CHARACTERIZATION OF SELECTED PHOTONIC MATERIALS AND SYSTEMS USING PHOTOACOUSTIC TECHNIQUE

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Characterization of Selected Photonics Materials and Systems Using Photoacoustic technique
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CERTIFICATE

Certified that the research work presented in the thesis entitled "***Characterization of Selected Photonic Materials and Systems using Photoacoustic Technique***" is based on the original work done by Mrs.Annieta PhilipK. under my guidance and supervision in the International School of Photonics, Cochin University of Science & Technology, Kochi 682 022 and has not been included in any other thesis submitted previously for the award of any degree.

Kochi
29-10-2004

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DECLARATION

Certified that the work presented in the thesis entitled "***Characterization of Selected Photonic Materials and Systems using Photoacoustic Technique***" is based on the original work done by me under the guidance and supervision of Dr. P Radhakrishnan, Professor, International School of Photonics, Cochin University of Science & Technology, Kochi 682 022, India and has not been included in any other thesis submitted previously for award of any degree.

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Annieta Philip K.

Preface

Spectroscopy is the measurement and interpretation of absorption and emission of electromagnetic radiation when atoms or molecules or ions move from one energy level to another. Because of its versatility, range and non-destructive nature, optical spectroscopy remains a widely used and most important tool for investigating and characterizing the properties of matter. The photoacoustic (PA) effect originally discovered by Alexander Graham Bell in 1880 has been developed into an elegant, sensitive and nondestructive method for measuring the optical and thermal properties of materials due to the availability of coherent optical sources and advancement in signal processing and data acquisition systems. In the simplest terms laser photoacoustic spectroscopy (PAS) involves the absorption of light energy by a molecule or atom and the subsequent detection of heat energy released by the molecule upon return to the ground state where as in conventional optical absorption spectroscopy the optical absorption properties of the sample are studied by measuring the light transmitted by it. Since PAS measures the internal heating of the sample, it is clearly a form of calorimetry as well as a form of optical spectroscopy. The sensitivity of laser photoacoustic spectroscopy arises from the inherently high efficiency of thermal conversion that occurs in most of these light absorption processes coupled with the efficiency of the sensing device that converts the pressure waves into a voltage pulse. One of the great advantages of the PA measurements is that it is a direct monitoring of the non-radiative relaxation channel and therefore the PA technique may complement the absorption and luminescence spectroscopic techniques.

The basic physical process in the PA effect is that, a medium converts part of the intensity modulated light energy absorbed by it into heat. Such heat can be detected either by measuring the time dependent pressure fluctuations induced in a coupling gas or by measuring the thermal stresses or strains in the absorbing medium. The pressure fluctuations can be detected using a microphone and the thermal stress can be detected using a piezoelectric transducer. The technique can be used to measure optical absorption coefficient in extremely wide range of frequencies, to get information on thermal properties of a material and as a tool for investigating nonradiative processes in matter. The sample can be in the form of gas, liquid or solid. It can be either bulk solid or in thin film form. The main advantage of this technique is that only a small volume of the sample is required and the detection mechanism is sensitive to weak absorptions.

In the studies reported here the gas microphone detection scheme is employed and the samples are mainly in the form of thin films. The thin film samples selected for investigations find several scientific and industrial applications.

The thesis is divided into seven chapters and the chapter-wise summary of the same is given below.

Chapter 1 gives a general introduction to photothermal phenomena giving emphasis to photoacoustics. A review of the history of photoacoustics and the development of photoacoustic technique is given. The photoacoustic spectroscopy can be used in the investigation of optical and thermal properties of materials and for non-destructive testing. This chapter gives an idea of the various branches of science and technology where PA technique can be used for material characterization. A review of the work done by various researchers using PA technique is also presented.

The **second chapter** describes in detail the absorption of radiation by matter and the subsequent generation of photoacoustic signal in condensed matter. A general theory for the photoacoustic effect in condensed media was formulated by Roscencwaig and Gersho. Later on many improvements were made to the theory by McDonald and Wetsel and others. However, all such modifications are valid only in certain specific cases and these refinements have not changed the basic results of RG theory in most of the experimental situations. The theoretical model of photoacoustic signal generation in condensed media formulated by Rosencwaig and Gersho (RG theory) is presented in this chapter.

