

STRUCTURAL, LUMINESCENCE AND ELECTRICAL CONDUCTIVITY OF
STRONTIUM TITANATE THIN FILMS PREPARED BY DIP COATING
METHOD

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Dedicated to

my beloved husband, parents, family, and friends.

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ABSTRACT

Strontium titanate (SrTiO_3) sol-gel was deposited onto glass substrates by dip coating method. The effects of deposition parameters such as different annealing temperatures and number of coating layers were studied. Strontium nitrate anhydrous ($\text{Sr}(\text{NO}_3)_2$), titanium isopropoxide ($\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$), ethylene glycol ($\text{HOCH}_2\text{CH}_2\text{OH}$) and nitric acid (HNO_3) were used to prepare sol-gel of strontium titanate. Ethylene glycol was added to promote polymerization between $\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$ and $\text{Sr}(\text{NO}_3)_2$. The samples structure were characterized by X-ray diffraction (XRD), atomic force microscopy (AFM), Fourier transform infra red (FT-IR) and photoluminescence (PL) spectroscopy. XRD analysis showed the process of the intermediate crystalline phase of $\text{Sr}(\text{NO}_3)_2$ with lattice parameter $a = b = c = 0.78$ nm for as-prepared sample. An amorphous inorganic phase was found for samples annealed at 300 and 400 °C. The samples began to change to SrTiO_3 phase after being annealed at 500 °C with lattice parameter $a = b = c = 0.39$ nm and peak orientation at planes (110), (111), (200) and (211). Thin films with double coating layers showed clearer peaks orientation. The microstructure study using AFM revealed the grain size in the range 220 – 460 nm and surface roughness in the range 33 – 69 nm at 500 °C for different number of coating layers. FT-IR analysis shows that SrTiO_3 crystallization phase was completely formed at higher temperatures of 500, 600 and 700 °C. PL spectroscopy of SrTiO_3 thin films appeared to have spectra emission in the region of 310 - 510 nm wavelength. The spectra also showed the different peaks appearing at different annealed temperatures. Van der Pauw technique was used to measure the electrical conductivity and activation energy for single and four coating layers samples at 500 °C annealing temperature. The results showed that the electrical conductivity for single layer was higher than the four coating layers. Activation energy in high temperature region were in the range of 0.656 meV and 0.446 meV whereas activation energy in low temperature region were in the range of 0.162 meV and 0.105 meV for single and four coating layers samples, respectively.

