

# Non-Contact Thickness Measurements with Terahertz Pulses

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**Abstract.** Via time-of-flight techniques, pulsed terahertz radiation determines the thickness of polymeric materials or paint layers, even in multi-layered samples. In reflection-mode measurements, terahertz pulses strike the object under test, each layer interface reflects a part of the incident pulse, and the time elapsed between the arrivals of pulse "echoes" from either side is directly proportional to the optical thickness of that layer.

We present two different time-domain terahertz systems designed for noncontact thickness measurements. Both systems employ industry-grade 1.5  $\mu$ m fiber laser technology and photoconductive antennas made of InGaAs, which convert the laser pulses into terahertz radiation with an industry-record average power of more than 50  $\mu$ W.

The first design employs a voice-coil delay stage in order to readout the terahertz pulses. The system achieves a measurement speed of up to 50 pulse traces or thickness values per second. Owing to the high precision of the delay stage, successive pulse traces can be averaged in order to reduce the noise level. This results in an excellent dynamic range and an ultrabroad bandwidth of 6 THz, and enables thickness measurements of layers down to 20  $\mu$ m with an accuracy on the order of 1  $\mu$ m.

The second setup is optimized with respect to measurement speed. The mechanical delay is eliminated completely and replaced by a technique dubbed ECOPS (Electronically Controlled Optical Sampling). To that end, two (rather than one) femtosecond lasers are employed and the pulse train of the "slave" laser is phase-locked to that of the "master" laser. The repetition rate of the "slave" is however varied slightly with respect to that of the "master", creating the time delay required for signal readout. The ECOPS system achieves measurement rates up to 10 kHz and therefore lends itself to "ultrafast" inspection tasks, e.g. to monitoring of samples on fast conveyor belts, in particular if a parameter mapping with high spatial resolution ("100% inspection") is desired.

## 1. Introduction

Contact-free thickness measurements play an increasingly important role in industrial process monitoring and quality control. Extruded polymers – e.g., as used in production lines of plastic bottles or pipes – often require a minimum wall thickness for mechanical or chemical stability, yet material costs increase significantly if the layers become too thick. In



automotive manufacturing, the exact thickness of paint and coating layers plays a crucial role not only for the overall appearance, but also for protection against UV radiation and corrosion. In pharmaceuticals, the coating thickness governs the release of the drug inside the body, with consequences on the efficacy and potential side effects. In chip production, polymer coatings act both as protection against moisture and dust and as stress absorbers, and a high homogeneity of the surface is required as well.

Over the past decades, a plurality of techniques for thickness assessments has been developed, including ultrasonic testing, x-ray CT, thermography, magnetic gauges and eddy-current measurements. Yet they all face challenges: either in terms of radiation hazards, or contact media required, or they are limited with respect to measurement speed and depth resolution, or they fail entirely when used with multi-layered systems. In an ideal setting, the measurement technique employed should be intrinsically safe, fast (i.e., provide few 10 .. few 1000 thickness readings per second), accurate to the micrometer level, and capable of resolving layers as thin as ~10  $\mu$ m, even in multi-layered samples.

A comparatively new technology involves short, broadband terahertz pulses. The "terahertz range" denotes electromagnetic radiation at frequencies between 100 GHz and 10 THz, or wavelengths between 3 mm and 30  $\mu$ m. Terahertz waves penetrate a variety of materials, including many plastics, textiles and paper. Pulsed, or time-domain (TD), terahertz systems can thus probe the thickness of polymeric materials or paint layers in a contact-free manner: Using reflection-mode optics, terahertz pulses illuminate the sample under test, and each interface generates a pulse "echo" by reflecting a fraction of the original pulse. If the optical properties of the material are known, time-of-flight measurements of the pulse echoes provide information on the thickness of the individual layers [1].

In recent years, TD-terahertz instrumentation has grown increasingly mature, having come a long way from bulky, cumbersome lab setups to flexible, high-performance systems that are about to make their way into "real-world" industrial applications. Of a variety of different terahertz generation and detection methods, photoconductive switches driven by femtosecond lasers have found particularly widespread use [2]. Today's terahertz platforms combine mature and cost-efficient telecom components on the laser side with fiber-coupled photoconductive switches that either generate or detect the terahertz pulses. In 2014, the authors already presented a TD-terahertz spectrometer with a peak dynamic range of 90 dB and a bandwidth of more than 4.5 THz [3], and demonstrated the suitability of the system for material analysis as well as terahertz imaging.

