passivating the Lewis acid sites. The selective binding is based upon the strong binding energies that develop between X-1P and AlF_3 . Without the addition of X-1P into ZDOL, the intensities of the catalytic fragments CF_3 and C_2F_5 increased more than one order with uncoated Al_2O_3 sliders compared to the DLC coated sliders. In addition, more hydrogen evolution occurred with the CH_x films as compared to the CN_x films, which generates more fragments from the catalytic reactions.

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 and OSA data show that the X-1P additive increases the mobility of AM3001 lubricant - a faster lubricant replenishment rate into the wear track provides more material to be decomposed so the carbon overcoat surface is protected for a longer duration against sliding.

In addition, the catalytic reaction associated with uncoated $\text{Al}_2\text{O}_3/\text{TiC}$ sliders has been prevented by using X-1P as an additive in ZDOL. X-1P passivates the catalytic activity of Al_2O_3 – the activity centers (Lewis acid) of Al_2O_3 are passivated by the nucleophilic (Lewis base) attack of the X-1P phosphazene ring. The usefulness of X-1P as an additive in ZDOL results from its ability to cover the catalytically active Al_2O_3 surface on the head, thus preventing the catalytic decomposition of ZDOL lubricants.

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CAPTIONS

Fig.1: optical surface analyzer (OSA).

Fig. 2: friction coefficient of UHV drag test on 12Å pure AM3001 lubricated CNx disk.

Fig.3: friction coefficient of UHV drag tests on 12Å AM3001/2% X-1P lubricated CNx disk.

Fig.4: friction coefficient of UHV drag test on 12Å AM3001/4% X-1P lubricated CNx disk.

Fig.5: (a) the OSA data on the lubricant thickness versus time graphs; (b) the AM3001 lube migration rate versus X-1P additive %.

Fig.6: wear durability of CNx disks with different X-1P additive % into AM3001 during UHV drag tests.

Fig.7: integrated degradation intensities of four major AM fragments during UHV drag tests on CNx disks with different X-1P additive %.

Fig.8: wear durability of CHx disks with w/o X-1P additive into Z-DOL during UHV drag tests.

Fig.9: integrated degradation intensities of four major AM fragments during UHV drag tests on CHx disks w/o X-1P additive.

