# 高分子添加剤の吸着膜構造がしゅう動時の潤滑膜形成に及ぼす影響

# Effects of Conformations of Adsorbed Films of Polymer Additives on the Formation of Lubrication Films during Sliding

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## 1. Introduction

To improve energy efficiency, attempts are being made to reduce friction loss by lowering the viscosity of lubricating oils. Due to decrease in dynamic pressure, which is proportional to the viscosity, boundary and mixed lubrications are becoming increasingly important. Adding additive molecules is one promising measure to reduce the friction in boundary and mixed lubrications, where additives are able to adsorb on the sliding surfaces to avoid contact with the sliding surfaces. Understanding the formation mechanism of adsorbed film is essential to clarify its lubricating performance. However, due to the limitations of the measurement methods, it is still unclear. Previously, we have proposed a novel method with vertical-objective-type ellipsometric microscopy (VEM) that enables ellipsometry to directly measure the thickness change of adsorbed films during the adsorption process and gap changes during sliding in-situ. By using the VEM, lubricating film formation abilities of adsorbed films with different conformations were investigated.

## 2. Experiment methods

The schematic setup for measuring adsorbed film thickness and sliding gaps with VEM is shown in Fig. 1. Previously, VEM was developed to measure the sliding gaps between slider and glass substrate[1]. Since VEM is based on ellipsometry, the incident light has to be obliquely illuminated onto the sample surface. To achieve that, the incident light is converged on an off-axis point on the back focal plane of the objective lens, which provides that a parallel light beam obliquely illuminates onto the glass substrate. Meanwhile, the observation system is set to be vertical to the sample surface, which provides high lateral resolution (0.1 μm order) and a wide field of view (100 μm in diameter). By analyzing the intensity image of the reflected polarized light captured by the CMOS camera, the gap between the glass substrate and the slider can be obtained.

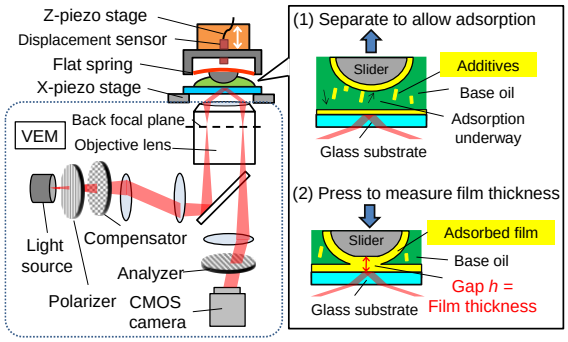


Fig. 1 Schematic setup for adsorbed film thickness and sliding gaps measurement with VEM

Recently, a new method based on VEM was developed to measure the thickness of adsorbed films[2]. In the conventional ellipsometry setup, the incident light cannot be reflected at the interface between the lubricant oil and adsorbed additive film due to the similar refractive indices of the additive and the lubricant oil. As a result, the polarization state of the reflected light would not change with the thickness of the adsorbed additive film. Therefore, the thickness of the adsorbed film cannot be measured in situ with the conventional ellipsometry setup. In order to apply the ellipsometry measurement, the adsorbed polymer films must be isolated from the lubricant oil.

In this method, additive films that adsorbed on the surfaces are isolated from the lubricant oil by squeezing the lubricant oil out of the gap between the surfaces. The procedures for measuring the thickness of the adsorbed film under pressure are as follows (Fig. 1): The slider is separated from the glass substrate, allowing the additives to adsorb onto the solid surfaces. After maintaining the separation for a certain period of time (~10 s), the slider is firmly pressed onto the glass substrate. The gap between the slider and glass substrate is then measured by VEM while the slider is under pressure. When the slider is pressed, only the adsorbed film remains in the contact area because the lubricant oil is squeezed out of the contact area between the slider and glass substrate during the pressing process. Therefore, the measured gap of the contact area at that moment is equal to the thickness of the adsorbed film. By repeating this procedure of separating and pressing, the temporal thickness change of the adsorbed film during adsorption can be obtained. In addition, after the adsorbed process is saturated, the gap change under reciprocating sliding is measured with the glass substrate laterally displaced by X-piezo stage, which can provide the lubricating film formation ability during sliding.

## 3. Experimental samples

The base oil used in this study was the group III mineral oil. The additive was the poly (2-ethylhexyl methacrylate) (PMA-

EH). Two kinds of PMA-EH/ group III mineral oil solutions with concentrations of 2.0 wt%, and 0.3 wt% were used. The glass with the refractive index of 1.93 was used as the substrate, and its thickness was 0.8 mm. The slider was the plano-convex glass lens, which was coated by the 53-nm thick stainless-steel by sputtering method. Its radius was 15.6 mm. The roughness in $R_q$ of the slider surface and the glass substrate was 1.1 nm and 0.4 nm, respectively.

