Chapter 5 Thickness Profile Measurement of Free-Standing Transparent Film

5.1 Background and objectives of the study

In the previous chapter, we proposed a method for measuring transparent films with a substrate (substrate coated film), such as semiconductor wafers and LCD substrates. However, in the case of films without a substrate (free-standing films) such as plastic films, it is difficult to keep the sample stable, and a simpler and more robust measurement method is required.

On the other hand, for the measurement of film thickness of plastic films and their coating films, various techniques have been used such as (1) infrared methods, (2) radiation methods (beta ray, gamma ray, x-ray, x-ray fluorescence, etc.), (3) spectroscopic methods, (4) optical interferometry, (5) capacitance methods, and (6) microwave methods, but most of them are point measurement, and in order to measure the film thickness profile, it is necessary to mechanically scan the target or measuring instrument.

Therefore, in this chapter, we propose a method for measuring the thickness profile of transparent films using a surface profiler [Kitagawa 2007a] and a simultaneous method for measuring film thickness and refractive index [Kitagawa 2007d].

5.2 Measurement of film thickness profile by transmission interferometry

5.2.1 Measurement principle

In the proposed method (hereinafter referred to as the TF method), a surface profiler is used to measure the thickness profile of a stand-alone transparent film by inserting it into one side of the interference light path and detecting the optical path difference due to the film. In other words, the film thickness is obtained from the change in height measured by the insertion of the film on a separately prepared flat surface as the measurement reference plane. For example, as shown in Fig. 5-1, when a transparent film is inserted into one part of the measurement field of view of the surface profiler and a flat reference surface is measured, an optical path difference due to the presence or absence of the film is generated, resulting in a step $D\ [= (n-1)t]$. Therefore, if the refractive index n is known, the film thickness t can be calculated by

$$t = D/(n-1) \tag{5–1}$$

The transparent film should be separated from the reference surface and kept outside the coherence length. This method can be applied to thin or multilayered films. In addition, the measurement is not affected by the position of the film or its waviness, and the measurement is stable.

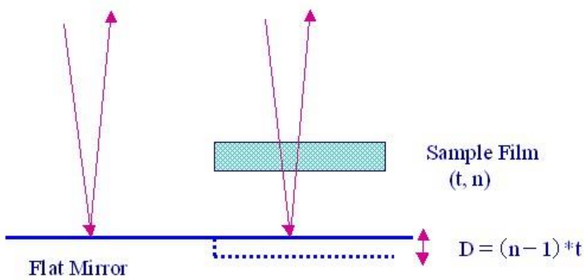


Fig. 5-1 Principle of TF method

5.2.2 Experiments with wide field-of-view interferometer

(1) Experimental conditions

A wide field-of-view surface profiler SP-530 (Fig. 5-2) developed by the authors, was used as the measuring device. This Michelson interferometer is a non-microscopic interferometer with a long working distance (WD) and a wide field of view (about 200 mm). Several polyester films (with a nominal refractive index of 1.65) with a nominal film thickness of 0.9 to 6.0 μm were attached to the slide mount, as shown in Fig. 5-3. A metallic mirrored hard disk drive substrate was used as the reference flat surface, and the vertical scanning method (scanning speed of 2.4 μm/s) was used to measure it.

