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**EFFECT OF H CONTENT IN THE CARBON OVERCOAT
ON THE CORROSION RESISTANCE OF THIN FILM MAGNETIC DISKS**

A Thesis

Presented to

The Faculty of the Department of Materials Engineering
San Jose State University

In Partial Fulfillment
of the Requirements for the Degree
Master of Science

By

Katesuda Chinmeteepitak

May 1996

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ABSTRACT

EFFECT OF H CONTENT IN THE CARBON OVERCOAT ON THE CORROSION RESISTANCE OF THIN FILM MAGNETIC DISKS

by Katesuda Chinmeteevitak

Corrosion resistance of lubricated and unlubricated thin film magnetic disks containing 0%, 16%, and 36% hydrogen in the carbon overcoat were compared. The disk samples were exposed in a mixed flowing gas system containing 2.6 ppb Cl_2 , 323 ppb SO_2 , 483 ppb NO_2 , and 37 ppb H_2S at 30°C and 80%RH. A light scattering technique was utilized to measure the extent of corrosion. Electrochemical measurements of the disks were also conducted. For the lubricated samples, atmospheric and electrochemical results agree that the higher H content disks have a better corrosion resistance than the lower H content ones. Atmospheric corrosion on unlubricated disk samples shows better corrosion resistance than the lubricated group, which is inconsistent with the electrochemical measurement result which shows better corrosion resistance for the lubricated group.

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CHAPTER 1

Introduction

1.1 Background

Among three magnetic recording devices in use today, the hard drive is the primary data storage device used in microcomputers. Advantages of the hard drive are an extremely fast and random data access compared to tape and a high storage capacity compared to flexible disk. Because fast access to stored data is a prime requisite for the hard drive, its disks are designed to rotate at 3000-5000 revolutions per minute. The read-write magnetic heads aerodynamically fly on a cushion of air created by disk rotation, preventing damage and data loss from physical head to disk contact. Data signals are obtained from magnetic fields traveling through the spacing between heads and disks. This spacing or "flying height" is required to be as small as possible in order to maximize the quality of read and write signals. A typical "flying height" is around 2 to 4 microinches (0.05 to 0.1 μm). Future designs have flying height spacings approaching zero, nearly contacting the disk surface.

One major cause of hard disk drive failure is disk corrosion. The problem of disk corrosion occurs when corrosion product asperities forming on the disk surface are large compared to the head flying height. The heads pick up

debris, leading to stiction problems and recording signal interference. Severe corrosion causes the head to crash into its disk, resulting in drive malfunction and data loss. As a future trend for higher storage capacity and lower flying height, the disk corrosion problem is becoming a critical issue.⁽¹⁾

The hydrogenated carbon overcoat is a new trend in hard disk design due to its diamond-like properties of high hardness and wear resistance. The high electrical resistivity of this type of film is also interesting from a corrosion point of view. A high resistivity film will reduce the amount of electron flow in the electrochemical processes involved with the underlying metallic material, and therefore improve disk corrosion resistance. Should it enhance the corrosion resistance of the hard disk, the benefit of using the hydrogenated carbon overcoat will be two-fold.

The hard disk, more properly called the "thin film magnetic recording disk," is generally composed of an aluminum alloy substrate serving as the main structure. Nickel phosphorous (NiP) is then electroless plated to give strength and hardness to the substrate structure. The NiP layer is textured to increase surface roughness, necessary for decreasing stiction between head and disk during take-off when the disk begins to spin. Textured thin film disk

surface roughness is around 37 to 48 nm from peak to valley, compared to 16 nm for the untextured disk.⁽²⁾ A magnetic layer, usually a cobalt alloy approximately 300Å thick, is then sputtered onto the NiP layer and serves as the data storage layer. Around 150-300Å of carbon is deposited over this magnetic layer to increase wear resistance and prevent physical damage to the underlying magnetic layer. Lastly, around 20Å of perfluoropolyether lubricant is dip coated onto the carbon layer for enhanced head flyability and wear resistance. A common perfluoroalkyl polyether lubricant has the following chemical structure:

Typical disk manufacturing process and disk structure are shown schematically in Figures 1 and 2, respectively.

Figure 1. Typical disk manufacturing process.

Figure 2. Schematic of cross-sectioned thin film magnetic recording disk, showing disk structure, layer thickness, and magnetic spacing.

Because strong recording signals are required, not only must there be a low physical spacing between head and disk, but also thin magnetic, carbon, and lubricant layers are needed in order to minimize the magnetic spacing. As shown in Figure 2, magnetic spacing is composed of mechanical spacing or "flying height," lubricant thickness, carbon thickness, and approximately half of the magnetic layer thickness. This aggregate explains why the carbon layer thickness is required to be as thin as possible while still serving its wear resistance function.

1.2 Carbon film properties and structure

Carbon occurs naturally in both crystalline and amorphous forms. Diamond and graphite are two major allotropes of the crystalline form. Other types of carbon, such as charcoal and carbon black, are amorphous forms consisting of the short range order, imperfect graphitic structures.

Diamond is a metastable phase based on the crystalline face centered cubic structure. Each of the four valence electrons of the carbon atom is arranged in a tetrahedral sp^3 hybrid orbital which forms a strong sigma bond with adjacent atoms. Sigma type bonding gives diamond a wide 5.5 eV band gap resulting in high electrical resistivity and optical transparency.

Graphite is the stable form of crystalline carbon; its crystal structure is hexagonal. Three out of four valence electrons are arranged in sp^2 planar trigonal hybrid orbital forming sigma bonds in the basal plane. The fourth electron is in the p_z orbital, which forms weak pi bonds with the adjacent atom. These pi bond electrons are non-localized and thus account for the good electrical conductivity of graphite.

There are various ways to deposit thin film carbon. Some of these methods include plasma enhanced chemical vapor deposition (PECVD), ion beam deposition and sputter deposition. In a magnetic recording application, sputtering is most desirable due to its high volume processing capability.⁽³⁾ These deposited films are amorphous carbon. X-ray diffraction analysis suggests that an amorphous carbon structure consists of short range order of the more stable sp^2 bonding in graphite.⁽³⁾ A wide range of these film properties can be obtained depending on deposition conditions. For example, increasing sputtering power from 0.25 to 25 W/cm² results in the reduction of density from 2.1 to 1.6 g/cm³, electrical resistivity from 2500 to 0.2 ohms-cm, and hardness from 2400 to 740 kg/mm².

With the unique properties of diamond being high hardness, high electrical resistivity, and chemical inertness, various methods to deposit a diamond-like carbon

film have been developed. Either high energy ions or the presence of hydrogen as hydrocarbon or hydrogen gas in a deposition chamber has been shown to enhance the sp^3 type bonding of diamond.^(3,4) High energy ions create spikes of pressure and temperature allowing the metastable bonding of diamond to form. The presence of hydrogen stabilizes the dangling bonds of surface carbon and induces sp^3 bonds; carbon later replaces the hydrogen which leaves the surface as H_2 .⁽⁴⁾ This group of amorphous hydrogenated carbon is designated by a-C:H.

It is not well understood how hydrogen atoms incorporate into the carbon structure. Some atoms could be present interstitially in the structure while others are bonded to the carbon as evidenced by the Fourier Transform Infrared (FTIR) spectrum in the 2900 cm^{-1} region.^(5,6)

Various research has been conducted on the properties of hydrogenated carbon as a function of hydrogen content.⁽⁶⁻¹⁰⁾ It has been shown that increasing hydrogen pressure in the sputtering chamber and hence hydrogen content in the carbon film results in an increase in the sp^3/sp^2 bonding ratio. The sp^3 bonding can be detected using Raman Spectroscopy by the shift of the G (around 1575 cm^{-1}) peak and the change in intensity ratio between D (around 1365 cm^{-1}) and G peaks, as shown in Figure 3. The shifting of the G peak to a lower frequency, for example, from 1575 cm^{-1} to

1555 cm^{-1} , indicates higher sp^3/sp^2 ratio. The lower intensity ratio of D to G peaks, for example $I(\text{D})/I(\text{G})$ from 1.4 to 0.7, indicates a reduction in number and size of graphitic microdomains.

Figure 3. Schematic of Raman spectra of carbon film at 0% and 5% hydrogen gas in the sputtering chamber.

