

All fiber THz spectrometer for plastics industry

R. WILK*

Menlo Systems, Am Klopferspitz 19a, 82152 Martinsried, Germany

Abstract. In this paper we present an all-fiber THz spectrometer based on novel OSCAT technique. We investigate its application to non-destructive testing in the polymer industry.

A set of polypropylene samples with different contents of TiO_2 as a filler is examined using THz waves.

Key words: fiber THz spectrometer, plastic industry.

1. Introduction

It has been shown that the THz technology is applicable to a non-destructive inspection of industrial goods and plastics [1, 2] as well as in the semiconductor industry, homeland security [3], biology and medicine. The polymer industry is of a special interest, while many polymers and additives are transparent to the THz waves. As shown it is possible to employ THz waves to in-line measurements of compound concentration, detection of defects, air bubbles, as well as foreign objects [4]. A harsh industrial environment includes strongly varying temperature, humidity and acoustic vibrations. Therefore, industry-specified systems are very demanding in terms of robustness and system stability. Key components of the conventional THz Time Domain Spectroscopy (THz-TDS) systems include the femtosecond laser source with a central wavelength around 780 nm, the LT-grown GaAs (LT-GaAs) antennae and a free-space mechanical delay line. The active region of typical LT-GaAs antenna is about $5 \mu\text{m} \times 10 \mu\text{m}$ big and its adjustment with free-space optics is very demanding and sensitive for acoustic vibrations. Thus, an application of free-space THz spectrometers in the industrial environment is somewhat limited. An alternative would be a THz system based on optical fibers and 1.55 μm femtosecond laser source. Fiber components originating from the telecom industry are well-developed, robust and cost-effective. Femtosecond fiber lasers are more efficient and typically less-expensive than Ti:Sapphire based lasers with emission wavelength around 800 nm. Recently developed LT-InGaAs material [5] with sub-ps carrier life time and sensitivity around 1.55 μm have sped up the development of all-fiber THz spectrometer. Up-to-date such fiber coupled THz spectrometers for industrial applications include external mechanical delay lines. This is usually the limiting element for system performance and stability. The scanning speed of typically used delay lines based on shakers, voice-coils or rotating mirrors is limited to a few ten Hz at a range of few ten ps. An alternative technique is based on two stabilized fs laser sources and Asynchronous Optical Sampling (ASOPS) [6] scheme. The ASOPS system offers virtu-

ally an unlimited range of the delay line with a scanning speed of several thousand scans per second. This solution overcomes many drawbacks of the conventional mechanical delay lines, but requires two femtosecond laser sources, synchronization electronics and increases an overall cost of the system.

The authors have recently presented an "Optical Sampling by Cavity Tuning" (OSCAT) measurement scheme for time resolved experiments in general [7, 8] and specifically OSCAT based THz spectrometer [9]. In this novel technique there is no need for an external moveable delay line, instead the temporal offset between the pulses in the pump and probe beam can be controlled by the repetition rate of the pulsed laser source. Although, the THz OSCAT spectrometer employs a single laser source, it presents two major improvements over conventional THz Time Domain Spectroscopy (THz-TDS). At first there is no need for the external moveable delay line. Secondly, there is, similar to the THz ASOPS systems, virtually unlimited scanning range covering the distance between the two adjacent pulses in the fs pulse train.

In this paper we present an all-fiber THz spectrometer based on the OSCAT technique. We investigate its application to non-destructive testing in the polymer industry and analyze a set polymer samples with TiO_2 fillers.

2. OSCAT

In conventional pump-and-probe experiments both the pump pulse and probe pulse originate from the same optical pulse which is split into two by an optical beam splitter. The temporal offset between them is set using mechanical delay lines. However, it is also possible to employ the pump and probe pulses that originate from the sequenced optical pulses when the pump and probe paths of the experimental setup are not of equal length. In such *unbalanced* experimental setup a change in the lasers source repetition rate f_{rep} modifies the time shift between the pump and probe pulses. The temporal shift $\Delta\tau$ between the pump pulse i and the subsequent probe pulse $i-a$ is determined by the repetition rate of femtosecond laser. A sweep of laser repetition rate from f_{rep} to $f_{rep} + \Delta f$ will

*e-mail: r.wilk@menlosystems.com

results in a continuous change of the temporal offset between the pulses:

$$\Delta\tau = a \left(\frac{1}{f_{rep}} - \frac{1}{f_{rep} + \Delta f} \right). \quad (1)$$

This scanning technique is called ‘‘Optical Sampling by Cavity Tuning’’ (OSCAT). As can be seen in Eq. (1), there are several important properties of the OSCAT scheme. Firstly, the temporal offset is being scaled with an index a (separation between the subsequent pulses in both arms of the experimental setup). In other words, one arm of the unbalanced interferometer has to be much longer than the other. Secondly, the tunable femtosecond laser source has to be characterized by low timing jitter. Moreover, it is preferable to choose possibly high repetition rate sources, where a small change in the oscillator length results in a significant repetitions rate shift Δf . For an extensive discussion about the OSCAT technique and its limitations, please refer to [7, 8].

