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Application News

Fourier Transform Infrared Spectrophotometer IRTracer™-100
Infrared Microscope AIMsight™
Infrared Raman Microscope AIRsight™

Mapping Measurement of Paints and Pigments with Thermoelectrically Cooled MCT Detector

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User Benefits

- A thermoelectrically cooled MCT detector and mapping software (optional product) can be used for mapping measurement of microscopic samples without the need for liquid nitrogen.
- Mapping measurement can create composite chemical images showing the distribution of paint and pigment constituents in a sample.

■ Introduction

Automobiles are coated with multiple layers of paint, each with a specific purpose and composition. So paint fragments at the scene of a traffic accident can provide critical information for forensic investigations. Infrared microscopy can analyze fragments that retain this multi-layer structure to identify the component constituents in each paint layer, and this can be used to identify the specific model and year of manufacture of an automobile involved in an accident.

Infrared microscopy is also used in scientific studies of valuable historical works of art to identify the pigment components and their distribution. This can reveal what techniques were used to create them, and it can assist in selecting the techniques and materials used to restore and conserve them.

The thermoelectrically cooled MCT (TEC MCT) detector is a new optional detector for infrared microscopes that can measure microscopic targets as small as 25 µm with high resolution and without requiring liquid nitrogen. But while AIMsight infrared microscope and the AIRsight infrared Raman microscope are equipped as standard with a T2SL detector, which can measure microscopic targets down to 10 µm, they require liquid nitrogen to do so. Due to the difficulty of procuring it and the dangers it can pose of frostbite and oxygen displacement, there is an increasing demand for analytical options that avoid using liquid nitrogen.

In this article, an infrared microscopy system (Fig. 1) equipped with a TEC MCT detector was used for a mapping measurement of an automobile coating and organic pigments.

Fig. 1 IRTracer™-100 and AIMsight™

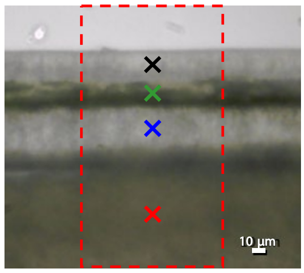
■ Automobile Paint Measurement

An automobile bumper was used as a sample. A 2-cm square section of the bumper was prepared with an electric saw, and a microtome (HistoCore AUTOCUT R, Leica Biosystems) was used to prepare 5-µm thick sections of the bumper. A holding jig was used to hold a section on an infrared microscope stage with the cut surface facing upwards, transmission methods was used to analyze the cross section of the automobile coating. The measurement conditions used are shown in Table 1.

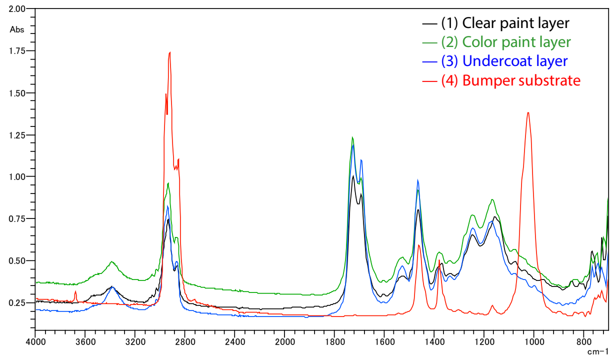
Table 1 Measurement Conditions

Instruments:	IRTracer-100, AIMsight
Resolution:	8 cm^{-1}
Number of scans:	100
Apodization function:	SqrTriangle
Aperture size:	25 × 100 µm
Step size:	3 µm
Mapping area:	186 × 100 µm
Detector:	TEC MCT

Fig. 2 shows a microscope image of the area that was mapped and four spectra recorded from different locations in the area. The microscopic image shows three layers of paint on the bumper substrate, where the uppermost layer (1) appears to be clear paint layer, the middle layer (2) appears to be color paint layer, and the bottom layer (3) appears to be an undercoat layer.