Increasing the sp^3 to sp^2 bonding ratio by adding hydrogen to the carbon structure, results in an increase in wear resistance and electrical resistivity. For example, wear resistance is measured by the number of scratches per millimeter on the disk surface after a drag test. Marchon

et al.⁽⁶⁾ found that the number of scratches reduced from 15 to 5 per millimeter when hydrogen gas in the sputtering chamber increased from 0 to 5 percent. Lee *et al.*⁽⁷⁾ found that increasing hydrogen content in the carbon from 4 to 28 percent resulted in an increase in the electrical resistance from 1 to 10^6 ohms. It was also found that at very high H content, i.e. greater than 20% H₂ in the sputtering chamber, the carbon film becomes soft and polymer-like due to the more C-H bonds in the structure.^(5,6)

The presence of hydrogen also increases the optical band gap of the carbon film. Compared to the value of -0.04 eV band gap in graphite, and 0.4 to 0.7 eV in evaporated a-C, hydrogenated carbon has the values of band gap range from 1.5 to 4.0 eV, which is evidently higher than the non-hydrogenated evaporated a-C. However, the density of a-C:H film is quite low, i.e. 1.4 to 1.8 g/cm³, compared to 2.0 in evaporated a-C, 2.2 in graphite, and 3.5 g/cm³ in diamond.⁽¹¹⁾

Void network is known to be a structural imperfection of amorphous films prepared by electrodeposition, evaporation, and sputtering. Carbon prepared by sputtering contains a network of pipe-like voids which do not penetrate through the carbon layer thickness.⁽¹²⁾ The size of these voids was found to be 10 - 20Å. The voids form due to trapped gases, with the probability of formation depending upon substrate surface roughness. The void formation

mechanism is believed to be the same as that of amorphous germanium.⁽¹²⁾ A TEM study was attempted by Sides⁽¹³⁾ to view intrinsic pore structures. At 100,000X magnification, discontinuous gaps about 5 nm by 100 nm were seen on a carbon sample prepared at a sputtering power of 3 W/cm² and an Argon pressure of 1.4 Pa.

In a hydrogenated carbon film where microdomains of sp³ and sp² regions exist, high intrinsic compressive stress was found in the diamond-like (sp³) region, with the stress decreasing toward the graphite-like (sp²) region.⁽¹⁴⁾ Because tensile stress in a film leads to crack initiation, high compressive stress in a high hydrogen content film provides a better film quality and lower the susceptibility to cracks and pores.

1.3 Disk corrosion mechanism

Corrosion is the destructive result of a chemical reaction between metals and their environments. When metals are extracted from minerals into engineering uses, the pure metal form is not stable. Corrosion is the reverse process that converts metals back into their naturally occurring oxide forms.

Most corrosion processes are electrochemical in nature. Electrons are exchanged from an anode, where the oxidation reaction occurs, to a cathode, where reduction takes place. The driving force for the process is the free energy change,

ΔG , which is associated with an electrochemical potential, E , at equilibrium through a thermodynamic relationship^(15,16)

$$\Delta G = -nFE \quad (1)$$

where n is the number of electrons exchanged in the reaction and F is Faraday's constant, 96500 coulombs per equivalent.

The electrochemical potential, E , is the combination of the standard half cell electromotive force (emf) potentials of both oxidation and reduction reactions. The standard emf series given in most corrosion textbooks are at equilibrium having electrolyte concentration at unit activity, which is one gram atomic weight of metal ions per liter. In the system where the electrolyte is not at unit activity, the potential can be determined using Nernst's equation:

$$e = e^\circ + 2.3RT/nF \log(a_{\text{oxid}}/a_{\text{red}}) \quad (2)$$

where e is the half cell emf potential, e° is the standard half cell emf potential, R is the gas constant, T is absolute temperature, a_{oxid} and a_{red} are the activities of oxidized and reduced species.

The Potential-pH or Pourbaix diagram is a thermodynamic tool that predicts stable phases in various conditions of an aqueous electrochemical system at equilibrium. One can use this plot to predict the spontaneous direction of a reaction, estimate the composition of corrosion products, and predict environmental changes that will prevent or reduce corrosion. The diagram for each metal is constructed

using the Nernst's equation and solubility data for constituent compounds. Pourbaix diagrams of most metals can be found in ref. 17. Figures 4(a) and 4(b) are simplified schematics of the diagrams for cobalt and nickel, respectively.

Thermodynamic information suggests reaction probabilities but gives no indication of corrosion rates; corrosion rates can be determined by the exchange current density. At equilibrium, when the rate of oxidation, r_{oxid} , is equal to the rate of reduction, r_{red} , the relationship between the corrosion rate, r , and current density, i_0 , is given by

$$r = r_{\text{oxid}} = r_{\text{red}} = i_0/nF \quad (3)$$

(a)

(b)

Figure 4. Schematic of Pourbaix diagrams for (a) cobalt and (b) nickel

1.3.1 The electrochemical method

Corrosion rates can be determined in practice by electrochemical measurement using a Tafel plot. The plot is obtained from the data of either anodic or cathodic polarization measurements. Cathodic polarization is preferable due to the ease of the experimental measurement. An electric circuit diagram for a cathodic polarization measurement is similar to that shown in Figure 5. Three electrode probes are used. The unpolarized or reference electrode (Ref) measures the potential of a metal sample called the working electrode (WE). Cathodic current is supplied to the working electrode via a third counter or auxiliary electrode (Aux), usually a material having a high corrosion resistance such as platinum.

Figure 5. An electric circuit diagram for a cathodic polarization measurement.

For a system of metal M immersed in an air-free acid solution, corrosion potential (E_{corr}) and corrosion rate (i_{corr}) are defined by mixed potential theory as shown in Figure 6(a). Oxidation of metal M is represented by the straight line with the positive slope, the reduction process of hydrogen with the negative slope. Before any current is applied, the reference electrode measures the corrosion potential, E_{corr} . When a cathodic current is applied, the excess electrons cause the potential to shift from the previous corrosion potential toward a more negative potential ($E_{\text{corr}} - E_c$). The anodic current is reduced from i_{corr} to i_a , while the cathodic current is increased to i_c as shown in Figure 6(a). The difference between i_c and i_a is simply the applied current, i_{app} ; $i_{\text{app},c} = i_c - i_a$. The plot of potential and i_{app} on a semilog scale gives a polarization curve similar to that shown in Figure 6(b). The curve becomes linear at approximately 50 mV beyond the corrosion potential, E_{corr} . This linear region is called Tafel region. Extrapolation of this linear region to the point where it crosses the E_{corr} yields the corrosion current (i_{corr}). Similarly, the anodic polarization curve can be obtained by the equation $i_{\text{app},a} = i_a - i_c$.

(a)

(b)

Figure 6. (a) Mixed potential diagram for metal M in acid solution, and (b) calculated Tafel plot.

1.3.2 Atmospheric corrosion

During atmospheric corrosion, chemical reactions can occur because of moisture adsorption. When exposed to moisture, any solid is capable of adsorbing moisture from the vapor phase.⁽¹⁸⁾ The adsorption phenomena occurs to reduce the surface free energy of the solid surface. The extent of adsorption depends on pressure, temperature, surface area, and surface reactivity. The forces that bring gas phase molecules to the solid surface are London's dispersion and short range repulsive forces. Gas molecules become attached to the solid surface as liquid phase with these physical forces and can no longer freely move in three dimensions. The further away from the solid surface, the weaker the molecule attaches. At some distance from the surface, when the heat of vaporization is greater than heat of adsorption, this phenomenon stops. Adsorption preferentially occurs at the pores or cracks, where more atoms encircle the gas phase molecules than the open surface.⁽¹⁸⁾

On ionic and metallic surfaces, electrostatic forces also enhance adsorption of the polar water molecules.⁽¹⁸⁾ Therefore, a more metal-like surface of sp^2 type bonding in the amorphous carbon should adsorb more moisture than the greater sp^3 type bonding of diamond-like carbon. Moisture adsorption on DC magnetron sputtered carbon, which contains

more sp^2 type bonding (4% H content), was compared with plasma enhanced chemical vapor deposition (PECVD) carbon containing more sp^3 type bonding (27% H content) by Goodson.⁽¹⁹⁾ No significant difference in water adsorption between the two types of carbon was found using the Quartz crystal microbalance. Both types of carbon film were found to adsorb approximately two monolayers (or 0.6 nanometers) of water at 70% RH.

Smallen et al.⁽²⁰⁾ studied water adsorption on disks at different H content using ellipsometry. It was found that on both lubricated and unlubricated disks, water adsorption increases as hydrogen content increases (0% to 30% C_2H_2 in the sputtering chamber). The author also found that the adsorption is independent of lubricant type and thickness.

Adsorbed water and dissolved contaminant gases provide basic conditions for an electrochemical process. Atmospheric cobalt corrosion and the effect of urban gases was studied by Rice.⁽²¹⁾ The gases studied were SO_2 , Cl_2 , NH_3 , NO_2 , H_2S , and O_3 . Micrographs from transmission electron microscope (TEM) suggested that moisture adsorbs on a complex oxyhydroxylated cobalt surface in clusters. Cl_2 first reacts with these clustered water molecules forming HCl . Later cobalt chloride is formed. Cobalt chloride is hygroscopic and stabilizes the water molecule clusters. SO_2 then dissolves in these stabilized water clusters and forms

sulfuric acid. For the gases studied, SO₂ and Cl₂ were found to have the greatest effect on cobalt corrosion rate. At a fixed water vapor pressure, the cobalt corrosion rate was also found to increase as the temperature decreased, corresponding to the amount of adsorbed water.

