Terahertz time domain spectroscopy for non-destructive testing of plastic parts

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Abstract. Dielectric materials, including most plastics, are transparent for electromagnetic radiation in the Terahertz (THz) spectral range. This offers great potential for novel non-destructive testing (NDT) methods for plastic parts. Here we present spatially-resolved measurements on different plastic parts using THz time domain spectroscopy (TDS) in reflection arrangement.

Based on weight optimization, more and more metal parts are substituted by plastics. In order to ensure the functionality and quality, suitable test methods are needed. In previous works it has already been demonstrated that THz TDS is a powerful tool for wall thickness measurements, moisture and filler content determination as well as to distinguish between different sorts of plastics [1]. However, all these measurements were performed in transmission arrangement for a high signal-to-noise ratio (SNR). Hence, this requires a two-side access to the sample which is often not feasible for practical applications in industrial environment.

Now we are using a reflection arrangement for the inspection of plastic parts with THz TDS as the next step towards increased practical relevance. First, we have designed and implemented an experimental test setup for spatially resolved imaging under dry air condition. This is crucial to suppress water absorption for getting a good SNR. Next, specially adapted algorithms were developed for data evaluation. For example, this allows the determination of water content without knowing the sample thickness. Furthermore, we have determined the fiber orientation in short glass fiber reinforced injection molded parts based on polarization sensitive measurements. We have also measured the layer thickness as well as the filler content.

In summary, we used THz TDS technology in reflection arrangement for the inspection of plastic parts with only one-sided sample access to determine a variety of material properties.

Introduction

Electromagnetic radiation with THz frequencies is spectrally located between microwaves and infrared radiation. Commonly, the THz range is specified between 100 GHz to 10 THz, corresponding to wavelengths of 3 mm to 30 µm. Unfortunately, there was a lack of powerful, feasible and coherent sources and detectors for a long time. Only the technological progress of the last decades enabled appropriate devices [2]. Today, various types of THz systems are established on the market and the technology is currently
acquiring more and more industrial applications [3-5]. On the one hand, all-electronic THz systems based on microwave technology enter the THz range from low frequencies. On the other hand optical THz systems using laser radiation for the generation of THz radiation penetrate from the high frequency range mostly in combination with semiconductor antenna modules [6]. Advantage of optical systems is the generation of frequency in a wide range between 30 GHz up to 40 THz [7].

THz radiation is very versatile and used in many different fields. Most dielectric materials and therefore many types of plastic can be well penetrated by THz waves [3]. Mainly plastics with non-polar bindings such as polyethylene (PE) or polypropylene (PP) are characterized by low absorption or good transparency [8-11]. In contrast, polyamides (PA) absorb THz radiation very strongly [12]. Some other materials like electrical conductors cannot be penetrated. As a consequence of the high transmissivity of most polymers, THz is a powerful tool for the NDT of plastic parts [13]. But water molecules have a strong absorbing effect on THz waves due to their dipole structure with rotational vibrational transitions in the THz region [14]. Especially at frequencies above 1 THz, the absorption due to humidity in air occurs more apparent. As a consequence, measurements in the laboratory are typically performed under dry air or nitrogen atmosphere. Liquid water acts strongly absorbing and reflecting.

THz technology has some decisive advantages compared to other NDT methods. In contrast to X-rays there is no ionizing effect on organic matter due to the low photon energy of only several meV. This means that no special safety precautions must be considered working with THz systems. Compared to ultrasonic testing, THz radiation has electromagnetic nature and can be well transmitted through air without the need of a special medium. Furthermore, also air-filled samples like plastic foams can be investigated without appreciable damping losses.

**Experimental**

**THz System**

All measurements shown here have been carried out with an optical-pulsed THz time domain spectrometer Tera K15 of Menlo Systems GmbH, Martinsried (Germany). This system uses a pulsed femtosecond fiber laser with a wavelength of 1550 nm to stimulate photoconductive switches for the generation and detection of broadband THz-pulses. The photoconductive antennas are based on a low-temperature grown InGaAs/InAlAs semiconductor structure equipped with a fiber connection for easy handling [15].

The laser beam path of the whole system is completely encapsulated in polarization-maintaining optical fibers. Only the mechanical delay unit, which is encapsulated itself, has a free laser beam path inside which is, however, not easily accessible. The mechanical delay line scans the THz pulses in a time range up to 285 ps with a sampling rate of 2 Hz. In comparison to systems with free laser beam, complex alignment procedures are avoided. The fiber coupling allows a high flexibility in the THz beam path. In particular, the antenna module along the optical axis can be rotated very easily, which was used for polarization dependent measurements with different directions.

