FABRICATION AND CHARACTERIZATION OF THE COPPER GALLIUM OXIDE THIN FILM AT DIFFERENT ANNEALING TEMPERATURES

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I hereby declare that the work in this thesis is my own except for quotations, equation, summaries and references, which have been duly acknowledged.

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ABSTRACT

The study of p-type semiconductor $CuGaO_2$ thin film was carried out to investigate the effects of various temperatures during and after the deposition in order to obtain the optimum result in achieving a good optical transparency and conductivity of the thin film. Majority of the transparent oxide semiconductors (TOS) which were developed do not possess p-type conductivity. Due to an excess oxygen in the acceptor levels and the created holes which are firmly localized at the oxygen sites, p-type oxide semiconductors are gaining more research attention. With the increase of demand in the novel components, $CuGaO_2$ has gained importance in research as a p-type semiconductor. Previous studies emphasized on the effects of lower temperature post-treatment of the $CuGaO_2$ thin film of temperature lower than 500°C. The potential applications of the $CuGaO_2$ thin films are very wide such as thin-film transistors, transparent diodes and light-emitting diodes which are growing to be more and more relevant in the current generation. The $CuGaO_2$ thin films were fabricated on quartz substrate via the RF magnetron sputtering technique with varying sputtering temperatures and annealing temperatures. The p-type thin films were deposited at temperatures of room temperature (RT), 50°C, 100°C, 150°C, 200°C and 250°C. Each samples of the individual deposition temperatures were also annealed at varying temperatures of 500°C, 600°C, 700°C, 800°C and 900°C. The XRD results showed that the thin films of 900°C annealing temperature has a peak approximately at 36.28° with the orientation of (012), which is based on rhombohedral unit cell with the space group R-3m (JCPDS card No. 41-0255). The crystallite size obtained is 18.040nm for the annealing temperature of 900°C while the Full-Wave Half Maximum (FWHM) value is 0.081. The optical band gaps obtained from the thin films ranged from 3.30-3.72 eV, which is in line with the results found in the general range of $CuGaO_2$ thin film optical band gaps of 3.30-3.60 eV. From the UV-Vis measurement, the high transparencies were observed to be approximately at 70-80%. The EDS measurement of the chosen parameters of 100°C with annealing temperature of 900°C showed that the Oxide weighted at 33.75%, Copper at 27.86% and Gallium at 19.13% while the remaining 19.13% belongs to Silicon which is a part of the quartz used as the substrate. Due to the optimum surface morphology of the annealing temperature of 800°C of deposition temperature 100°C, bandgap which is within the range of general range of $CuGaO_2$ and high transmittance are reasons suitable for diode fabrication.