Chapter 3 deals with the PA studies on certain ceramic materials. Details of the experimental setup and the open photoacoustic cell used for the studies are discussed in this chapter. PA technique has been used for the thermal characterization of Silicon carbide, alumina-zirconia, zirconia, barium titanate, barium tin titanate, lead magnesium niobate titanate(PMNPT) and lead zirconate titanate(PZT) tapes. Significance and uses of the materials selected for investigations and the details of the tape casting process are discussed. Both reflection and transmission geometry have been used for the evaluation of thermal parameters of the sample and the experimental details are given. The thermal diffusivity,. thermal conductivity, thermal capacity and effusivity of the ceramic tapes have been evaluated and the results are reported.

Chapter 4 describes the use of laser induced photoacoustic technique in the study of photobleaching of Rhodamine 6G and Rhodamine B mixture doped in polymethyl methacrylate (PMMA). Dye mixtures find application as laser media. Thin films are prepared by incorporating Rhodamine 6G and Rhodamine B mixtures into polymethyl methacrylate matrix. Energy transfer from a donor molecule to an acceptor molecule in a dye mixture affects the output of the dye. Details of investigations on the role of laser power, concentration of the dye, modulation frequency and the irradiation wavelength on the photosensitivity of dye mixture film are presented in this chapter. Photosensitivity is found to change with changes in donor acceptor concentrations. Dye mixture doped PMMA samples are found to be more photosensitive when the dyes are mixed in same proportion.

Chapter 5 deals with the non-destructive testing of multilayer coatings. Multilayer dielectric coatings are used as laser mirrors and antireflection coatings. PA technique finds application as an effective tool for nondestructive testing of materials. The feasibility of the PA technique for the characterization of weakly absorbing samples like reflection coatings is demonstrated. Details of the PA cell developed and its calibration are also discussed. Sample can be directly mounted on the cell and from the measurement of PA signal amplitude and phase as a function of modulation frequency of the excitation source; thermal parameters of the sample can be evaluated. Details of the optical sources used and the actual experimental setup used for the PA measurements are given. Photoacoustic signal is found to be sensitive to the presence of a boundary in a layered structure. PA signal generation in highly reflecting multilayer structure is very complicated and hence calculation of thickness and thermal parameters from the PA signal data is not possible. Since phase of PA signal is sensitive to the presence of interphases, an attempt was made on depth profiling. The number of layers present in the multilayer stack and the defective structure present in it are clearly brought out. Through the detection of transmitted and absorbed radiation, reflectance of the film is calculated. The average reflectance value calculated from PA measurement is in close agreement with the value measured using conventional techniques. Experimental details and results are presented.

Chapter 6 discusses the investigations carried out on cresyl violet (CV) incorporated in polymer film. Thin film samples are prepared by incorporating a laser dye- cresyl violet- into polyvinyl alcohol and gelatin matrices. Film preparation method and experimental details are presented in this chapter. Photostability of these samples are investigated using photoacoustic technique. Introductory studies are carried out on CV-gelatin film and detailed studies are carried out on CV-PVA film. Chopping frequency dependence of bleaching of cresyl violet in polyvinyl alcohol due to irradiation by laser light is investigated. The results indicate that photo degradation rates can be controlled by adjusting the modulation frequency. The influences of dye concentration and laser power on photo degradation rate are also discussed.

Chapter 7 gives general conclusions of the present investigations and future prospects of the work.

Papers published

- Photoacoustic study of periodic dielectric multilayer stack. *(Int. J.Optoelectronics, 8,4,493-499,1993)*
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- Photoacoustic studies on bleaching of cresyl violet in polyvinyl alcohol by laser light, National laser symposium, IIT, Madras, 1993.

Papers communicated

- Studies on Photostability of organic dye pairs in PMMA matrix using PA technique. (Communicated to PHOTONICS 2004)

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Annieta Philip K.

