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ENHANCEMENT OF FIELD ELECTRON EMISSION PROPERTIES OF CARBON NANOTUBES/ZINC OXIDE NANOCOMPOSITES USING SINGLE AND MULTI-STEP METHODS

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## THESIS SUBMITTED IN FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF MASTER OF SCIENCE (MASTER BY RESEARCH)

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#### ABSTRACT

This study aimed to enhance the field electron emission (FEE) properties of carbon nanotubes (CNTs) synthesized from waste cooking palm oil combined with zinc oxide (ZnO) to produce CNTs/ZnO nanocomposites. The methods used in this study were single and multi-step depositions. The single-step deposition method was done by directly mixing the CNTs and ZnO precursors and they were synthesized using thermal chemical vapor deposition (TCVD) method for 30 minutes. Meanwhile, the multi-step deposition process was carried out by combining TCVD and sol-gel immersion methods to fabricate CNTs/ZnO nanocomposites. There were three different ZnO nanostructures namely nanorods, nanoflowers and nanorods-nanoflakes which were composited with CNTs via multi-step deposition process. The obtained samples were analyzed using electron microscopy, energy dispersive X-ray, microdiffraction spectroscopy, photoluminescence spectroscopy, X-ray Raman spectroscopy and four-point probe current-voltage measurement. The field emission properties of the samples were also studied using FEE measurement. The findings showed that the turn-on and threshold fields of CNTs/ZnO nanocomposites decreased as compared to pristine CNTs. Other than that, different nanostructures of ZnO contributed to the FEE performance of CNTs/ZnO nanocomposites. The best FEE properties were given by the growth of CNTs on ZnO nanoflowers, which has the lowest turn-on field of 0.8 V/ $\mu$ m at current density of 1  $\mu$ A/cm<sup>2</sup> and a high field enhancement factor of 9417. Larger emission site density and lower screening effect in this sample were believed to affect the FEE performance. As a conclusion, the fabrication of CNTs/ZnO nanocomposites have successfully enhanced the FEE properties of CNTs. Implication of this study is that it provide a new insight on advancing the synthesis of CNTs/ZnO nanocomposites for electron emission devices.

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### PENINGKATAN SIFAT PEMANCARAN ELEKTRON MEDAN BAGI NANOKOMPOSIT NANOTIUB KARBON/ZINK OKSIDA MENGGUNAKAN KAEDAH TUNGGAL DAN BERPERINGKAT

### ABSTRAK

Kajian ini bertujuan meningkatkan sifat pemancaran elektron medan (PEM) nanotiub karbon (NTK) yang disintesis menggunakan minyak masak terpakai dengan menggabungkan zink oksida (ZnO) bagi menghasilkan nanokomposit NTK/ZnO. Kaedah yang digunakan bagi kajian ini adalah pemendapan tunggal dan berperingkat. Kaedah pemendapan tunggal telah dilakukan dengan mencampurkan secara langsung prekursor NTK dan ZnO kemudian disintesis menggunakan kaedah pemendapan wap kimia terma (PWKT) selama 30 minit. Sementara itu, proses pemendapan berperingkat dilakukan dengan menggabungkan kaedah PWKT dan rendaman sol-gel untuk fabrikasi nanokomposit NTK/ZnO. Terdapat tiga struktur nano ZnO yang berbeza iaitu nanorod, nanobunga dan nanorod-nanoemping yang telah dikompositkan dengan NTK melalui proses pemendapan berperingkat. Sampel yang dihasilkan dianalisis menggunakan mikroskop elektron, analisis penyerakan tenaga sinar-X, spektroskopi mikro-Raman, spektroskopi pembelauan sinar-X, spektroskopi kefotopendarcahayaan dan pengukuran prob empat titik arus-voltan. Sifat pemancaran medan daripada sampel juga telah dikaji melalui pengukuran PEM. Dapatan kajian menunjukkan bahawa nilai bagi medan permulaan dan medan ambang nanokomposit NTK/ZnO menurun berbanding dengan NTK tulen. Selain itu, struktur nano ZnO yang berbeza menyumbang kepada prestasi PEM nanokomposit NTK/ZnO. Sifat PEM yang terbaik diberikan oleh pertumbuhan NTK di atas ZnO nanobunga yang mempunyai medan permulaan terendah iaitu 0.8 V/µm pada ketumpatan arus 1  $\mu$ A/cm<sup>2</sup> dan faktor peningkatan medan yang tinggi iaitu 9417. Kesimpulannya, fabrikasi nanokomposit NTK/ZnO telah berjaya meningkatkan sifat PEM NTK. Implikasi kajian adalah ianya memberi gambaran baharu bagi pengembangan sintesis nanokomposit NTK/ZnO sebagai peranti pemancar elektron.