ABSTRAK

FABRIKASI DAN PENCIRIAN FILEM NIPIS COPPER GALLIUM OXIDE PADA SUHU PENYEPUHLINDAPAN YANG BERLAINAN

Kajian terhadap filem nipis semikonduktor $CuGaO_2$ jenis-p telah dijalankan untuk mengkaji kesan kepelbagaian suhu semasa dan selepas penmendapan, bagi mendapatkan hasil yang optimum dalam mencapai ketelusan optik dan kekonduksian yang baik bagi filem nipis. Kebanyakan semikonduktor oksida yang berlutsinar yang dihasilkan tidak memiliki kekonduksian jenis-p. Disebabkan oksigen yang berlebihan yang ada pada aras penerima dan juga lubang lohong yang bersetempat di tapak oksigen, semikonduktor oksida jenis-p telah menarik lebih perhatian untuk kajian dilakukan. Dengan permintaan yang semakin tinggi, $CuGaO_2$ menunjukkan kepentingan dalam penyelidikan sebagai semikonduktor jenis-p. Potensi aplikasi filem nipis $CuGaO_2$ sangat luas seperti transistor filem nipis, diod telus dan diod pemancar cahaya semakin terkenal dan lebih relevan pada generasi semasa. Filem nipis $CuGaO_2$ telah difabrikasi pada substrat kuarza menggunakan teknik pemercikan Magnetron Frekuensi Radio dengan suhu pemercikan dan suhu penyepuhlindapan yang berbeza-beza. Filem nipis jenis-p telah dimendapkan pada suhu yang berbeza iaitu pada suhu bilik, 50°C, 100°C, 150°C, 200°C dan 250°C. Setiap sampel yang dipercikkan juga melalui proses sepuh lindap pada suhu yang berbeza 500°C, 600°C, 700°C, 800°C dan 900°C. Hasil XRD menunjukkan bahawa filem nipis pada suhu 900°C mempunyai puncak secara hampir pada 36.28° dengan orientasi (012), berdasarkan sel unit rhombohedral dengan kumpulan ruang R-3m (kad JCPDS No. 41 -0255). Saiz kristal yang didapati adalah 18.040nm daripada suhu penyepuhlindapan 900°C manakala nilai maksimum Separuh Penuh Gelombang (FWHM) adalah 0.081. Jurang tenaga optik yang diperolehi daripada filem nipis adalah dari 3.30-3.72 eV, ianya selaras dengan hasil yang didapati dalam rangkaian umum filem nipis $CuGaO_2$ iaitu 3.30-3.60 eV. Berdasarkan pengukuran UV-Vis, ketelusan filem dicerap kira-kira 70-80%. Pengukuran EDS bagi parameter 100°C dengan suhu penyepuhlindapan 900°C menunjukkan bahawa peratus berat Oksida pada 33.75%, Tembaga pada 27.86% dan Gallium pada 19.13% manakala peratusan selebihnya sebanyak 19.13% adalah milik Silicon yang merupakan sebahagian daripada substrat. Oleh kerana morfologi permukaan yang optima pada suhu penyepuhlindapan 800°C dengan suhu pemercikan 100°C, jurang tenaga optic yang berada di rangkaian umum $CuGaO_2$ dan ketelusan filem yang tinggi adalah sebab-sebab yang sesuai untuk fabrikasi diod.

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LIST OF SYMBOLS

&	-	And
Å	-	Angstrom
Ag	-	Silver
As	-	Arsenic
α	-	Absorption Coefficient
β	-	Fixed constant
Bi	-	Bismuth
^{o}C	-	Celsius
cm^{-1}	-	Centimeter per unit
cm^{-3}	-	Centimeter per cubic
$CuAlO_2$	-	Copper Aluminium Oxide
$CuGaO_2$	-	Copper Gallium Oxide
$CuInO_2$	-	Copper Indium Oxide
Co	-	Cobalt
Cr	-	Chromium
Cu	-	Copper
Cu_2O	-	Cupric Oxide
d	-	distance between the near atomic planes/ film thickness
E_c	-	Conduction band
E_g	-	Band gap
E_v	-	Valence band
eV	-	Electron volt
hv	-	Photon energy
Ga	-	Gallium
GaAs	-	Gallium Arsenide
GaN	-	Gallium Nitrite
In	-	Indium
In_2O_3	-	Indium oxide
IPA	-	Isopropyl alcohol
σ	-	Electrical conductivity
K	-	Potassium
κ	-	Scherrer Constant

kÅ/s	-	Kilo angstrom per second
λ	-	Wavelength of electromagnetic spectrum
Li	-	Lithium
MeV	-	Mega electron volt
μm	-	Micrometer
n	-	Integer of the order of the diffracted beam/ allowed direct band
N	-	Nitrogen
Na	-	Natrium
NiO	-	Nickel Oxide
nm	-	Nanometer
O	-	Oxide
P	-	Phosphorus
Pd	-	Palladium
Pt	-	Platinum
PTFE	-	Poly (tetrafluoroethylene)
Sb	-	Antimony
Si	-	Silicon
sccm	-	Standard cubic centimetre per minute
T (%)	-	Transmittance
Θ	-	Angle of the incidence X-ray
V	-	Volt
V_i	-	Oxygen vacancies
W	-	Watt
Zn_i	-	Zinc interstitial
ZnO	-	Zinc Oxide

LIST OF ABBREVATIONS

CVD	-	Chemical Vapor Deposition
DC	-	Direct current
FWHM	-	Full width half maximum
ITO	-	Indium Tin Oxide
JCPDS	-	Joint Committee on Powder Diffraction Standards
LCD	-	Liquid Crystal Display
LED	-	Light Emitting Diode
PLD	-	Pulsed laser deposition
RF	-	Radio frequency
RMS	-	Root mean square
RPM	-	Revolutions per minute
RT	-	Room Temperature
TCO	-	Transparent Conductive Oxide
TV	-	Television
UV	-	Ultra-violet
WWII	-	World War 2
XRD	-	X – Ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background Study

The electronic industries particularly optoelectronics have intrigued the interest of many especially researchers due to its potential and demand in the marketplace. The optoelectronic devices found widely in many applications such as LCD TV, mobile phones, electronic tablets and laptops have been the typical necessities in most societies nowadays.