In this work, we report on recent progress accomplished with this system: owing to a new generation of photoconductive antennas, the attainable bandwidth now exceeds 6 THz. We apply the system to spectroscopic measurements, and examine the thickness of a bottle made of high-density polyethylene (HDPE). In addition, we present an alternative TD-terahertz technique that achieves significantly higher measurement speeds: Employing a technique dubbed "ECOPS" (Electronically Controlled Optical Sampling) [4, 5], we overcome the velocity limitations of conventional TD-terahertz assemblies and increase the measurement speed by more than two orders of magnitude.

#### 2. Instrumentation

#### 2.1 General Considerations

In a conventional TD-terahertz setup, the output of a femtosecond laser is split in two parts. One pulse travels to the transmitter and is converted into terahertz radiation. The terahertz pulse interacts with a sample, where it is transmitted or reflected, and finally arrives at the receiver. For spectroscopic measurements, one usually prefers transmission-mode geometries, whereas thickness measurements are more conveniently carried out in a reflection setting.

At the receiver, the incident terahertz pulse is sampled with a time-shifted copy of the laser pulse. This pump-probe concept requires a time delay. Whilst most of the "classical" TD-terahertz systems employ mechanical delay stages, the novelty of the ECOPS approach is a purely electronic delay scheme: Emitter and receiver are pumped by separate, but synchronized short-pulse lasers.

## 2.2 TD-Terahertz System with Mechanical Delay

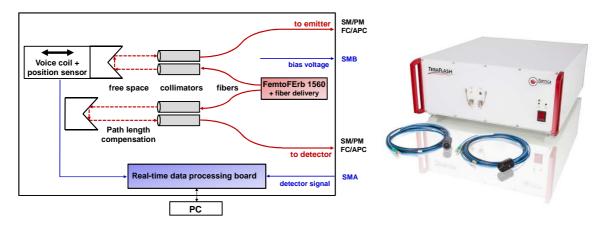


Fig. 1. Schematic diagram (left) and photograph (right) of the TeraFlash system. Red lines denote optical signal paths, blue lines represent electric connections.

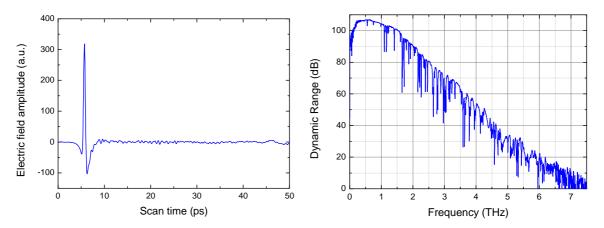
We have realized a compact, fiber-coupled TD-terahertz system (product name "TeraFlash"; for a detailed technical description cf. [3]). The instrument makes use of 1.5  $\mu$ m fiber laser technology and InGaAs/InAlAs photoconductive switches [6-9]. Due to a precise reconstruction of the time axis, the system drastically reduces jitter noise, which results in a high dynamic range and broad bandwidth.

Fig. 1 shows a sketch and a photograph of the TeraFlash. The entire system has a size of 180 x 450 x 560 mm (H x W x D). The built-in laser (FemtoFErb THz FD6.5, Toptica Photonics) emits pulses with a halfwidth of <60 fs, a repetition rate of 100 MHz, an average output power of 80 mW, and a center wavelength of 1.55  $\mu$ m. The pulses are delivered to the photoconductive antennas via a single-mode, polarization-maintaining fiber, with a 50 / 50 fiber splitter dividing the output into an emitter and a detector branch. The fiber assembly further includes dispersion-compensating means in order to prevent temporal broadening of the laser pulse, which would else reduce the quality of the terahertz signal.

Both the transmitter and the receiver paths feature fiber-coupled mechanical delay stages: the receiver path includes a slow, long-travel delay and the emitter path a fast, scanning delay. The long-travel delay introduces a constant timing offset and thus enables a flexible change of the terahertz path length: the two antennas can be separated by any distance between 15 cm and 110 cm.

The scanning delay consists of a voice-coil-driven corner-cube mirror combined with a digital position sensor. The sensor records 50000 time stamps per second with a resolution of 1.3 fs. Data acquisition is accomplished both during the forward and the backward movement of the mirror, which minimizes the "dead time" of the system. The precise timing resolution gives rise to the high accuracy of the time scale, which results in a superior dynamic range when multiple traces are averaged.

The photoconductive antennas were designed and assembled at Fraunhofer Heinrich-Hertz-Institute, Berlin. The emitter employes a high-mobility InAlAs/InGaAs multilayer heterostructure (MLHS) as described in [6-9]. The receiver antennas are based on LT-grown and Beryllium-doped InAlAs/InGaAs MLHS. A strip-line antenna geometry with a 100  $\mu$ m photoconductive gap was chosen for the emitter, and a dipole geometry with 10  $\mu$ m gap for the receiver. The average terahertz output amounts to 50-100  $\mu$ W [10].



