Novel Algorithm for Sample Material Parameter Determination using THz Time-Domain Spectrometer Signal Processing

Kirill I Zaytsev¹, Arseniy A Gavdush¹, Sergey P Lebedev² and Stanislav O Yurchenko¹

¹ Bauman Moscow State Technical University, 2nd Baumanskaya str., 5, Moscow, 105005, Russia
² A. M. Prokhorov General Physics Institute of the Russian Academy of Sciences, Russia

E-mail: kirzay@gmail.com

Abstract. In our research we have developed a novel sample material parameter determination algorithm which could be used for characterization of very thin flats. Utilizing this algorithm one could find optical properties of the media which are thin enough to cause appearance of the satellite pulses in TDS system signal, transmitted through the flat sample. The algorithm is based on the minimization of the Error function (minimization between the theoretical model of sample transfer function and the experimental data collected with THz pulsed spectrometer). The algorithm was implemented experimentally and tested by means of the test samples characterization. The results of the algorithm implementation were compared with the results of the same test sample studying using terahertz backward-wave oscillator spectroscopy.

1. Introduction and background

Terahertz (THz) time-domain spectroscopy (TDS) is a technique for measuring the complex refractive index (or the complex permittivity) of different media over a wide frequency range starting from 0.1 THz and goes up to 3.0 ... 30.0 THz depending on the methods which are used for generation and detection of short THz pulses. Since the appearance of THz pulsed spectroscopy in second half of XX century [1],[2] a lot of authors have presented their material parameter extraction algorithms working with solid and liquid media [3]-[6].

Usually for material parameter extraction, a THz pulse, transmitted through the sample of interest, is being compared with THz pulse obtained without the sample on the propagation path of THz beam. Flat sample acts as a Fabry-Perot resonator for the THz pulse, thus the measured signal exhibits multiple reflections of the THz pulse (satellite pulses) caused by the multipath interference phenomenon. A simplistic method for approximation of the optical material parameters considers a narrow time window filtering procedure for the sample waveform. The window helps to suppress all the satellite pulses in the sample signal of THz spectrometer, leaving the main sample pulse untouched [7],[8]. However, the isolation of the first pulse may not be possible in the cases of low refractive indexes and/or thin samples Therefore the novel sample material parameter extraction procedure should be developed.
The present paper describes a technique for sample material parameter characterization based on the method, described in [5]. The sample could have rather small refractive index and could be rather thin (up to 0.1 mm). Small thickness of the sample would not cause distortion of the reconstruction results. Let’s describe all stages of algorithm implementation step by step. The results of experimental studying of test sample will be considered and discussed. Reconstructed sample material parameter curves will be compared with the results of the same sample studying utilizing THz backward-wave oscillator spectroscopy.

2. Results
Sample thickness should be determined precisely using micrometer or Vernier compass before measurement of TDS signals would take place. After thickness determination, two signals are measured with THz pulsed spectrometer: \( E_s(t) \) is the signal transmitted through the sample and \( E_r(t) \) is the signal transmitted through the empty THz beam path. After detection, the waveforms are being filtered using the procedure of tukey window filtering, which helps to suppress noises caused by multiple reflections of the main THz pulse in THz spectrometer beam path (generator, beamsplitters, lenses, detector etc.). Note, the filtering procedure should not distort the main THz pulse and the satellite pulses caused by the multiple reflections in the sample flat.

Figure 1 contains initial waveforms and the result of filtration. The filter window size and the filter location are determined automatically during the TDS signal processing in time-domain. We have chosen two test samples to verify the algorithm experimentally: GaAs sample with thickness 0.56 mm and ceramic sample with thickness 1.37 mm. The material parameters of GaAs were fully characterized in [9]. Note, that the waveforms presented in the figure 1 correspond to the test GaAs sample.

![Sample and Reference Waveforms](image)

**Figure 1.** An example of sample and reference waveforms before Tukey window filtering (top curves) and after it (bottom curves).

Reliable frequencies for complex refractive index determination are lying in range from 0.2 to 2.5 THz for TDS spectrometer based on LT-GaAs photoconductive antenna emitter and ZnTe electrooptical detector. Certainly this restriction depends on the type of TDS system which was used to collect experimental data. After signals have been detected, one could find experimental transfer function of the sample according equation:
where \( \hat{E}_s(f) \) and \( \hat{E}_r(f) \) are Fourier spectrums of the sample \( E_s(t) \) and the reference \( E_r(t) \) signals, and \( F_t \) is a Fourier transform operator. This transfer function takes into consideration all satellite pulses of sample signal.

