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Application News

No. A541

Spectrophotometric Analysis

Reflectance Measurement of Thick Samples Using an Infrared Microscope

The AIM-9000 automated micro analysis system can normally measure samples with a thickness of up to approximately 10 mm by placing the sample on the stage. However, by removing the lower cassegrain (condenser-objective), samples with a thickness of up to 40 mm can be placed for reflectance and ATR measurement.

This article introduces examples of reflectance measurement of samples with a thickness larger than 10 mm.

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Attaching/Removing the Lower Cassegrain

The lower cassegrain can be easily attached and removed as necessary by following a guide which is displayed by selecting [Replace Lower Cassegrain] on the AIMsolution software menu.

In measurement by transmission microspectroscopy, the lower cassegrain (indicated by an arrow in Fig. 1) is necessary to condense infrared light on the sample surface. Conventionally, attaching a lower cassegrain requires manual alignment and height adjustment. However, the AIM-9000 features an automatic lower cassegrain adjustment function which automates this work and achieves smooth attachment.

Fig. 1 Lower Cassegrain

Contaminant Analysis on a Thick Metal Component

We first measured a stain and fiber-like contaminants on a metal component. Since the sample was 15 mm thick, reflectance measurement was performed with the lower cassegrain removed. Fig. 2 shows the instrument with the lower cassegrain removed.

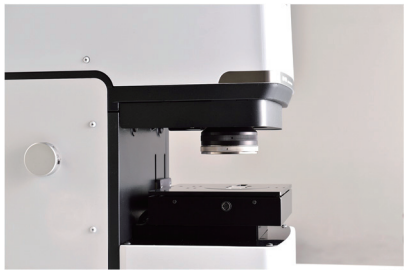


Fig. 2 With the Lower Cassegrain Removed

Upon attempting observation with the wide-field camera, observation proved difficult due to the camera light reflecting off the metal sample surface as indicated in Fig. 3. By adjusting the wide-field camera lighting as indicated in Fig. 4, the clear image shown in Fig. 5 was obtained. Fiber-like contaminants adhered on top of a circular stain can be observed.

The AIM-9000 allows adjustment of the wide-field camera lighting and brightness, as well as freedom in adjustment of the color balance, brightness, and contrast of the microscope camera. For details on the wide-field camera, refer to Application News No. A530.

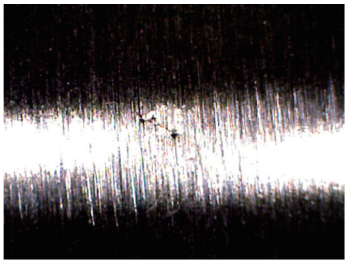
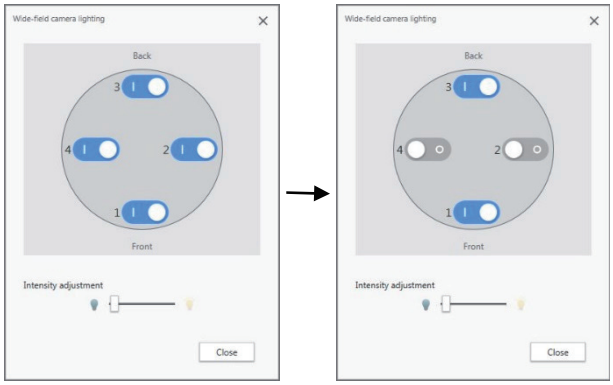


Fig. 3 Wide-Field Camera Image of the Contaminant (Before lighting adjustment)



(Before adjustment) (After adjustment)

Fig. 4 Adjustment of the Wide-Field Camera Lighting

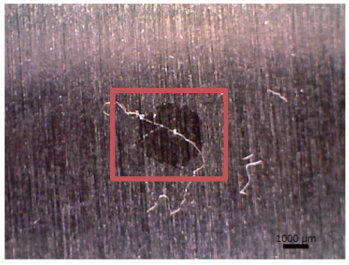
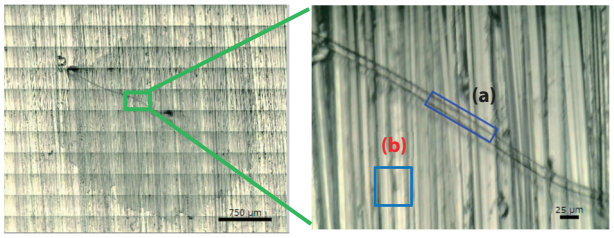


Fig. 5 Wide-Field Camera Image of the Contaminant (After lighting adjustment)

Next microscope camera images were acquired of the area indicated by a red frame in Fig. 5. The "Multi-image tiling" measurement support function was used to create a wide-field image by joining together microscope camera images. The multi-tiling image is shown as (A) in Fig. 6. Since measurement points can be set on multi-tiling images, it is possible to smoothly specify measurement points even when the object for measurement spans across a wide area.