**Fig. 2.** Left: Pulse trace of an InGaAs photoconductive switch. Right: TeraFlash spectrum. The peak dynamic range exceeds 100 dB, in a measurement time below 1 min. The dips are absorption lines of water vapour.

Fig. 2 depicts a typical time trace of a terahertz pulse, and the corresponding spectrum. A single trace of 50 ps can be acquired in only 44 ms. Via the control software, the sampling time can be flexibly adjusted between 20 ps and 200 ps. The software also allows for averaging multiple time traces, in order to reduce the noise level of the measurement. The peak dynamic range (PDR) of the spectrum, defined as the ratio between the maximum signal at approx. 0.5 THz and the noise floor, amounts to approx. 70 dB for a "single-shot" measurement. A factor of 10 in the number of averages increases the PDR by 10 dB. With 1000 averages, i.e. within a measurement time still well below one minute, the PDR exceeds 100 dB and the bandwidth becomes greater than 6 THz (Fig. 2, right hand side). To the best of our knowledge, these values represent a record for commercial TD-terahertz systems.

This ultrabroad bandwidth enables thickness measurements of very thin layers: depending on the material properties, layer thicknesses of 10-20 µm can still be resolved.

#### 2.2 TD-Terahertz System with Electronic Delay

System performance notwithstanding, the mechanical delay presents a bottleneck in terms of measurement speed. This becomes particularly relevant for rapidly moving samples (conveyor belts, papermaking machines, extrusion lines), in particular if measurements with high spatial resolution ("100% inspection") are required: In this case, a data rate of a few 10 Hz turns out to be insufficient. To give an example: if a sample moves at 10 m/s, and measurements with a spatial resolution of 1 mm are required, then the data rate needs to be as high as 10 kHz. This is no longer possible with a mechanical delay, due to the inertia of the moving masses involved.

A fast and flexible alternative is a system based on ECOPS [4], a scheme that employs two femtosecond lasers rather than just one, thus eliminating the mechanical delay altogether. In this concept, sketched in Fig. 3, the terahertz transmitter and antenna are driven by a separate laser each. In order to vary the time-of-arrival of the "readout" laser pulses at the receiver, the cavity length – and consequently, repetition rate – of one of the lasers is modulated with the help of a piezo crystal. To this end, one of the laser cavities (laser #1, "Master") features a fixed cavity length, whereas the other laser (laser #2, "Slave") includes a very short free-space path, where the light is coupled out of the fiber oscillator and directed onto a mirror attached to the piezo. By means of a fast feedback loop that acts on the cavity piezo, the repetition rate of the "slave" laser is phase-locked to that of the "master". An additional sinusoidal modulation varies the phase difference between the two laser frequencies, and therefore, the pulse train of laser #2 is periodically accelerated or delayed. The net effect is equal to that of a conventional, mechanical delay stage, yet the modulation frequency is hundreds of times faster.

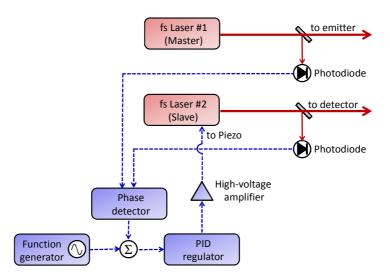
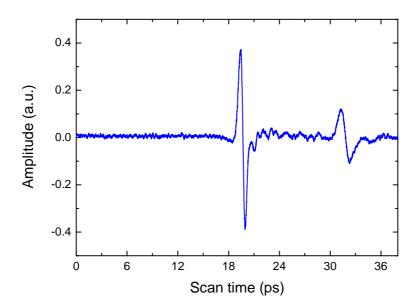


Fig. 3. Sketch of the ECOPS setup. Red: optical signals, blue: electric signals.



**Fig. 4.** "ECOPS" time trace of a terahertz pulse transmitted through a Silicon wafer. The first peak results from the terahertz pulse that travelled directly across the wafer and the second pulse arises from a double-reflection at the two surfaces, i.e. the pulse crossed the wafer three times. The acquisition time for the entire pulse trace was 100 µs.

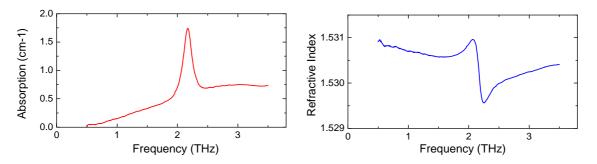


Fig. 5. Absorption coefficient (left) and refractive index (right) of a HDPE sample.