Fig. 5-2 Wide Field-of-View Surface Profiler SP-530

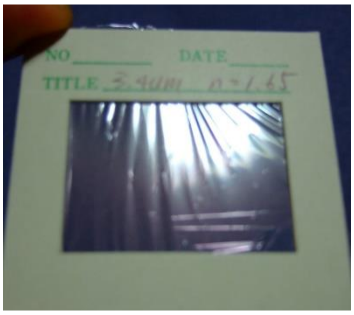


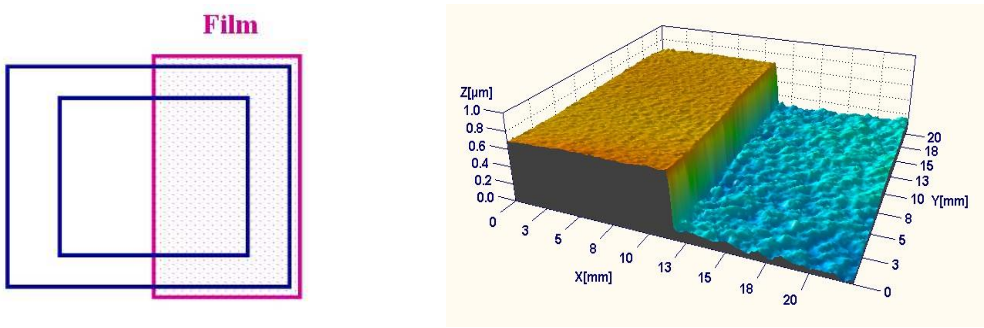
Fig. 5-3 Film sample fixed in a slide mount

(2) Experimental results and discussion

The measurement results for polyester film with a nominal film thickness of 0.9 μm are shown in Fig. 5-4. The average step difference of 0.58 μm and the refractive index of 1.65 yielded a film thickness of 0.89 μm.

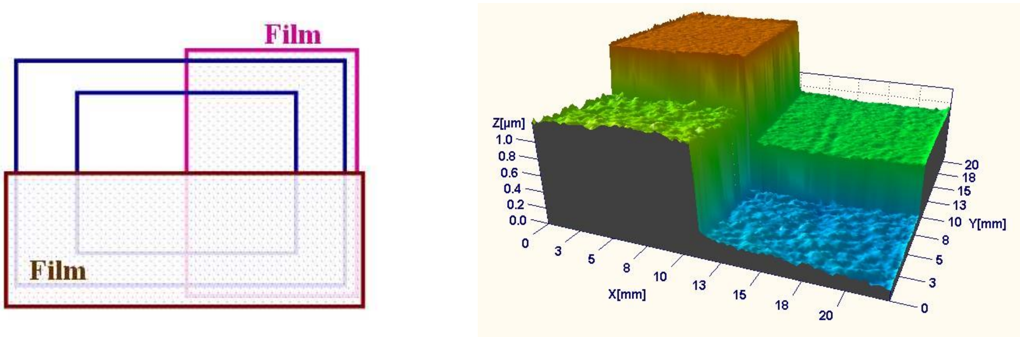
Next, Fig. 5-5 shows the results of cross-laying two films with a film thickness of 0.9 μm and 1.5 μm. The measured step values were 0.55 μm, 1.03 μm and 1.60 μm with the film-less area as the reference. These values were converted to film thicknesses of 0.84 μm, 1.58 μm and 2.46 μm, respectively.

The correlation between the nominal film thickness and the measured values for several film samples is shown in Fig. 5-6. Good correlations were obtained for samples with nominal film thicknesses of 0.9 - 6.0 μm and their superimposed samples, confirming the validity of the proposed method.



(a) Measured film sample (b) Measured surface

Fig. 5-4 Measured sample and surface profile of a mirror surface, with a PET film of a nominal 9-μm thickness inserted in the right half



(a) Measured film sample (b) Measued surface

Fig. 5-5 Measured sample and measured surface profile of a mirror surface, with two PET films inserted

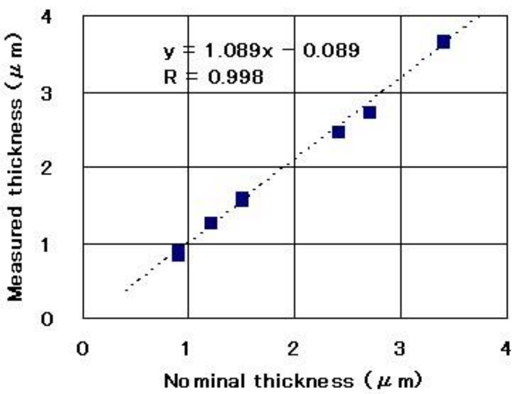


Fig. 5-6 Correlation between the measured values and the nominal values

5.2.3 Experiments with an interference microscope (low magnification)

(1) Experimental conditions

A microscopic surface profiler SP-500 (Fig. 2-10) was used as the measuring device. In the case of a low-magnification objective lens, the distance (WD) between the lens and the specimen is large, so that a transparent film can be inserted between them. However, it would disrupt the microscopic imaging system. Here, we used a 5X lens and changed the sample height as shown in Fig. 5-7 to investigate the effect on the measured values. The thickness of the slide glass used as spacers was about 1.3 mm.