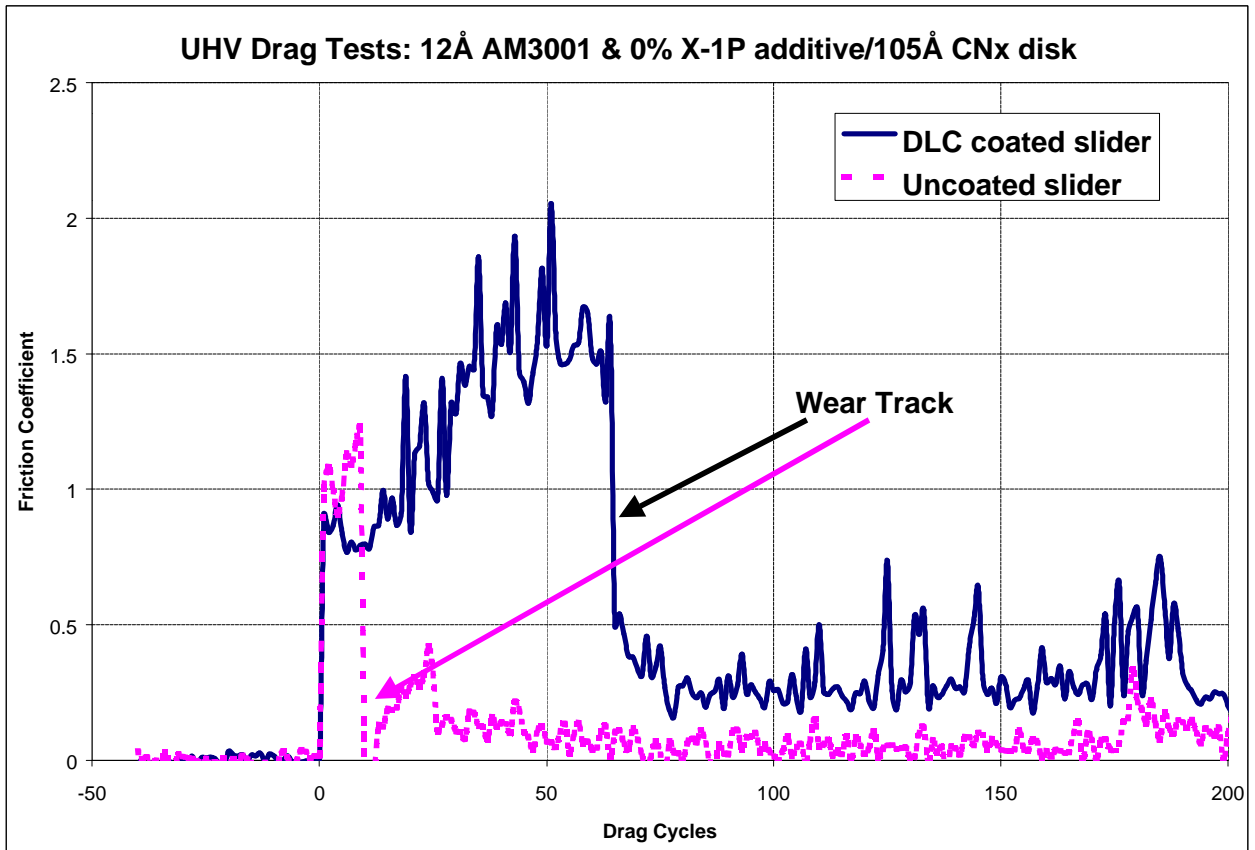


Fig. 2: Friction coefficient of 12Å pure AM3001 lubricated CNx disk in UHV drag tests.