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### TABLE OF CONTENTS

	Pages
DECLARATION	ii
ACKNOWLEDGEMENTS	iii
ABSTRACT	iv
ABSTRAK	v
TABLE OF CONTENTS	vi
LIST OF TABLES	xiii
LIST OF FIGURES	XV
LIST OF SYMBOLES AND ACRONYMS	xxiv
CHAPTER 1 INTRODUCTION	
1.1 Introduction	1
1.2 Research Background	2
1.3 Research Problems	5
1.4 Research Objectives	8
1.5 Scope of Studies	9
1.6 Significance of Research	10
1.7 Thesis Organization	11
	DECLARATION ACKNOWLEDGEMENTS ABSTRACT ABSTRAK TABLE OF CONTENTS CALE OF TOBLES LIST OF TABLES LIST OF FIGUES AND ACRONYMS CHAPTER 1 INTRODUCTION 1.1 Introduction 1.2 Research Background 1.3 Research Problems 1.4 Research Objectives 1.5 Scope of Studies 1.6 Significance of Research 1.7 Thesis Organization

### CHAPTER 2 LITERATURE REVIEW

### 2.1 Introduction

12

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIV**Ü**SITI PENDIDI N IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI F

	2.2	Introduction to Carbon Nanotubes	
		2.2.1 Structures and Properties of Carbon Nanotubes	13
		2.2.2 Synthesis of Carbon Nanotubes	18
		2.2.3 Waste Cooking Palm oil as Carbon Precursor for	20
		Carbon Nanotubes Production	
	2.3	Introduction to Zinc Oxide Nanostructures	
		2.3.1 Structures and Properties of Zinc Oxide	22
		Nanostructures	
		2.3.2 Synthesis of Zinc Oxide	26
	2.4	Introduction to Carbon Nanotubes/Zinc Oxide	
		Nanocomposite	
		2.4.1 Synthesis of Carbon Nanotubes/Zinc Oxide	31
		Nanocomposite	
		2.4.2 Application of Carbon Nanotubes/Zinc Oxide	34
		Nanocomposite	
	2.5	Characterization of Carbon Nanotubes/Zinc Oxide	
		Nanocomposite	
		2.5.1 Field Emission Scanning Electron Microscope	35
		2.5.2 Micro-Raman Spectroscopy	36
		2.5.3 X-Ray Diffraction	37
		2.5.4 Photoluminescence	39
		2.5.5 Four-Point Probe Current-Voltage Measurement	40
	2.6	Field Electron Emission	
		2.6.1 Theory of Field Electron Emission	42

