

## Investigation of Lubricating Grease using Terahertz Transmission Spectroscopy

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**Abstract**—A nondestructive and contactless screening method for selected lubricating grease stored in common plastic box has been examined using the terahertz (THz) time-domain spectroscopy. The THz wave has been transmitted through the commercially available lubricating grease in polystyrene filters. Greases have unique spectral features in the THz range, and the differences in the absorption coefficient and the refractive indices, which reflect the ingredients of organic compounds, suggested that the THz spectroscopy can be used to characterize the type and consistency of lubricating grease.

**Keywords**—terahertz; transmittance; lubricating grease

### I. INTRODUCTION

Lubricating grease is formulated from a base oil (typically 85%) and a thickener system at ~10% plus other possible ingredients imparting special properties. Grease is a viscoelastic plastic solid, therefore, a liquid or solid, dependent upon the applied physical conditions of stress and/or temperature [1, 2]. The grease in liquid or solid phase is always stored in plastic containers. Infrared spectroscopy has been used for a number of years in grease research to establish the nature and composition of the dispersion medium of grease [3]. The development of inspection methods for plastics containers containing grease has been the urgent issue on the manufacture and transportation. Mid-IR absorption and Raman scattering are possible methods of inspecting in grease store in transparent containers. These spectroscopic techniques, however, can not be applied for opaque plastic containers [4].

The terahertz (THz) band offers a plethora of fingerprints for many chemical and petroleum products. THz wave has some of the properties of radio light. It is to penetrate many non-conducting materials. Recently, there has been a remarkable effort in employing THz time-domain spectroscopy (THz-TDS) for investigating the properties of gasoline [5], diesel [6], oil-water complexes [7], lubricants [8-9], and organics [10]. THz-TDS differs from infrared Fourier transform spectroscopy (FTS) in a number of important aspects that give it some significant advantages. THz-TDS uses coherent pulsed sources, typically of 1 to 2 ps duration, while FTS uses continuous-wave non-coherent sources. THz-TDS can provide both absorption coefficient and refractive index of a sample with high signal-to-noise ratio (SNR) and without using the Kramers-Kronig relation [11-12].

In this paper, a nondestructive and contactless scanning method has been examined using THz-TDS for lubricating grease, which was stored in plastic box made of polystyrene (PS). Our results indicated that the transmittance and refractive index in the THz region were dependent on the type and consistency of commercially lubricating grease.

### II. EXPERIMENTAL METHODS

A THz-TDS system is illustrated schematically in Fig. 1. All measurements were performed with a conventional transmission setup for THz-TDS based on a ZnTe emitter for terahertz generation and electro-optic sampling [13]. THz transmission measurements were conducted for semi-solid lubricating grease stored in PS box in the path of the THz wave. By visible inspection, any macroscopic bubble, which would have influence on the results, was not found in the samples. The PS box was transparent for visible light with a side thickness of less than 1 mm and path length of 30 mm. Oils are classified in the SAE system, so grease is classified in the NLGL (National Lubricant Grease Institute of America) by means of penetration. The different kinds of

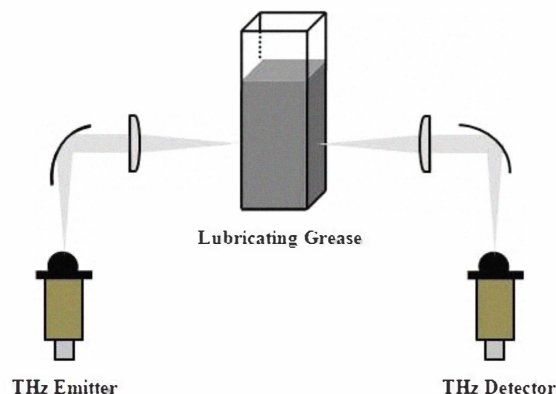


Figure 1. Schematic diagram of the transmission measurement of the THz-TDS. A polystyrene box containing lubricating grease is placed in the path of the THz wave.

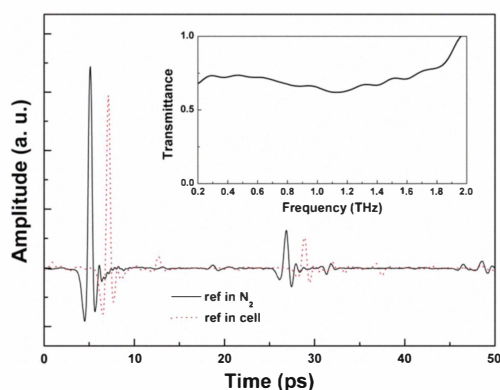


Figure 2. THz time-domain spectra of THz wave pass through nitrogen and empty polystyrene cell. Inset: THz transmittance of the empty PS cell in the frequency range extending from 0.2 to 2 THz.

lubricating greases are collected from Shell Tongyi (Beijing) Petroleum Chemical Co. Ltd. The samples are synthesized high temperature grease (SHTG) and molybdenum disulfide extreme pressure lithium grease (MoS<sub>2</sub>-EPLG) with NLGL number 0# (MoS<sub>2</sub>-EPLG0), 1# (MoS<sub>2</sub>-EPLG 1) and 2# (MoS<sub>2</sub>-EPLG2). The empty cell is used as the reference measurement. The experiment box was filled with nitrogen to prevent the absorption by atmospheric water vapor. The samples were investigated at room temperature and humidity was kept less than 3.0%.