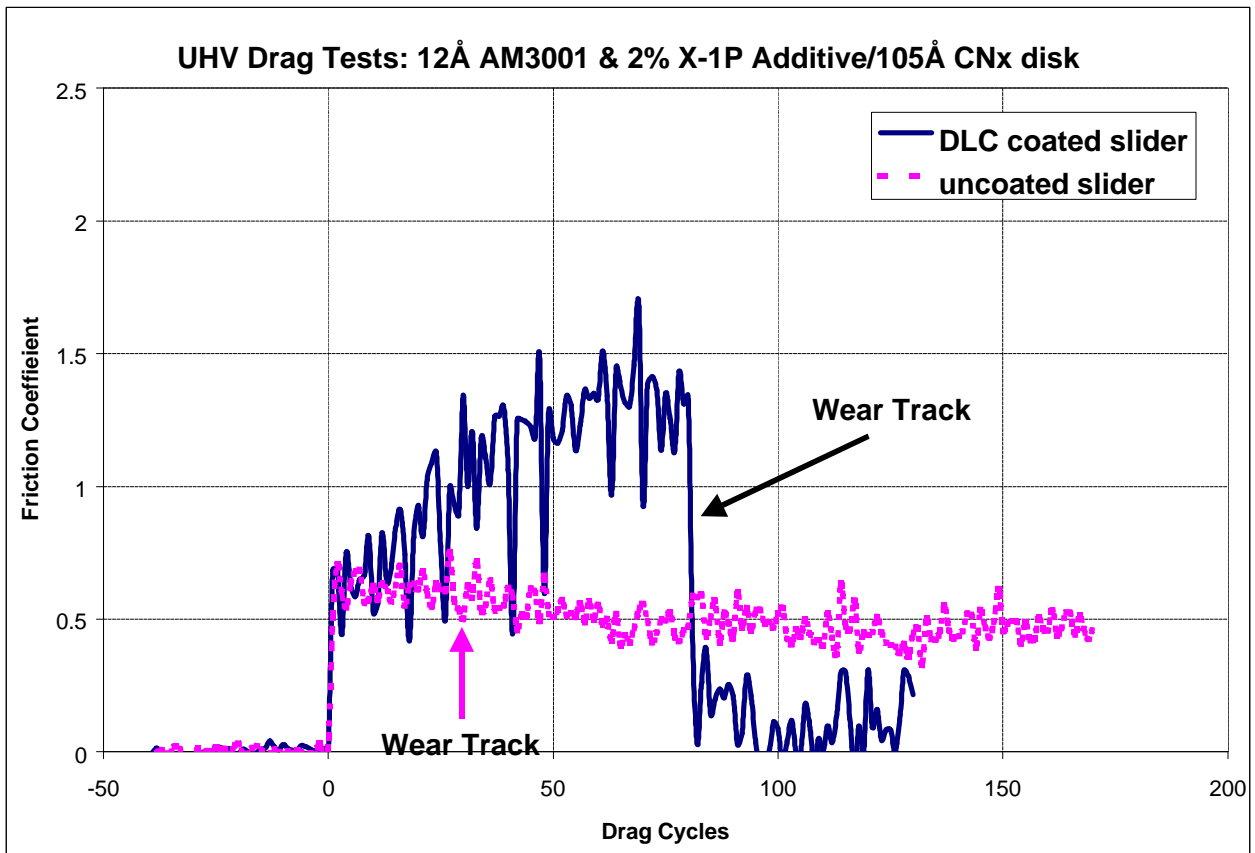


Fig.3: Friction coefficient of 12Å AM3001/2% X-1P lubricated CNx disk in UHV drag tests.