Transparent Conductive Oxides (TCO) are widely used not just due to its transparent properties but also their electrical properties in relevant devices which are commonly used in technological applications such as LEDs (Tsukazaki *et al*, 2004), flat panel displays (Bruce *et al*, 2005), sensors (Alias *et al*, 2012) and transistors (Presley *et al*, 2004), which form the basis of the optoelectronic industry. Essentially, optoelectronic devices require good transparency in the visible spectral range and low resistivity (Saad *et al*, 2015). Inorganic oxide-based materials of high transparency and electrically conductive of either electron or hole type were being worked on over the past two decades (Domaradzki, 2016). Hence, TCOs have become more popular in the modern society due to its practicality. A de-icer was the first device that involved a TCO material which was used in WWII as bomber windows (Bruce *et.al*, 2005). Most of the TCOs used are of the n-type semiconductors due to its good electrical properties such as SnO_2 (Presley *et.al*, 2004), In_2O_3 (Sahm *et.al*, 2007) and ZnO (Dhara and Giri, 2012). As an example, ZnO has a large exciton binding energy which is approximately 60 meV (Ajimsha *et.al*, 2014). In addition, due to its low solubility and self-compensating effect of the acceptor dopants, ZnO exhibit a n-type conductivity. Moreover, the development of ZnO homojunction was constrained as reported by Shen *et.al* (2014:345). Hence hybrid p-n heterojunctions such as p-type thin layers p-Si, p-NiO and p-polymer with n-type ZnO were considered. However, high quality heterojunctions are always difficult to obtain due the huge difference in lattice mismatch between the p-type and n-type (Shen *et.al*, 2014:345).

The delafossite group was reviewed and showed potential in improving the p-type conductivities. Therefore, it is important to research on this group in the development of the optoelectronic advancement. Several popular examples of the delafossite group which have been studied on such as $CuAlO_2$ (Reddy *et.al*, 2009), $CuInO_2$ (Sasaki and Shimode, 2003) and $CuGaO_2$ (Tsay and Chen, 2017). The discovery of the p-type Cu-bearing TCOs also presented opportunities for research on the p-n transparent junction devices (Domaradzki, 2016).

1.2 Problem Statement

A p-n junction of two types of oxide semiconductors combined leads to the creation of transparent optoelectronic devices and electronic circuits, which are also known as smart electronics. However, majority of the transparent oxide semiconductors (TOS) which were developed do not possess p-type conductivity (Tsay and Chen, 2017). Due to an excess oxygen in the acceptor levels and the created holes which are firmly localized at the oxygen sites, p-type oxide semiconductors are gaining more research attention. Copper based delafossite oxide semiconductors has a formula of $CuM^{III}O_2$, where M is group III elements such as Ga, Al, and Cr. These semiconductors were studied on the basis that they have room temperature p-type conductivity (Tsay and Chen, 2017). The copper-based delafossite oxide semiconductors are attributed to the ionized Cu vacancies and interstitial oxygen atoms. The introduction of the divalent cations for the trivalent cations at the octahedral sites of the delafossite structure might be a fruitful solution in increasing the hole concentration density, hence improving the electrical conductivity of the thin films (Tsay and Chen, 2017). Doped p-type were introduced in past researches. However, it was still difficult to obtain a stable p-type conductivity due to several factors such as deep acceptor level, low solubility of the acceptor dopant and of the native donor defects (Dhara and Giri, 2012).

With the increase of demand in the novel components, $CuGaO_2$ has gained importance in research as a p-type semiconductor (Yu and Lee, 2018). Previous studies emphasized on the effects of lower temperature post-treatment of the $CuGaO_2$ thin film, which were also limited with the use of glass that has a threshold of 550°C (Abu Bakar *et.al*, 2015). High formation temperature has been reported for delafossite structures such as $CuCrO_2$ (600°C) and $CuAlO_2$ (800°C) (Yu and Lee, 2018). Hence, annealing of the thin films were performed to improve the crystallinity.

