STUDY OF CHEMICAL CONTAMINATION AT HEAD/DISK INTERFACE

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Summary

This thesis studies the chemical contaminations induced by hydrocarbon or organic siloxane at head/disk interface (HDI) in terms of stiction, fly stiction, head smear and disk wear.

The whole work can be divided into two parts. In the first part, we studied the effect of hydrocarbon. Three chemicals were selected for study: hexadecane, octadecane and DOP (dioctyl phthalate). CSS (Contact-Star-Stop) tests or continuous flying tests were conducted at component level in the presence of each chemical with Z-Dol/X1-P lubricated disks. The results showed that hydrocarbon could build up liquid-like film condensation at HDI and induce high stiction or fly stiction. A long rest period was normally required for the high stiction to occur and prevent the motor from spinning up. However, the presence of hydrocarbon did not necessarily result in interface failures during the test. Analysis of AE (Acoustic Emission) signal in frequency domain revealed that the slider’s flying characteristics played an important role and the intermittent contacts of the slider with disk media at the trailing edge was a key factor in causing interface failures. Head smear and disk wear were much dependent on lubricant pick-up/depletion. The lubricant picked up by sliders could be another kind of contamination and degraded the HDI performance, causing the interface to be very unstable. The contaminants were mainly distributed at three locations: the trailing edge, the leading edge and the etch cavity area. We noted that X1-P additive was picked up by
sliders and accumulated as large liquid droplets at the negative pressure pocket. The cause of this phenomenon is unclear.

In the second part, effects of organic siloxane at HDI were examined using Z-Dol coated disks with or without X1-P additive. Tests with both kinds of disks led to interface failures with well defined wear track on disk surface due to silica formation on slider surfaces. Chemical identification of head smears using TOF-SIMS revealed that siloxane was converted to abrasive silicon oxides through friction/contacts at contact areas. Detection of metal (Al, Ti) fluorides on slider surfaces by TOF-SIMS supports the view that decomposition of Z-Dol lubricant catalyzed by Lewis acid had occurred, which is supposed to be one prerequisite for the formation of silica at HDI. X1-P is believed to be able to neutralize Lewis acids and maintain the structure integrity of lube system. However, our results suggest that low amounts of X1-P additive (less than 1 wt%) in current commercial disks is not enough to suppress the formation of silica at HDI. Therefore, new types of lubricant system have to be developed in view of the need for achieving long term reliability and durability against siloxane contamination.

In all of our studies, an acoustic emission sensor was used instead of strain gauge to monitor and evaluate HDI performance. It was shown that AE measurements were sensitive and reliable techniques for head/disk interference studies.
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CHAPTER 1

Introduction

1.1 Disk Drive Storage Systems

The disk drive technology commonly referred to as Winchester technology utilizes the rigid disk as a non-removable stack in a drive. The disks rotate at a constant angular speed, with concentric data tracks recorded on their surfaces. Above each side of disks, there is a read-write transducer which moves across the disk surface to retrieve and store data. Hard drives store data on disks made of aluminum or glass and coated on both sides with a thin layer of magnetic film. Figure 1.1 shows a schematic of the cross-section of a typical thin film magnetic rigid disk. The substrate of the magnetic disk is usually made of aluminum-magnesium alloy or glass ceramic with a thickness of about 1 mm. A nickel-phosphorus layer of about 10 μm is electroless plated on the substrate. On the top of this is the magnetic medium of 30-50 nm thickness followed by a 10-30 nm carbon overcoat to prevent wear and corrosion. Lubricant, with thickness of 1-2 nm, is also applied on the carbon overcoat for further protection (Bhushan [1996]).
Magnetic recording in rigid disk drives uses high frequency signals coupled with high medium speeds, small access time and high data reliability. In principle, the read-write transducer consists of two separate elements called the read and write heads which write data to and read data from the disks. The write head is a simple thin film coil structure which emits out a tiny magnetic field when a current is passed through it. The magnetic field establishes the polarity of the area of the disk that passes under the write head at the precise time the current is sent through the coil. Since the disk is rotating these polarized areas or bits are organized along concentric, circular tracks. The read head is a thin film, metalized structure that exhibits the magnetoresistive (MR) effect. MR refers to the change in resistivity of metals in the presence of a magnetic field. A small current is sent through the MR head and the change of resistance is sensed when a polarity reversal associated with a bit passes under it. Cutting edge and future read heads include a giant magnetoresistive (GMR) head, dual spin valve head, and tunnel junction head. These advanced heads utilize new technologies to enhance the MR effect.
and thus the read head sensitivity (Mallinson [1993], Wang and Taratorin [1998]).

The read and write head are fabricated integral with a larger structure called the slider. The slider serves several important functions. The slider face flying on the disk, called the air bearing surface (ABS) is designed to generate an air bearing and maintain certain fly height. The slider fly height above the disk places the read-write heads in close proximity to the disk for reading and writing data. The slider is attached to a suspension which in turn is attached to the actuator. Rotation of the actuator arm about the pivot is driven by a voice coil motor (VCM) (Mee and Daniel [1990]).

Since the innovation of the first rotating rigid disk for digital data storage in 1957, efforts in the research and development of magnetic rigid disk drives have continually focused on increasing the storage capacity. This has been primarily achieved by an increase in the areal density which is defined by the number of bits in one track (linear density) and the number of tracks in a unit length (track density). In order to increase storage density, improvement in materials and recording techniques, miniaturization of the recording components, advances in media properties, narrow-track head design and accurate head-positioning technology are required. Application of advanced read/write elements including GMR heads, have allowed areal densities to exceed 60 Gb/in$^2$. Now the areal density is growing at 100% per year. Figure 1.2 (Grochowski and Halem [2003]) shows the growth of areal density through the last four decades and the estimated growth in the coming decade.
From the theory of magnetics, highest signal resolution and storage density can be achieved when the read-write transducer is in direct contact with the magnetic layer (Jiles [1998]). However, in current disk drives, reliability considerations prevent such an idealized scenario from occurring. The contact between the read-write head and the magnetic disk can generate wear at the head/disk interface (HDI). These wear particles in turn can cause damage of the magnetic layer, resulting in loss of the stored signals. Therefore, Reliable operation of a modern magnetic disk drive depends critically on maintaining a controlled spacing between the magnetic recording head and the recording medium. This is mainly achieved by positioning the magnetic transducers at the end of a
small ceramic 'slider' with a carefully designed air bearings that is self-pressurized by the airflow of the rapidly spinning disk (see Fig. 1.3). On the disk surface, thin protective overcoat and lubricant are applied to the magnetic layer to resist corrosion and prevent direct contact of the magnetic media.

Figure 1.3 Schematic diagram of head-disk interface

Therefore the spacing between the magnetic transducer and the magnetic layer is composed of the air bearing, the lubricant layer and the overcoat. To increase the areal density, efforts have been concentrated on minimizing the fly height of the slider, the thickness of the lubricant and the overcoat while at the same time maintaining a spacing large enough to sufficiently prevent damage on the disk files during the drive’s warranted lifetime. This makes the HDI become the most critical and sensitive part in a hard disk drive. As a result, great attention has been paid to the reliable design of HDI. This involves the slider air bearing surface design which defines the slider flying
characteristics, optimizing the disk roughness and waviness, the thin protective overcoats on disk and slider, and the molecularly thin lubrication scheme to both avoid wear and minimize static (stiction) and dynamic friction. The requirements for reliable low-flying, long-term durability and low stiction have resulted in designs aimed at optimizing the interface as an integrated system.

1.2 Chemical Contamination at Head/Disk Interface

The constant upward trend in storage density requirements for hard disk drives has been achieved largely through fly height reduction and the use of GMR heads. This means that the internal operating environment of the drives has to be very clean with respect to particulates, outgassed species, non-volatile residues (NVRs), and ionic levels, or disk drive failures may result. In today’s drives, the fly height is as low as almost 10 nm. And it is expected that in the future the fly height will continuously decrease to about 5 nm. With such a low fly height, the HDI is extremely sensitive to minute level of contamination. Moreover, a low fly height significantly increases the possibility of head/disk contact which may activate a tribochemical process at the HDI, which finally leads to hard disk drive failures. Statistics have shown that contamination-related failures are a significant fraction of all failures. In manufacturing failures, contamination-related failures account for 30.4% (Mee et al. [2002]).

Actually, since the early days of hard disk drive design the long-term durability of the drive components and the HDI has been compromised by the presence of contamination. Several factors in the evolution of disk drive design have contributed to ongoing
importance of chemical integration in the HDI. The use of organic liquids as disk surface lubricants added to the robustness of the interface by reducing the surface wear of the media during head to disk contact. However, failures of the interface may sometimes be the result of degradation of these organic liquids followed by their accumulation, along with other debris, on the air-bearing surface (Fowler and Geiss [2000]). With the advent of metallic thin film heads and, later, thin film media, corrosion at the HDI had been an ongoing concern to reliability engineers. The development of protective, thin, amorphous carbon overcoats has minimized corrosion as a major HDI problem. The relentless reduction of the slider’s fly height to increase areal density leads to the need to remove ever-smaller particulate contamination via high-efficiency filters has been extremely effective. Internal particle filters continuously clean the internal airflow, adding to the high reliability of today’s disk drives. Lower fly height demands smoother disks to minimize head/disk contact. The negative side to smoother disks is higher contact friction or “stiction” when the slider lands on the disk. Today, stiction is controlled by landing only on specialized laser bump textures and by limiting the lubricant thickness to about a single molecular layer. Nevertheless, stiction is aggravated by the presence of the disk lubricant and by condensable organic contamination. Under the meniscus force (Gao et al. [1998], Gui and Marchon [1995]), these liquids can flow into the space between a landed slider and the disk and cause stiction failure of the drive. In the presence of multilayer, molecular films on both sides of the HDI, the measured increase in stiction possibly results from entanglement of molecules (Ruths and Granick [1998]). Furthermore, miniaturization and cost reduction in the hard disk drive industry have caused the movement away from standard mechanical fastening techniques such as screws.
Nowadays, pressure-sensitive adhesives (PSAs) and plastic materials are widely used in current products and replacing metal parts. Outgassing of monomers, solvents and additives from these materials can affect the tribological performance of the HDI via many different mechanisms, e.g. stiction, head and disk smearing, head and disk wear, pole tip corrosion. As a result, outgassing has been another great concern to hard disk drive reliability engineers (Raman et al. [2000], Xu et al. [2003], Segar and Jesh [1999], Koka [1995], Jesh and Segar [1999]). However, with increasing spindle speeds come higher temperatures in operating disk drives. Higher temperature results in increased outgassing from drive components. For all these reasons, the impact of chemical contamination at the HDI continues to be an important topic.

The majority of the chemical contaminants normally fall into the following three categories: ionics, particulates and organics/inorganics. Sources of these contaminants have been found both in internal components and the external environment where the drives or components were assembled or operated (Dauber [1998]). The major contribution of ionic contamination with the presence of moisture in the disk drive is corrosion of the pole material in the head and the media. The sources of ionic contamination are varied from the drive manufacturing process. Some sources of ionic contaminates: latex gloves, finger cots, cotton Q-tips, foam swabs, particulates, packaging materials, vapor or aerosols and the presence of humans in the manufacturing environment. Media, at times, has been found to contain sulfates primarily on the carbon layer. Additional sources of ionic contamination are from ionic surfactants that may contain sulfates or phosphates that may still remain on the parts. The end effect of ionic contamination is corrosion of the pole tip and the media that yields a failed drive
(Liew et al. [2003]). In the case of the phosphate containing surfactants the glass deposition on the head can be etched away exposing the poles. Particulates are separated into two groups, aerosols and solid particulates. Aerosols could be mists from oils, condensed volatiles from other organic materials and may deposit on the media surface causing stiction failure. Solid particulates may be from organic inorganic, metallic or magnetic materials. Examples of solid organic and inorganic particulates are powder from gloves, finger cots, make-up and human debris. Particulate residues embedded in media or sliders from lapping slurries used in processing are potential sources of HDI failure. Metallic particulates from stainless steel components and magnetic particulates when present will cause drive failure. Particulates that become free within the drive may cause the head to crash or fly erratically and lead to a drive failure. As for organics/inorganics, it can be further sub-divided into volatile and non-volatile residues. Volatile components from gaskets or adhesives or other sources which outgas may condense upon the media surface causing problems such as stiction, head smear, or corrosive attack of pole tip surfaces (Koka [1995], Segar and Jesh [1999], Smallen and Mee [1999], Jesh and Segar [1999]). Compounds which may be NVR sources can come from uncured adhesive components, mold release agents (polydimethyl-siloxanes), plasticizers (DOP), oils from motors and bearings, and monomers from tray materials. Almost all of the examples can form a film on the media and cause a stiction problem resulting in drive failure (IDEMA Standards [1998]).
1.3 Research Objectives

This research project will focus on organic chemical contamination at HDI. Ionic and particulate contamination will not be covered in this study. Several chemicals are selected as the research objects from two compound groups: hydrocarbon and siloxane which are commonly released from drive component outgassing (refer to Appendix B). The objectives of this research project are characterized in the following four areas:

(1) Perform reliability and durability tests at component level in the presence of chemical contamination. Do failure analysis on failed samples and study the failure modes and failure mechanisms.

(2) Investigate and evaluate the detrimental effects of different chemical contaminations on HDI.

(3) Use acoustic emission sensor instead of strain gauge to monitor the HDI performance. Develop new and reliable techniques for HDI performance analysis and evaluation.

(4) Contribute or lend support to the improvement of HDI design.

1.4 Dissertation Structure

This thesis contains 5 chapters. Chapter One comprises a brief introduction to the magnetic disk drive and its operations. An overview of the necessity and importance of the study of chemical contamination at HDI is discussed. Chapter Two describes the type of equipments used for the analysis done in this project and the principles and operations
behind the equipments. Chapter Three presents the studies of hydrocarbon contamination at HDI. Experimental approaches, results, analysis and discussion are also included. Chapter Four describes the results and discussions of another experiment and investigates the effect of organic siloxane on HDI performance. Chapter Five gives the conclusion.
CHAPTER 2

Analysis Techniques

In order to investigate the mechanisms of failures due to chemical contamination at HDI, we normally have to characterize the failure samples using various analysis techniques. Some of these techniques were utilized in this study and will be introduced here.

