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Application News

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Spectrophotometric Analysis

Thickness Determination of Film and Coating on Inside of Can Using IR Pilot

The Shimadzu IRSpirit launched recently is a compact Fourier transform infrared spectrophotometer characterized by the highest signal to noise ratio and the highest maximum resolution in its class (Fig. 1). The dimensions of the body are W390 × D250 × H210 mm and less than A3 in size. The IRSpirit adopts a unique design which allows not only lateral installation but also longitudinal installation so that it can be installed in a narrow space in width. The IRSpirit also has another feature that it can mount the existing accessories such as a single reflection type attenuated total reflection attachment and a diffuse reflection attachment and the commercially available accessories in addition to the accessories for the transmission measurement such as a demountable cell and a KBr pellet holder.

A dedicated program named IR Pilot to assist the analysis, which allows four measurement items - identification test, contaminant analysis, quantitative analysis and film thickness determination - to be easily operated, is equipped in the LabSolutions IR software which controls the IRSpirit and executes data manipulations. By using the IR Pilot, successive operations of measurement, analysis and print can be orderly performed following the on-screen instructions. The IR Pilot is comprised of twenty-three kinds of the dedicated program of which the often-used program up to four kinds can be registered in its main menu.

This article introduces the film thickness determination using the IR Pilot.

Y. Suzuki

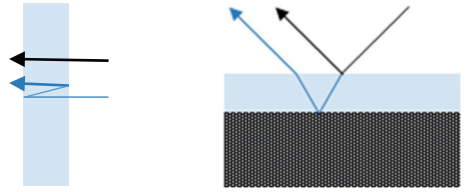
Fig. 1 Shimadzu IRSpirit compact FTIR

Principle of Film Thickness Determination

A variety of films are used in products used in daily life. For example, a multi-layer film used for the food packing laminates films to allow functions such as moisture impermeability and blocking performance against gases and light. For a touch panel in a smartphone, films providing the antireflection effect and the electric characteristic are laminated.

The film thickness determination performed by using interference fringes appearing in an infrared spectrum is one of the determination methods. Since infrared light doesn't damage a sample, the measurement can be conducted without destroying and touching it.

In the case of the transmission measurement of a film, a beam transferring to the direction of travel again after being reflected inside a film interferes with an incident beam (Fig. 2 a). For the reflection measurement of a coating on the metal surface, a beam reflected on the surface of the coating interferes with that reflected on the metal surface after transmitting the coating (Fig. 2 b).



(a) Transmission Measurement (b) Reflection Measurement

Fig. 2 Interference of Light

As a result, interference fringes whose intensities change like a sine wave as shown in Fig. 3 are observed in an infrared spectrum. Although interference fringes appear regardless of the existence or nonexistence of an absorption peak, they are clearly observed in the region where no peak exists.

Fig. 3 Interference Fringes

For both transmission spectroscopy and reflection spectroscopy, film thickness (t) is calculated using the following equation.

where n = refractive index of a sample, M = number of complete sine waves in the wavenumber range from ν_1 to ν_2 (order of interference), θ = incident angle. For transmission spectroscopy, θ is zero.

$$t = \frac{M}{2\sqrt{n^2 - \sin^2\theta}\,(\nu_1 - \nu_2)}$$

Film Thickness Determination Using the IR Pilot

After starting the IR Piot, "Spectrum" program in the LabSolutions IR is displayed on the screen and then the IRSpirit is automatically initialized. After the initialization, "Film Thickness Determination" in the main menu shown in Fig. 4 is selected as the purpose of analysis.

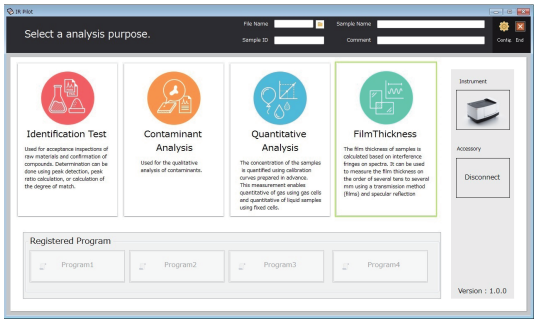


Fig. 4 Main Menu

Then incident angle and refractive index of a sample are input in a screen shown in Fig. 5 before the measurement. Zero degrees (transmission measurement) and 10 degrees (incident angle of the SRM-8000 specular refection attachment) are selectable. If a sample is measured at the other angle, the angle also can be input using "Custom".