1.3.3 *Thin film disk corrosion*

Numerous studies have been conducted on thin film disk structure corrosion.⁽²²⁻³³⁾ Disk corrosion has been found to occur through pores in the carbon overcoat,^(13,22-25) or pores related to the texture lines on the Ni-P layer.⁽²⁶⁾ Brusic et al.⁽²²⁾ has shown that corrosion products are localized on a carbon coated disk, and uniform on an uncoated one. Kawashima⁽²³⁾ showed that the anodic polarization curve of a disk sample is similar to that of the metallic underlayer, while the cathodic curve had the same shape of the carbon material. These results suggest that corrosion occurs through pores in the carbon overcoat.

Corrosion asperities most likely occur along texture lines or defects.^(22,26) Corrosion products were found to contain large amount of cobalt, nickel, oxygen, and some contaminant gases.⁽²⁶⁾ The presence of nickel in the corrosion product indicates that surface texturing on the Ni-P layer plays an important role in affecting the incomplete coverage of both Ni-P and cobalt alloy layers.

On a disk surface, the dissolution of contaminant gases in the adsorbed water reduces the solution pH and increases electron conductivity. This solution acts as an electrolyte in a corrosion cell consisting of cobalt or nickel anode and carbon cathode. Pourbaix diagrams of cobalt and nickel in Figure 4 show that these two elements are stable as ions at pH around 6 and lower. The metallic cobalt and nickel will dissolve into the electrolyte at pH below 6, freeing electrons which will subsequently be consumed by oxygen in a reduction process at the carbon surface. Cobalt and nickel oxide and hydroxide products then form on the surface. This corrosion model is also discussed by Novotny⁽²⁴⁾ and is shown in Figure 7. The electrochemical reactions are as follows:

Figure 7. Schematic of disk corrosion mechanism.

Disc corrosion is likely exacerbated by galvanic effect. By theory, two dissimilar metals or conductive materials in electrical contact and exposed to the same electrolyte will exhibit galvanic corrosion. Considering the sandwich structure of thin film disks, a porous carbon film is in contact with the cobalt alloy; both carbon and cobalt are exposed to the same environment. Therefore, galvanic action between the carbon overcoat and the underlying magnetic material is highly possible in a thin film disk structure. If the NiP layer is exposed to the environment, galvanic action between cobalt alloy and nickel will also occur. The small anode at the pore and the large cathode at the carbon surface can further increase the localized corrosion rate at the pores.

Galvanic action between the cobalt alloy magnetic layer and the carbon overcoat has been demonstrated.^(22,27) Modeling of a thin film disk structure found that cathodic galvanic current flows mainly to the areas of holes and defects. It was also found that the cathodic area was not important in determining galvanic current.⁽²⁷⁾ Brusic et al.⁽²²⁾ compared the corrosion behavior of a dielectric coated disk structure, AlMg/NiP/CoP/D, with a carbon coated structure, AlMg/NiP/CoP/D/C. Localized corrosion was found on the carbon coated sample, but not on the dielectric coated sample. The result means that there is galvanic

action between carbon and cobalt alloy layer, and that depositing a dielectric material as a barrier layer does not prevent the galvanic effect. To reduce the galvanic mismatch between magnetic and carbon layer, a highly corrosion resistant magnetic material, having high electromotive force potential, should be used.

Corrosion of various magnetic cobalt alloy compositions have been compared.^(23,28) Smallen et al. found that Co-20Cr has more corrosion resistance than Co-20Ni and electroless Co-P. In a ternary alloy, Kawashima et al. found that corrosion resistance decreased in the following order: Co-12Cr-2Ta > Co-24Ni-5Cr >> Co-10Pt-9Ni.

The presence of a top 20Å thin polymeric lubrication layer has not usually been considered in a corrosion study due to its non-conductive nature. However, this layer could alter the amount of water adsorbed on the surface, affecting overall corrosion rate.

CHAPTER 2

Research Objectives and Hypothesis

One property resulting from the high sp^3/sp^2 bonding ratio of the hydrogenated carbon overcoat is high electrical resistivity. Because the carbon layer participates in disk corrosion as a cathode, the higher resistivity of the overcoat lowers the disk corrosion rate due to the reduction of electron flow in the electrochemical process. Therefore, a disk containing higher hydrogen content in the overcoat should have a higher corrosion resistance than that with a lower hydrogen content.

The objective of this research is to compare the corrosion resistance of thin film disk samples at different hydrogen levels in the carbon overcoat. In order to see the effect of the outermost lubricant layer, corrosion resistance of unlubricated disk samples at different H levels are also compared. The polymeric lubricant does not participate in the electrochemical reaction, but could alter the amount of water adsorbed. Otherwise, the lubricant should somewhat protect the layers underneath from the environment, by limiting the amount of water to reach the underlayers through pores in the overcoat.

The experimental methods used were atmospheric corrosion and electrochemical tests, light scattering technique, optical microscopy, and electron spectroscopy for chemical analysis (ESCA).

CHAPTER 3

Experimental Methodology

3.1 Specimens

Three sets of 95 millimeter diameter lubricated disks with 0%, 16%, and 36% H content in the carbon overcoat were used. The disks were commercially prepared by a disk manufacturer contracted by Quantum Corporation, Milpitas, CA. The disk samples were reported from the vendor to have an average surface roughness (R_q) of $43\text{\AA}$, an average carbon thickness of $185\pm 10\text{\AA}$, and an average lubricant thickness of $25\text{\AA}$. The magnetic layer is a cobalt based alloy coated with sputtered carbon at varying hydrogen contents. The sputtering power, pressure, and the resulting hydrogen content, electrical resistivity, and carbon thickness are reported in Table 1.

Table 1. Sputtering conditions and the resulting carbon layer

Power (kW)	Pressure (mTorr)	H content (%)	Resistance (Ω)	C thickness ($\text{\AA}$)
2.8	7.1	0	< 0.01	150
2.5	2.8	16	0.08	173
2.9	8.0	36	16000	201

Because the effect of lubrication is not known, three samples of unlubricated disks at 0%, 16%, and 36% are also compared. The disks were prepared from the same lot as the lubricated disks but were withheld from the liquid lubrication coating process.

3.2 Atmospheric corrosion test

The conditions used in this experiment were $30 \pm 0.2^\circ\text{C}$, $80 \pm 2\%$ RH. While the percentage relative humidity is typical of most corrosion tests, a lower temperature of 30°C was used because cobalt corrodes faster at lower temperatures.⁽²¹⁾ Although standard gas species and concentration for disk corrosion tests have not yet been established by the ASTM standards, the corrosive gases used were similar to those used in Ref. 24, being 2.6 ppb Cl_2 , 36.9 ppb H_2S , 323 ppb SO_2 , and 483 ppb NO_2 , by weight. These test conditions were originally developed for contact and connector materials, having a precious metal overlaid structure. Interested readers can find detailed development in Refs. 34 to 36.

3.2.1 Mixed flowing gas environmental test system

Different gas species come individually prepared in plastic tube containers. Each species come in liquid form, whose vapors permeate through plastic tubing walls with a concentration gradient as the driving force. Each of the gas species was obtained from Metronics Inc., San Jose, CA

at a specified permeation rate. The rate was calculated from the intended concentration and the total air flow rate in the corrosion chamber. For example, the intended Cl_2 concentration is 2.6 ppb, and the total air flow rate is 10 liters per minute. The specified permeation rate of Cl_2 is therefore 2.6 ng/g times total air flow rate (10 l/min) times air density of 1.19 g/l. The result is 30.9 ng/min. If the concentration is specified by volume, the mass of each gas species can be converted to volume using its molar constant, 24.4 liters divided by its molecular weight.

The permeation rate depends on temperature, tube length, wall thickness, the type of plastic, and the nature of the liquid species. A specific permeation tube will therefore permeate at a rate totally dependent on the temperature. A permeation tube is usually calibrated at a fixed operational temperature, and is used at that temperature. When not in use, the tubes are stored in a freezer to minimize their permeation rates. The tubes were placed into a desiccator at ambient temperature at least one day before use. The actual permeation rate was then determined by observing the weight loss over a period of time. A full description of the calibration technique can be found in ASTM standard D-3609.⁽³⁷⁾

The environmental test chamber was the IFA+ Mixed Gas Atmospheric Corrosion Test Systems Model MGC-10QM specially

modified for Quantum Corporation by Interfact Associates, Ltd., PA. The system was designed to have an air flow rate of 10 liters/minute, and sized to have an air velocity over the specimen of 1 cm/sec. The system is shown schematically in Figure 8. There are three main parts in the system: the gas permeation chamber, the humidifier, and the test chamber. Dry air from the water aerosol filter passes through the carbon and HEPA filters to remove the organics and particulates sized larger than 0.2 micron. The clean dry air is then divided into two streams. The first air stream is bubbled through a bath of heated water in the humidifier, becoming water saturated air having a relative humidity of 100%. This air stream is called wet air. The second air stream goes through the permeation chamber containing gas permeation tubes and carries selected gases to the test chamber after mixing with the first stream (wet air) at the top of the humidifier.

