Transmission pulse photoacoustic set-up for characterization of solids

K.Lj. Djordjevic
S.P. Galovic
M.V. Nesic
Vinca Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia
katarina.djordjevic@vin.bg.ac.rs
bobagal@vin.bg.ac.rs
mioljub.nesic@vin.bg.ac.rs

D. Todorovic
Institute for Multidisciplinary Researches, University of Belgrade, Belgrade, Serbia
dmtodor@afrodita.rcub.bg.ac.rs

M.N. Popovic
D.D. Markushev
D.K. Markushev
Institute of Physics Belgrade - National Institute of the Republic of Serbia, University of Belgrade, Belgrade-Zemun, Serbia
maricanpopovic@gmail.com
dragan.markushev@ipb.ac.rs
dragana.markushev@vektor.net

Abstract—**Laboratory-built measuring device, based on pulse photoacoustic (PA) effect, which allows the measurement of optical and acoustic signals in time domain, is presented. The experimental setup is described and the device specifications are given in detail.**

Optical and PA signals are measured in a time range from 10 µs to 10 s, for different samples and conditions. As an example, the experimental results for Si square membrane (Si chip with 5 x 5 mm frame and 3 × 3 mm square membrane, 32 µm thick, used in micromechanic devices) are presented. The square membrane Si chips were prepared by wet anisotropic bulk micromachining process.

The applicability of the measured PA signal in the characterization of semiconductors and micromechanical structures is discussed.

Keywords – photoacoustic, photothermal, time-domain photoacoustics, dynamic and relaxation processes, micromechanical structure,

I. Introduction

Photoacoustic (PA) effect is suitable for the development of experimental non-contact and non-destructive methods and allows examining different characteristics of optical, thermal, carrier transport and elastic characteristics of solids and soft matter [1]-[25].

There are two basic mechanisms of PA signal generation in an optically excited solid sample: *thermodiffusion* (TD) *mechanism* and *thermoelastic* (TE) *mechanisms*. Both occur during optical excitacion and optical absorption in the sample, leading to heat generation and its propagation from different heat sources, forming a space-time temperature field. TD mechanism is determined by thermal diffusion of heat pulses passing through the sample. TE mechanism is determined by the induced elastic deformation impulses passing through the sample.

Optically generated acoustic response (sound) can be detected by an acoustic detector (microphone). This is the so-called solid-gas-microphone (SGM) detection configuration and the acoustic response detected by the microphone (time dependent variation of the electric output of this detector) is the well-known – *PA signal.*

Generally accepted interpretation of the cause of a*coustic signal in the PA cell* in SGM measurements is the *piston model*, which considers two distinct regions in the gas volume of the PA cell. First region is the thin isobaric gas layer expanding and contracting at the solid-gas interface, thus acting as a piston. Second region is the major part of the gas volume where adiabatic pressure changes are transmitted through the PA cell.

Microphone pressure variations depend on optical, thermal, elastic and other related properties of the sample, which is the basis for the use of PA methods in material characterization. However, the recorded signal depends on geometric properties of the cell, transfer functions of electronic components in the measuring chain and the characteristics of the microphone, which requires the development of calibration techniques for each experimental setup, in order to distinguish that part of the signal.

Photoacoustics provides dual applications, i.e. photoacoustics can be considered in frequency-domain [1]-[10] and in time-domain [11]-[19]. Both methods have main advantages and disadvantages in recording measuring and analyzing PA signals. Frequency-domain characteristics are: (modulated) CW sources modulation frequency, matched to detector, narrow band response, coherent detection, cost efficient set up, high signal-to-noise ratio (SNR), low laser fluence and technically simpler sources. Time-fomain characteristics are: pulsed sources in Optoacoustics (when scan duration is lower than 10ns) and in Thermoacoustics (when scan duration is 500 ns – 1 ms), broadband response, time of flight measurement, being expensive, but having high SNR and high energy pulses (10-100mJ/pulse).

The excitation of the optical source by pulse modulation is performed by current-voltage modulation where the pulse-pause ratios are adjusted. In pulsed photoacoustics, the microphone records pressure changes in time domain, while in some settings Fast Fourier's transform of recorded pulse response is introduced for easier distinguishing of temporal heat evolution through the sample from other mechanisms involved in PA signal formation. The most common applications of time-domain photoacoustics are given in [11]-[25].

II. Pulse PA set-up

Presented impulse PA set-up is a laboratory-built experimental device that allows the measurement of optical and PA signals in time domain..

As presented in the scheme, given in Fig. 1, the set-up consists of *Optical & Acoustical part* and *Electronic part.* The selection of the parts is adjusted to achieve maximum acoustic noise protection and to obtain good SNR.

