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DEPOSITION AND MEASUREMENT OF NANOSCALE COMPLEX HYDROCARBON FILMS ON MAGNETIC HARD DISK SURFACES

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ABSTRACT

Measurement of complex hydrocarbon film thickness on engineering surfaces such as magnetic hard disks is difficult if the film thickness is below the peak to valley distance of the surface roughness. Instrument sensitivity and detection accuracy for most of the common tools for this purpose is limited. We have developed a novel calibration technique that allows accurate measurement of such films within 5% - 10% accuracy.

INTRODUCTION

Magnetic hard disks generally have a surface roughness with a peak to valley distance of 1.5 nm – 2.5 nm. A disk is usually covered by a lubricant layer such as bi-alcohol functional-grouped perfluorocarbons (PFPE) with a thickness near one nanometer. Film thickness measurement of such films is usually made by Fourier transformed infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) where the CF_2 peaks can be accurately measured. Complex hydrocarbons have many advantages over PFPE in additive solubility, and lubricity but are limited by volatility and oxidation stability. Multiply alkylated cyclopentane (MAC) overcomes many of these shortcomings and is a reasonable candidate for many micro-system devices applications as well as magnetic hard disks. However, thickness measurement and control of such films on engineering surfaces below the peak to valley distance has proven to be a challenge.

We have tried many techniques such AFM, X-ray reflectivity, ellipsometry, FTIR, and XPS and their modifications. Results are inconsistent and reproducibility is poor. Because hydrocarbons have multiple peaks and the surface roughness interferes with some of the techniques, precise measurements cannot be obtained. Many of the techniques also require calibration constants to obtain film thickness and the multiple thin films of magnetic hard disks make it difficult to obtain these calibration constants.

APPROACH

We deposited a known amount of complex hydrocarbon dissolved in a high purity dilute solvent within a barrier film on a magnetic hard disk. The sample was then frozen and the solvent allowed to evaporate slowly, leaving a uniform film on the surface. By knowing the amount of MAC used, the film thickness can be calculated. From this calibration sample, constants for a variety of analytical techniques can be obtained.

Experimental procedures and materials

The hydrocarbon lubricant used in this study was a multiply alkylated cyclopentane (MAC) synthetic compound. The chemical structure of this compound is shown in Figure 1 and consists of three C20 alkyl groups (octyl-dodecyl) attached to a cyclopentane core (1).

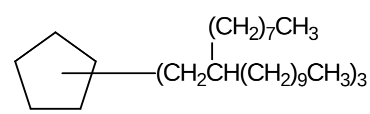

Figure 1 Chemical structure of MAC synthetic fluid

The combination of chain length and degree of branching results in a molecule that possesses a liquid nature over a wide temperature range but has a very low volatility. Dilute solutions of MAC in cyclohexane were created and disk samples were dip-coated to apply very thin films of MAC onto their surfaces. By changing the concentration of MAC in solution and the withdrawal speed, the thickness of MAC on the disk surfaces could be controlled. The relative amount of MAC on a disk surface was obtained using 85° grazing angle FTIR (Figure 2) to measure the intensity of the C-H stretch bond region. This provided a very sensitive measure of the relative amount of MAC present but calibration is required to give an absolute amount. A special technique was developed to create nanometer-scale thickness standards of MAC on actual hard disk surfaces.