Of the various sections we measured on Fig. 6 (A), the enlarged image of the region in the green frame is indicated as (B) of Fig. 6. The reflectance spectra of measurement sections (a) and (b), together with their search results are indicated in Fig. 7. Table 1 lists the instrument and analysis conditions. As for the aperture size, (a) was set to 100 × 20 µm, and (b) to 50 × 50 µm.

The infrared reflectance spectrum obtained from measurement section (a) indicates polyester and paraffin, while the one obtained from measurement section (b) indicates paraffin. We can therefore deduce that the contaminant analyzed is polyester fiber adhered to a paraffin oil stain.



(A) Multi-tiling Image (B) Enlarged View of Green Frame Section

Fig. 6 Microscope Camera Images of the Contaminant

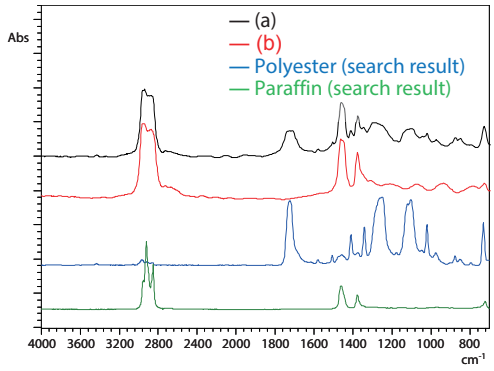


Fig. 7 Reflectance Spectra of Contaminant and Obtained Search Results

Table 1 Instrument and Analysis Conditions

Instrument	:	IRTracer-100, AIM-9000
Optical resolution	:	8 cm^{-1}
Accumulation times	:	40
Apodization function	:	Sqr Triangle
Detector	:	MCT

Contaminant Analysis on Non-flat Components

Samples that are spherical or have protrusions or depressions can be measured by holding the sample with a micro vise holder so as to keep the sample surface level. (Samples up to approx. 40 mm wide and 40 mm long can be held by adjusting the screw on each end of the holder.)

This time we performed reflectance measurement on an organic substance adhered to the metal prongs of a power plug. Fig. 8 shows the power plug held by the micro vise holder. Measurement was performed with the lower cassegrain removed since the power plug extends approx. 30 mm from the top surface of the micro vise holder.

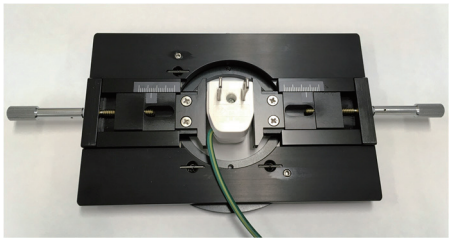


Fig. 8 Power Plug Held by the Micro Vise Holder

The microscope camera image is shown in Fig. 9. The reflectance spectra together with their search results are indicated in Fig. 10. The aperture size was set to 50 × 50 µm.

The organic substance is assumed to be a mixture of paraffin and lanolin.

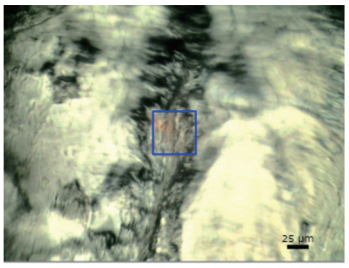


Fig. 9 Microscope Camera Image

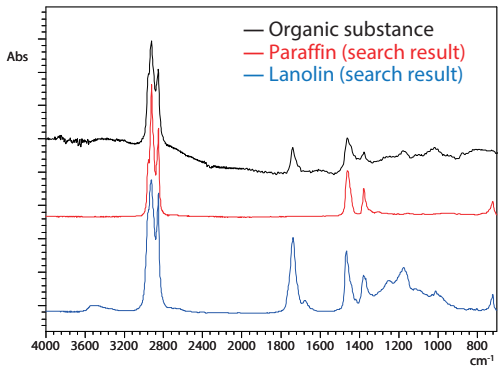


Fig. 10 Microscopic Reflectance Spectra and Obtained Search Results

Conclusion

We analyzed thick samples using the AIM-9000 automated micro analysis system. If the sample surface for analysis is exposed, the sample can be analyzed without preparations, such as cutting the sample, by removing the lower cassegrain.

References

1) Application News No. A530 "Observing and Measuring Contaminants - Advantages of the Wide-Field Camera -"

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