2.1 Optical Microscopy

A simple magnifying system uses a single lens unit to form an enlarged image of an object for viewing or projecting. When a magnification greater than that obtainable with a simple magnifier (30X to 40X) is desired, a compound magnifying system or an optical microscope is used. In this system, magnification takes place in two stages. The immediate image formed by a first lens (objective) is further enlarged by a second lens (eyepiece). The total magnification is the product of the magnification of the objective and the eyepiece lens. The object to be examined is placed just beyond the first focal point of the eyepiece that forms a virtual image. Microscope lenses are of relatively short focal lengths. The shorter the focal length, the greater will be the magnification at a given image distance. In a microscope, a high two-stage enlargement is attained over a relatively short optical path with such-focus lenses. The magnifying power of an objective is classified as 1x, 5x, 10x, and 20x, up to 200x. The magnifying power of the eyepiece
could be 5x, 10x or higher up to 30x. Optical microscopy is a very effective tool in tribological studies that can be used to identify surface topographies. These include localized defects, wear debris and corrosion spots.

2.2 Optical Surface Analyzer (OSA)

Figure 2.1 shows the working principle of OSA. An OSA uses P- and S-polarized light to measure thickness changes in both, lubricant and carbon layer of a thin-film disk. Polarized light passes through a lens and is focused into a 3µm by 6 µm beam on the disk surface. Polarized light interacting with the disk surface results in a combination of absorption, reflection, and scattering. The amount of reflected and scattered light is measured using two photodetectors: P/S scattered (with an integrating sphere) and P/S specular. OSA (Meeks et al. [1995]) is designed in such a way that S- and P- polarized light reflectivity will vary in different ways as a function of thickness of disk lubricant and carbon overcoat. Thinning of the lubricant increases the intensity of reflected s-polarized light, but decreases the intensity of reflected P-polarized light. The opposite is true for an increase of lubricant thickness. Both, S- and P- reflected light intensities increase in the case of carbon film thinning. Wear particle formation or surface roughness increase leads to a decrease in both, S- and P- polarized light specular components due to increased light scattering.
This is summarized in Fig. 2.2 called the 2-D histogram. The data in the first quadrant correspond to the complete wear of the lubricant plus some wear of the carbon overcoat. Data in the second quadrant correspond to lubricant thinning. Wear and contamination particles and roughness increase can be detected in the third quadrant, and lubricant pooling will generate data points in the fourth quadrant. The central part of the 2-D histogram consists of data points, shown as a circle in Fig. 2.2, with values within the noise level of the system. More distant points mean stronger signals and allow more accurate interpretation (Khurshudov et al. [1999]). A special software (Candela Instruments) enables trace-back of data-points on the 2-D histogram to their original locations on the S- and P- images and to find out the exact location of wear particles or areas of carbon wear and lubricant migration on the disk surface. Data from both S- and P- images can be utilized simultaneously to interpret the OSA images.
2.3 X-ray Photoelectron Spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA) is a technique used to study the surface chemical structures of inorganic and organic materials. Surface analysis by XPS is normally done by irradiating a sample in a vacuum with mono-energetic soft x-rays and analyzing the energy of detected electrons. Mg $K\alpha$ (1253.6eV), Al $K\alpha$ (1486.6eV), or monochromatic Al $K\alpha$ (1486.7eV) x-rays are usually used. These photons have limited penetrating power in a solid on the order of 1-10 micrometers. When they bombard a material, the photons interact with atoms in the surface region, causing electrons to be emitted from the sample surface by photoelectric effect. These electrons may be photoelectron or Auger electrons. The kinetic energy distribution of the ejected electrons is analyzed by a high resolution
electrostatic spectrometer. Figure 2.3 shows the schematic emission process of XPS. The energy of the incident photon (x-ray) minus the kinetic energy of the photoelectron line yields its binding energy. The binding energy could be regarded as the energy difference between the initial and final states after the photoelectron has left the atom. It is proportional to the distance below the line indicating the Fermi level, which corresponds to zero binding energy.

Figure 2.3 Schematic diagram of XPS emission process

In most cases, the binding energies of the core electronic levels are uniquely different for each element so that the spectra can be easily used for surface contamination identification, or source chemical composition determination. All elements, except Hydrogen and Helium, are detectable. Small shifts in element binding energies are commonly referred to as the chemical shifts due to the modification of the electronic levels caused by changes in the chemical environment of the atom of interest. From the binding energy and intensity of these shifts, the elemental identity, chemical state, and
quantity of an element are determined. The information XPS provides about surface layers or thin film structures is of value in many industrial applications including: polymer surface modification, catalysis, corrosion, adhesion, semiconductor and dielectric materials, electronics packaging, magnetic media, and thin film coatings used in a number of industries.

In addition, XPS goes beyond elemental analysis to provide information on chemical bonding. It can distinguish chemical arrangements such as silicon-to-silicon bonds from silicon-to-oxygen bonds. The following two figures are examples of XPS analysis. Figure 2.4(a) presents the wide scans of a lube and depicts the elemental information of the sample, whereas Fig. 2.4(b) gives the narrow scans around C1s indicating the chemical bonds at C1s.
Figure 2.4 (a) Wide scans of a lube and (b) Narrow scans at C1s

2.4 Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS)

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is a powerful tool for surface analysis with both high spatial and high mass resolution. It can provide sub-micron elemental, chemical and molecular characterization and imaging of solid surfaces and thin films for products such as semiconductors, hard disk drives, polymers, paint and other surface coatings. Manufacturing companies in the chemical, semiconductor and pharmaceutical industries may use this tool to improve product performance, conduct failure analysis and manage process control.

TOF-SIMS differs from Dynamic SIMS in that it can analyze the outermost one or two monolayers of a sample without etching the surface away. While D-SIMS provides primarily elemental information, TOF-SIMS analysis yields elemental and molecular data. It is ideal for organic or inorganic materials, and can be used to characterize both insulating and conductive samples.
With detection limits in the ppm to ppb range, shallow depth profiling capabilities and automated analysis, TOF-SIMS can be used to analyze surface contamination, trace impurities, thin films and delamination failures. It is also a valuable tool to investigate surface modification chemistry and catalyst surface composition.

TOF-SIMS uses a pulsed primary ion beam to desorb and ionize species from a sample surface. The resulting secondary ions are accelerated into a mass spectrometer, where they are mass analyzed by measuring their time-of-flight from the sample surface to the detector. Figures 2.5 and 2.6 depict how the secondary ion fragments are being created by TOF-SIMS and the working principle of TOF. An image is generated by rastering a finely focused beam across the sample surface. Due to the parallel detection nature of TOF-SIMS, the entire mass spectrum is acquired from every pixel in the image. The mass spectrum and the secondary ion images are then used to determine the composition and distribution of sample surface constituents.

Figure 2.5 Effect of the primary ion on the monolayer of the topmost surface
Figure 2.6 Working principle of TOF. Pulsed primary ion beam impacts the sample surface to desorb and ionize the species. This results in secondary ions ejected from the fragmentation area and accelerated into a mass spectrometer. The spectrometer analyzes and differentiates these secondary ions by measuring their time-of-flight from the sample surface to the detector.

TOF-SIMS provides spectroscopy for characterization of chemical composition, imaging for determining the distribution of chemical species, and depth profiling for thin film characterization.

In the spectroscopy and imaging modes, only the outermost (1-2) atomic layers of the sample are analyzed. To ensure the analyzed secondary ions originate from the outer surface of the sample, a primary ion dose of less than $10^{12}$ ions/cm$^2$ is employed. Below this "static limit," roughly less than one in one thousand surface atoms or molecules are struck by a primary ion. The actual desorption of material from the surface is caused by a "collision cascade" which is initiated by the primary ion impacting the surface. The emitted secondary ions are extracted into the TOF analyzer by applying a potential between the sample surface and the mass analyzer. TOF-SIMS spectra are generated using a pulsed primary ion source (very short pulses of $<1$ ns). Secondary ions travel
through the TOF analyzer with different velocities, depending on their mass to charge ratio \((ke=\frac{1}{2}mv^2)\). For each primary ion pulse, a full mass spectrum is obtained by measuring the arrival times of the secondary ions at the detector and performing a simple time to mass conversion. Meanwhile, chemical images are generated by collecting a mass spectrum at every pixel (256 x 256) as the primary ion beam is rastered across the sample surface. Figure 2.7 shows the samples of mass spectrum and chemical images taken by TOF-SIMS from a corroded disk media.

Figure 2.7 Spectrum and chemical images taken by TOF-SIMS from a corroded disk media. The above mass spectrum shows the counts of different fragment ions at the analyzed area. The distinct peaks observed at masses 52 and 59 indicate that \(\text{Cr}^+\) and \(\text{Co}^+\) definitely exist on the disk surface. Since \(\text{Cr}\) and \(\text{Co}\) are magnetic materials and should be in magnetic layer below disk
surface, the findings of these materials on disk surface mean that the disk media is already corroded. The two ion mapping images present the distribution of Cr$^+$ and Co$^+$ at the analyzed area. The brightness is proportional to the ion count.
CHAPTER 3

Effect of Hydrocarbon at Head/disk Interface

3.1 Introduction

It is well known that the high static frictional force required to initiate sliding at the HDI, commonly referred to as stiction, is mechanically detrimental to the magnetic HDI and has been one of the dominant failure mechanisms in today’s magnetic disk drives (Gao and Bhushan [1995], Bhushan [1996]). This is because the allowable margin for stiction is very small with smooth thin-film disks, i.e., about 0.1 N or less per HDI and primarily due to the small, low-power consuming motors in these drives. The stiction force is mainly due to meniscus formation at the HDI. Lubricant, adsorbed water, hydrocarbon contamination or other outgassing organic materials all contribute to meniscus formation (Bhushan and Dugger [1990], Gao et al. [1995], Gao et al. [1998]). The meniscus force is equivalent to an applied normal force, causing an additional friction force at the HDI. The excessive meniscus formation can cause high stiction force and its control is so critical that most magnetic disk surfaces have been purposely textured (roughened) either mechanically or using a laser to reduce the meniscus force with the compromise of wear durability (Gao and Bhushan [1995], Baumgart et al. [1995]). Generally speaking, menisci are formed around the contact and near-contact spots at the interface resulting in adhesive forces between the two surfaces. Depending on the
mobility of the accumulated liquid materials to form menisci at the HDI, a period of resting time (equivalent to a power off in a hard disk drive) of the head on the disk surface may be required for high stiction to occur.

Since the introduction of smooth thin-film disks in drives, it has been realized that organic contaminants can induce large stiction and wear at the interface, and there have been a few papers on this topic during the past years. Smallen et al [1990] found that there is a good relationship between the organic contaminant thickness on disks from drives and the stiction forces measured in the drives. Most of the contaminants they found on the disk were dioctylphthalate (DOP). Yamamoto et al. [1990] studied the influence of DOP and octamethyl cyclotetrasiloxane on a head flying on a particulate media disk. Volpe et al. [1992] demonstrated an interesting technique to study the effects of volatile contaminants on head/disk stiction and used DOP and trimethylpentadacane as the contaminants. In the latter two studies, head/disk interaction and large stiction forces could be measured only after complete condensation of the organic vapors occurred on the disks. Koka [1995] studied the effect of diethylhexylphthalate (DEHP) on the HDI and found that DEHP significantly increased the stiction during CSS tests. However, it did not introduce any smears or debris on the heads, and did not cause visible wear tracks to appear on the disks.

Not until recent years, the fly stiction phenomenon was reported. Fly stiction is a special type of HDI stiction that occurs only after a head has flown over a disk for an extended period of time. During flying, liquid materials transfer to the HDI and cause high stiction. When high stiction occurs, not only severe mechanical damage to the HDI may result due to excessive adhesive force at the drive start, but also magnetic signal
degradation during magnetic read-write may occur due to excessive material pick-up on
the magnetic head. Experimental and theoretical studies demonstrated that fly stiction
values, unlike the normal stiction values, exhibit a unique bi-/tri-model distribution (Gui
and Marchon [1998]). Gao et al. [1999] quantitatively studied the correlation between
lubricant build-up on the slider surface and the fly stiction performance. An exponential
dependence of the fly stiction force on the lubricant pick-up was found. Raman et al.
[1999] performed component level investigation of liquid accumulation on sliders. They
discussed the physical origins of fly stiction and the processes that lead to high stiction.
Zhao and Bhushan [2000] conducted long-sweep/flying tests with three different kinds of
sliders at various environmental humidities and studied the mechanism of fly stiction.

Typically, Contact-Start-Stop (CSS) test is used to assess the tribological
performance of a head/disk system. It is common to set a critical value for the stiction
force measured by strain gauge sensors during the CSS test to indicate failure of the
interface. As the recording densities of magnetic hard disk drives continue to increase, the
fly height of the slider reduces to around 10nm. This causes some potential durability and
contamination problems. One requires an in situ tool to monitor the interactions at the
head/disk interface in real time. This may include events like carbon micro-wear,
lubricant depletion and degradation, head slip and asperity contact, and slider pick-up and
drop-off lubricants or debris. However, the traditional strain gauge sensor has lost its
sensitivity towards these events not only because of limited bandwidth (up to 5k Hz), but
also because the strain gauge measures the average displacement of a strain arm caused
by the shear force at the HDI during a rotational movement. On the other hand, the AE
technique provides higher bandwidth response (≥ 1 MHz). Also, this technique can more
accurately detect contact events at the head/disk interface because of the acoustic waves generated in a solid body due to high velocity pseudo-impact collisions of head-disk-asperities. Each contact event results in a geometrical distortion of the slider, hence generation of an acoustic wave (Jiaa et al. [1997]). In this study, we studied the effect of hydrocarbon on HDI using an acoustic emission sensor. CSS tests and continuous-flying tests were performed in the presence of hydrocarbon and AE rms signal was recorded for analysis. The interface failures were investigated by correlating the AE response to the basic mechanisms of contacts events at the interface. The slider and disk samples were examined by optical microscope and OSA after the CSS tests or continuous-flying tests.

3.2 Experimental

Figure 3.1 shows the experimental setup for this study. All tests were performed in a single disk component tester which was enclosed in a glass chamber. The tester consists of a computer controlled spindle on which the disk is clamped. The slider-suspension assembly is attached to a load beam supported by a XZ two-dimension translation stage. The position of the slider on the disk surface is controlled by adjusting the micrometers of the translation stage. A mini acoustic emission sensor is attached to the upper surface of the load beam to generate AE signal during the tests; hence, the HDI performance and the head-disk interaction can be monitored. The AE signal is pre-amplified and high band pass filtered. The filtered signal is further amplified and then sampled by a DAQ card manufactured by National Instruments at a sampling frequency of 2500Hz. The AE rms
values of the raw signal are programmed to be calculated with a time constant of 10ms and recorded accordingly.

![Schematic diagram of the experimental setup](image)

**Figure 3.1** Schematic diagram of the experimental setup

All the sliders used in this study were sub-ambient pressure tri-pad pico sliders with a fly height of 14 nm and a pre-load of 2.5g. These sliders had a diamond like carbon overcoat and a Si seedlayer on Al$_2$O$_3$-TiC substrate. Commercial magnetic recording disks with a diameter of 95mm were used in all the tests. The disk samples were overcoated with 15 nm nitrogenated carbon and had a top layer of Z-dol lubricant with X1-P additive of 1.0 nm. Between the radius of 16.5 mm and 19.5 mm the disk had a laser textured landing zone. The crater-shaped laser bumps were 10 µm in diameter and
10 nm in height and arranged in spiral pattern. Pictures of the sliders and the laser textured landing zone of the disks used in this study are shown in Fig. 3.2.

