Transmittance of High-density Polyethylene from 0.16 THz to 15 THz

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ABSTRACT

High-density Polyethylene (HDPE), with a density above 0.95 g/cm3, has been widely used in terahertz systems. The advantages of low absorption loss, low refractive index and high rigidity make HDPE an ideal material for cryostat window, focus lens and substrate. HDPE can be machined easily and be used as a substrate material for components such as metal mesh filters and polarizers. What's more, it is quite inert and can be used at cryogenic temperatures. On account of these applications, we need to characterize the dielectric property of HDPE precisely in a wide frequency range. In this paper, we present the transmittance measurements of a 2 mm thick HDPE sheet from 0.1 THz to 15 THz. Three kinds of measurement methods are employed to cover the whole frequency range. A vector network analyzer (VNA) combined with a quasi-optical transmissometer has been used to measure the transmittance and dielectric constant of HDPE from 0.16 THz to 0.18 THz at 300 K and 4 K. A Time Domain Spectrometer (TDS) is employed to cover the frequency range from 0.2 THz to 3 THz since the VNA can't work upon 1 THz. A Fourier Transform Spectroscopy (FTS) has been used for the measurement from 3 THz to 15 THz since the TDS can't achieve broad band and fast scan speed. The measured transmittance of HDPE is nearly 0.93 below 1 THz and decrease to 0.3 when the frequency increase to 15 THz. A rather elusive absorption band at 2.2 THz has also been observed. The dielectric constant of HDPE has been measured by VNA and TDS, showing a frequency independency from 0.1 THz to 3 THz.

Keywords: High-density Polyethylene, transmittance, THz, TDS, FTS, vector-network-analyzer

1. INTRODUCTION

The terahertz (THz) region of the electromagnetic spectrum has proven to be one of the most elusive. Terahertz radiation is loosely defined by the frequency range of 0.1 to 10 THz (~3 mm - 30 μm), situating between infrared light and microwave radiation. Terahertz technology is an extremely attractive research field, with interest from sectors as diverse as the semiconductor, medical, manufacturing, space and defense industries. In comparison to visible or infrared waves THz radiation can penetrate organic materials safely due to its low photon energy. High-density Polyethylene (HDPE), with the advantages of low absorption loss, low refractive index and high rigidity, has been widely used in terahertz systems as an ideal material for cryostat window, focus lens and substrate. What's more, HDPE is quite inert and can be used at cryogenic temperatures. On account of these applications, the dielectric property of HDPE should be characterized precisely in a wide frequency range.

Terahertz spectroscopy allows a material's optical properties to be determined as a function of frequency. This information can yield insight into material characteristics for a wide range of applications. Many different methods exist for performing THz spectroscopy. Broadband frequency-domain methods based on vector-network analyzers (VNA) [1] can describe the material properties from the lower microwave band to around 1 THz, with high dynamic-range and high spectral resolution. However, the VNA's dynamic range and broadband sweeping ability above 1 THz is dramatically reduced. Terahertz time domain spectroscopy (TDS) [2], based on direct measurements of the amplitude of ultrashort electromagnetic pulses has proved to be a versatile tool for quantitative determination of both spectral refractive index and spectral attenuation coefficient of a sample as well as providing high dynamic range at sub-millimeter wave bands. Although the spectral resolution of THz-TDS is much coarser than vector-network analyzers (VNA), its spectral range can increase to 5 THz. At higher frequency range, Fourier transform spectroscopy (FTS) [3] is perhaps the most common technique. It has the advantage of an extremely wide bandwidth, enabling material characterization from THz frequency

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to well into the infrared. In this paper, we present the transmittance measurements of a 2 mm thick HDPE sheet from 0.1 THz to 15 THz with the THz spectrometers mentioned above.