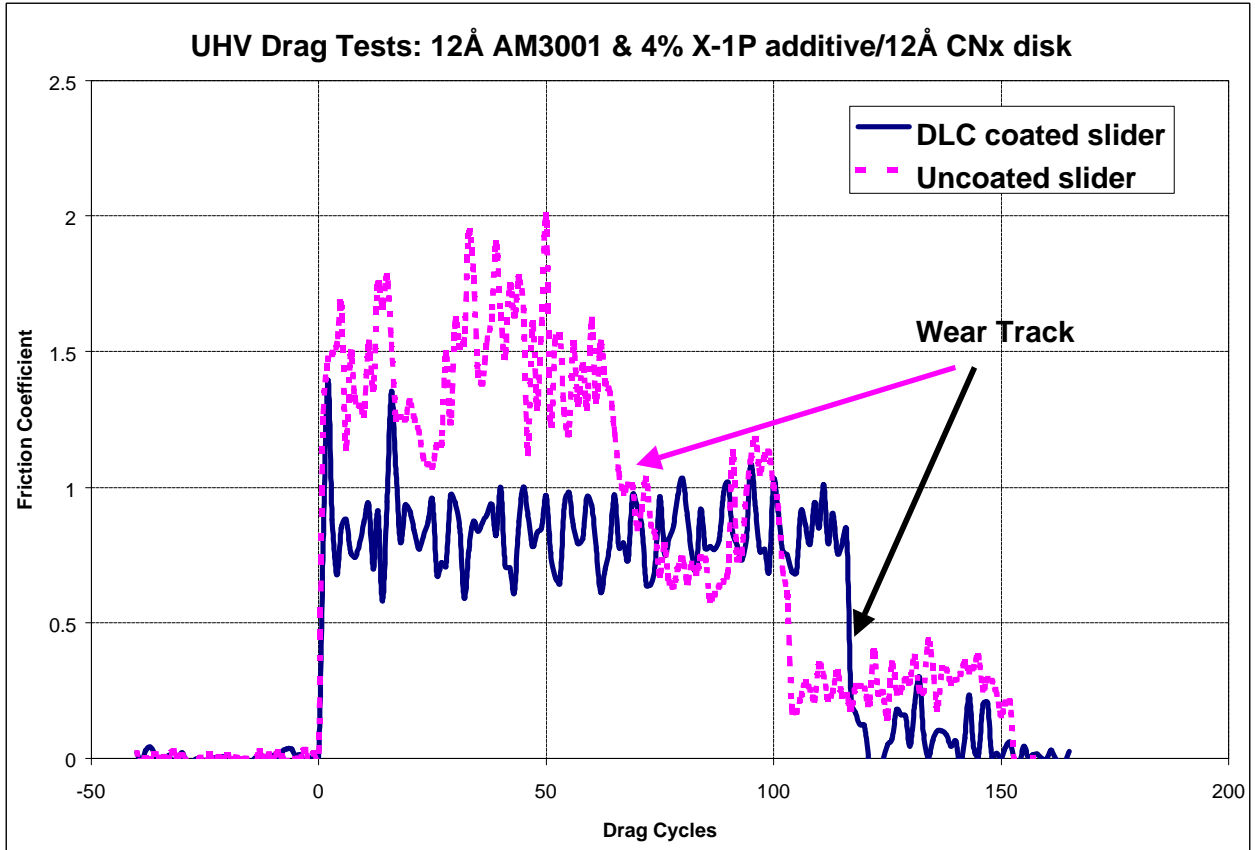


Fig.4: Friction coefficient of 12Å AM3001/4% X-1P lubricated CNx