ABSTRAK

Sol-gel strontium titanat (SrTiO_3) telah dimendapkan ke atas substrat kaca dengan menggunakan kaedah salutan celupan. Kesan parameter pemendapan seperti perbezaan suhu penyepuhlindapan dan bilangan salutan lapisan telah dikaji. Anhidrat strontium nitrat ($\text{Sr}(\text{NO}_3)_2$), titanium isoproposida ($\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$), etilena glikol ($\text{HOCH}_2\text{CH}_2\text{OH}$) dan asid nitrik (HNO_3) telah digunakan untuk menghasilkan sol-gel strontium titanat. Etilena glikol telah ditambah untuk menggalakkan pempolimeran diantara $\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$ dan $\text{Sr}(\text{NO}_3)_2$. Struktur sampel telah dicirikan dengan menggunakan pembelauan X-ray (XRD), mikroskopi daya atom (AFM), transformasi Fourier infra merah (FT-IR) dan spektroskopi fotopendarcahaya (PL). Analisis XRD telah menunjukkan proses fasa pertengahan kristal bagi $\text{Sr}(\text{NO}_3)_2$ dengan parameter kekisi $a = b = c = 0.78$ nm untuk sampel pra-persediaan. Fasa bukan organik amorfus telah diperolehi bagi sampel sepuhlindapan pada 300 and 400 °C. Sampel mula berubah kepada fasa SrTiO_3 selepas sepuhlindapan pada 500 °C dengan parameter kekisi $a = b = c = 0.39$ nm dan orientasi puncak pada satah (110), (111), (200) dan (211). Saput tipis dengan dwi lapisan salutan menunjukkan puncak-puncak orientasi yang paling jelas. Kajian mikrostruktur melalui AFM menunjukkan saiz butiran pada julat 220 – 460 nm dan permukaan kasar pada julat 33 – 69 nm pada suhu 500 °C untuk bilangan lapisan salutan yang berbeza-beza. Analisis FT-IR menunjukkan fasa kristal SrTiO_3 telah terhasil secara menyeluruh pada suhu lebih tinggi 500, 600 and 700 °C. Spektroskopi PL untuk saput tipis SrTiO_3 menunjukkan pancaran spektra pada panjang gelombang dalam lingkungan 310 – 510 nm. Spektra yang dihasilkan juga menunjukkan kemunculan puncak yang berbeza pada suhu sepuhlindapan yang berbeza. Teknik Van der Pauw telah digunakan untuk mengukur konduktiviti elektrik dan tenaga aktif untuk sampel satu dan empat lapisan salutan pada suhu penyepuhlindapan 500 °C. Keputusan menunjukkan konduktiviti pada sampel satu lapisan adalah lebih tinggi berbanding sampel empat lapisan. Tenaga aktif pada julat suhu tinggi berada dalam lingkungan 0.656 meV dan 0.446 meV manakala tenaga aktif pada julat suhu rendah berada dalam lingkungan 0.162 meV dan 0.105 meV masing-masing untuk sampel satu dan empat lapisan salutan.

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LIST OF SYMBOLS

FT-IR	-	Fourier Transform Infrared Spectroscopy
XRD	-	X-ray Diffraction
AFM	-	Atomic Force Microscopy
PL	-	Photoluminescence
VDP	-	Van der Pauw
SrTiO ₃	-	Strontium Titanate
Sr(NO ₃) ₂	-	Strontium Nitrate
OHCH ₂ CH ₂ OH	-	Ethylene Glycol
C ₁₂ H ₂₈ O ₄ Ti	-	Titanium Isopropoxide
JCPDS	-	Joint Center Power Diffraction Standard card
σ	-	Electrical Conductivity
σ_0	-	Pre-exponential of Electrical Conductivity
E_a	-	Activation Energy
k	-	Boltzmann constant
d	-	Spacing between the planes in the atomic lattice
d_{hkl}	-	Interplanar spacing for planes with Miller indices (hkl)
λ	-	Wavelength
θ	-	Angle between the incident ray and the scattering planes
ν	-	Frequency (Hz)
k	-	Force constant of the bond (Nm ⁻¹)
μ	-	Reduced mass (kg)
m_1	-	Relative atomic mass of M ₁ x u
m_2	-	Relative atomic mass of M ₂ x u
u	-	atomic mass unit = 1.66 x 10 ⁻²⁷ kg
h	-	Planck's constant
c	-	Speed of light

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Phenomenon of thin film can be describe when a layer of oxide appeared on a material. Thus, scientist used thin as a term to explain about a very thin layer appeared on a material that can be characterize and applied in technology development. This phenomenon has been discovered by Faraday last 1838 using an electrolysis process, followed by Bunsen & Grove on 1852 using chemical reaction, in 1857 Faraday have discovered thermal evaporation process and further studied have been carried out intensively after world war two (Samsudi Sakrani, 2002).

Thin film is a layer of solid material such as metal, semiconductor or insulator deposited on a substrate with appropriate thickness in range from 10-1000 nm (Suriani, 2005). For layer greater than 1000 nm, it will be grouped as a thick film or film. It will be known as ultra film for layer thinner than 100 nm. In thin films study, variable of thickness shown different characterization result. A substrate is use as a medium to deposit thin film. To produce a good quality and

interact with film in hygiene surface. The main applications in thin film development are in electronic devices and optical coatings.

