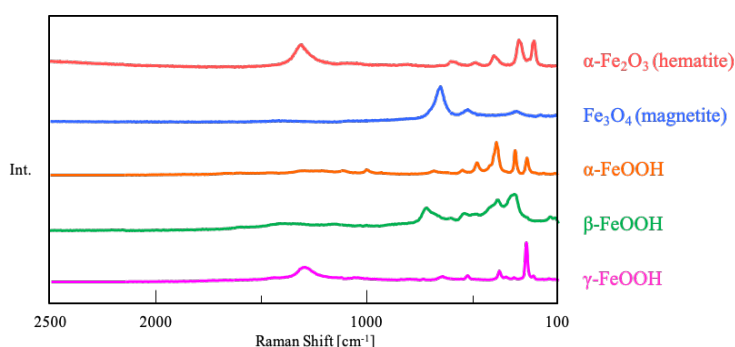


## Raman measurement of surface iron oxidation states

### Introduction

Rust that occurs when iron materials corrode has various oxidation states and crystal structures, such as  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ , and  $\text{FeOOH}$ , resulting in differences in Raman spectra (Figure 1). Since the type of rust that occurs depends on the exposure environment, analysis of the oxidation state of the rust can lead to elucidation of the causes of corrosion, and the necessary steps to prevent it. In this note, we introduce the results of analysis of oxidation states on the surface of iron materials using a micro Raman spectrometer.



**Figure 1.** Raman spectra of iron oxides and hydroxides

### Keywords

Raman spectroscopy, Surface Scan Imaging (SSI), Iron oxide, corrosion

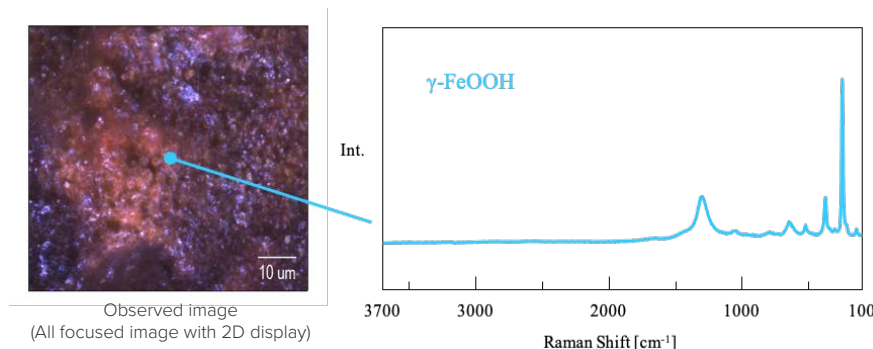
### Experimental

**Samples:** Two samples\* taken from different environments (indoor and outdoor) were analyzed.

\*Details of the environment in which the samplers were previously exposed, both indoors and outdoors at the time of sampling are unknown.

**Measurement procedure and results:****1. Point measurements for each rust sample**

Multiple spectra from different oxidation states such as  $\alpha\text{-Fe}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$  were obtained from samples collected indoors, with  $\gamma\text{-FeOOH}$  being mostly detected from samples collected from outdoor samples (Figure 2).



**Figure 2.** Results of samples collected outdoors

**2. Imaging measurement of samples collected indoors (evaluation of component distribution)**

Three different spectra were obtained. Here, the typical spectrum of each component was used as the reference spectrum (the spectra are shown in Figure 4), and a chemical image was created using the correlation (similarity) with the reference spectrum. Using a false-color map to create a chemical image, it is possible to visualize the component distribution that may be difficult to determine visually, which is thought useful for analyzing the cause of corrosion.

**Parameters for imaging and point measurement**

Since the surface of rusted metal has a lot of irregularities, Surface Scan Imaging (SSI) was used for imaging measurement along the irregularities. (Reference: Application Note 260-AN-0017). SSI has the ability to quickly image and map rough or uneven samples.