disk in UHV drag test.

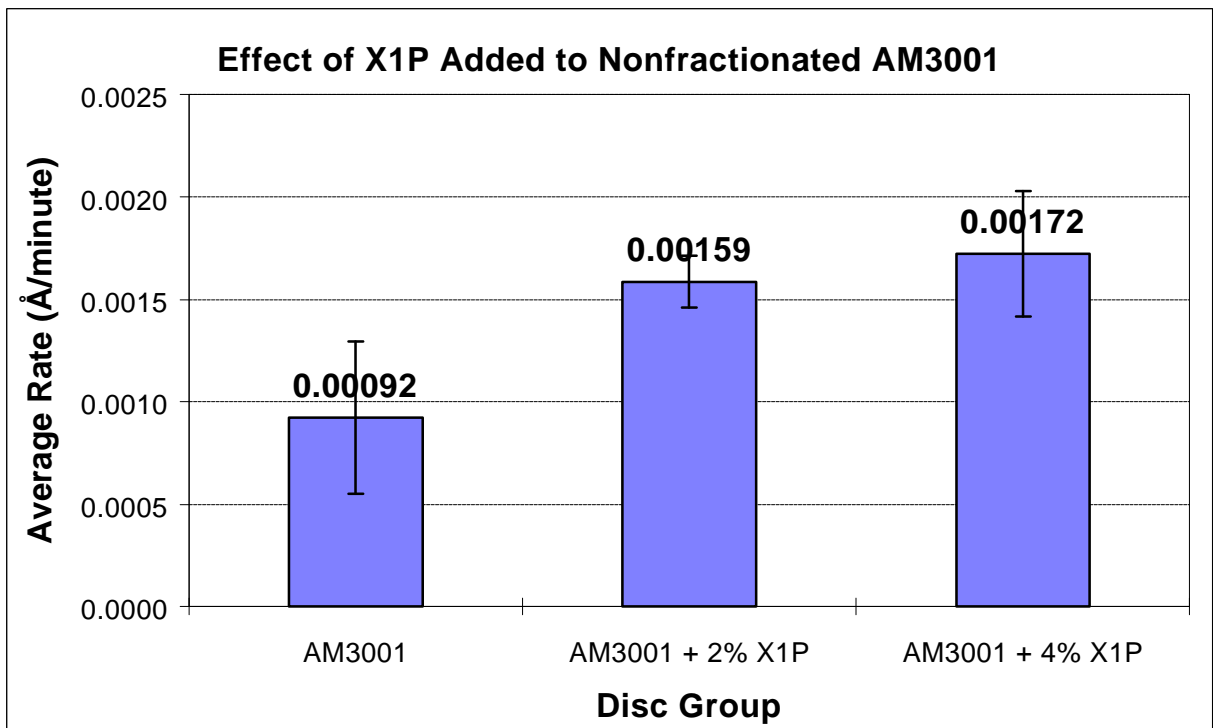
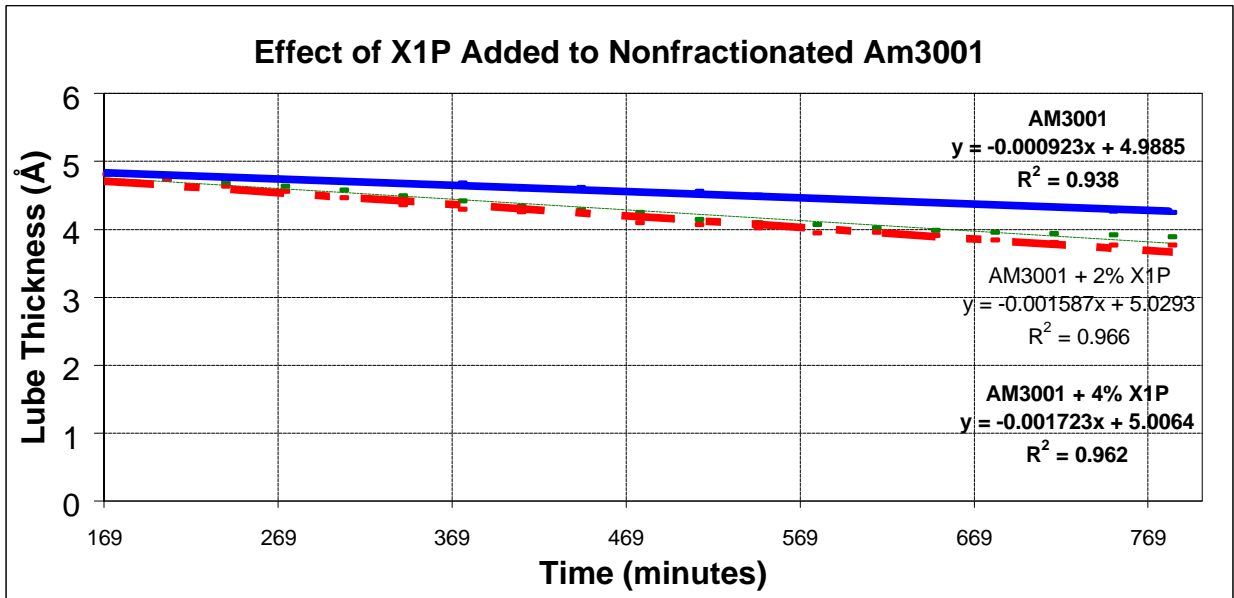


