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Fabrication of photoacoustic cell and thermal diffusivity measurement of coal carbon black using it

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A B S T R A C T

Photoacoustic spectroscopy (PAS) is an important technique for the characterization of materials by measuring heat generation in the sample. The photoacoustic (PA) signal detection is rather difficult due to the weaker signal intensity and unavailability of sensitive detectors of acoustic waves. In this work, authors have fabricated a photoacoustic cell and made parametric studies to get the maximum signal. For the optimum signal, the volume of acoustic cell cavity was found to 9.42×10^{-7} m^3. After the optimization, thermal diffusivity of coal Carbon black was measured and it was found to be 2.206×10^{-4} m^2/s which is very close to the standard diffusivity value of coal carbon.

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1. Introduction

In 1880, Alexander Graham Bell shown that when very thin disc was exposed through a beam of mechanically chopped sunlight, the sound wave was generated by the disc. Later, Bell revealed that materials can also produce sound when they exposed to IR or UV region of the solar spectrum which was a novel observation by A. G. Bell but this effect was forgotten at that time [1]. After the discovery of microphones, Viengerov was able to observe photoacoustic effect in gaseous samples also but growth of this new branch of spectroscopy still remains in a very less state due to many experimental limitations and sensitiveness [2]. The development of lasers in early sixties confirmed a new way in the Photoacoustic spectroscopy.

In 1976, Rosencwaig and Gersho had formulated a complete theoretical explanation to this effect in condensed media [3]. The future is moving for nanomaterial-based optoelectronic devices so it will be very beneficial to study the conversion of light energy into thermal energy by using Photoacoustic technique [4]. This spectroscopy technique can also be used for the characterisation of rare earth doped materials [5,6]. This technique is very simple and easy to establish in the laboratory with least instrumentation and it is applicable to materials in any phase of the sample (solid, liquid, and gas), and have a large impact value [7]. Photo-acoustic spectroscopy (PAS) is playing a vital role in the investigation of thermal as well as the optical properties of the samples [4]. Many thermal parameter for examples thermal diffusivity, thermal conductivity and heat capacity can be measured using a very simple design of Photoacoustic cell [3]. Thermal diffusivity (α) can be defined as the rate of change of diffusion of heat in a material and can be given as:

$$\alpha = k/rc \tag{1}$$

Where k is thermal conductivity (watts per meter-Kelvin or Wm^{-1}-K^{-1}), ρ is density (kg/m^3), c is specific heat capacity ($J\ kg^{-1}\ K^{-1}$). By Gilliam and Morgan (1987) in assessing the suitability of a place for a durable disposal, one of the individuality that may change the site of constancy is the generation of heat resulting in thermal stresses which could change the existing directions. Then, for an evaluation of waste disposal potential, data of thermal diffusivity and thermal expansion are required [8]. The Photoacoustic mentions that the generation of acoustic wave by the sample due to the interaction of modulated electromagnetic radiation with it [9]. If I be the intensity of the modulated incident light into the sample which kept inside the cell could be stated by the equation

$$I = I_o/2.(1 + cos\omega t) \tag{2}$$

Where, ω represent the modulation frequency of the chopper and I_o is the maximum beam intensity where the chopper frequency is

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zero. The sample generate periodic heat (H) due to the incident beam define by the equation

$$H = \beta I_o / 2.e^{\beta x}(1 + cos\omega t) \quad (3)$$

Here x is the distance away from the surface of the sample where heat produced and β is the absorption coefficient. The pressure variation was developed inside the cell cavity by the sample according to the chopper frequency which generates acoustic noise. If θ be the temperature developed along the sample thickness x, then it characterized by the thermal diffusion equation and it given by Rosencwaig and Gersho [10].

