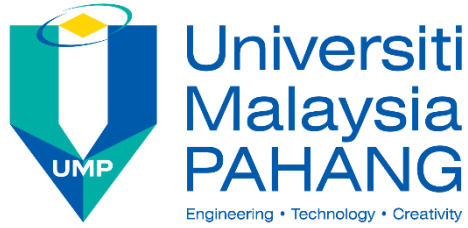


THE IMPROVEMENT OF THE PHYSICAL
PROPERTIES OF INDIUM TIN OXIDE THIN
FILM THROUGH ANNEALING PROCESSES

MEHDI QASIM ZAYER

DOCTOR OF PHILOSOPHY (PHYSICS)

UNIVERSITI MALAYSIA PAHANG



SUPERVISORS' DECLARATION

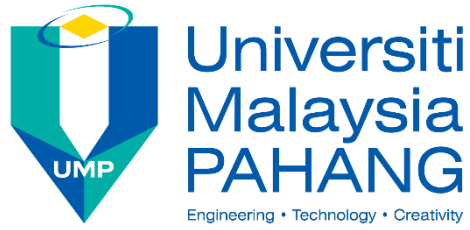
We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy in Physics.

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I hereby declare that the work in this thesis is my own except for quotations and summaries, which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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MEHDI QASIM ZAYER

Thesis Submitted in Fulfillment of the Requirements for the Award of the
Degree of Doctor of Philosophy in Physics Nanotechnology

FACULTY OF INDUSTRIAL SCIENCES & TECHNOLOGY
UNIVERSITI MALAYSIA PAHANG

November 2016

DEDICATION

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ
اللَّهُ لَا إِلَهَ إِلَّا هُوَ الْحَيُّ الْقَيُّومُ لَا تَأْخُذُهُ سِنَّةٌ وَلَا نَوْمٌ لَمْ يَلَمْسْ فِي السَّمَوَاتِ وَمَا
فِي الْأَرْضِ مَنْ ذَا الَّذِي يَشْفَعُ عِنْدَهُ إِلَّا بِإِذْنِهِ يَعْلَمُ مَا بَيْنَ أَيْدِيهِمْ وَمَا
خَلْفَهُمْ وَلَا يُحِيطُونَ بِشَيْءٍ مِنْ عِلْمِهِ إِلَّا بِمَا شَاءَ وَسِعَ كُرْسِيُّهُ السَّمَاوَاتِ
وَالْأَرْضَ وَلَا يَئُودُهُ حِفْظُهُمَا وَهُوَ الْعَلِيُّ الْعَظِيمُ

البقرة 255

IN GOD WE TRUST

ACKNOWLEDGMENTS

In the name of ALLAH, the Most Benevolent, the most Merciful. First of all, I wish to record immeasurable gratitude and thankful fullness to the One and The Almighty Creator, the Lord and Sustainer of the universe, and the Mankind, in particular; peace and blessings of Allah be upon the noblest of the Prophets and Messengers, our Prophet Mohammed and upon his family, companion and who follows him until the last day. First of all I would like to thank my Almighty God to complete this research and this thesis appearance in this form. Moreover, it is ardently desired that this little effort be accepted by Him to be of some service to the cause of humanity. I am truly and deeply indebted to so many people that there is no way to acknowledge all or even any of them properly.

I would like to express my deep and grateful thanks to my supervisor Dr. Mohamad Ashry Bin Jusoh, time and patience throughout my work. Also, my sincere thanks go to Dr. Jinan Basheer Ahmed Al-dabbagh, Dr. Gurumurthy Hegde and Dr. Nasser Ahmad for their constant guidance and encouragement to me throughout this doctoral study.

I would like to express my sincere gratitude to Universiti Malaysia Pahang (UMP) for providing laboratory facilities. It was a wonderful place to work, and the staffs are very dedicated people. Furthermore, special thanks to the academic, management and technical staff in the Faculty of Industrial Sciences & Technology and the staff of Institute of Postgraduate Studies (IPS) in UMP. I thank all my friends and colleagues for every bit of support. I am also grateful to Iraqi Ministry of Higher Education and Scientific Research and University of Technology for giving me the permission for this study.