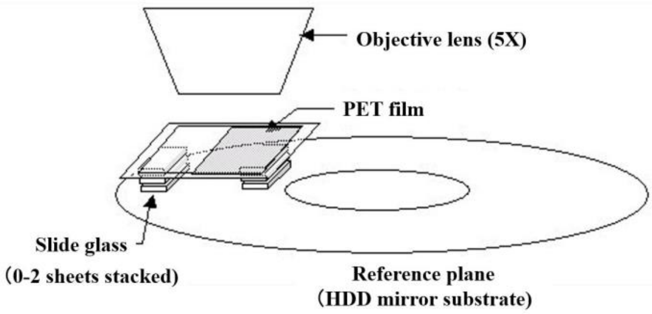


Fig. 5-7 Test method of effect of object position

(2) Experimental results and discussion

Figure 5-8 shows the height profile of a sample with a nominal thickness of 0.9 μm inserted in the right half of the field of view of the microscope, and the number of spacers is changed. The height change in the horizontal direction is displayed based on the height of the left edge of the filmless part.

As the number of spacers increases, the step edges become duller, and the correct step cannot be measured with two spacers. This corresponds to a disorder of the microscopic imaging system and blurred edges.

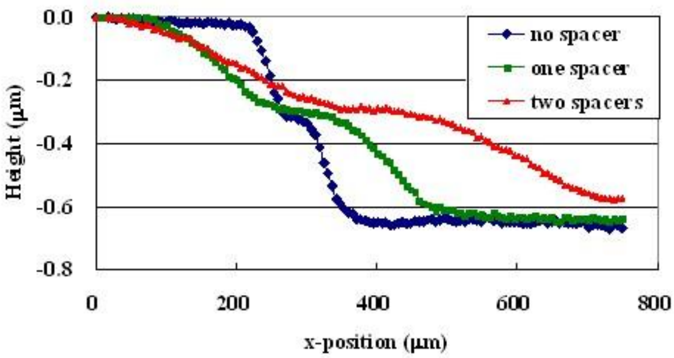


Fig. 5-8 Effect of object position

5.2.4 Experiments with an interference microscope (high magnification)

(1) Experimental conditions

In the case of a high-magnification objective lens, it is difficult to insert a partially transparent film between the field of view of the objective lens and the specimen because the distance (WD) between the lens and the specimen is small. Therefore, we inserted the transparent film into the entire field of view and obtained the film thickness from the difference in measured heights before and after. A sample with a nominal film thickness of 0.9 μm was measured using a 20X lens.

(2) Experimental results and discussion

The measurement results before insertion of the sample are shown in Fig. 5-9, the results after insertion are shown in Fig. 5-10, and the film thicknesses obtained from the differences are shown in Fig. 5-11. The average measured film thickness was 0.85 μm compared to the nominal thickness of 0.9 μm.

