Sensitive Determination of Layer Thickness by Waveguide Terahertz Time-Domain Spectroscopy

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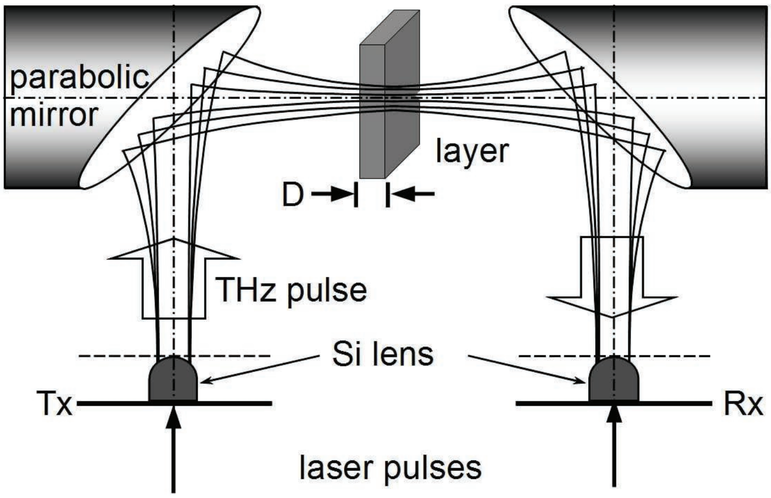

Figure 1: Terahertz time-domain spectroscopy system (THz-TDS)

Thin film sensing and layer thickness determination are important for various industrial processes. Non-destructive testing procedures are needed to control, for example, production parameters, like the drying time or the amount of applied substances. Also in final inspection precise sensors are needed to determine coating thicknesses. The terahertz (THz) frequency band (located between 100 GHz and 10 THz, corresponding to wavelengths between 3000 µm and 30 µm in free-space) efficiently penetrates most dielectrics. Thus, THz non-destructive testing can be used to measure the thickness of common plastics and paint layers in a contact-free way [1]. Located in the electromagnetic spectrum between the microwaves and the infrared, THz radiation is completely harmless as it is non-ionizing. No safety measures have to be taken when applying THz waves.

Unlike standard incoherent systems, where only the intensity attenuation can be measured, THz time-domain spectroscopy (THz-TDS) [2] can measure the time-resolved amplitude of the electric field. So also the phase information is accessible, which is a measure for the pulse delay. A standard THz-TDS system is shown in Fig. 1. It consists of a femtosecond laser source, which emits pulses in the near infrared. These pulses are used to gate a transmitter (Tx) and receiver (Rx), respectively. Comparable to a pump-probe experiment, the THz electric field can be sampled, if the relative temporal delay is shifted between pump and detector laser pulse. The optical path of the THz wave begins at the transmitter, which is a semiconductor material with a low carrier lifetime (lt-GaAs). An attached silicon lens collects the THz radiation. A pair of parabolic mirrors forms a frequency-dependent beamwaist of 1 cm diameter. The receiver (also lt-GaAs) produces a current, if the laser pulse and the THz pulse are overlapping in time. This current is directly proportional to the THz electric field.

If now a sample is put in the THz beam path, the changes due to absorption, dispersion or optical thickness can be measured by comparing the scan with inserted sample to a reference scan. As we are interested in thickness information, the pulse delay is evaluated. This can either be the relative pulse delay of the peak maximum or the zero crossing of the electric field. The delay is connected to the geometrical thickness by the refractive index of the material in the THz range according to:

$$\Delta t = \Delta t_{sample} - \Delta t_{reference} = (n - 1)D/c$$

with t the delay time, n the refractive index, D the sample thickness and c the velocity of light. Typical indices of refraction are 1.52 for PE and 1.7 for PET.

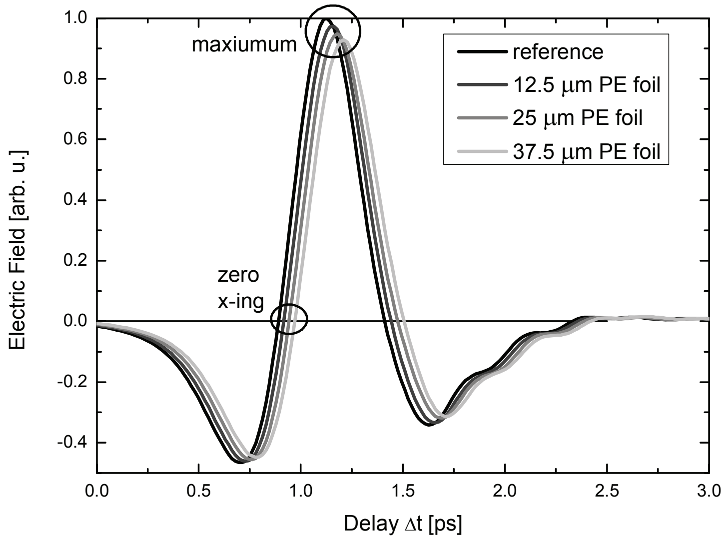

Figure 2: THz electric fields measured in transmission through PE foils of varying thickness.