Lastly, most importantly present to my parents, brothers, sisters, wife, daughters, son and all family members. I just want to say that I love you all very much and that I dedicated this thesis to all of you. I am also indebted to my lovely wife for all her sacrifices and patience during the past three years, and thanks to my kids for adding a sugary flavor to my life.

Mehdi Qasim Zayer

TABLE OF CONTENTS

	Page
DECLARATION	
TITLE PAGE	i
DEDICATION	ii
ACKNOWLEDGMENTS	iii
ABSTRACT	iv
ABSTRAK	v
TABLE OF CONTENTS	vi
LIST OF TABLES	xii
LIST OF FIGURES	xiii
LIST OF SYMBOLS	xix
LIST OF ABBREVIATIONS	xx
CHAPTER 1 INTRODUCTION	
1.1 Introduction	1
1.2 Problem Statement	4
1.3 Objectives of Thesis	5
1.4 Scope of Thesis	6
1.5 Organization of the Thesis	6
CHAPTER 2 LITERATURE REVIEW	
2.1 Introduction	8
2.2 Indium Tin Oxide Material	8
2.3 ITO Structural and Electrical Properties	10
2.3.1 ITO Structure	10
2.3.2 ITO Electrical Band Structure	11
2.4 ITO Optical Properties	13
2.4.1 Transmittance	13
2.4.2 Absorbance	14
2.4.3 Reflectance	15

2.5	ITO Deposition Techniques	15
2.5.1	RF Magnetron Sputtering Technique	15
2.5.2	Thermal Evaporation Technique	21
2.5.3	Spin Coating Technique	24
2.6	Heat Treatment	27
2.6.1	Normalizing Heat Treatments	27
2.6.2	Hardening Heat Treatments	27
2.6.3	Annealing Treatment	28
2.7	Thermal Oil	31
2.7.1	Thermal Oil Chemistry	31
2.7.2	Types of Thermal Oils	32
2.7.3	Thermal Oil Selection	33
2.8	Annealing Effect on Physical Properties	33
2.8.1	Annealing Effect on Structural Properties	33
2.8.2	Annealing Effect on Optical and Electrical Properties	35
2.9	Adhesion Strength Testing Method	37
2.10	Thickness Measurement Method	38
2.11	Four-Point Probe Method (FPPM)	40
2.12	Applications of ITO Thin Films	42
2.13	Summary	42

CHAPTER 3 RESEARCH METHODOLOGY

3.1	Introduction	43
3.2	Design Procedure	44
3.3	Growth Techniques in Preparation of (ITO) Thin Films	46
3.3.1	Substrate Preparation	46
3.3.2	RF Magnetron Sputtering Technique	47
3.3.3	Thermal Evaporation Technique	48
3.3.4	Spin coating Technique	49
3.4	Processes of ITO Heat Treatment	50
3.4.1	Gas Thermal Annealing Technique	50
3.4.2	Oil Thermal Annealing (OTA) Technique	51

3.5	Characterization and Measurements	52
3.5.1	Structural characterization of ITO thin films by XRD	53
3.5.2	Surface Morphology by Field Emission Scanning Electron Microscope	53
3.5.3	Surface Roughness by Atomic Force Microscope	54
3.5.4	Optical Characterization of ITO Thin Film	55
3.5.5	Electrical measurements by Four Point Probes	57
3.6	Thickness Measurements	58
3.7	Adhesion Strength Testing	59
3.8	Summary	60

CHAPTER 4 RESULTS AND DISCUSSION

4.1	Introduction	61
4.2	Surface Morphology Properties	62
4.2.1	Surface Morphology of ITO Film Coated by RF Magnetron Sputtering	62
4.2.2	Surface Morphology of ITO Film prepared by Thermal Evaporation	71
4.2.3	Surface Morphology of ITO Film Coated by Spin Coating	80
4.3	Structural Properties	89
4.3.1	Structural Properties of ITO Film Coated by RF Magnetron Sputtering	89
4.3.2	Structural Properties of ITO Thin Film Coated by Thermal Evaporation	92
4.3.3	Structural Properties of ITO Thin Film Coated by Spin Coating Technique	95
4.4	Optical and Electrical Properties	98
4.4.1	Optical and Electrical Properties of ITO Thin Film coated by RF Magnetron Sputtering	98
4.4.2	Optical and Electrical Properties of ITO Thin Film prepared by Thermal Evaporation	106