![Micrographs of (a) the sub-ambient pressure tri-pad slider and (b) the laser textured disk landing zone](image)

Figure 3.2 Micrographs of (a) the sub-ambient pressure tri-pad slider and (b) the laser textured disk landing zone

For all experiments except the control run, chemical vapor of hydrocarbon was continuously introduced into the test chamber. This was achieved by placing a known amount of hydrocarbon liquid in a flask heated by hot water, the temperature of which was kept at 80℃ by a recirculator. All tests in the presence of hydrocarbon usually started 2 hrs later after the beginning of introduction of hydrocarbon into the test chamber. All experiments were carried out at the ambient environment conditions without control of the temperature and relative humidity. However, both the temperature and the relative humidity were real-time monitored during each test. The temperature was around 24℃ and the relative humidity was in the range of 40–55%RH. All tests including the assembly of the tester components and materials were done in a clean room to minimize the effect of ambient contamination.
Three kinds of hydrocarbon were used in this study: hexadecane, octadecane and diocetylphthalate (DOP), which were commercially available from Merck Inc. Alkane is one kind of hydrocarbon that is ubiquitous in the outgassing of disk drive components. The outgassed alkane is mainly made up of compounds with 12 or more carbon atoms (Fowler and Geiss [2000]). Hence, we select hexadecane and octadecane. While, DOP was the main contaminant found on disks by Smallen et al. [1990]. The chemical and physical properties of these three chemicals are listed in Table 3.1.

Table 3.1 Chemical/physical properties of the tested chemicals

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Formula</th>
<th>Density, g/cm³</th>
<th>Melting point, °C</th>
<th>Boiling point, °C</th>
<th>Vapor pressure, mmHg @ 25°C</th>
<th>Vapor phase concentration @ 25°C, g/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexadecane</td>
<td>C₁₆H₃₄</td>
<td>0.77</td>
<td>18</td>
<td>287</td>
<td>0.003</td>
<td>0.036</td>
</tr>
<tr>
<td>Octadecane</td>
<td>C₁₈H₃₈</td>
<td>0.78</td>
<td>26-29</td>
<td>317</td>
<td>0.00007</td>
<td>0.00095</td>
</tr>
<tr>
<td>DOP</td>
<td>C₂₄H₃₈O₄</td>
<td>0.98</td>
<td>−50</td>
<td>385</td>
<td>0.0000026</td>
<td>0.000054</td>
</tr>
</tbody>
</table>

Three groups of tests, named Group A, B and C respectively, were conducted. In Group A, a 30K cycles CSS test was performed for each sample of head/disk combination. The CSS test was performed at disk contact landing zone and the slider was positioned at a radius of 18 mm. Each CSS cycle consisted of a 3 sec rapid on, a 3 sec hold at 5400 rpm and a 3 sec rapid off followed by a 3 sec rest. During the CSS test, the AE rms data profile of each CSS cycle was reported and the slider’s take-off peak value was also recorded in a separate data file. The take-off peak value here is referred to the maximum AE rms value or the AE rms amplitude recorded when the slider was taking
off. Group B also included several CSS tests. The only difference was that, in the middle of each CSS test (after 15K CSS cycles) the slider was parked at the landing zone and rested for 12 hrs before the next CSS cycle. Differently, Group C consisted of a group of continuous-flying tests. Before each continuous-flying test, a CSS circle was conducted at the landing zone and the AE rms profile was recorded and then the slider was manually moved to the OD of the disk at a radius of 40 mm and flew over the disk surface for up to 96 hrs. When the extended flying was finished, the spindle stopped spinning and the slider was manually moved back and parked at the landing zone. After a 12 hr rest, the slider ran another CSS test circle and the profile was also recorded. During the continuous flying, the AE rms response was recorded every 15s with a sampling time of 2s. The average value of these 5000 data points was reported as one data point.

The test conditions for all test samples in this study are listed in Table 3.2. Control runs were conducted in the absence of chemical contamination and repeated at least twice. The samples listed in either group (A, B, C) were run only once for each because we intended to study these chemicals as a group not by individual because they all belong to the same category - hydrocarbon. If we put all these tests together, they may form a test matrix. In this matrix, tests were performed 3 times for each chemical and also 3 times for each test routine no matter what condition it is. All the tests had some common outputs like AE rms signal and slider images (slider smear) to ensure the reproducibility of the experiments.
Table 3.2 Test conditions for all samples in this study

<table>
<thead>
<tr>
<th>Group</th>
<th>Sample #</th>
<th>Chemical</th>
<th>CSS cycles or continuous flying time</th>
<th>Fly height (nm)</th>
<th>Spinning speed (rpm)</th>
<th>Flying location (radius mm)</th>
<th>Park time before the test (hrs)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control run</td>
<td>1</td>
<td></td>
<td>53k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>Repeated twice</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>2</td>
<td>Hexadecane</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td>Without rest time in the middle of the CSS test</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>Octadecane</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>DOP</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>5</td>
<td>Hexadecane</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td>After 15k CSS cycles, the slider parked and rested for 12 hrs before the next cycle</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>Octadecane</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>DOP</td>
<td>30k</td>
<td>14</td>
<td>5400</td>
<td>18</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>8</td>
<td>Hexadecane</td>
<td>96 hrs</td>
<td>14</td>
<td>5400</td>
<td>40</td>
<td>2</td>
<td>One CSS cycle + 96 hrs continuous-flying + 12 hrs rest + another CSS cycle</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>Octadecane</td>
<td>96 hrs</td>
<td>14</td>
<td>5400</td>
<td>40</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>DOP</td>
<td>96 hrs</td>
<td>14</td>
<td>5400</td>
<td>40</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>

To minimize or eliminate the interference due to the chemical introduced in the previous tests, before each test, precautions were taken to completely clean out the tester and the enclosure with iso-propyl alcohol and subsequently thoroughly air-dried in an air stream.

After the CSS tests or the continuous-flying tests, the disk landing zone or the flying track was examined by OSA. The contamination patterns of the heads were observed by the optical microscope. The data from the AE transducer, OSA and contamination patterns provide insights of the head-disk interactions and the interface failure mechanisms.
3.3 Results and Discussion

3.3.1 CSS tests without rest period

3.3.1.1 CSS performance in the absence of hydrocarbon

Up to 53k CSS cycles were performed in the control runs in the absence of hydrocarbon. All the control runs showed similar results, which means our experiments were repeatable. Figure 3.3 presents an example of the similarity and reproducibility of those AE rms data profiles recorded for the 1st and 50kth CSS cycles in the control runs. From this figure we can see that there were only very small changes in the shape of AE profile even after the HDI had undergone 50k CSS cycles. The AE take-off peak values for all CSS cycles are plotted along CSS cycle number in Fig. 3.4. It was observed that the AE take-off peak values in the control run experiment were quite stable for such a long CSS test. The relative stabilities of the AE rms profiles and the peak values indicate that the interface had good CSS performance in the absence of hydrocarbon and under the cleanroom test environment. This view is also supported by the optical micrographs taken on the slider ABS shown in Fig. 3.5. It was observed that the slider surface was essentially clean and had only a little debris on the leading edge. The debris was supposed to be caused by particulate contamination from the test environment.
Figure 3.3 AE rms profiles for the (a) 1st and (b) 50kth CSS cycles recorded for sample #1

Figure 3.4 AE take-off peak value profile over the entire CSS test for sample #1
Figure 3.5 Optical micrographs of slider #1 after CSS test. (a): graph of the trailing edge region; (b) graph of the leading edge region.

3.3.1.2 CSS performance in the presence of hydrocarbon

Figure 3.6 shows the AE rms data profiles for the 1st, 5kth, 6kth and 30kth CSS cycles recorded for sample #2. These figures definitely display the degradation of CSS performance of the interface as drastic changes of the AE rms data profile for sample #2 were observed during the CSS test. In Fig. 3.6(a), the signal at the noise floor shows an increased level and higher fluctuation compared to that observed in the control run. This might be due to the high initial stiction caused by the introduction of hydrocarbon and indicates the unstable flyability of slider #2 even at the beginning of the CSS test. Moreover, we noted a small peak after 2 sec in the first slider take-off profile (indicated by an arrow in Fig. 3.6(a)) which was not observed in the control run. This small peak should be attributed to a hard collision between the tailing edge of the slider and the disk media. As the CSS cycle number increased, the amplitude of this peak also increased and additional peaks also came up at flying conditions, indicating that more contacts occurred
even at flying conditions. Meanwhile, the amplitudes of the slider take-off profiles also dropped quickly (see Fig. 3.6(b) and (c)). The AE take-off peak value profile is plotted in Fig. 3.7 along CSS cycle number. It is observed that the AE rms peak value generally decreased from around 2.5 to around 0.6 between cycle 3800 and cycle 5500, and after that began to climb up again slightly. All these observations suggest an interface failure presumably due to a large amount of organic liquid and debris picked up by slider and their accumulation on slider ABS. This view is supported by the micrographs of the slider ABS taken by optical microscope following the test. These graphs showed heavy accumulation of liquid material on the center pad of the slider as well as in the etch cavity region (Fig. 3.8). Although some debris is also observed attached at the leading edge of the slider, the liquid material was prominent over the trailing edge of the slider. An OSA image shown in Fig. 3.9(a) of the tested disk #2 reveals a wear track with severe lubricant depletion and degradation at the laser textured zone (the area between radius 16.5mm and radius 19.5mm). The width of the wear track is in good agreement with that of the smear on the center pad of the slider.
Figure 3.6 AE rms profiles for the (a) 1st, (b) 5000th, (c) 6000th and (d) 30000th CSS cycles recorded for sample #2

Figure 3.7 AE take-off peak value profile over the entire CSS test for sample #2
Figure 3.8 Optical micrographs of slider #2 after CSS test showing severe contamination on slider surfaces: (a) graph of the slider’s trailing edge region; (b) graph of the slider’s leading edge region; (c) magnified view of the pole tip area; (d) graph of the trailing center pad.
Figure 3.9 S- and P-polarized OSA images of the laser textured zone of (a) disk #2 and (b) disk #3. Carbon wear, lube pooling or debris smear are observed. The width of the images is 3mm (radius 16.5 ~ 19.5 mm). Disk spinning direction: from right to left.

Figure 3.10 and Fig. 3.11 give the AE rms results collected for sample #3 from similar experiment. A small peak after 2 sec was also detected in the AE profile plot for each CSS cycle. However, the amplitude of this peak decreased as the cycle number increased and almost disappeared at the end of the CSS tests. It can be deduced that there were also collisions between the trailing edge of the slider and the disk, but the intensity of this interaction was weak and decreased gradually. The AE take-off peak values were observed to increase slightly as the CSS cycle number increased. From the AE CSS profile plot, it is found that both the take-off and landing off AE signals at the end of the test were much noisier and stronger than those observed at the beginning. This might be due to debris and lubricant build up on the slider surface (Knigge and Talke [1999]).
The optical micrographs taken for slider #3 after the CSS test are shown in Fig. 3.12. The debris and lubricant build up on slider #3 is not so serious as that observed on slider #2 and the distributions are also different. The fact that only a small amount of organic liquid is observed at the trailing edge of the slider lends a hand to the above mentioned inference that weaker interaction occurred at the trailing edge of the slider with the disk.
media. However, more debris is found at the slider etch cavity area. These debris should be generated by wear at the head/disk contact area and then sucked into and trapped at the etch cavity area due to the existence of a negative pressure zone. Figure 3.9(b) presents the OSA image of the laser textured zone on disk #3. No wear track is visible, indicating less lubricant depletion. However, a dark line is notable on the S-polarized image. This line should be due to accumulation of contamination debris which dropped from slider surface and smeared the disk surface.

Figure 3.12 Optical micrographs of slider #3 after CSS test: (a) graph of the slider’s trailing edge region; (b) graph of the slider’s leading edge region.

The organic contaminant on both slider #2 and #3 was analyzed by X-ray Photoelectron Spectroscopy (XPS). The XPS results are shown in Fig. 3.13 and the six atomic elements that are most abundant in the contaminated area are listed in order. The results indicate that the contaminants on both sliders are a mixture of disk lubricant and hydrocarbon. However, the contaminant on slider #2 is much thicker and has a much higher percentage of disk lubricant, indicating that severe lubricant depletion and degradation occurred.
Figure 3.13 XPS results for organic contaminant on (a) slider #2 and (b) slider #3

TOF-SIMS analysis was also performed on slider #2 and #3 to map the distribution of lubricant and hydrocarbon. The results of slider #2 are shown in Fig. 3.14. Two areas were selected for ion mapping. One is focused on the contamination spot on the trailing center pad as this spot is the largest, thickest and conspicuous one. The mapping area was located at the edge of the spot so that we could get a comparison between the on smear and off smear areas. The other mapping area is also near the center pad and located at the junction between the shallow etch region and the deep etch region. This area was supposed to be the head/disk contact area where smears were found to form streaks indicating sliding contact between the slider and the disk media. CF$^+$, CFO$^+$ and C$_2$F$_5$$^+$ are typical ion fragments from disk lubricant while C$_2$H$_3$$^+$, C$_2$H$_5$$^+$, C$_3$H$_5$$^+$ and C$_3$H$_7$$^+$ are fragments from hydrocarbon. The brightness is directly proportional to the ion count. The ion mapping images clearly show that the smears on slider #2 were Z-Dol lubricant. Only
a weak signal of hydrocarbon was captured at the mapping area. This indicates that a large amount of disk lubricant was picked up by the slider during head/disk intermittent contacts and was attached to the slider surfaces, finally leading to interface failure.