$$\frac{\partial^2 \theta}{\partial x^2} - \frac{1}{\alpha}\left(\frac{\partial \theta}{\partial t}\right) + Ae^{\beta x}(1 + e^{i\omega t}) = 0 \quad (4)$$

$$\text{Where}, A = \frac{\beta I_o}{2\alpha\rho c} \quad (5)$$

Here α, ρ, c are the thermal diffusivity, density and specific heat of the powder sample respectively [3]. By knowing the transition frequency and the thickness of the specimen under investigation, the thermal diffusivity (α) can be evaluated using the expression,

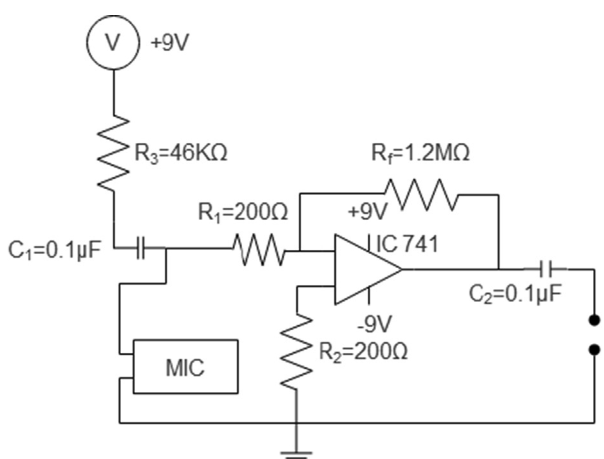

Fig. 1. Pre-amplifier circuit diagram.

$$\alpha = f_c l^2 \quad (6)$$

Where f_c is the critical frequency and l is the sample thickness [11]. Above formula is valid for thermally thick samples with known thickness. In order to measure thermal diffusivity various theoretical models have been proposed with specific sample conditions [12]. In the present case thin pellets of the samples were made.

2. Experimental techniques

2.1. Instrumentation for the spectrometer

A common PA spectrometer consists of four parts: a light source with an optical chopper for illumination, photoacoustic cell, amplifier, and the data acquisition system. In this experiment authors have used a diode laser of wave length 980 nm (Model PSU-H-LED, Changchun New Industries Optoelectronics tech. Co. Ltd, China). For modulation of the incident light a mechanical light chopper (Model SR 540, Stanford Research Systems, INC) in frequency range of 10 Hz. to 400 Hz was used. As the signal detected by the microphone is very week, a voltage pre-amplifier was designed to amplify the weak signal from condenser microphone. To obtain the desired signal, authors have designed a pre-amplifier using IC 741, as shown in Fig. 1. Here ± 9 V batteries are used as a biasing voltage of the amplifier and microphone. Single stage pre-amplifier circuit shown in Fig. 1 is used to amplify the weak signal of microphone (Brüel & Kjær, Model-4966, Sensitivity 50 mV/Pa and frequency range 5 Hz–20 kHz). For data recording a double channel digital storage oscilloscope (DSO) (Model GDS-2102E) was used.

2.2. Design of the Photoacoustic cell

In the Photoacoustic spectrometer, the cell is one the most important components of the system. The Photoacoustic cell design must have some restriction according to the theoretical studies made by Rosencwaig and Gersho [3]. These requirements should be followed with care during the fabrication of the cell [3]. The laboratory fabrication of photoacoustic cell permits flexibility to the users according to the need. It has two parts; one the main block where there is a place for sample chamber & is connected through a small channel to a sensitive condenser micro-