4.4.3	Optical and Electrical Properties of ITO Thin Film Prepared by Spin Coating	114
4.5	Final Comparison of ITO Thin film Production	122
4.5.1	Comparison of ITO Thin Films Prepared by RF Magnetron Sputtering, Thermal Evaporation and Spin Coating Techniques	122
4.5.2	Comparison of Adhesion Strength of ITO Thin Film Prepared by Thermal Evaporation and RF Magnetron Sputtering Technique	123
4.5.3	Thickness Effect	124
4.5.4	Cost Estimation	126
4.6	Heat Treatment Process Model	127
4.6.1	Gases Heat Treatment Process Model	128
4.6.2	Thermal Oil Heat Treatment Process Model	129

CHAPTER 5 CONCLUSION AND FUTURE RECOMMENDATION

5.1	Conclusion	131
5.2	Contributions	132
5.2.1	Fabrication technologies	132
5.2.2	Treatment Effect	132
5.3	Future Work	132

REFERENCES	135
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APPENDIX	157
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A.	AFM AND FESEM IMAGES	157
A.1	AFM and FESEM Images by using RF Magnetron Sputtering Technique	157
A.2	AFM and FESEM Images by using Thermal Evaporation Technique	161
A.3	AFM and FESEM Images by using Spin Coating Technique	165
B.	Electrical information	169
	Table B1: I-V data for ITO thin film prepared by RF- sputtering technique.	169

Table B2: I-V data for ITO thin film prepared by TE technique.	170
Table B3: I-V data for ITO thin film prepared by SC technique.	171
C. Thickness Information	172
C.1. Thickness at 100 nm	172
Table C.1. 1 RF Magnetron Sputtering Technique	172
Table C.1. 2 Thermal Evaporation Techniques	173
Table C.1. 3 Spin Coating Techniques	174
C.2. Thickness 150 nm	175
Table C.2. 1 RF Magnetron Sputtering Technique	175
Table C.2. 2 Thermal Evaporation Techniques	176
Table C.2. 3 Spin Coating Techniques	177
C.3. Thickness 250 nm	178
Table C.3. 1 RF Magnetron Sputtering Technique	178
Table C.3. 2 Thermal Evaporation Techniques	179
Table C.3. 3 Spin Coating Techniques	180
C.4 Thickness 300 nm	181
Table C.4. 1 RF Magnetron Sputtering Technique	181
Table C.4. 2 Thermal Evaporation Techniques	182
Table C.4. 3 Spin Coating Techniques	183
D. Material Cost	184
D.1 Total Cost of Commercial Sample	184
D.2 RF-Magnetron Sputtering Technique	184
D.3 Thermal Evaporation Technique	185
D.4 Spin Coating Technique	186
E. Temperature distribution	188
E1. Figure shows Temperature distribution of gas	188
E2. Figure shows Temperature distribution of oil	189
F. Chapter Three Information	190
F1 Adhesion strength	190
F.2 (XRD) X-Ray Diffractometer	190
F.3. (FESEM) Field Emission Scanning Electron Microscope	191
F.4. (AFM) Atomic Force Microscopy	191
G. Journal Publications	192

H.	International Conferences	193
I.	Patent & Awards	194

LIST OF TABLES

Table No.	Title	Page
4.1	Comparison of surface roughness for ITO thin film prepared by RF magnetron sputtering technique with different treatment types.	69
4.2	Comparison of surface roughness for ITO thin film prepared by thermal evaporation technique with different treatment types.	78
4.3	Comparison of surface roughness for ITO thin film prepared by SC technique with different treatment types.	87
4.4	Comparison of sheet resistance and transmittance for ITO thin film prepared by RF magnetron sputtering technique.	105
4.5	Comparison of sheet resistance and transmittance for ITO thin film prepared by thermal evaporation technique.	114
4.6	Comparison of sheet resistance and transmittance for ITO thin film prepared by spin coating technique.	121
4.7	Comparison of cost estimation.	127