2. MEASUREMENT SETUP

2.1 Vector-network analyzers (VNA)

Vector Network Analysis has been extensively used in microwave measurement systems. It is a heterodyne receiver that is capable of measuring the amplitude and phase-difference between a test and reference path to very high accuracy and phase-stability. Ongoing advances in microwave electronics has extended operation of the VNA beyond low millimetre wave bands upto 1 THz. Vector Network Analysis has distinct advantages over Time Domain Spectroscopy (TDS) and Fourier transform spectroscopy (FTS). These are superior spectral resolution (down to 1MHz), a huge dynamic range (100 to 140 dB at low sub-THz band to 50-80 dB near 1THz) and, a rapid sweeping efficiency (spot rate less than picoseconds) [1].

The diagram of the vector network analyzer (VNA) combined with a quasi-optical bench is shown in Figure. 1. Two biconvex focus lenses (L1 and L2) are employed as quasi-optical collimators. Their sizes and positions are well designed to have a Gaussian beam-waist at the sample location and a beam waist smaller than the radius of the sample. The harmonic generator generates a RF signal through a sweepable centimeter source (S1) which provides a nominally flat output power in the frequency range of 8-18 GHz. The RF signal is focused by a lens (L1) to the sample which located at the symmetry center of the quasi-optical bench. The transmitted signal is then focused by another lens (L2) onto the receiver horn, where the incoming signal is mixed. The detected signal is PLL-controlled by the source signal (S1) with a frequency offset in MHz range. The mixed IF signal is then transferred to the VNA to extract the amplitude and phase spectrum. The transmittance spectrum is measured by recording the ratio of the complex spectrums of transmitted (with sample) and incident (without sample) signals. Dividing the complex transmittance spectrum will cancel all effects that not caused by the sample, except the phase shift ($e^{i2\pi fl/c}$, where l is the thickness of sample and f the frequency) due to the optical path inside the sample. The permittivity can be deduced from experimental data by looking at the observed phase or amplitude variation, more detail can be found in [1]. In this paper, we employ a VNA's post-processing function to calculate the average permittivity in the operating frequency range. A Fabry-Perot fitting has also been done with the measured data after Fourier-spectrum manipulation. A cryostat has been used for low temperature measurements at 4 K by injecting liquid Helium. The scanning frequency range is from 160 GHz to 180 GHz.

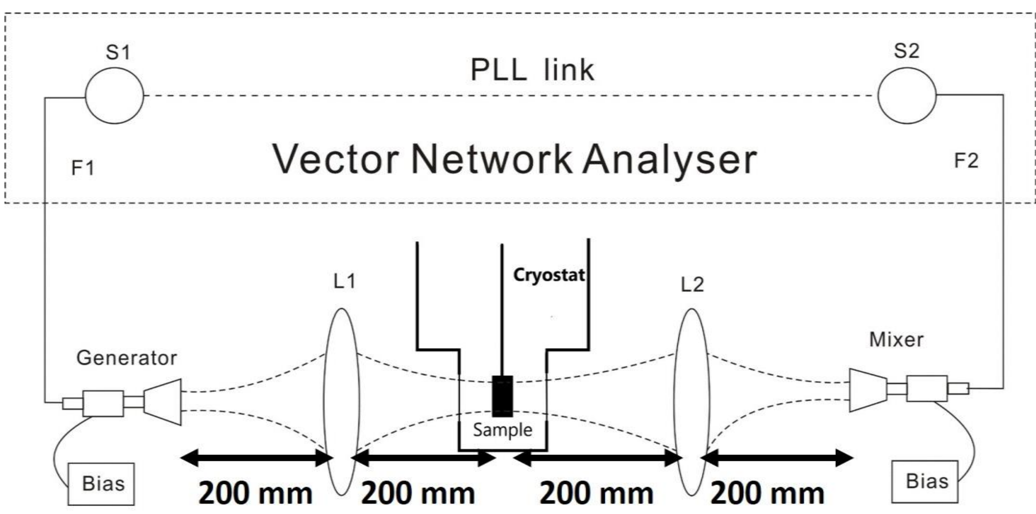

Figure 1. Schematic of the quasi-optical VNA setup.

