UNIVERSITI PUTRA MALAYSIA

THERMAL DIFFUSIVITY MEASUREMENT USING PHOTOACOUSTIC AND THERMAL LENS TECHNIQUES

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By

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The open photoacoustic cell (OPC) technique was used for measuring thermal diffusivity of solid samples. It is based upon the measurement of the photoacoustic signal as a function of the modulation frequency in the region where the thermal diffusion length equals to the sample thickness. The measurements were carried out at room temperature for samples metal, alloy, semiconductor, polymer and superconductor. The measured thermal diffusivity values for metal, semiconductor and polymer samples are in good agreement with the values reported previously by other researchers. The thermal diffusivity values of the Ag_xAu_{1-x} alloys decrease from 1.48 cm^2/s (pure Ag) to a minimum value at around x = 0.70 before increasing towards the value of 1.28 cm^2/s (pure Au). In $Au_xCu_{(100-x)0.7}Ag_{(100-x)0.3}$ alloy system, the thermal diffusivity values decrease with the increasing of the weight fraction x and reaching a minimum at around x = 90 composition. Then, the thermal

diffusivity values tend to increase to the value of 1.28 cm^2/s for the pure Au metal. For superconductor samples in the normal state, the measured thermal diffusivity decreases with the increase of Zn content in the $Bi_2Pb_{0.6}Sr_2Ca_{2-x}Zn_xCu_3O_\delta$ system, However, the thermal diffusivity values increase with the increasing of Ba dopant in the $Bi_2Pb_{0.6}Sr_2Ca_{2-x}Ba_xCu_3O_\delta$ system.

The OPC detection was also used to monitor the evaporation time of the liquid samples. The evaporation time for 10.60 mm^3 of acetone, chloroform, methanol and ethanol samples were recorded as 236.7 s, 578.8 s, 436.2 s and 869.2 s respectively. The results also show that the liquid evaporation time is inversely proportional to the laser power.

The laser beam power in the range of (2-16) mW was monitored by using OPC, closed photoacoustic cell (CPC) and piezoelectric (PZT) detections. In each case, the photoacoustic (PA) signals were found to be linear up to the laser power of 16 mW. In comparison, the power meter responsivity for CPC detection always higher than OPC and followed by PZT detection.

Finally, the thermal lens technique was used to determine the thermal diffusivity of liquid samples. The phenomenon of thermal lensing is due to the refractive index change with temperature in a liquid medium causes by the periodic photothermal heating. The change of the refractive index will turn the heated medium into a lens. By measuring the time dependence of the laser intensity change after passed though

the thermal lens, the thermal diffusivity of the sample can be obtained. In this work, the measurements were carried out for various solvents, fuel, palm oils and chitosan at different concentration. It was found that the thermal lens technique was suitable for measuring thermal diffusivity value of liquids in the range of $(9.09 \times 10^{-4} - 12.1 \times 10^{-4})$ cm^2/s.

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PENGUKURAN PEKALI RESAPAN TERMA DENGAN MENGGUNAKAN TEKNIK FOTOAKUSTIK DAN TEKNIK TERMA KANTA

Oleh

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Teknik fotoakustik sel terbuka (OPC) ditunjukkan untuk menentukan nilai pekali resapan terma bahan pepejal. Konsepnya adalah berdasarkan pengukuran isyarat akustik sebagai fungsi frekuensi termodulasi di mana jarak peresapan terma sama dengan ketebalan sampel. Pengukuran dibuat pada suhu bilik bagi bahan-bahan logam, aloi, semikonduktor, polimer and superkonduktor. Hasil nilai-nilai ukuran bagi bahan-bahan logam, semikonduktor dan polimer amat menyetujui dengan nilai yang telah dilaporkan oleh penyelidik-penyelidik lain. Nilai-nilai pekali resapan bagi aloi Ag_xAu_{1-x} berkurang dari 1.48 cm^2/s (Ag tulen) ke suatu nilai minima lebih kurang pada $x = 0.70$ sebelum meningat ke 1.28 cm^2/s (Au tulen). Bagi sistem aloi $Au_xCu_{(100-x)0.7}Ag_{(100-x)0.3}$, nilai-nilai pekali resapan berkurangan dengan penambahan nilai nisbah berat x dan mencapai nilai minimanya lebih kurang pada $x = 90$. Kemudian, nilai-nilai ini bertambah sehingga mencapai 1.28 cm^2/s iaitu nilai

pekali resapan bagi sampel Au tulen. Bagi sampel superkonduktor pada keadaan normal, nilai pekali resapan berkurangan dengan penambahan kandungan Zn pada sistem $Bi_2Pb_{0.6}Sr_2Ca_{2-x}Zn_xCu_3O_\delta$. Akan tetapi, nilai pekali resapan bertambah dengan penambahan kanduangan Ba pada sistem $Bi_2Pb_{0.6}Sr_2Ca_{2-x}Ba_xCu_3O_\delta$.