LIST OF FIGURES

Figure No.	Title	Page
2.1	Indium Tin Oxide structure (a) Indium Oxide and (b) Tin Oxide	9
2.2	Crystal structure of In_2O_3	11
2.3	The schematic diagram of the mechanism levels (a) optical band gap and (b) levels of the valance and conduction band	12
2.4	Schematic diagram of plasma discharge	16
2.5	Schematic of the possible effects with ion bombardment	17
2.6	Schematic diagram of glow discharges.	19
2.7	Simple reactive sputtering process	20
2.8	Schematic diagram of a typical magnetron sputtering system	21
2.9	Schematic diagram of the thermal evaporation technique	24
2.10	Four different stages of Spin Coating process.	26
2.11	The schematic diagrams in (a) annealing by vacuum and (b) annealing by gas flow.	29
2.12	Schematic of the three stages of liquid quenching	30
2.13	DOWTHERM Thermal oil operating temperatures.	32
2.14	Mechanism for thermal annealing effect on the ITO structural status.	34
2.15	XRD patterns of the ITO thin films before and after annealing heat treatment.	35
2.16	Effect of annealing temperature on the electrical resistivity and carrier concentration of the ITO thin film	36
2.17	Effect of annealing temperature on optical transmittance of the ITO film	37
2.18	Schematic diagram of adhesion Strength test.	38
2.19	Schematic diagram of thin film thickness measurement by the Michelson interference method.	39
2.20	Schematic diagram of the interference fringes.	40
2.21	Schematic diagram of four-point probe method	41
3.1	General design methodology procedure of ITO thin film.	45
3.2	Substrate preparation sequence.	46
3.3	Schematic diagram of RF magnetron sputtering technique.	48

3.4	Schematic diagram of the thermal evaporation technique.	49
3.5	The spin coating technique (a) image and (b) step.	50
3.6	A schematic diagram of gas thermal annealing treatment process.	51
3.7	Photo of the experimental setup for gas thermal annealing treatment process.	51
3.8	Experimental setup of oil thermal annealing.	52
3.9	A schematic diagram AFM principle.	54
3.10	Interaction of light with ITO thin film.	55
3.11	Set up for Spectrophotometer.	56
3.12	Spectrophotometer measure methods	57
3.13	Schematic diagram of four point probes measurements.	58
3.14	Schematic diagram of the Michelson interference for the ITO thin film thickness measurement.	59
3.15	Adhesion Strength Testing (a) Wilson Hardness (Tukon 1102 Knoop/Vickers Tester) and (b) Schematic diagram of ITO thin film adhesion strength testing.	60
4.1	(a) AFM and (b) FESEM images for As-deposited of ITO thin film prepared by RF magnetron sputtering technique.	63
4.2	AFM and FESEM images of ITO thin film prepared by RF magnetron sputtering technique after treated by OTA.	65
4.3	AFM and FESEM images of ITO thin film prepared by RF magnetron sputtering technique after treated by argon gas.	66
4.4	AFM and FESEM images of ITO thin film prepared by RF magnetron sputtering technique after treated by nitrogen gas.	67
4.5	Comparison of surface roughness at R_{max} and R_a for ITO thin film prepared by RF magnetron sputtering technique.	70
4.6	Variation of surface Roughness with annealing time for RF magnetron sputtering techniques.	71
4.7	(a) AFM and (b) FESEM images for As-deposited of ITO thin film prepared by TE technique.	72
4.8	AFM and FESEM images of ITO thin film prepared by thermal evaporation technique after treated by OTA.	74
4.9	AFM and FESEM images of ITO thin film prepared by thermal	75