## 4. Experimental procedure

In this study, two type experiments were conducted, as shown in Fig. 2. In the type A experiment, the temporal change in thickness of adsorbed films formed by 0.3 wt% PMA-EH/ group III mineral oil was measured. After the thickness of the adsorbed film was saturated, the sliding gap changes during the reciprocating sliding was measured with the laterally-displacing glass substrate at a constant speed of 0.6 mm/s under different loads.

In the type B experiment, the temporal change in thickness of adsorbed films formed by 2 wt% PMA-EH/ group III mineral oil was measured first. After the thickness of the adsorbed film was saturated, the lubricant oil between the slider and the glass substrate was changed to 0.3 wt% PMA-EH/ group III mineral oil. Next, the gap change during sliding was measured at a constant sliding speed of 0.6 mm/s under different loads. In both type A and B experiments, the amplitude of the triangular-wave sliding motion was set at 15 μm.

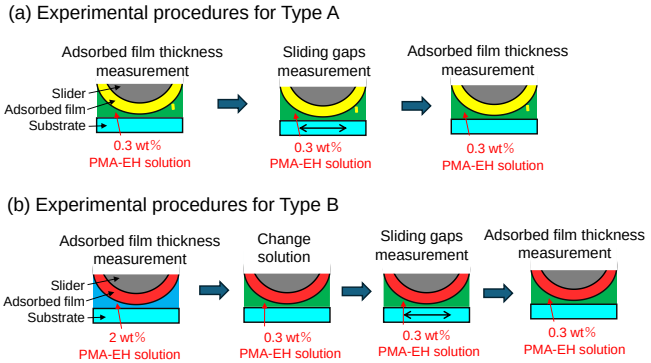


Fig. 2 (a) Experimental procedures for type A, and (b) experimental procedures for type B

## 5. Results

Figure 3 (a) shows measured thickness of adsorbed film formed by different concentration solutions. The saturation thickness formed by 0.3 wt% solution and 2 wt% solution was 1.8 nm, and 3.0 nm, respectively, indicating different conformations of adsorbed films. In the case of 2 wt% solution (Type B experiment), the saturation thickness decreased to 2.3 nm after changing the concentration of solution to 0.3 wt% (red filled circle in Fig. 3(a)). Figure 3 (b) shows sliding gap changes during sliding in the Type B experiment. As mentioned in experimental procedures, the solution was replaced from 2 wt% to 0.3 wt% before the start of the sliding. Therefore, Fig. 3(b) indicated the ability of lubricating film formation of adsorbed film formed by 2.0 wt% solution. Figure 3(c) shows sliding gap changes during sliding in the Type A experiment, where the concentration was kept at 0.3 wt% before and during sliding. Therefore, Fig. 3(c) indicated the ability of lubricating film formation of adsorbed film formed by 0.3 wt% solution. The sliding gaps generated by adsorbed film formed by 2.0 wt% solution (Fig. 3 (b)) was significantly larger than that generated by adsorbed film formed by 0.3 wt% solution (Fig. 3 (c)). These results show that the conformations of adsorbed films strongly affect the lubricating film formation ability during sliding.

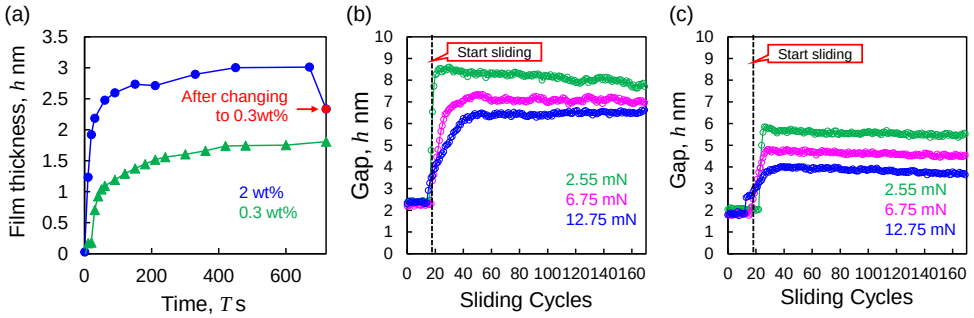


Fig. 3 (a) Temporal changes in thickness of adsorbed films formed by 2 wt% and 0.3 wt% solutions, (b) sliding gap changes with sliding cycles for Type B experiment, and (c) sliding gap changes with sliding cycles for Type A experiment

## 6. Summary

The method for in-situ measuring adsorbed film thickness with VEM was introduced. The lubricating film formation abilities during sliding for different conformations of adsorbed polymer films with the method were investigated.


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### References


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2) Song, Yuxi, et al. "In-situ measurement of temporal changes in thickness of polymer adsorbed films from lubricant oil by vertical-objective-based ellipsometric microscopy." *Tribology International,* 165 (2022): 107341.