The test chamber is cylindrical in shape, with a diameter of 5.5 inches and three feet long. The mixed air flows through fritted glass at one end of the chamber and is exhausted through a tube on the chamber door located at the other end of the chamber.

1. Mass flow controller	4. Humidifier
2. Pressure sensitive switch	5. Test chamber
3. Gas permeation chamber	

Figure 8. Schematic of the mixed flowing gas system.

The relative humidity in the corrosion chamber is controlled by the amounts of wet and dry air. The percentage relative humidity is gauged by dew point measurement using the mirror type hygrometer. An air sample is drawn from the air stream flowing to the test chamber through a chilled mirror sensor; a dew point temperature is

read when condensation occurs. This dew point measurement should be made when there are no gas tubes present in the permeation chamber because the gases can contaminate the mirror sensor and cause false readings.

Water vapor pressures at various temperatures are shown in Table 2. To find the operating dew point, the vapor pressure at an operating temperature (30°C) was determined from the table. This is the saturated vapor equivalent to 100% RH. The vapor pressure (31.824) is then multiplied by desired relative humidity (80) and divided by 100. The temperature at this resulting vapor pressure (25.459) is the dew point temperature (26.2) at the desired relative humidity, which was determined from the same table. The temperatures of the test and permeation chambers are controlled by recirculation of temperature controlled water between the reservoirs and the chamber water jackets.

Table 2. Water vapor pressure at 25°C to 30°C (mmHg)

Temp. (°C)	0.0	0.2	0.4	0.6	0.8
25	23.756	24.039	24.326	24.617	24.912
26	25.209	25.509	25.812	26.117	26.426
27	26.739	27.055	27.374	27.696	28.021
28	28.349	28.680	29.015	29.354	29.697
29	30.043	30.392	30.745	31.102	31.461
30	31.824	32.191	32.561	32.934	33.312

3.2.2 *Light scattering method*

The extent of corrosion was measured by detecting the changes in surface roughness due to corrosion asperities using a light scattering technique. This technique has been proven a sensitive monitor for studying the corrosion of thin films.⁽³⁸⁾ It should be noted that the term "scattering" may be different from that used in physics, where light is absorbed and released due to electron oscillation. Reflection is one such type of scattering. However, in this research the term "scattering" means diffuse reflection. Light falling on a rough surface is reflected by any area big enough to be a reflection plane, i.e. larger than the wavelength of incident light.

The equipment used in this experiment was the IFA+ Dark Field Scattering Meter made by InterFact Associates, Ltd., Dresher, PA. The equipment consists of three main parts: A white light source connected to two fiber optic light pipes, a sample housing, and a power meter. The schematic of the apparatus is shown in Figure 9. The sample housing was placed above the disc surface to be measured, with the two fiber optics held on both sides of the sample housing, making 45 degrees with the disc surface. The fiber optics were in such positions that their combined beams focused on the sample underneath. Sharp edges of the illuminated area indicated that both beams were in focus. The focused,

combined beams produced an elliptical shape of about 1 x 1.5 cm. illuminated area.

A light sensor was placed on top of the sample housing, making a 90 degree angle to the disc sample surface lying underneath, to detect scattered light from the disc surface. The other end of the sensor was attached to the power meter.

Figure 9. Schematic of the light scattering method.

The power meter used was the Coherent Model 212, with an intensity range from 0.1 microwatts to 300 milliwatts, a response time of 7 μ s to 70 ms, and an accuracy of $\pm 5\%$. Its spectral sensitivity is in the range of 440 nm to 1.1 μ m, covering the entire visible and part of the ultraviolet light range.

On a smooth mirror-like surface, all the 45 degree incident light will reflect forward at an equal angle of 45 degrees to the surface. Therefore, minimal light will reach a detector positioned at 90 degrees to the sample plane. When corrosion occurs, corrosion product asperities scatter light randomly in all directions, and the light sensor will detect some of the scattered light. Larger numbers and size of the asperities increase the amount of light scattering and therefore the amount of light detected. The correlation between light intensity and extent of corrosion in terms of asperity areal density is shown in Figure 10. Asperity areal density was obtained from an optical micrograph of a corroded surface of disks by summing the area of all corrosion spots and dividing by the total area of the micrograph.

A standard alumina plate was used to calibrate the light intensity at the beginning and during the measurement, to insure the incoming light intensity remained fixed. Alumina is a chemically inert material; when polished to a smooth surface its smoothness is constant with time. The fixed light intensity recommended for disc surface measurements is 0.3 to 0.5 mW. The meter range used with the standard alumina and the disk samples are 1 x 1 mW and 10 x 1 μ W, respectively.

Figure 10. Light intensity as a function of corrosion asperity areal density on corroded disks.

3.2.3 Corrosion chamber calibration

Uniformity and reproducibility of the corrosive environment are important for comparing the corrosion resistance of one sample to another. Air velocity and gas concentration indicate how much and how fast the contaminant gases are brought onto a tested sample. Standard practice of chamber calibration can be found in the ASTM B-827-92.⁽³⁹⁾ In this experiment, the uniformity region was determined by comparing the corrosion resistance of equivalent disk samples. Two experiments (U2, U3) were conducted in order

to find a uniform region in the chamber. Design of the sample holder is important, to present the least obstructive effect on the laminar air flow over the samples. The disk holder used was a pole that support the disks at their inner diameters. The disk samples were stacked using plastic rings as separators. The distance between each sample is 75 mm. The schematic of the stack assembly is shown in Fig 11(a).

Figure 11. (a) Disk holder with disks stack, and (b) observed locations on a tested disk.

Five disks were assembled in the first (U2) experiment. Three disks were used for the second (U3) as guided by results from the previous experiment. Two dummy disks were placed on the top and bottom of each stack, to create the same environment for all disk samples being investigated.

The disk stack was preconditioned in an oven at 45-50 °C for 15 minutes. This step is necessary to prevent water condensation on the sample surfaces, which will cause deviation in the atmospheric corrosion behavior from the conditions controlled in the test chamber. Eight positions on a disk were observed, four on each surface, each position being 90 degrees apart.

Corrosion resistance of the disks manufactured from the same lot were compared using a light scattering technique (see section 3.2.2). The initial light intensity readings of all eight positions per disk were recorded. Location numbers were designated relative to the air flow direction in the test chamber as shown in Figure 11(b). The numbers shown in parentheses are the locations on the opposite sides (bottom surfaces) of the disks.

After preconditioning, the test assembly was transferred to the corrosion chamber having locations 1 (top) and 5 (bottom) facing the air flow. The disks were removed every six hours to measure the change in light intensity readings, using the same locations as the initial

readings. The experiment was allowed to progress for at least 24 hours, and the changes in light intensities at each location of all five disks were plotted as a function of exposure time. While not being tested, the disks were stored in a desiccator to limit the amount of moisture in contact with the samples.

Different positions on a disk will see different gas concentrations. At the leading edge (positions 1 and 5), some gases molecules will interact and become part of the corrosion products. The amount of gases at the trailing edge will be less and hence give a slower corrosion rate. Therefore the tested disks can be compared only at the same location, e.g. location 1 on disk 1 should be compared with location 1 on other disks. Here, the leading edge positions (1 and 5) were chosen to represent the results which are shown in Appendix A. All other position results are used only for qualitative comparison. The results showed three middle positions in the test chamber to be essentially uniform in corrosion response, when comparing the same location on each disk.

3.2.4 Corrosion test of disks with different H levels

The three uniform locations in the chamber were used to compare corrosion resistance of disks with 0%, 16%, and 36% H contents. The procedures were the same as those of the uniformity test above. The experiment of the lubricated

disks group was repeated three times, the positions of the samples being switched within the uniform region each time. For example, if the 0%H disk was placed on the top position in the first run, it was placed at the middle and bottom positions in the second and third run respectively, and so on for the others. Only one experiment was conducted on an unlubricated disk group. The experiment name, disk group, and the disk positions from the top of the stack are tabulated in Table 3.

Table 3. Experiment name, disk group, and disk position

Experiment name	Disk group	Disk position		
		Top	Middle	Bottom
H1	Lubricated	0%	16%	36%
H2	Unlubricated	0%	16%	36%
H3	Lubricated	36%	0%	16%
H4	Lubricated	16%	36%	0%

Besides the light scattering measurement, optical micrographs were taken after every six hours of exposure on the H3 and at some exposure times on the H1 and H2 experiments using a Leco AH-3 system with Olympus Vanox AHMT-3 lens, Optronics engineering DEI-470 color video printer and Sony color video monitor.

3.3 Electrochemical test

In order to confirm the atmospheric corrosion test result, an electrochemical study was conducted on both lubricated and unlubricated prime disks at 0%, 16%, and 36% H contents using the EG&G PAR potentiostat model 273. The electrolyte used was 1N. Na_2SO_4 . A glass joint tube held the electrolyte and electrodes on top of a disk as shown in Figure 12. The tube and its O-ring was placed directly onto the disk surface, and held firmly clamped by two plates of acrylic plastic and four sets of screws.