Quasi Fabry-Perot resonator model was chosen as a theoretical model of sample transfer function. Theoretical model of transfer function has several parameters which need to be determined before starting the material parameter extraction. As it was mentioned above sample thickness \( l \) should be known \textit{a priori}. Also we need to know the number of satellite pulses in sample signal \( \delta \), and the interval of possible values of real and complex part of sample refractive index. The formulation of theoretical model can be written as follows:

\[
H_{\text{theor}}(f) = 4 \cdot \exp \left( \frac{j \cdot 2\pi f l (\tilde{n}_0 - \tilde{n}_1)}{c_0} \right) \cdot \left( \frac{\tilde{n}_1}{\tilde{n}_1 + \tilde{n}_0} \right)^2 \cdot \left[ 1 + \sum_{l=1}^{\delta} \left( \frac{\tilde{n}_0 - \tilde{n}_1}{\tilde{n}_1 + \tilde{n}_0} \right)^2 \cdot \exp \left( \frac{-2j \cdot 2\pi f l}{c_0} \right) \right]^{-1},
\]  

where \( f \) is frequency, \( c_0 \) is the speed of light in vacuum, \( \tilde{n}_1 \) and \( \tilde{n}_0 \) are the complex refractive index of sample and air, respectively. Figure 2 illustrates the modulus and the phase of the experimental and the theoretical transfer functions.

The sample material parameter determination is produced during the minimization of an error functional based on the experimental \( H_{\text{exp}}(f) \) and theoretical \( H_{\text{theor}}(f) \) transfer functions:

\[
\tilde{n}_1(f) = \arg \min_{\tilde{n}_1(f)} \{ \text{Err}(f, \tilde{n}_1(f), l) \}
\]

The error functional can be described with the following equation:
where
\[
\begin{align*}
M(f) &= |H_{\text{theor}}(f)| - |H_{\text{exp}}(f)|, \\
A(f) &= \angle H_{\text{theor}}(f) - \angle H_{\text{exp}}(f), \\
\eta &= 10 \cdot \xi.
\end{align*}
\]

Coefficients $\eta$ and $\xi$ helps to correct differences in order of the functions $M(f)$ and $A(f)$. In the figure 3 the dependence of an error function on the test ceramic sample refraction index, absorption coefficient and on the frequency of the incident electromagnetic wave is shown.

Inaccurate sample thickness determination would lead to the distortion of the reconstruction results. These distortions have the form of the periodic fluctuations appeared in the material parameter curves of the sample. To predict the appearance of these mistakes and to reduce the required accuracy of thickness determination one could use the procedure of numerical thickness correction during the THz spectrometer signal processing. According to this procedure the problem of functional minimization (3) needs to be solved for all possible sample thicknesses from the interval $[l_0 - \Delta l; l_0 + \Delta l]$, where $l_0$ is the thickness measured with the micrometer or the Vernier compass, and $\Delta l$ is a possible mistake of the sample thickness measurement. The result of this solution is functions $n_1(f, l)$ and $\alpha_1(f, l)$ (the material parameter curve dependence on the sample thickness). The criteria of the curve smoothness:

\[
R(l) = \left[ \int_{f_{\text{min}}}^{f_{\text{max}}} \left( \xi \left( \frac{\partial n_1(f, l)}{\partial f} \right)^2 + \zeta \left( \frac{\partial \alpha_1(f, l)}{\partial f} \right)^2 \right) df \right]^{-1}
\]

is used to choose the smoothest pair of material parameter curves, where $\zeta$ and $\zeta$ are the coefficients used to regulate the impact of the functions $n_1(f, l)$ and $\alpha_1(f, l)$ on the $R(l)$, $f_{\text{min}}$ and $f_{\text{max}}$ define the range of integration and correspond to the reliable frequencies for complex refractive index determination. This sample thickness clarification approach helps to significantly improve the accuracy of reconstruction especially if the relation between $\zeta$ and $\zeta$ is defined by:

\[
\zeta = 10 \zeta.
\]

Figure 3 illustrates several minimum branches of an error function. One of the presented minima of the error functional corresponds to the desired sample material parameters. Other minima (side branches) appear in correspondence to the multiple of $2\pi$ phase shifts of the time-domain signal Fourier-harmonics. In consequence, in the figure 4 the optical properties of the test samples are presented. This dependence of optical properties on frequency was obtained as the result of the global minimization of the sample pulse function in a wide range of the sample material parameter values.

Note, that additional conditions were used during the minimization procedure to prevent choosing of the false functional minimum. These conditions are based on the criteria of the optical material parameter curves smoothness. The points marked on the material parameter curves (figure 5) illustrate the results of the same test samples studying with THz backward wave oscillator spectrometer [10],[11]. One could see that difference between the results of the same sample studying with two different types of THz spectroscopy is lower than 1%.
Conclusions
The new algorithm for material parameter curves determination was presented and implemented. GaAs and ceramic samples were studied experimentally. The procedure for sample thickness numerical clarification was offered. It significantly improves the accuracy of the material parameter curves reconstruction and reduces the required accuracy of the sample thickness priori measurements. The material parameter curves of two samples were reconstructed and compared to the results of the same sample studying utilizing THz backward wave oscillator spectroscopy. The presented algorithm could provide reconstruction error smaller than 1%.
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