

Study of adsorption states for lubricant molecule using hard X-ray photoemission spectroscopy

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Abstract

The adsorption states for lubricant molecules have been investigated using hard X-ray ($h\nu = 7.95$ keV) photoemission spectroscopy (HX-PES). This method has the advantage for the organic molecules to be able to measure damage few. Being aware of the fact that P atoms exist only in cyclotriphosphazene base, we measured the take-off angle dependence of the P1s spectra. Each spectrum consists from two peaks, that is, substrate NiP peak and cyclotriphosphazene P peak. The cyclotriphosphazene P peak rapidly disappears with increasing take-off angle. We have also measured C1s spectra. Combining these experimental results, we have found that the adsorption state of cyclotriphosphazene end group is undergoing.

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1. Introduction

The successful operation of hard-disk drive product is critically dependent upon the formation of a tribologically robust interface between the read–write head and the magnetic recording disk. As a result of continuous demands for increasing area density in the hard disc (HD) the space between the magnetic head and the disc surface has been reduced down to sub 10 nm. To minimize the wear resulting from any high-speed intermittent contacts between these two surfaces, the magnetic recording disk and the read–write head are typically overcoated with a hard amorphous carbon film and the surface of the disk is lubricated with a molecularly thin film of a perfluoropolyether (PFPE) oil of relatively low molecular weight ($M_w < \sim 5000$ g/mol). The chemical structure of this lubricant is presented below in the experimental section. While this lubricant material has been successfully used in the computer disk industry for over 10 years, it is unclear whether it will be capable of meeting the increas-

ingly stringent demands that will be placed on the lubricant in future storage devices [1,3]. A novel lubricant molecule which consists of a PFPE structure terminated on one chain end by a hydroxyl end group and on the other end by a cyclotriphosphazene derivative is being developed for the next generation HD systems [2,4]. That is designed to be resistive to high temperature and have a function of self repair in case the main chain of the molecule is damaged. However, it is not clear how it sticks on the diamond-like carbon (DLC) surface. In order to clarify the adsorption states in relation to this lubricant molecules and DLC substrate, it is important to investigate these electronic structures for being improve the tribological reliability of future HD devices.

In this report, we have performed angle resolved hard X-ray photoemission (HX-PES) measurements on the samples covered with monolayer of the lubricant molecule. Recently, high resolution HX-PES has successfully been realized at the third generation high-energy synchrotron radiation facility, Spring-8 [5–7]. In light chemical elements, this method has the advantage for the organic molecules to be able to measure damage few due to low cross-section by high excitation energy. The electronic states concerning with their interaction of substrate are discussed, and the model of adsorption state are reported.

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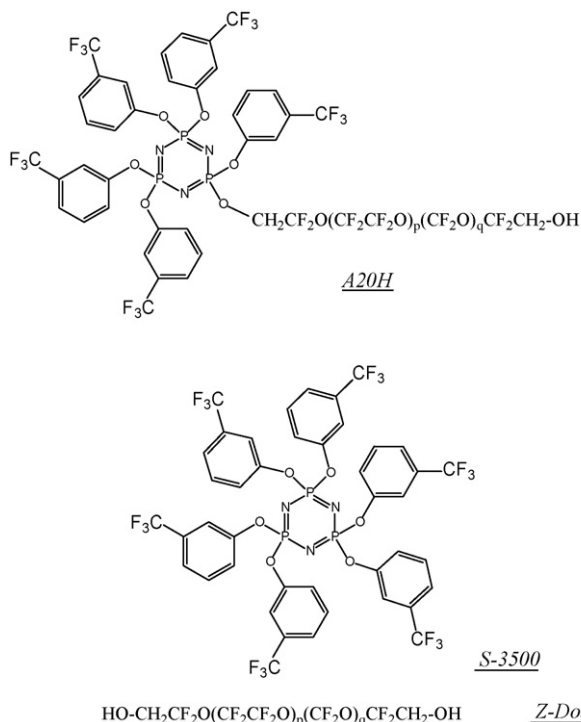


Fig. 1. Monomer units of chemical structure. “A20H” which is an end-functionalized PFPE based on the “Fomblin Z-Dol” backbone (random copolymer of perfluoroethylene oxide and perfluoromethylene oxide repeat units). “S-3500” which is organized by only cyclotriphosphazene.

