X-RAY REFLECTOMETRY FOR ABSOLUTE THICKNESS MEASUREMENT OF PERFLUORO-POLYETHER LUBRICANT ON HARD DISK SURFACE

In a hard disk drive, magnetic write/read heads fly with a spacing of about 10 nm over the magnetic disk surface at very high speed. Therefore, direct contact between head and disk sometimes makes irreversible damage on a recording surface. Perfluoro-polyether (PFPE) liquid lubricant is widely used as a protective coating against fatal damage (Fig. 1).

Precise measurement of the absolute thickness of liquid lubricant film is a very important issue to understand the mechanism of lubricity and to design a high-performance lubricant layer. X-ray reflectometry at beamline **BL19B2** was applied to measure the absolute thickness of PFPE on a Si wafer in the nm range. A standard laboratory X-ray is not suitable for this purpose, since the brightness of the X-ray is not enough to analyze nm range thickness from X-ray reflectivity.

In this study, we have chosen two differently structured PFPE oils on Si wafer as typical thin film lubricant on a hard disk surface (Table 1).

We used a multi-axis diffractometer installed in BL19B2 for the measurement. The sample coated on a Si wafer was mounted in a chamber filled with He gas with a cover of Kapton film for reducing the background noise from air scattering.

Reflectivity of the Si wafer with lubricants was measured as a function of the reflection angle 20.



Fig. 1. Cross section of head-disk interface of hard drive.

Table 1.	Molecular	structure	of PFPE	lubricant
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Absolute thickness was calculated by a curve fitting method with computer simulation based on the multilayered film model (Fig. 2). The lube thickness of each sample was also measured by ellipsometry for comparison.

At first, for the measurement of the no-lube sample, a model of two native oxide silicon layers was necessary for suitable matching of the experimental reflectivity and simulated curves. Therefore, in cases of lubricated sample measurements, the same model of two oxide silicon layers was applied for a lubricant layer simulation. The simulation was performed on a multilayered thin film model that is simulated with three parameters for each layer. Film thickness, electron density and interface roughness were adjusted as parameters. For example, in the case of the lubricant A20H in Fig. 2, calculated parameters for each layer are shown in Table 2.

A correlation between the thickness calculated from X-ray reflectivity and the thickness measured by ellipsometry is shown in the Fig. 3. Both measurements have a good relationship in thickness.

Table 2. Simulation results for A20H film on Si

A20H	Thickness [nm]	Electron Density	Roughness [nm]
Lubricant	1.14	1.58	0.288
SiO ₂ (1)	0.84	1.51	0.091
SiO ₂ (2)	1.30	2.28	0.182
Si wafer	∞	2.33	0.009

Industrial Applications

Thus, the ellipsometry that has been widely used in the hard disk industry can be applied even in a very thin thickness range below 5 nm. However, the absolute thickness of lubricant measured by X-ray reflectometry is 1.56 times thicker than that measured by ellipsometry. Therefore, the ellipsometry should be calibrated by X-ray reflectometry in order to obtain precise values. Reflectometry gives electron density and interface roughness simultaneously. They are also very important information to evaluate thin film properties in the nm range.

In this study, a Si wafer was chosen as the base substrate for PFPE lubricant in order to obtain high reflectivity for an X-ray. However, on an actual hard disk, PFPE lubricant is coated on a diamondlike-carbon (DLC) overcoat. The interaction between lubricant molecules and a DLC surface will be different from that between lubricant molecules and a Si surface. Therefore, X-ray reflectometry will be tried on a disk DLC surface directly in a subsequent experiment.





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