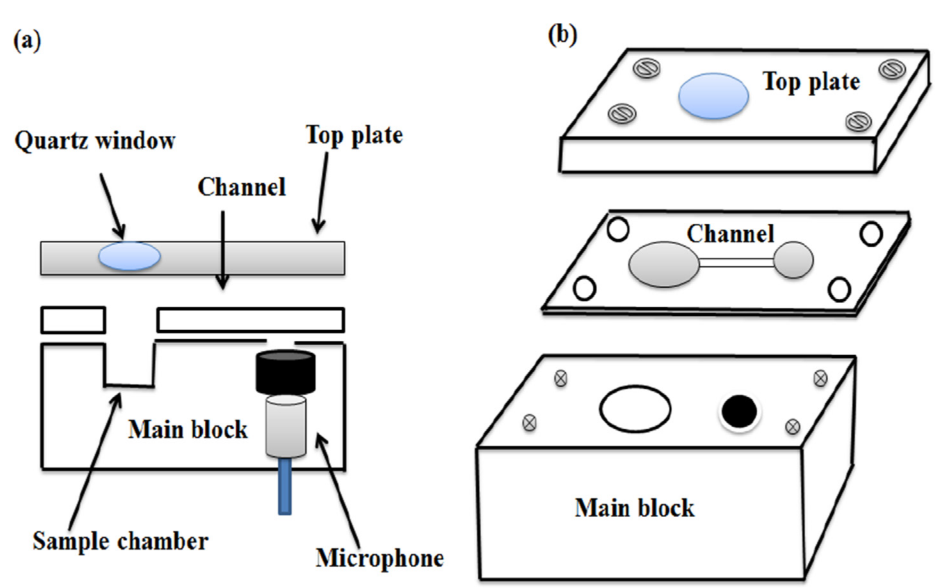

Fig. 2. Design of a Photoacoustic cell. (a) Cross-sectional view of the cell. (b) Vertical view of the cell.

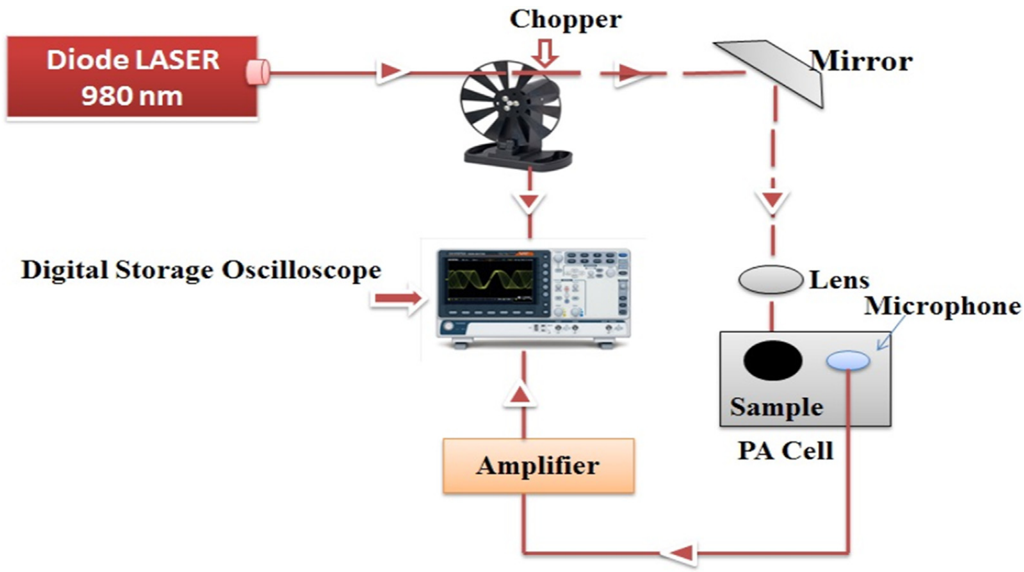

Fig. 3. Experimental setup for the Photoacoustic spectrometer.

phone and second one is the covering top plate. The top plate consists of a quartz glass window to pass the light. Both of these are made by aluminium block. To minimize the outside interference Photoacoustic spectrometer was fully covered through thick foam layer. Fig. 2(a,b) shows the horizontal and vertical views of the acoustic cell. Complete experimental setup ready for measurement is shown in Fig. 3.

