Application Note

Measurement of embedded foreign material using an NRS-4100 Raman Spectrophotometer

Introduction

Currently, infrared microscopy is used extensively as one of the methods for identifying microscopic contamination or foreign materials in a sample matrix. Infrared spectroscopy has well established and large database of identified sample spectra and is ideal for the identification of unknown foreign materials. However, the drawbacks of infrared microscopy are that the measurement has a spatial resolution limited to only a few micrometers and extensive sample preparation can be required if the target material is buried inside the sample. Laser Raman spectroscopy is a technique being widely adopted for measurement of foreign materials in combination with infrared microscopy.

Keyword : Raman, Confocal Raman Analysis
Polymer analysis, NRS-4100, Terpene resin
Cellulose, Glass

NRS-4100

JASCO INC.
28600 Mary's Court, Easton, MD 21601 USA
Tel: (800) 333-5272, Fax: (410) 822-7526
Application Library: http://www.jascoinc.com/applications

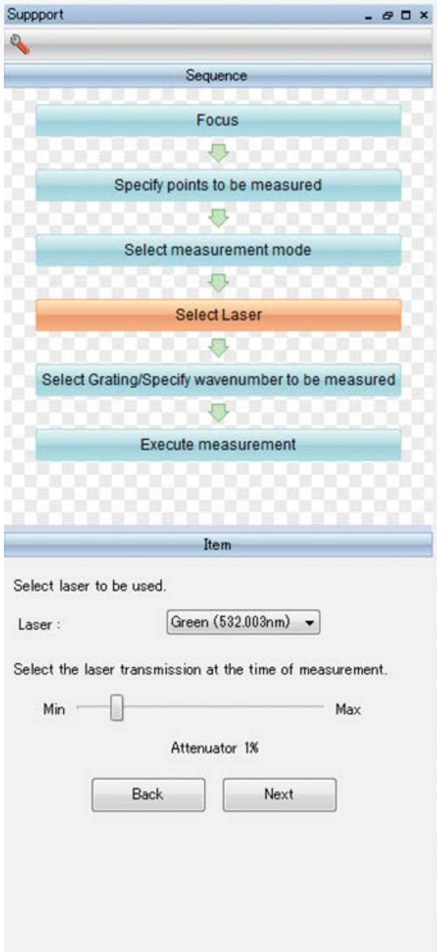
Raman spectroscopy is similar to IR microscopy in that it can identify molecular structures by analyzing molecular vibrations, but Raman spectroscopy offers several key advantages.

1. Excellent spatial resolution down to 1 μm using a visible laser.

2. Raman spectroscopy offers quick and easy non-destructive measurement without sample pretreatment.

3. Identification of inorganic samples is simplified because sample measurements can be obtained in a low wavenumber range (below 400 cm-1).

The potential for measuring foreign materials using Raman spectroscopy is expanding, and JASCO has developed a new, compact, easy to use laser Raman spectrometer. The JASCO NRS-4100 is designed to be used as a complementary technique to FTIR microscopy. In this application note, we will illustrate some of the key features of the NRS-4100 for the identification of foreign materials buried in a polymer film.



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Features of the NRS-4100

The NRS-4100, as shown in Fig. 1, is a Raman spectrometer, incorporating a high performance spectrograph, sample compartment, detector and laser light source in a very small optical bench just 60 cm square, which can be installed on a standard laboratory bench (no requirement for an anti-vibration table). The instrument requires no extra space as the sample compartment door moves up and down for open/close operations. In addition, the NRS-4100 meets the Class 1 laser safety standard.

Up to three lasers, including the standard configuration of 532/785 nm plus a compact 457 nm laser can be installed inside the instrument. The spatial resolution for the NRS-4100 is as small as only 1 μm in the XY and 1.5 μm in the Z direction, enabling high spatial resolution and fluorescence minimization, which are important for the measurement of foreign materials.

The “Measurement Assist” function aids the user in setting up the NRS-4100 for sample measurement; a simple sequence guide takes the user through setup and optimization of measurement parameters with helpful advice and tips, such as a warning if 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 and includes an array of Raman specific tools and analysis functions.