2.2 Time domain spectroscopy (TDS)

The THz-TDS uses short pulses of broadband THz radiation, which are typically generated using ultrafast laser pulses. The THz TDS system, shown in Figure 2, employing a conventional configuration for free-space THz generation and detection [4, 5]. The system uses two channels of ultra-short pulse lasers with either biased output (for THz generation) or signal input (for THz detection). The generated THz beam are collimated and focused on the target using parabolic mirrors. After transmission through the target, the THz beam is collimated and re-focused on the THz detector. The optical probe beam is used to gate the detector and measure the instantaneous THz electric field (both amplitude and phase).

A unique optical sampling method, utilizing phase-modulated dual-laser-synchronized control technology without a mechanical optical delay line, enables extremely high-speed terahertz spectroscopy. A Cherenkov THz source module that utilizes non-linear optical crystal $LiNbO_3$ waveguides was applied to pump and detect the THz wave, with the central wavelength of 1550 nm and pulse duration of 50 fs. An all-in-one photoconductive THz detector complete with hyper-hemispherical silicon lens and fiber pigtailed was housing compactly.

The dynamic range of the THz spectroscopy system was around 70dB in amplitude, and the usable bandwidth was from 200 GHz to 3 THz. The generated THz pulse has an output power of about 30 mW, and the resolution of 7.6 GHz with a scan speed of 8 ms/scan. The optical configuration was designed to provide a focused THz beam for imaging and a parallel beam for spectroscopy. The THz spectrum is then obtained from the data by a Fourier transform. Since the absorption coefficient and the refractive index of the material studied are directly related to the amplitude and phase respectively of the transmitted field, both parts of the complex permittivity can be obtained by THz TDS.

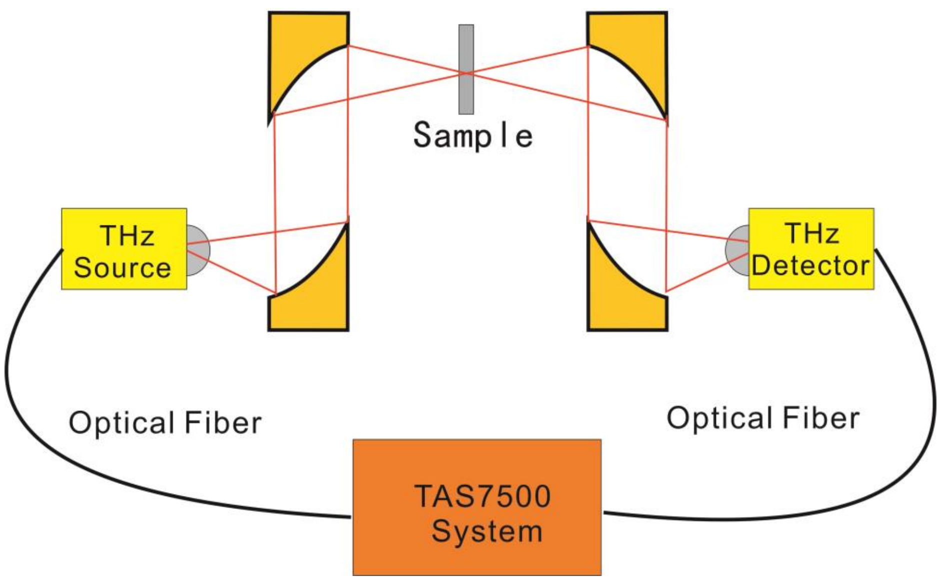

Figure 2. A schematic drawing of the THz TDS system.

