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# Analyzing sub-100-μm samples with transmission terahertz time domain spectroscopy

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ABSTRACT

The authors propose a method for the extraction of material parameter and thickness information from sub-100-μm thin samples using non-differential transmission terahertz time domain spectroscopy. The approach relies on an additional Fourier transform of the frequency dependent material parameters to a quasi space regime. In this quasi space, periodic Fabry–Perot oscillations from the frequency domain, which originate from multiple reflections inside the sample, correspond to discrete peaks. By iterative minimization of these peaks, the highly precise thickness information along with the refractive index and absorption coefficient of the sample can be determined. Experimental verification of the approach is also provided.

## 1. Introduction

Investigating thin samples with thicknesses well below 100 μm in a terahertz (THz) time domain spectroscopy (TDS) system usually requires a differential data acquisition technique [1,2]. Unfortunately, differential setups are strongly susceptible to misalignment errors and therefore an accurate mechanical alignment is essential. Furthermore an excellent thickness homogeneity of the substrate carrying the films which are characterized is vital. In addition a multiple modulation technique has to be utilized to improve the accuracy of the derived material parameters [3], which reduces the amplitude of the measured signals. Even in conjunction with modern data extraction algorithms [4–7], which consider multiple reflections within the sample, non-differential measurement schemes are still limited to the regime of thicknesses above 100 μm.

In this paper, we present a material parameter extraction and thickness determination algorithm for samples as thin as a few μm. The smallest determinable thickness is only restricted by the bandwidth of the employed TDS system. Note that since the uncertainty of the extracted parameters rises with decreasing spectral amplitude, the useable bandwidth is determined by the finite signal to noise ratio of the sample's measurement [8]. The algorithm makes use of the Fabry–Perot (FP) oscillations of both the measured transfer function and the resulting dielectric material parameters by applying an additional Fourier transform to the frequency domain data which yields information in a quasi space domain. In this quasi space (QS), the FP oscillations modulated on the frequency domain spectra appear as discrete peaks with a certain broadening induced by dispersion of the sample's material parameters as well as non-ideal surface conditions. By iteratively varying the assumed sample thickness and minimizing the peak amplitude in the quasi space, the real sample thickness, the absorption coefficient $\alpha$, and the refractive index $n$ can be determined, simultaneously.

The remainder of the paper is structured as follows: After a detailed discussion of the algorithm, we present experimental verification of the approach by analyzing thin samples of three well characterized materials: Firstly, a high resistivity silicon (HR Si) wafer with an approximate thickness of 54 μm. Secondly, a thin sheet of polyamide nylon 6 (PA6) with an approximate thickness of 25 μm and thirdly, to also demonstrate the suitability of the approach for relatively thick samples, a substrate of silica with an approximate thickness of 700 μm.

## 2. Algorithm

A key problem of measuring thin samples with electromagnetic radiation in transmission geometry, is the presence of FP oscillations. Thus, a standard data extraction, usually employed in TDS Systems [9], cannot be applied, because the resulting material parameters would be superimposed with a spectral modulation.

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Hence, more sophisticated, numerical analysis algorithms have been supposed, e.g. in [4–6], which we employ as basis for the algorithm introduced in this paper.

First, a theoretical transfer function of the sample, which depends on the refractive index $n$, the absorption coefficient $\alpha$, the samples thickness $L$ and the number of measured multiple reflections $M$, is derived. By assuming an approximate thickness $L_0$ and calculating the number of multiple reflections $M_0$ in a preprocessing step, considering the finite signal to noise ratio of the system, the values for $n$ and $\alpha$ of the sample can be computed by a numerical optimization, in which the theoretical transfer function is compared to the measured one. Although this procedure results in material parameters n and $\alpha$ which mathematically describe the measurement, they are a function of the assumed thickness. In accordance to [5], the transfer function exhibits a basic shape

$$H(\omega) = A_0 \cdot exp\left(-i\frac{\omega}{c_0}[\tilde{n}-1]L\right) + A_1 \cdot exp\left(-i\frac{\omega}{c_0}[3\tilde{n}-1]L\right) + \cdots. \tag{1}$$

Here, $c_0$ denotes the speed of light in free space, $\omega$ the angular frequency of the wave, and $\tilde{n}$ the complex refractive index. The amplitudes $A_i$ are functions of the Fresnel coefficients and can easily be calculated [5]. We point out that the arguments of the exponential functions in (1) are linearly independent. This implies that an incorrect thickness induces oscillations on both the calculated refractive index and the absorption. Only if the correct thickness is considered in the calculation, the FP-oscillations disappear. Thus, by iteratively changing the assumed thickness and minimizing the FP-oscillation amplitude, the correct thickness of the sample can be determined.