Optical & Acoustical part consists of: Optical source (LED), Photo detector and PA cell; *Electronic processing part* consists of: LED Driver, Digital Acquisition Unit (DAQ), Preamplifier (Preamp), and Signal & Control Processing Unit.

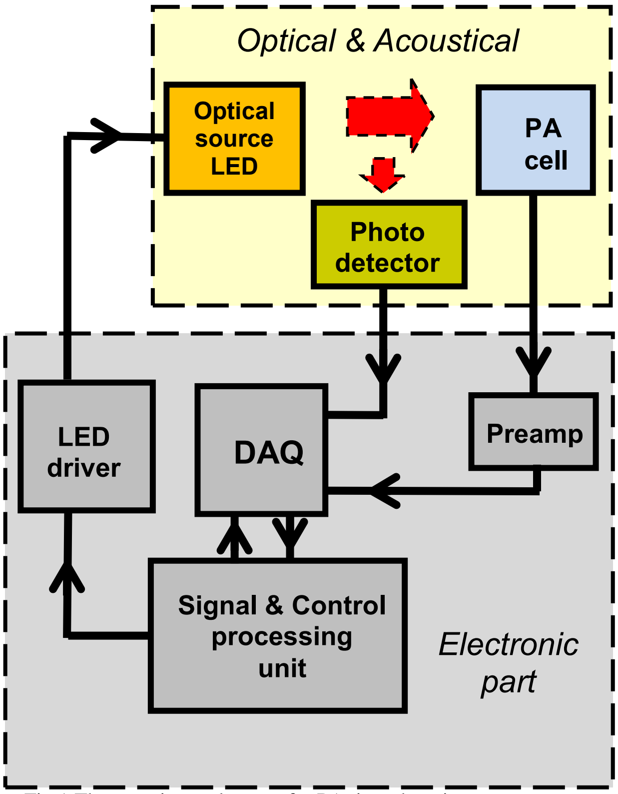

Fig 1.The experimental set-up for PA time-domain measurements.

Optical source is the modulated high power LED (~640 nm) which enables the optical excitations of the sample in the PA cell. Optical excitation is in the form of a series of rectangular pulses with the possibility of adjusting the intensity, the width, and the repetition rate of the pulse.

Photo detector enables measuring the optical excitation signals, i.e. the waveform of the optical signals by using the fast Si photodiode (10 ns).

PA cell is the original construction of PA SGM transmission configuration with minimal acoustic detection volume, using an electret microphone as acoustic detector.

The construction of the PA cell is optimized to get maximum PA signal (with minimum generation and detection acoustic volume), maximum acoustic protection from the surroundings and good signal/noise ratio (SNR).

LED Driver is a specially designed device that electronically controls high power LED in the optical source, in order to provide optical excitation as described above. It is constructed as a virtual instrument with full computer (software) control, enabling the generation of optical excitation pulses, as defined in optical source .

Digital Acquisition Unit (DAQ) is a commercial fast digital acquisition instrument (National Instruments DAQ - NI USB 6361 DAQ. Device specifications:NI 6361 - X Series Data Acquisition: 2 MS/s, 16 AI, 24 DIO, 2 AO. Analog Input: Number of channels 8 differential or 16 single ended and ADC resolution 16 bits. Sample rate: Single channel maximum 2.00 MS/s, Multichannel maximum (aggregate) 1.00 MS/s, Minimum No minimum, Timing resolution 10 ns and Settling Time for Multichannel Measurements ±0.2V, ±0.1V @ 2μs.

Preamplifier (Preamp) is the low-noise, wide-band (0.1 - 100 kHz) and sensitive (100 μV) preamplifier.

Signal & Control processing unit (Electronic processing unit) consists of a computer (PC) with the software specifically developed for control measuring procedure (measuring procedures enable full control of the measuring process: pre-set parameters, initialization, recording the data, etc).

