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Nondestructive evaluation of multilayered paint films in ambient atmosphere using terahertz reflection spectroscopy

Jindoo Choi, Won Sik Kwon, Kyung-Soo Kim *, Soohyun Kim *

Department of Mechanical Engineering, Korea Advanced Institute of Science and Technology, Daejeon 305-701, Republic of Korea

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ABSTRACT

The reliability of material characterization with terahertz (THz) waves is typically compromised by irrelevant signals present in the THz measurements. These are well observed in the investigation of layered materials in humid environments, causing difficulty in accurate analyses. In this article, we suggest a realistically employable method of analyzing angled THz reflection measurements in ambient atmosphere for qualitative monitoring and evaluation of multilayered paint films. In particular, we present successive decomposition of THz sample signals for individual identification of each constituent layer in the samples. It is shown that the extracted spectrum of each paint layer presents sufficient information for effective monitoring of the multilayered structures. In this process, THz spectra of the marine paints EH2350, Interthane 989, BEA777, and ENA300 are reported for the first time. Our experimental findings show promise in highly effective nondestructive inspection of layered structures for real-world evaluation applications.

1. Introduction

Pulsed terahertz (THz) broadband systems have become remarkably important tools for the investigation of numerous materials over frequencies from a few hundred GHz to several THz. Significant developments in THz science [1–3] have enhanced the performance of material characterization and triggered extensive applications in security [4], medicine [5], semiconductors [6], quality control [7,8], etc. In particular, an area that would considerably benefit from these advancements is nondestructive evaluation of various materials in industrial applications [9–12]. However, the inspection results are generally affected by various artifacts present in the THz measurements, such as electronic noise, signal fluctuation from atmospheric water vapor absorption [13], and echo signals arising from internal reflections from the sample or system components [14]. These complications are frequently risen in analyses of layered materials in ambient atmosphere, hence, causing hardship in accurate characterization.

In the last decade, several authors proposed insightful approaches to overcome these obstacles. The temporal peak analysis [8] and the inverse electromagnetic problem [15–17] are most commonly used, but they require several reflections of the THz pulse and are typically applicable only to homogeneous, low absorption, and single-layered materials. THz signal manipulation techniques, such as the removal of multiple echo signals [18–20] and signal reconstruction from atmospheric attenuation [21,22] are greatly competent in gaining fluctuation-free THz signals but face complex signal processing procedures and inevitably possess the possibility of data alteration. The recent dispersion model-based methods [23,24] provide extremely accurate material analyses, but sometimes require prior knowledge about the sample or undergo necessary parameterization schemes. Therefore, these methods are mostly valid only in limited situations and still remains relatively unused in real-world applications.

In our earlier report [25], we have introduced a simple and intuitive model-based approach that can be universally applied for the decomposition of THz transmission signals. The method showed distinctive advantages in that it did not require any prior information of the sample under investigation or incorporate removal/reconstruction techniques of any kind. Here, we extend the findings from our previous report and present qualitative monitoring and evaluation of materials with layered structures. Particularly, we suggest a realistically employable method of analyzing angled reflection measurements, the most feasible way of nondestructively testing various samples. The article encompasses the investigation of multilayered paint films, verification of the THz signal decomposition algorithm in uncontrolled temperature and humidity settings, and THz reflection time-domain spectroscopy. A brief description and background of the proposed method is given, followed by experimental results and comprehensive performance assessment.

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* Corresponding authors.

E-mail addresses: kyungsookim@kaist.ac.kr (K.-S. Kim), soohyun@kaist.ac.kr (S. Kim).

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2. Experimental details

2.1. Terahertz reflection time-domain spectrometer

The THz spectrometer used in this experiment is similar to the setup of Ref. [26] and is consisted of a femtosecond laser, photoconductive THz emitter and an electro-optic detector (Fig. 1). Pumped by a continuous wave 532-nm diode-pumped solid state laser, the mode-locked Ti:Sapphire femtosecond laser (pulse duration = 25 fs, center wavelength = 800 nm, average power = 250 mW) emits a femtosecond pulse, divided into a pump and probe beam by a beam splitter. The pump beam generates pulsed THz radiation upon excitation of a low-temperature-grown GaAs photoconductive antenna (TERA-SED10, Laser Quantum) modulated by a square wave (frequency = 70 kHz, amplitude = 20 Vpp) used as the reference signal of a discrete signal processing lock-in amplifier. The diverging, p-polarized THz wave is focused onto the sample at an incident angle of 45° and the reflected wave is recollected onto the detector using four off-axis parabolic gold mirrors. THz amplitude is detected by the probe beam at a 1 mm-thick ZnTe crystal, where a quarter-wave plate and Wöllaston prism enable balanced detection [27]. A complete temporal scan of the THz wave is recorded using a motorized delay stage.