Figure 12. Electrochemical test setup

A standard Calomel reference electrode wrapped by a spiral 304 stainless steel wire was placed into the electrolyte filled glass tube. The level of the electrolyte covered the tip of the reference electrode and the spiral portion of the steel wire. The high corrosion resistant steel spiral was used as a counter or auxiliary electrode; its spiral shape was used to increase total surface area for electron current flow without disturbing the voltage measurement of the reference electrode. The disk itself is the working electrode. The contacting point on the disk was abraded using sandpaper to insure good electrical contact with the metal layers underneath. All three electrodes were connected to the PAR equipment.

A scan from $-50 \text{ mV}_{\text{sce}}$ to $+50 \text{ mV}_{\text{sce}}$ below and above the rest or corrosion potential was done on a Tafel mode. The scan rate used was 1 mV/sec , the sample area 5.5 cm^2 . The software used to calculate tafel slopes and corrosion rates came supplied with the EG&G PAR model 352.

3.4 Chemical and Image Analyses

3.4.1 ESCA analysis for corrosion product

To compare the corrosion product composition on the 0% and 36%H disks, two samples were submitted for Electron Spectroscopy Chemical Analysis (ESCA) at Surface Science Laboratories. Because the high oxygen content in the lubricant interferes with the corrosion product spectrum,

only the corroded 0% and 36% unlubricated disk samples were submitted. An uncorroded 16% unlubricated disk was analyzed for control.

ESCA is a surface analytical technique that provides elemental and chemical bonding information of all the elements, with the exception of hydrogen and helium. The technique is applicable to both conductive and nonconductive materials. A surface irradiated with an X-ray photon energy ($h\nu$) ejects an electron with a certain kinetic energy, depending on its binding energy. By measuring the kinetic energy of this emitted electron, the electron binding energy can be determined through the equation $h\nu = E_{\text{kin}} + E_{\text{B}}^{\text{v}}(k)$, where E_{kin} is the kinetic energy of the electron. The $E_{\text{B}}^{\text{v}}(k)$ is the binding energy of the k^{th} level electron at vacuum level. This binding energy is characteristic of an individual element, and can be used to determine atomic species within a sample.

All spectra were collected using the SSX-100 ESCA Spectrometer equipped with a monochromatic Al Ka x-ray source. The x-ray spot size was approximately $600\ \mu\text{m} \times 1000\ \mu\text{m}$. ESCA survey and high resolution spectra were acquired with energy resolutions of 1.1 eV/channel and 0.16 eV/channel respectively. The information was from approximately the top 50 to 100 Å of the sample surface, with the sensitivity of 0.05 to 0.5 atomic percent.

3.4.2 Pore count and image analysis

Brusic and co-workers⁽²²⁾ suggested a method called "pore decoration technique," originally developed by W.H. Abbott (unpublished), to determine pore density in the carbon overcoat of a magnetic disk. Because pores cannot be seen in deposited film by optical microscope, exposing disk samples to an environment containing 10 ppb Cl_2 at 25°C, 70%RH for 24 hours yields corrosion products that can be seen at the pore locations at 1,000x magnification. The number of pores can then be determined by counting the number of corrosion sites.

Because the pores in the carbon overcoats of corroded disks exposed to the condition used in this experiment are already decorated, the number of corrosion sites on the disks represent the number of pores on those disks. These decorated pores are susceptible to localized corrosion and are the ones of interest. The number of corrosion spots were counted on 1860x magnification optical micrographs of the H3 group disks taken at 24 hours exposure time. At 24 hours, it is assumed that all the pores are sufficiently decorated.

In order to discern the corrosion mechanism between the disks containing hydrogen (16% and 36%H) and the ones containing no hydrogen (0%H), an image analysis of the sample surfaces was conducted. The histogram of size

distributions of the 0%, 16%, and 36% disks was constructed from optical micrographs taken at 1860x magnification.

3.4.3 Contamination analysis

Contamination on a disk surface prior to atmospheric corrosion test could affect the disc corrosion resistance. In order to compare the level of inorganic and organic anion contaminants on the lubricated and unlubricated disks, 16%H lubricated, 16%H unlubricated, and 36%H unlubricated samples were rinsed with 60 ml. DI water. The wash from each disk rinse was analyzed using Ion Chromatograph model DX-300 with the IONPAC AS4A analytical column and AI-450 Chromatography software made by DIONEX Inc., Sunnyvale, CA.

CHAPTER 4

Results and Discussion

4.1 Lubricated disks

The extent of corrosion versus exposure time of three H levels of disks at locations 1 and 5 in experiments H1, H3, and H4 are compared in Figures 13 to 18. A higher light intensity value indicates a greater extent of corrosion. It can be clearly seen from all six results that the 36% disk has the least extent of corrosion, while four out of six results show that the 0% disk has the most extent of corrosion. The difference between the 0% and the 16% at locations 5 of the H1 and H4 experiments cannot be clearly distinguished (Figures 14 and 18). Results from all other locations (not shown) agree that extent of corrosion for lubricated group from high to low are: 0% > 16% > 36%.

It should be noticed that curves from all the positions from experiment H1 (Figures 13 and 14) flatten out at 36 hours of exposure, which seem to indicate some kind of time dependent passivation.

The extent of corrosion is evident by representative optical micrographs of the disks at locations 1 from the H3 experiment after 24 hours of exposure as shown in Figure 19.

Figure 13. Extent of corrosion of lubricated disks at location 1, having positions from top to bottom as 0%, 16%, 36% H content.

Figure 14. Extent of corrosion of lubricated disks at location 5, having positions from top to bottom as 0%, 16%, 36% H content.

Figure 15. Extent of corrosion of lubricated disks at location 1, having positions from top to bottom as 36%, 0%, 16% H content.

Figure 16. Extent of corrosion of lubricated disks at location 5, having positions from top to bottom as 36%, 0%, 16% H content.

Figure 17. Extent of corrosion of lubricated disks at location 1, having positions from top to bottom as 16%, 36%, 0% H content.

Figure 18. Extent of corrosion of lubricated disks at location 5, having positions from top to bottom as 16%, 36%, 0% H content.

(a)

(b)

(c)

Figure 19. Optical micrographs of lubricated disk surfaces after 24 hours exposure at 1860 magnification: (a) 0%, (b) 16%, and (c) 36% H content disk.

Electrochemical results for the lubricated disk group are shown in Table 4. The 36% disk sample has the lowest corrosion current (I_{corr}), and the 0% the highest. This result is in good agreement with the atmospheric corrosion test.

Table 4. Electrochemical results for the lubricated disks.

Samples	E(I=0) (mV)	I_{corr} (nA/cm ²)	Tafel slopes (V/decade)		Chi square
			β_a	β_c	
0%	-109.4	21.1	1.39E-1	5.16E-2	2.770
16%	-100.4	5.97	1.15E-1	7.04E-2	0.637
36%	+141.0	0.72	8.94E-2	5.43E-2	0.230

It is clear for the lubricated group that the higher H content disks have a better corrosion resistance than the lower H content ones. However, we do not know whether high electrical resistivity alone plays a role in enhancing corrosion resistance. The carbon film structure of the high H content may contain fewer pores, limiting the access of moisture and contaminants to the cobalt alloy film underneath. The slightly thicker carbon film (50Å) in the high H content disks as reported by the manufacturer could also be responsible for enhancing the corrosion resistance.

The results from Enke's work as mentioned earlier led to the possibility that high H content disks could contain fewer pores due to the higher intrinsic compressive stress in the carbon film. Because the number of corrosion spots also refers to the number of pores in the overcoat as suggested by the pore decoration technique,⁽²²⁾ it can be seen in Figure 19 that there are more pores on the 0% disks than the 36% disks. The number density of corrosion spots or pores are numerically shown in Table 5. Pore diameters range in size from 0.5 to 2.1 micrometers.

Table 5. Number density of the corrosion sites for the lubricated disks at 24 hours of exposure.

Samples	Number density (μm^{-2})
0%	19.39×10^{-2}
16%	7.49
36%	5.02

Electrochemical results in Table 4 also show a very high potential value of 141 mV in the 36% sample compared to the negative (more active) values for the 0% and 16% disks. The high potential value suggests that the 36% sample has a much better coverage, or fewer pores, than the 0% and 16% samples. The correlation between the potential value and carbon coverage has been confirmed experimentally and also discussed in ref. 22.

4.2 Unlubricated disks

Extent of corrosion for the unlubricated group (H2) is shown in Figures 20 and 21. Optical micrographs of the disks at location 1 taken at 24 hours of exposure are shown in Figure 22. The result shows that the extent of corrosion for three different H level disks are not as distinguishable as those for the lubricated group. The difference is only $1.2 \mu\text{W}$ between the lowest (16%H) and the highest (0%H) at 24 hours for position 1. The difference between the highest and the lowest in position 5 is only $0.6 \mu\text{W}$, while the difference in the uniform region from the uniformity test (U3) is $0.4 \mu\text{W}$ at position 1 and 5. In the lubricated group (H1), the difference between the lowest and highest for lubricated samples is $3.0 \mu\text{W}$ at location 1 and the same exposure time.

