SHIMADZU
Excellence in Science

Application News

Fourier Transform Infrared Spectrophotometer

Measurement of Grease Using FTIR

Kazuki Sobue and Yasushi Suzuki

User Benefits

- The attenuated total reflection (ATR) method is a convenient and suitable method for qualitative analysis of base oils in grease.
- In the case of grease with strong light absorption, ATR measurement techniques such as applying a thin layer of grease or changing the Ge prism are used. Advanced ATR correction can be used as an analysis technique to obtain spectra similar to that of the transmission method.

■ Introduction

Grease is used on various machine parts (mainly shafts and bearings) to help smooth their operation. Grease is defined as a base oil (lubricating oil) with thickeners dispersed to achieve a semi-solid or solid state. There are various types of base oils and thickeners, which are used according to the given environment and application.

Fourier transform infrared spectrophotometers (FTIR) are used to check and identify base oils contained in grease and to investigate their degree of degradation. In particular, measurements using the ATR unit can be performed simply by applying grease to a prism. However, some base oils of grease have strong light absorption, which requires using special techniques for measuring or analyzing data. In this article, the FTIR single-reflection ATR method (ATR method) is compared to the transmission method for measuring commercial greases.

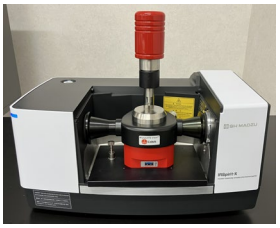

Fig. 1 IRSpirit™-TX + Microm ATR

■ Measurement of Grease

Three types of grease with different base oils were prepared and measured by the ATR method and the transmission method using a liquid cell. Fig. 2 shows the sample placement. With the ATR method, semi-solid (viscous) materials such as grease can be measured by simply spreading the sample onto the prism area without using the attached clamp to press it against the prism. With the ATR method, the penetration depth of light entering the sample is determined by the prism material, the angle of incidence, and the refractive index of the sample. For example, if the prism material is diamond (Dia), the angle of incidence is 45 degrees, and the refractive index of the sample is 1.5, then light penetrates 5 µm at 400 cm^{-1} and 0.5 µm at 4,000 cm^{-1}. On the other hand, with the transmission method, the thickness of the sample directly affects absorption. Since grease has low volatility and high viscosity, the samples were measured by applying a thin layer to a single window plate. Fig. 3 to 6 show the measurement results under the conditions indicated in Table 1.

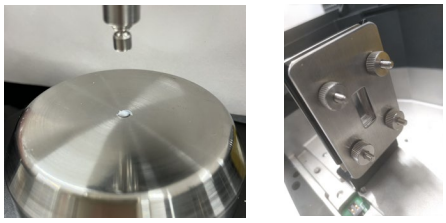

Fig. 2 Left: Sample Applied to Prism, Right: Liquid Cell

Table 1 Measurement Conditions

Instruments:	IRSpirit-TX, Microm ATR (Diamond) Liquid cell (KBr)
Wavenumber range:	4,000 to 400 cm^{-1}
Resolution:	4 cm^{-1}
Accumulation:	40
Detector:	DLTGS

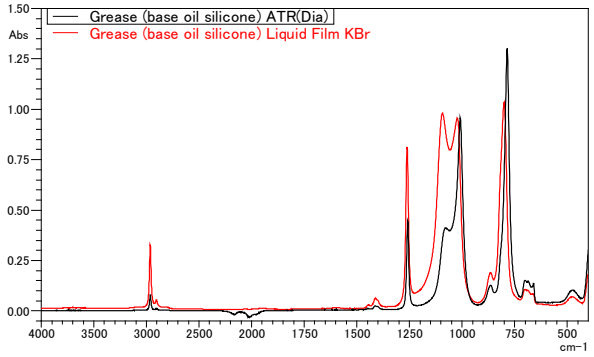

Fig. 3 Infrared Spectra of Commercial Grease (Base Oil : Silicone)

Fig. 3 shows the measurement results of grease with a silicone base oil. In the case of the ATR method, multiple peak positions at 1,300 to 700 cm^{-1} shifted toward lower wavenumbers. In addition, the peak intensity ratio at 1,200 to 1,000 cm^{-1} differed between the ATR method and the transmission method. Such peak shifts and intensity ratio changes occur with the ATR method for samples with strong absorption, due to the difference in refractive index between the prism and the sample. This suggests that silicone-based samples have strong absorption. The difference in the peak intensity ratio between the ATR method and the transmission method across the entire measurement wavenumber range is due to the penetration depth of the light with the ATR method.