In this study, the effect of higher annealing temperature ranging from 500°C, 600°C, 700°C, 800°C, and 900°C of the nature of $CuGaO_2$ thin film has been studied to attain better efficiency of an optoelectronic material. Chun-Tsung and his research team report the common use of quartz glass as substrate for $CuAlO_2$ thin film (Chun-Tsung *et al*, 2011). The use of quartz enables the samples to be heated up to higher temperatures by RF magnetron sputtering (Kumar *et al*, 2012). Hence, the use of quartz has been applied for this research.

1.3 Research Aims

This research aims to study on the effect of the higher annealing temperature with quartz as the substrate compared to that of a lower heat tolerance glass slab of the deposition of $CuGaO_2$ thin film. The deposition method used in this research is RF magnetron sputtering due to its high purity via the high vacuum process and the ability to control the thickness at nanoscale. Fabrication of different parameters will produce different outcome, namely the structural or morphological property, the optical property and the crystalline structure of the $CuGaO_2$ thin film.

1.4 Research Objectives

The objectives of this research are:

1) To fabricate the $CuGaO_2$ thin films with different deposition temperature and different annealing temperature via RF magnetron sputtering technique.
2) To characterize the of $CuGaO_2$ thin films at different deposition temperature and different annealing temperature in terms of structural, surface morphology and optical properties.

3) To determine the optimum deposition and annealing temperature of $CuGaO_2$ thin films as a p-type TCO.

1.5 Research Scope

Using various parameters in fabricating a semiconductor sample will bring about different results, likewise with different deposition methods. Hence, this research aims to ascertain the optimum parameters there is during and after the deposition, which in this research via RF magnetron sputtering. The experiments include depositing semiconductor materials on quartz substrate with deposition temperature of room temperature (RT), 50°C, 100°C, 150°C, 200°C and 250°C for 30 minutes on each sample. Subsequently, this study also involves the annealing process from 500°C, 600°C, 700°C, 800°C, and 900°C for each sample at a fixed time of 3 hours and leaving one of the parameters as non-annealed for comparison. Hence, the sample size comprises of 36 samples as each samples of individual deposition temperatures were annealed individually from the parameters mentioned above. The characterization methods are demonstrated in terms of the crystal structures, surface morphologies and optical properties. Methods used includes XRD and EDS to study on the crystal structure, FESEM and AFM to study on the surface morphology, lastly UV-Vis to study on the optical properties of the thin film.

1.6 Thesis Outline

This thesis consists of five chapters with different contents. In chapter 1, the introduction of the project is discussed starting with uses of TCO devices being discussed. In addition, the limitations of achieving high quality TCO devices are also included. The aim of the research, objectives and the scope of the thesis are written in this chapter.

Chapter 2 provides the fundamental theory behind the sophisticated device. The emphasis of this research deals mainly with semiconductor physics hence most of the elaborations are based on it. Previous researches were also reviewed concerning the development of TCO from the primary aspect to the up-to-date progress.

Chapter 3 discusses the methods used in carrying out the experiments, with a brief explanation of the steps taken beginning with cleaning of the substrate and the proper way of handling the samples. The characterization methods are also discussed in terms of the crystal structures, surface morphologies and optical properties. Methods used includes XRD and EDS to study on the crystal structure, FESEM and AFM to study on the surface morphology, lastly UV-Vis to study on the optical properties of the thin film.

Chapter 4 details the results obtained, and data tabulated. The characterization of the finished product from the experiment is done and a discussion based on past research is provided so that the current research can be justified.

Finally, chapter 5 summarizes and concludes the effectiveness of the research with its objectives fulfilled. A future work is also suggested so that there is an advancement in this field.

CHAPTER 2

LITERATURE REVIEW

2.1 Solid Classification

In semiconductor physics, solid is classified into three forms categorized as conductors, semiconductor and insulator. The main difference among the three is the electrical conductivity.

2.1.1 Conductors

When electrons experience an applied electric field, the electrons are accelerated and are able to move into a new energy state. Hence, there must be empty energy states vacant for electrons to fill for charge transport to happen (DasGupta and DasGupta, 2007). The outermost shell of the atom is not completely occupied by electrons; hence it is possible for current to flow through a solid when there is a potential difference across the conducting material. The higher bands overlap such that more conduction happens through the empty states which are vacant. Types of conductors includes alkali metals which are sodium and potassium and metals such as aluminum and mercury (Edwards-Shea, 1996).