In order to get a virtually unlimited temporal scanning range, the $\Delta\tau$ has to cover the whole spacing between the two adjacent pulses in a pulse train. In a case of typical experimental setup (250 MHz femtosecond laser with 1% tuning range), the index a has to be in a range of 100. This corresponds to the length difference of 120 m between the pump and probe arms of the experimental setup. Such distances are impractical for free-space approach. Therefore, the experimental setup was based on optical fibers components.

3. Experimental setup

The experimental setup is shown in Fig. 1. The core component is the femtosecond fiber laser with 250 MHz repetition rate and a tuning range of 2.5 MHz (model M-Fiber, Menlo Systems GmbH). The M-Fiber laser emits sub-90fs pulses with a central wavelength of 1550 nm. The output power of the oscillator is split into two fiber coupled ports. Both ports are dispersion pre-compensated for three meters of smf28e fiber.

A port A is connected directly to the fiber-coupled THz emitter antenna based on LT-InGaAs semiconductor (model Tera15-SL25-FC, Menlo Systems GmbH). A port B is connected to the 80 m long fiber link and afterward to the fiber coupled detector antenna (model Tera15-SL25-FC, Menlo Systems GmbH). The 80 m long fiber link consist of two different fiber types in order to archive zero dispersion operation. The antennae are illuminated with 34.6 mW and 38.1 mW for the emitter and detector, respectively. At such power levels nonlinearities in the optical fiber do not play a significant role. Therefore, the pulse shape and its duration is not significantly altered after the propagation in the optical fiber. The pulse duration after a 3 m fiber in port A and after a 83 m fiber in port B was measured to be around 80 fs. The emitter antenna is biased with a bipolar square-wave with amplitude of ± 20 V and a frequency of 15 kHz. The detector antenna is connected to the current-voltage amplifier and lock-in amplifier. The THz radiation is guided with four polymer lenses. The laser repetition rate is measured with a frequency

counter (model 53181A, Agilent). The setup does not include any moveable external delay line.

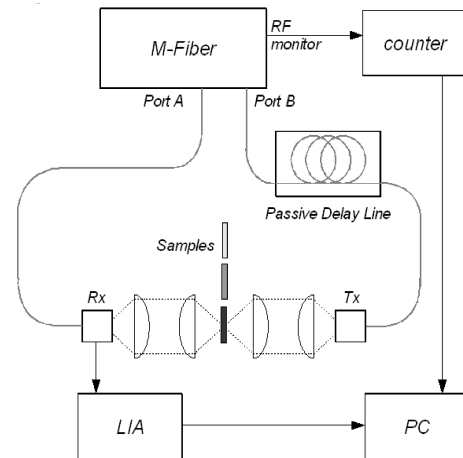


Fig. 1. Experimental setup

In order to investigate the application of all fiber THz OSCAT spectrometer to non-destructive testing in the polymer industry, a set of polymer samples with different contents of TiO_2 was analyzed. The filler contents were 0%, 5%, 10% and 20% of the weight, respectively. The 2 mm thick Polypropylene (PP)/ TiO_2 samples were measured in the spectrometer in transmission geometry.

4. Results and discussion

Figure 2 shows a pair of reference-sample THz pulses travelling through air and the PP plate, respectively.

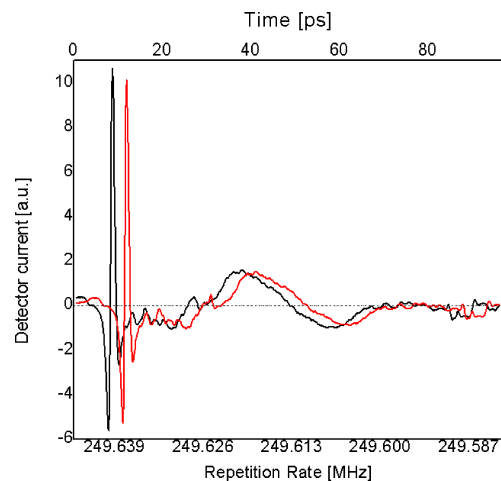


Fig. 2. THz pulses travelling through air (black line) and PP sample (red line)

The measurements are shown in laser repetition rate domain, which is characteristic for OSCAT measurements. In order to record the THz pulse, the lasers repetition rate was swept from 249 644 550 Hz to 249 582 580 Hz. The 62 kHz difference in a repetition rate corresponds to 97 ps in the time domain. The top axis shows re-calculated time axis, as given by Eq. (1). The pulse traveling through the PP sample is

delayed in time and has somewhat smaller amplitude due to the Fresnel reflections at the sample-air interfaces and small absorption of the PP material. The calculated frequency dependent refractive index of all measured samples is shown in Fig. 3. The PP refractive index dependency on the TiO₂ filler contents is shown in Fig. 4. Each point corresponds to the integrated refractive index over 200 GHz and 1 THz. The dependence of the measured PP/TiO₂ refractive index on the weight content of TiO₂ filler is nearly linear. The THz refractive index increases from 1.50 to 1.65 for pure PP sample and PP with 20 wt.-% content of TiO₂, respectively. Therefore, the filler concentration can be easily and non-destructively measured using the all fiber THz OSCAT spectrometer. The presented results are consistent with similar measurements conducted with conventional THz TDS technique, as reported in [4].

