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Measurement of nanometer-thick lubricating films using ellipsometric microscopy

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ABSTRACT

A method based on vertical-objective-based ellipsometric microscopy (VEM) is presented for measuring lubricant film thickness in nanometer sliding gaps. It provides an image of nanometer-thick lubricating films in real time at high lateral and thickness resolutions without any special layers. The ellipsometric image is directly converted into the film image by using a piezo-stage displacement method combined with a rotating compensator ellipsometry method. The accuracy of thickness measurement is about 1 nm. The VEM-based method revealed that nanometric deformation of the sliding surfaces arises in nanometric gaps even if the load is low, which significantly affects the lubrication properties in small gaps. This method is useful for clarifying the lubrication phenomena in nanometric sliding gaps.

1. Introduction

Along with progress in processing technology, narrower sliding gaps are needed to improve the performance of advanced machines, which requires more precise lubrication technology for the narrower gaps or the lubrication with very thin lubricant films [1,2]. For example, the gaps between the head and disk in computer hard disk drives are now of the order of 1 nm. In addition, since reducing lubricant viscosity can meet social demands for energy saving, the lubrication in small gaps has attracted much attention, such as lubrication with lower viscosity lubricants for automotive engines. However, the lubrication in nanometric sliding gaps has not been fully established yet, because liquids that confined in nanometric gaps exhibit properties different from those of the bulk [3–7].

Measurement of gap shapes or thickness distribution of lubricating film is essential for clarifying the lubrication phenomena in small gaps. Optical-interferometry-based methods have been widely used for this purpose and have provided fruitful results in elastohydrodynamic lubrication in which sliding gaps or film thicknesses are more than about 100 nm; however, the sensitivity of such methods is too low for nanometer-thick film in principle. Since the difference in the light path length is needed to be at least half of the wavelength to give rise to optical interference, it is difficult to measure film thickness less than one-quarter wavelength of the light. If a typical visible light (wavelength of 500–600 nm) is used, the one-quarter wavelength is around 100 nm. That is why the measurement of film thickness less than about 100 nm is difficult for conventional optical interferometry.

A number of methods have been proposed to overcome this problem. One method, which is called spacer-layer imaging or ultra-thin film interferometry, uses a spacer layer consisting of a transparent layer such as a silica layer and a semi-reflective layer such as a thin chromium layer [8,9]. The sliding surface is coated with a spacer layer to elongate the light path. Thickness of the transparent silica layer is needed to adjust so that its optical path length is equal to the one-quarter wavelength. Another method, which is called relative optical interference intensity method, coats the sliding surfaces with a semi-reflective metal film such as a thin chromium layer [6]. This semi-reflective metal film shifts the phase of the light that reflected from the sliding gap, and increases the sensitivity. In the former method, the test sliding surface is needed to be coated with a multilayer film (spacer layer). In the latter method, since the measurement accuracy significantly depends on optical properties of the semi-reflective chromium layer, the precise control of the thickness and refractive index of the chromium layer is needed [10,11]. In Ref. [11], it was reported that a thickness of the chromium layer is needed to be 5 nm for precise thickness measurement. Thus, both methods require coating of test sliding surfaces with precisely-controlled

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special layers, which limits variety of test sliding surfaces and makes test surfaces different from practical ones.

Ellipsometry is widely used for measuring the thickness of thin films on substrates without any modification of the samples, such as adding the special layer. Ellipsometers can provide the thickness of nm-thick films at a single sample point at a thickness resolution of 0.1 nm or less [12,13]. Aimed at improvement in the measurement speed, ellipsometric microscopy (EM), or imaging ellipsometry, uses an imaging device such as a CCD camera instead of a photodetector [14,15]. Such methods could provide the thickness distribution at a time as an image; however, they have low lateral resolution. Oblique illumination is needed to obtain ellipsometric signal as in conventional ellipsometers. Conventional ellipsometric microscopes use oblique observation corresponding to the oblique illumination. In this setup, the focal plane of the objective lens is not normal to the sample plane, which narrows the focus region. This problem is more serious when a higher resolution lens is used. For example, the focus region is only about 1 μm when an objective lens with a numerical aperture of 0.9 is used. Therefore, the lateral resolution is larger than about 10 μm.

Vertical-objective-based EM (VEM) improves the lateral resolution [16–20]. A lateral resolution of 0.1 μm order was achieved for thickness measurement of thin films coated on substrates by combining vertical observation with off-axis Kőhler illumination. It was demonstrated that this VEM can visualize nm-thick solid thin films [16,17] and nm-thick liquid thin films in real time [18–20]. In this paper, we first proposed a method that applies VEM to measurement of nanometric lubricant thickness in sliding gaps, which does not require special layers such as spacer layers and semi-reflective chromium layer. This VEM-based method can provide real-time imaging of lubricating film in nm-sliding gaps at high lateral resolution along with high thickness resolution.

