The initial results of THz spectroscopy non-destructive investigations of epoxy-glass composite structure

Robert Panowicz[1], **Danuta Miedzińska**[2], **Norbert Pałka**[3] **and Tadeusz Niezgoda**[4]

[1]*Department of Mechanics and Applied Computer Science, Military University of Technology, Faculty of Mechanical Engineering*
Kaliskiego Street 2, 00-908 Warsaw, Poland
e-mail: rpanowicz@wat.edu.pl

[2]*Department of Mechanics and Applied Computer Science, Military University of Technology, Faculty of Mechanical Engineering*
Kaliskiego Street 2, 00-908 Warsaw, Poland
e-mail: dmiedzinska@wat.edu.pl

Institute of Optoelectronics, Military University of Technology, Department of Optoelectronic Technology
Kaliskiego Street 2, 00-908 Warsaw, Poland
e-mail: npalka@wat.edu.pl

[4]*Department of Mechanics and Applied Computer Science, Military University of Technology, Faculty of Mechanical Engineering*
Kaliskiego Street 2, 00-908 Warsaw, Poland
e-mail: tniezgoda@wat.edu.pl

Abstract

The paper presents the initial results of the epoxy-glass composite samples analyses carried out with the use of terahertz spectroscopy. Firstly, the standard specimens made of epoxy resin and glass fibers were researched. The first sample consisted of one layer of glass fiber, the second one had two such layers. To compare the results the results of the composite material sample shut with the 7.62 mm caliber bullet THz analyses were shown. The terahertz spectroscopy uses the well known spectroscopy research methodology however with the use of the new radiation scope. Terahertz bandwidth is mostly defined as (0.1 ÷ 10) THz. Terahertz waves penetrate many materials apart from metals or polar mediums (like water). Therefore, terahertz radiation allows to research many materials such as composite.

Keywords: composites, damage, experimental mechanics, laminates

1. Introduction

Recent advances in generation and detection of terahertz radiation with conjunction with spectroscopy methodology have enabled the undertaking of new non-destructive investigations of composite materials. The range of terahertz radiation has many definitions in literature. Terahertz bandwidth is defined as (0.1 ÷ 10) THz, what corresponds to wavelength (3000 ÷ 30 μm)[2].

Development and implementation of terahertz technology is connected with unique features of terahertz radiation. Frequency of terahertz radiation corresponds well to frequency of a normal mode (vibrational and rotational) of oscillating molecules of chemical compounds in room temperature and acoustic vibrations of molecules. Accordingly, chemical compounds of organic materials are characterised by specific absorption and emission spectra in the terahertz range.

Terahertz waves penetrate many materials apart from metals or polar mediums (like water). Therefore, terahertz radiation can "see" through many materials such as composite. Transmissivity varies significantly dependently on different types of composite. It is greater in glass composite than carbon composite.

The next major factor contributing to this interest is that THz radiation poses minimal health risk to a suspected person or the system's operation due to the fact that photon energy is very small (4,4 meV @ 1THz)[2].

2. THz radiation

The electromagnetic spectrum runs from long-wavelength radio at one end to high-energy, short-wavelength X-rays and gamma rays on the other. Between microwaves and X-rays, in the least explored region of the spectrum, lie T-rays, or terahertz radiation, the most common form of radiation in the universe.

In physics, terahertz radiation refers to electromagnetic waves propagating at frequencies in the terahertz range. It is synonymously termed submillimeter radiation, terahertz waves, terahertz light, T-rays, T-light, T-lux, THz. The term typically applies to electromagnetic radiation with frequencies between high-frequency edge of the microwave band, 300 gigahertz (3×10^{11} Hz), and the long-wavelength edge of far-infrared light, 3000 GHz (3×10^{12} Hz or 3 THz). In wavelengths, this range corresponds to 0.1 mm (or 100 μm) infrared to 1.0 mm microwave. The THz band straddles the region where electromagnetic physics can best be described by its wave-like characteristics (microwave) and its particle-like characteristics (infrared). According to some authors the THz band is also designated as Tremendously high frequency or THF.