### III. RESULTS AND DISCUSSION

The time-domain spectra of the THz wave propagated through nitrogen and empty cell are shown in Fig. 2. Because of the Fabry-Perot reflection effect, one supplementary peak appears behind the main peak. Furthermore, one sees that the amplitude of the THz pulse in the time domain is attenuated when the THz pulse passes through the empty cell. As calculated from measured THz time-domain spectra, the transmittance of empty PS cell is more than 88% in the frequency range extending from 0.2 to 2 THz. The transmittance of polyethylene and polyethylene terephthalate bottles investigated by T. Ikeda are below 80% and decrease as the THz frequency increases within the 0-60 cm<sup>-1</sup> [14]. The results show that PS is an excellent material as a filter for THz spectroscopy and THz imaging.

In Fig. 3, the THz transmittance spectra (=Esam+PS(v)/EPS(v)) of the greases stored in PS boxes were calculated [15], where Esam+PS(v) and EPS(v) are complex THz fields transmitted through a grease-containing box and an empty box at a frequency v, respectively. The difference in the THz transmittance spectra for various greases depended on the species and consistency of the grease. These greases consist of different kinds of hydrocarbon ingredients, soaps and some performance additives, leading to the different intensities of the THz absorption. In addition, the difference in the THz transmittance spectra for various greases implied the possibility for sample classification using the THz-TDS.

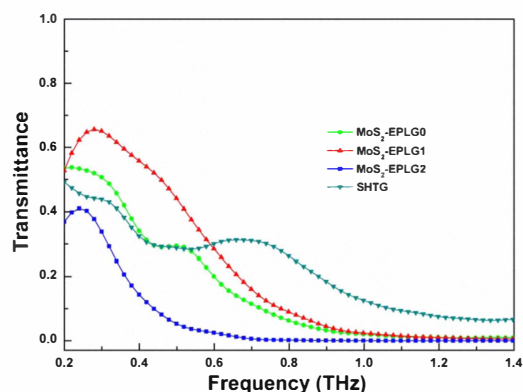


Figure 3. THz transmittance of lubricating greases filled in the PS boxes.

The power absorption coefficients and refractive indices of the investigated lubricating greases were calculated as shown in Fig. 4. Previously, molybdenum disulfide lithium greases with different consistency were studied, and the interaction of layers and resonance of own layers of MoS<sub>2</sub> give rise to the strong absorption [16]. Due to the high absorption of MoS<sub>2</sub>, the MoS<sub>2</sub>-EPLG also has a strong absorption in the effective spectral range 0.2-1.4 THz. There is no obvious absorption peak in the absorption spectrum of SHTG and MoS<sub>2</sub>-LG and the absorption coefficients of four greases show a slow increase as the THz wave frequency increases. The spectra of these samples may be ascribed to both intramolecular and intermolecular vibration modes in experimental range. The refractive index has no change as the frequency increases. The refractive indices varying from 1.5 to 1.92 display average values of 1.513, 1.915, 1.846 and 1.822 for SHTG, MoS<sub>2</sub>-EPLG0, MoS<sub>2</sub>-EPLG1, and MoS<sub>2</sub>-EPLG2, respectively. The refractive indices of greases with different NLGL grades can be identified and predicted using the Lorentz-Lorenz relation [16]. Additional dissolved

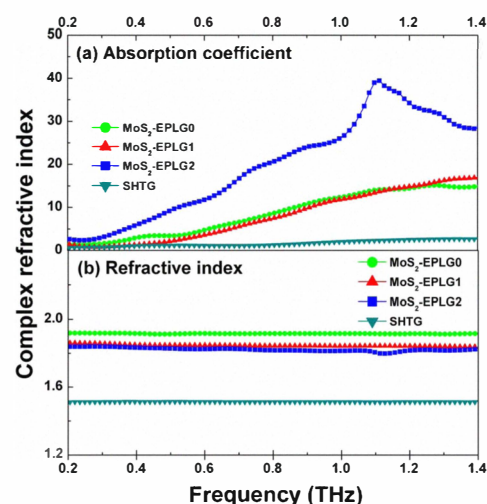


Figure 4. The frequency dependence of the complex refractive index of lubricating greases with different type and consistency. substance of the components to increase consistency of greases will change the refractive index.

#### IV. CONCLUSIONS

In summary, we have examined the feasibility of application of the THz-TDS to the nondestructive and contactless screening method for lubricating greases stored in the polystyrene filters. The transmittance of empty PS cell has a remarkably high value more than 88% and little variation as a function of frequency. Furthermore, the commercially available lubricating greases with different type and consistency in the PS filters can be distinguished by optical properties. The differences in the transmittance and the refractive index imply that the THz-TDS can be used for identification of lubricants.

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