UNIVERSITI PUTRA MALAYSIA

THERMAL DIFFUSIVITY AND ELECTRICAL CONDUCTIVITY STUDIES OF POLYANILINE BASED MATERIALS AND SELECTED CERAMICS

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THERMAL DIFFUSIVITY AND ELECTRICAL CONDUCTIVITY STUDIES OF POLYANILINE BASED MATERIALS AND SELECTED CERAMICS

By

JOSEPHINE LIEW YING CHYI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Master of Science

May 2003

DEDICATION

To my beloved parents Vincent Liew and Jennifer Tan

for their love and concern……

To my beloved Richard Koo Wee Yeow

for his love, support, understanding and care……

To my friends (too many)

for their wonderful encouragement and support……

To my lecturer Prof. W. Mahmood bin Mat Yunus

For his guidance, advice, understanding and endless support……

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairman: Professor W. Mahmood Mat Yunus, Ph.D.

Faculty: Science and Environmental Studies

In this work, the photoflash and four–point probe techniques were respectively applied for thermal diffusivity and electrical conductivity measurement on polyaniline, polyaniline blends, polyaniline composite and ceramic at room temperature.

In the photoflash technique, the signal was initially generated by a high intensity camera flash as an excitation source. A fast response K–type thermocouple was used as a detector to monitor the temperature at the rear surface of the sample. The photoflash signal was captured as a function of time and the half rise time; $t_{0.5}$ of each sample was then analyzed to determine the thermal diffusivity. The photoflash setup was first calibrated with the sample of known thermal diffusivity. The results indicate that the thermal diffusivity of the calibration sample correlated well with the results from literature.

For the four–point probe technique, the electrical conductivity measurement was carried out on emeraldine salt (ES), polyaniline blends (ES/PMMA), polyaniline composite (ES/ZnO) and doped polyaniline (EB/ZnO doped with acid H_2SO_4) sample. By applying a constant current source to pass a steady current through the two outer probes, the voltage drop across the two inner probes was measured. When I–V characteristic curve was plotted, the gradient of the I–V curve was used to calculate the electrical conductivity value of the samples.

The effect of particle size, applied pressure, heat treatment temperature and composition of samples of polyaniline, polyaniline blends, polyaniline composite and doped polyaniline composite on the thermal diffusivity and electrical conductivity value was investigated in detail. It was found that the thermal diffusivity and electrical conductivity values of the sample is dependent on the particle size, applied pressure, heat treatment temperature and composition. The way that the thermal diffusivity behaves towards the change of dopant concentration and sintering time was also investigated for ceramic sample. The measured thermal diffusivity value of the ceramic samples was found to be very dependent on the dopant atom and dopant concentration but not dependent on the sintering time.

Since the variation of thermal diffusivity and electrical conductivity can not be concluded, the Fourier Transform Infrared (FTIR) spectra, X–Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were carried out in order to support and explain the changes of the thermal diffusivity and electrical conductivity result.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

KAJIAN RESAPAN TERMA DAN KEKONDUKSIAN ELEKTRIK PADA BAHAN BERDASARKAN POLYANILINE DAN SERAMIK TERPILIH

Oleh

JOSEPHINE LIEW YING CHYI

Mei 2003

Pengerusi: Profesor W. Mahmood bin Mat Yunus, Ph.D.

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Dalam ujikaji ini, teknik sinaran lampu kilat dan penduga empat titik masing–masing telah digunakan untuk pengukuran resapan terma dan kekonduksian elektrik polyaniline, adunan–adunan polyaniline, komposit polyaniline dan seramik pada suhu bilik.

Dalam teknik sinaran lampu kilat, isyarat awal telah dihasilkan oleh kamera lampu kilat yang berkeamatan tinggi sebagai satu sumber pengujaan. Satu termogandingan jenis K yang bertindak balas cepat telah dipilih sebagai satu pengesan untuk mengukur suhu pada permukaan belakang sampel. Isyarat lampu kilat ini telah diambil sebagai satu fungsi kepada masa dan masa setengah hayatnya, $t_{0.5}$ daripada setiap sampel akan dianalisis untuk menentukan nilai resapan terma. Susunan peralatan sinaran lampu kilat telah ditentusahkan mula–mula dengan menggunakan sampel yang diketahui nilai resapan termanya. Keputusan yang diperolehi menunjukkan bahawa nilai resapan terma yang diperolehi daripada sampel tentukur bersetuju baik dengan keputusan yang dilaporkan dalam kajian lepas.