3. Results and discussion

3.1. 3.1Thermal diffusivity measurement of coal carbon black

The thermal diffusion length is the function of chopping frequency of the chopper. Here amplitude of the generated acoustic signal was measured by varying the chopping frequency. The tran-

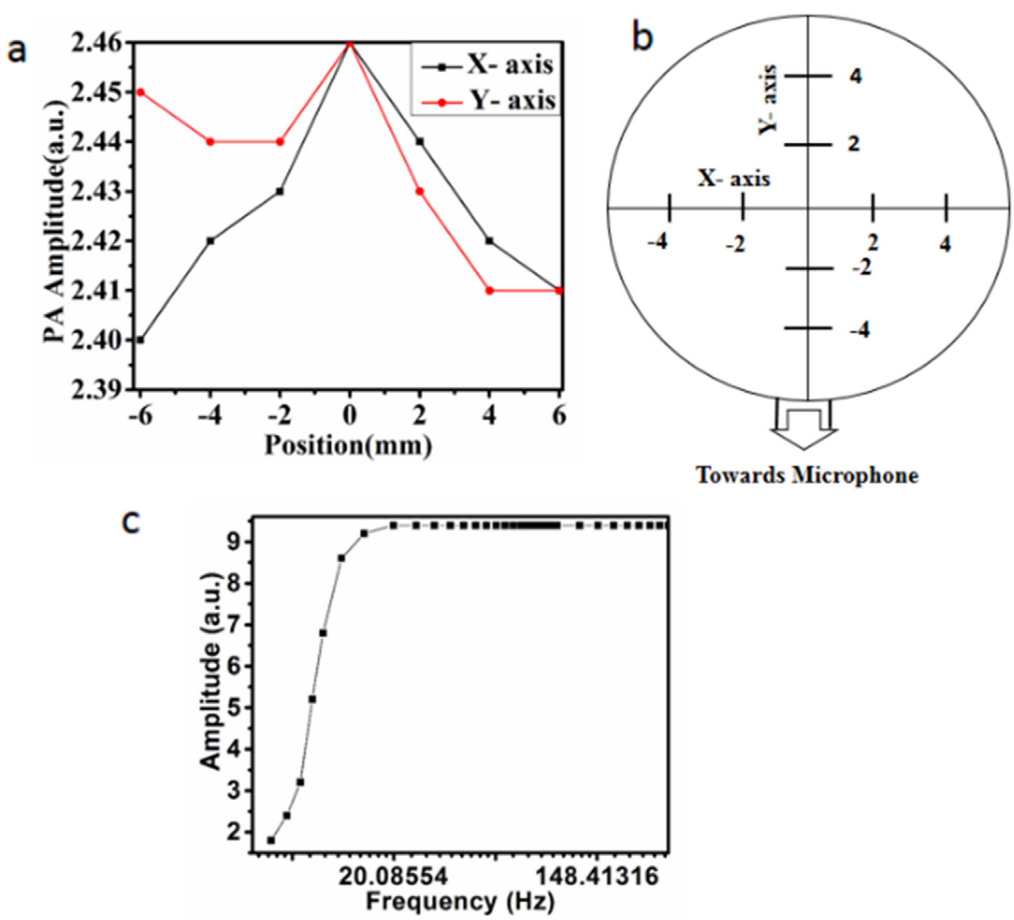

Fig. 4. (a) Variations of the acoustic signal strength with position of the incident light, (b) representation of different position of the cell (c) frequency response of the microphone.