2. Experimental

The cyclotriphosphazene-terminated PFPE lubricant used in this study was synthesized and purified by the Matsumura Oil Research Co., Japan. The lubricant is designated “A20H”, which is an end-functionalized PFPE based on the “Fomblin Z-Dol” backbone (random copolymer of perfluoroethylene oxide and perfluoromethylene oxide repeat units). To make comparative study, “S-3500” which is organized by only cyclotriphosphazene is also object substance in this investigation. These monomer units of chemical structure are shown in Fig. 1. These lubricants with monolayer ($\sim 1 \text{ \AA}$) were applied to substrate using standard dip-coating technique [8]. The substrate is amorphous carbon films (5 nm) with diamond-like carbon (DLC) on NiP (200 μm)/Al plate. In particular, DLC films are currently used in the production of hard coatings because of their extreme hardness, excellent tribological, corrosion and adhesive properties.

The HX-PES measurements were performed at an undulator beamline, BL47XU in SPring-8. Hard X-rays were monochromatized to 7.95 keV with the bandwidth of 80 meV by a Si 1 1 1 double crystal monochromator and additional a Si 3 3 3 channel-cut monochromator [9]. IMFPs at the kinetic energy of 8 keV range from 5 to 15 nm and are almost five times larger than those at 1 keV [10]. Available photon flux is about 2×10^{11} photons/s with the spot size of 0.2 mm (vertical) \times 1 mm (horizontal).

A hemispherical electron analyzer (R-4000 Gammadata Scienta Co.) was modified to measure high kinetic energy photoelectrons up to 10 keV. In order to gain photoelectron efficiency,

the detection angle of photoelectrons is set parallel to the polarization vector of incident X-rays. The vacuum of the analysis chamber was 10^{-6} Pa during measurements. No surface treatment was carried out and all the samples were investigated as they were inserted. All measurements were performed at room temperature. Normally, binding energy is referred to Fermi-edge estimated from the spectra of an Au plate. Total energy resolution was estimated to 294 meV.

3. Results and discussion

The high resolution C1s spectrum of thin A20H film is shown in Fig. 2(a) reveals the presence of two broad peaks. The spectrum is similar to the C1s XPS spectrum of A20H/CNx reported by Waltman et al. [1,2]. Their proposed assignments are based on comparison to FTIR and SCF/3-21G[d] calculation of thin A20H films. The assignment number of the peak is marked in Fig. 2, and the C1s spectrum of thin S-3500 film, thin Z-Dol film and DLC substrate are also shown in Fig. 2(b–d), respectively. These features in Fig. 2(a) of A20H are able to classify by comparison with each spectrum as followings. The largest peak S is mainly due to C–C bonding states of DLC substrate because of same largest peak is observed in each spectrum of Fig. 2. The characteristic features 1 in shoulder of peaks S are observed

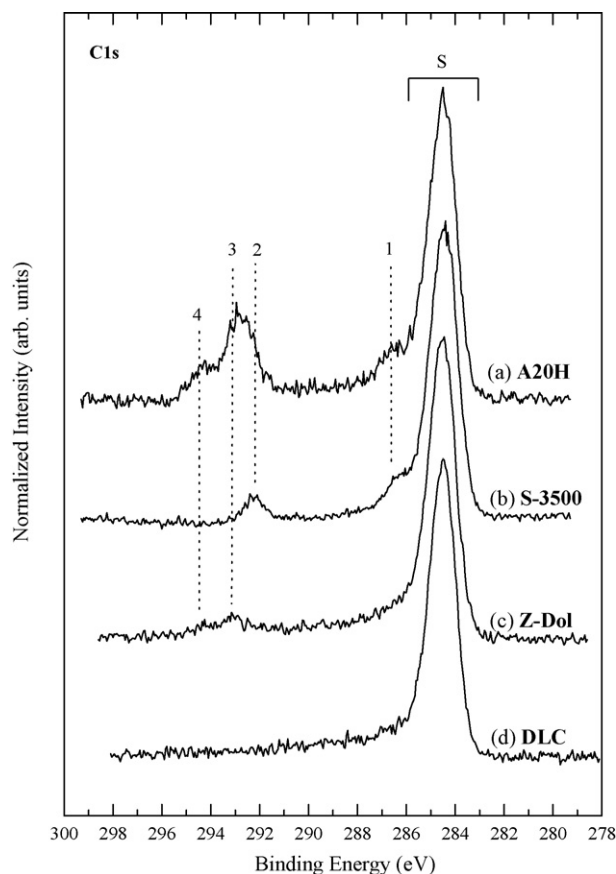


Fig. 2. The C1s spectra of thin A20H film (a), S-3500 (b), Z-Dol (c) and DLC (d). These intensities are normalized to the intensity of DLC (d) substrate which is organized by only carbon. A Shirley background is subtracted, and the assignment number of the peak is also marked, respectively.

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