**THz beam setup**

Almost all THz TDS systems only have a single pixel transmitter and receiver, respectively. Performing spatially-resolved measurements, a setup was established to test different plastic components in reflection arrangement. In order to get a perpendicular incident of the radiation for measurements in 0° reflection arrangement, a 3.5 mm thick
silicon wafer was used as THz beam splitter (Figure 1). The THz beam emitted by the transmitter is collimated by a plano-convex lens made out of polymethylpentene (PMP), and focused by a second lens on the sample. The beam width in the focal plane depends on the frequency and measures 0.9 mm between 300 to 400 GHz. The reflected radiation is collimated by the lens and is directed via the beam splitter on the detector antenna with a third lens. The sample is mounted on a 2D x/y linear raster scan unit in order to realize spatially-resolved characterization.

To avoid absorption due to humidity, the whole test stand is enclosed and flushed with dry air (dew point < -50 °C). Polarization dependent measurements were realized by rotating the linearly polarized antenna modules along their optical axis. Thus, it is possible to vary the angle between the emitted and detected linearly polarized electric field.

**Data Analysis**

*Evaluation in time domain*

A schematic diagram of a reflected THz signal in time domain is shown in Figure 2. Two pulses are derived from the reflection at the front (pulse 1) and rear surface (pulse 2) of the sample.

The pulse energy $E$ can be calculated from the measured amplitude in time domain $A(t)$ by

$$ E = \int A(t)^2 \, dt. \quad (1) $$
The integration limits are set to the respective pulse. The absorption $\alpha$ is defined by the ratio of the pulse energies

$$\alpha = -\frac{1}{2w} \ln \left( \frac{E_{\text{pulse}_2}}{E_{\text{pulse}_1}} \cdot \frac{1}{(1-R)^2} \right).$$  \hspace{2cm} (2)

where $R$ is the reflectance calculated by the Fresnel equations and $w$ the sample thickness. The group delay can be determined by

$$t_G = \int A(t)^2 \cdot dt \cdot \frac{c}{E_{\text{pulse}}},$$  \hspace{2cm} (3)

The refractive index $n$ can be calculated from the difference in group delay and the speed of light $c$.

$$n = \frac{(t_{G,\text{pulse}_2} - t_{G,\text{pulse}_1}) \cdot c}{2w}.$$  \hspace{2cm} (4)

**Thickness correction for moisture measurements**

The knowledge of the layer thickness is crucial to calculate the correct values of the material parameters. But in industrial applications ideally plano-parallel surfaces are very uncommon. Usually the layer thickness varies with different positions. Therefore, a correction thickness algorithm was developed. Considering the variation of moisture in polymers, the change in refractive index is negligible compared to the change in the absorption. Hence, the layer thickness $w$ can be calculated from the difference in group delay $\Delta t_G$, the speed of light $c$ and refractive index $n$:

$$w = \frac{\Delta t_G \cdot c}{2n}.$$  \hspace{2cm} (5)

This allows the correct determination of spatially-resolved absorption based on the data evaluation of the THz pulse train in each pixel together with the calculated thickness. With the help of calibration data the moisture content can be determined spatially-resolved.

**Analysis in the frequency domain**

Time domain analysis, as described above, provides average material parameters in the entire frequency band in a simple and fast way. However, an evaluation by means of Fast Fourier Transform (FFT) is used to achieve additional spectral information [16]. First, each of the two pulses, pulse 1 and pulse 2 in Figure 2, is windowed with a Blackman filter function in time domain. After FFT in frequency domain the ratio of spectrum’s amplitudes relate to the absorbance for a known sample thickness. The phase information together with the sample thickness provides the refractive index.

**Determination of fiber orientation**

A spatial order of short glass fibers in a plastic matrix results in an optical anisotropy of the material. This fiber orientation can be determined by measuring the refractive index under different polarization angles [17]. If the THz polarization is parallel or perpendicular relative to the fiber orientation, the refractive index has a maximum or minimum value,
respectively. An ellipsoid can be spanned by measuring at least three different polarization angles (Figure 3). The orientation of the resultant major axis is referred to the preferred orientation of the fibers.

![Figure 3: Schematic drawing of the orientation ellipsoid](image)

Here we have measured three angle-dependent refractive indices \(n(0°), n(45°)\) and \(n(90°)\) to define the ellipse. The angle \(\gamma\) between the major axis of the ellipse and the 0° direction represents the fiber orientation (Figure 3). It has to be noted that the layer thickness of the part is not required, since only the relative variation of the refractive index is evaluated. The group delay time difference could be used alternatively instead of the refractive index.