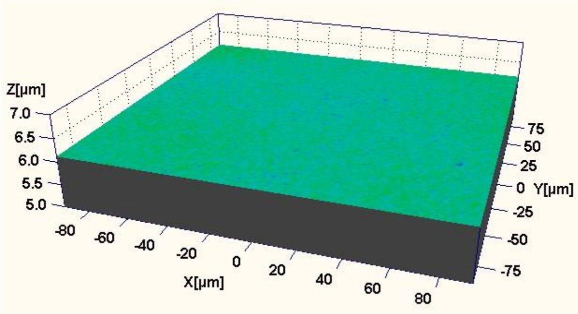


Fig. 5-9 Measured surface profile without sample

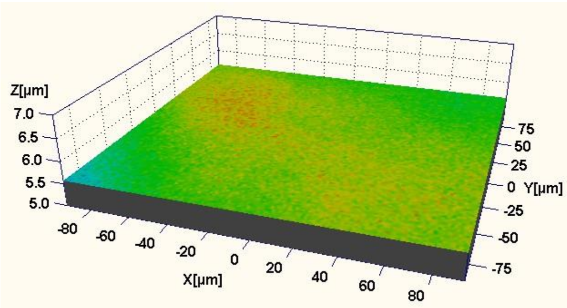


Fig. 5-10 Measured surface profile with sample

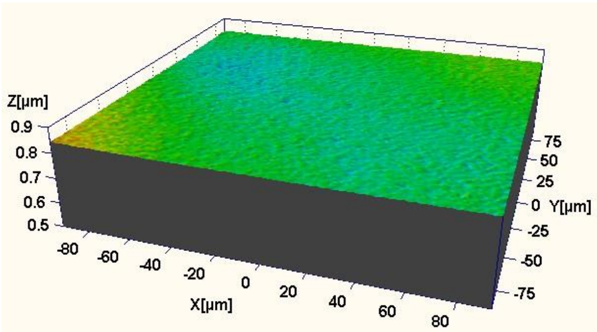


Fig. 5-11 Measured thickness profile

The problem with this method is the possible change in the reference surface height between the two measurements. To solve this problem, we measured again the reference surface, and the average height of the first and third measurements was adopted as the reference plane height. The variation was 23 nm.

5.2.5 Comparison with KF method

The proposed method is compared with the KF method described in Chapter 4.

(1) Experimental conditions

A lens with magnification 5X was used, and the sample with a nominal thickness of 1.5 μm was placed close to the reference plane with the sample occupying the right half of the field of view. The vertical scanning speed was set to 1.2 μm/s.

(2) Experimental results and discussion

The film thicknesses of the same samples were measured using two different methods: the TF method is shown in Fig. 5-12 and the KF method is shown in Fig. 5-13. The average film thickness profile of the 50 central lines by the two methods is shown in Fig. 5-14.

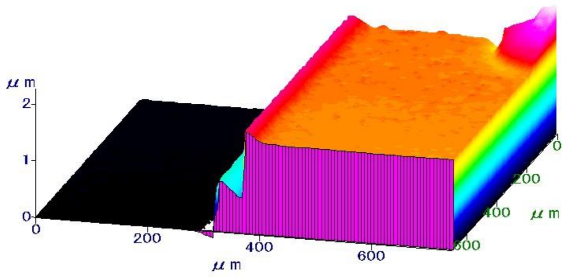


Fig. 5-12 Film thickness profile by the TF method. The upper right protrusion is a marker.

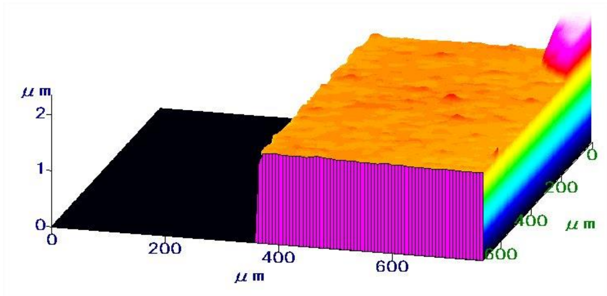


Fig. 5-13 Film thickness profile by the KF method

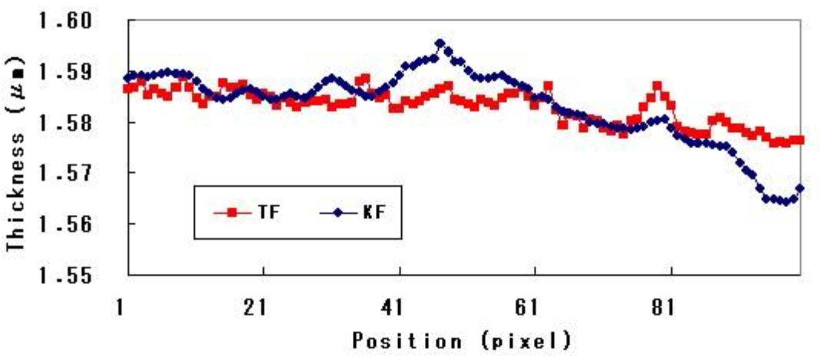


Fig. 5-14 Film thickness profiles by two methods

Except for the turbulence at the edge of the film, the TF method was comparable to the KF method and gave stable results. The partial difference in the thickness of the two films may be due to either (1) unevenness of the reference surface or (2) refractive index error.