Figure 3.14 TOF-SIMS ion mapping images taken from slider #2 (the mapping area is enclosed in the blue square). (a) showing the location of one mapping area at the edge of the largest contamination spot; (b) ion mapping images of the area indicated by (a); (c) another mapping area covering the junction between the shallow etch region and deep etch region where smear streaks formed; (d) ion mapping images of the area indicated by (c). The distribution of lubricant and hydrocarbon are focused in these mapping images. Note that the shape of the contamination smears matches very well with the distribution of lubricant.
Figure 3.15 shows the ion mapping images of slider #3. The debris on slider #3 was not accumulated and mainly distributed at the etch cavity region. Hence, the selected mapping area is located in the negative pressure pocket. A high intensity signal from hydrocarbon ion fragments was captured this time. In addition, strong signal from Z-Dol lubricant was also detected. It is suggested that the etch cavity area was covered by a thin film of hydrocarbon and lubricant. However, it is interesting to note that the distribution of hydrocarbon and lubricant doesn’t match each other. Akamatsu and Ohtani [2002] studied the desorption energy of \textit{n-Heptadecane} on a-C:H surface and on a-C:H surface with PFPE. They found that \textit{C17} is more likely to attach to a-C:H surface than to a-C:H surface with PFPE. Hence, it is speculated that hydrocarbon absorbed and lubricant picked up on the slider surface repelled each other and as a result, distributed differently on the slider surface. Another explanation for the different distribution is that the slider surface was first covered by a thin film of hydrocarbon and later some areas were contaminated by picked-up lubricant. Slider #2 and #3 were sent to the TOF-SIMS vacuum chamber at the same time. However, slider #3 was first analyzed. Considering that hydrocarbon should evaporate quickly in vacuum environment, a weak signal from hydrocarbon detected on slider #2 might be reasonable. In addition to lubricant and hydrocarbon, some other ions (Ti\(^+\), Ni\(^+\), Al\(^+\)) were also mapped. It is noted that very small particles of Ni related products were detected near the contact pad in the mapping area. The Ni particles should be generated by wear and transferred from other locations.
Figure 3.15 TOF-SIMS ion mapping images taken from slider #3. (a) showing the ion mapping area (the mapping area is enclosed in the blue square); (b) ion mapping images of the area indicated by (a). Note that lubricant contaminant was not prominent over the mapping area. Strong signals from hydrocarbon were detected.

Figure 3.16 and Fig. 3.17 present the AE rms results collected for sample #4 during the CSS test in the presence of DOP. A very small peak after 2 sec was also detected in the 1st cycle. However, this peak disappeared after less than 1000 CSS cycles. Except for this, no other notable changes were observed over the entire test. Comparing the AE profiles of sample #4 with those of sample #1, #2 and #3, we have to note that both the amplitude and the noise floor of the AE profile of sample #4 were quite high. It is surprising that such an interface did not fail.
Figure 3.16 AE rms profiles for the (a) 1st, (b) 1000th and (c) 30000th CSS cycles recorded for sample #4

Figure 3.17 AE take-off peak value profile over the entire CSS test for sample #4
After the test, only some small organic liquids were observed at both the trailing edge and the leading edge of the slider (see Fig. 3.18). No visible debris was found at the center pad and the etch cavity area. Examination of disk #4 at the landing zone by OSA also suggested that no wear track was observable. These observations indicate that the CSS performance of sample #4 was relatively stable.

![Image](image.png)

Figure 3.18 Optical micrographs of slider #4 after CSS test: (a) graph of the slider’s trailing edge region; (b) graph of the slider’s leading edge region.

3.3.1.3 Discussion

In the above tests we used an AE sensor to monitor the CSS performance of HDI in the presence of hydrocarbon. Degradation of CSS performance and interface failures were observed for sample #2 and #3. However, these two interface failures were different and one was more severe than the other. This can be correlated to the different AE rms
profiles, different contaminant distribution on slider surface and different contaminant composition. These useful information can help us find out the ways leading to the different failures.

For sample #2, a small peak with increasing intensity was detected after 2 sec during slider take-off over the entire test. These peaks suggest that a hard pseudo-impact event occurred regularly at the trailing edge of the slider with the disk, which resulted in serious disk lubricant depletion and degradation. The degraded lubricant mobile fraction accumulated along with hydrocarbon liquid and was picked up by the slider at the trailing edge. As the CSS cycle number increased, the accumulated organic liquid also increased in amount and was dispersed away on the center pad by the head/disk contacts. The thick liquid might also be knocked off onto the disk and picked up again by the leading edge of the slider and smeared the taper area of the leading edge. When sufficient organic liquid was deposited on the slider surface, this reduced direct solid-solid asperity head/disk contact and provided damping which resulted in lower AE rms amplitude and higher stiction. Meanwhile, as the interface was more and more filled up with liquids, it became less and less stable, and eventually unstable. It is believed that the amount of lubricant (or liquids in general) in the interface is the most sensitive factor that reduces the height of the energy barrier that has to be overcome by either an internal or an external energy perturbation to make the interface unstable (Gui and Marchon [1998]). At this stage, the slider lost the rising force to take off smoothly and had quite poor flyability which resulted in more collisions even at flying conditions. The high AE response and more peaks observed at the noise floor for sample #2 reflect the enhanced head/disk interaction.
The end result of this enhanced interaction is early failure and severe contamination or scratches on the third pad and two ABS rails.

In contrast, the interface failure for sample #3 behaved in a different way. The contact at the trailing edge of the slider with the disk for sample #3 was not so strong as that for sample #2. This is supported by the observed AE response. This resulted in less lubricant depletion and degradation. Also due to the lower initial stiction and less organic liquid at the head/disk contact area, less organic contaminant was picked up at the trailing edge of the slider. As a result, more wear debris should be sucked into the negative pressure zone and trapped at the etch cavity area. The debris deposited at the etch cavity area can potentially re-deposit into the head-disk interface and result in higher AE response.

For sample #4, no notable degradation of CSS performance was observed in a 30k cycle CSS test in the presence of DOP, although DOP may cause stiction at HDI. This could be due to the fact that the concentration of DOP inside the tester chamber was not high enough to degrade CSS performance. This result is in agreement with the studies conducted by Volpe et al. [1992] who found that DOP had little effect on HDI unless a much higher concentration was achieved and complete condensation of the chemical vapor occurred. In contrast, hexadecane and octadecane have much higher vapor pressure than DOP.

A key point that is common to the interface failures of sample #2 and #3 is the intermittent hard contacts at the trailing edge of the slider. This factor, along with other factors, e.g. high stiction/friction, presence of organic contaminants, degrades the slider’s flying characteristics and finally leads to interface failure. For further study, a short slice...
of AE signal after several thousand CSS cycles was recorded by digital oscilloscope and analyzed in the frequency domain for sample #2 and sample #3.

Figure 3.19 shows the power spectra generated for raw AE signals recorded by the Lecroy Oscilloscope during slider take-off. The sampling frequency was 5 MHz and the sampling duration of each short slice of raw AE signal was 20,0004 ms. In the power spectrum of sample #3, one weak intensity peak is detected at around 1621 kHz. Finite element analysis shows that the first bending mode and the first torsional mode of pico sliders should occur at around 1.6 MHz and 1.25 MHz, respectively (Wang et al. [1998], Harris et al. [1998]). Hence, the peak detected at 1621 kHz refers to the first bending mode assigned to the contacts on the center pad at the trailing edge. However, no notable peaks are detected at about 1.25 MHz related to the torsional mode assigned to the contacts on two ABS rails at the leading edge. This indicates that head/disk contacts occur mainly in the tri-pad region for tri-pad sliders (McMillan and Talke [1998]). Some intensity peaks also appear at very low frequency ranges (less than 250 kHz). These peaks should be excited by the bump pattern or related to the air bearing vibrations. By contrast, in addition to bump pattern excitation and bending mode, more peaks are also detected in the low frequency range of 200 kHz to 900 kHz in the power spectrum of sample #2. Several peaks with high intensity are also observed in low frequency range for sample #2 at flying conditions (Fig. 3.20), some of which are detected even at the bump pattern frequency and are supposed to occur only in the slider take-off and land-off profiles. Peaks at low frequencies may indicate a transition state from intermittent contact with disk to continuous touching with disk for flying sliders (Kita et al. [1980]). Therefore,
these additional peaks are strong evidence of an unstable flying state caused by enhanced head/disk interactions due to heavy contamination at the slider ABS.

Figure 3.19 Power spectra of short slice of AE signals recorded during slider take-off for sample #2 and #3 (left side: the raw AE signals; right side: the power spectra). The sampling frequency was 5 MHz.
Figure 3.20 Power spectra of short slice of AE signals recorded at flying conditions for sample #2 and #3 (left side: the raw AE signals; right side: the power spectra). The sampling frequency was 5 MHz.

### 3.3.2 CSS tests with rest period

#### 3.3.2.1 Results

Similar CSS tests were also performed with those samples listed in Group B. In order to evaluate the effect of the stiction developed during the CSS tests, after 15k CSS cycles each slider of Group B was parked at the landing zone of the disk and rested for 12 hrs before the next CSS cycle. The AE rms data profiles recorded for some CSS cycles of
sample #5 are presented in Fig. 3.21. It is noted that, unlike the AE data recorded for Group A, no small peaks after 2 sec were detected in each cycle at the beginning of the CSS tests of sample #5. This indicates that slider #5 had better take-off performance than sliders in Group A. However, after 15k CSS cycles and a 12 hr rest period the AE rms data profiles underwent great changes. In cycle 15001, no peaks were detected and the value of the AE response was very close to zero over the entire cycle. It is suggested that high stiction occurred and prevented the motor from spinning up. In cycle 16759, although two main peaks were detected, the peak excited by slider take-off appeared after 2 sec. That means the slider’s take-off was delayed due to high stiction. Deformed AE rms data profile was also observed for cycle 16910, where only one big peak was detected at 6 sec. Similar deformed AE rms data profiles were also recorded for sample #6 (see Fig. 3.22) and sample #7 (see Fig. 3.23). All these observations suggest the existence of high stiction at the HDI, which prevented the motor from spinning up or degraded the slider’s take-off performance.
Figure 3.21 AE rms profiles for the (a) 1\textsuperscript{st}, (b) 15000\textsuperscript{th} and (c) 15001\textsuperscript{st}, (d) 16759\textsuperscript{th}, (e) 16910\textsuperscript{th} and (f) 30000\textsuperscript{th} CSS cycles recorded for sample #5.
Figure 3.22 AE rms profiles for the (a) 1\textsuperscript{st}, (b) 15000\textsuperscript{th} and (c) 15001\textsuperscript{st}, (d) 15011\textsuperscript{th}, (e) 16300\textsuperscript{th} and (f) 30000\textsuperscript{th} CSS cycles recorded for sample #6
Figure 3.23 AE rms profiles for the (a) $1^{st}$, (b) $15000^{th}$ and (c) $15001^{st}$, (d) $15050^{th}$, (e) $15060^{th}$ and (f) $30000^{th}$ CSS cycles recorded for sample #7
Figure 3.24, 3.25 and 3.26 present the AE rms peak values recorded during the slider’s take-off over the entire CSS test for sample #5, #6 and #7, respectively. From these figures we can also see the effect of stiction on the HDI performance. In these figures, the AE rms peak value dropping to near zero suggests an interface failure due to high stiction at the HDI causing the motor’s spinning up or the slider’s take-off to be unsuccessful. One can clearly see that this kind of interface failure occurred more times with sample #5, followed by sample #6 and #7. This might be due to the fact that hexadecane has much higher vapor pressure than octadecane and DOP. More hexadecane vapor was introduced into the test chamber. Hence, higher stiction could be observed in the presence of hexadecane.

![Figure 3.24 AE take-off peak value profile over the entire CSS test for sample #5](image)

Figure 3.24 AE take-off peak value profile over the entire CSS test for sample #5
Figure 3.25 AE take-off peak value profile over the entire CSS test for sample #6

Figure 3.26 AE take-off peak value profile over the entire CSS test for sample #7

The optical micrographs for slider #5, #6 and #7 taken after the CSS tests are shown in Fig. 3.27, 3.28 and 3.29, respectively. Compared with sliders in Group A, it is noted that these sliders, especially slider #7, are relatively clean. Few smears or droplets were found accumulated at the trailing edge of the sliders. This observation correlates well with the good take-off performance of the slider before the rest period. This is also reflected in the AE rms profile. That shows no hard impacts at the trailing edge of the slider with the
It is therefore suggested that the interaction between the trailing edge of the slider and the disk media was weak. Therefore, less wear debris was generated and less lubricant was picked up at the trailing edge. This suggestion is also supported by the OSA images of these disks. Only light lubricant depletion or degradation was observed and no visible carbon wear track was found. It seems that all these observations lead us to the same conclusion drawn by Koka [1995] that hydrocarbon does not introduce any smears or debris on sliders and does not cause visible wear tracks to appear on disks either.

Figure 3.27 Optical micrographs of slider #5 taken after the CSS test in the presence of hexadecane contamination: (a) graph of the slider’s trailing edge region; (b) magnified view of the pole tip area; (c) magnified view of the supposed smear; (d) graph of the slider’s leading edge region. Only two contamination spots were detected and the contamination was very thin.
Figure 3.28 Optical micrographs of slider #6 taken after the CSS test in the presence of octadecane contamination: (a) left side of the trailing edge region; (b) right side of the trailing edge region; (c) left side of the leading edge region; (d) right side of the leading edge region. Note that smears were mainly distributed at the leading edge region and the trailing edge region was quite clean.
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3.3.2.2 Discussion

The test conditions for Group B and Group A were the same except that a long rest period was introduced to Group B, but the test results were totally different. Despite the degradation of CSS performance and the severe liquid accumulation at the slider surface, the motor’s incapability to spin up was not observed for the test samples in Group A. By contrast, no severe liquid accumulation was observed for sliders of Group B. However, serious failures in motor’s spinning up and slider’s take-off due to high stiction were detected. It is interesting to note that all these interface failures occurred after the long rest period.
period. Therefore, in CSS test a long rest period is normally necessary for high stiction. This conclusion is also supported by the studies conducted by Li et al [1990], in which it was found that stiction increases substantially after a long parking time. This stiction is called rest stiction and resulted from the formation of meniscus bridges at the asperity contacts during the rest period. During the CSS test, the condensed hydrocarbon liquid and the degraded lubricant fraction might wet both the slider and the disk media. When the slider was brought to park at the landing zone, the liquid spread itself, flooded the HDI and formed menisci around the contacting points. The meniscus force and the applied load could cause the contacting asperities to undergo elastic response deformation. The spontaneous deformation could continue until the entire interface was in full contact. Therefore, an excessive force had to be overcome to initiate sliding at the HDI. In addition, the stiction can gradually increase during the CSS tests. As the CSS cycle number increases, the disk landing surface is burnished and as a result, an even smoother landing zone is produced. Smooth surfaces cause higher contact friction or “stiction”. This also contributes to the high rest stiction at the HDI.

In the micrographs of slider #5, we can clearly see a black area near the center pad. TOF-SIMS analysis results (Figure 3.30) identify that the slider surface was covered by a thin film of lubricant whereas, the chemical identity of the black area indicated on the optical micrographs is unclear. We speculate that this black area is a deformation caused by the high meniscus force during the rest period or at the drive start. This deformation shows that a large area of wet asperity contacts at the HDI is evident.
Figure 3.30 TOF-SIMS analysis of the smear on slider #5. (a) graph showing the mapping area (enclosed in the blue square); (b) positive ion spectra and (c) ion mapping images for C⁺, Al⁺ and CF⁺. Note that the mapping area was reasonably clean except for a thin film of lubricant. However, the mismatch between the shape of the black smear and the lubricant distribution indicates that the smear was not lubricant contaminant.

