Paper:

Thickness Determination of Plastic Products using Continuous Wave Terahertz Frequency-domain Spectroscopy

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Abstract. In this paper, a novel method for thickness determination of plastic products using continuous-wave (CW) THz frequency-domain spectroscopy is proposed. This method begins with the refractive index measurement of target material over THz frequencies. The optimal frequency band with high SNR and minimum water vapor affection is then selected for thickness prediction. For samples with unknown thickness, the THz wave's optical path is initially reconstructed from the phase delay of the recorded data. The calibrated refractive index is then applied to calculate the physical thickness of the sample. The proposed method is exemplified by the widely used plastic material Teflon. The thicknesses predicted by THz-FDS measurements are coincided with conventional caliper measurements perfectly.

Keywords: Terahertz waves, Non-destructive inspection, Plastic industry, Thickness determination

1. INTRODUCTION

Terahertz (1 THz = 10^{12} Hz) waves are recognized as electromagnetic radiations whose frequencies are located between microwaves and infrared light [1]. In the past two decades, terahertz (THz) spectroscopy has been proved to be an excellent alternative for conducting in-line, non-destructive inspection for the plastic industry [2, 3]. The major components in the plastic products are polymer materials which are generally transparent or semi-transparent for the THz radiation [4]. Thus, the THz wave is able to pass through the plastic products while carrying the inside information, such as thickness, refractive index, absorption features, dielectric constants, birefringence, and even water content [5–7]. Those valuable information leads to promising industrial applications, including the polymeric compounding process monitoring for the synthesis optimization, defects and uniformity inspection for quality control, and adhesive bonds evaluation at welding joints for in-service performance modification [8–10]. Most of the previous THz investigations were performed by adopting the classical THz time-domain spectroscopy (THz-TDS), where a picosecond pulse is generated and coherently recorded as a time trace by the combination of the femtosecond laser, semiconductor antennas pair, and the mechanical delay line [11]. Despite the fact that the application of THz-TDS was well established in the past two decades in terms of data processing method and experimental setup performance, the fragile femtosecond laser is too bulky and expensive to be chosen by the industry against existing inspection methods such as ultrasonic, FT-IR and X-ray techniques. Aiming to lower the system cost as well as improve the robustness of the THz setup, THz frequency-domain spectroscopy (THz-FDS) was developed [12] where the continuous-wave (CW) THz radiation is generated by two NIR lasers with adjustable wavelength combing with the semiconductor antenna referred to here as photo-mixer. The frequency of the CW THz wave is determined and tuned by the wavelength difference between two NIR lasers. The detection of the CW THz wave can be performed by a bolometer or photo-mixer. The latter configuration equipped with a pair of photo-mixers is capable of directly measuring THz waves' phase information based on its coherent detection nature. However, the shortcoming of this novel photo-mixing THz-FDS is that the data processing method is comparatively not systemically established [13, 14], especially for the extraction of optical parameters, including refractive index and thickness. In this paper, a novel method for thickness determination of polymer materials using CW THz-FDS is proposed. The major novelty of this study is the establishment of the relationship between the raw signal and phase information for the THz-FDS experiment. Our method starts with the calibration of the refractive index over THz frequencies for the target material. The optimal frequency band with higher SNR and minor affection from water vapor is then selected for thickness estimation. For samples with unknown thicknesses, the optical path which THz wave travels through is initially extracted from the phase delay of the recorded CW THz photo-current. The obtained refractive index is then applied to calculate the physical thick-

ness of the sample. The proposed method was exemplified with the widely used plastic material polytetrafluoroethylene (PTFE), also known as Teflon. Two sets of samples were prepared, out of which the first group was used to measure the refractive index of PTFE while the second group was used to evaluate the thickness determination accuracy.