	evaporation technique after treated by argon gas.	
4.10	AFM and FESEM images of ITO thin film prepared by thermal evaporation technique after treated by nitrogen gas.	76
4.11	Comparison of surface roughness at R_{max} and R_a for ITO thin film prepared by TE technique.	79
4.12	Variation of surface roughness and annealing time with different treatment type for ITO thin film prepared by thermal evaporation techniques.	80
4.13	(a) AFM and (b) FESEM images for As-deposited of ITO thin film prepared by SC technique.	81
4.14	AFM and FESEM images of ITO thin film prepared by spin coating technique after treated by OTA.	83
4.15	AFM and FESEM images of ITO thin film prepared by spin coating technique after treated by argon gas.	84
4.16	AFM and FESEM images of ITO thin film prepared by spin coating technique after treated by nitrogen gas.	85
4.17	Comparison of surface roughness at R_{max} and R_a for ITO thin film prepared by spin coating technique.	88
4.18	Variation of surface roughness with annealing time of ITO thin film prepared by spin coating techniques.	89
4.19	XRD patterns results for ITO thin film prepared by RF magnetron sputtering techniques after treated by OTA.	90
4.20	XRD patterns results for ITO thin film prepared by RF magnetron sputtering techniques after treated by argon gas.	90
4.21	XRD patterns results for ITO thin film prepared by RF magnetron sputtering techniques after treated by nitrogen gas.	91
4.22	Variation of grain size of ITO thin film prepared by RF magnetron sputtering technique with annealing time at temperatures 150 °C And 300 °C.	92
4.23	XRD patterns results for ITO thin film prepared by thermal evaporation techniques after treated by OTA.	93
4.24	XRD patterns results for ITO thin film prepared by thermal evaporation techniques after treated by argon gas.	93

4.25	XRD patterns results for ITO thin film prepared by thermal evaporation techniques after treated by nitrogen gas.	94
4.26	Variation of grain size for ITO thin film prepared by thermal evaporation technique with annealing time.	95
4.27	XRD patterns results for ITO thin film prepared by spin coating technique after treated by OTA.	96
4.28	XRD patterns results for ITO thin film prepared by spin coating technique after treated by argon gas.	95
4.29	XRD patterns results for ITO thin film prepared by spin coating technique after treated by nitrogen gas.	97
4.30	Variation of grain size for ITO thin film prepared by spin coating technique with annealing time.	97
4.31	Variation of transmittance for ITO thin film prepared by RF magnetron sputtering technique after treated by OTA at (a) 150 °C and (b) 300 °C.	99
4.32	Variation of transmittance for ITO thin film prepared by RF magnetron sputtering technique after treated by argon gas at (a) 150 °C and (b) 300 °C.	100
4.33	Variation of transmittance for ITO thin film prepared by RF magnetron sputtering technique after treated by nitrogen gas at (a) 150 °C and (b) 300 °C.	101
4.34	Comparison of transmittance of ITO thin film prepared by RF magnetron sputtering technique at temperatures 300 °C and 10 minutes.	102
4.35	Sheet resistance of ITO thin film prepared by RF magnetron sputtering techniques after treated by OTA.	103
4.36	Sheet resistance of ITO thin film prepared by RF magnetron sputtering techniques after treated by argon gas.	103
4.37	Sheet resistance of ITO thin film prepared by RF magnetron sputtering techniques after treated by nitrogen gas.	104
4.38	Comparison of sheet resistance for ITO thin film prepared by RF magnetron sputtering technique with different annealing treatment type.	104

4.39	Transmittance versus wavelength for ITO thin film prepared by thermal evaporation technique after treated by OTA at temperatures (a) 150 °C and (b) 300 °C.	107
4.40	Transmittance versus wavelength for ITO thin film prepared by thermal evaporation technique after treated by argon gas at (a) 150 °C and (b) 300 °C.	108
4.41	Transmittance versus wavelength for ITO thin film prepared by thermal evaporation technique after treated by nitrogen gas at (a) 150 °C and (b) 300 °C.	109
4.42	Comparison of transmittance of ITO prepared by thermal evaporation technique at 300 °C and 10 min for different annealing treatments.	110
4.43	Sheet resistances for ITO thin films prepared by thermal evaporation technique after treated by OTA.	111
4.44	Sheet resistances for ITO thin film prepared by thermal evaporation technique after treated by argon gas.	111
4.45	Sheet resistances for ITO thin film prepared by thermal evaporation technique after treated by nitrogen gas.	112
4.46	Comparison of sheet resistance for ITO thin film prepared by thermal evaporation technique with different annealing treatment type.	113
4.47	Transmittance versus wavelength for ITO thin film prepared by spin coating technique after treated by OTA at temperatures (a) 150 °C and (b) 300 °C.	115
4.48	Transmittance versus wavelength for ITO thin film prepared by spin coating technique after treated by argon gas at (a) 150 °C and (b) 300 °C.	116
4.49	Transmittance versus wavelength for ITO thin film prepared by spin coating technique after treated by nitrogen gas at (a) 150 °C and (b) 300 °C.	117
4.50	Comparison of transmittance for ITO prepared by spin coating technique at 300 °C and 10 min for different annealing techniques.	118