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI FENDIDIKAN SULTAN IDRIS KAN SULTAN IDRIS KAN

		2.6.2	Applica	tion of	Carbon	Nanot	ubes/zi	nc Oxid	e	45
			Nanoco	mposite a	as Emitte	er				
			2.6.2.1	Carbon	Nanotu	bes in	Field	Electro	n	45
				Emissic	on Applic	cation				
			2.6.2.2	Zinc Ox	tide in Fi	ield Ele	ectron E	Emission		48
				Applica	tion					
			2.6.2.3	Carbon	Na	notube	/Zinc	Oxid	e	50
				Nanoco	mposite	in	Field	Electro	n	
				Emissic	on Applic	cation				
	2.7	Summ	nary							52
CHAPTE	ER 3	METH	HODOLC	OGY						
	3.1	Introd	uction							53
	3.2	Substr	ates Prep	aration						54
	3.3	Synthe	esis of Ca	arbon Nai	notubes					54
	3.4	Synthe	esis of C	arbon Na	anotubes	Zinc (	Oxide V	Via Singl	e	58
		Step P	rocess							
	3.5	Synthe	esis of Ca	arbon Na	notubes/Z	Zinc O	xide via	a Multipl	e	58
		Steps	Process							
		3.5.1	Synthes	is of Zine	c Oxide I	Nanost	ructures	8		59
			3.5.1.1	Synthes	is of Zir	nc Oxi	de Nan	orods an	d	59
				Nanoflo	owers o	on M	lagnesiu	um Zin	с	
				Oxide S	leeded C	atalyst				
			3.5.1.2	Synthes	is of Z	Zinc C	Dxide 1	Nanorods	-	62
IKAN SUI	TAN 1	IDRIS	UNIV	/FRSITI F	FNDIDI	AN SI	JITAN	IDRIS	UN	IVFF

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			Nanoflakes on	Aluminum Zinc	Oxide	
			Seeded Catalys	t		
		3.5.1.3	Synthesis of Z	inc Oxide Nano	flowers	64
			on Silicon Diox	ide Seeded Catal	yst	
	3.5.2	Formatio	on of Carbon	Nanotubes/Zinc	Oxide	67
		Nanocor	nposite			
	3.5.3	Spin Co	ated Carbon Na	nnotubes on Zinc	oxide	68
		Nanoflo	wers Grown on	Magnesium Zinc	oxide	
		Seeded (	Catalyst			
3.6	Sampl	e Charact	erization			
	3.6.1	Field En	nission Scanning	Electron Micros	cope	68
	3.6.2	Transmi	ssion Electron M	licroscope		69
	3.6.3	Micro-R	Raman Spectrosc	ору		70
	3.6.4	X-Ray D	Diffraction			71
	3.6.5	Photolur	ninescence Spec	troscopy		71
	3.6.6	Four Poi	nt Probes Curren	nt-Voltage Measu	rement	72
	3.6.7	Field Ele	ectron Emission	Measurement		73

3.7 Summary 76

# CHAPTER 4 RESULTS & DISCUSSIONS

4.1	Introduction						
4.2	Preparation	of	Carbon	Nanotubes/Zinc	Oxide	78	
	Nanocomposi	tes S	ynthesized	Using Single and	Multi-		

# Step Deposition Process

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDID N IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI F

- 4.2.1 Synthesis of Carbon Nanotubes/Zinc Oxide 78Nanocomposites Using Single-Step DepositionProcess
- 4.2.2 Synthesis of Zinc Oxide-Coated Carbon 80
  Nanotubes Nanocomposites Via Multi-Step
  Deposition Process Using Magnesium Zinc
  Oxide Seeded Catalyst
- 4.2.3 Synthesis of Carbon Nanotubes-Coated Zinc83Oxide Using Multi-Step Deposition ProcessUsing Magnesium Zinc Oxide Seeded Catalyst
- 4.2.4 Structural and Electrical Properties of Carbon 86Nanotubes, Zinc Oxide and Their CompositeMaterials
- 4.2.5 Field Electron Emission of Carbon 96 Nanotubes/Zinc Oxide Synthesis via Single- and Multi-Step Deposition Process
- 4.3 The Effect of Different Nanostructured Zinc Oxide on 104Properties and Field Electron Emission Performance ofZinc Oxide/Carbon Nanotubes Nanocomposites
  - 4.3.1 Combination of Different Zinc Oxide/Carbon 104Nanotubes Nanocomposites Via Multi-StepDeposition Process
    - 4.3.1.1 Synthesis of Zinc Oxide 105 Nanoflowers/Carbon Nanotubes Using

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDID N IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI FENDIDIKAN SULTAN IDRIS UNIVERSITI F