Such a measurement working in single-pass transmission is presented in Fig. 2. It shows the pulses recorded with layers of increasing thicknesses in the beam. Clearly, a temporal shift of the pulses is seen along with a small reduction of peak amplitude. The shifts due to multiple layers (12.5 µm each) can already be observed by the eye. An evaluation of the individual scans is summarized in Fig. 3.

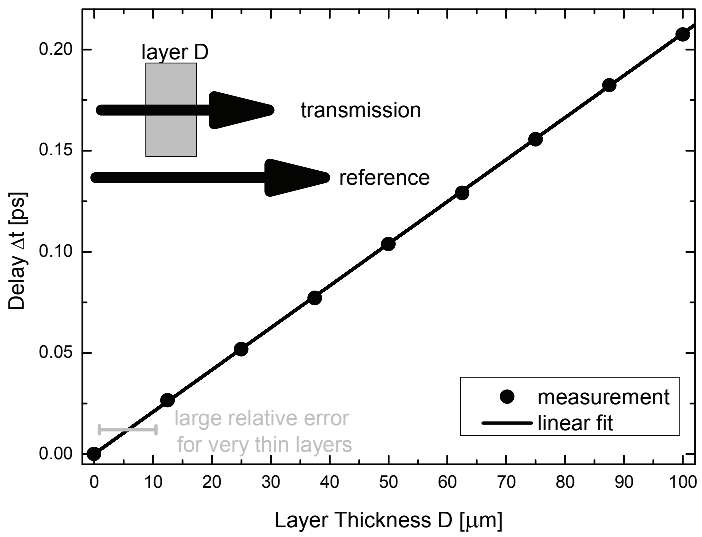

Figure 3: The pulse delay is proportional to the layer thickness D.

A linear behavior of delay as a function of thickness D is obtained, in good accordance to theory. But the measurement process is also subject to errors like the hysteresis of the mechanical delay line, thermal drift of optics and mounts. These drifts accumulate to a few microns, which result in a large error bar for measurements below 10 µm thickness. So in that case a standard single-pass transmission measurement does not give a reliable and stable measurement quantity for the layer thickness. Long-term stability prevents the precise measurement of very thin layers, if periodic reference scans cannot be implemented in the process. So to measure even thinner layers, we investigated waveguide geometries to enhance the sensitivity.

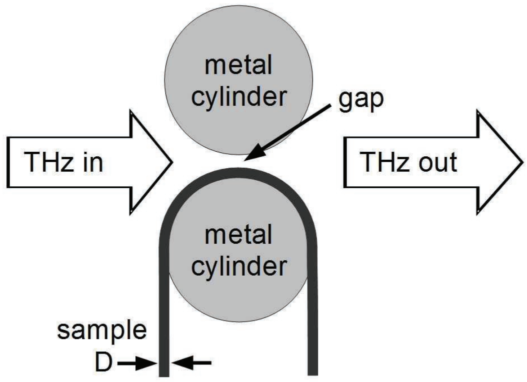

Figure 4: Sketch of the two-cylinder sensor used for layer thickness determination.