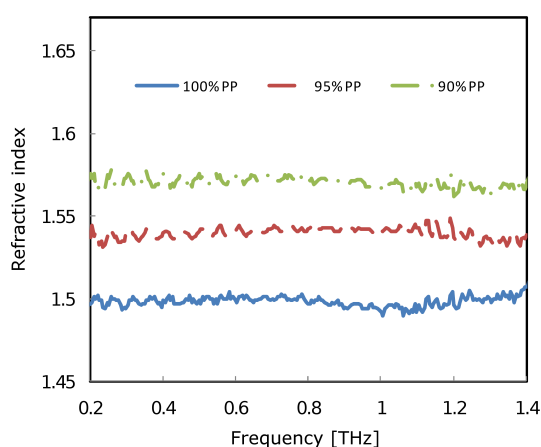


Fig. 3. THz refractive index of the measured polypropylene samples

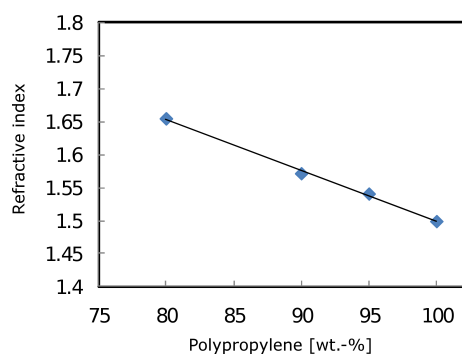


Fig. 4. Refractive index of the PP with TiO₂ additive for different compound concentration

5. Conclusions

In this paper we have employed novel THz OSCAT spectrometer to the quality control of the polymer compounds. The presented all-fiber THz spectrometer operates without any moveable extracavity delay line, and therefore is capable of the in-line measurements in the industrial environment. Measurements performed by the THz OSCAT spectrometer agree well with the measurements performed using the standard THz-TDS spectrometer.

Acknowledgements. The author would like to thank to R. Holzwarth, M. Mei from Menlo Systems and T. Hochrein from SKZ – Das Kunststoff-Zentrum for fruitful discussions

REFERENCES

- [1] R. Wilk, F. Breithfeld, M. Mikulics, and M. Koch, "A continuous wave THz spectrometer as a non-contact thickness measuring device", *Applied Optics* 47, 3023–3026 (2008)
- [2] J.S. Witkowski, P. Jarzab, K. Nowak, R. Wilk, M. Mikulics, and E.F. Plinski, "Fourier transform in THz measurements of refractive index", *IRMMW 1*, CD-ROM (2009).
- [3] R. Ryniec, M. Piszczek, and M. Szustakowski, "Multicriterial analysis of explosives in the THz range", *Acta Physica Polonica A* 118, CD-ROM (2010).
- [4] S. Wietzke, C. Jansen, F. Rutz, D. Mittleman, and M. Koch "Determination of additive content in polymeric compounds with terahertz time-domain spectroscopy", *Polymer Testing* 26, 614–618 (2007).
- [5] H. Roehle, R.J.B. Dietz, H.J. Hensel, J. Böttcher, H. Künzel, D. Stanze, M. Schell, and B. Sartorius, "Next generation 1.5 μm terahertz antennas: mesa-structuring of InGaAs/InAlAs photoconductive layers", *Opt. Express* 18, 2296–2301 (2010).
- [6] D. Stehr, C. Morris, C. Schmidt, and M. Sherwin "High-performance fiber-laser-based terahertz spectrometer", *Opt. Lett.* 35, 3799–3801 (2010).
- [7] T. Hochrein, R. Wilk, M. Mei, R. Holzwarth, N. Krumbholz, and M. Koch, "Optical sampling by laser cavity tuning", *Optics Express* 18 (2), 1613–1617 (2010).
- [8] R. Wilk, T. Hochrein, M. Koch, M. Mei, and R. Holzwarth "OSCAT: novel technique for time-resolved experiments without moveable optical delay lines", *J. Infrared, Millimeter and Terahertz Waves* 32, 596–602 (2011).
- [9] R. Wilk, T. Hochrein, M. Koch, M. Mei, and R. Holzwarth, "THz spectrometer operation by laser repetition frequency tuning", *J. Opt. Soc. Am. B* 28, 592–595 (2011).