2.3 Fourier transform spectroscopy (FTS)

A FTS system based on Martin-Puplett interferometer [6] was employed to measure the transmittance of HDPE above 3 THz. The schematic of this FTS is shown in Figure 3. The input signal is horizontal polarized at Polarizer 1, and is then divided by a 45° wire-grid, Polarizer 2, into two beams (reflected and transmitted) which have an angle of ±45º with horizontal plane respectively. Then they are reflected by a roof mirror and the polarization direction deflects 90 degrees. Therefore, both of them can pass through Polarizer 2 and no signal return to input port. Compared to a Michelson interferometer, a Martin-Puplett interferometer can provide a coupling efficiency much higher and a frequency dependence negligible since the wire grid is used instead of the dielectric film as beam splitter. The input ports of the FTS is 77 K blackbody. Roof mirror 1 is fixed and Roof mirror 2 is moveable. The instrument used DLATGS (deuterated L-alpha-alanine doped triglycine sulfate) pyroelectric detectors operated at ambient temperature. The spectral resolution was set by the interferometer scan length and the weighting applied for apodization. The scan length was ± 12.5 mm relative to the zero path difference (ZPD) position of the interferometer, and a Blackman weighting function was applied in processing. This yielded an instrumental resolution function with a full width at half maximum of 13.8 GHz. FTS measures only the field intensity, and therefore provides only the absorption coefficient. Although the refractive index can be calculated from the FTS data by using K-K relations, the calculation is not straightforward and has many potential sources of error.

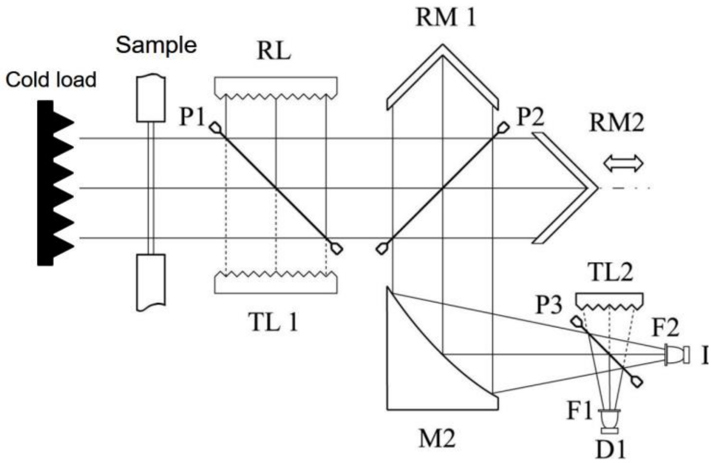

Figure 3. A schematic drawing of the Martin-Puplett FTS system.

3. RESULTS AND ANALYSES

Figure. 4 presents the measured and fitted complex transmittance spectrum of HDPE at 300 K from 156 GHz to 178 GHz by vector network analyzer. The phase-angle is modified to the zone of +180 to -180 degrees. The measured amplitude and phase of transmittance spectrum together with the simulated results are plotted in the same picture. The transmittance spectrum behaves as a Fabry-Pérot resonator, the observed periodic character is due to the interference between signal multi-passes in the sample. Obviously, as illustrated in Figure 4, the simulated curves coincide well with the measured results, indicating fairly high accuracy of measurement. The calculated dielectric constant and loss tangent at 300 K and 4 K have been listed in Table 1. It is easy to find that they don't change with the temperature.