Contents

Chapter 1

Introduction

Abstract

This chapter gives a general introduction to the different processes resulting from light matter interaction. Different non-radiative relaxation processes that lead to photo-thermal phenomena in general, giving emphasis to photoacoustic effect, is discussed in detail. A review of the work done by various researchers and the applicability of photoacoustic technique for the characterization of photonic materials are also included.

1.1. Interaction of radiation with matter

Everything we know about light comes from its interaction with matter. As the radiation falls on matter, the electrical vector of radiation interacts with the atoms and molecules of the medium. The nature of interaction depends on the properties of the material. There are a number of processes by which radiation may interact with matter. Some of the phenomena that are likely to occur when electromagnetic radiation passes through matter are dispersion, reflection, refraction, scattering, absorption, polarization etc. The radiations passing through matter are not essentially completely absorbed and it is possible that some of them may pass into matter and undergo scattering or reflection. Sometimes the radiations are neither absorbed nor scattered. In such cases they only undergo changes in orientation or polarization. In some cases the excited molecules formed due to absorption do not de-excite quickly but lose the excess energy only after some time. In such cases, the energy is re-emitted in the form of electromagnetic radiations mostly of larger wavelength than the wavelength of the absorbed radiation. This happens in the case of fluorescence and phosphorescence. When the energy of the radiation corresponds to appropriate values they may be absorbed by matter, electronic, vibrational or rotational energy changes occur. Sometimes a combination of these changes may take place. The absorption of photons by atoms or molecules will result in a series of processes or effects in a material (1-6). When atoms or molecules absorb energy they go to excited states, which are unstable, and give out the excess energy quickly through radiative processes such as spontaneous emission and stimulated emission or through non-radiative relaxation processes. The non-radiative relaxation processes mainly results in heat generation. Depending on the strength of the interacting electric field of the electromagnetic radiation, materials exhibit several linear and non-linear phenomena (7,8).

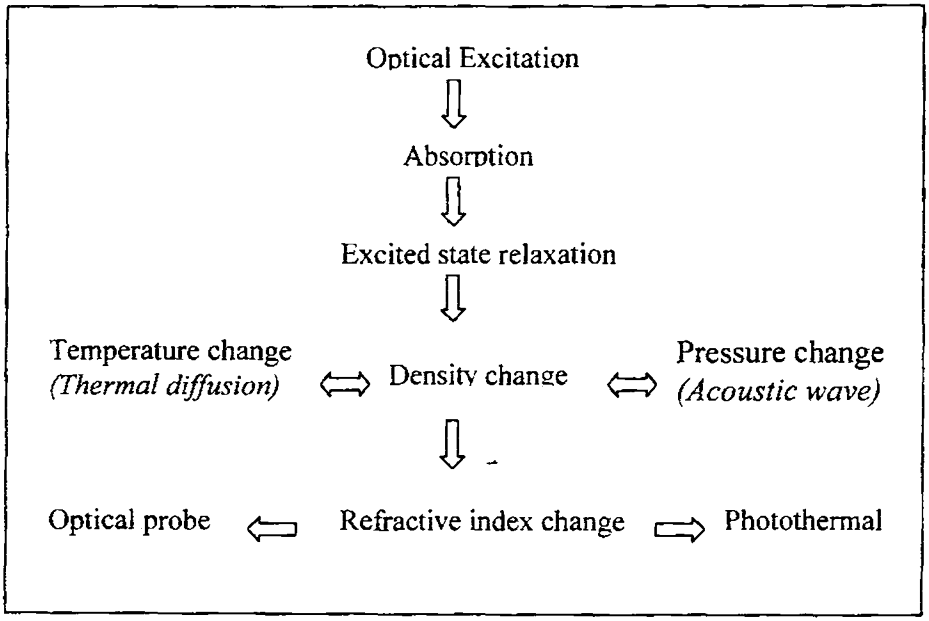

Figure1. Basic processes responsible for the photothermal signal generation.