Fig.5: (a) lubricant thickness versus time graphs from OSA data; (b) the AM3001 lube migration rate versus X-1P additive %.

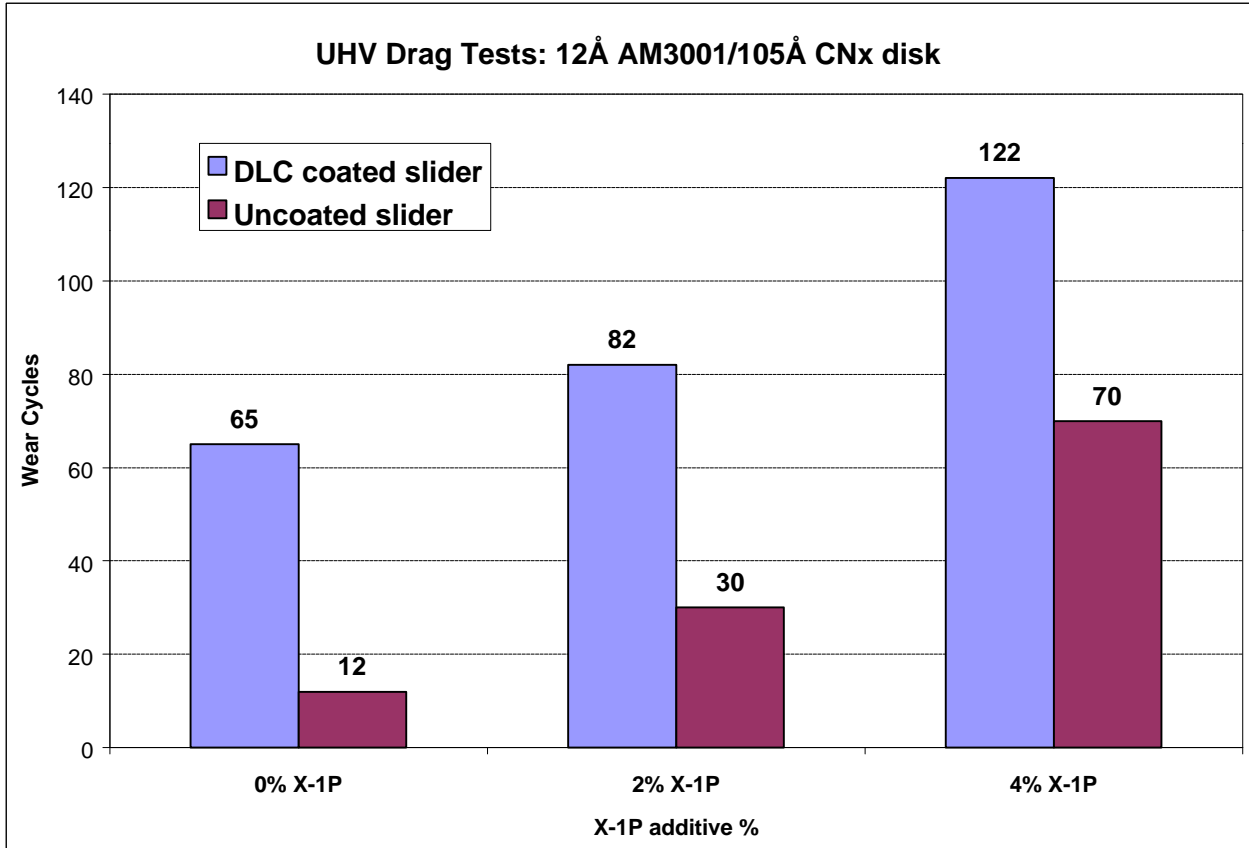


Fig.6: wear durability of CNx disks with different X-1P additive % in AM3001 in UHV drag tests.

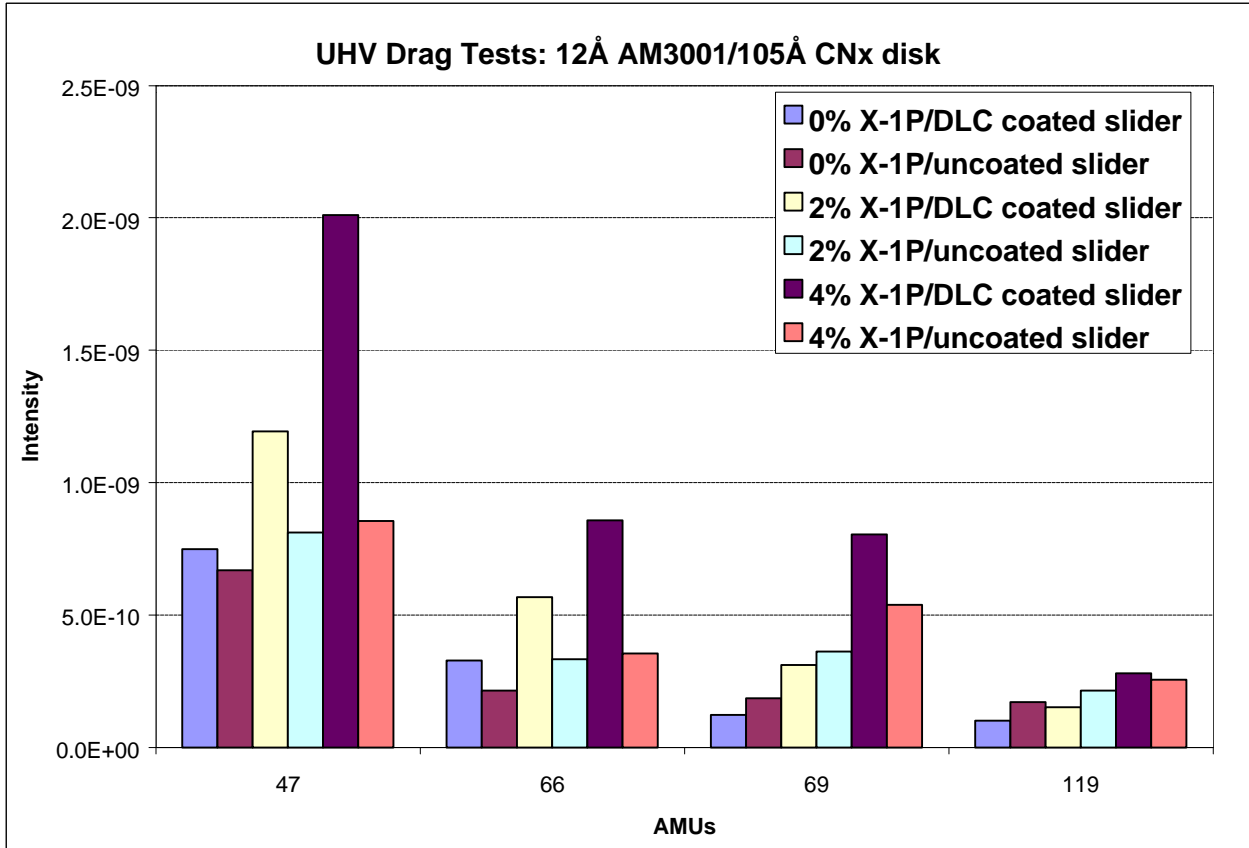


Fig.7: integrated degradation intensities of four major AM fragments during UHV drag tests on CNx disks with different X-1P additive %.

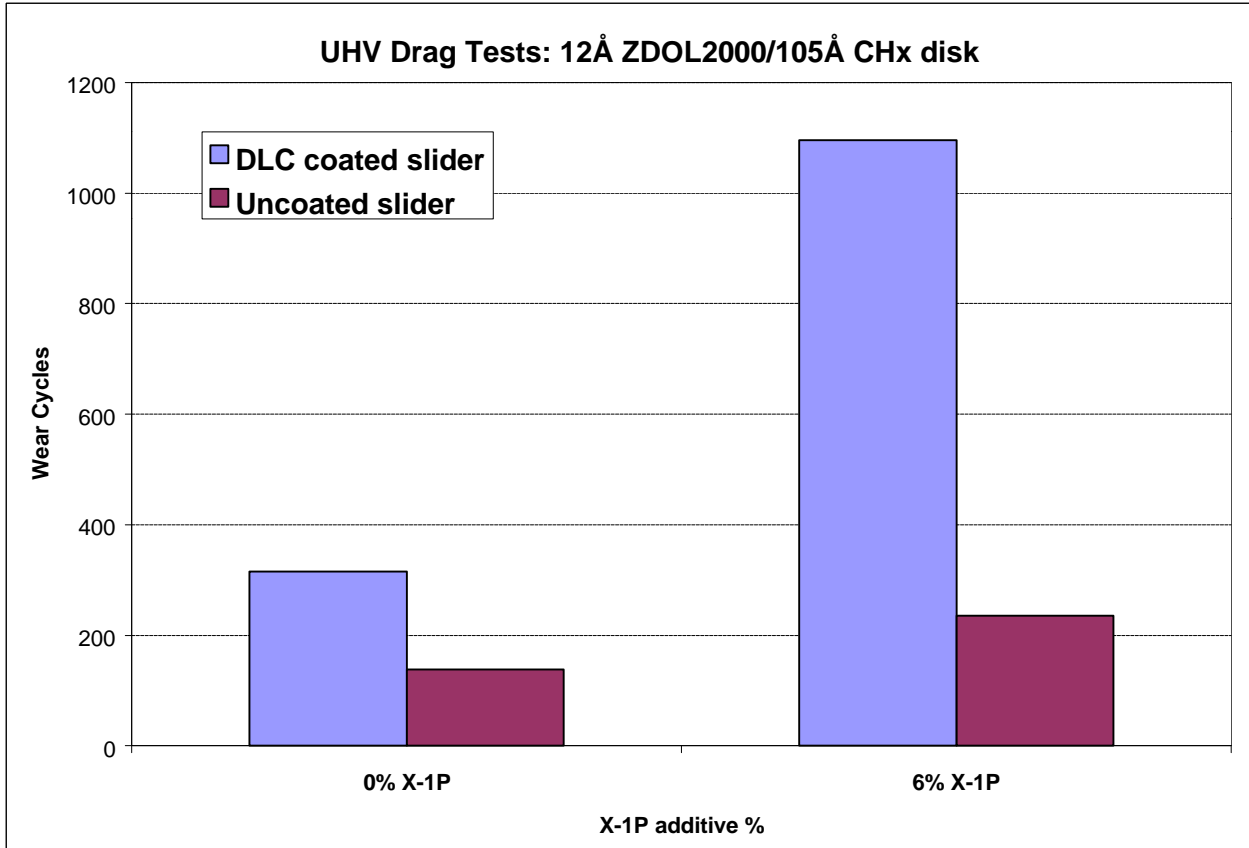


Fig.8: wear durability of ZDOL/CHx disks with X-1P additive in UHV drag tests.

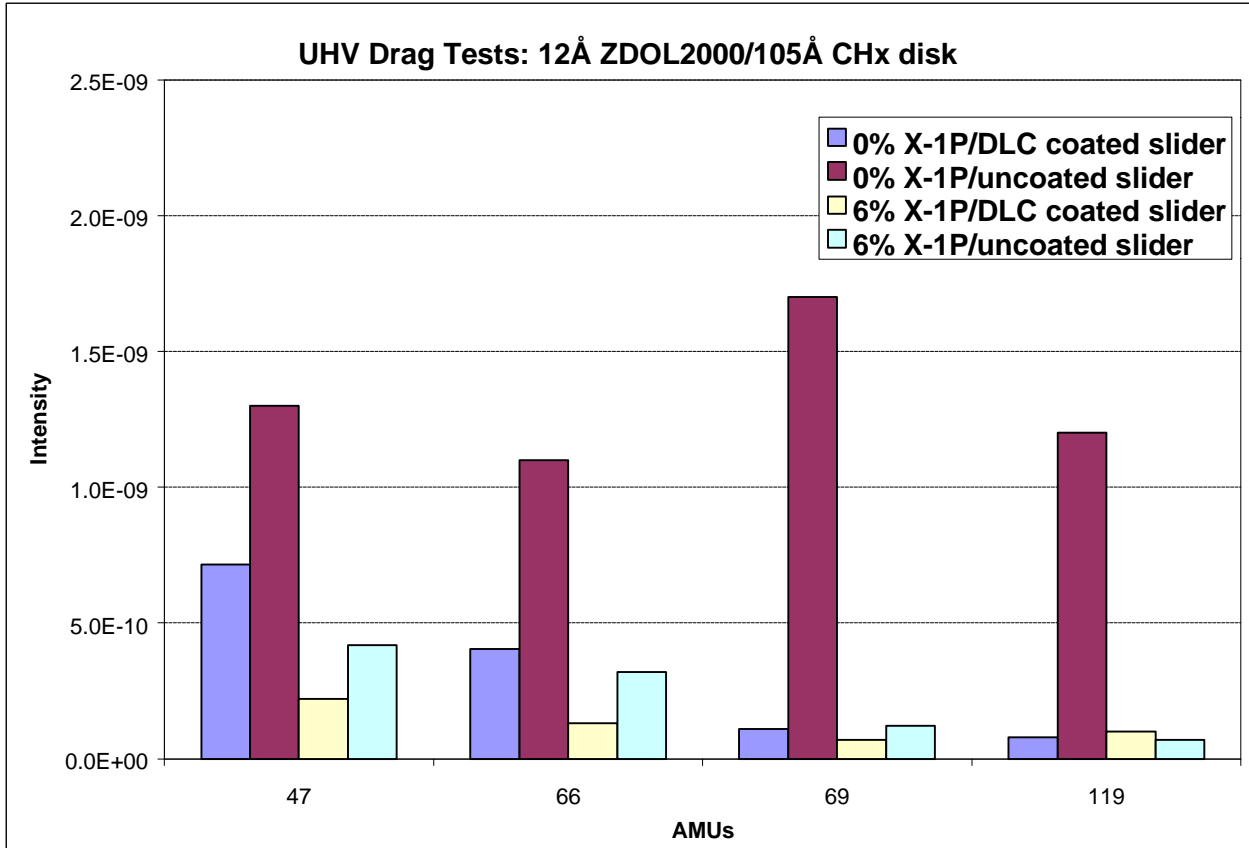


Fig.9: integrated degradation intensities of four major ZDOL fragments generated during UHV drag tests of CHx disks with X-1P additive.