(a) Microscope Image of Automotive Coating



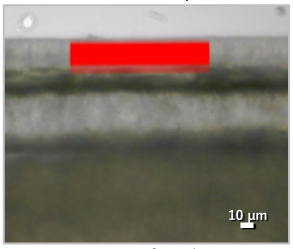
(b) Infrared Spectrum of Each Paint Layer and Bumper Substrate

Fig. 2 Microscopic Image (a) and Infrared Spectra (b) of Section of Automotive Bumper

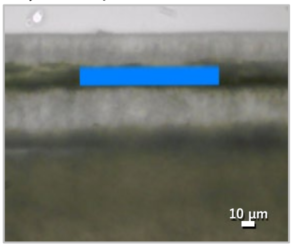
The three paint layers all gave a similar spectral profile (Fig. 2 (b) (1), (2), and (3)) and a spectral library search matched all three as acrylic polymer. N-H and C-O peaks characteristic of polyurethane and (weak) C≡N and C=C-H peaks characteristic of acrylonitrile butadiene styrene (ABS) were also detected in the three layers, suggesting that all the layers used an acrylic-based paint mixed with polyurethane and ABS. A spectral library search for the bumper substrate spectrum (4) produced a match for polypropylene (PP) and hydrated magnesium silicate (talc). This suggests to show the bumper substrate was made of PP with an additive to improve impact resistance and rigidity.

Fig. 3 shows chemical images prepared by mapping measurement overlaid on microscopic images of the sample section. Mapping measurement shows the distribution of constituent materials in a sample otherwise not visible in a microscopic image. Using spectra recorded during mapping measurement, colors are added in areas of the microscopic image based on selected parameters, such as peak height, peak area, data from multivariate analysis (PCR/MCR), and the degree of similarity with reference spectra. In this example, four composite chemical images were created showing the degree of similarity between the mapped area and the infrared spectrum of the three paint layers and the bumper substrate. In Fig. 3, the areas where the coloring is more opaque represent areas of greater similarity (Fig. 3).

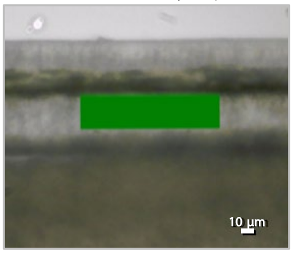
At a glance, there appears to be very little difference between the spectral profiles of the three paint layers (Fig. 2(b)), but mapping measurement based on the degree of spectral similarity produced composite chemical images showing clearly defined color paint layer that were 20 µm thick (Fig. 3). This shows that mapping measurement and the resulting composite images can provide a visual representation of the ratios and types of polymer in each layer of paint. Accordingly, infrared microscopy can be used to visualize and identify the type of paint used in each layer of a multi-layer sample.



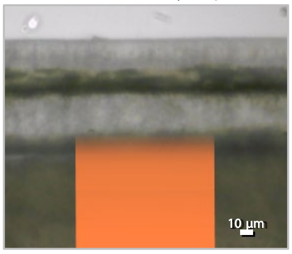
(a) Degree of Similarity
with the Clear Paint Layer Spectrum



(b) Degree of Similarity
with the Color Paint Layer Spectrum



(c) Degree of Similarity
with the Undercoat Layer Spectrum



(d) Degree of Similarity
with the Bumper Substrate Spectrum

Fig. 3 Composite Chemical Images of a Section of an Automobile Bumper

■ Organic Pigment Measurement

Yellow organic pigment A and yellow organic pigment B were prepared, dissolved in ethanol, mixed, and used as a sample. The mixture was spread on an aluminum mirror and ethanol dried before reflection measurements were recorded. The measurement conditions used are shown in Table 2.