> Magnesium Zinc Oxide Seeded Catalyst

- 4.3.1.2 Synthesis of Zinc Oxide Nanorods Nanoflakes/ Carbon Nanotubes Using
   Aluminum Zinc Oxide Seeded Catalyst
- 4.3.2 Structural and Electrical Properties of Different 112
  Zinc Oxide/Carbon Nanotubes on Magnesium
  Zinc Oxide and Aluminum Zinc Oxide Seeded
  Catalyst
- 4.3.3 Field Electron Emission Properties of 121
  Nanostructured Zinc Ozide/Carbon Nanotubes
  Nanocomposites Grown on Magnesium Zinc
  Oxide and Aluminum Zinc Oxide Seeded
  Catalysts
- 4.4 Effect of Different Seeded Catalyst on Growth of Zinc 128Oxide Nanoflowers/Carbon Nanotubes and Its FieldElectron Emission
  - 4.4.1 Synthesis of Zinc Oxide Nanoflowers/Carbon 128Nanotubes on Different Catalysts of MagnesiumZinc Oxide and Silicon Dioxide
  - 4.4.2 Structural and Electrical Properties of Zinc 132
    Oxide Nanoflowers/Carbon Nanotubes
    Nanocomposites Grown on Magnesium Zinc
    Oxide and Silicon Dioxide Seeded Catalyst

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIV**XÜ**SITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI P

	4.4.3 F	Field Electro	on Emission	of Zinc	c Oxide	138
	Ν	Vanoflowers/	Carbon Nanotu	ibes on Ma	agnesium	
	Z	Zinc Oxide	and Silicon	Dioxide	Seeded	
	C	Catalysts				
4.5	Growth	Mechanism	of Zinc Oxi	de-Coated	Carbon	143
	Nanotub	es				
4.6	Synthesi	s of Spin Coa	ted Zinc Oxide	/Carbon N	anotubes	149
	Nanocon	nposites Via I	Multi-Step Dep	osition Pro	ocess	
4.7	Summar	у				154
CHAPTER 5	CONCL	USION AND	FUTURE WO	RK		156

REFERENCES	160
APPENDIX	177

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI FENDIDIKAN SULTAN IDRIS KAN SULTAN IDRIS KAN

### LIST OF TABLES

Table		Pages
2.1	Physical properties of wurtzite ZnO structure at $T = 300$ K	23
2.2	FEE characteristic of CNTs investigated by many researchers	46
2.3	FEE properties of ZnO nanostructures	49
2.4	FEE properties of CNTs/ZnO nanocomposites	51
3.1	The chemical used in this study for the growth of ZNRs and	62
	ZNFs on the MgZnO seeded catalyst	
3.2	The chemical used for the production of Al doped ZNRs-FLs	63
	on AlZnO seeded catalyst	
3.3	The chemical used to synthesis ZNFs on SiO <sub>2</sub> seeded catalyst	66
4.1	Micro-Raman peak position and $I_D/I_G$ ratio of pristine CNTs	87
	and CNTs/ZnO synthesized via single and multi-step	
	deposition	
4.2	XRD peaks position of pristine CNTs and CNTs/ZnO	90
	nanocomposites	
4.3	The electrical properties of pristine CNTs, ZnO and	96
	CNTs/ZnO nanocomposites	
4.4	FEE properties of CNTs and CNTs/ZnO nanocomposite via	103
	single and multi-step deposition	
4.5	Diameter of nanostructured pristine ZnO and ZnO/CNTs	110
	nanocomposite	
4.6	The $I_D/I_G$ ratio of CNTs grown on different ZnO	116
	nanostructures	

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNI**VER**SITI PENDID

N IDRIS UNI\

4.7	The XRD peaks position of the ZnO/CNTs nanocomposite	118
	grown on different ZnO nanostructures	
4.8	The electrical properties of ZnO/CNTs nanocomposites	120
	grown on different ZnO nanostructures	
4.9	FEE properties of ZnO/CNTs nanocomposite on different	123
	ZnO nanostructures	
4.10	The diameter of pristine ZNFs and ZNFs/CNTs grown on	131
	MgZnO and SiO <sub>2</sub> seeded catalysts	
4.11	The peaks position and $I_D/I_G$ ratio of the pristine ZNFs, CNTs	135
	and ZNFs/CNTs grown on different seeded catalysts	
4.12	The peaks position of XRD analysis of ZNFs/CNTs	136
	nanocomposite grown on different seeded catalysts	
4.13	The conductivity of ZNFs/CNTs nanocomposites grown on	138
	different seeded catalysts	
4.14	FEE properties of ZNFs/CNTs grown on different seeded	141
	catalysts	
4.15	The FEE properties of ZNFs/CNTs samples obtained via	154
	multi-step deposition by directly depositing process and spin	
	coating-assisted deposition process	

