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Application News

Infrared/Raman Microscope AIRsight™

Analysis of Stain-like Deposits on the Surface of Metal Machined Parts for the Transportation Industry Using the AIRsight Infrared/Raman Microscope

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User Benefits

- AIRsight makes it easy to quickly switch between infrared and Raman measurements without moving the target.
- Materials up to 40 mm thick can be measured directly and non-destructively.
- Raman spectroscopy is ideal for measuring micron-scale areas and rough surfaces.

■ Introduction

When manufacturing metal machined parts, anti-rust oil or cleaning fluid from machine tools may remain on metal parts, potentially leading to stain-like deposits. These stains can cause issues, such as reacting with lubricating oil or causing poor plating adhesion.

This Application News presents a case study of measuring stain-like deposits found on the surface of metal machined parts for the transportation industry using the infrared-Raman microscope AIRsight.

■ Analysis of Stain-like Deposits

The choice of analytical instrument for contaminant analysis varies depending on the size and thickness of the deposits. Additionally, accuracy and speed are also required.

If the stain-like deposits on the surface of metal machined parts are inorganic, then instruments like an Electron Probe Micro Analyzer (EPMA) are effective. For organic deposits, infrared microscopes or Raman microscopes are effective. The infrared spectroscopy method using an infrared microscope can measure samples without damaging them since they are irradiated with infrared light. For very thin stain-like deposits, the highly sensitive reflective method using a Grazing Angle Objective (GAO) can be useful. Besides, Raman spectroscopy using a Raman microscope allows the measurement of tiny regions on the micron scale by using a visible light laser as the light source.

When using AIRsight, incorporating the Raman unit inside the infrared microscope makes it possible to perform both infrared and Raman measurements with a single instrument. This microscope is extremely useful for contaminant analysis as it allows rapid switching and acquisition of both infrared and Raman spectra without moving the sample.

■ Sample

In this case study, the brown stain-like deposits on the surface of a circular metal machined workpiece for the transportation industry ("circular workpiece") were measured. A photograph of the stain-like deposits is shown in Fig. 1.

Fig. 1 Brown Stain Adhered on the Surface of the Circular Workpiece

By removing the lower condenser mirror, AIRsight can perform reflection or ATR measurements on samples up to 40 mm thick (Fig. 2). The circular workpiece used in this analysis has a thickness of about 35 mm and can be analyzed by placing it directly on the stage without destruction.

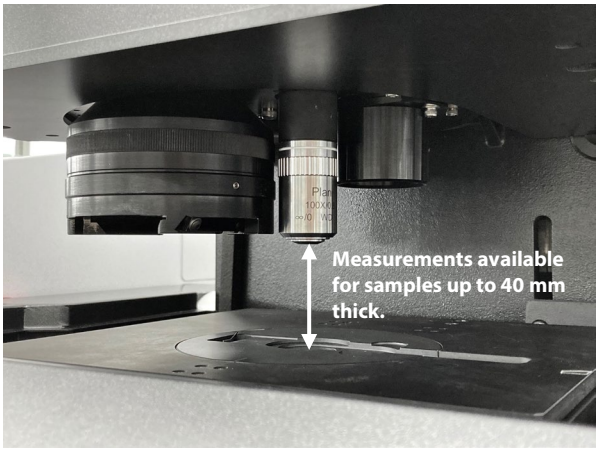

Fig. 2 Sample Stage of AIRsight

■ Sample Surface Condition Observation

Fig. 3 shows an image of the brown stains using the wide-field camera. The wide-field camera allows observation with a field of view up to 10×13 mm and has a 5x zoom function. This makes it easy to identify relatively thickly adhered areas suitable for measurement by confirming the distribution of the stains over a wide range. Additionally, since the reflective objective for infrared measurement and the objective lens for Raman measurement share positional information with the wide-field camera, it is possible to smoothly switch to the infrared objective or Raman lens and set the measurement area without any shift in the field of view.

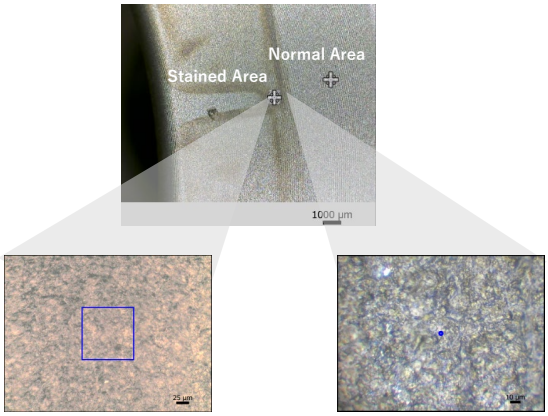

Fig. 3 Observation Images of Brown Stains by the Wide-Field Camera (Top), Reflective Objective for Infrared Measurement (Bottom Left), and Objective Lens for Raman Measurement (Bottom Right)

■ Analytical Conditions

Infrared and Raman spectroscopic measurements were performed at the same location for both the stained and normal areas. The analytical conditions for each are shown in Table 1.

Table 1 Analytical Conditions

Instrument:	IRXross™, AIRsight
Infrared Spectroscopy	
Resolution:	8 cm^{-1}
Number of Scan:	45
Apodization Function:	SqrTriangle
Aperture Size:	100 µm × 100 µm
Detector:	T2SL
Raman Spectroscopy	
Number of Scan:	45
Exposure Time:	10 sec
Objective Lens:	50x
Excitation Wavelength:	532 nm
Detector:	CCD

■ Infrared Spectroscopy Measurement

Infrared spectroscopy measurements were conducted for both stained and normal areas. The measurements were performed using two types of objective lenses: a 15x reflective objective and a GAO. The measurement results are shown in Figs. 4 and 5.

