SHIMADZU APPLICATION NEWS

LA103-E008

● SPECTROPHOTOMETRIC ANALYSIS

No.A344

Micro Contaminants Analysis with Infrared Microscope Micromanipulator System

Infrared microscopes are widely employed for the analysis of micro items or micro areas. Depending on sample conditions and analysis objectives, transmission or reflectance methods can be selected. In particular, the specular reflectance and attenuated total reflectance (ATR) methods, variations of the reflectance method, are capable of non-destructive analysis without sample pretreatment. However, with these methods, applicable samples are restricted because the measurement is affected by the surface conditions or thickness of the sample, as well as by the substrate.

In contrast, transmission methods can be applied to a wider range of samples and can achieve higher-sensitivity measurements on account of the small loss of the light intensity. However, in most cases, pretreatment procedures such as sampling the target object are required.

The Infrared Microscope Micromanipulator System introduced in this Application News allows all processes from sample observation to sampling and analysis to be conducted with a single microscope.

■ Infrared Microscope Micromanipulator System

Fig. 1 shows the infrared microscope system that combines the FTIR-8400S and the AIM-8800 infrared microscope equipped with the MMS-77 micromanipulator.

The MMS-77 micromanipulator conducts sample pretreatment by operating the metal probe attached to the end of the arm using a joystick. This allows control in the micro meter order, eliminating errors caused by manual operation. Therefore, even a sample as small as 10μm or less can be easily sampled, which was impossible with manually operated tweezers or needles. Also, the metal probe can be replaced with a microcutter (bio-cutter or sheet-cutter) or diamond tool, to cut out or dig out contaminants from film or sheet samples.

This micromanipulator can be attached to other commercially available microscopes. However, by combining the micromanipulator with the Shimadzu AIM-8800 infrared microscope, sample observation, sampling and measurements are all performed with a single microscope, eliminating the risk of contamination, or the possibility of losing the sample during transfer from the optical microscope to infrared microscope. In addition, the combination of micromanipulator's position memory function and the AIM-8800's auto-stage allows seamless operations from sampling to sample setting on the infrared transmittance cell.

Fig.1 FTIR-8400S, AIM-8800 with MMS-77

■ Analysis of Micro Contaminant on Quartz Oscillator

Fig. 2 to 4 show a contaminant found on a quartz oscillator that was picked up and transferred onto a diamond cell using the micromanipulator with a metal probe having a tip radius of 2.5µm (pictures taken with x10 objective lens). The diameter of the contaminant is about 25µm.

If the contaminant is on metal, or it has a flat surface and sufficient thickness, it can be directly analyzed by the specular reflection method. However, if the contaminant is on quartz, glass or plastic, the spectrum obtained will be affected by reflectance from the substrate, making analysis difficult. But in such case, the use of the micromanipulator enables micro contaminants to be picked out and transferred to a position convenient for analysis, as shown in Fig. 2 to 4.

Fig. 5 shows the transmission spectrum of the contaminant. This shows that the contaminant is a silicate containing a small amount of organic matter.

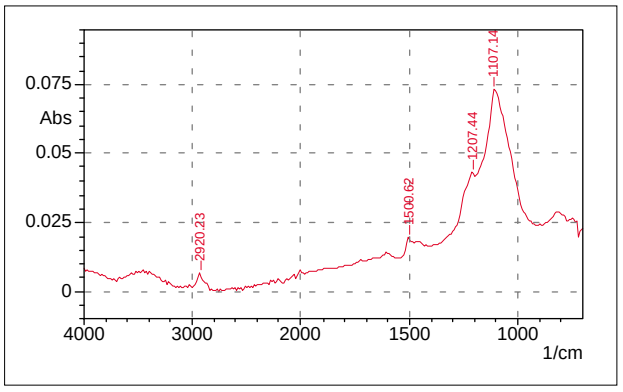

Fig.5 Infrared Spectrum of a Small Contaminant

■ Analysis of Micro Contaminant Near Electrical Contact

Fig. 6 and 7 show a contaminant found near a contact point on a circuit board, which was picked out using the micromanipulator fitted with a metal probe having a tip radius of 2.5µm (pictures taken with × 20 objective lens). The contaminant size is about 10 × 25 µm.

Fig. 8 shows the transmission spectrum obtained by analyzing the contaminant transferred onto a diamond cell. The result shows that the contaminant is rosin.

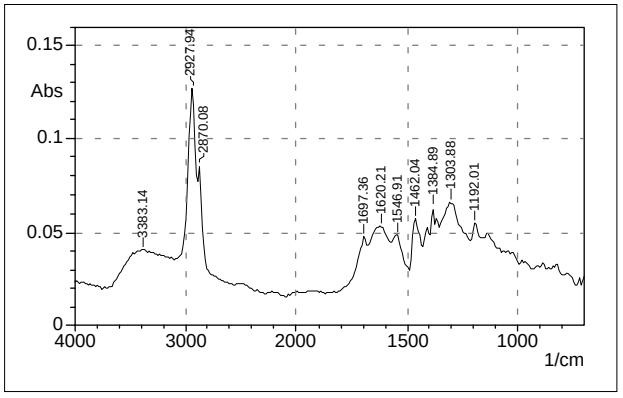

Fig.8 Infrared Spectrum of a Small Contaminant

Table 1 Analytical Conditions

Resolution	: 8cm^{-1}
Accumulation	: 100
Detector	: MCT

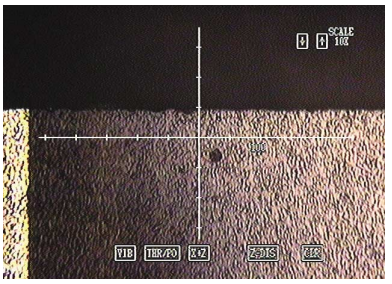

Fig.2 Micro Contaminant on Quartz Oscillator

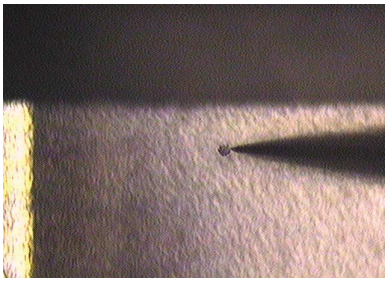

Fig.3 Picking out Micro Contaminant using Metal Probe

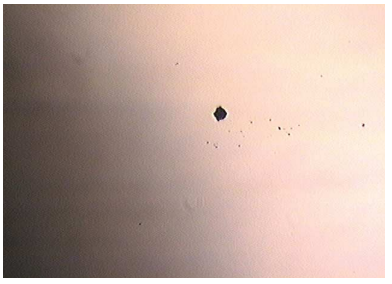

Fig.4 Setting on Diamond Cell

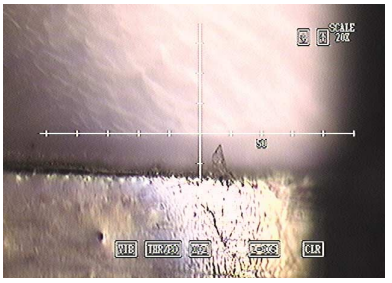

Fig.6 Micro Contaminant near Electrical Contact

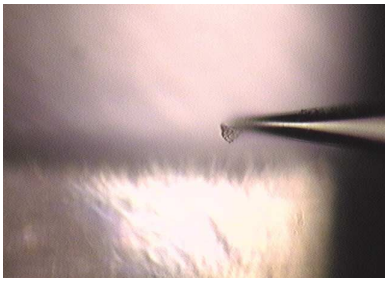

Fig.7 Picking out Micro Contaminant using Metal Probe

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