In the following, we will extend the scope of the THz technique from free-space propagation to waveguides, measuring thin coatings. The geometry uses dielectrically coated metal cylinders as sample mounts. The components of the two-cylinder waveguide sensor are shown in Fig. 4. The experimental layout operates as an exchangeable device for a standard THz-TDS system. The film under investigation is wrapped on the surface of one metal cylinder. Together with the opposing metal cylinder (both 63 mm diameter) the THz optics are formed with the coated and the uncoated cylinders mounted in direct contact or with a preset gap defined by spacers. This approach uses concepts of adiabatic THz wave compression and the advantages of THz waveguides [3].
Using the two-cylinder waveguide sensor, the approaching THz wave is spatially compressed from a wavelength dependent spot-size to a sub-wavelength line focus (less than 10 µm). After the THz wave propagates through the partially filled gap, the metal surfaces of the two facing cylinders continue to act like a horn, as the expanding wave is guided out of the sensor. The high efficiency coupling is due to the slowly varying beam pattern of the funnel-shaped geometry [4]. This adiabatic (which means slow with respect to the wavelength) behavior allow to compress the THz beam even to sub-wavelength spotsize without causing major losses or reflections. The overall coupling ratio remains sufficient which allows recording the transmission at a high signal-to-noise ratio.
In this arrangement the THz wave has a much longer propagation length within the film compared to the single-pass case, thereby giving rise to a considerably increased delay. The amount is given by the integrated filling factor, which is a quantity that gives a measure on the intensity fraction of the wave propagating in the film or in the air gap, respectively. The filling factor is the higher the closer the cylinders are and the more dense the wave is compressed. Even a small gap reduces the delay of the THz pulse by reducing the filling factor, especially in the strong interaction area of the gap.
The results of thin layers wrapped on top of one metal cylinder comprising the two-cylinder sensor are shown in Fig. 5. Even the thinnest available layers of BoPET with a thickness of 2.5 µm are clearly resolvable [5]. A considerable sensitivity increase of up to a factor of 150 is obtained for layers down to 2.5 micron thickness by applying the two-cylinder waveguide sensor. The results measured with the two-cylinder sensor are in good agreement to the calculated theory curves. In perfect contact (air gap 0 µm), theory predicts larger delays than observed, especially for thin layers. This discrepancy is caused by the surface roughness of the cylinders. Although the soft flexible plastic foil can further reduce the gap, due to the limited foil thickness, there are still small air spaces remaining. An averaged tiny gap of 2 µm in the order of the surface roughness shows good agreement with the measured values. A second series was recorded with spacers between the cylinders forming a 50 µm gap. These gaps are used to calculate the theoretical values of the delay, assuming a simple model using formulae known for partially filled parallel-plate waveguides. The delay is integrated from the initial compression through the intense interaction at the propagation through the gap and to the exit expansion. For the in-contact cylinders and the corresponding theory curve (gap 2 µm), the values match for thin layers, while for thick layers a transition into saturation can be observed. Working with a larger gap of 50 µm, theory predicts a nearly linear behavior of the delay as a function of coating thickness, which is also in good agreement with the measured values. This type of measurement can be used to obtain a simple calibration curve and good sensitivity at the same time. The sensitivity increase given by the ratio of measured stretched delay of the two-cylinder waveguide sensor to the theoretical single-pass delay reaches up to 150 for the 2.5 µm coating and the cylinders in contact, dropping to 66 for a 24.5 µm coating. In the case of a 50 µm gap, the pulse-delay factor is 74.1 fs/µm which corresponds to a sensitivity increase by a factor of 32.

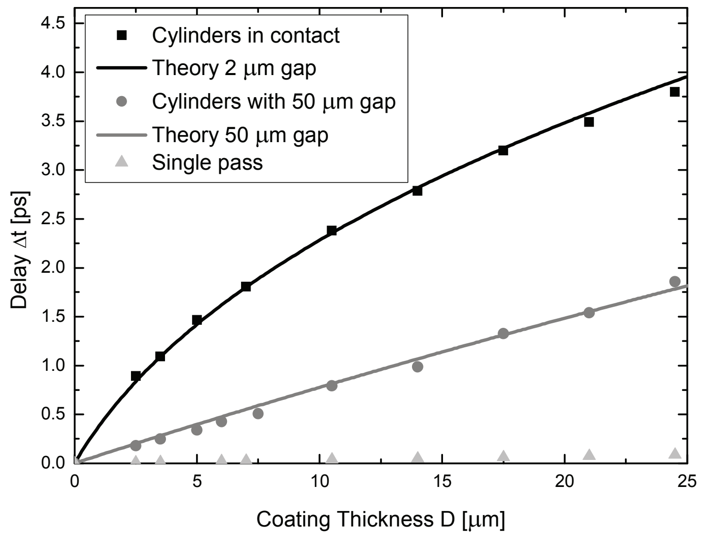

Figure 5: Sensitivity enhancement for thin film determination using the two-cylinder sensor.

Despite the presence of a gap, the caused delay applying the two-cylinder sensor is much higher for all layer thicknesses. The larger delay values minimize the relative errors of the technique. They are up-scaled from a fraction of a picosecond to multiple picoseconds. System drifts take less influence on the signals, increasing the long-term stability of the system.

As a further advantage, also the measurement of thin dielectric coatings on metallic foils is now possible, even if the perpendicular reflection cannot resolve the two originating echo pulses. These measurements would be performed by attaching the foil to the sample cylinder with the metallic side in direct contact with one cylinder. The chosen geometry of commercial metal cylinders is reproducible, easy to align, and does not require additional optical components. No tricky alignment of lenses is necessary. Also the sample changing process is fast. In an industrial application, the senor could be directly working attached to a deflector roll, guiding the product. Online production control even in harsh environments is possible by using fiber-based THz systems [6].

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