Terahertz radiation is emitted as part of the black body radiation from anything with temperatures greater than about 10 kelvin.

Much of the recent interest in terahertz radiation stems from its ability to penetrate deep into many organic matrials without the damage associated with ionizing radiation such as X-rays (albeit without the spatial resolution). Also, because terahertz radiation is readily absorbed by water, it can be used to distinguish between materials with varying water content—for example, fat versus lean meat. These properties these applications are still in the research phase, although TeraView (Cambridge, England), which is partially owned by Toshiba, has developed a technique for detecting the presence of cancerous cells that is currently in human trials.

Terahertz radiation can also help scientists understand the complex dynamics involved in condensed-matter physics and processes such as molecular recognition and protein folding. lend themselves to applications in process and quality control as well as biomedical imaging. Tests are currently under way to determine whether terahertz tomographic imaging can augment or replace mammography, and some people have proposed terahertz imaging as a method of screening passengers for explosives at airports [5].

3. THz spectroscopy

There are two main techniques used for measurement of transmission spectra in the THz range: Fourier Transform Infrared Spectroscopy (FTIR) and Time Domain Spectroscopy (TDS). In both cases there were measured spectra of the material pellet and the reference pellet [3].

A typical far-infrared Fourier transform spectroscopy consists of a source - an incoherent high-pressure mercury arc lamp useful for the THz range and a piroelectric (DTGS) or a bolometer detector and a scanning Michelson interferometer (a far-IR beam splitter, mirrors, a motorized delay line), samples, and a data acquisition system. Because of the short coherence of the source, during scanning of the mirror an interferogram is recorded as a function of mirror position. Next, the data should be processed by means of Fourier transform to obtained the spectrum (Fig. 1).

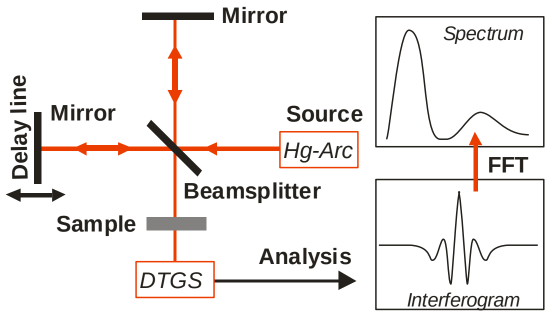

Figure 1. Fourier Transform Infrared Spectroscopy setup [1]

A standard Time Domain Spectrometry setup (from EKSPLA) with pulses generated by an 800 nm femtosecond laser is presented in Fig.2. The laser beam was split into a pump and probe and directed through a system of mirrors to an emitter and a detector. Low temperature grown GaAs dipole antennas with gap distance of about 6 µm were applied as the emitter and the detector. The pump beam was focused on the biased emitter antenna to generate THz pulses through a photoconductive phenomenon. The emitted THz pulses were collimated by a Silicon lens. The pellets were placed in the middle of the distance between the emitter and detector, perpendicularly to the incident THz beam. The transmitted THz beam was detected by means of the detector antenna gated using the laser probe beam and a mechanical delay line. A lock-in amplifier and LabView-based software was used to collect and process data. The system was purged with dry nitrogen gas to eliminate water vapor.

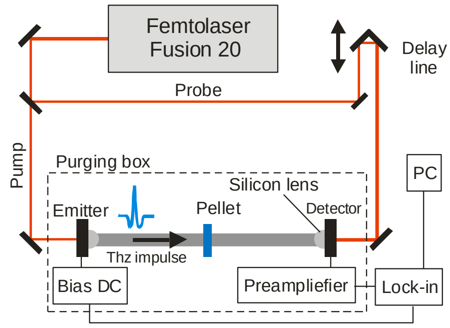

Figure 2. Time Domain Spectrometry setup [2]

4. Research equipment

The research presented in the paper was carried out with the use of TDS Spectra 3000 from TeraView with a reflection imaging module. TeraView's terahertz spectrometers utilize powerful, ultra-fast laser sources and semiconductor based detection systems to supply full access to the 0.06 – 4 THz (2 cm-1 - 120 cm-1) spectral region. Furthermore, TeraView's spectrometers do not require liquid helium cooling or any vacuum systems for its operation, enabling results to be obtained in a minute or less, all while operating under ambient temperature conditions.