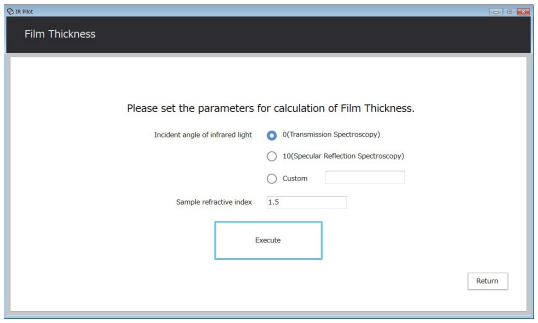


Fig. 5 Screen to Input Incident Angle and Refractive Index of Sample

At first, a polystyrene film was measured in transmission spectroscopy and its thickness was determined. The wavenumber range used for the film thickness calculation is chosen in a measured spectrum using a screen shown in Fig. 6. The wavenumber range where no absorption peaks are observed and only interference fringes appear should be selected. Here, the wavenumber range from 3833 cm^{-1} to 3205 cm^{-1} was selected.

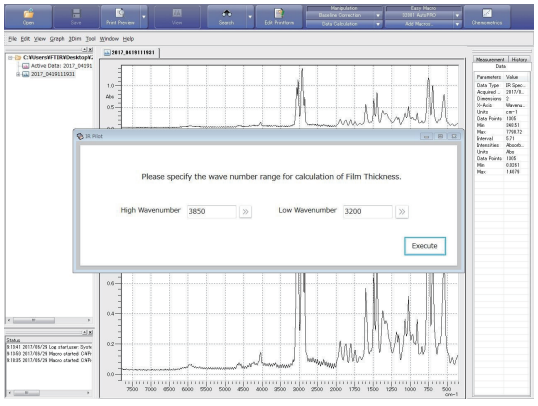


Fig. 6 Screen to Specify Wavenumber Range of Interference Fringes and Its Expanded One

The number of complete sine waves in the input range is automatically obtained. Then the film thickness is calculated using the selected incident angle, the input refractive index and the obtained number of waves. Fig. 7 shows a screen for the calculation result. The measured polystyrene film stood at 57 μm in thickness.

Fig. 7 Result of Film Thickness Determination

Next, the film thickness determination by reflection spectroscopy was performed. The coating on the inside of the bottom plate of a commercially available juice can was measured with the SRM-8000 specular reflection attachment (incident angle : 10 degrees). The infrared spectrum and the calculated result are shown in Fig. 8. The thickness was 21 μm.

A reflection measurement attachment with other incident angle is also available because the film thickness determination can be calculated with an arbitrary incident angle.

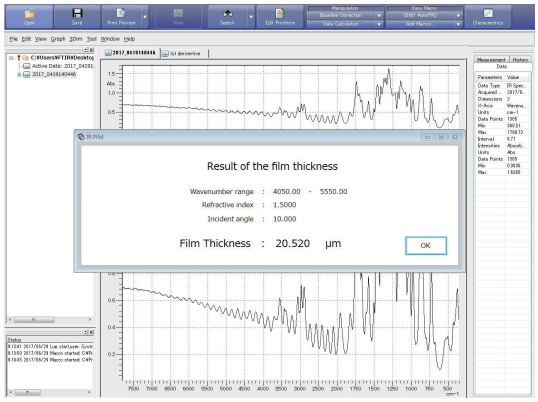


Fig. 8 Measurement of Coating on Inside of Beverage Can by Reflection Spectroscopy

Conclusion

The film thickness determination can be applied to a variety of samples such as a film, a coating and a semiconductor. An easy operation for the film thickness determination introduced in this article allows the labor-saving and efficient measurement operation. The IR Pilot can execute successive operations of the setting of measurement parameters, the background measurement, the sample measurement, the data manipulation and the print automatically and facilitate the film thickness determination. The IR Pilot allows a beginner of FTIR and a user conducting the routine measurement to use the IRSpirit more comfortably according to the proper procedures. Please try to use it.

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Shimadzu Corporation
www.shimadzu.com/an/