UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT AND APPLICATIONS OF PHOTOFLASH-PVDF TECHNIQUE IN THERMAL DIFFUSIVITY MEASUREMENT AT LOW TEMPERATURES

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DEVELOPMENT AND APPLICATIONS OF PHOTOFLASH-PVDF TECHNIQUE IN THERMAL DIFFUSIVITY MEASUREMENT AT LOW TEMPERATURES

By

MEHDI HAYDARI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

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DEDICATION

To my beloved parents

Hossein Haydari & Leila Nikbakht

They are the first and the best in my life

and

My supervisor Professor Dr. Mohd Maarof H. A. Moksin
for his guidance, advice and endless supports

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in Fulfilment of the requirement for the degree of Doctor of Philosophy

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Chairman: Professor Mohd Maarof H. A. Moksin, Ph. D.

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The photoflash technique is developed and used for measuring thermal diffusivity of various types of material, at temperature range from ~77K to ambient temperature. It uses a cheap and simple camera flash and polyvinlidene difluoride (PVDF) film as signal generating source and detector, respectively.

The theoretical signal was derived based on the square pulse approximation of the camera flash that replaced Dirac-δ function approximation employed in other studies. Comparative studies on these two different approximations have been performed on SiC/B_4C composites. Although the camera flash temporal shape is closer to square pulse, Dirac-δ function approximation is still valid for the limited

case of PVDF signal that is significantly longer than camera flash pulse duration.

The square wave approximation model was further used in determining the thermal diffusivity of superconductors, semiconductors, magnetoresistances, carbon nanotubes, ceramics, composites, polymers and porous samples.

The thermal diffusivity for SiC/B_4C composites and SiC doped with Al decreased with increasing temperature. This suggests that thermal diffusivity is basically influenced by phonon interaction within lattice that determines the phonon mean free path.

In case of superconducting materials, thermal diffusivity measurements were carried for BSCCO, doped with Samarium (Sm) at Bi, Sr and Cu sites and sintered for 24, 48 and 100hrs respectively. The results were explained in terms of electron–phonon and phonon-lattice interactions in association with the sample grain size.

The magneto-resistive of LCMO doped with Er at La site was also studied in this study. Thermal diffusivity measurements revealed that the transition from metallic to insulator and from insulator to semiconductor behavior in the materials, were closely matched to the results obtained from electrical resistivity measurement of other researchers.

The thermal diffusivity of carbon nanotubes (CNTs) decreased when the temperature was increased from low to room temperature. Besides, there were also double slope phenomena in the way the thermal diffusivity changed with composition of CNT in the range of temperature covered in the measurement.

In the case of polymers of Emeraldine Base (EB) and Emeraldine Salt (ES), the thermal diffusivity changed with temperature as in other insulating materials.

Finally, the effect of porosity on thermal diffusivity was studied using Nickel Copper Zinc Ferrite samples. The thermal diffusivity decreased with increasing porosity of the sample. The results also showed that porosity has a greater effect on thermal conductivity of the material than its thermal capacity.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN DAN PENGGUNAAN TEKNIK FOTOKILAT-PVDF DALAM PENGUKURAN PERESAPAN TERMA PADA SUHU-SUHU RENDAH

Oleh

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Teknik fotokilat telah dibangunkan dan digunakan untuk mengukur resapan terma yang mempunyai bidang yang lebar daripada suhu rendah ~77K kepada suhu ambien untuk pelbagai jenis bahan. Ia menggunakan 'camera flash' dan filem polyvinlidene difluoride (PVDF) yang murah dan sederhana, masing-masing sebagai sumber penjana dan pengesan isyarat.

Isyarat teori telah diterbitkan berdasarkan penghampiran kepada denyut segi empat untuk camera flash menggantikan penghampiran fungsi Dirac-δ yang biasa diguna pakai dalam kerja-kerja sebelum ini. Kajian perbandingan bagi kedua-dua penghampiran ini telah dijalankan ke atas beberapa sampel komposit SiC/B_4C. Walaupun bentuk tempohan daripada camera flash lebih hampir kepada bentuk denyut segi empat, namun penghampiran fungsi Dirac-δ masih sah digunakan

untuk kes yang terhad kepada isyarat PVDF yang jauh lebih panjang berbanding dengan tempoh denyut kilat camera flash dalam kejian ini.

Model penghampiran bagi gelombang segi empat adalah seterusnya digunakan untuk menentukan pemalar peresapan terma bagi bahan superkonduktor, semikonduktor, magnetoresistan, nanotiub karbon, seramik, polimer dan bahan berliang.