4.51	Sheet resistance for ITO thin film prepared by spin coating technique and treated by OTA.	119
4.52	Sheet resistance for ITO thin film prepared by spin coating technique and treated by argon gas.	119
4.53	Sheet resistance for ITO thin film prepared by spin coating technique and treated by nitrogen gas.	120
4.54	Comparison of sheet resistance for ITO thin film prepared by spin coating technique with different annealing treatment type.	120
4.55	Final comparison of the deposition technique.	122
4.56	Comparison of ITO adhesion strength for RF and thermal evaporation preparation techniques at temperature 150 °C and 300 °C with annealing time.	123
4.57	Effect of thickness on ITO transmittance and sheet resistance.	125
4.58	Heat treatment processes by gas, (a) filling gas, (b) starting heat source, and (c) stable annealing temperature 300 °C at 10 minute annealing time.	129
4.59	Heat treatment processes by OTA (a) filling oil, (b) starting heat source, and (c) stable annealing temperature 300 °C at 10 minute annealing time.	130

LIST OF SYMBOLS

ϵ_0	dielectric constant in vacuum
ϵ_r	relative permittivity
β	broadening factor
θ	Bragg angle, peak angle position of diffracted X-ray
λ	X-ray wavelength
μ	mobility of free carriers
μ_N	mobility
ρ	Fluid Density;
σ	Electrical conductivity
τ	mean size of the crystalline domain
$\alpha (\lambda)$	absorption coefficient,

LIST OF ABBREVIATIONS

Symbol	Description
a	angle of attack
a_0	effective Bohr radius
AFM	Atomic Force Microscopy
1-D	one direction
CF	correction factor determined by the ratio of the
DMSO	Dimethylsulfoxid-6
d	Distance between atomic planes
DC	Direct Current
D_λ	Dark intensity at wavelength λ
e	evaporation rate
e	electron charge,
EBP	Emslie, Bonner, and Peck
ESCA	Electron Spectroscopy for Chemical Analysis
E_f	Fermi energy
eV	electron Volt
$F(\zeta_d)$	screening function
FPP	Four point probes
f_1	finite sample thickness
f_2	sample width correction factors
FESEM	Field Emission Scanning Electron Microscopy
g	Acceleration Due to Gravity
h	Planck's constant
I	current
$I(hkl)$	measured intensity of a particular (hkl) plane,
$\ln(hkl)$	intensity of the nth peak
ITO	Indium Tin Oxide
m	film viscosity
K	shape factor,
L	mean-free path length
\ln_2O_3	Indium Oxide
m	fluid viscosity

m	mass flux of solvent
m^*	effective mass of the conduction electrons
n	significant peaks observed in the corresponding diffractogram
N	number of pixels within a given area
N	carrier concentration
nc	density
N_e	number of molecules evaporated from the unit area per second,
N_i	density of the charged scattering centers
N_n	concentration of neutral impurities
w	angular velocity
O	doubly charged vacancies
O_2	oxygen
P	Pressure;
P_e	Equilibrium vapor pressure (in Torr)
PVD	Physical Vapor Deposition
q	fluid flow rate
r	fluid density
r	radial distance
RF	Radio Frequency
RMS	Root Mean Square
R_λ	Reference intensity at wavelength λ
sccm	Standard Cubic Centimeters per Minute
s	fluid surface tension
s	probe spacing
SC	Spin Coating
SEM	Emission Scanning Electron Microscope
Sn	extrinsic dopants
SnO ₂	tin dioxide
S_λ	Sample intensity at wavelength λ
r	solvent density
t	film thickness,
TCO	Transparent Conducting Oxide
TEM	Transmission Electron Microscopy

h	thickness
UV	Ultraviolet-Visible
V	potential difference , Voltage
v	substrate velocity
w	angular velocity
XRD	X-ray diffraction
Y	sputtering yield
z	Height
Zm	mean surface level within a given area,
$R(\lambda)$	normal incidence reflectivity,
$k(\lambda)$	extinction coefficient
$n(\lambda)$	real refractive index
TCOs	Transparent conducting oxides