Generally thin film can be prepared either in chemical (Desai *et al.*, 2006) or physical method (Dantus *et al.*, 2011). Examples of chemical method are dip coating and spray pyrolysis techniques. Other examples of physical method are sputtering and vacuum evaporation techniques. There are various methods of preparing nanostructure thin films such as sol-gel process (known as dip coating method and spin coating method), metal organic decomposition (MOD), liquid phase deposition (LPD), spray pyrolysis and thermal evaporator under vacuum. A lot of researchers are competing in producing thin films with a simple technique, large surface area, inexpensive and non-pollution for industrial user. Dip coating with sol-gel process have been widely used to prepared thin films and ceramic powder. This type of method has an capability to control the stoichiometric literally at the molecular level.

ABO₃ family is known as cations elements for A and B and anion element for O or oxygen. Recently a lot of ABO₃ family for examples barium titanate, barium strontium titanate and strontium titanate have been attracted due to their ferroelectric and electro-optic properties (Soledade *et al.*, 2002 and Goa *et al.*, 2002). Strontium titanate is a paraelectric material with cubic perovskite structure (Zanetti *et al.*, 1997 and Benthem *et al.*, 2001). Theoretically, strontium titanate material is known to has properties such as high dielectric constant, good thermal stability, second order phase transformation and stress induce phase transition photo-activity (Kim *et al.*, 2007). These advantages makes strontium titanate as the best material and widely used for application in memory cell capacitors for non-volatile memories or dynamic random access application (DRAM), microelectronic, solar and sensor technology in recent years usually in bulk material or thin films technology development (Brankovic *et al.*, 2004).

1.2 Problem Statement

Nowadays, strontium titanate has attracted many researchers because of its wide application and favorable performance in industry. With this purpose, numerous of work have been done in different environment and condition to develop a good quality of strontium titanate thin films with a simple step, low cost and environmental friendly.

In recent years, works on sol-gel preparation with dip coating technique to deposit strontium titanate thin films have been actively considered. Even though there are several report on strontium titanate thin films, there is no clear-cut understanding on the relation between numbers of coating layer thin films and annealing temperatures with several characterization. Thus, this experiment will be done by various deposition parameters such as number of coating layer on corning glass substrate and at different annealed temperatures. By changing the parameters in strontium titanate thin films, one can expect to obtain new characterization result and may give better understanding on structural and electrical conductivity. Thus, the change in behavior of these films can be used in specific applications and development.

1.3 Research Objectives

Research objective of this study are:

- i. To prepare strontium titanate thin films by using sol-gel dip coating method at different numbers of coating layer and annealed temperatures.
- ii. To characterize the structural behavior of strontium titanate thin films in coating layer and at annealing temperatures by using X-ray diffraction

(XRD) analysis, atomic force microscopy (AFM), Fourier Transformation Infrared Spectroscopy (FT-IR) and photoluminescence spectrometer (PL).

- iii. To investigate the electrical conductivity of coating layer thin films by using van der Pauw (VDP) technique.

1.4 Scope of Studies

The structural and chemical bonding of strontium titanate was characterized by using x-ray diffraction (XRD) analysis while infrared spectroscopy (FT-IR) was used to confirm the existence of strontium titanate crystallization. Atomic force microscopy (AFM) was used to characterize the morphology of the films. Meanwhile, emission and electrical conductivity behavior of the films were investigated by using photoluminescence spectroscopy (PL) and Van der Pauw (VDP), respectively.

This thesis has covered five chapter. Chapter 1 includes background, problem statement, objective and scope of studies. Chapter 2 discussed about literature review regarding the physical properties of strontium titanate, thin films methodology in previous work and properties of strontium titanate thin films. This covers theory of sample characterization of strontium titanate thin films, on the structural and electrical conductivity properties. The methodology and instrumentation in chapter 3 covered all the experimental and characterizations process. Next, chapter 4 discussed the results of experimental conducted in the research. Lastly, chapter 5 summarizes the studies and some suggestion are presented for future works.

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