Destructive changes such as vaporization of the material and plasma generation may take place as a result of photon-matter interaction at very high power densities of the incident light (9-15). When the strength of interacting field is of the order of atomic field, materials show different nonlinear optical properties such as harmonic generation, hyper polarizability, higher order susceptibility etc.(16). If the photon energy is high enough, photochemical changes such as photo-decomposition, photo-ionization etc. may take place (17). Chemical changes may be irreversible or reversible. Measurement of the energy absorbed or released through any of the relaxation channels facilitates the study of various properties and parameters of the sample. During the last two decades, many researchers have explored the non-radiative path of de-excitation of specimen after irradiating with a chopped optical radiation to investigate the thermal, optical, transport and structural properties of materials (18-30).

1.2. Photothermal methods

Thermo optic spectroscopy is a branch of spectroscopy, which has been developed to study those materials that cannot be studied using conventional spectroscopic techniques. The peculiarity of this technique over the conventional spectroscopy is that even though the incident energy is in the form of photons, the interaction of these photons with the material is studied not through subsequent detection and analysis of photons, but through a direct measure of the energy absorbed by the material as a result of its interaction with the light beam. Direct consequence of the non-radiative relaxation is a change in temperature of the sample or the coupling fluid, which is in contact with the sample. If the temperature change occurs in a faster time scale than the time required for the fluid to expand, the rapid temperature change will result in a pressure change in the sample. The non-destructive or non-intrusive photothermal methods are based on the detection of a transient temperature change that characterizes the thermal waves generated in the sample after illumination with pulsed or chopped optical radiation.

Since absorption of electromagnetic radiation is required for the generation of photothermal signal, light that is scattered or transmitted is not detected and hence does not interfere with the photothermal techniques. Another advantage is the capability of obtaining optical absorption spectra of materials that are completely opaque (31, 32). It is a photo-calorimetric method that measures how much of the electromagnetic radiation absorbed by a sample is converted into heat. It can be used to obtain the absorption or excitation spectrum, the lifetime of excited states and the energy yield of radiative processes. It can also be used to measure the thermal and elastic properties of materials, study chemical reactions, measure thickness of thin films and for non-destructive testing of materials (33-43). The magnitude of photothermal signal depends on the specific method used to detect the photothermal effect and on the type of the sample analyzed.

The common detection techniques that are employed in photothermal methods are shown in table.1 Even though all these techniques are based on the same principle, the detected parameter changes from one technique to other.

In photothermal calorimetry, the change in temperature of the sample due to non-radiative magnitude of the photothermal signal depends on the specific method used to relaxation processes is directly measured.(45-50).The temperature change is measured directly by monitoring the infrared emission and it can be used in situations where a large temperature change has occurred. This technique can be used for non-destructive testing and analysis of materials. Thermal imaging of samples can also be carried out using this technique. The major advantage of this technique is that remote sensing of the signal is possible and investigations can be carried out on samples of any shape.

Thermodynamic parameter	**Measured property**	**Detection technique**
Temperature	Temperature	Calorimetry
	Infrared emission	Photothermal radiometry
Pressure	Acoustic wave	Photoacoustic spectroscopy
Density	Refractive index	Photothermal lens
		Photothermal Interferometry
		Photothermal deflection
		Photothermal refraction
		Photothermal diffraction
	Surface deformation	Surface deflection
Pressure	Acoustic wave	Photoacoustic spectroscopy

Table1.Common detection techniques used in photothermal spectroscopy.

In the photopyroelectric method (51-53), simultaneous measurement of different thermal parameters is possible. In this case a pyroelectric sensor is used. When thermal waves reach the pyroelectric sensor, it detects an electric current, which contains information about the structure, thermal and optical properties of the sample. Calibration of the detector is necessary for the accurate measurement of data.

The temperature change due to non-radiative de-excitation can produce changes in the volume and density of the specimen. Photothermal interferometry directly measures the refractive index changes through the

If the pressure change produced in the coupling gas due to the transient temperature change in the sample is measured, the detection scheme is known as opto-acoustic or photo-acoustic technique (68-70).

Two types of pumping mechanisms can be used for the excitation of the sample. They are the pulsed laser excitation and modulated continuous wave (cw) laser excitation. Pulsed excitation produces transient signals of large amplitude immediately after the excitation and it decays as the sample approaches thermal equilibrium through heat diffusion. Usually the transient signals last for few microseconds in gas phase and several milliseconds in condensed matter. By performing the transient waveform analysis, one can obtain a variety of sample properties. If the excitation is carried out using a periodically modulated cw laser, the signal will be periodic and its magnitude and phase will be functions of modulation frequency. The frequency dependent phase-shift information is essentially equivalent to that contained in the time dependent signal transients obtained from pulsed excitation.