2. EXPERIMENTS

2.1. Experimental Setup

The experimental data acquisition in this paper was conducted on a commercially available THz-FDS system (TeraScan 1550, TOPTICA PHOTONICS, Germany) based on photo-mixing. Fig. 1 presents the schematic diagram of the THz-FDS setup. The system applies two distributed feedback (DFB) lasers as the frequency mixing light source whose initial wavelength is centered at 1533 nm and 1538 nm, respectively. As mentioned before, the frequency difference between two DFB lasers is located in the THz frequency band. Consequently, a beat signal oscillates at THz frequency is generated by the laser coupler. After beam splitting, one of the beat signals irradiates on the photo-mixer antenna at the transmitter. CW THz radiation with exact the difference frequency of the lasers is output under the applied bias voltage. The THz radiation meets another beat signal on the photo-mixer antenna at the detector. A photo-current whose magnitude is related to both the amplitude and phase of the THz electric vector is then recorded by a lock-in amplifier. The photo-mixers applied in this system are based on InGaAs. The frequency tuning is realized by modulating the wavelength difference of two DFB lasers. The minimum modulation frequency can reach 1 MHz, and the reliable spectral band is 200 - 1200 GHz. The THz-FDS setup used in this study adopts the all-fiber design for the purpose of real industrial application.

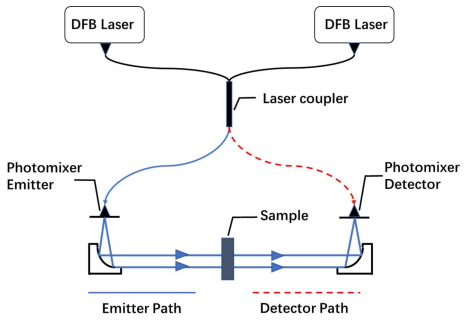


Fig. 1. THz-FDS experimental setup

Table 1. Prepared pellets in this study.

Sample Label	Sample information	
	Thickness (mm) [a]	Weight(mg)
RI-PTFE-1	1.61±0.01	479.9
RI-PTFE-2	1.76±0.02	530.3
RI-PTFE-3	1.91±0.01	577.7
RI-PTFE-4	2.11±0.01	631.9
RI-PTFE-5	2.21±0.01	668.8
PD-PTFE-1	1.64±0.02	486.3
PD-PTFE-2	1.81±0.02	536
PD-PTFE-3	1.93±0.01	580.4
PD-PTFE-4	2.07±0.01	617.6
PD-PTFE-5	2.25±0.02	679.9

[a] Thickness uncertainties originated from triplicate caliper measurements

2.2. Sample Preparation

In this study, PTFE was selected to exemplify our thickness determination method for its widespread applications in the plastic industry. PTFE was purchased from Micro Powders (FLUO 625F) in the form of white powder. Experimentally investigated samples were prepared as compressed round pellets. For each preparation, the PTFE powder was pressed into 13 mm diameter pellets by applying a stainless-steel die in conjunction with a hydraulic press. Prior to compression, a moderate vacuum was applied to the mixture within the die in order to minimize porosity. After the vacuum was applied for 2 minutes, the PTFE pellet was produced by applying 5 tons of pressure to the die for 5 minutes. Freshly compressed pellets were removed from the die and placed in a desiccator for 2-3 days to ensure pellets were fully relaxed prior to collection of the THz data. Mass, thickness, and diameter were measured for each relaxed pellet. Following such procedure, ten PTFE pellets were prepared in total using masses ranging from 479.9 to 668.9 mg resulting in the thicknesses ranging from 1.607 to 2.253 mm. Those pellets were equally divided into two groups. The first group was designed to calibrate the refractive index (RI) of PTFE, while the second group was prepared to evaluate the thickness prediction (PD) of our method. The details of all the prepared pellets are listed in table 1.

2.3. Data Collection

The photo-current data were collected with a step-size 0.05 GHz and an integration time of 3 ms. Data acquisition for data ranging from 200-1200 GHz with this step-size/integration-time combination could be finished in less than 30 seconds. It would be faster in the thickness determination application if a proper wavelength selection was settled. For each pellet, a reference signal for which the THz wave passes through the air is recorded first, followed by a measurement of the sample signal where the pellet was mounted on a sample holder located in the THz wave propagation path. Triplicate measurements were conducted on each sample to obtain an average photo-current spectrum. It should be noted that all the data collection was conducted under an ambient environment to test the robustness of the proposed method against water vapor affect.