Compared with the KF method, the TF method has the following features.

(1) In principle, there is no limit to the measurement range

(2) Only the film thickness can be measured (surface topography cannot be measured)

(3) Resistant to external disturbances such as sample vibration

5.2.6 Effect of wavelength dispersion

In general, the refractive index of transparent films is not constant, but has a wavelength dependence (wavelength dispersion). When the phase shift method is used for measurement, there is no effect of wavelength dispersion because the illumination is monochromatic. When white-light interferometry is used, as in the present experiment, it is necessary to take into account the effect of wavelength dispersion.

When the refractive index of a sample with a film thickness of t changes from n to $n+\Delta n$, the optical path difference change is $t\Delta n$. In the wavelength band used for white-light interferometry, since the wavelength dispersion Δn of general materials is on the order of 1/1000, the peak position change due to the optical path difference change is on the order of 1 nm per 1 μm of film thickness. This is sufficiently small compared to the 300 nm period of the interference waveform and does not pose a problem for interference measurements.

5.2.7 Comparison with other interference methods

The proposed method is also applicable to the case of the Fizeau interferometer (Fig. 1-5). On the other hand, the Mach-Zehnder interferometer, shown in Fig. 5-15, is known as an optical interferometer capable of measuring the thickness of transparent films. This method is a transmission interferometry, which is the same as the TF method in principle and does not require a reference plane. However, it is not available on the market as a general product. Also, it is not easy to realize the microscopic system.

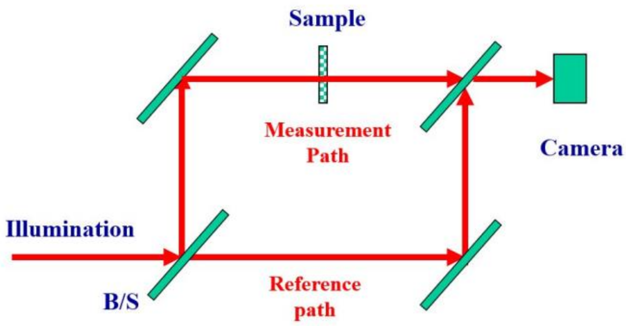


Fig. 5-15 Mach-Zehnder interferomter

5.3 Simultaneous measurement of film thickness and refractive index

5.3.1 Background and objectives of the study

In the previous sections, a method for measuring the thickness profile of transparent films was proposed. However, it is not possible to separate the refractive index from the film thickness. The film thickness is usually obtained with the refractive index as a known value.

However, the refractive index may be unknown, and there is a need for simultaneous measurement of film thickness and refractive index. Several methods have been proposed so far, such as the combination of confocal and interferometric optics [Fukano et al. 1996, Maruyama et al. 2002], the polarization analysis method, and the multiple incidence angle method. They require special optics. In addition, since most of them are point measurement, mechanical scanning is necessary to measure the distribution.

We propose a simultaneous measurement of the film thickness and refractive index of stand-alone transparent films using an optical interferometer [Kitagawa 2007d].

5.3.2 Algorithm

When measuring a transparent film sample, the optical film thickness nt is obtained by the KF method and the optical path difference (OPD) is obtained by the TF method. By combining the information obtained by these two methods, it is possible to measure both the film thickness and the refractive index separately. That is, if the optical film thickness is T and the step is D, then

$$T = nt \tag{5–2}$$

$$D = (n-1)t \tag{5–3}$$

Therefore, the film thickness and refractive index can be obtained independently from the following equation.

$$t = T - D \tag{5–4}$$

$$n = T/(T-D) \tag{5–5}$$

This method was originally devised by us when we were discussing the results of the KF and TF methods, but a survey of the literature shows that the principle is briefly described in the specification of an old patent [Kalliomaki et al. 1987]. However, there is no experimental data and little is known about it.