**Results**

**Filler content**

The quantitative determination of the filler content of plastic compounds is based on the linear relationship between volumetric filler content and refractive index in THz frequency range [14]. First, measurements on samples with defined filler content have been performed in transmission geometry for calibration purposes. To demonstrate the potential of THz technology, components with locally differing filler content were made out of different compounded granules with by compression molding. The samples were mechanically reworked to ensure a homogeneous layer thickness and smooth surfaces.

Figure 4 a) shows a photo of a component filled with 16 wt.-% calcium carbonate (\(\text{CaCO}_3\)) including internal letters with a different filler content of 26 wt.-% than the surrounding material. Figure 4 b) shows the phase image calculated from the group delay difference of the pulses. It is clearly seen that the group delay in the area of the letters, since a higher filler content, results in a higher pulse delay difference. Figure 4 c) depicts the ratio of the pulse energies of the front and back reflection. As a consequence of the discontinuities between letters and matrix of the sample diffraction effects occur. The spatially-resolved filler content (Figure 4 d) was calculated using the calibration data and refractive index. The calculated values correlate very well with the nominal values. The apparently lower filler content in the peripheral areas is originated from a still deviation of the component thickness.
Water strongly absorbs THz radiation. This effect is exploited for moisture detection in plastic components. Again, also calibration measurements are necessary for a quantitative specification. Therefore, reference components with defined geometry were used with defined moisture content by soaking water, whereat the moisture content was determined gravimetrically. The calibration measurements were performed in transmission arrangement under dry air atmosphere to achieve a high SNR.

Spatially resolved THz images were evaluated pixel by pixel in time domain including the algorithm for thickness correction described above. To verify the thickness correction algorithm a completely dried PA sample with three different layer thicknesses was characterized, shown in Figure 5 a). It can be clearly seen, that each of the three thicknesses (1, 2 and 4 mm) does not have any moisture content and the thickness correction is working correctly. For each thickness, the absorption and therefore the moisture content were calculated correctly. The deviations at the edges result from diffraction effects.

Figure 5 b) shows the moisture content of another PA 6 component with two different thicknesses (1 mm and 2 mm) which has been partially immersed into water. It
was found that even in the regions which were above the water level also moisture was detected. This is due to migration effects of water over time inside the plastic part. Furthermore, in the thin area (1 mm) the highest water content was measured. In contrast, the thicker part (2 mm) has lower moisture content since the water has to penetrate through almost identical size of surface in a larger volume.

Fiber orientation

Figure 6 a) and b) show the calculated fiber orientation in a false color plot and as orientation vectors for two injection molded PP samples with a thickness of 4 mm and filler content of 30 wt.-% short glass fibers. The direction of mold injection is indicated with an arrow above. Sample a) has an additional obstacle within the melt flow to guide the melt front. The THz measurements were performed under three different polarization angles in reflection arrangement. For the calculation of fiber orientation the concept of the orientation ellipsoid was applied pixel by pixel.

![Figure 6](image.png)

**Figure 6:** Determination of the preferred fiber orientation on an injection molded PP component with 30 wt. % short glass fibers. a) b) e) f) µCT images have been additionally taken to verify the THz measurements.

In case of the component with melt flow obstacle, a complex orientation of the fibers can be observed. Due to the obstacle two melt fronts are formed which recombine again behind the barrier.

Verification measurements were done by X-ray micro computed tomography (µCT) to review the results (Figure 6 left and right). The µCT images are taken from a depth plane centered in the cross section of the component. The results of the µCT measurements confirm the results of the THz-imaging. However, it has to be noted that the THz measurements give an integrated value over the thickness cross-section, whereas µCT allows the investigation in any depth layer.

**Summary and Outlook**

THz-TDS imaging in reflection arrangement has great potential for NDT of plastic components since only a one-sided access to the sample is needed. But, compared to measurements in transmission arrangement, a lower SNR as well as a strong dependence of the measured signal on the incident angle and surface inhomogeneity occurs. Therefore, special algorithms for data analysis are required.
Here we have successfully demonstrated different analysis methods for the evaluation of measurement data in reflection geometry. THz imaging in 0°-reflection arrangement allows the inspection of plastic components regarding filler and moisture content as well as the fiber orientation. With the help of special algorithms, it is possible to perform a simultaneous spatially-resolved thickness measurement and moisture determination. For a quantitative determination of filler content, the knowledge of the layer thickness is required.

Measurements in reflection arrangement are an important step towards novel industrials applications of this technology. In the vast number of possible measurement scenarios a two-sided sample access is hard to realize.

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