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● SPECTROPHOTOMETRIC ANALYSIS
No.A350

Pretreatment Methods for Eliminating the Interference Fringes

As already introduced in Application News A313 and A335, interference fringes appearing in film transmission spectra are used for calculating film thickness, because their period vary depending on the light's incidence angle and the film's refractive index and thickness.

However, interference fringes also affect peak positions and intensities. Therefore, they can impede not only quantitation, but also qualification and identification of unknown substances.

This Application News introduces a pretreatment method that will decrease the effect of interference fringes in transmission measurements. Some caution points will be also indicated.

■ Interference Fringes

Interference fringes are caused by light being reflected inside the sample. Fig. 1 shows an overview of how light transmits through a sample. Infrared light directed to the sample is split into light (A) that passes through the sample, light (B) that is reflected at the front surface of the sample, light (C) that is reflected at the back surface, light (D) that is reflected twice at the front and back surfaces, and light (E) that is reflected three or more times at the front and back surfaces. In case of the sample is polymer film, the percent reflectance at the atmospheric interface is 10% or less. Therefore, lights (B) through (E), especially light (E), are much weaker than to the transmitted light (A).

Among these lights (A) through (E), the lights (A) and (D) are directed to the detector. However, (D) has a different phase from (A) due to multiple reflections in the film. The existence of lights with different phases as shown here causes interference fringes. So by removing light (D) the interference fringes can be eliminated.

One method to reduce (D) is to make the sample surface rough by using a sand paper. By making the sample surface rough, regular reflection is reduced due to scattering at the sample surface, and thereby (D), and in turn interference fringes are reduced. However, transmitted light (A) is also affected by scattering, especially the shorter its wavelength is the bigger the influence by scattering is. This leads to baseline lowering at higher-wavenumber ranges in the display of transmittance.

This report introduces a pretreatment method where the sample is enclosed with an infrared transmitting material KBr. The sample is sandwiched between two KBr plates and encased using a method similar to pressing tablets. Using this method, interference fringes can be eliminated without the effects of light scattering. Fig. 2 shows a schematic view of the analysis.

Lights are scarcely reflected at an interface between materials having the same refractive index. The refractive indices of general organic materials and the atmosphere are around 1.5 and 1.0 respectively. Therefore, several percent of surface reflectance occur. Because KBr's refractive index is 1.52, the reflection at the boundary between KBr and the sample is extremely small. This means light (D) that is reflected inside the sample becomes extremely small, and thereby interference fringes are eliminated.

Fig. 3 shows the transmission spectra for commercial wrapping film. The upper spectrum is obtained by conventional analysis method, and the lower spectrum is obtained by an analysis where the sample was encased with KBr. It is clear that the interference fringes are completely eliminated by KBr encasing. The peak intensities in the spectrum for the encased sample are smaller than those in the upper spectrum. This is thought to be due to the film thickness being pressed thinner by the encasing process.

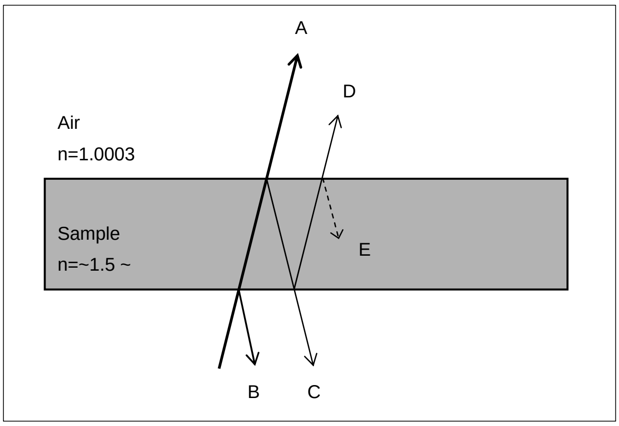

Fig.1 Schematic Diagram of Transmission Spectroscopy

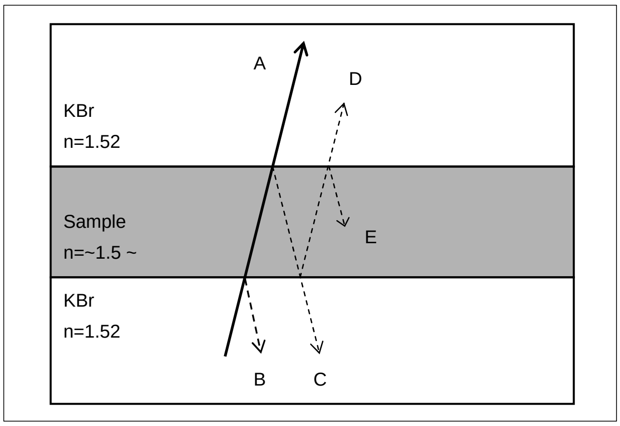

Fig.2 Schematic Diagram of Transmission Spectroscopy using KBr Plates

■ Applying the Method to Infrared Microscope Measurements

Interference fringes also affect analysis using an infrared microscope, causing difficulty in analyzing spectrum in the analysis of foreign matter or laminated films. In particular, when cutting out a cross section of a laminated film using a microtome for analyzing each layer, the smoother and cleaner the cut surfaces are, the greater the affect of interference fringes. In this case, interference fringes can be also reduced or eliminated by encasing the sample in KBr.