The experimental apparatus described above allows different PA time-domain measurements. Typical PA measuring conditions are:

- optical pulse excitation
  - rectangular pulse train;
  - repetition rate (frequency), f (f = 0.5 – 50 Hz);
  - pulse period, T (T = 20 - 2000 ms);
  - pulse width, T_W (T_W / T = 0.1 – 100 %);
  - intensity I_0(I_0 = 0.05 – 5 mW / cm2);
- digital acquisition:
  - scan rate, F_S= 1e3 – 1e6 sample/s;
  - scan duration, (measuring sequence, T_S= 500 – 5000 ms);
  - number of samples, $N_S = F_S T_S$;
  - number of periods, N_T (N_T = 5 – 100 periods);
  - samples / periods, N_{ST} (N_{ST} = 1000 samples, min);
  - time resolution, T_{RES}(T_{RES} = 1 μs, max);

III. Experimental photoacoustic signals vs time

As an example, the experimental results for the micromechanical structure - Si square membrane - are presented here. The sample is Si membrane 32 μm thick (Si chip with frame 5 x 5 mm and square membrane 3 × 3 mm). The Si chips with square membrane were prepared from 3 inch, 390 μm thick, double side polished, 3-5 Ω·cm n-type (100) Si wafers. Si membranes were fabricated by wet anisotropic bulk micromachining process. Potassium hydroxide (KOH) solution in water is used as echant of Si.

The measurement conditions are given below:

- repetition rate (frequency), $f = 8$ Hz;
- pulse period, $T = 125$ ms;
- pulse width, $T_W = 62.5$ ms (T_W/T=50%);
- time resolution, $T_{RES} = 100$ µs);
- scan rate, F_S= 2000 samples/s;
- number of periods, N_T ($N_T = 5 – 100$ periods);

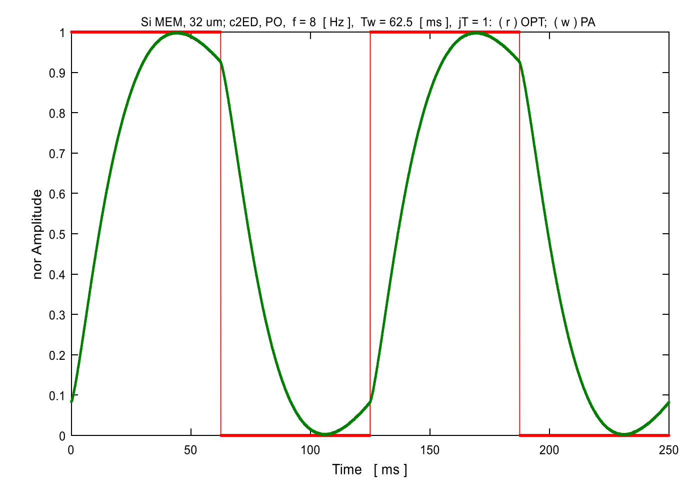

Fig. 2. Normalized and synchronized optical and PA signals (two periods) for a micromechanical structure - Si membrane 32 µm thick (Si chip with frame 5 x 5 mm and square membrane 3 × 3 mm). (red) optical signal; (green) PA signal.

Experimental signals, optical excitation and PA response (in Fig.2 given as the rectangle pulse train and the periodic-green line, respectively,) have been measured versus as the function of time. Each experimental signal is obtained as the averaged result of at least three measurements. Optical and PA signals are normalized by the following formula:

$$S_{nor} = \frac{S - \min(S)}{\max(S) - \min(S)} \qquad (1)$$

The PA effect in semiconductor structures depends on the photogeneration of electron-hole pairs, i.e. plasma waves. The carrier thermalization, surface and bulk recombination, as thermal sources, cause thermal wave generation. The propagation of thermal waves through the sample and through the surrounding gas, which is in contact with the sample, produces acoustic waves (sound) in the gas. This is the so-called thermodiffusion (TD) mechanism of PA generation. Also, the thermal waves cause elastic displacements of the sample surfaces and then it acts as a mechanical piston, producing sound in the surrounding gas - the thermoelastic (TE) mechanism of PA generation. Also, semiconductor materials exhibit mechanical strain when electron-hole plasma is generated – the plasmaelastic (PE) mechanism of PA generation. It means that general theoretical analysis of the transport processes in a semiconductor and semiconductor micro-mechanical systems (MMS) requests modeling the complex system of thermal, elastic and plasma wave phenomena [8-10]. This theoretical treatment enables quantitative accounting for amplitude and phase of the carrier density, temperature and elastic displacement as well as describing their functional dependence on time and thermal, elastic, and carrier transport properties of the sample.

By comparing experimental measurements of PA signal (Fig.2) with theoretical predictions thermoelastic, optical, and relaxation processes in MMS can be obtained. This is the subject of further investigations of the group.

IV. Conclusion

Transmission pulse photoacoustic measuring set-up for characterization of solids was developed and tested. The experimental PA pulse setup was described and the device specifications were given in detail.

The experimental results for the micromechanical structure - Si square membrane - were presented. These results showed that the developed laboratory apparatus is suitable for the characterization of solids, ie for investigating various dynamic and relaxation processes in solid material and micromechanical structures together with the corresponding theoretical-mathematical model, which remains the subject of further research.

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