

# Evaluation of adsorbed film formation of additives in lubricant oil with vertical-type-objective ellipsometric microscope

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By using the vertical-type-objective ellipsometric microscope (VEM), we proposed a new method for in-situ measuring the thickness of additive adsorbed film in the lubricant oil. The formation process of PAMA polymer additive adsorbed film was successfully observed.

**Keywords:** adsorbed film, polymer additive, ellipsometric microscope, boundary lubrication

## 1. Introduction

Understanding the formation mechanism of adsorbed film is critical to clarify the boundary lubrication. Most studies are unable to quantify the thickness change in the process, which could help reveal the film structure. Ellipsometry is a suitable technology to measure the thickness of thin film in air. Nevertheless, it is difficult to measure the adsorbed film thickness in lubricant oil due to their similar reflective indices. In this study, we proposed a novel method that enables ellipsometry to directly measure the thickness change of adsorbed film during the adsorption process in-situ.

## 2. Experiment Setup

Figure 1 shows the schematic setup of nano-thick film thickness measurement with the vertical-type-objective ellipsometric microscope (VEM). The incident light is converged on an off axis point on the back focal plane of the objective lens, which produces a parallel beam of light illuminates obliquely onto the glass substrate to obtain the ellipsometry signal. The nano-gaps between the metal-coated plano-convex glass lens and glass substrate can be measured by analyzing the ellipsometric images captured by the CCD camera. In addition, the load applied on the lens could be controlled by the flat spring, displacement sensor and piezo stage.

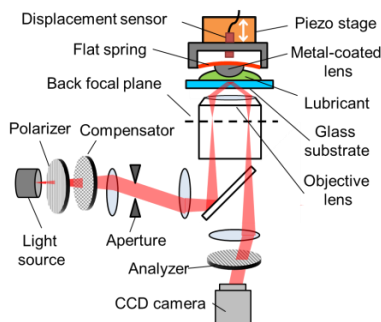


Figure 1: Schematic setup of VEM

## 3. Experiment Procedure and Materials

Without lubricant, the metal-coated lens was firmly pressed on the glass substrate by a high load so as to make the gap between them zero. Then, the base oil with additive was injected into the space between the lens and the glass substrate. After that, metal-coated lens and glass substrate were separated to let additives adsorb onto the surfaces, and the separation was maintained for 10 s. Then, the high load was applied again on the metal-coated lens to squeeze the base oil

out. At this moment, only the adsorbed additive remained between the gap of the metal surface and the glass substrate. Therefore, the measured gap equals to the thickness of the adsorbed film. Next, by repeating the separation and the pressing procedure, the formation process of the adsorbed film could be obtained. In this study, the polymer named non-functionalized polyalkylmethacrylate (PAMA) was used as the additive. The base oil is group III mineral oil; the concentration of all solution is 2 wt%. Poly- $\alpha$ -olefin (PAO) was also used as the control group.

## 4. Results

The measured temporal changes in film thicknesses of different lubricant oils are shown in Fig. 2. The horizontal axis is the total separation time, and the vertical axis is measured film thickness. The measured film thicknesses of PAO4, and base oil of PAMA solution are less than 0.2 nm, indicating no adsorbed film formed. In contrast, the measured film thickness of PAMA solutions increases dramatically in the first 50 s. In addition, with different molecular weights, the adsorption processes are also different. When the molecular weight is high (200 k, 90 k, 60 k), the saturation thickness is around 3.5 nm. As for the low molecule weight (20 k) PAMA, the adsorbed film thickness is quickly saturated with the thickness of 1.5 nm. These results suggest that the adsorbed additive films adopt different structures between small molecules (20 k) and larger ones, which should affect properties of friction reduction performance.

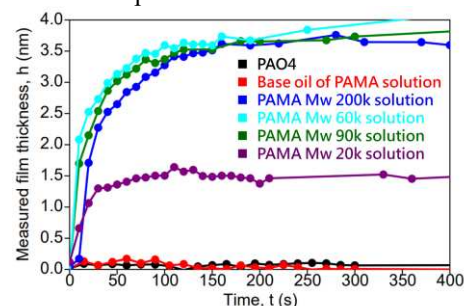


Figure 2: Measured temporal film thickness change of different lubricant oils

## 5. Acknowledgements

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