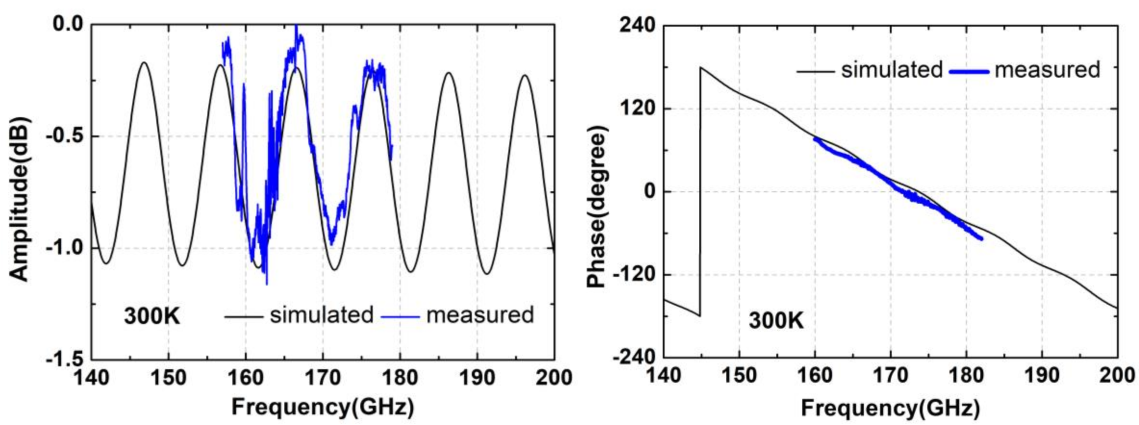

Figure 4. Transmittance spectrum of amplitude (left) and phase (right) at 300 K from 156 GHz to 178 GHz. The phase-angle is modified to the zone of +180 to -180 degrees.

Table 1. Measured complex permittivity of HDPE at different temperatures.

	Dielectric constant	**Loss tangent (10^{-4})**
300 K	2.361	7.8
4 K	2.360	9.2

The measured transmittance and phase spectrum of 2 mm HDPE sheet by TDS has been presented in Figure. 5. The measured transmittance of HDPE is nearly 0.93 below 1 THz and decrease to 0.75 when the frequency increase to 3 THz. A rather elusive absorption band at 2.2 THz has also been observed. The dielectric constant has been calculated by the measured phase spectrum, good result has been obtained due to the accurate phase measurement. The measured dielectric constant is around 2.37 and is nearly constant in whole operating frequency range.

The transmittance of 2 mm HDPE sheet measured by FTS system was illustrated in Figure. 6. The transmittance decrease from 0.75 to 0.3 when the frequency increase from 3 THz to 15 THz.

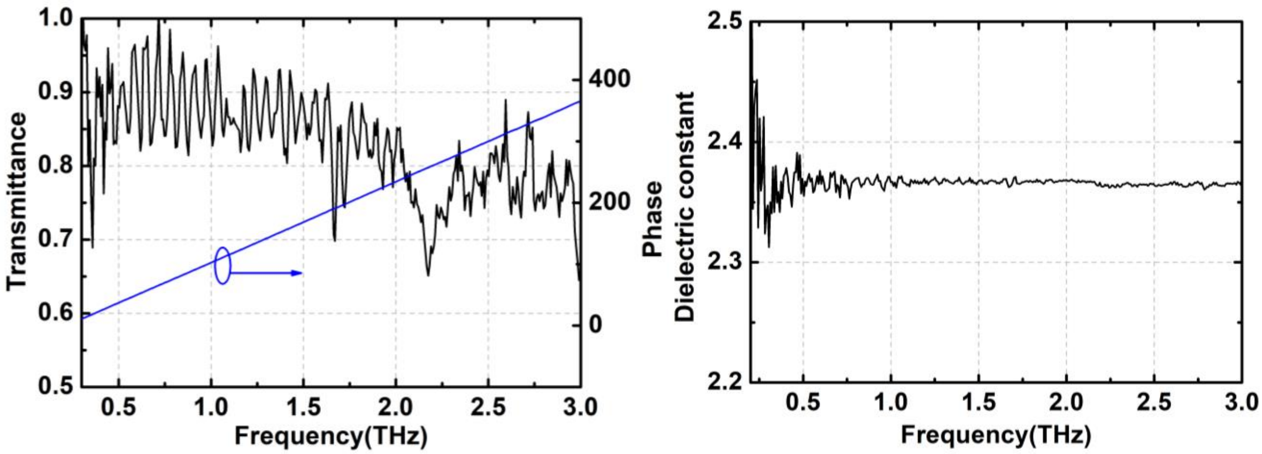

Figure 5. Measured transmittance and phase spectrum of HDPE by TDS from 0.2 THz to 3 THz (left); Calculated dielectric constant from 0.2 THz to 3 THz (right).