Bagi teknik penduga empat–titik, pengukuran kekonduksian elektrik telah ditumpukan pada emeraldine salt (ES), adunan–adunan polyaniline (ES/PMMA), komposit polyaniline (ES/ZnO) dan polyaniline dop (ES/ZnO didop dengan asid sulfurik). Dengan menggunakan sumber arus tetap untuk mengalirkan satu arus yang stabil melalui dua titik luar, kejatuhan voltan melalui 2 titik bahagian dalam akan diukur. Apabila graf pencirian I–V dilukis, nilai kecerunan graf I–V akan digunakan untuk mengira nilai kekonduksian elektrik sampel–sampel.

Kesan saiz zarah, tekanan gunaan, suhu rawatan haba dan komposisi sampel–sampel polyaniline, adunan–adunan polyaniline, komposit polyaniline dan polyaniline dop pada nilai resapan terma dan kekonduksian elektrik telah dikaji secara teliti. Didapati bahawa nilai resapan terma dan kekonduksian elektrik bagi sampel yang dikaji adalah sangat bergantung pada saiz zarah, tekanan gunaan, suhu rawatan haba dan komposisi. Cara resapan terma bertindak balas terhadap perubahan kepekatan pendopan dan masa pensinteran juga diselidik bagi sampel seramik. Niai resapam terma terukur pada sampel seramik telah didapati sangat bergantung pada atom pendopan dan kepekatan pendopan tetapi tidak bergantung kepada masa pensiteran.

Disebabkan perubahan resapan terma dan kekonduksian elektrik tidak boleh dirumuskan, spektra Transformaasi Fourier Inframerah (FTIR), Belauan Sinar–X (XRD) dan Pengimbasan Mikroskop Elektron (SEM) telah dijalankan untuk menyokong dan menerangkan perubahan–perubahan yang berlaku pada keputusan resapan terma dan kekonduksian elektrik.

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I am gratefully acknowledge the award of the PASCA Scholarship from the Universiti Putra Malaysia, which enable me to undertake this work. Last but not least, my sincere thanks to all my friends, seniors and family members, who involved directly or indirectly towards the success of this project.

I certify that an Examination Committee met on 30th May 2003 to conduct the final examination of Josephine Liew Ying Chyi on her Master of Science thesis entitled "Thermal Diffusivity and Electrical Conductivity Studies of Polyaniline, Polyaniline Blends, Polyaniline Composite and Ceramic Samples" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as partial fulfilment of the requirements for the degree of Master of Science. The members of the Supervisory Committee are as follows.

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

JOSEPHINE LIEW YING CHYI

Date: 11/9/2003

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LIST OF ABBREVIATION

–B–	benzenoid ring
=Q=	quinoid ring
AFM	Atomic Force Microscopy
ANICSA	aniline camphorsulfonate
ANIHCl	aniline hydrochloride
Au	Gold
B_4C	Boron Carbide
B_c	cis benzenoid unit
Bi	Bismuth
BSCCO	Bi–Sr–Ca–Cu–O system
C	Carbon
$C_6H_5NH_2$	aniline
Ca	Calcium
Cu	Copper
Cu-O	Copper Oxide
DC	Direct Current
DMTA	Dynamic Mechanical Tensile Analysis
DSC	Differential Scanning Calorimetric
DSO	Digital Storage Oscilloscope
DVM	Digital Voltmeter
EB	Emeraldine Base
EB/PMMA	Emeraldine Base blends with Poly(methyl methacrylate)
EB/ZnO	Composites of Emeraldine Base and Zinc Oxide
ECA	Electrochemical–Assembly

ECP	Electrically Conductive Polymer
ES	Emeraldine Salt
ES/PMMA	Emeraldine Salt blends with Poly(methyl methacrylate)
ES/ZnO	Composites of Emeraldine Salt and Zinc Oxide
ESR	Electron Spin Resonance
FTIR	Fourier Transform Infra–Red
GPIB	General Purpose Interface Bus
H_2SO_4	Acid Sulfuric
HCl	Acid Hydrochloric
HP	Hydraulic Press
HRP	Horseradish Peroxidase
HTSC	high–T_c superconductor
IBM	International Business Machines Corporation
ICP	Intrinsically Conductive Polymers
IR	Infrared
KBr	Potassium Bromide
La	Lanthanum
LB	Leucoemeraldine Base
MMA	methylmethacrylate
MOR	Modulated Optical Reflectance
N	Nitrogen
Nd	Neodymium
Nd/YAG	Neodymium (3+) – doped Yttrium Aluminum Ganet
NH_3	Ammonia
NMP	N–methylpyrrolidinone