3.3.3 Continuous-flying tests

3.3.3.1 AE signal analysis

Figure 3.31 presents the recorded AE rms average response profile of each sample during the continuous-flying tests. The experiment was conducted in the presence of 3 different contaminants: hexadecane, octadecane and DOP. We can clearly see some differences among these 3 AE profiles, reflecting different head-disk interaction behaviors possibly resulted from the presence of different contaminants.
Figure 3.31 AE rms average value profiles recorded for sample #8, #9 and #10 during the continuous flying tests. The test period of each sample was 96 hrs.
For sample #8, the AE rms average value rises in a short time and then decreased continuously. After the slider had flown over the disk surface for about 1200 mins, the AE rms average response remained stable to the end of the test. For sample #9, the AE rms average response stabilized after 4000 mins but many sudden peaks were observed during the flying test. Whereas, the AE rms average response of sample #10 fluctuated and was completely unstable over the entire test. The instability and sudden changes of AE average response suggested that some events could have occurred at the HDI. These events may include lubricant depletion and degradation, head slip and asperity contact, and slider pick-up and drop-off of liquid or debris (Jiaa et al. [1997]). From this view, it is concluded that these events occurred mainly at the initial stage of flying for sample #8. However, for sample #9 and #10, more head-disk interactions occurred during the entire continuous-flying. The sudden peaks in AE rms average observed for sample #9 indicate much stronger slider/disk interactions.

For further studies, the raw AE signals collected from these three samples are also analyzed in the frequency domain (see Fig. 3.32). The raw AE signals were recorded in the middle of the continuous flying tests. Each slice of raw signal was collected with a sampling frequency of 5 MHz and a sampling period of 0.0004 ms. One can clearly see that sample #9 and #10 have peaks of high intensity in the low frequency ranges (less than 300 kHz). Typically, a slider’s air bearing vibrations lie in the range of 100-300 kHz (Takahashi et al. [1998]). It is suggested that the air bearing vibrations of sample #9 and #10 were much stronger than sample #8. It is also detected that sample #10 has a notable peak at around 1.62 MHz which refers to the first bending mode of the slider, indicating
that slider #10 had strong interactions at the trailing edge with the disk. This peak is, however, weak or unobservable for sample #8 and #9.

Figure 3.32 Raw AE signals and the corresponding power spectra of sample #8, #9 and #10 (left side: the raw AE signals; right side: the power spectra). The raw AE signals were recorded in the middle of the continuous flying tests. For
each slice of raw signal, the sampling frequency was 5 MHz and the sampling period was 20,0004 ms.

### 3.3.3.2 Lubricant modulation and redistribution

Figure 3.33 presents the S-polarized OSA images of the flying tracks on disk #8, #9 and #10. The disk samples initially had about 10 Å of partially bonded film of Z-Dol/X-1p lubricant distributed evenly on the carbon overcoat. After a long flying test period of up to 96 hrs, one can clearly see the tracks due to the redistribution of the lubricant. The dark lines in these images indicate decreased intensity of reflected S-polarized light caused by lubricant pooling resulted from the degradation and debris accumulation of the lubricant. There are three notable dark lines on disk #9 and #10. The areas between these dark lines are of lighter color, indicating light lubricant depletion. It appears that the lubricant was pushed away from underneath the sliders along the flying tracks, creating lubricant tracks due to the two rails and the one central pad. This strong force of “pushing” should originate from the high pressure generated at the slider air bearing surface. When a disk is spinning at a very high speed, the pressure underneath the trailing pad of the air bearing surface may be as high as 0.5-1 MPa even if the head does not make contact with the disk surface (Pit et al. [2001]). Such a high pressure will deform the lubricant film locally and push the lubricant around by air-shear force and pressure gradient. As a result, the redistribution of free lubricant is quite dependent on the air bearing pressure between slider and disk. The reflectivity signal indicating lubricant
depletion is weak and there was also no high amplitude AE response observed. It is concluded that no solid direct head/disk contact occurred during the flying test.

Figure 3.33 S-polarized OSA images of the flying track on (a) disk #8, (b) disk #9 and (c) disk #10. The area of the image is between radius 38.5mm and 41.5mm and from 0°~360°. Disk spinning direction was from right to left.
Figure 3.34 Cross section of lube modulation along the radial direction from 39.3 ~ 40.8 mm in S-polarized images. The increase of lubricant thickness decreases the reflectivity of S-polarized light. The valleys of the curves are where lubricant pooled.

Figure 3.34 presents a cross section of the lube modulation along the radial direction. The valleys of reflectivity are where lubricant pooling formed. One can clearly see that the lubricant pooling on disk #9 and #10 are much more notable than on #8. Similar results are also obtained by performing lubricant degradation analysis. If the total area analyzed by OSA is considered as a whole, lubricant degradation rate is defined as the percentage of the area where the lubricant is degraded. The OSA analysis results suggest that the lubricant degradation rates for these flying tracks are in the following sequence: #9 (0.12%) > #10 (0.05%) > #8 (0.0067%). This should be attributed to more interactions at the HDI for sample #9 and #10 due to stronger air bearing vibrations during flying. This result correlates very well with the analysis done from the AE rms average data and
raw AE signal. Sample #9 and #10 had strong air bearing vibrations at the HDI and this contributed to more lubricant being pushed around underneath the slider and accumulated under the two rails and the central pad.

3.3.3.3 Lubricant pick-up and the induced fly stiction

Optical micrographs taken for these sliders after the continuous-flying tests are shown in Fig. 3.35, 3.36 and 3.37.
Figure 3.35 Optical micrographs of slider #8 taken after 96 hrs continuous-flying test in the presence of hexadecane contamination. (a) and (b): graphs of the trailing edge region; (c) and (d): graphs of the leading edge region; (e): magnified view of the pole tip area; (f): magnified view of the contamination spots. Note: large liquid droplets were accumulated at the etch cavity area.
Figure 3.36 Optical micrographs of slider #9 taken after 96 hrs continuous-flying test in the presence of octadecane contamination. (a) and (b): graphs of the trailing edge region; (c) and (d): graphs of the leading edge region; (e): magnified view of the pole tip area; (f): magnified view of the contamination spots. Note: large liquid droplets were accumulated at the etch cavity area. The observations of slider #9 are quite similar to those of slider #8.
Figure 3.37 Optical micrographs of slider #10 taken after 96 hrs continuous-flying in the presence of DOP contamination. (a) and (b): graphs of the trailing edge region; (c) and (d): graphs of the leading edge region; (e) and (f): magnified view of the pole tip area. Note: the slider picked up many fine liquid particles at the trailing edge area.

It is noted that all three sliders were heavily contaminated and these contamination can very well explain the variations of the AE rms average data. Visual examination reveals that smears are mainly distributed at three principal locations: the trailing edge, the etch cavity area close to the trailing edge, and the leading edge. Minute droplets are also seen over the air bearing surface which are aligned parallel to the long axis of the slider in the direction of the air stream. Large contaminant spots were observed in the etch cavity area on slider #8 and #9. These spots might have originated from aerosol particulates that consist of condensed hydrocarbon, lubricant and other environmental contaminants. As all sliders used in this study were negative-pressure sliders, these particulates could have been sucked into the negative-pressure zone and trapped by the slider surface when they entered the HDI during the continuous-flying. This event should occur mainly at the early stage of the continuous-flying for sample #8 as indicated in the AE rms average profile. In addition, some small liquid droplets were also found at the
trailing edge near the pole tip. For slider #10, by contrast, quite a lot of very fine liquid droplets are seen accumulated at the trailing edge and over the pole tip. These droplets could have been picked up by the trailing edge of the slider from the media surface or the air flow. From the large amount of droplets accumulated at the trailing edge and the instability of the AE rms average, it can be inferred that the pick-up event had occurred over the entire test process. This observation also correlates well with the analysis results obtained from the raw AE signal. The first bending mode of sample #10 was stronger than the other two samples and contributed to the large amount of fine droplets picked up by the trailing edge. It is also noted that liquid streaks are observed on all three sliders. This could be the consequence of either of the following two cases: (1) collisions and sliding between slider and liquid materials; (2) spreading of large liquid droplets on the sliders once they became unstable during flying.

![Figure 3.38 AE rms profiles for CSS cycle: (a) before and (b) after continuous flying recorded for sample #8](image-url)
Figure 3.39 AE rms profiles for CSS cycle: (a) before and (b) after continuous flying recorded for sample #9

Figure 3.40 AE rms profiles for CSS cycle: (a) before and (b) after continuous flying recorded for sample #10

Although stiction value was not measured in this study, we speculated that high stiction had occurred at the HDI when sliders first took off the landing zone after the long continuous-flying test followed by a 12 hrs of rest period. This is because liquid had
accumulated on the sliders’ surface. This conclusion is supported by the comparison between the AE rms data profiles of the CSS cycles performed before and after the continuous-flying. These profiles are shown in Fig. 3.38, 3.39 and 3.40 for sample #8, #9 and #10, respectively. For sample #8, the AE rms profiles were almost identical before and after the continuous-flying except for a small reduction in the amplitude. By contrast, sample #9 underwent great changes in the AE rms profile. The AE rms amplitude during take-off decreased from around 2 before the continuous-flying to about 0.5 after the continuous-flying. An explanation for this is that sufficient liquid and contaminants were picked up during the long continuous-flying test. Similar observations were also made for sample #10 and the percentage of the reduction in rms amplitude was even higher. The changes observed between the “before” and “after” AE rms profiles suggest that slider #9 and #10 should have experienced high stiction at the start of taking off resulted from the liquid contaminant picked up during the continuous flying test.

This type of stiction, developed during the continuous-flying tests, is called fly stiction, which occurs only after a head has flown over a disk for an extended period of time (Gui and Marchon [1998]). It is different from that observed in the CSS tests of Group B, which we called “conventional stiction”. The main difference between fly stiction and conventional stiction is that wet asperity contact in fly stiction is mainly caused by the liquid droplets formed during flying. Whereas, in conventional stiction, wet asperity contact originates from the contact at the interface (Zhao and Bhushan [2001]). Stiction force is mainly due to meniscus formation at the HDI. For a typical wet asperity at the head/disk interface as a sphere above a flat sphere surface with liquid in between, the meniscus force can be obtained by the meniscus force model as discussed by Li et al.
The meniscus force is proportional to liquid surface tension and increases with increasing liquid thickness. The surface tensions of hexadecane, octadecane and DOP at 20°C are 27.5mN/m, 28.5mN/m and 31.1mN/m, respectively. This shows that the 3 chemicals used in my study do not have significant difference in terms of surface tension. We also assume that liquid thickness is proportional to liquid amount. Therefore, the fly stictions in my study are mainly dependent on the amount of liquid materials picked up by the sliders. Theoretical studies have revealed that fly stiction is an interface instability event triggered by an excess amount of liquid material that is carried into the interface by the head. The outcome of a fly stiction measurement could be either low or high, depending on whether the amount of liquid that transfers into the interface is sufficient to trigger the instability (Gui and Marchon [1998]).

Figure 3.41 shows a schematic side view of a flying slider picking up contaminant particulates at its surface. Contaminant particulates illustrated in Figure 3.41 could be condensed hydrocarbon, water, lubricant fraction scattered by the shear and expansion forces from the air bearing pressure or other environmental contaminants. They are carried into the HDI by the air flow. These particulates at the HDI may interact with the head/disk in different ways. Some particulates that hit the slider taper area may be attached and agglomerate at the taper area. Some may be attached to the disk surface and leave liquid streaks on the slider surface during the collisions with slider. Larger particulates that cannot pass through the HDI are more likely to be sucked into the negative-pressure zone and attached into the etch cavity area. Whereas, small particulates may pass through the small space between the head and the disk but most of them will be
trapped at the trailing edge as whiskers due to the strong vortex that exists at the trailing edge.

Figure 3.41 Schematic side view of a flying slider picking up liquid contaminant

Figure 3.42 (Henshaw, W. D. et al. [1991]) shows the streamlines of the fluid around the slider. The vortex at the trailing edge at three different periods of time is shown in Fig. 3.42 (a) and a magnified view is shown in Fig. 3.42 (b). Very close to the trailing edge side, streamlines under the head slider go up while the streamlines from the top go down. A recirculation region can be observed behind the slider with a stagnation point above the corner. It is believed that the vortex at the trailing edge is a key factor in causing fly stiction. First, fine particulates passing through the HDI cannot flow away quickly because of the vortex and will stagnate at the trailing edge. Second, a segment of the local liquid film located on the disk surface and underneath the trailing edge of the head slider can be blown away as a result of a sufficiently strong vortex and results in scattered liquid molecules. Therefore, air flow containing a relatively high concentration of liquid
molecules forms around the head slider. If the fly height between the head and the disk surface is close enough and the vortex is strong enough, the liquid molecules can be readily picked up by the sliders at the trailing edge. This air flow also makes it possible for the cavity area to pick up liquid molecules. Liquid droplets form at the trailing edge and/or the cavity area on the slider surface, and then migrate to the rail surfaces. A meniscus between the asperities at the interface forms during the rest time of the slider on the disk surface and leads to high stiction when the disk restarts up, thus causing fly stiction.

(a)
Figure 3.42 Streamlines around the head slider with (a) the vortex at the trailing edge side at three different periods of time and (b) a magnified view of the trailing edge side

3.3.3.4 Chemical analysis of the liquid contamination on slider surfaces

TOF-SIMS analysis was performed on the head smears. Figure 3.43 shows the positive ion spectra at different mass ranges taken from the smears on slider #8. Strong peaks observed at the masses 31 (CF\(^+\)), 47 (CFO\(^+\)), 50 (CF\(_2\)\(^+\)), 69 (CF\(_3\)\(^+\)), 100 (C\(_2\)F\(_4\)\(^+\)) and 119 (C\(_2\)F\(_5\)\(^+\)) are fragments from the main chain of Z-Dol lubricant; whereas, the peaks at masses 95 (FC\(_6\)H\(_4\)\(^+\)), 126 (CF\(_2\)C\(_6\)H\(_4\)\(^+\)), 145 (CF\(_3\)C\(_6\)H\(_4\)\(^+\)), 902, 952, 1002 and 1052 are typical fragments from the X-1P additive. In addition, some peaks from hydrocarbon fragments (29 (C\(_2\)H\(_5\)\(^+\)), 43 (C\(_3\)H\(_7\)\(^+\)), and 57 (C\(_4\)H\(_9\)\(^+\))) are also detected. The spectra results indicate that the head smears mainly consist of lubricant and hydrocarbon.
Figure 3.43 Positive ion TOF-SIMS spectra taken from the smears on slider #8
Figure 3.44 TOF-SIMS ion map images of the surface around the contamination spots on slider #8: (a) graph showing the mapping area (enclosed in the blue square); (b) ion mapping images of $C^+$, 126.031(amu), 1002.124(amu), $C_2H_5^+$ and $C_3H_7^+$. The results indicate that the contamination spots are X-1P droplets.