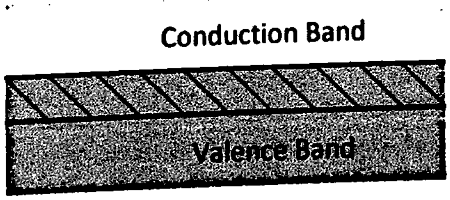

Figure 2.1: (a) A narrow bangap and (b) overlapping band of a conducting material

Source : Edwards-Shea, 1996

2.1.2 Insulator

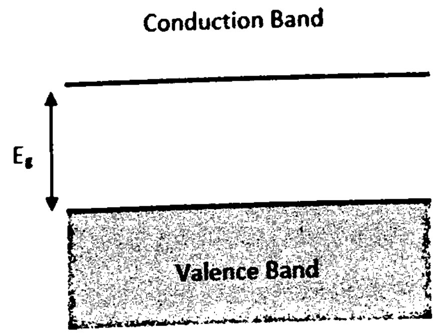

Figure 2.2: Band diagram of an insulated material

Source : Edwards-Shea, 1996

Insulators have very few electrons present in the conduction band due to the valence band being almost filled and hence less available empty states for electrons to fill. The energy gap is big and hence conduction of electricity is unlikely to happen (DasGupta and DasGupta, 2007). Even in high temperature up to several hundred kelvins, insulators are either almost filled or the other way around which is almost completely empty. When an electric field is applied across these insulating materials, the electrons cannot be accelerated in the field since there are no available energy states which the electron energies can be increased. Hence, the force exerted by the field is too small for the electrons to cross the gap from the valence band to the conduction band (Edwards-Shea, 1996).

2.1.3 Semiconductor

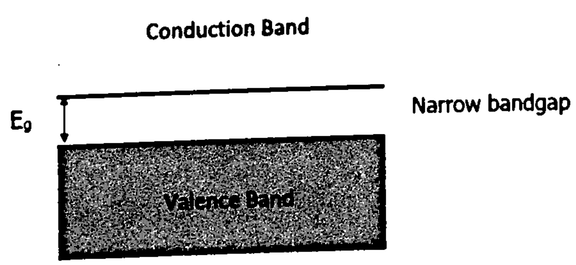

Figure 2.3: Band diagram of a semiconducting material

Source : Edwards-Shea, 1996

Semiconductors have similar energy gap just like that of the insulators where the valence band is filled, and the conduction band is empty at 0 K. The main difference between them are the size of the band gap which is bigger for insulators. The small gap between the valence and conduction bands in semiconductors enables electrons in the filled valence band to be excited and fill the empty conduction bands by thermal excitation at room temperature. Some examples of semiconductors are Germanium, Silicon, Indium Phosphide etc. (DasGupta and DasGupta, 2007). The significance of the narrowness of the bandgap between the valence and conduction band is that it can gain enough heat energy even at room temperature so that the electrons can be excited and emerge from the valence band to the conduction band (Edwards-Shea, 1996).

At temperature close to 0 K, semiconductors act like an insulator in which they do not conduct. This is due to the insufficient thermal energy for the electrons break their bond, hence staying in the valence band. However, semiconductors in temperatures more than 0 K can conduct which source its energy from the thermal excitation of electrons, leaving the valence band only party empty.

2.2 Classification of Semiconductors

Generally, semiconductors consist of two types, intrinsic semiconductor and extrinsic semiconductor. An intrinsic semiconductor is a perfect crystal with no impurities or defects (DasGupta and DasGupta, 2007) while an extrinsic semiconductor has certain impurities added to it so that its conductivity is improved (Edwards-Shea, 1996).

2.2.1 Intrinsic Semiconductor

In many instances intrinsic semiconductors are found with some impurities. This may have been caused by the processing methods in manufacturing factories which have incorporated the impurities. Ideally intrinsic semiconductors comprise just atoms of its own which form solids as they bond together.

Elemental semiconductors like silicon and germanium have four valence electrons on the outermost orbit of their atom which is in a tetravalent configuration.

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