2. Film thickness measurement method based on ellipsometric microscopy

2.1. VEM-based measurement of lubricating film thickness

Fig. 1 shows a schematic setup of film thickness measurement based on VEM. As shown in Fig. 1(a), setting the observation system vertical to the sample surface can provide diffraction-limited lateral resolution of the order of 0.1 μm. Oblique illumination is needed to obtain an ellipsometric signal as with conventional ellipsometers. To achieve this in VEM while remaining compatible with the vertical observation system, the illumination light is focused onto an off-axis point on the back focal plane of the objective lens as shown in Fig. 1(b). This enables oblique and parallel light illumination to be generated, and provides a sufficient ellipsometric signal. Since our method is based on ellipsometry, it has sufficient sensitivity for nanometer-thick lubricating films. In addition, it enables the films to be visualized in real time.

In this measurement, a plano-convex glass lens was used as the slider to make the surface smooth on the nm scale, and the lens surface was coated with a metal layer (stain-less steel layer) to simulate the practical surfaces. Note that this metal coating layer does not require special adjustment for improvement of measurement accuracy, unlike the spacer-layer and semi-reflective layer that described in Section 1. Therefore, for example, a steel ball with a smooth surface can be used as the slider. A glass plate was used as the substrate, as shown in Fig. 1. The light from the light source was reflected at the gap between the metal-coated lens and sliding glass substrate, which is filled with the lubricant. If the amplitude reflectivity ratio of the p- and s-polarization lights are r_p and r_s, respectively, the complex reflectivity ratio of p- and s-polarization lights, ρ, is given by Ref. [12]

$$\rho = \frac{r_p}{r_s} = \tan\Psi(h)e^{i\Delta(h)}, \tag{1}$$

where $\tan\Psi$ and Δ are defined as the absolute value and argument of reflectivity ratio ρ, respectively. Since ρ changes with lubricating film thickness h, the intensity of the reflected light from the gap reflects film thickness h; therefore, film thickness h can be obtained by analyzing the intensity of images obtained by VEM, called “ellipsometric images” here.

2.2. Conversion of ellipsometric image to lubricating film image

Direct conversion from the image intensity to the film thickness was attempted. Direct conversion enables acquisition of the film image at a high frame rate, which can be equal to the maximum frame rate of the imaging device. The conversion curve or the relationship between the image intensity and film thickness is needed. This calibration requires a sample with known film thicknesses or gaps. As shown in Fig. 2(b), a simple approach is to prepare different gap filled with the lubricant by changing the separation between the lens and substrate with a precise drive mechanism such as a piezo stage. With this approach, the physical quantity obtained is the stage displacement, which is not the gap or film thickness but its change. A contact point can be used to convert the displacement into the film thickness. At the points of contact between the lens and substrate, the film thickness is equal to zero; therefore, the piezo displacement at the contact point gives the difference between the displacement and film thickness. However, the gap around the contact point is generally difficult to obtain precisely by using piezo displacement, because the contact surfaces generally deform due to adhesion on the nm-scale. Therefore, another approach was introduced to measure the gap or film thickness around the contact point.

A rotating compensator ellipsometry (RCE)-based method was introduced for measuring the film thickness around the contact point. For

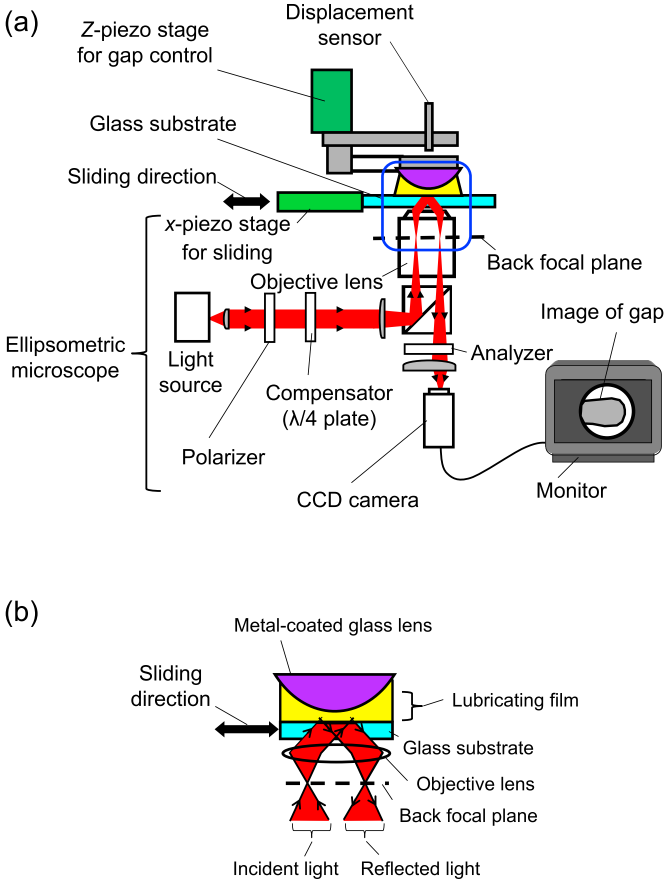

Fig. 1. Schematic setup for measuring lubricating film thickness by VEM: (a) setup and (b) magnified view around gap (region enclosed in blue line in (a)). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)