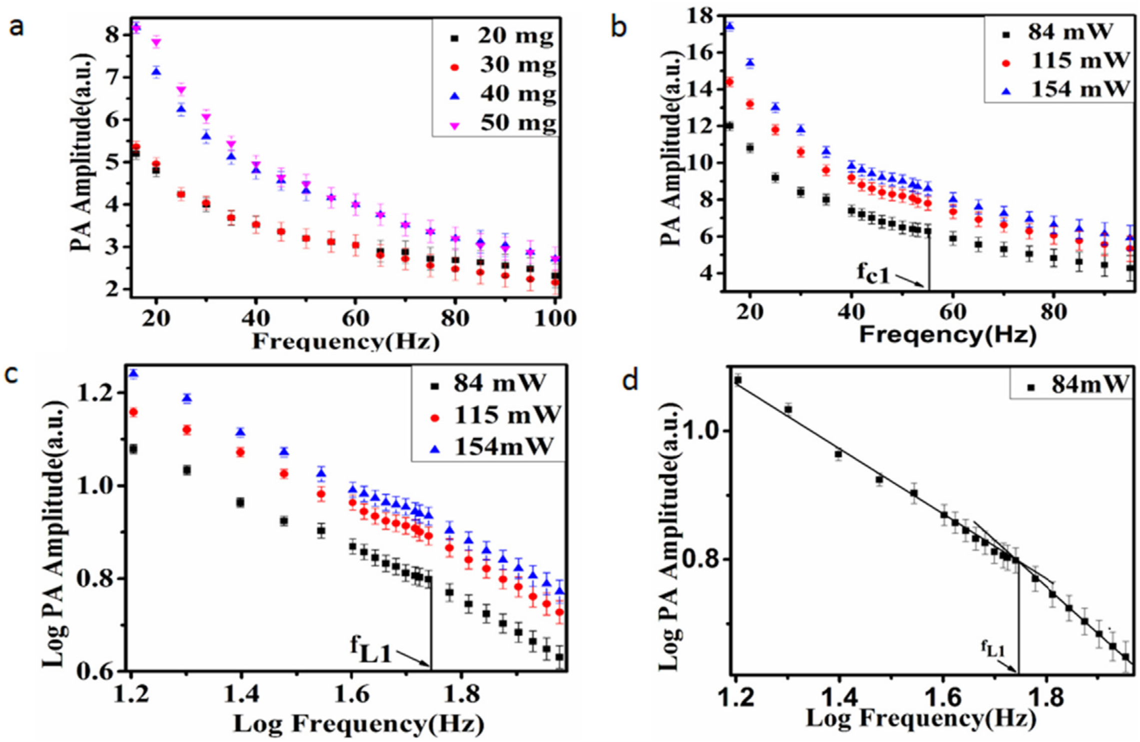

Fig. 5. (a) PA Signal for different volume of lamp black Carbon, (b) Variation of PA Signal for Coal carbon black with frequency, (c) Variation of PA Signal log amplitude with log frequency for Coal carbon black, (d) Linear fitting of the log PA amplitude with log frequency for 84 mW.

Table 1
Records of the transition frequency from Log-Log graph.

Corresponding Power (mW)	f_L Log frequency (Hz)	Transition f_c (Hz)	Thermal Diffusivity α (m^2/s)	Mean value of α (m^2/s)
84	4.0270	56.092	2.24368×10^{-4}	$\approx 2.206 \times 10^{-4}$
115	4.0119	55.251	2.21004×10^{-4}	
154	3.9916	54.141	2.1656×10^{-4}	

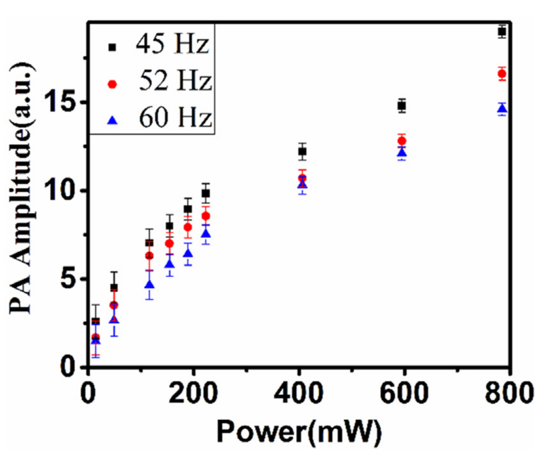

Fig. 6. PA amplitude with power of incident laser beam.