Moreover, the order is not consistent from position to position. At nearly all locations on the disks, the 36% H disk has the highest extent of corrosion, the 16% H the lowest. Location 1 has the order of 0%, 36%, 16% and location 5: 36%, 16%, 0% from high to low extent of corrosion at 24 hours. Because the results vary from different locations, one cannot conclude which percentage H content disk has the best corrosion resistance.

Figure 20. Extent of corrosion of different H content unlubricated disks from Experiment H2 at location 1.

Figure 21. Extent of corrosion of different H content unlubricated disks from Experiment H2 at location 5.

(a)

(b)

(c)

Figure 22. Optical micrographs of unlubricated disk surfaces after 24 hours exposure at 1860 magnification: (a) 0%, (b) 16%, and (c) 36% H content disk.

It should be noted that some scratches and defects were found in the 36% unlubricated disk sample along the circumference of radius 1.1 inches, Figure 23. While the scratches themselves cannot explain the unexpected results, they can worsen the overcoat coverage and corrosion resistance. It also raises a question of sample integrity as a whole.

Figure 23. Scratches found on the 36% unlubricated disk surface, x1860 magnification.

Electrochemical results of the unlubricated group are shown in Table 6. The 36% disk has the lowest corrosion current, the 0% the highest. As expected, the high electrical resistivity of the 36% disk lowered the flow of electrons and reduced corrosion rate. The result is also consistent with the lubricated group.

Table 6. Electrochemical results for the unlubricated disks.

Samples	E(I=0) (mV)	I _{corr} (nA/cm ²)	Tafel slopes (V/decade)		Chi square
			β_a	β_c	
0%	-146.8	75.69	2.07E-1	6.02E-2	2.900
16%	-158.1	43.93	1.77E-1	6.59E-2	0.245
36%	-138.8	11.02	1.37E-1	1.48E-1	0.153

The film porosity determined by the number density of corrosion sites from representative micrographs in Figure 22 is shown in Table 7. It should be kept in mind that the coverage depends on both number and size of the pores. Greater number density in the 0% sample may have a smaller pore size, thus the number density may not be directly compared with the coverage interpreted from the electrochemical values.

Table 7. Number density of corrosion sites on the unlubricated disks at 24 hours of exposure.

Samples	Number density (μm^{-2})
0%	11.05×10^{-2}
16%	3.41
36%	4.95

The number of corrosion sites from Table 7 indicates that there are more pores in the 36% disk than the 16% disk. Potential values of the 36% unlubricated disk from the electrochemical measurement show -138 mV, which is much different than that of the 36% lubricated sample having +141 mV. These results suggest that the coverage in the 36% unlubricated sample is much less than the 36% lubricated sample. The lubricant alone would not have this much effect. This explains why the 36% unlubricated sample corroded at a faster rate than the 36% lubricated disk.

From Table 6 the potential values of all three disks are about the same, which indicates that the coverage of all three samples are about equal. This can explain why the corrosion resistance from the environmental test of all three disks are similar and cannot be readily distinguished.

From the inconsistency of atmospheric and electrochemical results from the unlubricated group, it is likely that carbon overcoat coverage or porosity plays a more important role in atmospheric corrosion than the film

resistivity property. The film resistivity shows more effect in the electrochemical results. Therefore, electrochemical measurements give a more predictable corrosion behavior concerning H content affecting carbon film electrical resistivity. However, the result cannot always predict the atmospheric corrosion behavior.

4.3 Comparison of lubricated and unlubricated disks

Figure 24 compares the extent of corrosion between the lubricated (H1 at location 1) and unlubricated group (H2 at location 1). The extent of corrosion for all the unlubricated samples fall between those of 16% and 36% of the lubricated group. This means that the unlubricated samples have a better corrosion resistance than the lubricated group, except for the 36% sample which has a higher corrosion extent than that of the lubricated group. If the lubricant has a capability to adsorb more moisture from the atmosphere than the carbon material, it can explain why the 0% and 16% lubricated samples have more extent of corrosion than the unlubricated samples. More experiments should be done to confirm this result.

Figure 24. Extent of corrosion comparison between lubricated (H1) and unlubricated (H2) group, at location 1.

Surface contamination was expected to play a role in the abnormally high corrosion rate of the 36% unlubricated sample, and the higher corrosion rate in the lubricated group. Ion Chromatograph analysis result in Table 8 shows that the contamination level of the 36% unlubricated sample is about the same as the 16% unlubricated sample. Contamination levels on both unlubricated samples are higher than the lubricated sample. Therefore, contamination is not the culprit. However, if the oxalate anion found only on the lubricated sample has more effect on disk corrosion, the result of higher corrosion rate in the lubricated group can be explained. In the unlubricated sample set, the low coverage of carbon film for the 36% H content disk is likely to be the cause of its higher corrosion rate.

Table 8. Contamination levels on prime disk samples.

Anions	Concentration (ppb)		
	16%lubed	16%unlubed	36%unlubed
Fluoride	2.7	0	0
Formate	2.4	31.8	39.7
Chloride	2.4	4.8	4.0
Nitrite	33.4	55.6	47.8
Nitrate	0	9.3	7.6
Sulfate	22.4	42.6	38.7
Oxalate	58.1	0	0

Tafel plots from the electrochemical measurement of both lubricated and unlubricated disks at different H levels are compared in Figure 25. As can also be seen from Tables 4 and 6, general corrosion rates of the unlubricated group are higher than those of the lubricated group. As expected, the presence of lubricant helps protect the disks from exposure to the electrolyte, reducing the rate of corrosion.

Results from an electrochemical and an atmospheric corrosion test do not always agree. Disk corrosion occurs locally at pores in the overcoat. In the atmospheric corrosion study, physical changes due to localized corrosion are detected. In an electrochemical test, the overall number of electrons flowing through the system is measured. The localized electrons being exchanged within the pores may not be detectable by the electrochemical measurement.

Figure 25. Tafel plot of different H content lubricated and unlubricated disks.

4.4 Corrosion products and size distributions

Corrosion product composition on the 0% and 36% unlubricated specimens as determined by ESCA are shown in Table 9. Comparable amount of nickel and cobalt were detected. The elemental distributions are not distinguishable between the two samples.

Table 9. ESCA scan of the 0% and 36% corroded unlubricated disk samples.

Sample	Atomic percent of element detected					
	Co	Ni	O	Cl	S	N
0%	0.5	0.5	17	0.2	1.0	0.7
36%	0.2	0.3	18	0.4	0.6	1.6
control	-	-	9.7	-	-	0.3

The presence of nickel indicates that NiP layer was also exposed to the environment, allowing nickel in the layer to participate in the electrochemical process. Because most corrosion sites occur along the texture lines and nickel participates in the reaction, it is likely that susceptible pores in the carbon overcoat are the result of surface roughness from the texturing process. A more compressive nature of the high H content carbon film probably gives a better coverage at these rough surfaces. It is rationalized that these pores are macroscopic and are responsible for the corrosion susceptibility. The intrinsic voids in the amorphous film can be responsible only when

they combine to form a through-thickness pore. A new trend of smoother disk surface in the future would certainly be beneficial in improving disk corrosion resistance.

Corrosion product size distribution is interesting in a way that it may give some clue to the mechanism differentiation among these samples. The distributions of 0%, 16%, and 36% of the lubricated and unlubricated samples are shown in Figures 26 and 27. The size distribution pattern of the H content disks is slightly different than the disk with no H content. The 16% and 36% disks seem to have greater ratios of the larger corrosion product than the 0% disks. An exception is made for the 36% unlubricated sample; the distribution is closer to the 0% type.

It is likely that there are competing processes between corrosion nucleation and growth. Because there are greater numbers of pores in the 0% disks, more reaction energy is spent on corrosion nucleation rather than growth. In the 16% and 36% disks where there are fewer pores, added contaminant gases cooperate more into corrosion growth, since a limited number of nucleation sites are available. However, in the 16% carbon the electrical resistivity is low enough that an electron can mobilize over a bigger area, resulting in a larger corrosion spot size. In the 36% sample, the resistivity is higher; fewer numbers of large corrosion sites can form, compared to the 16% sample.

(a)

(b)

(c)

Figure 26. Size distribution of different H content lubricated disks from Experiment H3 at location 1: (a) 0%, (b) 16%, and (c) 36% H.

(a)

(b)

(c)

Figure 27. Size distribution of different H content unlubricated disks from Experiment H2 at location 1: (a) 0%, (b) 16%, and (c) 36% H.