The conventional approach to this optimization is the Total Variation (TV)-minimization [4], in which the deviation of neighboring $n$- and $\alpha$-values is added up and the sum is minimized in a second step. But for thin samples, where the FP period extends over several hundred GHz (thus often several thousand sample points), the dielectric dispersion and the noise are the major contributions to the TV-value and therefore, the method fails for such thin samples. In literature, the thinnest sample investigated by TV is a 540 μm thick silicon wafer with an optical thickness (refractive index times the thickness) of around 1.8 mm [5].

Due to these limitations, we abandon the TV approach and introduce a quasi space optimization. As the FP oscillations are periodic in the frequency space, we apply a Fourier transform to the material parameter data, entering into the QS, where the periodicity of the FP oscillations directly corresponds to a discrete peak. According to the finite Fourier transform, the discrete QS values $QS_k$ can be computed by the formula

$$QS_k = \sum_{n=0}^{N-1}\left[y(\omega_n)exp\left(-i\frac{2\pi}{N}kn\right)\right], \quad k = 0, \ldots, N-1, \tag{2}$$

where $y(\omega_n)$ represents one spectral material parameter (either the refractive index, the extinction coefficient or the absorption coefficient) and $N$ is the amount of sampling points. In most the cases it is advantageous to utilize the refractive index for the calculation since this value is less sensitive to amplitude fluctuations [8]. The abscissa $x_{QS}$ of the QS value is given by the following relation:

$$x_{QS} = \frac{2\pi}{\omega}. \tag{3}$$

Thus, the transformation leads to a unit of time but since the period of the analyzed oscillations is assigned to the optical thickness of the sample, it is suggestive to normalize the $x$-axis as follows:

$$L_{QS} = x_{QS}\frac{c_0}{2}. \tag{4}$$

This way, the abscissa is directly correlated to the specific optical thickness.

The region of analyzable thicknesses as a quantity of the measured discrete data can be estimated by

$$L_{samp,max} = \frac{1}{df}\frac{c_0}{4n} = c_0\frac{T_{max}}{4n} \tag{5}$$

and

$$L_{samp,min} = \frac{c_0}{2n\Delta f}, \tag{6}$$

where $df$ is the spectral resolution, $\Delta f$ the bandwidth and $T_{max}$ the time windows of the measurement. While (5) is equivalent to the Nyquist–Shannon sampling theorem and the occurrence of at least one echo pulse in the time window of the measurement, (6) accounts for the distinguishability of the QS peak from its neighboring values and consequently for the condition of comprising at least one single period of oscillation within the spectral window. Therefore, the spectrometer's bandwidth, determined by the signal to noise ratio, is the limiting factor in regards to the minimum determinable sample thickness. While a 9 μm silicon layer can already be characterized in a standard 5 THz bandwidth TDS system, recently introduced ultra-broadband spectrometers with multiple ten terahertz of bandwidth [10–12] hold the potential to access material parameters and thickness information of even sub-micron thin samples using the QS extraction algorithm.

## 3. Measurements

To validate the QS algorithm, we measured three thin samples, consisting of well characterized materials: A high resistivity silicon (HR-Si) wafer, a thin sheet of polyamide nylon 6 (PA6), and a substrate of silica. If the thickness is determined correctly, the material parameters derived with the QS method are expected to agree with literature values obtained from thick samples, so that this comparison directly allows to judge the accuracy of the QS algorithm. The measurements were performed using a standard THz time domain spectrometer [13]. The HR Si sample has a specified thickness of 54 ± 1 μm. The derived value of the normalized QS peak is plotted as a function of the thickness in Fig. 1 (left). The QS value exhibits a clear minimum at 54.49 μm. Here, the resulting refractive index at 1 THz is 3.418 ± 0.002, which is in concordance with the literature value [13]. To further illustrate the principle behind the proposed algorithm, Fig. 1 (right) shows the QS values over the normalized abscissa $L_{QS}$ for three different assumed thicknesses. Caused by the definition of $L_{QS}$ (4), the QS peak is located at the position of the sample's optical thickness. For both, the smaller and the larger thickness, the QS values are relatively high and the oscillations on

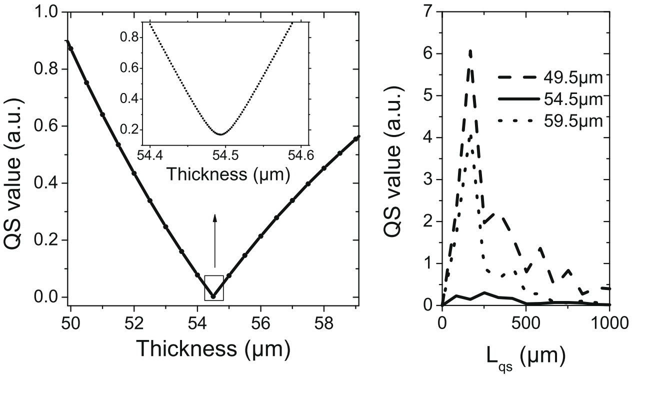

**Fig. 1.** The QS peak values for differing thicknesses of a HR-Si wafer (left) and the QS values over the normalized length $L_{QS}$ (right).