sition frequency at which a sample changes from thermally thin to the thermally thick regime can be known from the amplitude spectrum of the PA signal. In the Fig. 4(a) and (b), authors shown the variations of photoacoustic voltage with different position of the incident beam into the cell. We have consider that the origin present at the middle of the sample holder. Here from the recorded data, we found that at the middle of the sample holder, our cell shows maximum sensitivity. Fig. 4(c) shows the frequency response of the microphone at low frequency range, it is clear that our microphone works in between a specific range and it has less performance at very low frequency (less than 15 Hz) of the acoustic signal. From this we conclude that our microphone works properly within the range. Our first purpose is to optimize the cell. As the sensitivity of the cell depends on the cavity volume, we have performed experiment for different volume of the lamp carbon black samples and plotted these data with the modulation frequencies. In Fig. 5(a), it has been shown that the sensitivity of the cell depends on the inert volume of the cavity and for sample of volume 4.71×10^{-7} m^3 corresponding to the 50 mg for excitation wavelength 980 nm is more sensitive compare to the others less volume of the same sample.

Finally to perform the experiment, we choose same volume of coal carbon as a sample to get better performance of the cell. Here, Fig. 5(b) shows the data record for coal carbon with the same excitation laser light for different modulation frequency. Now we obtained the transition frequency f_c from the plot 5 (c) log–log plots of amplitude and frequency. We have record the data for three different power of the laser like 84, 115, 154 mW etc. and evaluated the transition frequencies from log plots of each of the graph. Here transition f_{c1} (56.092 Hz) is obtained from the linear fitting in Fig. 5(d) of the log–log plots corresponding to the incident source of power 84mW. Finally, obtained the thermal diffusivity is 2.24368×10^{-4} m^2/s by using equation (6). Similarly, the transition frequencies are obtained f_{c2} (55.251 Hz) and f_{c3} (54.141 Hz) for incident laser power 115 and 154mW respectively and calculate thermal diffusivity for each power of incident beam follow the same way as the previous one. All calculated data are given below in Table 1. Finally, authors have measured average value of the thermal diffusivity of the coal carbon black which was 2.206×10^{-4} m^2/s by using Photoacoustic spectroscopy.

3.2. Table

In Fig. 6, we have also study the acoustic signal variations with different power of the incident laser beam for three different modulation frequencies 45 Hz, 52 Hz and 60 Hz respectively and here from the result, we conclude that the acoustic signal strength increases with the increases of the power of laser beam but it decreases with increases of the modulation frequency, as maximum photon blocked by the chopper at high frequency compare to the low frequency and it was effected on phonon transition for non-radiative decay.

4. Conclusion

Authors have successfully fabricated and optimized the Photoacoustic spectrometer. Fabrication of Photoacoustic spectrometer is done on the basis of theoretical model of Rosencwaig and Gersho (R-G theory). Authors have optimized the spectrometer by using the carbon Lamp black sample. We also study the sensitivity of the cell for different volume of the samples and suggested that for the maximum sensitivity the volume of the inert cavity should be less. We have study the frequency response of the microphone at low frequency range and from the recorded data we have seen that our microphone sense the acoustic signal within the specific range and it has less sensitivity at low frequency less than 15 Hz. Finally, thermal diffusivity of Coal carbon black is measured by our designed Photoacoustic spectrometer which was 2.206×10^{-4} m^2/s. Also from the study of the acoustic signal with power, we have seen that for a fixed power of incident light, microphone sense large acoustic signal at low frequency compare to high frequency of the chopper. So we could say that the number of phonon for responsible of generation of acoustic signal due to non-radiative decay also decrease with incises of modulation frequency. Photoacoustic is an effective technique for thermal characterization of solid sample.

CRediT authorship contribution statement

Minarul I. Sarkar: Conceptualization, Methodology, Data curation, Investigation, Writing – original draft. **Kaushal Kumar:** Conceptualization, Methodology, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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