TDS spectra 3000 from TeraView (Fig. 3) is the world's first commercial terahertz spectrometer capable of performing both transmission and attenuated total reflection (ATR) measurements. Its modular sample compartment also accepts reflectance imaging modules, variable temperature samples holders and cryostat accessories.

Figure 3. Terahertz (THz) spectrometer with modular sample compartment for transmission, ATR analysis, cryostats, variable temperature cells and reflection modules for imaging

Key features of the ThZ equipment are as follows:

- Terahertz transmission and reflectance measurements on solids, liquids, suspensions, slurries and films
- Spectral Range – 0.06 THz to 4 THz (2 cm^{-1} - 120 cm^{-1})
- Accomodates standard IR sampling accessories
- ATR modules for optimized measurements on as little as 1 mg solid sample
- Solid-state emitter and detector for ambient temperature operation
- Bespoke optics casing with controlled alignment optics for system optimization

5. Research results

The researched samples are presented in Fig. 3. Sample 1 is built of a single glass fiber layer, sample 2 – of two such layers. Those samples were tested with the use of the terahertz spectrophotometer TeraView. The results were saved and the image processing was carried out.

Figure 3. Researched samples

The data after initial processing are presented in Fig. 4. The border layers (front layer in blue, back layer in red) are very clearly marked. Due to the very high value of the signal coming from those surfaces to the detector, they were eliminated from the image before further processing (Fig. 5).

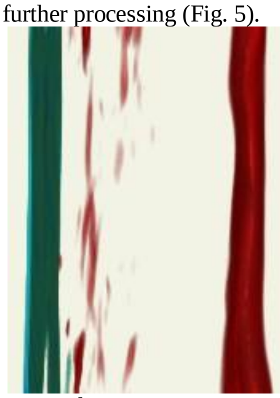
Figure 4. Data after initial processing

The shape of the back surface of the sample did not allowed for the total elimination of the reflected signal. Instead of that fact the internal area contrast improvement was reached. The particular elements of the structure are better visible. Changing the visualization surface position the variations in structure composition can be analysed.

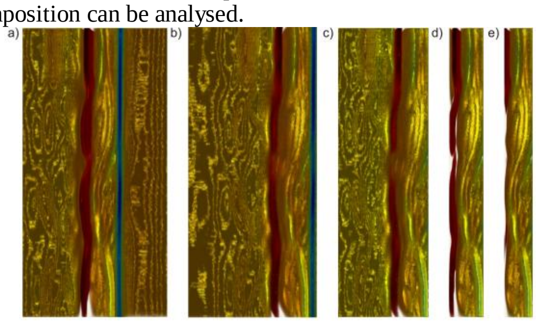

Figure 5. Image processing of one-layer sample

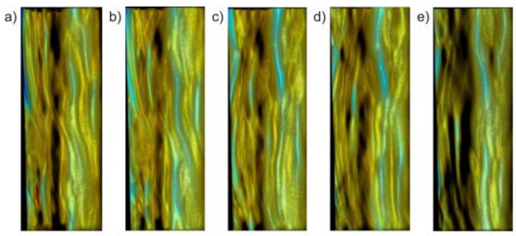

Figure 6. Image processing of two-layer sample

Fig. 6. presents the terahertz spectrogram of the second sample. Besides the external surfaces (as in the previous analysis) the internal areas of the increased deflection level are visible. Analogically to the described methodology allowed to get the image of the internal structure of the sample (Fig. 6).

The main difference between Figures 5 and 6 can be observed in the middle area of the image in Fig. 6. A lot of black areas are visible what can be assumed as the existence of the border between two glass fibre layers in the sample.