In a proof-of-principle study of the ECOPS technology [5], some of us demonstrated scan ranges of 180 ps and 20 ps at 2 kHz and 8 kHz speed, respectively. Using an improved setup, we meanwhile recorded traces of 38 ps length at data rates of 10,000 traces per second. Fig. 4 shows the time trace of a pulse transmitted through a silicon wafer (refractive index  $n \sim 3.42$ ): The time interval of approx. 12 ps between the two pulses corresponds to a wafer thickness of 525 µm, in excellent agreement with a mechanical thickness reading.

#### **3.** Application Examples

#### 3.1 Spectroscopy

In this section, we demonstrate the suitability of the TeraFlash system for spectroscopic measurements, thickness measurements and terahertz imaging.

Fig. 5 shows the absorption coefficient and refractive index of a disc of high-density polyethylene (HDPE). The measurement was carried out in an enclosure purged with dry air, in order to eliminate any spurious water vapour signatures in the terahertz spectra.

The prominent absorption line at 2.2 THz is clearly seen, both in the absorption coefficient and in the dispersive shape of the refractive-index spectrum.

Practical applications of spectroscopic measurements are manifold. In polymers, the variation of the refractive index with temperature helps uncover minute structural changes [11]. For fiber-reinforced plastics, the refractive index yields information on the orientation of the fiber strands [11]. Terahertz spectroscopy provides insights into the molecular dynamics of liquid crystals [12], and reveals semiconductor parameters such as conductivity and carrier density [13].

#### 3.2 Wall Thickness

Fig. 6 depicts pulse traces obtained with a bottle made of HDPE. The amplitude of the main lobe of the reflected terahertz pulses is negative at the first interface (air – HDPE) and positive at the second interface (HDPE – air). The negative and the positive peak maxima are separated by 4.35 ps (blue trace, see dashed lines and arrow) and 8.60 ps (red trace). The wall thickness is thus 426  $\mu$ m and 843  $\mu$ m, respectively. In other words, the wall thickness of this particular bottle varies by as much as a factor of 2 at different locations.

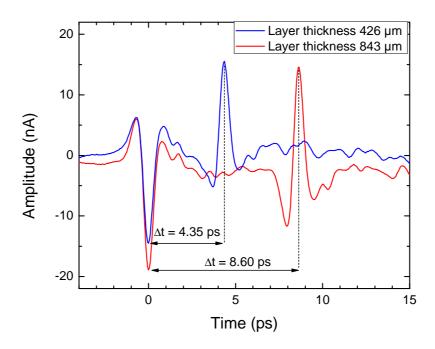


Fig. 6. Wall thickness measurements on two points of the same HDPE bottle.

# 3.3 Imaging

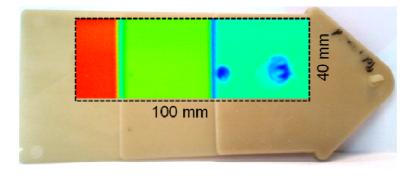


Fig. 7. Photograph and terahertz image overlay of a step-wedge made of polyamide.

Figure 7 shows a photograph of a polyamide step-wedge and a 100 mm x 40 mm terahertz overlay. The image was acquired in a pixel-by-pixel fashion, with the sample being scanned across the focus of the terahertz beam. The scan speed was 2 mm / s, limited by the velocity of the x/y translation stages. The step size was 0.3 mm and the total measurement time was 65 min. The transmitted signal intensity is shown in false colors; red and blue regions denote sample areas of high and low transmission, respectively. The transmission thus scales inversely with the thickness of the sample, but more interestingly, the terahertz image also reveals the presence of two air bubbles, which are not visible from the outside. Terahertz imaging can thus pinpoint voids, delaminations and sub-surface cracks in polymer samples.

## 4. Conclusion

We have designed and characterized two fiber-coupled time-domain terahertz time-domain systems, operating at an optical excitation wavelength of 1.5  $\mu$ m. Both instruments combine mature telecom technology and photoconductive switches based on InAlAs/InGaAs multi-layer heterostructures. The first system ("TeraFlash") employs a precise, voice-coil driven mechanical delay stage. It achieves a bandwidth of >6 THz and a peak dynamic range of 100 dB. The TeraFlash thus lends itself to broadband spectroscopic measurements, as well as the characterisation of thin layers, where thickness values of 10-20  $\mu$ m can still be quantified. The second setup ("ECOPS") is optimised with respect to measurement speed: the mechanical delay stage is replaced by an electronic concept, where two lasers are synchronised to one another, and the repetition rate of one of the lasers is modulated across a small range with the help of a piezo crystal. The ECOPS technique achieves data rates of up to 10,000 pulse traces per second and is the method of choice for rapidly moving samples, or for measurement tasks that require a high spatial resolution, e.g. the 100%-inspection of extruded plastic materials.

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