CHAPTER 5

Conclusions

For the lubricated group, the atmospheric and electrochemical results are consistent and are as expected. That is, higher hydrogen content in the carbon overcoat gives a better corrosion resistance than the lower hydrogen content. The effect of hydrogen is to induce the sp^3 bonding type in the carbon layer which results in higher electrical resistivity and hence hinders corrosion electron flow in the overcoat of sandwich structure thin film disks. Hydrogen might also affect the carbon structure by reducing the number of pores due to the intrinsic high compressive stress in the diamond-like region. The low number of pores is evident by very high coverage in the 36% lubed sample as indicated by the electrochemical measurement.

The atmospheric experiment on the unlubricated samples shows no clear distinction in corrosion resistance between the different H level disks. These results are inconsistent with the electrochemical measurement, which suggests better corrosion resistance for the higher H content disks than the lower H content ones.

Comparing the lubricated and unlubricated groups under equivalent atmospheric corrosion conditions shows that the unlubricated disks have a higher corrosion resistance than the lubricated disks. Anomalous behavior is noted for the

36% unlubricated sample. This latter result is most likely due to the low coverage of the carbon film of the particular 36% unlubricated disk investigated. The general result is inconsistent with the electrochemical results which show better corrosion resistance, as measured by i_{corr} , in the lubricated group, as expected. This inconsistency leads to a conclusion that atmospheric and electrochemical results do not always agree. However, more experiments should be done to clarify this.

Overall, atmospheric and electrochemical results show that hydrogen in the overcoat enhances disk corrosion resistance. The one caveat is the inconclusive results from the atmospheric corrosion test on the unlubricated disks.

CHAPTER 6

Recommendations for Future Research

Although studying actual disks is beneficial in learning about true situations, sometimes all parameters are difficult to control in a manufacturing environment. In order to learn specifically the effect of hydrogen in the overcoat, all other parameters such as overcoat thickness and surface roughness, need to be kept precisely constant. Therefore, experimental samples should be prepared in a laboratory. In order to learn more about disk corrosion, the areas suggested below should be examined.

1. Perform more experiments on unlubricated disks at different H levels.
2. Compare corrosion resistance of lubricated and unlubricated disks at the same H level.
3. Compare corrosion resistance of the different H level disks without texturing.
4. Measure corrosion potential of high and low H content carbon films by electrochemical method.
5. Compare the amount of water adsorbed between unlubricated disks and different types of lubricated disks.
6. Compare both microscopic (floating, x800k) and macroscopic (cross section, x45k) porosity of high and low H content carbon films using TEM.
7. Study the effect of oxalate anions on disk corrosion.

CHAPTER 7

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APPENDIX A
Uniformity Test Data

Uniformity test (U2)

<u>Disk</u>	<u>Loc</u>	<u>Initial</u>	<u>3hr</u>	<u>9hr</u>	<u>15hr</u>	<u>21hr</u>	<u>27hr</u>	<u>33hr</u>	<u>39hr</u>	<u>45h</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
1	1	0.4	0.4	0.5	0.6	1.4	2.1	3.0	3.9	4.0
	2	0.4	0.4	0.5	0.6	1.2	1.7	2.5	3.1	3.2
	3	0.4	0.4	0.5	0.5	1.0	1.4	2.0	2.5	2.5
	4	0.4	0.4	0.5	0.5	1.0	1.6	2.4	3.3	3.4
	5	0.4	0.4	0.5	0.7	1.7	2.6	4.0	5.2	5.3
	6	0.4	0.4	0.5	0.5	1.3	2.0	3.5	4.8	4.8
	7	0.4	0.4	0.5	0.5	0.9	1.4	2.3	3.3	3.4
	8	<u>0.4</u>	<u>0.4</u>	<u>0.5</u>	<u>0.6</u>	<u>1.3</u>	<u>2.0</u>	<u>3.3</u>	<u>4.5</u>	<u>4.5</u>
2	1	0.4	0.4	0.5	0.7	1.7	2.4	4.4	6.1	6.5
	2	0.4	0.4	0.5	0.7	1.7	2.6	4.7	6.6	7.0
	3	0.4	0.4	0.5	0.6	1.3	2.1	3.8	5.3	5.7
	4	0.4	0.4	0.5	0.6	1.5	2.2	4.3	6.1	6.3
	5	0.4	0.4	0.5	0.7	1.7	2.7	4.6	6.2	6.4
	6	0.4	0.4	0.5	0.6	1.5	2.6	4.8	6.6	6.9
	7	0.4	0.4	0.5	0.6	1.2	2.0	3.7	5.2	5.4
	8	<u>0.4</u>	<u>0.4</u>	<u>0.5</u>	<u>0.7</u>	<u>1.7</u>	<u>2.6</u>	<u>4.3</u>	<u>6.8</u>	<u>6.8</u>
3	1	0.4	0.4	0.6	1.5	3.2	4.3	5.6	7.0	7.3
	2	0.4	0.4	0.5	0.9	2.2	3.3	4.6	5.9	6.2
	3	0.4	0.4	0.5	0.7	1.7	2.5	3.7	4.8	5.2
	4	0.4	0.4	0.5	1.0	2.1	3.2	4.5	5.8	6.1
	5	0.4	0.4	0.6	1.6	3.2	4.4	5.9	7.1	7.2
	6	0.4	0.4	0.5	1.0	2.2	3.4	5.0	6.3	6.1
	7	0.4	0.4	0.5	0.8	1.7	2.5	3.7	4.7	4.8
	8	<u>0.4</u>	<u>0.4</u>	<u>0.5</u>	<u>1.0</u>	<u>2.2</u>	<u>3.4</u>	<u>4.8</u>	<u>6.0</u>	<u>5.9</u>
4	1	0.4	0.4	0.6	1.7	3.3	4.5	6.0	7.3	7.4
	2	0.4	0.4	0.5	1.1	2.4	3.4	4.8	5.8	5.8
	3	0.4	0.4	0.5	0.7	1.6	2.4	3.5	4.5	4.6
	4	0.4	0.4	0.5	1.0	2.1	3.2	4.6	5.9	5.8
	5	0.4	0.4	0.6	1.7	3.4	4.5	6.0	7.2	7.3
	6	0.4	0.4	0.5	1.1	2.4	3.6	5.4	6.8	6.8
	7	0.4	0.4	0.5	0.7	1.7	2.8	4.2	5.3	5.4
	8	<u>0.4</u>	<u>0.4</u>	<u>0.5</u>	<u>1.1</u>	<u>2.4</u>	<u>3.4</u>	<u>4.9</u>	<u>5.8</u>	<u>5.9</u>
5	1	0.4	0.4	0.6	1.5	3.0	4.4	5.8	6.8	6.9
	2	0.4	0.4	0.5	1.5	3.1	4.5	6.0	6.9	6.9
	3	0.4	0.4	0.5	1.2	2.5	3.8	5.1	6.0	6.0
	4	0.4	0.4	0.5	1.0	2.3	3.5	4.9	6.0	5.9
	5	0.4	0.4	0.7	1.5	2.8	3.8	5.1	5.9	6.1
	6	0.4	0.4	0.5	0.7	1.4	2.0	3.0	3.7	3.6
	7	0.4	0.4	0.5	0.9	1.5	1.8	2.2	2.5	2.5
	8	<u>0.4</u>	<u>0.4</u>	<u>0.6</u>	<u>1.4</u>	<u>2.6</u>	<u>3.5</u>	<u>4.4</u>	<u>5.0</u>	<u>5.1</u>

U2 - Location 1

U2 - Location 5

Uniformity test (U3)

<u>Disk</u>	<u>Loc.</u>	<u>Initial</u>	<u>6hr</u>	<u>12hr</u>	<u>18hr</u>	<u>24hr</u>	<u>30hr</u>	<u>36hr</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
1	1	0.65	0.80	1.40	2.90	4.60	5.90	7.20
	2	0.65	0.75	0.80	1.25	2.20	3.50	4.80
	3	0.65	0.75	0.80	1.10	1.70	2.40	2.90
	4	0.65	0.75	0.80	1.50	2.70	3.90	5.00
	5	0.70	0.80	1.50	3.10	4.90	6.50	8.30
	6	0.70	0.75	0.85	1.70	3.30	5.20	7.10
	7	0.65	0.70	0.80	1.35	2.40	3.80	5.50
	8	<u>0.70</u>	<u>0.70</u>	<u>0.80</u>	<u>1.65</u>	<u>3.25</u>	<u>5.00</u>	<u>7.30</u>
2	1	0.70	0.80	1.40	2.90	4.50	6.10	7.70
	2	0.70	0.75	0.90	1.70	3.20	4.90	6.65
	3	0.65	0.70	0.80	1.45	2.50	3.80	5.10
	4	0.65	0.70	0.80	2.00	3.80	5.40	6.80
	5	0.70	0.80	1.50	3.00	4.70	6.20	7.70
	6	0.70	0.80	0.90	2.10	4.10	6.00	7.80
	7	0.70	0.75	0.90	1.50	2.75	4.15	5.70
	8	<u>0.70</u>	<u>0.75</u>	<u>0.90</u>	<u>1.60</u>	<u>3.05</u>	<u>4.60</u>	<u>6.40</u>
3	1	0.70	0.80	1.20	2.70	4.30	6.90	8.90
	2	0.70	0.75	1.00	2.00	3.30	5.60	8.30
	3	0.70	0.70	0.90	1.60	2.40	4.20	6.50
	4	0.70	0.75	1.00	2.00	3.30	5.80	8.50
	5	0.70	0.80	1.30	3.00	4.60	7.10	8.80
	6	0.70	0.75	1.00	2.00	3.40	5.80	8.20
	7	0.70	0.70	0.90	1.50	2.40	4.00	6.00
	8	<u>0.70</u>	<u>0.75</u>	<u>1.00</u>	<u>2.10</u>	<u>3.50</u>	<u>5.90</u>	<u>8.60</u>