5.3.3 Measurement results

(1) Thin glass plates

Two kinds of thin glass plates (nominal thicknesses of 30 μm and 50 μm; nominal refractive index of 1.523) were measured by using a surface profiler SP-500. The refractive index and thickness profile of a 30-μm nominal thickness sample is shown in Fig. 5-16, and the refractive index and thickness profiles obtained from these data are shown in Fig. 5-17. Although both are obtained as 3D data, here we show the profile data on one line. The refractive indices of two glasses of different thicknesses were measured as nearly identical.

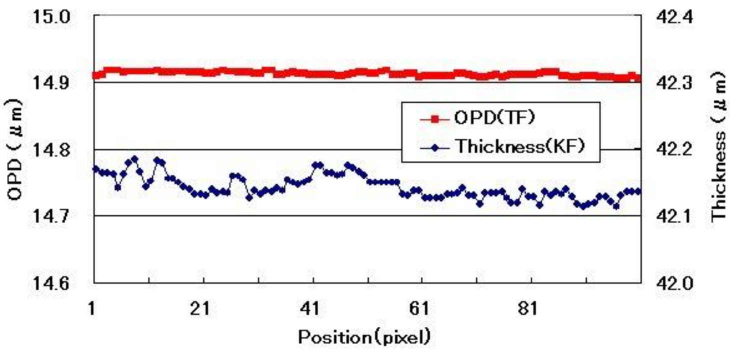


Fig. 5-16 Profiles by the KF and TF methods

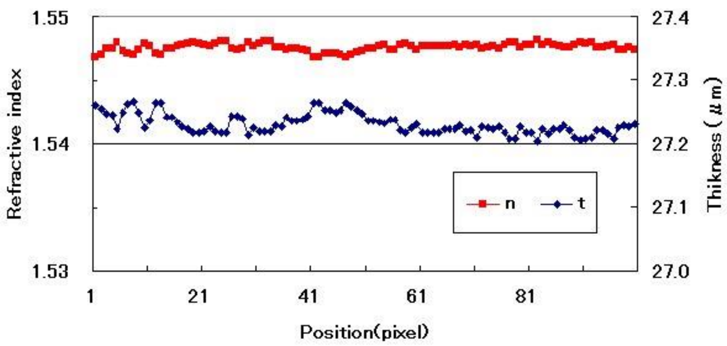


Fig. 5-17 Thickness and refractive index profiles of glass

Table 5-1 Test results of glass plates

Nominal thickness	Thickness given by supplier	Measured thickness Mean ± in-plane σ	Nominal refractive index	Measured refractive index Mean ± in-plane σ
30 μm	33 μm	27.23 μm ±0.02	1.523	1.547 ±0.000
50 μm	51 μm	50.66 μm ±0.03	1.523	1.545 ±0.000

Note: The nominal refractive index is of the D-line (589nm) n_D.

(2) Plastic films

Polyester (PET) film with a nominal film thickness of 1.5μm and a nominal refractive index of 1.65, and polyethylene (PE) film with an unknown film thickness and a nominal refractive index of 1.53 (commercially available wrapping film) were attached to the slide mount as shown in Fig. 5-18. The optical film thicknesses measured by the KF method and the step difference by the TF method are shown in Fig. 5-19. The left is the PET film, the right is the PE film, and the center is the area without a film. The refractive index and film thickness profiles obtained from these data are shown in Fig. 5-20. The difference in refractive index is clearly measured. The nominal and measured (in-plane average) values are shown in Table 5-2. Both the film thickness and refractive index were close to the nominal values. Note that the disturbance in the refractive index profile of the PET film is due to the thin film thickness, which causes wrinkles and disturbs the film thickness data of the KF method.

