Application Note

050AT0246

Deterioration analysis of industrial grease by using ATR method

Introduction

Lubricants, such as oil and grease, are used for reducing the friction between moving parts such as gears in mechanical systems. Over time, the efficiency of the lubricant degrades due to oxidation by air and heat, and contamination metal fragments from the wearing of the metal. Inspection and maintenance are periodically required to examine the quality of the lubricant. By monitoring the quality of the grease or oil, the user can predict when the lubricant should be replaced. This can keep the machine in good working condition thus minimizing down time and a loss of revenue.

FTIR is a proven method, as demonstrated in ASTM methods using a transmission cell, for the evaluation of lubricants used in industrial environment. However, this method has some disadvantages including: (1) proper sample volume is necessary, (2) difficulty in measuring a highly viscous sample such as grease.

In order to address these issues, JASCO can offer the evaluation method using the ATR technique. ATR is a sampling technique for measuring the sample surface in contact with the high refractive index prism. Therefore, this technique can offer the micro-volume measurement without sample preparation or pretreatment. In addition, the penetration depth of surface measurement depends on two measurement conditions: (1) incident angle of infrared light, (2) refractive indices of the prism and sample (See FT/IR Application Note: 280-AT-0003). This ATR technique can give both qualitative and quantitative information.

This application note shows the ATR measurement result of various grease samples (before and after use), and the evaluation result of moisture, oxidation and oil-separation. As a sample, the industrial grease containing the urea-based thickener was measured.

Measurement

Instruments:	FT/IR-4600
Resolution:	4cm^{-1}
Method:	ATR
Detector:	DLATGS
Accumulation:	16 times (Approx. 20 sec)
Accessory:	ATR PRO ONE (Fig. 1)
Prism:	Diamond

Fig.1 ATR PRO ONE

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Result

<Moisture evaluation>

The broad absorption peak attributed to O-H stretching vibration (3500 - 3000 cm^{-1}) indicates water is included in the lubricant. Figure 2 shows the spectra of O-H region. As shown in the figure, there is the difference between the spectra of the grease before and after use. This result indicates that the water penetrates into the lubricant during movement of the mechanical parts.

One can also evaluate the tendency of moisture absorption in the grease sample quantitatively. This requires the creation of a calibration curve using known standard concentrations. Jasco's Spectra Manager II software allows for both classical least squares calibrations as well as a variety of chemometric techniques.

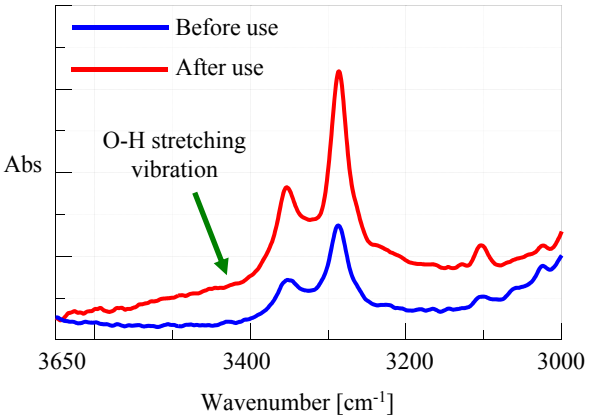

Figure 2 Spectra of grease (O-H region)
(Blue: before use, Red: after use)

<Oxidation evaluation>

The Grease generally consists of a base oil, thickener, and an additive. If the base oil oxidizes, the carbonyl and aldehyde are generated. An increase and/or decrease of the absorption peak attributed to C=O stretching vibration (near 1720 cm^{-1}) can be interpreted oxidation of the base oil. Figure 3 shows the resultant spectra of C=O region. As demonstrated in the figure, there is the distinct difference between the spectra of grease before and after use.

In addition, plotting the peak height or area enables one to evaluate the oxidation quantitatively.

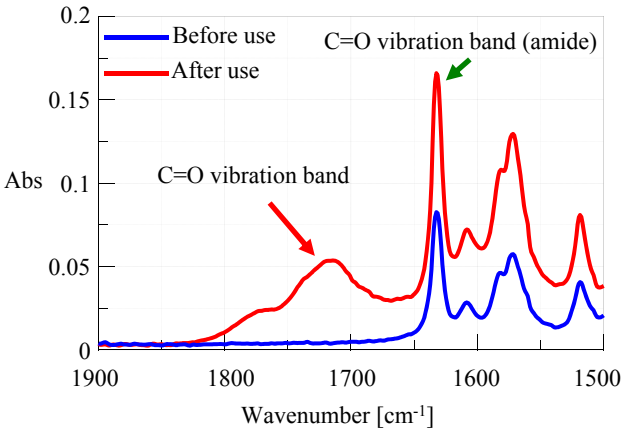

Figure 3 Spectra of grease (C=O region)
(Blue: before use, Red: after use)

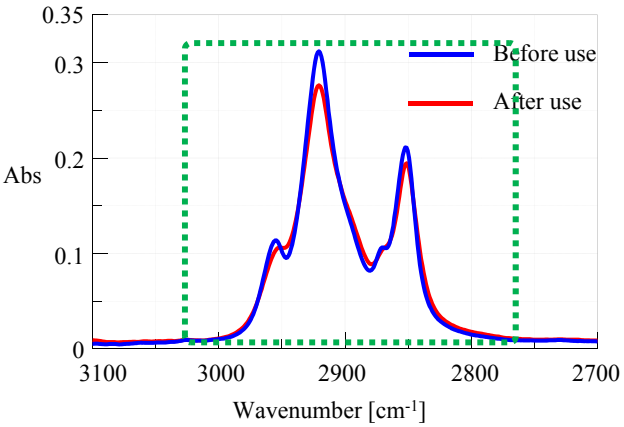

Figure 4 Spectra of grease (C-H region)
(Blue: before use, Red: after use)

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<Oil-separation evaluation>

With the movement of mechanical parts, the base oil is separated from the lubricating grease. This decreases the performance of the lubricant. One can use FTIR spectroscopy to evaluate this separation using the following formula:

$$Area\ ratio = \frac{\text{Amide C = O peak (1630 cm}^{-1}\text{) of the urea - based thickener}}{\text{C - H peak (around 2900 cm}^{-1}\text{) of base oil and thickener}}$$

The calculation result of the grease before use was 28.9, and the result after use was 16.3. This result shows that the base oil was separated from the lubricating grease significantly.

In order to assist with the above evaluation methods, JASCO can provide FTIR measurement applications within Spectra Manager II to ease in the implementation of these methods and providing a turn-key solution to the analyst.

Conclusion

This report demonstrates the ATR method can evaluate deterioration of lubricants. The ATR method can perform the small-volume sample measurements both rapidly and simply for the monitoring of lubricant degradation including:

(1) Monitor the decrease of the additive (such as oxidation inhibitor) concentration by calculating subtraction spectra between the spectra of grease before use and the one after use.

(2) Evaluate the foreign material by metal build up in the lubricant by micro-reflection measurement.

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