Table 2 Measurement Conditions

Instruments:	IRTracer-100, AIMsight
Resolution:	8 cm^{-1}
Number of scans:	50
Apodization function:	SqrTriangle
Aperture size:	50 × 50 µm
Step size:	50 µm
Mapping area:	550 × 1,100 µm
Detector:	TEC MCT

Fig. 4 shows the pigment A spectrum and pigment B spectrum overlaid.

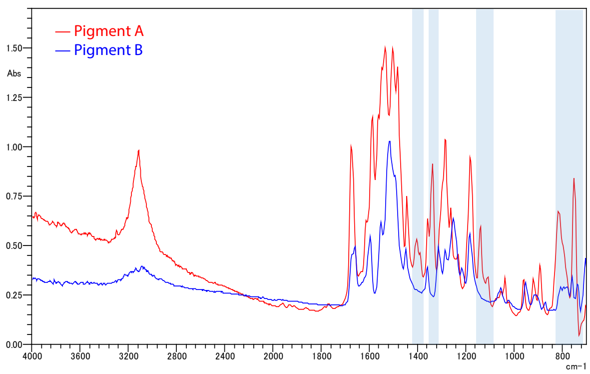
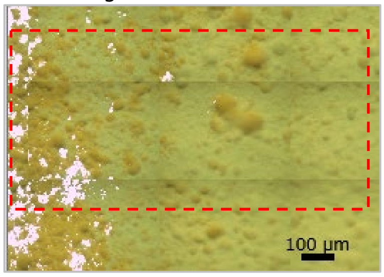
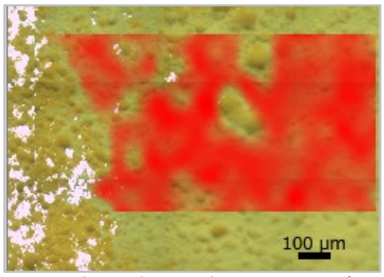


Fig. 4 Pigment A and Pigment B Infrared Spectra

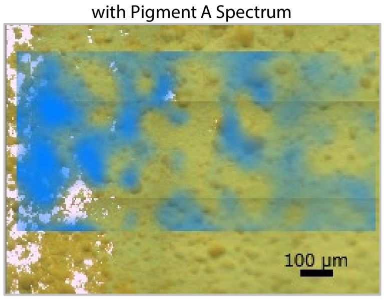
Fig. 4 shows that even when pigments were A and B mixed, the wavenumber regions shaded in blue can be used to distinguish them. Similar to the above chemical mapping of an automobile coating, composite chemical images were prepared of the mapping area (Table 2) based on the degree of similarity with the spectra of pigment A or B (Fig. 4). These composite chemical images are shown in Fig. 5.



(a) Area of Chemical Mapping (Area Inside Dashed Red Line)



(b) Composite Chemical Image Showing Degree of Similarity
with Pigment A Spectrum



(c) Composite Chemical Image Showing Degree of Similarity
with Pigment B Spectrum

Fig. 5 Distribution of Pigment A and Pigment B (Composite Chemical Images)

The composite chemical images in Fig. 5 show a greater distribution of pigment A on the right side of the mapping area and a greater distribution of pigment B on the left sides Some pigment B is also interspersed in the area with a large amount of pigment A.

Although slight differences in yellow coloration are visible at the microscopic image in Fig. 5(a), mapping measurement by infrared microscopy revealed detailed information about the distribution of constituent components not present in the microscopic image.

■ Conclusion

An infrared microscopy system equipped with a TEC MCT detector was used to perform mapping measurement of an automobile coating and organic pigments. The TEC MCT detector could perform mapping measurement over a wide target area with good sensitivity and without the need for liquid nitrogen. But for mapping measurement that requires a smaller aperture or if it needs to be quicker and use fewer scans, the T2SL detector, which requires liquid nitrogen is recommended.

<Related Application News>

1. Sensitivity Evaluation and Example Analysis of Microscopic Targets with Thermoelectrically Cooled MCT Detector: Application News No. 01-00826

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