2.2. Sample preparation

Paints EH2350 (KCC Corporation), Interthane 989, and BEA777 (International®), frequently used for the protective coating of marine ships, were separately coated onto a layer of ENA300 (International®), producing three types of multilayered samples. Each paint layer was a few hundred microns in thickness. All samples were prepared on $9 \times 9\ \mathrm{cm}^2$ steel plates to allow reflection measurements, in which the paint thickness variation across the sample area was limited to 2–5% according to eddy current measurements. The paints used in this work were sufficiently stirred to a homogeneous consistency before preparation of each sample. A gold mirror was used to obtain reference data. Experiments were conducted in an environment with a temperature of ~21 °C and relative humidity of ~61% to involve the effects of atmospheric absorption of THz waves. These natural settings were not artificially controlled or modified.

3. Terahertz signal decomposition theory

3.1. Assumptions and notations

Throughout this paper, the prepared samples are assumed to be homogeneous (layer-wise) and planar (at least for the region illuminated by the THz beam) to neglect scattering effects or incident angle errors. This will ensure the spatial and directional consistency of the sample characteristics. We denote the propagation, transmission, and reflection coefficients as $P_a(x)$, T_{ab} and R_{ab}, respectively. $P_a(x)$ represents the propagation of a THz wave in material a after length x. T_{ab} and R_{ab} respectively describe the transmission and reflection coefficients at the interface of materials a and b when the wave propagates from material a to material b:

$$P_a(x) = \exp\left(i2\pi f \frac{\tilde{n}_a x}{c}\right), \tag{1}$$

$$T_{ab} = \frac{2\tilde{n}_a \sec\theta_2}{\tilde{n}_a \sec\theta_1 + \tilde{n}_b \sec\theta_2}, \tag{2}$$

$$R_{ab} = \frac{\tilde{n}_a \sec\theta_1 - \tilde{n}_b \sec\theta_2}{\tilde{n}_a \sec\theta_1 + \tilde{n}_b \sec\theta_2}, \tag{3}$$

where f indicates the frequency and c the speed of light in vacuum. $\tilde{n}_a$ and $\tilde{n}_b$ are the complex refractive indices of materials a and b, respectively. θ_1 and θ_2 are the incidence and refraction angles, respectively, when the THz wave propagates from material a to material b.

3.2. Sample transfer function model

A sample under investigation can be characterized in the THz range by comparing the THz sample and reference signals, in which the former possesses sample information (by traveling

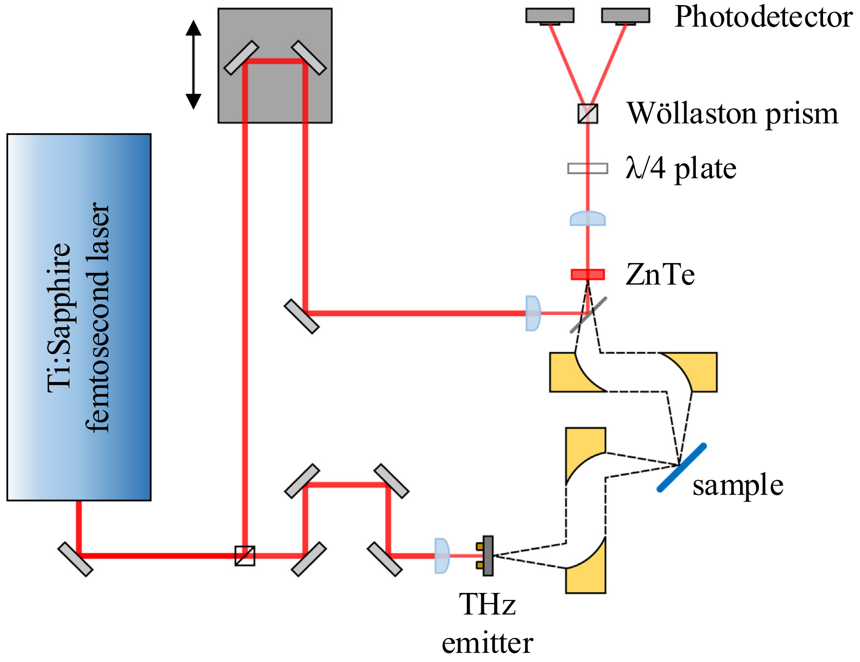

Fig. 1. THz reflective time-domain spectrometer apparatus.