Figure 3.44 presents the ion mapping images of the surface around the large contamination spots on slider #8. In TOF-SIMS ion mapping, brightness is directly proportional to the ion count. As we can clearly see, the area where fragments with masses of 126 and 1002 are accumulated correlates very well with those large contamination spots in shape and distribution. Therefore, it is concluded that these contamination spots are almost purely X1-P droplets. By contrast, lubricant and hydrocarbon are distributed almost evenly on the surface. It is suggested that a large amount of X1-P additive was picked up by the slider from the disk and accumulated as
droplets during the continuous-flying test. Similar observations were also made from the surface of slider #9.

However, no X1-P droplets were found on slider #10 and the TOF-SIMS analysis results suggest that the smears on slider #10 were Z-Dol lubricant. Figure 3.45 presents the ion spectra taken from smears on slider #10. The typical fragment ions from X1-P at masses 95, 126 and 145 were not detected and the intensity signal for ions at masses 952 and 1002 was also very weak.

Figure 3.45 Positive ion TOF-SIMS spectra taken from the smears on slider #10
Relating the TOF-SIMS analysis results to the sliders’ micrographs, we find that, on sliders #8 and #9 where X1-P droplets formed, less Z-Dol lubricant was picked up. Whereas, on slider #10 where no X1-P droplets formed, more Z-Dol lubricant was accumulated. C. Mathew Mate et al. [1998] studied the interaction of X1-P with disk and slider surfaces. They found that in order to lower surface energies X1-P molecules are more likely to attach to carbon overcoat than Z-Dol lubricant so as to increase the lubricant mobility by mediating its interaction with the disk. X1-P also improves disk lubrication by blocking and passivating the catalytic Lewis acid sites such as powered Al$_2$O$_3$ on slider and disk surfaces, preventing catalytic decomposition of the perfluoropolyether lubricant. Since the sliders used in this study were made of Al$_2$O$_3$ and coated with DLC, it is inferred that X1-P molecules are more likely to be picked up by sliders during the flying tests. But it is difficult to explain why the pick-up of X1-P did not occur on slider #10. Published studies (Hara et al. [2001], Kang et al. [1999]) have shown that micro-phase separation between X1-P and Z-Dol could occur due to their insolubility in each other. Do our observations of X1-P droplets on slider #8 and #9 suggest that disk #8 and #9 had already experienced this micro-phase separation before or during the continuous flying test? Is it due to the presence of different hydrocarbon? Or due to any other reasons? Due to the limitations of time and equipments, these hypotheses were not verified. Further studies are required to understand this more completely.
3.4 Summary

In this study we investigated hydrocarbon and lubricant as two main sources of liquid contamination at HDI. CSS and continuous-flying tests were performed at ambient conditions in the presence of hydrocarbon that was specially introduced into the test system. Our experiments lead us to the following observations or salient points:

(1) Existence of intermittent hard head/disk contacts is a key factor that initiates and accelerates interface failure. The contacts allow slider to pick up liquid materials which accumulate at the trailing edge. As more and more liquid materials are picked up, the slider’s flyability become unstable and interface failure occurs.

(2) The presence of hydrocarbon should have detrimental effects on interface performance but it doesn’t necessarily lead to interface failure. Slider’s flying characteristics play an important role.

(3) High rest stiction was observed in the presence of hydrocarbon. The stiction was gradually developed during the CSS tests. A long rest period was necessary for high stiction to occur.

(4) Hydrocarbon alone does not cause smear on slider surfaces. Head smear and disk wear are caused by lubricant pick-up or lubricant depletion.

(5) Lubricant modulation and redistribution was observed during continuous-flying and analyzed. This was attributed to strong air bearing vibrations and air pressure.

(6) Lubricant pick-up by sliders and its induced fly stiction was observed for continuous-flying tests. The picked up materials were distributed mainly at three locations: the trailing edge, the etch cavity area near the trailing edge, and the leading
edge.

(7) X1-P additive was observed to have been picked up from disk media and accumulated as liquid droplets at the slider etch cavity area. The mechanism of this phenomenon is unclear and needs further study.

(8) AE response is sensitive to HDI interactions. It provides useful insights into understanding the flying characteristics of sliders.

(9) The presence of hydrocarbon affects the HDI by increasing stiction and aggravating contamination on slider surface. However, its effects are not quantitatively analyzed in this study.
CHAPTER 4

Effect of Organic Siloxane at Head/Disk Interface

4.1 Introduction

Silicone is one source of undesirable contamination from the standpoint of interface reliability. A common source of this chemical is the release liners in adhesives. The use of pressure sensitive adhesives in assembly operations, as gaskets, as protective seals against the environment in drives and in perimeter sealing of hard disk drive clamshells are commonplace in the disk drive industry. Published information on the deleterious effects of silicones for the HDI is limited. In an early work, Yamamoto et al. [1991] presented results for a particulate disk with perfluropolyether lubricant and Alumina-TiC as the head. Octamethyl cyclotetrasiloxane was used as the source to simulate the organic siloxane. In the presence of this atmosphere, head-disk contacts increased during seeking, leading to a crash. The formation of silicon oxide particles at the interface was explained by the generation of heat at the HDI. X-ray photoelectron spectroscopy (XPS) of the crashed disk surface led the authors to conclude that the siloxane on the disk surface was oxidized by the heat generated by head-disk contacts. Golden et al. [1995] examined the role of volatile organic compounds released from adhesives and showed that they caused disk wear and resulted in failures during contact start/stop (CSS) tests. More recently, Segar and Jesh [1999] developed a test procedure to assess the effects of adhesive
outgassing on head-disk interface tribology. A total of 17 pressure sensitive adhesives (PSA) were evaluated and their effects on stiction and head smearing were characterized. The principal results were: (a) all PSA’s had detrimental effect on stiction and varying effects on head smearing; (b) HDI durability was largely unaffected; (c) no correlation could be established between the total outgassing from a PSA and either stiction or head smearing. The tests involved a combination of temperature and humidity cycling procedures as well as CSS cycles and seeks over the disk surface. However, there was no chemical identification of the head smears that formed during tests or information relating to chemistry that occurred at the head-disk interface. Raman et al. [2000] investigated flyability failures due to organic siloxanes at the HDI in a flyability performance test system by making use of capacitance measurements. Both pure octamethyl cyclotetrasiloxane liquid and metallized polymer adhesive tape were used as the source of organic siloxane in this study and similar flyability failures were observed. More recently, J. Xu et al [2003] developed in-situ techniques for observing and studying the development process of organic siloxane contamination. Silicon oxide particles were also found on the slider surface. The principal results were: (a) the development process of contamination consisted of two stages: initiation of a contamination seed and growth of the seed. Initiation time was much longer than growth time; (b) contamination always initiates at the slider’s trailing edge and the development process was activated by contact/friction; (c) as the slider’s fly height decreases, the initiation time decreases significantly, while the growth time decreases only slightly. Investigations by Kasai et al [2000] had revealed how silicon oxide could form in the disk drive environment. Two prerequisites for silicon oxide formation were proposed: (a) the presence of siloxane; (b)
generation and presence of fluorocarboxylic acid which is a product of the disproportionation reaction of Z-Dol lubricant catalyzed by Lewis acids. The degraded lube would repolymerize D4 (octamethyl cyclotetrasiloxane) back into PDMS at the HDI area. With the heat generated through head/disk contacts, PDMS undergoes oxidation process and forms silicon oxide at HDI.

In this study, we examine the effect of siloxane contamination in hard disk drives using accelerated CSS tests at component level. One specific focus of this work is to examine the processes that lead to failures under CSS test conditions. Chemical identification was performed on the contamination on slider surfaces to understand the tribochemical processes at the head/disk interface and how the interface failure occurs. Tests were performed using disks lubricated with pure Z-Dol or a Z-Dol/X1-P mixture to evaluate the effect of X1-P on improving the durability and reliability of the disk system against organic siloxane contamination. AE response techniques were also utilized to study the head/disk interactions.

4.2 Experimental

4.2.1 Apparatus

Figure 4.1 shows the experimental setup in this study. The tester is the same one used for hydrocarbon contamination study. However in this experiment, the recirculator is removed and an environmental chamber is introduced for two purposes: 1) to elevate temperature and accelerate the chemical evaporation; 2) to generate various testing
conditions. The chemical contamination source is changed to siloxane this time. A glass dish containing a known amount of pure liquid siloxane is placed beside the tester in the glass chamber. The liquid should evaporate quickly at high temperatures and spread inside the whole chamber. Some of them might be absorbed or condensed onto slider/disk surface.

![Figure 4.1 Schematic diagram of the experimental setup](image)

4.2.2 Test components and chemical samples

Similarly, all the sliders used in this study were sub-ambient negative pressure pico sliders with two side pads or one center pad. They had a fly height of 14 nm and a pre-load of 2.5g. These sliders were overcoated with diamond like carbon and had a Si seedlayer on Al$_2$O$_3$-TiC substrate. Commercial magnetic recording disks with diameter of 95mm were used for all tests. The disk samples were overcoated with 15 nm nitrogenated
carbon and had a top layer of Z-dol lubricant of 1.0 nm with or without X1-P additive. The disks with X1-P additive had a laser textured landing zone fabricated between the radius of 16.5 mm and 19.5 mm, and the crater-shaped laser bumps were 10 µm in diameter and 10 nm in height and arranged in spiral pattern. By contrast, the landing zone of disks without X1-P additive were not textured.

The source of the organic siloxane used in this study was pure octamethyl cyclotetrasiloxane from Merck Inc., available as a colorless liquid at the 98% purity level. Its chemical and physical properties are listed as follows:

Chemical name: Octamethyl cyclotetrasiloxane

Formula: C₈H₂₄O₄Si₄

Density: 0.95 g/cm³

Boiling point: 176 °C

Melting point: 18 °C

Vapor pressure(@ 20°C): 0.978 mmHg

Vapor phase concentration (@ 20°C): 15.83 g/m³

For each experiment, a glass dish containing about 10ml pure siloxane liquid is placed beside the tester inside the glass chamber.

4.2.3 Test procedure

After the head, disk and chemical sample are loaded onto the tester, the tester enclosed in the glass chamber is placed in the environmental chamber. The temperature inside the chamber is cycled from 30° to 70° C two times over a period of one day to promote vaporization of the chemical and condensation onto the heads and disks. The temperature is held at the temperature extremes for six hours with a 1.5 hour heating or cooling period in between. During this time, the disks are stationary with the heads
parking at the landing zone. Following the chemical evaporation precondition, the tester remains in the chamber and CSS tests are performed under different environmental conditions. In all tests, an AE sensor is used to monitor possible contacts at the HDI. The test may last up to 50k cycles unless an interface failure occurs. A single CSS consists of a 3 sec rapid on, a 3 sec hold at 7200 rpm and a 3 sec rapid off followed by a 3 sec rest. During the CSS test, the AE rms data profile of each CSS cycle was reported. And in order to evaluate the slider’s flying characteristics, another two values were recorded and monitored as a function of time. One is the AE rms peak value of the slider’s take-off profile; the other one is the average value of AE rms response at flying conditions for each CSS cycle (from 3\textsuperscript{rd} sec to 6\textsuperscript{th} sec).

All tests were performed in an environmental chamber. However, the sample loading into the glass chamber was done under a class 100 flow hood before the glass chamber was moved into the environmental chamber. Additionally, in order to minimize or eliminate the interference due to the chemical introduced in the previous tests, precautions were taken to completely clean out the tester by wiping/cleaning the enclosure with iso-propyl alchohol and subsequently thoroughly air-dried in a clean dry air stream. To ensure the reproducibility, our experiments were repeated at least once for each test condition.

After each test, the disk surface is examined by OSA to check if a notable wear track exists. The slider is also examined by optical microscope to define the contamination patterns on ABS. The chemical identification of the contamination on slider surface is analyzed by TOF-SIMS.
4.3 Results

4.3.1 Test results in the absence of siloxane

Three Control runs were first performed to evaluate the CSS durability of HDI in the absence of siloxane contamination. All control runs gave us the similar results, indicating that our experiments had good reproducibility. Figure 4.2 presents an example of the AE rms results taken for a CSS test conducted with a Z-Dol/X1-P-coated disk under 50°C/40%RH environmental conditions. As we can see that the AE rms peak values and average values were relatively stable over the entire test with up to 50k CSS cycles indicating that no interface failure had occurred. Examination of the slider following the test reveals that the slider surface was clean and free of contamination (see Fig. 4.3). And no visible wear tracks were detected at the disk contact zone. All these information suggests that the head/disk combination had good CSS durability in the absence of siloxane contamination.
Figure 4.2 (a) The AE take-off peak value profile and (b) the AE rms average value profile recorded for the CSS test conducted in the absence of siloxane under 55°C/40%RH. The lube was Z-Dol/X1-P mixture. The results suggest good CSS performance in the absence of siloxane contamination.

Figure 4.3 Optical micrographs of the slider surface after the control run, showing that the slider surfaces were quite clean. (a): graph of the pole tip and center pad area; (b): graph of the leading edge region; (c) and (d): graphs of the junction areas of the leading edge and the side rails.
4.3.2 Test results in the presence of siloxane using pure Z-Dol-coated disks

Figure 4.4 shows a representative example of test data taken for CSS tests conducted with disks without X1-P additive in the presence of siloxane contamination. For the convenience of comparison, this test sample is labeled as sample A. The siloxane source in this experiment was 10ml liquid of octamethyl cyclotetrasiloxane placed at the bottom of the tester. The test was performed at ambient conditions after one day of preconditioning. During the test, both the AE rms peak value of each take-off profile and the AE rms average value at flying conditions of each CSS cycle were monitored as shown in Fig. 4.4(a) and 4.4(b) as a function of CSS cycles. From Fig. 4.4(a) we can see that the AE rms peak value remained quite high almost throughout the entire test except for a sudden drop after 6000 CSS cycles. It is suggested that there was always a solid and hard contact between the slider and the disk media during the slider’s take-off. By contrast, Fig. 4.4(b) provides an irregular AE rms average profile which indicates that the interface became unstable after 3000 CSS cycles when direct head/disk contacts occurred intermittently even at flying conditions. It was observed that, after around 6600 CSS cycles, the AE rms average value increased continuously and eventually varied erratically (see the magnified view of the AE rms average data at the onset of failure).
Figure 4.4 (a) The AE take-off peak value profile and (b) the AE rms average value profile and the magnified view of the data at the onset of failure recorded for the CSS test conducted in the presence of siloxane under 30°C/25%RH. The lube was pure Z-Dol. The results indicate a head crash.

Figure 4.5 shown below presents the AE rms value profiles recorded for several CSS cycles. As we can see, the AE rms profiles underwent great changes from the beginning to the end of the test, indicating that the slider’s flyability was totally degraded.
Figure 4.5 The AE rms profiles recorded for (a) 1st cycle; (b) 4400th cycle; (c) 6800th cycle and (d) 7500th cycle of the CSS test conducted in the presence of siloxane under 30°C/25%RH.

Combining these figures with the AE rms average profile over the entire test, we presume that the failure was due to the accumulation of reaction products on the slider ABS at certain key locations. The process leading to failure evolved as follows: as CSS cycles increased, the head/disk contacts induced the formation of reaction products at the contact area. The reaction products decreased the fly height and resulted in asperity contacts at HDI even at flying conditions. However, these products could have also been
removed by head/disk contacts; hence, the solid contacts occurring at flying conditions only took place randomly. As more and more CSS cycles were conducted, more and more reaction products were formed and stayed firmly at the slider ABS. The fly height thus decreased continuously and the slider’s flyability degraded significantly. As a result, the slider made contacts with the disk media directly and continuously even at flying conditions as can be seen in Fig. 4.5(d).

Figure 4.6 shows the micrographs of the slider ABS taken by optical microscope following the test. The two graphs on the right side are magnified views of sub-areas. These graphs reveal an asymmetrical contamination pattern on the slider ABS: although some little contamination spots and smears were also found at the etch cavity region and the right side of slider ABS, the dark brown debris material, which looks like white feathery substance under dark field conditions, was prominent on the left side pad and left side rail (corresponding to the side flying at higher disk radius). It is also observed that the contamination went through the pad surface, shaping a strip from the leading edge to the trailing edge, indicating a large sliding area causing asperity contacts at HDI.
Figure 4.6 Optical micrographs showing a crashed slider due to silica formation at the HDI. (a)–(c): graphs showing the distribution of the contamination on the trailing edge region, middle part and leading edge region of the slider surfaces; (d): magnified view of the smears on the left side pad; (e) magnified view of the smears on the left side rail.

It is worth noticing that heavy contamination on the slider ABS is different from the liquid contamination discussed in the previous chapter because it didn’t cause high stiction at HDI, which is supported by the high AE rms peak values recorded during each slider’s tak-off. Sufficient liquid contamination might reduce direct solid-solid asperity head/disk contact and provide damping which results in lower AE rms amplitude. However, the siloxane induced contamination should affect the HDI differently: it aggravated the head/disk solid contacts and resulted in a crash.
Figure 4.7 presents both the S- and P-polarized OSA images of the CSS test area on the disk. From the bottom two images we can clearly see that the specific disk had two notable carbon wear tracks (the two linear light zones on the S-polarized image and the two linear dark zones on the P-polarized image), corresponding to the contact areas of the slider’s two side rails with the disk media. The outer track is believed to cause the final interface failure because it was wider and much deeper than the inner one. This observation correlated very well with the asymmetrical debris distribution on the slider surface.
Analysis of the debris contamination accumulated on the slider ABS was performed by TOF-SIMS. Three locations were analyzed as indicated in Fig. 4.8. Location 1 covers a small junction area between the left pad and the etch cavity area. Location 2 is located at the left rail surface. Whereas location 3 is in the etch cavity area and has an overlap with location 1. The TOF-SIMS ion mapping images of these three locations are presented in Fig. 4.9, 4.10 and 4.11 respectively.
Figure 4.9 TOF-SIMS ion mapping images for location 1
It is worth noting that the ions Co$^+$, Cr$^+$ and Ni$^+$ were detected at all three locations. This indicates that the contamination debris was comprised of disk magnetic materials. It is suggested that the carbon wear observed on the OSA images was so deep that it reached the disk magnetic layer. The Z-Dol lubricant was also detected. However, it was prominent only at location 1 and 2, indicating that the lube was picked up through head/disk contacts. Another thing we notice is the distribution of silicon related ions. The mapping images of these ions are listed in the third and the fourth rows of each figure. The ions 73.06 (SiC$_3$H$_9^+$), 206.854, 221.033 and 280.935 are all representative positive
ions originating from octamethyl cyclotetrasiloxane. Whereas, Si$^+$ and SiOH$^+$ originate from siloxane reaction products. The mapping images reveal that siloxane distributed differently from Si$^+$ and SiOH$^+$. It is observed that siloxane mainly distributed at the etch cavity area and only a weak signal of siloxane was detected on the pad and rail surface. By contrast, strong signals of Si$^+$ and SiOH$^+$ were detected at all three locations. Additionally, we noted that the distributions of Si$^+$ and SiOH$^+$ at location 3 formed a narrow strip. We concluded that Si$^+$ and SiOH$^+$ should originate from the side rail surface instead of the slider’s Si seedlayer based on the following information: (1) Si$^+$ and SiOH$^+$ distributed differently from Al$^+$ and Ti$^+$. Slider material itself also contains these ions. (2) The etch cavity area should be free of contact and no visible wear track was detected under optical microscope. (3) The strip formed by Si$^+$ correlates well with the narrow contact region on the side rail surface in width and spreading direction. Therefore, it is inferred that Si$^+$ and SiOH$^+$ at the etch cavity area were pushed or removed from the side rail surface through head/disk contacts. All these observations reveal the fact that siloxane contamination on the slider ABS had transformed to other products through chemical reactions initiated by the contact and friction. The reaction products must be SiO$_x$ as found in previous studies (Yamamoto et al. [1991], Raman et al. [2000], Xu et al. [2003]). We finally came to the conclusion based on the above analysis that the debris contamination on the slider ABS were made up of a collection of disk lubricant, disk magnetic materials along with reaction products (SiO$_x$) of siloxane.
Figure 4.11 TOF-SIMS ion mapping images for location 3

Figure 4.12 shows similar AE rms results obtained from another experiment performed under similar conditions with a center padded negative pressure slider. We label this test sample as sample B. As before, the AE rms peak values remained high during the entire CSS test. In the initial 5000 CSS cycles, the AE rms average value was very low and stable. After that, small peaks were observed now and then until around the 10850th cycle from where the AE rms average value started to increase rapidly and
continuously, indicating that the slider’s flyability was significantly degraded and continuous head/disk contacts were taking place.

Figure 4.12 (a) The AE rms peak value profile and (b) the AE rms average value profile and the magnified view of the data at the onset of failure recorded for the CSS test conducted in the presence of siloxane under 50°C/45%RH. The lube was pure Z-Dol. The results indicate that the interface finally went to a failure. Note: the slider used is a different type from the previous data set.
Figure 4.13 Optical micrographs showing the accumulation of silica debris on the surface of slider B. (a) graph of the trailing edge region; (b) graph of the leading edge region; (c) magnified view of the area at the step of center pad; (d) graph of (c) under dark field; (e) magnified view of leading edge area; (f) graph of (e) under dark field.
Figure 4.14 S- and P-polarized OSA images of the contact landing zone on disk B. Carbon wear tracks are also notable. The width of the images is 3mm (radial direction). Disk spinning direction: from right to left.

The optical micrographs of this slider ABS (Fig. 4.13) indicate that this slider surface was relatively cleaner than the previous one. However, it is also evident that fine filamentary materials and small particulates were present at the center pad and the taper area of the leading edge. These materials appear as a white feathery substance in a light microscope under dark field conditions. At the left side of the leading edge, the contamination materials formed a streak, indicating that the slider had been sliding on the disk which resulted in a notable wear track in the disk OSA images (Fig. 4.14). Chemical analysis of the contamination performed by TOF-SIMS also reveals the presence of silicon oxides, the reaction products of the organic siloxane. The number of CSS cycles before this interface failure took place was about twice as much as that of the failed sample described previously. It was apparent from the several sets of tests that there was a fairly large variation in the failure times of different head-disk pairs for tests performed under these conditions.
Generally speaking, all the test samples produced fundamentally the same results in terms of the propensity to produce failures during CSS tests in the presence of siloxane contamination.

4.3.3 Test results in the presence of siloxane using Z-Dol/X1-P-coated disks

Previous reports have indicated that disks lubricated with X1-P additive have vastly improved tribological performance over disks lubricated with only Fomblin Z-Dol. It was shown by Kasai et al. [1991] that Z-Dol decomposes because of its inferior stability towards Lewis acid surfaces such as alumina; whereas, X1-P maintains lubricant structural integrity by covering those Lewis acid surfaces and preventing the disproportionation reaction (Peretti et al. [1996]). In view of this, it was hoped to check the performance of disks lubricated with X1-P additive with specific attention to its propensity to produce SiO$_x$ on sliders.

Accordingly, CSS tests were conducted on disks lubricated with X1-P additive in the presence of organic siloxane under similar environmental conditions. For all disks used in this study, the amount of X1-P in the lubricant accounts for less than 1% in weight. A representative example of the AE rms results is presented in Fig. 4.15. For convenience, this test sample is labeled as sample C. It is observed that the AE rms average values increased slightly and were relatively stable during the first 8500 CSS cycles. However, after that the AE rms average data began to increase rapidly and continuously and finally stayed at around 2.6, indicating that the slider’s flyability was degraded and an interface failure occurred earlier than sample B. At the onset of failure, the AE rms amplitude
jumped to a higher level, indicating strong head/disk interactions. It is interesting to notice that the AE rms average data curve of sample C was much smoother than that of sample B. No small peaks were detected as signs of failure before the onset of failure. However, an interface failure was still inevitable. The optical micrographs (see Fig. 4.16) taken following the test reveal the accumulation of contamination on slider C, which is similar to that observed on slider B described in previous section.

Figure 4.15 (a) The AE rms peak value profile and (b) the AE rms average value profile recorded for the CSS test performed in the presence of siloxane under 50°C/40%RH. The lube was Z-Dol/X1-P mixture. The results indicate that the interface finally went to a failure.
Figure 4.16 Optical micrographs showing the accumulation of silica debris on the surface of slider C. (a) graph of the trailing edge region; (b) graph of the leading edge region; (c) magnified view of the area at the step of center pad; (d) graph of (c) under dark field; (e) magnified view of leading edge area; (f) graph of (e) under dark field.
Figure 4.17 S- and P-polarized OSA images of the contact landing zone on disk C. Carbon wear tracks are observed. The width of the images is 3mm (radial direction). Disk spinning direction: from right to left.

Figure 4.17 presents the S- and P-polarized OSA images of the contact landing zone on disk C. As we can see, many contamination smears were observed along the CSS tracks due to lubricant pooling and wear debris accumulation. However, the wear rate was much lower than that of sample B as analyzed by OSA. This might be due to fewer CSS cycles conducted on this sample and the termination of the test at the initial stage of the interface failure. Additionally, the observed red smears on the P-polarized OSA image indicate lubricant degradation as suggested by OSA.

The above experiment results led us to the conclusion that the X1-P additive in disk lubricant does not necessarily prevent interface failures arising from the formation of silicon oxides at HDI in the presence of organic siloxane.
4.4 Discussion

In this study, CSS tests were conducted on disks lubricated with and without X1-P additive in the presence of organic siloxane. In both cases, the tests led to the formation of abrasive silicon oxide particles on slider surfaces. The formation of this product resulted in rapid HDI failure with a well defined wear track on disk contact zone signifying failure. Here, we’ll discuss the failure mechanisms in more detail.

4.4.1 Formation of SiO$_x$ at HDI

The degradation process of PDMS has been examined before by several researchers (Balykova and Rode [1969], Kucera and Lanikova [1961]). It was concluded that in the presence of air and heat (with temperature up to 300$^\circ$C), the PDMS undergoes a depolymerization process or an oxidative process where the methyl groups are oxidatively replaced with cross linking –Si-O-Si bonds. The simplified view of this process is that PDMS in the presence of oxygen and heat leads to the formation of silicon oxide at the HDI. The temperature needed for this reaction may be achieved due to flash temperature arising from disk asperity contacts with the flying slider. The study conducted by Paul H. Kasai and Frederick P. Eng [2000] proposed two prerequisites for the formation of silicon oxide in disk environment: (1) the presence of siloxane; (2) the generation and presence of the fluorocarboxylic acid (degraded lubricant), which would repolymerize siloxane back into PDMS at the HDI area. Thus, lubricant degradation plays a great role in silicon oxide formation in disk environment.
In the past, quite a lot of research work has been dedicated to the studies of the decomposition and degradation of disk lubricant (Kasai et al. [1991], Wei et al. [1998], Kasai [1999], Kasai and Raman [2003], Chen et al. [2000], Bhatia [1999], Chen et al. [2000]). Based on previous studies, the decomposition of Z-Dol lubricant is initially caused by friction/thermal actions which cleave the main chain of the Z-Dol molecule at the linkage (C-O-C) that has the weakest bond energy. If Al₂O₃ is present, the decomposition products react with Al₂O₃ to produce a strong Lewis acid, AlF₃, which functions as a catalyst, and then rapid catalytic decomposition of Z-Dol lubricant occurs on the AlF₃ surface. The catalytic decomposition mechanisms of Z-Dol on the Al₂O₃-TiC surface are summarized as follows (Wei et al. [1998]):

1. Friction shear breaks the main chain of Z-Dol at weak bond sites and forms CF₂O:

   \[ -O-\ldots CF_2\ldots O-\ldots CF_2\ldots O-\ldots CF_2- \]

   \[ \rightarrow CF_2 = O + CF_2 - CF_2 - O - . \]

2. Partial CF₂=O reacts with Al₂O₃ and forms AlF₃:

   \[ Al_2O_3 + 3CF_2O \rightarrow 2AlF_3 + 3CO_2. \]

3. Rapid decomposition reactions along the main chain of Z-Dol take place on the AlF₃ surface, and form methoxy (CF₃-O-), ethoxy (CF₃CF₂-O-) compounds and acyl fluoride (R-CF=O):

   \[ R-CF_2-O-CF_2-R \rightarrow R-CF = O + CF_3 - R \]

   \[ (CF_3-R: CF_3-O-, CF_3CF_2-O-). \]

4. The resulting fluorocarbonyl end-group, if it is adjacent to a CF₂ unit, would readily convert to a strong acidic fluorinated carboxylic acid on encounter with a water molecule.
and generate HF. HF can react with Al\textsubscript{2}O\textsubscript{3} and also produce AlF\textsubscript{3}, which expedites the catalytic decomposition of Z-Dol.

\[ R - CF_{2} - CF' = O + H_{2}O \rightarrow R - CF_{2} - COOH + HF \uparrow . \]

\[ Al_{2}O_{3} + 6HF \rightarrow 2AlF_{3} + 3H_{2}O . \]

Once AlF\textsubscript{3} is produced, coordinated bonds (O→Al) between PFPEs and AlF\textsubscript{3} are formed by electron transfer from O to Al atoms, because the O atoms in PFPEs have unshared electron pairs and the Al atoms have empty orbitals. Strong coordinated bonds can be formed on the AlF\textsubscript{3} surface for those PFPEs without branch groups, owing to no spatial hindrance. Subsequently rapid catalytic decomposition of PFPEs takes place on the AlF\textsubscript{3} surface, which involves an intramolecular disproportionation reaction and results in the formation of O=CF-, CF\textsubscript{3}-O- and CF\textsubscript{3}CF\textsubscript{2}-O- groups (Wei et al. [1998]).