For the comparison the terahertz spectrogram of the multilayer glass composite sample shut with the bullet of 7.62 mm calibre was carried out. The sample shown in Fig. 7 was made of epoxy glass composite (E-53) reinforced with glass mat (density 316 g/m^2). The bevelling angle of the layers was 45 degrees and the thickness of the sample was 11 mm. In the first step of the research the resolution of the spectrogram was limited what allowed to reduce the number of registered data, the scanning time and to get the image of the sample larger area. The initial results are presented in Fig. 8.

Figure 7. Multilayer glass composite sample shut with the bullet – real photo

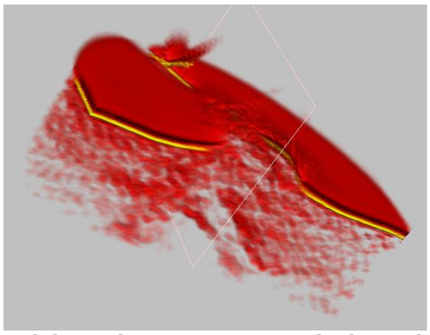
Figure 8. Multilayer glass composite sample shut with the bullet of 7.62 mm caliber – limited resolution ThZ spectrogram

The presented sample was than researched with the higher resolution of the ThZ spectroscope.

The high resolution spectroscopy of the sample described above was the next step of the research. To achieve more exact visualization of the damaged structure the spectroscopy was made two times: for the inlet and outlet side of the sample. The steps of the processing are presented in Fig. 9 as reference data, raw and deconvolved and filtered data. Such deconvolution process is often employed to extract the impulse function of the sample of interest [4].

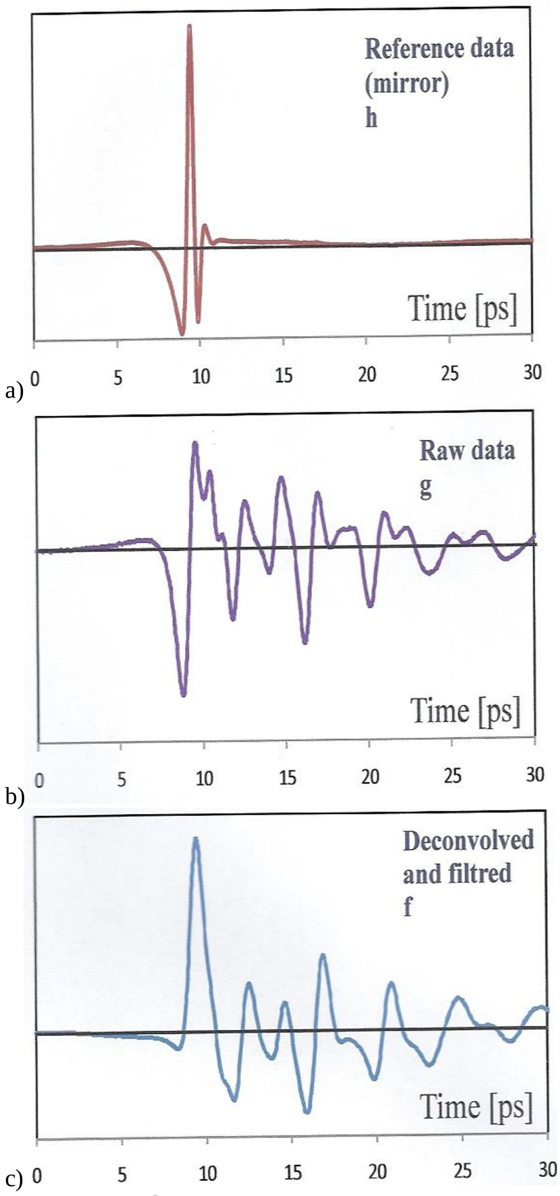

Figure 9. Signal processing

The results of the spectroscopy are presented in Fig. 10 – for the bullet inlet and in Fig. 11 – for the bullet outlet as real photos, B-scans (a vertical cross section), C-scans (horizontal cross section) images, and 3D visualization. In the pictures the regions of delamination as well as transitional and puncture regions are clearly visible. Also the layers of the composite can be observed.