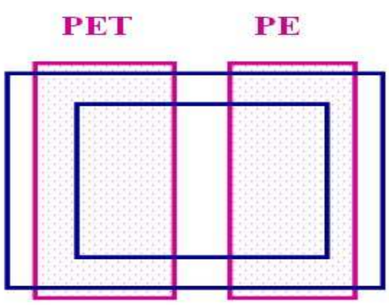


Fig. 5-18 Two film samples of PET and PE fixed on a mount

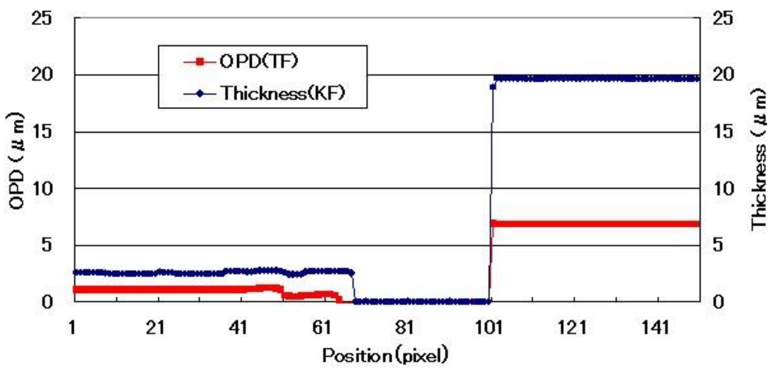


Fig. 5-19 Profiles by the KF and TF methods

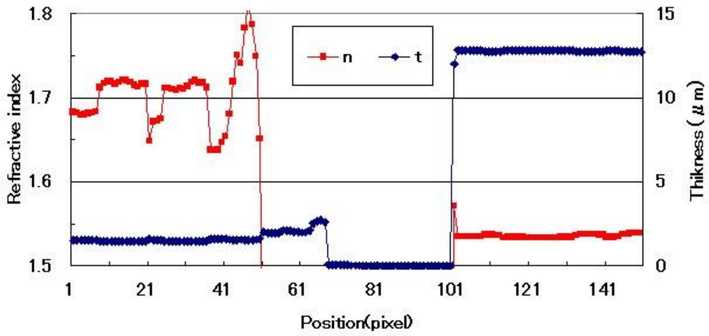


Fig. 5-20 Film thickness and refractive index profiles calculated from the results of the KF and TF methods

Table 5-2 Test results of plastic films

Sample	Nominal thickness	Thickness by contact probe	Measured thickness	Nominal refractive index	Measured refractive index
PET	1.5	1.6	1.51	1.65	1.69
PE	-	12	12.8	1.53	1.53

Note: Thickness is in μm. Thickness by contact probe is 5-point average.

5.4 Summary

We have proposed a method of determining the thickness profile of a stand-alone transparent film by inserting it into the optical path of an interference system and determining the film thickness from the change in the measurement results of the reference surface. First, the basic principle was confirmed by the wide-field-of-view interferometer. In addition, in the microscope-type interferometer, the effect of the sample height was investigated, and the practicality of the two-step measurement method was confirmed. This method can easily and stably measure the thickness profile of a transparent film using a commercially available reflective-type surface profiler, without being affected by the position or waviness of the sample. This method can also be applied to ultra-thin and multi-layered films.

Next, we proposed a method for simultaneous measurement of the film thickness and refractive index of transparent films and plates using white-light interferometry. The effectiveness of the method was confirmed by measuring thin glass plates and plastic films using a commercially available surface profiler. It is characterized by the ability to obtain four types of three-dimensional data, including the surface profile, back surface profile, film thickness profile, and refractive index profile of the transparent film, in a non-destructive, non-contact manner.

This is an English translation of the article:
Katsuichi Kitagawa, "Chapter 5: Thickness Profile Measurement of Free-Standing Transparent Film",
in "Study on industrial surface profile measurement method based on optical interferometry", pp. 50-59, Doctoral Dissertation, University of Tokyo (2011)
(original in Japanese; translated by the author in Oct. 2020)