Kasai and Eng [2000] examined the effect of Z-DIAC (Fomblin Z with carboxylic acid end groups) on D4 (Octamethyl cyclotetrasiloxane). When D4 was heated with 10 wt. % Z-DIAC at 120°C for 24h with stirring, PDMS formed. This result clearly supports the plausibility of the PDMS formation at the HDI in the presence of degraded lubricant.

Figure 4.18 shows TOF-SIMS narrow mass spectra around 27, 46, 48 and 67 amu at location 3 on slider A. The positive ions Al\textsuperscript{+}, AlF\textsuperscript{+}, Ti\textsuperscript{+} and TiF\textsuperscript{+} are clearly observed. This result is a good evidence of the formation of metal (Al, Ti) fluorides by the reaction of slider materials (Al\textsubscript{2}O\textsubscript{3}-TiC) with the PFPE lubricant (Numata et al. [2003]). As a result, we can conclude that catalytic decomposition of the Z-Dol lubricant occurred at these Lewis acid sites and the degraded lubricant made it possible for the conversion from siloxane to silicon oxides.
Chapter 4
Effect of Organic Siloxane at Head/disk Interface

All the commercial sliders (Al$_2$O$_3$-TiC) used in this study are coated with a thin layer of diamond-like carbon at ABS. The carbon coated ABS makes up a very small fraction of the whole slider. One might say that these sliders should have very low reactivity. However, the tribochemical reactions can be stimulated by friction. Under the action of friction, the protective carbon overcoat on the alumina surface could be destroyed through wear, and a fresh surface is exposed. Meanwhile, surface deformation produces many defects. Since the deformed surfaces have very high reactivity, the formation rate of Al$_2$O$_3$ and its catalytic activity become greatly increased (Wei et al. [1998]). Moreover,
friction also provides the heat needed for the conversion from siloxane to PDMS and the oxidation of PDMS. To prove these hypotheses, continuous flying tests, where friction or head/disk asperity contacts are vastly decreased, were also performed at disk OD (Outer Diameter) for disks lubricated with Z-Dol without X1-P additive in the presence of siloxane under similar environmental conditions (refer to Appendix C). Acoustic emission sensor was also used to monitor the potential head/disk contacts. The AE response, which was quite stable during a long test period, suggested that the sliders maintained good flying characteristics and no interface failure was observed. Close examination of the slider also revealed that the slider surfaces were free of silicon oxide debris. All these results lend good support to the above hypotheses that friction plays a great role in lubricant decomposition and silicon oxide formation in disk drive environment.

4.4.2 Effect of X1-P additive

Our tests using Z-Dol/X1-P-coated disks also generated similar interface failures due to silica formation at HDI as observed in tests using pure Z-Dol-coated disks. One might say that X1-P doesn’t improve lubricant system reliability and durability. This is not the truth.

X1-P, partially fluorinated hexaphenoxy cyclotriphosphazene, is advocated as an additive to PFPEs in disk file applications. Great attention has been paid to the remarkable improvement in the performance of disk lubricant with X1-P additive. It has been shown that the addition of X1-P to PFPEs, Z-Dol in particular, results in a
significant increase of the durability of the system (Zhao [1999], Perettie [2003]). However, there are two important aspects of their results we need to note here. One is that these results were obtained under normal environmental conditions free of siloxane contamination. The other one is that disks used in these studies had more than 5 wt% or up to 10 wt% X1-P in lubricant. By contrast, the lubricant of the disks used in our studies had less than 1 wt% X1-P additive and the tests were performed in a harsh environment where chemical vapor of siloxane might be saturated in the atomosphere.

C. Mathew Mate et al. [1998] investigated the interaction of X1-P with disk and slider surfaces and tried to find out how X1-P acts to improve the disk lubrication. They found that X1-P improved the disk lubrication in two ways. One way is that X1-P molecules tend to attach themselves to sites on the carbon overcoat surface for the purpose of lowering surface energies. They prevent the Z-Dol molecules bonding to those sites and mediate their interaction with the disk so as to increase their mobility. The other way is that X1-P is able to passivate the catalytic Lewis acid sites on slider and disk surfaces, thus preventing catalytic decomposition of the PFPEs lubricant. The chemical formula of X1-P is given as follows:

\[
\begin{array}{c}
\text{P} \quad \text{O} \\
\text{N} \quad \text{N} \\
\text{P} \quad \text{O} \\
\end{array}
\]

\[
\begin{array}{c}
\text{P} \quad \text{N} \\
\text{N} \quad \text{P} \\
\text{C}_\text{F}_3
\end{array}
\]

It is demonstrated that the efficacy of X1-P as an additive to Z-Dol results from the nucleophilic nature of the hexaphenoxy cyclotriphosphazene ring, which acts as a Lewis base capable of neutralizing Lewis acids.
V. Raman et al. [2000] proved that flyability tests for disks lubricated with pure X1-P resulted in satisfactory flyability with no signs of interface failure after long run times in the presence of siloxane contamination. It is clear that X1-P is able to avert the catalytic degradation process and has excellent thermal oxidative stability in the presence of slider materials like Al$_2$O$_3$-TiC. A simplified view is that in the absence of degraded lubricant, the reaction process is curtailed and no abrasive silicon oxides debris forms at the HDI. Thus, it may be possible to suppress the formation of silicon oxides at HDI by increasing the amount of X1-P in the lubricant system. However, this brings us another problem – micro-phase separation between X1-P and Z-Dol due to their insolubility to each other as reported recently (Hara et al. [2001], Kang et al. [1999]). Therefore, it is necessary to develop new types of lubricant system with a view of achieving long term reliability and durability of HDI. More recently it has been found that ZDPA (Fomblin Z terminated with dipropyl amine end groups), compared to a Z-Dol/X1-P mixture, exhibits good durability and reliability against organic siloxane (Kasai and Raman [2002]). When disks were coated with ZDPA, the formation of silicon oxide was completely suppressed, even in drives saturated with dimethyl siloxane oligomers.

4.5 Summary

The principal experimental information generated in this study can be briefly summarized as follows:

(1) CSS tests in the presence of organic siloxane with disks lubricated with Z-Dol lead to the formation of “SiO$_x$” on slider surfaces. The formation of this product leads to HDI
failures with a well defined wear track on the disk surface signifying failure.

(2) The failure process was characterized by a rapid and continuous increase in AE rms average data followed by drastic changes. The increase in AE rms average data and its sustained high values indicate that the slider’s flyability had been vastly degraded and continuous slider/disk contacts occurred at flying conditions. The application of AE techniques in characterizing this kind of failure is relatively new in published papers.

(3) Chemical identification of the contamination on the slider surface using TOF-SIMS revealed different distributions of reaction products and organic siloxane, essentially indicating that siloxane was converted to silicon oxides through friction/contacts at contact area. This is a new application of TOF-SIMS in characterizing organic siloxane. Detection of metal (Al, Ti) fluorides on the slider surface by TOF-SIMS supports the view that catalytic decomposition of Z-Dol lubricant had occurred.

(4) Similar tests for disks lubricated with a Z-Dol/X1-P mixture also resulted in interface failures which were attributed to the very low percentage of X1-P additive in the lubricant system.

(5) Continuous-flying tests performed on disks lubricated with Z-Dol didn’t result in interface failure in a test period up to 4 days and the slider surfaces were free of SiO_x debris. These new results suggest that friction/contacts play an important role in lubricant decomposition and the formation of SiO_x at HDI.

(6) It was reported in the literature that X1-P based lubricants do not undergo catalytic degradation. Our results also suggest that the reaction of PDMS to form SiO_x on sliders is dependent on the type of the disk lubricant and the lubricant degradation is an important factor in causing the formation of silica at HDI. Thus, in order to achieve
long term durability and reliability against siloxane contamination, it is necessary to develop new types of lubricant system that can hinder catalytic degradation effectively.

(7) AE measurements are reliable and can be utilized to detect HDI failures and offer us a method to monitor long term stability of the slider flying characteristics.
CHAPTER 5

Conclusions

The focus of this project is to study chemical contamination at HDI and investigate its effects on interface performance. Two kinds of chemicals, hydrocarbon and siloxne, were selected as our main research subjects in this study due to their common release from the outgass of disk drive components. Throughout our studies, AE sensors were used to evaluate HDI performance and help us investigate the failure mechanisms. It is found that AE response is sensitive to HDI interactions and provides useful insights into understanding the flying characteristics of sliders.

5.1 Effect of hydrocarbon at head/disk interface

Hydrocarbon contamination at HDI was studied by continuously introducing hydrocarbon chemical vapor into the drive operating environment to evaluate the effect of hydrocarbon on interface performance. An experiment setup was developed for this purpose and three kinds of chemicals were used: hexadecane, octadecane and DOP. Three groups of tests were designed and conducted: A. CSS tests without rest period in the middle; B. CSS tests with rest period in the middle; C. continuous-flying tests followed by a 12-hr rest period at the contact landing zone. All tests were performed under ambient environmental conditions (24-28°C/40-55%RH). Following each test, slider and disk
surfaces were examined and analyzed using optical microscope and OSA, respectively. The contamination on slider surfaces were analyzed using TOF-SIMS or XPS.

Head smear, disk wear, high rest stiction and fly stiction were all observed in our experiments. Our test results show that the presence of hydrocarbon has detrimental effects on interface performance but it doesn’t necessarily lead to interface failures. The slider’s flying characteristics play a great role. Existence of intermittent head/disk contacts is a key factor that initiates and accelerates interface failures. Hydrocarbon affects the HDI by increasing stiction and aggravating contamination on the slider surface. However, hydrocarbon does not necessarily induce head smear and disk wear. Head smear and disk wear are caused by lubricant pick-up or lubricant depletion. Disk lubricant, if picked up by the slider and accumulated on the slider surfaces, can smear the head, decrease fly height and cause high rest stiction or fly stiction, and finally lead to interface failure.

5.2 Effect of organic siloxane at head/disk interface

In this study we have investigated the effect of organic siloxane at head/disk interface. CSS tests were performed using Z-Dol- or Z-Dol/X1-P-coated disks in the presence of siloxane contamination under various environmental conditions (30-60°C, 25-50%). Our experiment results suggest that the HDI exhibited good CSS durability in the absence of siloxane contamination. By contrast, in the presence of siloxane contamination interfaces failures were observed. The failure process was characterized by a rapid and continuous increase in AE rms average data followed by drastic changes. The
increase in AE rms average data and its constantly high values indicate that the slider’s flyability had been vastly degraded and continuous slider/disk contacts were taking place at flying conditions. Analysis by TOF-SIMS on chemical contaminations on head surfaces reveals that the siloxane was converted to silicon oxides through friction/contacts at contact area, which resulted in HDI failures. Therefore, our conclusion is siloxane is harmful to HDI. It can fail the interface by converting itself to abrasive SiO$_x$ at the HDI and causing severe disk wear.

5.3 Future work

Due to the limitations of time and experiment conditions, the following areas are not covered in this study or need further studies.

1. In chapter 3, the different effects of each individual chemical on HDI and their mechanisms were not comprehensively analyzed and studied. This needs further studies to get better understanding.

2. The root cause for X1-P droplets picked up by sliders during the continuous-flying tests was not clear as mentioned in chapter 3. Was it due to micro-phase separation between X1-P and Z-Dol or attributed to the effect of hydrocarbon? Further studies are required to understand this more completely.

3. The effect of X1-P on improving lubricant durability against siloxane contamination was not fully evaluated in chapter 5. New type of lubricant system needs to be developed to achieve long term reliability and durability of HDI.
(4) In all my experiments of this study, the sources of contamination are all pure chemicals purposely introduced into the test environment. The effect of outgassing from real materials adopted in hard disk drives, as another source of contamination, needs to be studied so that our studies are more practical and meaningful.
References


10. TriboScan 3000 Software from Candela Instruments.


Appendix A

IDEMA Standards

Microcontamination

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<th>Transfer Modes</th>
<th>Mechanism</th>
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*BP means a chemical breakdown product.

**Proven means that either experiments were preformed or source removed and problem was eliminated.

Speculative means that focused attention on the contaminant reduced the problem.

Failure Mechanisms: Corrosion, Stiction, HDI, Attenuated Fly Height, Thermal Asperities, Disk Damage.
Appendix B

Outgass Compounds from Different Disk Drive Components

Adhesive 1

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**Appendices**
* All data are taken from the report of DSI internal project: Determination of outgassed contaminants of drive components.
Appendix C

Results Obtained in Continuous-Flying Tests in the Presence of Organic Siloxane

I. The following figures from Appendix D.1 to Appendix D.3 reveal the results for one sample.

Figure Appendix D.1 The AE rms average value profile recorded for a continuous flying test (96 hrs) conducted in the presence of siloxane under 50°C/40%RH. The lube was pure Z-Dol. The results suggest that the interface was stable.
Figure Appendix D.2 Optical micrographs of the slider surface after 96 hrs continuous flying test. No silica particles were detected on the slider surface.

Figure Appendix D.3 S- and P-polarized OSA images of the flying track on disk surface. The width of the images is 3mm (radial direction). Disk spinning direction: from right to left. The fly track due to lube modulation was observed, but no carbon wear was detected.

II. The following figures from Appendix D.4 to Appendix D.6 reveal the results for another sample.
Figure Appendix D.4 The AE rms average value profile recorded for another continuous flying test (66 hrs) conducted in the presence of siloxane under 50°C/40%RH. The lube was pure Z-Dol. The results suggest that the interface was also stable.

Figure Appendix D.5 Optical micrographs of the slider surface after 66 hrs continuous flying test. No silica particles were detected on the slider surface.
Figure Appendix D.6 S- and P-polarized OSA images of the flying track on disk surface. The width of the images is 3mm (radial direction). Disk spinning direction: from right to left. Lube modulation was observed. There was no carbon wear.
Appendix D

Graphical User Interface of Instrumentation Developed by Labview

1. GUI for CSS test profile
2. GUI for continuous flying test profile

* The plot in blue indicates the AE rms signal; The curve in white is the AE rms average data profile; The red and green curves plot the temperature and relative humidity profiles, respectively.