U3 - Location 1

U3 - Location 5

APPENDIX B
Hydrogen Level Test Data

Hydrogen levels test (H1)

<u>%H</u>	<u>Loc.#</u>	<u>Initial</u>	<u>6 hr</u>	<u>12 hr</u>	<u>18 hr</u>	<u>24 hr</u>	<u>30 hr</u>	<u>36 hr</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
0%	1	0.70	0.90	1.70	3.10	4.40	6.25	6.65
	2	0.70	0.85	1.20	2.00	3.10	4.45	4.60
	3	0.70	0.80	1.10	1.70	2.40	3.35	3.05
	4	0.70	0.90	1.20	2.10	3.20	4.60	4.85
	5	0.70	0.95	1.80	3.05	4.30	5.95	6.45
	6	0.70	0.90	1.20	2.00	2.90	4.00	4.40
	7	0.70	0.80	1.00	1.50	2.00	2.65	2.40
	<u>8</u>	<u>0.70</u>	<u>0.90</u>	<u>1.20</u>	<u>1.85</u>	<u>2.80</u>	<u>3.80</u>	<u>4.10</u>
16%	1	0.75	0.90	1.20	2.20	3.70	5.65	6.10
	2	0.75	0.85	0.90	1.20	1.90	3.30	3.35
	3	0.75	0.85	0.85	1.10	1.50	2.40	2.05
	4	0.75	0.85	0.90	1.35	2.10	3.55	3.70
	5	0.75	0.90	1.50	3.25	5.00	6.85	7.20
	6	0.75	0.85	1.00	1.70	2.70	4.20	4.40
	7	0.75	0.85	1.00	1.65	2.40	3.60	3.25
	<u>8</u>	<u>0.75</u>	<u>0.85</u>	<u>1.00</u>	<u>1.65</u>	<u>2.60</u>	<u>4.05</u>	<u>4.25</u>
36%	1	0.85	0.90	0.90	1.15	1.55	2.10	2.10
	2	0.85	0.85	0.85	0.90	1.10	1.60	1.60
	3	0.85	0.85	0.85	0.90	1.00	1.50	1.20
	4	0.85	0.90	0.90	0.95	1.20	1.80	1.65
	5	0.85	0.90	1.00	1.35	2.00	2.40	2.70
	6	0.85	0.90	0.85	0.90	1.20	1.70	1.55
	7	0.85	0.85	0.85	0.90	1.10	1.50	1.30
	<u>8</u>	<u>0.85</u>	<u>0.85</u>	<u>0.85</u>	<u>0.90</u>	<u>1.15</u>	<u>1.65</u>	<u>1.60</u>

Hydrogen levels test (H2 unlubricated disks)

<u>%H</u>	<u>Loc.#</u>	<u>Initial</u>	<u>6 hr</u>	<u>12 hr</u>	<u>18 hr</u>	<u>24 hr</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
0%	1	0.70	0.70	1.10	2.00	3.50
	2	0.70	0.70	0.70	0.90	1.50
	3	0.70	0.70	0.70	0.95	1.55
	4	0.70	0.70	0.70	1.00	1.60
	5	0.70	0.70	0.80	1.35	2.20
	6	0.70	0.70	0.70	0.90	1.25
	7	0.70	0.70	0.70	0.80	1.05
	<u>8</u>	<u>0.70</u>	<u>0.70</u>	<u>0.70</u>	<u>0.85</u>	<u>1.25</u>
16%	1	0.75	0.75	0.90	1.45	2.35
	2	0.75	0.75	0.75	0.75	0.85
	3	0.75	0.75	0.75	0.75	0.80
	4	0.75	0.75	0.75	0.75	1.05
	5	0.75	0.80	0.95	1.65	2.80
	6	0.75	0.75	0.75	0.75	0.90
	7	0.75	0.75	0.75	0.75	0.80
	<u>8</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.85</u>
36%	1	0.90	0.90	1.30	2.00	2.85
	2	0.90	0.90	1.00	1.40	2.00
	3	0.85	0.85	0.90	1.20	1.60
	4	0.85	0.85	1.00	1.60	2.45
	5	0.85	0.90	1.25	2.10	2.95
	6	0.95	0.95	1.10	1.80	2.70
	7	0.95	0.95	0.95	1.30	1.70
	<u>8</u>	<u>0.95</u>	<u>0.95</u>	<u>1.00</u>	<u>1.45</u>	<u>2.05</u>

Hydrogen levels test (H3)

<u>%H</u>	<u>Loc.#</u>	<u>Initial</u>	<u>6 hr</u>	<u>12 hr</u>	<u>18 hr</u>	<u>24 hr</u>	<u>30 hr</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
36%	1	0.80	0.80	0.90	1.00	1.30	1.80
	2	0.80	0.80	0.80	0.80	0.80	1.10
	3	0.80	0.80	0.80	0.80	0.80	1.00
	4	0.80	0.80	0.80	0.80	0.90	1.25
	5	0.80	0.80	0.90	1.00	1.30	1.55
	6	0.80	0.80	0.80	0.80	0.90	1.20
	7	0.80	0.80	0.80	0.80	0.80	1.00
	<u>8</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>1.00</u>
0%	1	0.70	0.80	2.50	4.20	6.80	9.10
	2	0.70	0.70	0.90	1.80	3.30	4.90
	3	0.70	0.70	0.80	1.50	2.70	4.10
	4	0.70	0.70	1.10	2.30	4.10	5.90
	5	0.70	0.70	1.50	2.70	4.60	6.30
	6	0.70	0.70	0.80	1.40	2.40	3.70
	7	0.70	0.70	0.70	1.00	1.60	2.30
	<u>8</u>	<u>0.70</u>	<u>0.70</u>	<u>0.75</u>	<u>1.15</u>	<u>1.90</u>	<u>2.90</u>
16%	1	0.80	0.80	1.25	2.20	4.10	6.20
	2	0.80	0.80	0.80	1.00	1.40	2.40
	3	0.80	0.80	0.80	0.90	1.10	1.80
	4	0.80	0.80	0.85	1.10	1.70	2.90
	5	0.80	0.80	1.30	2.30	4.20	6.30
	6	0.80	0.80	0.85	1.10	1.80	3.05
	7	0.80	0.80	0.80	1.00	1.30	2.20
	<u>8</u>	<u>0.80</u>	0.80	<u>0.80</u>	<u>1.00</u>	<u>1.40</u>	<u>2.35</u>

Hydrogen levels test (H4)

<u>%H</u>	<u>Loc.#</u>	<u>Initial</u>	<u>6 hr</u>	<u>12 hr</u>	<u>18 hr</u>	<u>24 hr</u>
		<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>	<u>μW</u>
16%	1	0.75	0.80	1.10	1.80	2.30
	2	0.75	0.75	0.85	1.10	1.50
	3	0.75	0.75	0.80	1.00	1.30
	4	0.75	0.75	0.80	1.10	1.40
	5	0.75	0.90	1.40	2.70	4.00
	6	0.75	0.75	0.80	1.30	2.00
	7	0.75	0.75	0.80	1.00	1.50
	<u>8</u>	<u>0.75</u>	<u>0.75</u>	<u>0.80</u>	<u>1.10</u>	<u>1.60</u>
36%	1	0.80	0.80	0.90	1.00	1.40
	2	0.80	0.80	0.80	0.80	0.90
	3	0.80	0.80	0.80	0.80	0.90
	4	0.80	0.80	0.80	0.85	1.05
	5	0.80	0.80	0.90	1.05	1.40
	6	0.80	0.80	0.80	0.80	0.90
	7	0.80	0.80	0.80	0.80	0.80
	<u>8</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.80</u>	<u>0.85</u>
0%	1	0.65	0.85	2.20	4.10	5.80
	2	0.70	0.70	0.80	1.70	2.80
	3	0.70	0.70	0.80	1.60	2.60
	4	0.70	0.75	1.05	2.50	3.80
	5	0.70	0.80	1.30	2.50	3.70
	6	0.70	0.75	0.85	1.60	2.35
	7	0.70	0.70	0.75	1.20	1.70
	<u>8</u>	<u>0.70</u>	<u>0.70</u>	<u>0.80</u>	<u>1.30</u>	<u>1.85</u>