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November 2016

ABSTRACT

Many optoelectronic devices such as mobile screens, flat panel displays, solar cells and optical storages use Indium Tin Oxide (ITO) due to its ability to retain its good physical properties during the thin film preparation. Therefore, it is of great importance to study the related issues in applications utilizing ITO such as transparency and resistance as well as the underlying cost. These problems can be overcome by the enhanced annealing treatment which is called the Oil Thermal Annealing (OTA) technique in the current work. In this study, ITO thin films of thickness 200 nm are deposited on glass substrates by three preparation techniques, i.e. RF magnetron sputtering, thermal evaporation, and spin coating. The 200 nm ITO thin film is selected due to its relatively lower sheet resistance and higher transparency as compared to those thin films of thicknesses ranging from 100 nm to 300 nm. The RF magnetron sputtering technique is the best candidate in improving the ITO thin film properties. Upon the preparation process, the thin film is treated by OTA, argon and nitrogen operating at temperature range: $150\text{ }^{\circ}\text{C} < T < 300\text{ }^{\circ}\text{C}$ with the annealing time of 2, 6 and 10 min. Measurements and calculations are conducted before and after the preparation and treatment processes. The structural properties and surface morphology of ITO thin films are examined by X-Ray Diffraction (XRD), Atomic Force Microscopy (AFM) and Field Emission Scanning Electron Microscopy (FESEM) before and after the annealing process. The XRD analysis reveals that a polycrystalline structure of maximum diffraction intensity at $2\theta = 30.5^{\circ}$ and (222) orientation exists in the ITO thin film. FESEM and AFM analyses show that the formation of continuous and homogeneous films are fully covered by pyramidal shaped particles, and roughness value decreases with increasing annealing time. It is also observed that by using OTA treatment, the sheet resistance of ITO thin films prepared by the RF magnetron sputtering technique reduces significantly to $20.8\text{ }\Omega/\text{sq}$ after 10 min of annealing time. On the other hand, the sheet resistance values of $70.8\text{ }\Omega/\text{sq}$ and $72.6\text{ }\Omega/\text{sq}$ are obtained via argon and nitrogen gas treatments, respectively. The As-deposited sheet resistance is $2.8\text{ k}\Omega/\text{sq}$. The visible spectrum obtained from the spectrophotometer has shown high transparency values of 95.6 %, 89.9 % and 85 % for RF sputtering which are treated by OTA, argon and nitrogen respectively. OTA gives the highest transparency value, due to the fact that the thermal distribution on the ITO thin film surface treated by OTA is better than those by using argon and nitrogen gases. The OTA treatment process is shown to be successful in improving the ITO thin film properties, which is valuable for many optoelectronic applications. The OTA process can be applied in nano-layers of various sizes and thicknesses at minimum production cost. The total costs incurred after the preparation process (via RF magnetron sputtering technique) and the annealing process (performed at temperature $300\text{ }^{\circ}\text{C}$ with time 10 min annealing time) using OTA, argon and nitrogen are RM 4.40, RM 46.60 and RM 46.06, respectively. From the current work, it is found that the adhesion strength of the ITO thin film prepared by the RF magnetron sputtering technique and treated by OTA is the highest. Also, the structural, optical and electrical properties can be improved as well by using the proposed technique.