Main Unit: NRS-5500 Raman Spectrometer			
Ex wavelength	532 nm	Data interval	2 $\mu\text{m}$
Exposure time	15 seconds	Accumulation	1 time
Objective lens	100x, long work distance for dark field observation		



**Figure 3.** NRS-5500 Raman Spectrometer

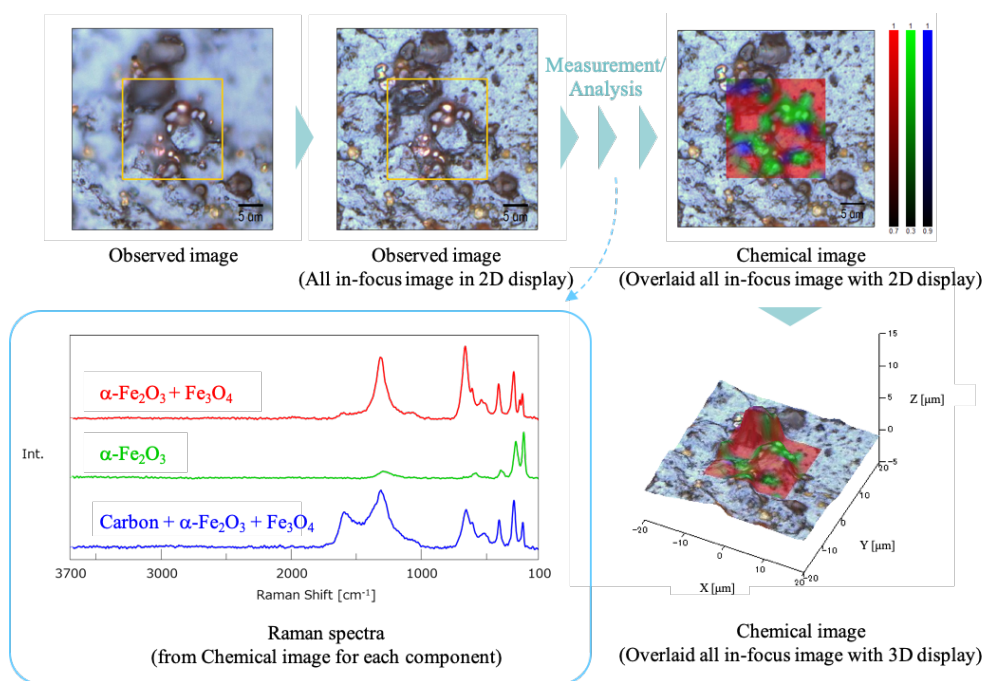


Figure 4. Measurement results of sample collected indoors

## Results

Using Raman spectroscopy it is possible to analyze the oxidation state and crystal structure of inorganic substances, such as rust. In addition, SSI is a very effective tool for imaging measurement of samples with an uneven surface such as rust.

System Configuration				
	Model	Description	P/N	Remarks
Main Unit	NRS-5500	Raman Spectrometer	7119-J051A	532 nm laser 100 mW laser was used, but a standard 50 mW is also available.
Options	RXY-57	Automatic XYZ stage	6947-J478B	
	SSI-457	Surface Scan Imaging	7118-J378A	
	RHG5-600B500	600 gr/mm grating	6947-J293A	
	RMIX57	MIX observation unit	7119-J255A	For dark observation and MIX observation <sup>*1</sup>
	LMPLFLN100xBD	Objective lens for dark field observation, x100, long work distance, for Vis range	1052-0196	Choose the lens according to the sample.

<sup>\*1</sup> MIX observation is useful for colored samples.

Note: this is a factory option, field upgrade is not possible.

An objective lens for dark observation is also required.

<About the measurement of Figure 4>

- Measurement time, approx.30 minutes.To avoid changing the rust condition, weaken the laser intensity and increase the exposure time, it takes a little longer for wide-range imaging.

- If the surface roughness is not significant, the standard imaging function can be used instead of SSI.

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