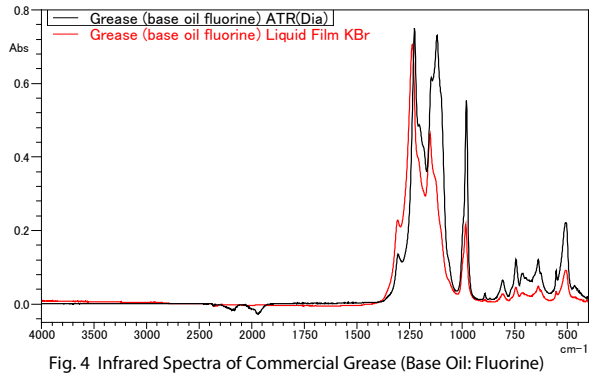

Fig. 4 Infrared Spectra of Commercial Grease (Base Oil: Fluorine)

Fig. 4 shows the measurement results of grease with a fluorine base oil. As with the silicone grease, it shows peak shifts and intensity ratio changes with the ATR method. That suggests that fluorine grease also has strong absorption. Fig. 5 shows transmission spectrum search results obtained using KnowItAll software from John Wiley & Sons, Inc.

Fluorine-based materials, such as PTFE (Fig. 5 bottom red line) and PFP (perfluoropolyether, Fig. 5 bottom green line) were identified in the sample, where they might be mixed.

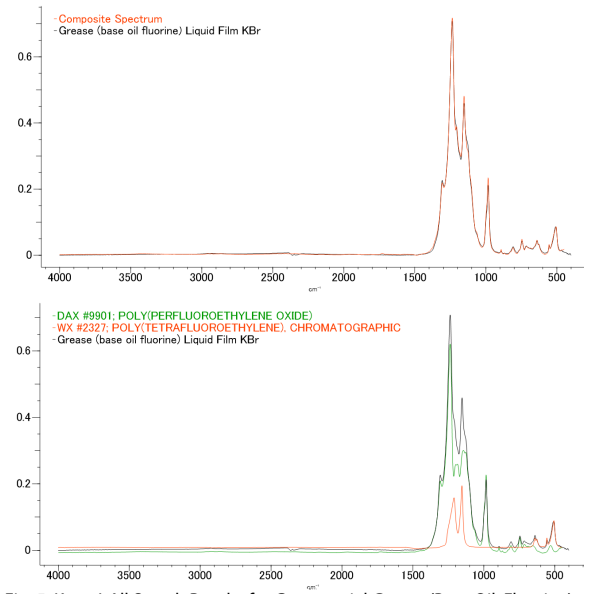

Fig. 5 KnowItAll Search Results for Commercial Grease (Base Oil: Fluorine)
Top: Comparison with Composite Spectrum,
Bottom: Comparison with Individual Spectra

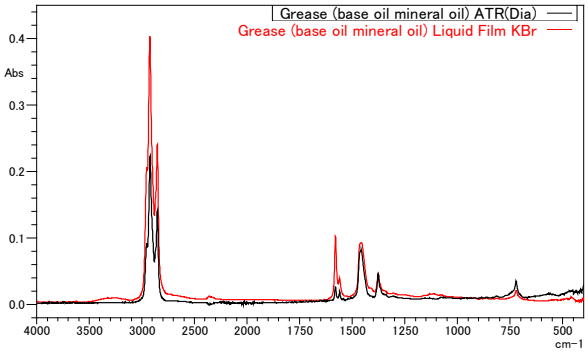

Fig. 6 Infrared Spectra of Commercial Grease (Base Oil: Mineral Oil)

Fig. 6 shows the measurement results for a mineral oil based grease. For this grease, the ATR method showed the signal with the same peak positions as the transmission method. A spectral search confirmed that the main component of the mineral oil was paraffin.

With the ATR method, grease can be measured easily by simply spreading it onto the prism. However, in the case of samples with strong absorption, it is necessary to be careful because the results may differ from the transmission method.

■ Measurement Techniques

If a thick sample of ordinary plastic or rubber is placed in close contact with the prism and the sample is thicker than the penetration depth, then the ATR spectrum intensity will not increase further even if the sample thickness is increased. However, in the case of samples with strong absorption, peaks will shift toward a lower wavenumber and the intensity ratios will change more than if measured by the transmission method. Therefore, for samples with strong absorption, it may be possible to obtain spectra similar to the transmission method by applying an extremely thin layer for the ATR method or by changing the prism to germanium (Ge). Fig. 7 shows an illustration of the penetration depth. If the sample is sufficiently thinner than the penetration depth, then even if the absorption of the sample is strong, the peak shift and change in the intensity ratio are also suppressed, though the overall intensity will decrease. Furthermore , since Ge has a higher refractive index than Dia, the penetration of light in Ge is about 1/3 of that in Dia*1, given an incident angle of 45 degrees, for example.

Fig. 8 to 11 show the measurement results under the conditions indicated in Table 1, where silicone and fluorine greases were applied very thinly to the Dia prism, and under the conditions shown in Table 2, where the prism was changed to Ge.