ABSTRAK

Banyak peranti optoelektronik seperti skrin mudah alih, paparan panel rata, sel suria dan penyimpanan optik menggunakan Timah Indium Oksida (ITO) kerana kemampuannya untuk mengekalkan ciri-ciri fizikal yang baik semasa penyediaan filem nipis. Oleh itu, adalah amat penting untuk mengkaji isu-isu yang berkaitan dalam aplikasi menggunakan ITO seperti ketelusan dan rintangan. Masalah-masalah ini boleh diatasi dengan rawatan penyepuh Lindapan dipertingkatkan yang dipanggil teknik penyepuh Lindapan haba minyak (OTA). Dalam kajian ini, filem nipis ITO berketebalan 200 nm didepositkan pada substrat kaca dengan tiga teknik penyediaan, iaitu percikan pemagnetan RF, penyejatan haba, dan putaran saduran. Filem nipis ITO 200 nm dipilih kerana rintangan lembaran yang rendah dan ketelusan yang lebih tinggi berbanding dengan filem-filem nipis berketebalan antara 100 nm hingga 300 nm. Percikan pemagnetan RF adalah teknik yang baik dalam meningkatkan sifat filem nipis ITO. Setelah proses penyediaan, filem nipis dirawat dengan OTA, argon dan nitrogen beroperasi pada julat suhu: $150\text{ }^{\circ}\text{C} < T < 300\text{ }^{\circ}\text{C}$ dengan masa penyepuh Lindapan daripada 2, 6 dan 10 minit. Pengukuran dan pengiraan dijalankan sebelum dan selepas proses penyediaan dan rawatan. Sifat-sifat struktur dan permukaan morfologi filem nipis ITO diperiksa oleh pembelauan sinar-X (XRD), Mikroskop Daya Atom (AFM) dan Mikroskop Pengimbas Elektron Emisi Medan (FESEM) sebelum dan selepas proses penyepuh Lindapan. Analisis XRD menunjukkan bahawa terdapat struktur polihabluran dengan keamatan puncak maksimum pada $2\theta = 30.5^{\circ}$ dan orientasi (222) wujud dalam filem nipis ITO. Analisis FESEM dan AFM menunjukkan bahawa pembentukan filem yang berterusan dan seragam dilindungi sepenuhnya oleh puncak piramid berbentuk zarah, nilai kekasaran permukaan lebih tinggi dengan peningkatan masa penyepuh Lindapan. Ia juga diperhatikan bahawa dengan menggunakan rawatan OTA, rintangan helai daripada ITO filem nipis yang disediakan oleh teknik percikan pemagnetan RF berkurang dengan ketara kepada $20.8\ \Omega/\text{persegi}$ selepas 10 minit masa penyepuh Lindapan. Sebaliknya, nilai rintangan helai pada $70.8\ \Omega/\text{persegi}$ dan $72.6\ \Omega/\text{persegi}$ diperolehi masing-masing melalui rawatan gas argon dan nitrogen. Rintangan helai As-deposit adalah $2.8\ \text{k}\Omega/\text{persegi}$. Spektrum nampak diperolehi daripada spektrofotometer menunjukkan nilai-nilai ketelusan yang tinggi pada 95.6 % , 89.9 % dan 85 % untuk percikan pemagnetan RF yang dirawat masing-masing oleh OTA, argon dan nitrogen. OTA memberikan nilai ketelusan tertinggi, disebabkan oleh fakta bahawa pengagihan haba di permukaan filem nipis ITO yang dirawat oleh OTA adalah lebih baik daripada menggunakan gas argon dan nitrogen. Proses rawatan OTA ditunjukkan berjaya dalam meningkatkan sifat-sifat filem nipis ITO, yang bernilai untuk banyak aplikasi optoelektronik. Proses OTA boleh digunakan dalam pelbagai saiz dan ketebalan nanolapisan pada kos pengeluaran yang minimum. Jumlah kos yang ditanggung selepas proses penyediaan (melalui teknik percikan pemagnetan RF) dan proses penyepuh Lindapan (dilakukan pada suhu $300\text{ }^{\circ}\text{C}$ dengan masa masa 10 minit penyepuh Lindapan) menggunakan OTA, argon dan nitrogen adalah masing-masing RM 4.40, RM 46.60 dan RM 46.06. Dari kajian semasa, didapati bahawa kekuatan lekatan filem nipis ITO yang disediakan dengan teknik percikan pemagnetan RF dan dirawat dengan OTA adalah yang tertinggi. Juga, sifat-sifat struktur, optik dan elektrik boleh diperbaiki dengan menggunakan teknik yang dicadangkan.

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