3. THICKNESS DETERMINATION ALGORITHM

The recorded photo-current intensity I_{det} is related to the terahertz electric field [12] by equation 1:

$$I_{det}(f) \propto E_{thz}(f) cos(\frac{2\pi f l}{c}) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

, where E_{thz} is the terahertz electric vector intensity, f is the frequency, and c is the speed of light in a vacuum. As an important parameter for thickness determination, l is the length difference between the emitter path and detector path indicated in Fig. 1. In our system, l is the THz wave path length and keeps fixed during each data collection. Hence, the raw photo-current recorded by the system would exhibit the characteristic of periodic oscillation against the change of frequency f according to the phase term $\frac{2\pi f l}{c}$ in equation 1, as shown in Fig. 2. Assuming f_m is the mth extreme of the photo-current, then the phase should follow:

$$m\pi = \frac{\pi f_m l}{c} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

It should be noticed that equation 2 is valid for both reference signal and sample signal:

$$m = \frac{f_{r,m} l_r}{c} = \frac{f_{s,m} l_s}{c} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

, where $f_{s,m}$ and $f_{r,m}$ is the frequency of the mth extreme of the photo-current for reference and sample signal. By setting the sample in the beam path, the variation in the refractive index makes the THz path-length change from l_r to l_s. For terahertz spectroscopy with coherent detection, either THz-FDS applied in this paper or classical THz-TDS, the essence of measuring the refractive index n_s or thickness d_s of the sample is to extract the optical path variation Δl from the measured spectra, as described in equation 4:

$$\Delta l = l_s - l_r = (n_s - 1) d_s \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Combination of equation 3 and 4 gives:

$$n_s = 1 + \left(\frac{f_{r,m}}{f_{s,m}} - 1\right) \frac{l_r}{d_s} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (5)$$

and

$$d_s = \left(\frac{f_{r,m}}{f_{s,m}} - 1\right) \frac{l_r}{n_s - 1} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (6)$$

, in which l_r can be extracted from the reference signal according to equation 3. The extreme point frequency $f_{r,m}$ and $f_{s,m}$ is accessed by applying a peak search algorithm to the reference and sample signal, respectively. The refractive index n_s of the target polymer needs to be calibrated from samples with known thicknesses according to the equation. All the parameters on the right side of equation 6 are accessible with the refractive index obtained. Therefore, the physical thickness prediction for plastic samples is available. For better description of our proposed thickness determination method, a flowchart of the general process is presented in Fig. 3.

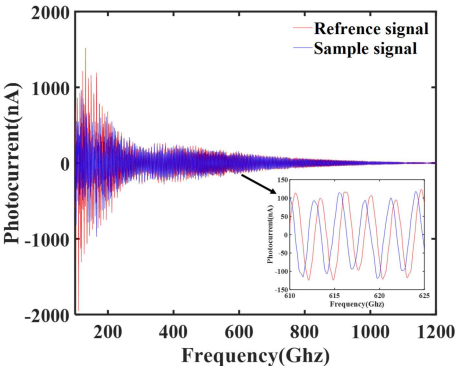


Fig. 2. Reference (blue) and sample (red) signal initially collected with THz-CW system. Spectra between 610-625 GHz was enlarged to show the oscillation mismatch corresponding to the phase information contained in the raw data.

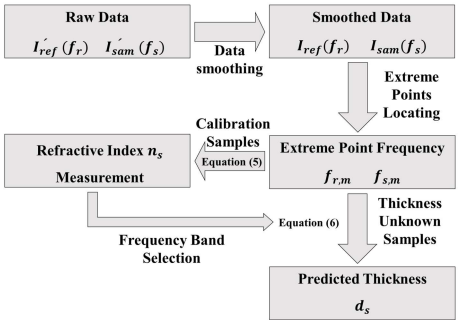


Fig. 3. Flowchart for thickness determination of plastic products using continuous wave terahertz frequency-domain spectroscopy.