1.3. Photoacoustic technique

The term opto-acoustics (OA) or photo-acoustics (PA) usually refers to the generation of acoustic waves by modulated optical radiation. In its broader sense, photoacoustics can mean the generation of acoustic waves or other thermo-elastic effects by any type of energetic radiation, including electromagnetic radiation from radio frequency to X-ray, electrons, protons, ions and other particles. Intensity modulation of the light source is a necessary requirement for the production of PA signal as it is the periodic heating and cooling of the sample, following absorption, which induces the measurable acoustic signal. The absorbed optical energy, which ultimately causes the photoacoustic signal, is that fraction of the total absorbed energy, which is converted to heat via non-radiative de-excitation processes in the sample. It is Alexander Graham Bell who discovered the PA effect in 1880 (71). Later on many theories are developed by several others and the technique has been effectively used by many others in diverse areas of physics, chemistry and medicine (72-81). With the advent of high power lasers, sensitive detection schemes and data acquisition systems, the versatility of PA technique has paved way to several innovative experiments.

The PA generation can be classified as direct or indirect. In direct PA signal generation, the acoustic wave is produced in the sample whereas in the indirect PA signal generation, the acoustic wave is generated in the coupling medium adjacent to the sample. In the direct PA signal generation, acoustic wave generated due to transient temperature change is measured using a piezoelectric transducer kept in contact with the specimen. Since the volume expansion of solids and liquids is less than that of gases, microphone detection scheme is more sensitive for these types of samples. In the case of powdered sample or gels, the piezoelectric detection is not applicable. In such cases, the microphone detection scheme is used. The simple and elegant microphone version of the PA technique can measure a temperature rise of 10^{-6} to 10^{-5} °C . Different versions of microphone based photoacoustic technique are used for the characterization of condensed matter (83-85). Depending on the position of the microphone in the PA cell cavity, the PA technique can be employed in two configurations viz., reflection configuration and transmission configuration, details of which are given in later chapters. The transmission configuration is found to be more useful in evaluating the thermal properties of material (86). The disadvantage of the microphone version of the PA technique is that the response time of the detector is limited by the transit time of the acoustic wave in the PA cell cavity and the low frequency response of the microphone.

1.4. Photonic materials

The advent of advanced lasers has led to significant break-through in basic science and the emergence of key technologies. These serve as the foundation for the development of new photonic materials and devices, as well as novel concepts for storage, display, communication and sensing. Photonic materials are used for generation, guiding, amplification and switching of light. Semiconductors, dielectrics, organic dyes, ceramics etc. are the commonly used materials in photonics industry.

Some of the potential applications of nonlinear optics include quantum computing, quantum communications, quantum imaging, all-optical switching, optical power limiting, and nonlinear optical image processing. However, the implementation of these applications has historically been held back by the limited availability of materials with the required properties of large optical non-linearity combined with high optical transparency and high resistance to laser damage. Several strategies have traditionally been exploited in an attempt to create new materials with more desirable optical properties. These strategies include the synthesis of new chemical compounds with intrinsically large optical response and the creation of composite materials that can combine the desirable characteristics of two or more constituent materials to create a new material with tailored optical properties. Of particular interest is the hope of developing artificial materials and structures with optical properties fundamentally different from those of naturally occurring materials.

Nanocomposite materials are especially well suited for photonics applications because they can be constructed in such a manner as to produce enhanced nonlinear optical response. Some of these materials are formed by the random association constituents, whereas others are formed with deterministic properties through various fabrication methods. But, mere development of a new material with some specific features will not be sufficient for its effective use in any of the applications. A complete characterization of materials is necessary for a comparative study of different materials to identify the most appropriate one. Photonic materials, being directly related to the generation or detection of photons, optical techniques are the most suitable for their characterization. Because of the high sensitivity, photothermal methods can be effectively employed for the characterization of photonic materials.

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