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIV**x**esiti pendid

### LIST OF FIGURES

Figures				
2.1	(a) Illustration of $sp^2$ hybridization of graphene which consist	14		
	of $\sigma$ and $\pi$ bonds (Jorio, M. Dresselhaus, Saito, & G.			
	Dresselhaus, 2010) and (b) structure of SWCNTs (left) and			
	MWCNTs (right).			
2.2	The labelled atoms on the graphene layer. The (n,m) notation	15		
	and unit vectors of two-dimensional lattice are shown (P. J.			
	Harris & P. J. F. Harris, 2001).			
2.3	Structure of the SWCNTs with different chirality types	16		
	(a) armchair (b) zigzag and (c) chiral.			
2.4	Illustration of bottom-up growth mechanism of CNTs produced	21		
	from WCPO (Suriani et al., 2010).			
2.5	Hexagonal wurtzite structure of ZnO. Oxygen atom is shown as	22		
	larger white sphere, while the smaller and dark represent zinc			
	atom (Vaseem et al., 2010).			
2.6	The local-density appoximation band structure of bulk wurtzite	24		
	ZnO (Vogel, Krüger, & Pollmann, 1995).			
2.7	The schematic band diagram of red emission from ZnO (V.	27		
	Kumar et al., 2013).			
2.8	(a) Tunneling electron microscope image, and (b) high and (c)	32		
	low magnification of SEM image of MWCNTs coated ZnO			
	nanoparticles (Klanwan et al., 2010).			

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDID

2.9	The FESEM images of MWCNTs-drop casted on the aligned	34
	ZnO nanowires. By increasing the number of drops, more	
	densely MWCNTs were deposited and covered the ZnO	
	nanowires (Dutta & Basak, 2009).	
2.10	Schematic diagram of Bragg diffraction from the crystalline	37
	lattice plane when exposed with X-rays (Ashcroft & Mermin,	
	2005).	
2.11	The XRD pattern of (a) pristine CNTs, (b) pristine ZnO and (c)	38
	CNTs/ZnO nanocomposite (Klanwan et al., 2013).	
2.12	The schematic diagram of four points probe which is contacted	41
	on the substrate for the <i>I-V</i> measurement.	
2.13	The I-V curve of SiC sample measured using four-point probe	42
	(Chandra et al., 2011).	
2.14	The potential energy diagram for electrons at a metal surface	43
	with the presence of applied electric field (Olson, 1999).	
2.15	(a) Typical J-E curve to determine turn-on and threshold field	45
	and (b) the F-N plot of the ZnO nanowires sample (Yao et al.,	
	2011).	
3.1	Schematic of the experimental setup for the growth of CNTs	55
	consist of TCVD furnace and argon gas. The position of	
	precursor and substrate were also illustrated.	
3.2	Preparation of precursor and catalyst for the CNTs production.	56
3.3	Two-stage furnace used to synthesize CNTs.	57

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNI**XVI**SITI PENDID

N IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS

3.4	(a) Spin coater to deposit MgZnO and (b) water bath used for	60
	immersion process to synthesize ZnO.	
3.5	Schematic of the sol-gel-assisted immersion method for the	61
	growth of ZNRs and ZNFs.	
3.6	Schematic views of oxidation system of Si substrate to fabricate	64
	SiO <sub>2</sub> thin layer using single stage furnace.	
3.7	Schematic diagram of the oxidizing procedure, involving	65
	heating, oxidation and cooling process.	
3.8	Illustration of ZNFs on SiO <sub>2</sub> production via immersion method.	66
3.9	Illustration of (a) CNTs-coated ZnO configuration and (b) ZnO-	67
	coated CNTs configuration.	
3.10	FESEM used to investigate the morphology of as-prepared	69
	samples.	
3.11	Micro-Raman spectroscopy was performed to analysis the	70
	scattering ability of as-prepared samples.	
3.12	PL spectrometer used for the optical properties investigation.	72
3.13	Four point probe setup for the <i>I-V</i> measurement.	73
3.14	Schematic diagram of FEE measurement system.	74
3.15	The flow chart of all-prepared sampels in this study.	75
4.1	(a)-(b) top view and (c)-(d) side view of CNTs/ZnO	79
	nanocomposite produced via single-step TCVD method, (e) top	
	and (f) side view observation of pristine CNTs.	
4.2	EDX spectra of CNTs/ZnO nanocomposite obtained via single-	80
	step TCVD method.	