4. RESULTS AND ANALYSIS

4.1. Refractive Index Calibration and Frequency Band Selection

The refractive index spectra ranging from 200-1200 GHz of PTFE measured with five pellets with different thicknesses in the refractive index calibration RI group is presented in Fig. 4. The overlapped spectra from tablets at different thicknesses indicate the reproducibility of our THz-FDS setup as well as the refractive index calculation method. The magnitude of the refractive index is comparative to the previously reported value [4]. Several dips marked with numbers can be noticed in all five spectra. Those features originated from the well-known water vapor strong absorption lines in the investigated spectral range centers at 557GHz, 747GHz, 996GHz, 1099GHz, and 1157GHz, respectively [15]. The frequency band selection was performed to minimize the affection arising from the water vapor and make our method more suitable for industrial application. The mean values, along with

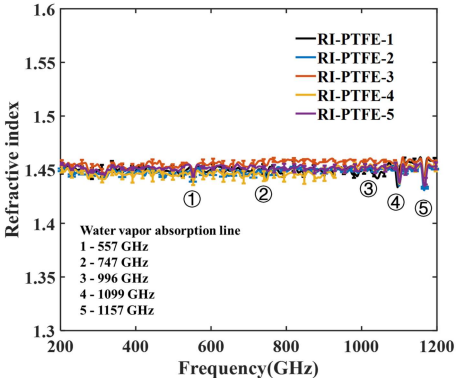


Fig. 4. Refractive index spectra of five PTFE pellets. Numbered dips indicate the corresponding positions of water vapor absorption features with specified central frequencies.

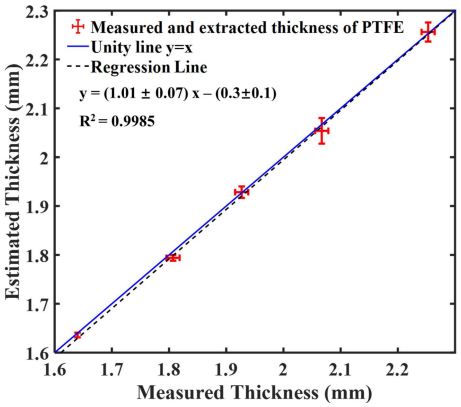


Fig. 5. Correlation plot between caliper measured thicknesses and THz-FDS predicted thicknesses. Error bar indicates the standard deviation between same sample triplicate measurements, both for caliper and THz-FDS.

Table 2. Refractive index result for frequency band selection

Frequency Band (GHz)	Refractive index results		
	Refractive index	Relative uncertainty (%)	Comments [a]
200-300	1.455±0.003	0.199	Low SNR
300-400	1.452±0.003	0.204	
500-600	1.453±0.003	0.207	V. A.
700-800	1.453±0.003	0.193	V. A.
800-900	1.454±0.003	0.171	
900-1000	1.455±0.003	0.253	V. A.
1000-1100	1.458±0.005	0.308	V. A.
1100-1200	1.457±0.004	0.296	V. A.

a. V. A. is short for vapor attenuation.
Water vapor attenuation features in investigated spectral range centers at 557GHz, 747GHz, 996GHz, 1099GHz, and 1157GHz, respectively.

the relative uncertainties of five samples' frequency averaged refractive index at eight bands, are listed in table 2. The frequency band 800-900 GHz outperformed other bands with its good SNR performance and absence of water vapor attenuation.

4.2. THz-FDS Thickness Determination Results

The thickness prediction was conducted on the PD group with the calibrated refractive index of PTFE. The resulting correlation plot is presented in Fig. 5 showing the individual points, the regression line as well as the ideal unity line. As shown in the figure, linear regression with R^2 = 0.9985 was presented between measured and predicted thickness for PTFE pellets. The standard difference of the calculated value compared to the measurement can be obtained from Equation 7, and the relative difference is given by Equation 8 [3]. For the values plotted in Fig. 5, the standard difference is 0.01 mm, and the average relative difference is 0.45%.

$$SE = \sqrt{\frac{\sum_{i=1}^{N}\left(d_{caliper,i} - d_{THz-FDS,i}\right)^2}{N}} \quad . \ . \ . \quad (7)$$

$$RE = \frac{\sum_{i=1}^{N}\left|\frac{d_{caliper,i} - d_{THz-FDS,i}}{d_{caliper,i}}\right|}{N} \times 100\% \quad . \ . \ . \quad (8)$$

5. CONCLUSION

A novel THz frequency-domain spectroscopic method is described for determining the thickness of the plastic subject in this paper. A series of Teflon samples were prepared to exemplify our method. The thickness prediction results are coincide with the conventional caliper measurements, with the overall relative difference being 0.45%. We expect that our method could make THz spectroscopy more competitive among existing quality inspection tools in the plastic industry.

Acknowledgements

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