*1 When the prism was changed to Ge, the measurable wavenumber range narrowed toward the lower wavenumber end.

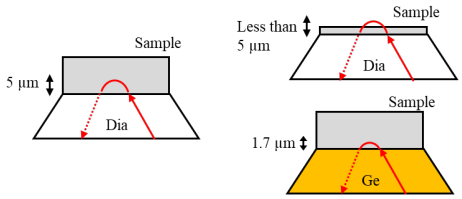

Fig. 7 Illustration of Penetration Depth for the ATR Method
with a Dia Prism (Left), with Grease Applied Very Thinly
to a Dia Prism (Upper Right), and with a Ge Prism (Lower Right)

Table 2 Measurement Conditions

Instruments:	IRSpirit-TX, Microm ATR (Ge)
Wavenumber Range:	4,000 to 600 cm^{-1}
Resolution:	4 cm^{-1}
Accumulation:	40
Detector:	DLTGS

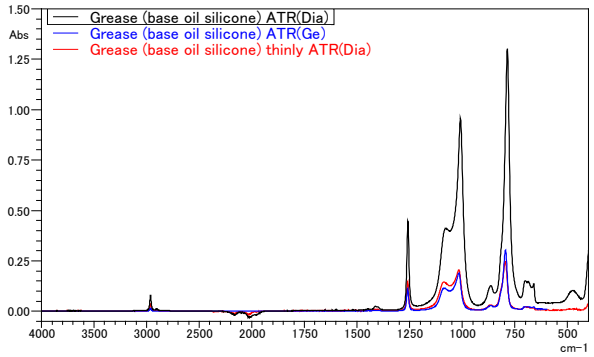

Fig. 8 Infrared Spectra of Commercial Grease (Base Oil: Silicon)

Fig. 8 shows the measurement results of grease with a silicone base oil. If the grease is applied thinly to a Dia prism or if the prism is Ge, the penetration of light is shallower and the absorption is less than the case where no special measures are taken.

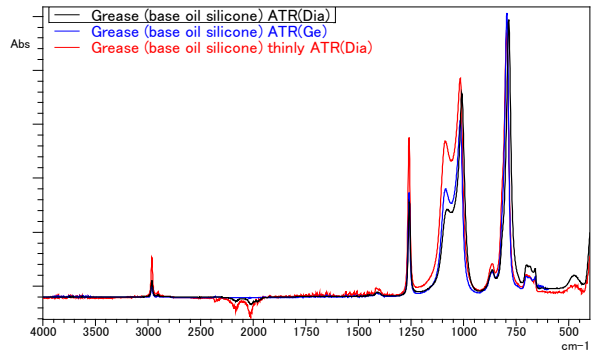

Fig. 9 Normalized Fig. 8 Spectra

Fig. 9 shows the results from normalization of the spectra in Fig. 8. In the case where the grease is thinly applied to a Dia prism or the prism is Ge, the results confirmed that the peaks in the 1,300 to 700 cm^{-1} range had a smaller shift toward the lower wavenumber end and approached the peak wavenumbers obtained using the transmission method.

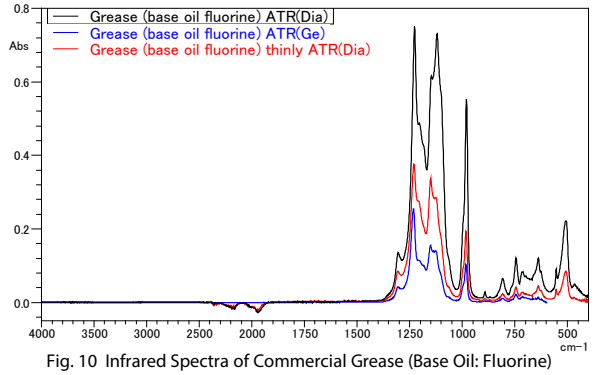

Fig. 10 Infrared Spectra of Commercial Grease (Base Oil: Fluorine)

Fig. 10 shows the measurement results for grease with a fluorine base oil. Similar to the silicone base oil, it shows that the penetration depth and absorption decreased when the grease was applied thinly to the Dia prism or when the prism is Ge.

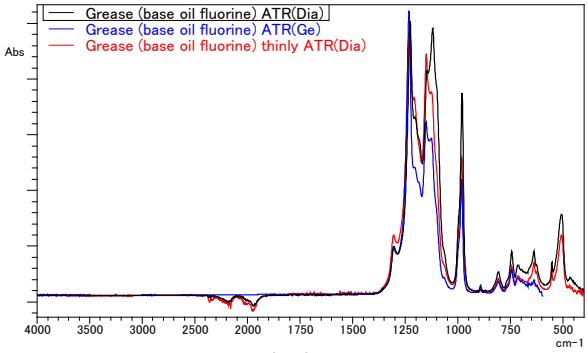

Fig. 11 Normalized Fig. 10 Spectra

Fig. 11 shows the results from normalization of spectra in Fig. 10. When the Dia prism was thinly coated or when the prism was Ge, the peaks in the 1,300 to 900 cm^{-1} range showed a smaller shift toward the lower wavenumber end and were closer to the peak wavenumbers of the transmission method.