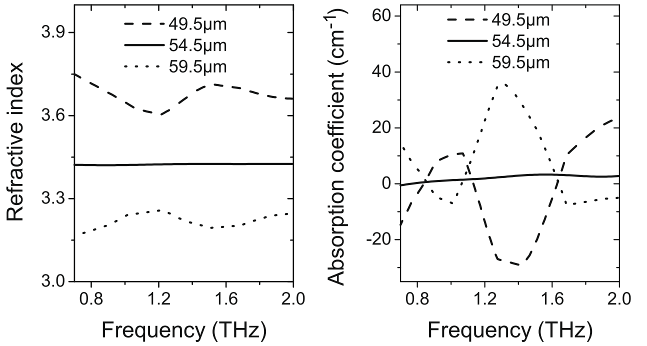

**Fig. 2.** The refractive indices (left) and the absorption coefficient (right) of the HR-Si wafer for three assumed thicknesses.

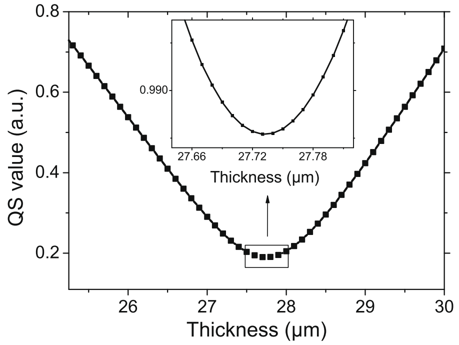

**Fig. 3.** The QS value of the polyamide (PA) sheet.

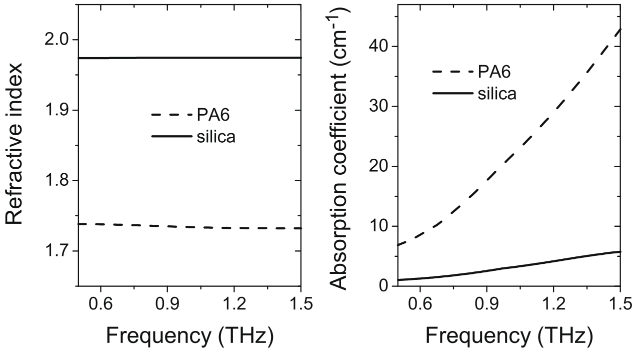

**Fig. 4.** The refractive index (left) and the absorption coefficient (right) for the PA6 sheet and the silica substrate.

the refractive index and the absorption coefficient (Fig. 2) become clearly visible. When the correct thickness is chosen, the oscillations vanish and the QS values reach a minimum as expected from theory.

Additionally, we investigated a $25 \pm 5$ μm PA6-sheet. In this case, the QS minimum is located at a thickness of 27.73 μm as shown in Fig. 3. Again, the resulting refractive index n at 1 THz ($1.734 \pm 0.01$), which is plotted in Fig. 4, agrees well with the literature value [9]. Note that the total optical thickness (refractive index times the thickness) of the PA6 sample was less than 50 μm.

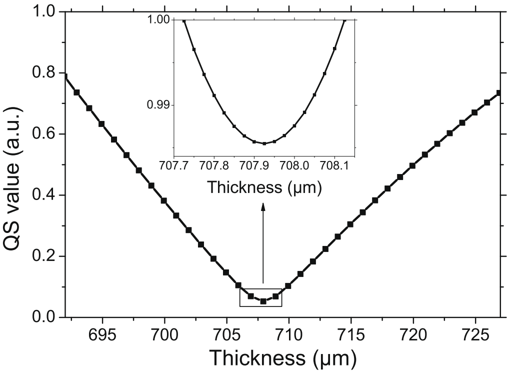

**Fig. 5.** The QS value of the silica substrate.

To demonstrate the broad applicability of the QS algorithm, we furthermore investigated a relatively thick glass sample ($700 \pm 10$ μm, silica). Here, the derived thickness is 707.93 μm (Fig. 5). The resulting refractive index at 1 THz $n = 1.974 \pm 0.003$ agrees well with the data published in [14]. The determined material parameters are shown in Fig. 4.

## 4. Conclusion

To conclude, we introduced an algorithm, which enables the thickness determination and material parameter extraction from thin samples (< 100 μm) with non-differential THz TDS measurements, which represents an order of magnitude improvement compared to existing methods. The algorithm is based on applying a Fourier transformation to the frequency dependent material parameters derived from a theoretical transfer function. In the resulting quasi space domain, a minimization of the Fabry–Perot peak by an iterative variation of the sample thickness delivers the desired information. The minimum sample thickness which can be investigated with this approach is only restricted by the bandwidth of the TDS system and the noise level.

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