Application Note

260-AN-0010

Measurement of buried foreign material by Raman Spectrophotometer NRS-4100

Introduction

Currently, the infrared microscope is used extensively as one of the identification approaches for micro foreign materials. Since in microscopic infrared spectroscopy there is a huge database, it work very well on the identification of foreign materials, while the infrared microscope has several problems for the measurement such that the spatial resolution is limited to only a few micrometer and the sample preparation is necessary if the foreign material is buried in the sample. Therefore, the Laser Raman spectrophotometer is now often used for measurement of foreign materials in combination with infrared microscope.

Raman spectroscopy is a method to analyze molecular structure by molecular vibration as well as infrared spectroscopy, but there are following advantages in Raman spectrophotometer.

(1) The spatial resolution is as small as 1 μm by using visible laser.
(2) The Raman spectrophotometer allows quick and easy measurement of the sample with non-destructive manner without sample pretreatment.
(3) For inorganic samples, it is easy to indentify because of the easy measurement in low wavenumber range.

The potential of measuring foreign materials by using Raman spectrophotometer is expanding, and JASCO has developed a new laser Raman spectrophotometer, NRS-4100 with compact design and ease of use to be used together with FTIR. In this application note, the features of NRS-4100 and identification of foreign materials buried in the polymer film are illustrated.

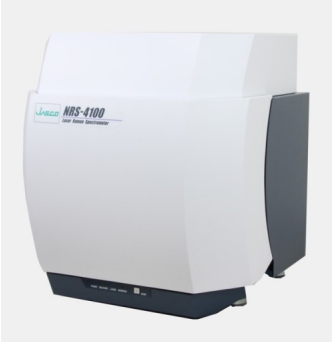

Fig. 1 NRS-4100

Features of NRS-4100

The NRS-4100 as shown in Fig. 1 is a Raman Spectrophotometer, incorporating high performance spectrograph, sample compartment, detector and laser light source in a space as small as 60 cm square, which can be installed on the ordinary laboratory bench other than anti-vibration bed, with no extra space because the door of sample compartment moves up and down for open/close In addition, the NRS-4100 meets the laser safety standards of Class 1.

Maximum three lasers such as 457nm and 785nm as well as 532nm can be mounted, and the spatial resolution is as small as only 1 μm in XY and 1.5 μm in Z direction, enabling the high spatial resolution and fluorescence minimization, which are important for the foreign material measurement.

The "Measurement assist function" aids the user in setting up the NRS-4100 for sample measurement; a simple sequence guide takes you through setup and optimization of measurement parameters with helpful advice and tips, such as a warning if you have the laser intensity set too high. The new "Sample Search" function is used with the automated XYZ stage. A new algorithm developed by JASCO (patent pending) analyzes the microscopic image and automatically selects measurement position(s) based on the size, contrast and/or color of the target material described by the user, then simply click the measurement button to execute spectral measurements of the automatically identified sample positions. Spectra Manager II includes a wealth of user-selectable options for data analysis, as well as the usual tools like opening single or multiple spectra, zooming, normalization, a variety of arithmetic data processing functions, there is a variety of Raman specific tools and analysis functions.

Fig. 2
Measurement assist function

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System configuration

- NRS-4100 Raman Spectrometer
- Automatic imaging system
- 532 nm laser (100 mW)

Foreign material measurement/Analysis

The foreign material buried in the multi layer substrate (Glass/Adhesion layer/Transparent film) shown in Fig. 3 was measured. It is difficult to measure such foreign material by using infrared microscope, because it is difficult to cut the foreign material in section due to the presence of glass and the adhesion layer may be picked up together. On the other hand, Raman spectrophotometer with the confocal optical system can obtain the spectrum of laser focused point selectively. As a result, it is possible to measure the inside of the sample in non-contact and non-destructive manner without lousy sample pretreatment. In this report, the position where the target foreign material is located was measured in the depth direction (Z axis direction) and also each layer's information was obtained. The major spectrum obtained from each layer is shown in Fig. 4.

Measurement parameters

Ex wavelength: 532 nm Grating: 900 gr/mm Exposure time: 5 sec. (Accumulation: 2 times)

Fig. 3 Observation image

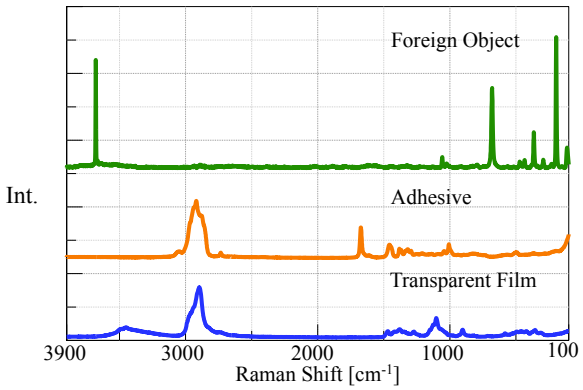

Fig. 4 Spectrum for each layer

In the obtained spectrum of the foreign material, the C-H peak at around 300 cm^{-1} is not shown, and so it is quite different from the spectrum of transparent film and adhesion layer. In order to analyze the result in further details, the spectrum of foreign material was tried to be indentified by using database as shown in Fig. 5, and the foreign material was found to be talc (hydrated magnesium silicate). In addition, it is known that the transparent film is made of cellulose and the adhesion layer is made of terpene resin.

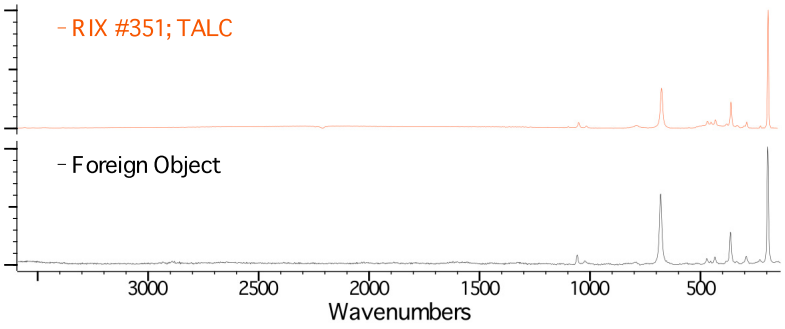

Fig. 5 Search result of the database

Summary

NRS-4100 can measure the buried sample in non-contact and non-destructive manner without lousy sample pretreatment. By using Infrared microscope together, the analysis of more complex foreign materials can be done.

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