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- 4.3 FESEM images of (a) VACNTs on Si substrate, (b) uneven
  MgZnO seeded catalyst on the VACNTs, (c) the growth of
  ZNFs on ZNRs by using VACNTs as the template and (d) side
  view observation of ZnO-coated CNTs nanocomposite.
- 4.4 Illustration of ZnO-coated CNTs with MgZnO seeded catalyst. 82
- 4.5 FESEM images of (a) MgZnO thin layer as seeded catalyst, (b)
  84 top view and (c) side view observation of ZNRs grown on
  MgZnO seeded catalyst, (d)–(e) as-grown CNTs on the ZNRs and (f) side view observation of CNTs-coated ZnO grown on
  MgZnO seeded catalyst.
- 4.6 The EDX spectra of (a) pristine ZNRs and (b) CNTs-coated 85 ZnO on the MgZnO seeded catalyst.
- 4.7 Micro-Raman spectra of pristine CNTs, CNTs/ZnO fabricated 86 via single-step TCVD method, ZnO-coated CNTs and CNTs-coated ZnO fabricated via multi-step deposition.
- 4.8 Micro-Raman spectra ranged from 100-1000 cm<sup>-1</sup> of 88 CNTs/ZnO synthesized via single-step deposition (left) and CNTs-coated ZnO synthesized via multi-step deposition process (right).
- 4.9 XRD pattern of (a) CNTs, CNTs/ZnO direct mixing, ZnOcoated CNTs and CNTs-coated ZnO fabricated via multi-step deposition, (b) magnified of XRD pattern of CNTs/ZnO direct mixing and (c) CNTs-coated ZnO samples.

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81

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4.10 *I-V* curves of (a) all prepared samples, (b) pristine ZnO, (c) 92 ZnO-coated CNTs and (d) CNTs-coated ZnO. 4.11 92 Electron transport route in (a) ZnO-coated CNTs and (b) CNTscoated ZnO samples. 4.12 I-V curves of CNTs-coated ZnO and ZnO-coated CNTs 94 samples with and without UV illumination. 4.13 Possible electrons transfer in (a) ZnO-coated CNTs and (b) 95 CNTs-coated ZnO under UV illumination. 97 4.14 The typical J-E curves of pristine CNTs, CNTs/ZnO synthesized via single-step deposition process, ZnO-coated CNTs and CNTs-coated ZnO. Typical F-N plot of (a) pristine CNTs, (b) CNTs/ZnO 4.15 98 synthesized via single-step, (c) ZnO-coated CNTs and (d) CNTs-coated ZnO. Current emission stability measurement of (a) pristine CNTs, 101 4.16 (b) CNTs/ZnO synthesized via single-step, (c) ZnO-coated CNTs and (d) CNTs-coated ZnO. 4.17 Illustration of the proposed emission of non-uniform tube-102 length CNTs. The tube may damage by the heat produced during the measurement process resulted a shortening of a tube. 4.18 FESEM images of (a) MgZnO seeded catalyst, (b) ZNFs, (c) 106 top view of ZNFs/CNTs nanocomposite, (d) dense CNTs grown on ZNFs, (e) and (f) side view observation of ZNFs/CNTs nanocomposite.

UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI F

4.19	Different position of MgZnO-coated substrate in the ZnO	107
	solution resulted ZNRs (at the top) and ZNFs (at the bottom).	
4.20	FESEM images of (a) AlZnO thin layer, (b-c) ZNRs-FLs, (d-e)	109
	as-grown CNTs on ZNRs-FLs and (f) side view of Al-doped	
	ZNRs-FLs/CNTs nanocomposite.	
4.21	EDX spectra of ZNFs/CNTs and ZNRs-FLs/CNTs	111
	nanocomposites.	
4.22	The PL spectra of ZnO nanostructures and ZnO/CNTs	112
	nanocomposite.	
4.23	The micro-Raman spectra of ZnO and ZnO/CNTs	114
	nanocomposites samples.	
4.24	Micro-Raman spectra ranged from 100-1000 cm <sup>-1</sup> of	115
	nanostructured ZnO/CNTs nanocomposites.	
4.25	XRD pattern of ZNRs/CNTs, ZNFs/CNTs, and ZNRs-	117
	FLs/CNTs samples.	
4.26	The I-V curves of pristine ZnO and ZnO/CNTs	119
	nanocomposites.	
4.27	Typical J-E curves of different morphologies of ZnO/CNTs	123
	nanocomposites.	
4.28	Typical F-N plots of different morphologies of ZnO/CNTs	122
	nanocomposites.	
4.29	The current emission vs time measurement of (a) ZNRs/CNTs,	125
	(b) ZNFs/CNTs and (c) ZNRs-FLs/CNTs nanocomposites.	

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- 4.30 The atomic percentage of element presence in the ZnO/CNTs 126 nannocomposite sample.
- 4.31 The band diagram of CNTs at higher (right) and lower (left) 127 oxygen content.
- 4.32 FESEM images of (a-b) the as-grown ZNFs on SiO<sub>2</sub> seeded 130 catalyst, (c) modified ZnO nanorods into nanoneedles structure after deposition of CNTs, (d) top and (e) side view image of ZNFs/CNTs nanocomposite and (f) CNTs grown on SiO<sub>2</sub>/ZNFs. (g)-(h) The ZNFs/CNTs grown on MgZnO seeded catalyst.
- 4.33 The PL spectra of ZNFs grown on MgZnO and SiO<sub>2</sub> seeded 133 catalysts.
- 4.34 The micro-Raman spectra of pristine ZNFs and ZNFs/CNTs 134 grown on MgZnO and SiO<sub>2</sub> seeded catalysts.
- 4.35 The XRD pattern of ZNFs/CNTs grown on MgZnO and SiO<sub>2</sub> 136 seeded catalysts.
- 4.36 The *I-V* curves of ZNFs/CNTs grown on (a) MgZnO and (b) 137SiO<sub>2</sub> seeded catalysts.
- 4.37 The *J-E* curves of ZNFs/CNTs grown on MgZnO and SiO<sub>2</sub> 139 seeded catalysts.
- 4.38 HRTEM images of CNTs grown on MgZnO/ZNFs 139 nanostructures.
- 4.39 The F-N plots of ZNFs/CNTs grown on MgZnO and SiO<sub>2</sub> 140 seeded catalysts.

NIVERSITI PENDIDIKAN SULTAN IDRIS UNI**XXII**SITI PENDID

4.40 Current stability measurement of ZNFs/CNTs grown on (a) 141MgZnO and (b) SiO<sub>2</sub> seeded catalysts.

- 4.41 (a) Fe particles deposited on the Si substrate, (b) the vapor 144 materials from WCPO were decomposed on the Fe particle, (c) the carbon diffused in the Fe particle, (d) the growth of CNTs and (e) the uneven surface of CNTs produced.
- 4.42 (a) Illustration of agglomerated MgZnO particles on the 144
   VACNTs (b) side view observation of VACNTs with the uneven surface and (c) uneven MgZnO seeded catalyst deposited on the VACNTs.
- 4.43 (a) ZnO solution before the sonication process, (b) ZnO 145 solution after 30 min of sonication process which resulted in well-mixed zinc nitrate-HMT and (c) Zn-HMT complex formation after the stirring process.
- 4.44 (a) Position of VACNTs/MgZnO-coated substrate during the 146 immersion process, (b) interaction of Zn-HMT complex with the MgZnO seeded catalyst, (c) the OH<sup>-</sup> ions reacted with CNTs during the formation of ZNRs, (d) the formation of ZNFs due to the presence of CNTs, (e) final product (ZnO-coated CNTs sample) and (f) top view FESEM image of the sample.
- 4.45 (a) Fe particles deposited on the as-grown ZNRs, (b) Fe particle
  was suggested to be diffused in the ZNRs, (c) the incoming
  hydrocarbon on the ZNRs-Fe template, (d) due to low catalytic
  activity, the CNTs tended to grow in agglomeration and (e) the