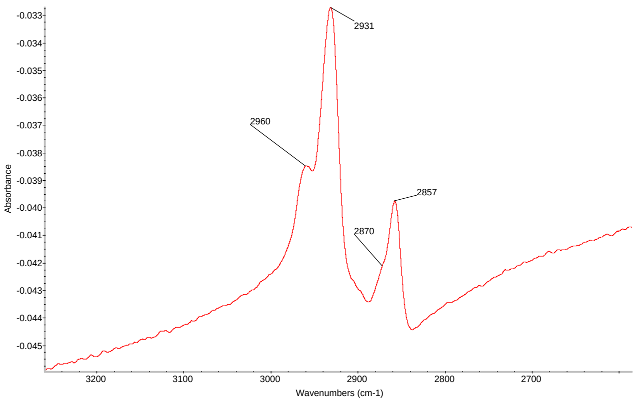

Figure 2 FTIR grazing angle C-H stretch region for MAC

Known concentrations of MAC in cyclohexane were confined to known areas of magnetic hard disk surfaces and quickly frozen in place with liquid nitrogen. The solvent was evaporated from the frozen solution and the remaining MAC deposited uniformly onto the surface. The uniformity of the deposition, checked using XPS, was better than 5% over a 28 mm diameter area.

Figure 3 Dilute solution of MAC in cyclohexane deposited on a disk surface

A series of FTIR measurements performed on standards of different thickness produces a calibration curve for MAC on that particular disk (Figure 4).

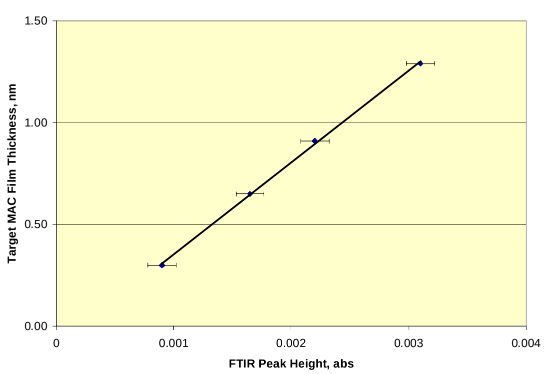

Figure 4 FTIR calibration curve using master calibration standards

Using the calibration, the relationship between film thickness and the concentration of MAC in solvent and the dip retrieval speed can be provided in quantitative terms (Figure 5). Knowing these relationships, a particular thickness of MAC on the disk can then be obtained by choosing an appropriate combination of concentration and retrieval speed.

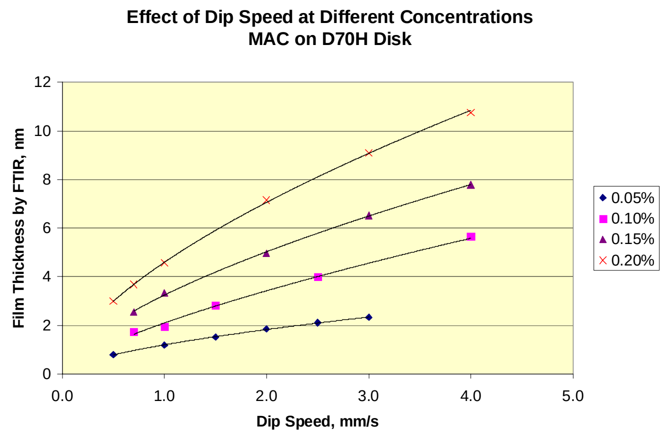

Figure 5 Film thickness as a function of MAC concentration and dip retrieval speed

CONCLUSIONS

A comprehensive method has been developed to allow controlled deposition and thickness measurement of MAC hydrocarbon synthetic lubricant on magnetic hard disks at the nanometer scale. Films can be deposited by dip coating using dilute solutions of MAC in cyclohexane. Film thickness can be controlled by careful selection of the proper combination of MAC concentration and dip retrieval speed. Thickness of the resulting films can be confirmed using 85° grazing angle FTIR. Calibration of the FTIR measurements was made utilizing standards produced using a novel freeze-evaporation technique capable of producing uniform, nanometer-thick films on hard disk surfaces.

The ability to deposit known and controlled thicknesses of hydrocarbon films on magnetic hard disk surfaces has significant impact on the potential exploration of this branch of lubricant chemistry. A wide variety of mobile and bonded lubricant mixtures may now be examined for their effects on nanotribology at this scale.

REFERENCES

(1) C.G.Venier and E.W.Casserly, Lubrication Engineering 47 (1991) 586-591