Teknik OPC juga digunakan untuk mengukur masa pengewapan bagi bahan-bahan cecair. Masa pengewapan untuk 10.60 mm^3 acetone, chloroform, methanol dan ethanol masing-masing dicatatkan sebagai 236.7 s, 578.8 s, 436.2 s and 869.2 s. keputusan juga menunjukkan bahawa masa pengewapan adalah berkadar songsang terhadap kuasa laser.

Kuasa alur laser dalam julat (2-16) mW juga dikaji dengan menggunakan teknik OPC, teknik fotoakustik sel tertutup (CPC) dan teknik piezoelektrik (PZT). Dalam setiap kes, isyarat akustik didapati berkadar terus dengan kuasa laser sehingga 16 mW. Secara perbandingan, kuasa tindakan bagi CPC teknik sentiasa lebih daripada teknik OPC and diikuti oleh teknik PZT.

Akhirnya, teknik kanta terma digunakan untuk menentu nilai pekali resapan terma bagi bahan-bahan cecair. Fenomena kanta terma adalah berdasarkan perubahan indek biasan terhadap suhu pada suatu medium cecair akibat pemanasan fotothemal secara berkala. Perubahan indek biasan akan mengubah medium yang telah dipanaskan ke suatu kanta. Oleh itu, dengan mengukur perubahan keamatan laser terhadap masa selepas menembusi kanta terma, pekali resapan terma bagi sampel

cecair boleh diperolehi. Dalam kajian ini, pengukuran telah dilakukan terhadap pelbagai pelarut, bahan api kenderaan, minyak kelapa sawit and chitosan. Teknik kanta terma ini didapati amat sesuai untuk mengukur nilai pekali resapan terma bagi sampel cecair dalam julat $(9.09 \times 10^{-4} – 12.1 \times 10^{-4})$ cm^2/s.

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CHAPTER 1

INTRODUCTION

Photothermal and Photoacoustic

Photothermal science is a cumulative name for a class of phenomena that involve the generation of heat caused by the absorption of modulated optical energy. In fact, when the optical energy is absorbed, the excited states in atoms or molecules loose their excitation energy by a series of non-radiative transitions that result in a general heating in the material.

The early concept of the photoacoustic effect (as cited by Favier, 1997) was discovered by Alexander Graham Bell in 1880 when he tried to explain the operation of his photophone. It was named photoacoustic because the photothermal heating effect was detected by an indirect acoustic method. He also studied the photoacoustic effect in solids, liquids and gases where the modulated light was used to illuminate the sample. Among the scientists who involved in studying this studying phenomena were Rayleigh, Rontgen, Mercadier and Tyndall. Due to the limitation of hearing tubes used as detectors in the early experiments, interest in the field of photoacoustic died down. However, some interesting conclusions could be made. For example, Mercadier who performed photoacoustic spectroscopic studies on various materials, came to the conclusion that the maximum effect was found to be produced by the red rays and by the invisible ultra red rays. Likewise, Rontgen stated that the sounds in question are due to the bending of the plates under unequal heating.

It was only at beginning of the 1970s that the photothermal and photoacoustic research was rediscovered mainly due to three major factors:

1. Development of intense light sources; such as lasers and high pressure arc lamps, such as xenon lamps.
2. Development of fast and sensitive detection equipment; such as electret microphones and piezoelectric detectors.
3. Development of more sensitive signal processing equipment; such as filters, phases sensitive detectors and ultimately lock-in amplifiers.

The improvements in the above three areas enabled the photoacoustic phenomena to be explored and studied further as partly shown in the present work, and hence higher sensitivity and greater selectivity photoacoustic spectroscopy could be performed.

Now, further development of the photoacoustic spectroscopy techniques and their applications become more interesting for measuring the optical properties and thermal characterization of various materials. The attractive features of photoacoustic spectroscopy can be listed as follows:

1. Requires minimal sample preparation
2. Enables measurements of thermal and optical properties on highly absorbing and scattering media
3. Non-contact and non-destructive

4. Measurements can be carried out on a broad range of material (gases, liquids, solids, powders, gels, thin films, etc.)
5. Can be used to determine a very wide range of absorption coefficient magnitudes (10^{-3} to 10^{-5} m^{-1})
6. A range of complimentary photothermal detection methods
7. An increase in signal-to-noise ratio with data processing capability and increasing in input light power.

Photothermal Detection Schemes

The heat generated in sample results in physical changes in and around the sample. Figure 1.1 shows the resulting of the phenomena caused by illumination of a surface by a modulated beam of light. Beside the change in temperature of the sample, it's also produced infrared, acoustic waves, thermal waves, thermoelastic waves; surface expansion, surface reflectivity modulation and refractive index gradient in the medium in contact with the heated surface. All of these effects have been used to probe the photothermal response of an enormous number of materials – solids, liquids and gases.

The thermal wave detection techniques were classified into three groups i.e. acoustic, thermal and optical. Acoustic detection techniques employ either a gas condenser microphone for the detection of pressure variations in air or a piezoelectric transducer for the detection of thermoelastic waves in solid media. Thermal detection methods include the use of thermocouples, thermistors, infrared detectors