System configuration

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

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Foreign material measurement/Analysis

The foreign material embedded in the multi layer substrate (glass/adhesion layer/transparent film) shown in Fig. 3 was measured. It is difficult to measure such foreign material using an infrared microscope because it is difficult to cut the foreign material into a cross-section due to the presence of glass and the adhesion layer, which may result in the two materials being sampled together. On the other hand, a Raman instrument with a confocal optical system can selectively obtain a spectrum of the sample at the focused laser point. As a result, it is possible to measure the inside of the sample in a non-contact and non-destructive manner without difficult and time-consuming sample treatment. In this report, the position where the target foreign material is located was measured by depth profiling (Z axis direction) and the spectral information for each layer was obtained. The major spectra 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

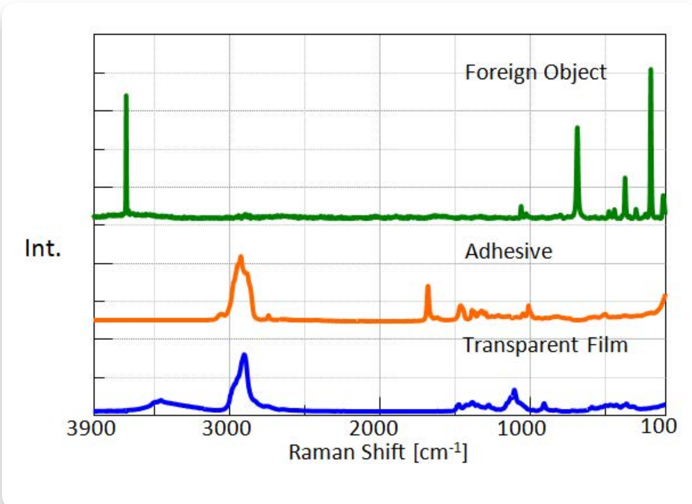

Fig. 4 Spectrum for each layer

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In the spectrum obtained of the foreign material, a C-H peak at around 3000 cm-1 is not shown and thus it is quite different from the spectra of the transparent film and adhesion layers. In order to analyze the result in further detail, the spectrum of the foreign material was searched and identified using a 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 a terpene resin.

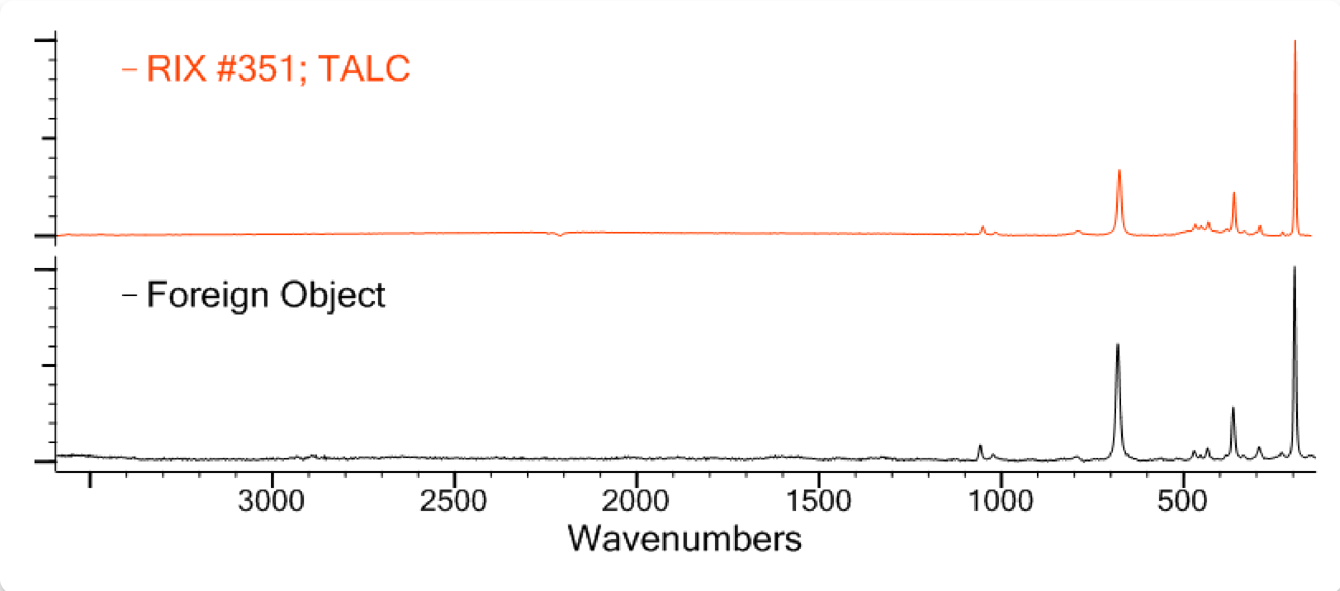

Fig. 5 Search result of the database

Summary

NRS-4100 can measure embedded samples in a non-contact and non-destructive manner without the requirement for extensive and difficult sample preparation. By using Raman in combination with an Infrared microscope, the analysis of more complex foreign materials can be easily accomplished.

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