When a sample with strong absorption is measured by the ATR method, a spectrum with peak positions similar to that of the transmission method may be obtained by applying the sample very thinly or changing the prism to Ge.

■ Analysis Technique

In some cases of samples with strong absorption, spectra comparable to the transmission method may be obtained by performing advanced ATR correction*2 after using the ATR method. For data obtained using a Dia/Ge prism (Fig. 1, 2, 8, and 10), advanced ATR correction was performed and the result was compared to the transmission method, as shown in Fig. 12 to 13.

*2 Advanced ATR Correction: This correction is used to make ATR measurement results the same as those obtained by the transmission method.

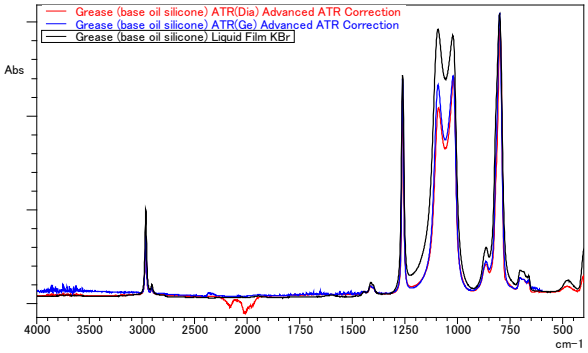

Fig. 12 Infrared Spectra Analysis of Commercially Available Grease (Base Oil: Silicone)

Fig. 12 shows the results from analyzing grease with a silicone base oil. By performing advanced ATR correction, not only the intensity ratio of overall measurement wavenumbers (such as the intensity ratio correction for 3,000/750 cm^{-1}) but also the peak intensity ratio at 1,200 to 1,000 cm^{-1}, which uniquely corresponds to silicone base oils, is corrected correctly and the results are equivalent to those obtained by the transmission method.

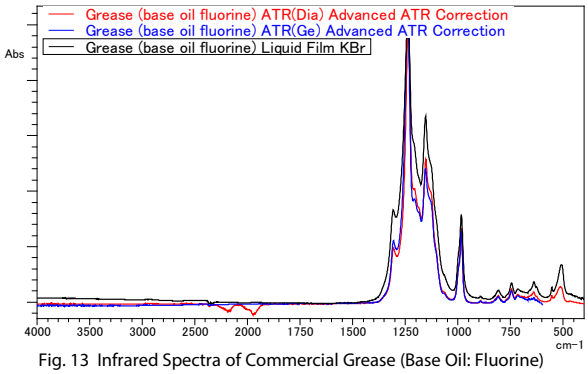

Fig. 13 Infrared Spectra of Commercial Grease (Base Oil: Fluorine)

Fig. 13 shows the results from analyzing grease with a fluorine base oil. As in the case of silicone grease, the advanced ATR correction corrects not only the intensity ratio of the overall measured wavenumbers but also corrects the intensity ratio of peaks in the 1,300 to 900 cm^{-1} range, which uniquely correspond to fluorine grease, to achieve results equivalent to the transmission method.

Thus, for samples with strong absorption, advanced ATR correction may provide a spectrum with peak positions and intensity ratios comparable to the transmission method.

■ Conclusion

Commercially available greases were measured by the ATR method and the transmission method.

For mineral oil greases, the ATR method and the transmission method yielded spectra with similar peak positions. However, because silicone and fluorine greases have strong absorption, the ATR method yielded spectra with different peak positions than the transmission method.

As a measurement technique, it was confirmed that spectra with similar peak positions as the transmission method could be obtained with the ATR method for silicone and fluorine greases by applying an extremely thin layer or changing the prism to Ge.

The results confirmed that using advanced ATR correction as an analysis technique could provide spectrum with the same peak positions/intensity ratios as the transmission method.

Thus, when comparing samples measured by different methods, it is necessary to selectively use different measurement and analysis techniques as necessary.

Related Applications

1. Advanced ATR Correction to Convert ATR Spectra to Transmission Spectra, Application News No. A476
2. Degradation Analysis of Lubricants Based on ASTM E2412 by Fourier Transform Infrared Spectrophotometer FTIR, Application News No. A603

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

01-01096-EN First Edition: May 2026

› Please fill out the survey

Related Products Some products may be updated to newer models.

› IRSpirit-X Series

Related Solutions

› Hydrocarbon Processing Industry (Petrochemical, Ch

› Petroleum refinery

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry