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C. Singh Bhatia, Joachim Stöhr,
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Photoemission Electron Microscopy and Near Edge X-Ray
Absorption Fine Structure Spectroscopy**

Simone Anders,¹ Thomas Stammmler,¹ C. Singh Bhatia,²
Joachim Stöhr,³ Walton Fong,⁴ Chao-Yuan Chen,⁴ and David B. Bogy⁴

¹Advanced Light Source Division
Ernest Orlando Lawrence Berkeley National Laboratory
University of California, Berkeley, California 94720

²SSD/IBM, 5600 Cottle Road, San Jose, California 95193

³IBM Research Division, Almaden Research Center,
650 Harry Road, San Jose, California 95120

⁴Computer Mechanics Laboratory, Department of Mechanical Engineering,
University of California at Berkeley, Berkeley, California 94720

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**STUDY OF HARD DISK AND SLIDER SURFACES
USING X-RAY PHOTOEMISSION ELECTRON MICROSCOPY
AND NEAR EDGE X-RAY ABSORPTION FINE STRUCTURE SPECTROSCOPY**

SIMONE ANDERS¹, THOMAS STAMMLER¹, C. SINGH BHATIA², JOACHIM STÖHR³,
WALTON FONG⁴, CHAO-YUAN CHEN⁴, and DAVID B. BOGY⁴

¹Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720,
SAnders@lbl.gov

²SSD/IBM, 5600 Cottle Road, San Jose, CA 95193

³IBM Research Division, Almaden Research Center, 650 Harry Road, San Jose, CA 95120

⁴Computer Mechanics Laboratory, Dept. of Mechanical Engineering, University of California at
Berkeley, Berkeley, CA 94720

Light Source Note:

Author(s) Initials	S.A.	4/3/98	Date
Group Leader's initials	JMP	4/5/98	Date

ABSTRACT

X-ray Photo Emission Electron Microscopy (X-PEEM) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy were applied to study the properties of amorphous hard carbon overcoats on disks and sliders, and the properties of the lubricant. The modification of lubricants after performing thermal desorption studies was measured by NEXAFS, and the results are compared to the thermal desorption data. The study of lubricant degradation in wear tracks is described. Sliders were investigated before and after wear test, and the modification of the slider coating as well as the transfer of lubricant to the slider was studied. The studies show that the lubricant is altered chemically during the wear. Fluorine is removed and carboxyl groups are formed.

INTRODUCTION

X-ray Photo Emission Electron Microscopy (X-PEEM) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy are two modern analysis methods based on the availability of tunable X-ray radiation produced by a synchrotron. X-PEEM combines high spatial resolution with the ability to obtain local NEXAFS spectra. NEXAFS yields information about the elemental composition and bonding structure of the elements in the sample, and orientation of molecules (if a preferred orientation is present). All this information is obtained in a PEEM with submicron resolution, in addition to topological contrast which is present in a PEEM also. NEXAFS as measured by electron yield detection is surface sensitive, the probing depth is 2-10 nm depending on the material. But due to its elemental specificity, PEEM can also study buried layers of a different material if the top layer is not thicker than the electron escape depth.

Both methods have been applied to study the tribological behavior of the head/disk interface of magnetic storage devices. For storage devices with increasingly smaller spacing between slider and disk, approaching pseudo-contact and contact-recording, the tribochemical properties of the system consisting of the carbon overcoat of the disk, the lubricant, and the slider surface (carbon coated or uncoated) become more and more important. PEEM is a suitable tool to study these processes due to the sensitivity of NEXAFS to chemical changes. In this paper we describe the investigation of disk and slider surfaces before and after thermal desorption tests and wear tests using PEEM and NEXAFS.

EXPERIMENTAL

Experiments have been performed at beamline 8.0 of the Advanced Light Source (electron synchrotron) in Berkeley. This is an undulator beamline equipped with a monochromator with three different gratings covering the energy range of 200-1500 eV with a spectral resolving power $\Delta E/E$ of about 5000 for the present study. The monochromatized X-rays are focused by a Kirkpatrick-Baez pair of mirrors into a 200 μm diameter spot on the sample. The PEEM working principle is based on the electron emission caused by the X-ray illumination of the sample which is proportional to the X-ray absorption. The absorption is a function of the elemental and chemical state of the sample and the X-ray wavelength. An electron optical column forms an image of the emitted electrons. It is possible to obtain locally resolved NEXAFS spectra with a resolution given by the resolution of the electron optics by tuning the X-rays through the absorption edge of the element of interest and taking images at incrementally increased photon energy. The resolution of the electron optics is about 200 nm for the experiments described in this paper. The microscope is a two-lens system operating at a nominal voltage of 10 kV, and it is described in detail elsewhere [1]. For the present studies we used the microscope without energy filter.

RESULTS AND DISCUSSION

Studies of hard disks exposed to thermal desorption experiments

Samples from disks which were coated with 7.5 nm sputter deposited, nitrogenated amorphous hard carbon (CN_x) and lubricated with 0.85 nm perfluoropolyether (Z-dol) were exposed to a thermal desorption experiment. They were heated in UHV (base pressure $<10^{-6}$ Pa) up to 425°C in 10 min. The desorbed gaseous products were measured using a quadrupole mass spectrometer to determine the processes leading to the degradation of the lubricant due to thermal effects. These results will be published in a separate paper. We have studied the disks before and after the thermal desorption experiments using (not spatially resolved) NEXAFS spectroscopy in the total electron yield detection mode.

Figures 1, 2 and 3 show the carbon K edge, fluorine K edge, and nitrogen K edge spectra before and after heating of the disk samples. The spectra were normalized to the incident X-ray flux so that their relative intensity reflects the relative amount of the corresponding element on the surface of the disk. The probing depth of total yield NEXAFS is given by the electron escape depth, which is about 10 nm for carbon [2]. It can be seen in Figure 1 that the peaks which are characteristic for $\text{C}=\text{C}$ π^* bonds at 285 eV and σ bonds (broad peak around 300 eV) as well as the $\text{C}=\text{N}$ peak around 286.2 eV [3] and the $\text{C}=\text{O}$ peak around 288.1 eV [2] are not influenced by the heat treatment.

Figure 1: Carbon K edge NEXAFS spectrum of hard disks before and after thermal desorption experiments.

Figure 2: Fluorine K edge NEXAFS spectrum of hard disks before and after thermal desorption experiments.

Figure 3: Nitrogen K edge NEXAFS spectrum of hard disks before and after thermal desorption experiments.

The peaks at 294.5 and 296.3 eV, respectively, which are C-F bonds [4, 5], practically disappear after the heating. The nitrogen spectra (Fig. 3) show no significant difference within the accuracy of the measurements, but the fluorine signal (Fig. 2) is reduced to 10% of its original value by the heating. The data indicate that the CN_x film is not modified in structure and composition by the heating, but 90% of the fluorine from the lubricant has been removed. This is in good agreement to FTIR measurements that were performed before and after the heating to determine the thickness of the lubricant, which showed a thickness of 0.85 nm before and 0.08 nm after heating.

Study of wear tracks on disks

Supersmooth disks were coated with 5 nm cathodic arc deposited amorphous hard carbon and lubricated with 0.85 nm perfluoropolyether (Z-dol). They were used in a tribochamber wear test. The tribochamber is equipped with a spindle in UHV (base pressure < 10⁻⁶ Pa) and a quadrupole mass spectrometer to measure the erosion products during the wear test. The disks were worn in a continuous drag test with 50% negative-pressure sliders at a speed of 0.2 m/s and a load of 30 mN. Two kinds of sliders were used: uncoated sliders (Al₂O₃/TiC) and sliders coated with sputter deposited CH_x. After the wear test the disks were studied using the PEEM microscope.

Fig. 4a shows a PEEM image of the edge of the wear track. The scratch is caused by one of the rails of the slider. The image was taken at a photon energy of 280 eV which is below the carbon K edge, therefore the image contrast is mainly topological. Local NEXAFS spectra were taken in the undamaged area of the disk, at the edge of the wear track caused by the rail, and in the area between the rails. Fig. 4b shows the carbon K edge spectra and Fig. 4c the fluorine K edge spectra.

Figure 4: (a) PEEM image of the edge of a wear track taken at a photon energy of 280 eV. The field of view is 150 μm . (b) Local carbon K edge NEXAFS spectra of indicated areas. (c) Local fluorine K edge NEXAFS spectra of indicated areas.

The spectra show that the carbon overcoat/lubricant are identical in the undamaged area and between the rails. At the edge of the wear track in the scratch caused by one of the rails the fluorine is almost completely removed and the carbon spectrum shows a new peak at 288.5 eV which can be attributed to the formation of carboxylic bonds [2]. The formation of these bonds was not observed for an analogue experiment using an unlubricated disks. In this case we found that the carbon overcoat thickness was reduced but the structure of the overcoat was not modified. A comparison of the tracks created by the coated and uncoated sliders showed that the modification of the lubricant and the formation of carboxyl bonds is stronger for the uncoated slider than for the coated slider. This is in agreement with the fact that the $\text{Al}_2\text{O}_3/\text{TiC}$ material of the uncoated slider surface leads to catalytic reactions which decompose the lubricant faster than interactions which occur at the interface between the lubricant and the carbon coated slider [6].

Study of slider surfaces

The sliders which were used to wear the disks in the tribochamber for the experiments described above were also investigated using the PEEM microscope. Figure 5 shows the PEEM image of a scratch found on one of the rails of an uncoated slider after the wear test. The image is taken at 280 eV below the carbon K edge and shows therefore mainly topological contrast. Figures 6-8 show the local NEXAFS spectra at the carbon K edge, the oxygen K edge, and the titanium $L_{2,3}$ edge taken in the scratch and outside the scratch.

Figure 5: PEEM image of a scratch on one of the rails. Image taken at 280 eV, field of view 150 μm .

Figure 7: Local oxygen K edge NEXAFS spectra inside and outside the scratch.

Figure 6: Local carbon K edge NEXAFS spectra inside and outside the scratch.

Figure 8: Local titanium $L_{2,3}$ edge NEXAFS spectra inside and outside the scratch.

The carbon spectra show basically no carbon on the undisturbed slider surface but a strong signal in the scratch. The spectrum is very similar to the one observed at the edge of the wear track on the disk (Fig. 4b). The oxygen spectrum outside the scratch shows one peak probably connected to the Al_2O_3 of the slider material, but a strong, sharp additional peak around 539 eV appears inside the scratch that can be correlated to the presence of carboxyl bonds [2]. The titanium signal is much weaker in the scratch indicating that some material has been deposited on the surface. All these data led us to conclude that material has been transferred from the disk to the slider, and this material is the same as the material at the edge of the wear track. It is probably some form of degraded lubricant which is formed during the drag test.

On the coated slider we found undamaged CH_x in most of the areas. We observed the same kind of carbon K edge spectrum with the additional peak around 288.5 eV in scratches of the uncoated and coated sliders, and at the edges of the wear tracks of the disks.

CONCLUSIONS

The experiments which are presented here demonstrate that NEXAFS and PEEM are useful tools in studying problems of the head/disk interface. Due to the elemental and bonding specificity it is possible to detect changes in the elemental composition and bonding structure of disk and slider surfaces with a high spatial resolution.

It was found that the heating of disks lubricated with perfluoropolyether (Z-dol) up to 425°C in 10 min. does not change the CN_x structure and elemental composition, but 90% of the lubricant is removed during this process.

Wear of disks lubricated with perfluoropolyether leads to a strong reduction of the fluorine content in the worn areas and to the formation of new carboxyl bonds. The formation of these bonds is not observed on unlubricated disks. Degraded lubricant showing the carboxyl peak in the carbon K edge spectrum was found also in scratches of coated and uncoated sliders.

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