Fig. 4 shows a micrograph of a cross section of a laminated film having four layers that is placed on a KBr plate. It was sliced using a microtome to a thickness of 14µm. The two inner layers are polyvinylidene chloride (an acrylic copolymer) and polyethylene, and both are about 20µm thick.

Fig. 5 and 6 show the transmission spectra for these two layers. Fig. 5 shows the spectra for samples placed on a KBr plate and Fig. 6 shows the spectra for samples encased in KBr. In Fig. 5, the baselines show large fluctuations due to interference fringes. However, in Fig. 6, the baselines are almost flat, not affected by interference fringes.

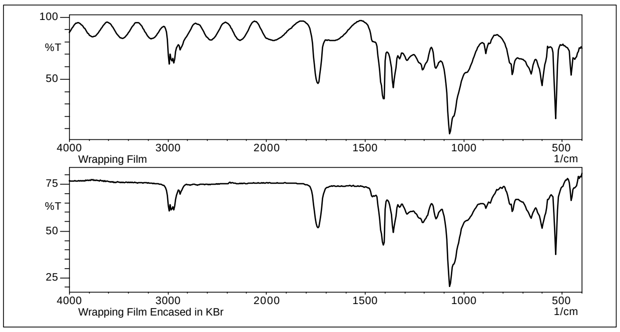

Fig.3 Transmission Spectra for Wrapping Film Upper: Without KBr Encasing, Lower: Encased in KBr

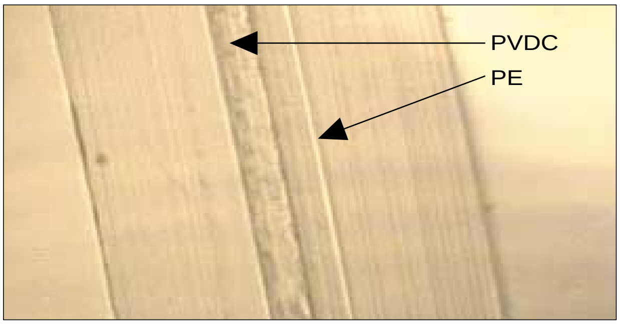

Fig.4 Micrograph of Cross-section of Laminated Film

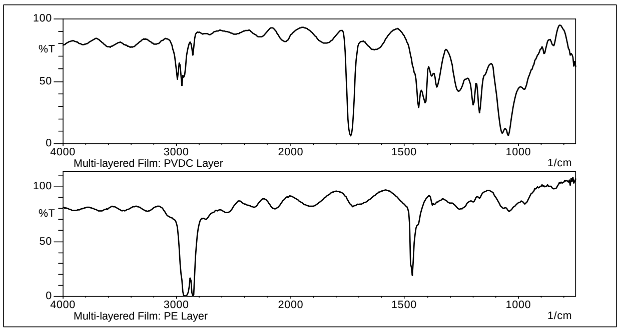

Fig.5 Transmission Spectra for Cross-section of Laminated Film Placed on KBr Plate

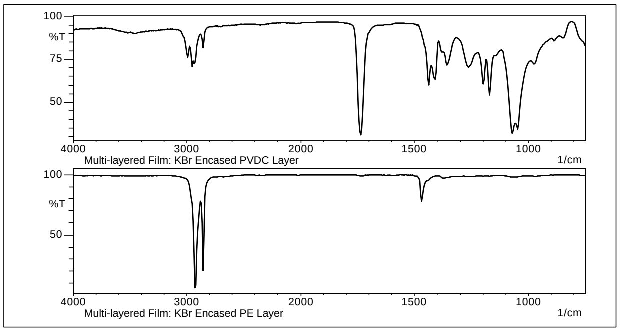

Fig.6 Transmission Spectra for Cross-section of Laminated Film Encased in KBr

■ Points to Note

The pretreatment method introduced here is very effective for reducing or eliminating the effect of interference fringes. However, there are several points those should be considered.

First, this method involves pressing the sample between two KBr plates, so the sample becomes thinner. This will cause the peak intensities to be smaller. Also, in this method, it is not possible to observe the sample during the pressing process, which is possible when pressing micro-sized objects with a diamond cell by using a microscope. Therefore, care must be taken not to press the sample excessively thin.

Second, when measuring micro samples using an infrared microscope, observation is difficult for encased samples. Even when the sample is clearly observed on a KBr plate or diamond cell, the contrast decreases by KBr encasing. In addition, if the sample is encased at an angle, only a small portion of the sample can be focused, making observation even more difficult. The cloudiness of the KBr plates is another obstacle to observation that especially affects white or transparent samples.

Finally, special care must be taken when recovering samples encased in KBr. KBr easily dissolves in water, so it can be easily removed by washing the sample with pure water. However, if the sample or substances contained in the sample are water soluble or affected by water, the sample cannot be recovered in their original state. A care is also needed for contamination.

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