6. Conclusions

The presented researched technique is currently at the initial stage. It allows to get the internal structure visualization of some kind of materials such as composites. The correct interpretation of the results needs a lot of experience. In this case, the improvement of the terahertz spectroscopy image and data processing software development is the most important. The results will be used for numerical models and analyses verification.

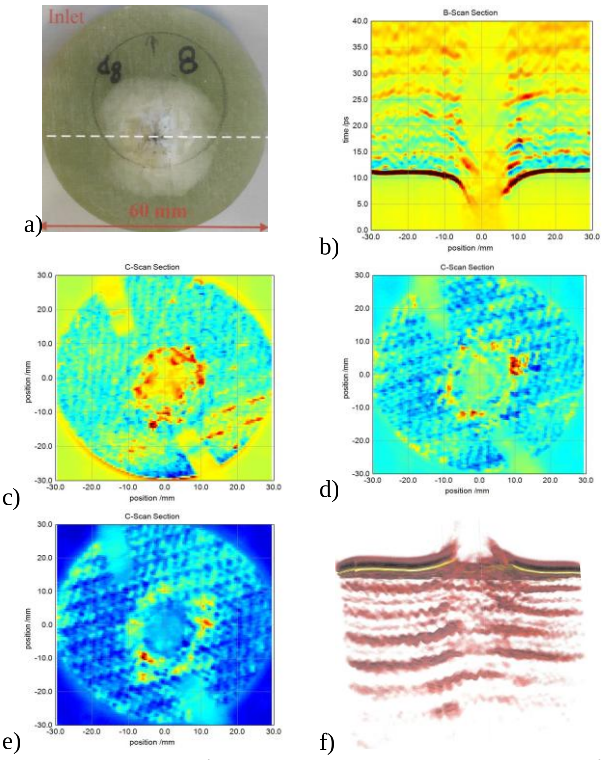

Figure 10. Results of the high resolution ThZ spectroscopy for the Multilayer glass composite shut sample for the bullet inlet: a) real photo, b) B-scan, c) C-scan for the signal passing time 13 ps, d) C-scan for the signal passing time 17 ps, e) C-scan for the signal passing time 25 ps, f) 3D visualization

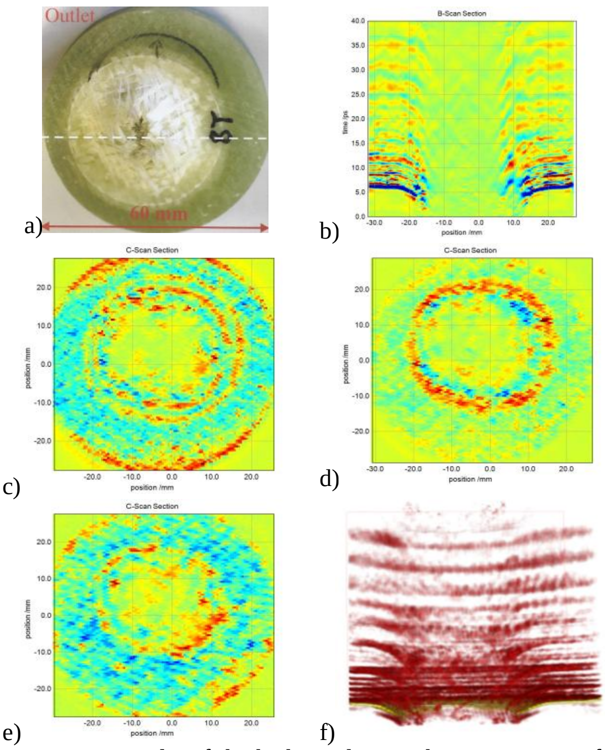

Figure 11. Results of the high resolution ThZ spectroscopy for the Multilayer glass composite shut sample for the bullet outlet: a) real photo, b) B-scan, c) C-scan for the signal passing time 9 ps, d) C-scan for the signal passing time 16 ps, e) C-scan for the signal passing time 20 ps, f) 3D visualization

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