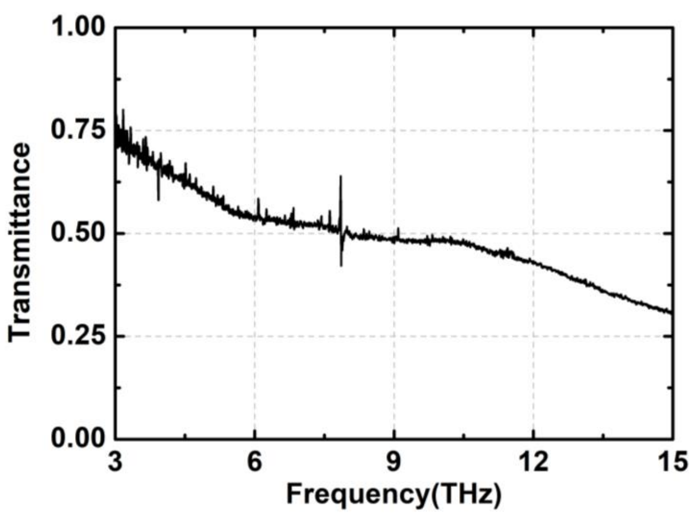

Figure 6. Measured transmittance of HDPE by FTS from 3 THz to 15 THz

4. CONCLUSION

In this paper, the dielectric property of HDPE has been characterized precisely in a wide frequency range. Three kinds of measurement methods are employed to cover the whole frequency range. A vector network analyzer (VNA) combined with a quasi-optical transmissometer has been used to measure the transmittance and dielectric constant of HDPE from 0.16 THz to 0.18 THz at 300 K and 4 K. The calculated dielectric constant and loss tangent at 300 K are 2.361 and $7.8*10^{-4}$ respectively, they change little when the temperature decreases to 4 K.A Time Domain Spectrometer (TDS) is employed to cover the frequency range from 0.2 THz to 3 THz since the VNA can't work upon 1 THz. The measured transmittance of HDPE is nearly 0.93 below 1 THz and decrease to 0.75 when the frequency increase to 3 THz. A rather elusive absorption band at 2.2 THz has also been observed. The dielectric constant of HDPE measured by VNA and TDS shows a frequency independency from 0.16 THz to 3 THz. A Fourier Transform Spectroscopy (FTS) has been used for the measurement from 3 THz to 15 THz since the TDS can't achieve broad band and fast scan speed. The measured transmittance decreases from 0.75 to 0.3 when the frequency increase from 3 THz to 15 THz.

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REFERENCES

[1] P. Goy, M. Gross, S. Caroopen, J. Mallat, J. Tuovinen, A. Maestrini, G. Annino, M. Fittipaldi, and M. Martinelli, "Millimeter - submillimeter vector measurements in free space, and in resonant structures. Application to dielectrics characterisation," Proceedings of Material Research Society, Spring 2000 Meeting, San Francisco.

[2] Grüner, George, and Claus Dahl, "Millimeter and submillimeter wave spectroscopy of solids," Vol. 200. Berlin: Springer, 1998.

[3] Griffiths, Peter R., and James A. De Haseth, "Fourier transform infrared spectrometry," Vol. 171. John Wiley & Sons, 2007.

[4] M. C. Beard, G. M. Turner, and C. A. Schmuttenmaer, "Terahertz spectroscopy," J. Phys. Chem B, vol. 106, pp. 7146–7159, 2002.

[5] M. Hangyo, T. Nagashima, and S. Nashima, "Spectroscopy by pulsed terahertz radiation," Meas. Sci. Technol., vol. 13, pp. 1727–1738, 2002.

[6] Sumner P.Davis, "Fourier Transform Spectrometry", Academic Press , 2001.