Hasil yang diperolehi untuk komposit SiC/B_4C dan SiC yang didop dengan Al menunjukkan peresapan terma mengecil dengan kenaikan suhu yang mana memberi petunjuk yang resapan terma pada asasnya dipengaruhi oleh saling tindak antara fonon dengan kekisi yang akan menentukan lintasan bebas min untuk fonon.

Dalam hal bahan superkonduktor, pengukuran resapan terma telah dilakukan ke atas BSCCO yang didop dengan Samarium (Sm) pada tapak Bi, Sr dan Cu yang masing-masing disinter selama 24, 48 dan 100 jam. Hasil yang diperolehi dihuraikan dari aspek saling tindak lektron-fonon dan fonon-kekisi yang ada kaitannya dengan dengan sais butir sample.

Sampel magneto-resistif bagi LCMO yang didop dengan Er pada tapak La telah juga dikaji. Hasil pengukuran resapan terma yang diperolehi telah menyerlahkan gelagat peralihan dari logam ke penebat dan dari penebat ke semikonduktor dalam bahan, hamper sama dengan yang diperolehi dari hasil pengukuran rintangan elektrik yang dilakukan oleh orang lain.

Sampel karbon nanotiub (CNT) menunjukkan resapan terma dari suhu rendah ke suhu bilik adalah mengecil dengan kenaikan suhu sebagaimana yang berlaku pada bahan penebat yang lain. Disamping itu terdapat fenomena dua cerun bagaimana resapan terma berubah dengan komposisi CNT pada bidang suhu yang diliputi dalam pengukuran ini.

Dalam hal polimer Emeraldine Base (EB) dan Emeraldine Salt (ES), resapan terma berubah dengan suhu sebagaimana dalam bahan penebat yang lain.

Akhir sekali, kesan liang pada resapan terma telah dikaji dengan menggunakan sampel Nickel Copper Zinc Ferrite. Resapan termanya menurun dengan kenaikan keliangan sampel. Hasil pengukuran juga menunjukkan keliangan memberi kesan yang lebih besar kepada konduktiviti terma bahan dibandingkan dengan kapasiti termanya.

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LIST OF ABBREVIATIONS

EB	Emeraldine Base
El	Electron
Er	Erbium
ES	Emeraldine Salt
Fe	Iron
FM	Ferromagnetic Metal
I	Insulator
IR	Infra Red
La	Lanthanum
latt	Lattice
LCMO	La-Ca-Mn-O system
M	Metal
Mn	Manganese
N.A	Normalized Amplitude
Ni	Nickel
O	Oxygen
OPC	Open Photoacoustic Cell
OTTER	Optotheraml Transient Emission Radiometry
PD	Photodeflaction technique
P^2E	Photopyroelectric effect
PA	Photoacoustic
PM	Paramagnetic

PVDF	Polyvinlidene Difluoride
SEM	Scanning Electron Microscopy
SiC	Silicon Carbide
Sm	Samarium
Sr	Strontium
TC	Temperature Controller
Y	Yttrium
YBCO	Y-Ba-Cu-O system
Zn	Zinc
AFM	Antiferromagnetic
B_4C	Boron Carbide
Ba	Barium
BCS theory	Bardeen, Cooper and Schrieffer theory
Bi	Bismuth
BSCCO	Bi-Sr-Ca-Cu-O system
C	Carbon
Ca	Calcium
CMR	Colossal Magnetoresistance
CNTs	Carbon Nanotubes
CPC	Closed Photoacoustic Cell
Cu	Copper
Cu	Copper
CW	Continues wave

LIST OF SYMBOLS

t_c	Characteristic rise time of the rear face temperature
θ	Debye temperature of the lattice
ρ	Density
ε	Dielectric constant
δ	Dirac symbol
ε	Emissivity of the surface
E_0	Energy/unit area absorbed in the materia!
$\vec{q}$	Heat flow
Q	Heat pulse energy
θ_0	Initial temperature rise at the surface
$\beta(\lambda)$	Optical absorption coefficient
$A(\lambda)$	Optical absorption length
l_{Phonon}	Phonon free path
υ_{Phonon}	Phonon velocity
τ	Pulse time duration
σ	Stefan's constant
$\vec{\nabla}T$	Temperature gradient
ℓ	The distance, where temperature reaches to maximum at a time ξ after the excitation
α	Thermal diffusivity
ξ	Thermal transit time