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final product of CNTs-coated ZNRs.

- 4.46 FESEM images of spin coated ZNFs/CNTs nanocomposites 151(a)-(b) n=3, (c)-(d) n=10.
- 4.47 Micro-Raman spectra of (a) spin coated ZNFs/CNTs, micro152 Raman spectra ranged from (b) 400-500 cm<sup>-1</sup> of ZNFs/CNTs-3 and (c) 100-1000 cm<sup>-1</sup> of ZNFs/CNTs-10 samples.
- 4.48 *J-E* curves of (a) ZNFs/CNTs-3 and (b) ZNFs/CNTs-10 153 samples and F-N plots of (c) ZNFs/CNTs-3 and (d) ZNFs/CNTs-10 samples.

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## LIST OF SYMBOLS AND ACRONYMS

θ	-	The angle between the incident rays and the surface of the crystal
χ	-	Electron Affinity
φ	-	Work Function
μm	-2	Micrometer
AlZnO	-	Aluminum-Doped Zinc Oxide
Ar	-	Argon
at%	-	Atomic Percentage
cm	-	Centimeter
CNTs	-	Carbon Nanotubes
CVD	-	Chemical vapor Deposition
D	-	Defect Peak
EDX	-	Energy-Dispersive X-Ray
FEE	-	Field Electron Emission
FESEM		Field Emission Scanning Electron Microscopy
F-N	-	Fowler-Nordheim
G	-	Crystalline Graphite Peaks
G'	- 1	Second-Order Raman Peaks
HMT	-	Hexamethylenetetramine
$I_{\rm D}/I_{\rm G}$	-	The Integrated Intensity ratio of the D and G Peaks
I-V	-	Current-Voltage
J-E	-	Current Emission-Electric Field
MgZnO	-	Magnesium Zinc Oxide

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mm	-	Millimeter
MWCNTs	-	Multi-Walled Carbon Nanotubes
nm	-	Nanometer
$O_i$	-	Oxygen Interstitial
PL	-	Photoluminescence
S cm <sup>-1</sup>	-0	Siemen per centimeter
SiO <sub>2</sub>	-	Silicon Dioxide
SWCNTs	-	Single-Walled Carbon Nanotubes
TPa	-	Terapascal
Vo	-	Oxygen Vacancy
$V_{Zn}$	-	Zinc Vacancy
wt%	-	Weight Percentage
XRD	-	X-Ray Diffraction Spectroscopy
ZnO	- *	Zinc Oxide
β	-	Field Enhancement Factor

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### **CHAPTER 1**

### **INTRODUCTION**

#### 1.1 Introduction

"There's plenty of room at the bottom" was brilliant idea that has been presented by Feynman in 1959, which opened up the possibility to work and manipulate materials at nanometer scale. The possibility to manipulate and modify a nanomaterial as well as to produce large surface area without changing its dimension makes these studies beneficial for further application devices. The word "nanotechnology" is introduced for the first time by Taniguchi (Taniguchi, 1974). Since then, numerous studies and investigations, both on nanomaterial and nanotechnology, have been extensively explored (Drexler & Minsky, 1990; Gohel, Chin, Zhu, Sow, & Wee, 2005). To date, the application of nanotechnology has been applied in large areas such as electronic (Liu & Guo, 2012), energy production (Y. Zhang et al., 2009), aerospace component

(Cabrera & Miranda, 2014) and medicine (Sui, Zhang, Sheng, Huang, & She, 2013). UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN DRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PENDIDIKAN SULTAN IDRIS UNIVERSITI PEN