In the measurements taken with the 15x reflective objective, small peaks characteristic of CH_2 from organic substances were observed around 2,920 and 2,850 cm^{-1} (Fig. 4). The GAO measurements yielded similar results (Fig. 5). However, in both cases, the peak intensities were very weak, and peaks other than those from CH_2 were not detected. Thus, the cause of the stains failed to be identified. This is likely due to the surface roughness of the sample, which reduces the amount of reflected light reaching the detector.

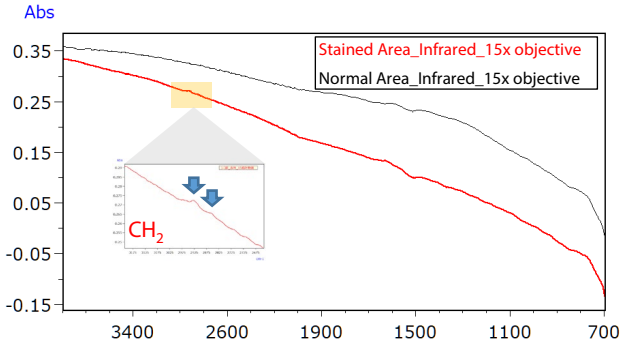

Fig. 4 Infrared Spectra of the Stained and Normal Areas Measured Using a 15x Reflective Objective

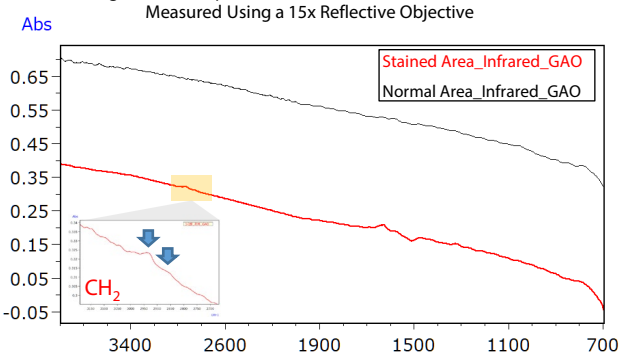

Fig. 5 Infrared Spectra of the Stained and Normal Areas Measured Using GAO

■ Raman Spectroscopy Measurement

Raman spectroscopy measurements were performed on both the stained and normal areas. The results are shown in Fig. 6. Clear peaks specific to the stained area can be observed around 2,900 cm^{-1} and 1,450 cm^{-1}. The Raman spectroscopy measurements with a 50x objective lens indicate its effectiveness for samples with irregular surfaces, where peak detection is difficult with infrared spectroscopy due to the measurement region being 5 µm or less.

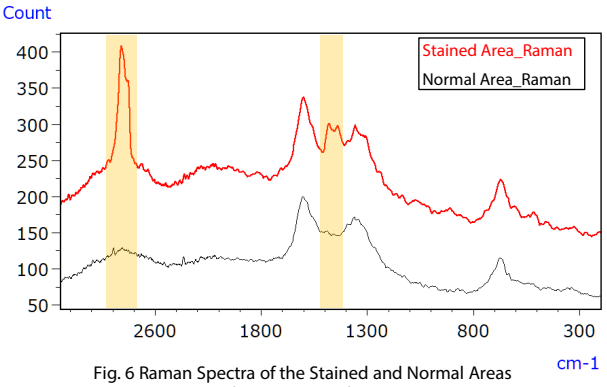

Fig. 6 Raman Spectra of the Stained and Normal Areas Measured Using a 50x Objective Lens

Next, to identify the cause of the stain, a search was conducted using the Raman spectra obtained from the stained area with the Wiley Raman spectra database. The search was limited to the wavenumber regions highlighted in Fig. 6, and as shown in Fig. 7, ethyl decanoate was identified. Since ethyl decanoate, found in the search, is not a component of the anti-rust oil or cleaning fluid used for the circular workpiece, it is assumed to be a residual from the reaction between the two components or something that adhered from the outside.

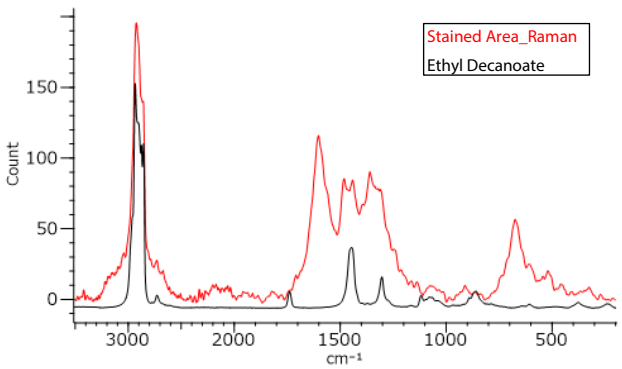

Fig. 7 Search Results of the Raman Spectrum of the Stained Area

■ Conclusion

The analysis of stain-like deposits discovered on the surface of metal machined parts for the transportation industry using AIRsight was introduced. Raman spectroscopy allowed the qualitative identification of the stain components, which was difficult with infrared spectroscopy due to the surface's unevenness. AIRsight is very useful for the qualitative analysis of unknown samples by allowing rapid switching and measurement of both infrared and Raman spectroscopy without moving the sample. Additionally, it can measure thick samples like metal machined parts non-destructively.

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