STRUCTURAL AND OPTICAL PROPERTIES OF STRONTIUM TITANATE THIN FILMS PREPARED BY SPRAY PYROLYSIS

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This thesis is especially dedicated to:

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Bonda Che Mah binti Mat Ali,
Abang Long and Kak Long,
Angah and Kak Ngah,
Achik and Kak Su,
Ateh and Cik La,
Abbasi and Syafiqa,
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ABSTRACT

Strontium titanate ($SrTiO_3$) thin films sample were prepared by using spray pyrolysis technique onto a glass substrate. The thin films were deposited using various concentrations of 0.2 – 1.0 M strontium nitrate ($Sr(NO_3)_2$) and titanium tetraisopropoxide (TTIP) at constant temperature 400ºC and a flow rate of 3 mL/min oxygen carrier gas. The films were annealed at temperature range of 400ºC to 650ºC. The films were characterized using UV-Visible spectrophotometer, X-ray diffractometer (XRD), atomic force microscope (AFM), Fourier transform infra-red spectrometer (FTIR) and photoluminescence (PL) spectrometer. The samples showed a high transmittance of more than 75% in the visible region, with band gap energy in the range of 3.4 eV to 4.0 eV. The band gap energy of the films decreased from 3.9 eV to 3.5 eV as the annealing temperature increased from 400ºC to 500ºC. The films deposited at 400ºC showed an amorphous phase. At an annealing temperature of 450ºC, the thin films sample turned into polycrystalline phase. The intensity of (110) orientation was the dominant phase of strontium titanate at annealing temperature of 650ºC. FTIR measurements showed that symmetrical OH absorption band exists in all the films and Ti-O bonding only can be observed at an annealing temperature 500ºC above. The grain sizes and surface roughness of the films increased with increasing annealing temperatures. In this study, the strontium titanate ($SrTiO_3$) thin films did not show luminescent characteristic.

ABSTRAK

Saput tipis strontium titanat ($SrTiO_3$) telah disediakan dengan menggunakan teknik semburan pirolisis ke atas substrat kaca. Saput tipis telah dimendapkan dengan larutan strontium nitrat ($Sr(NO_3)_2$) dan titanium tetraisopropoxide (TTIP) yang berbeza kepekatan daripada 0.2 - 1.0 M pada suhu pemendapan 400ºC dan kadar alir gas pembawa oksigen 3 mL/min. Saput tipis telah disepuh lindap pada suhu 400ºC hingga 650ºC. Saput tipis telah dianalisis dengan menggunakan spektrofotometer cahaya nampak, pembelauan sinar - X (XRD), mikroskop daya atom (AFM), spektrometer Fourier transformasi infra-merah (FTIR) dan spektrometer fotoluminesen (PL). Saput tipis menunjukkan kehantaran yang tinggi lebih daripada 75% pada kawasan cahaya nampak dengan tenaga jurang jalur dalam julat 3.4 eV hingga 4.0 eV. Tenaga jurang jalur saput tipis berkurangan daripada 3.9 eV ke 3.5 eV apabila suhu sepuh lindap bertambah daripada 400ºC hingga 500ºC. Saput tipis yang dimendapkan pada suhu 400ºC menunjukkan fasa amorfus. Pada suhu sepuh lindap 450ºC, saput tipis bertukar menjadi hablur. Keamatan pada orientasi (110) adalah fasa dominan bagi strontium titanat pada suhu 650ºC. Pengukuran FTIR menunjukkan penyerapan ikatan simetri OH wujud pada semua saput tipis dan ikatan Ti – O hanya boleh diperhatikan pada suhu sepuh lindap melebihi 500ºC. Saiz butiran dan permukaan kekesatan saput tipis bertambah apabila suhu sepuh lindap bertambah. Dalam kajian ini, saput tipis strontium titanat ($SrTiO_3$) tidak menunjukkan ciri luminesen.

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LIST OF SYMBOLS

α	-	Absorption coefficient
A	-	Absorption intensity
c	-	Speed of light
d	-	Atomic spacing
d	-	Sample thickness
E_g	-	Optical energy gap
e	-	Electron charge
eV	-	Electron Volt
ΔE	-	Width of the band tails
ε	-	Permittivity
hkl	-	Crystal plane orientation index
μ	-	Mobility
μ	-	Permeability
v	-	Speed
$\bar{v}$	-	Wavenumber
ω	-	Frequency
θ	-	Angle
λ	-	Wavelength

LIST OF ABBREVIATIONS

CB	-	Conduction band
FTIR	-	Fourier transformed infrared
g	-	gram
hr	-	hour
IR	-	Infrared
JCPDS	-	Joint Comitee on Powder Diffraction Standard
mm	-	Millimetre
mV	-	millivolt
NIR	-	Near infrared
nm	-	nanometer
UV	-	Ultraviolet
Vis	-	Visible
VB	-	Valence band
XRD	-	X-ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Strontium titanate is an oxide of strontium and titanium with the chemical formula $SrTiO_3$. At room temperature, it is a centrosymmetric paraelectric material with a perovskite structure. At low temperatures it approaches a ferroelectric phase transition with a very large dielectric constant $\sim 10^4$ but remains paraelectric down to the lowest temperatures measured as a result of quantum fluctuations, making it a quantum paraelectric. It was long thought to be a wholly artificial material, until 1982 when its natural counterpart discovered in Siberia and named tausonite. The name tausonite was given in honour of Lev Vladimirovich Tauson (1917-1989), a Russian geochemist. Dissused trade names for synthetic product include "*strontium mesotitanate*", "*Fabulite*", "*Diagem*" and "*Marvelite*". Pure strontium titanate occurs in nature material (an extremely rare mineral) as the well-known naturally occurring very tiny crystals (Muller and Burkard, 1979).

Strontium titanate, $SrTiO_3$ is one of the most widely used materials in thin films because it has numerous applications, such as dielectric thin film capacitors in microwave monolithic integrated circuits, high-*k* transistor gate dielectric for minimized metal-oxide semiconductor field-effect transistors, microelectronics, solar and sensor technology, dynamic random access memory (DRAM) for very large scale integrated devices, and insulating layers for limiting current in thin film electroluminescence displays (Paul and Arvids, 2001). In general, strontium titanate exhibits high dielectric constant, good thermal stability, second order phase transformation, stress-induced phase transition, high charge storage capacity, good insulating properties, chemical stability and photoactivity (Chang and Shen, 2006).

There are many techniques to prepared strontium titanate thin films, including sputtering, pulsed laser deposition (PLD), chemical vapor deposition (CVD), sol gel and spray pyrolysis. Unlike many other films deposition techniques, spray pyrolysis is interested due to very simple and relatively cost effective processing method. The films produced from this method is always with fine particles. In addition, it is extremely easy technique to preparing films of any composition and does not required high-quality substrates or chemicals. It is important to use deposition technique with low deposition temperature and low cost especially with regards to equipment costs. The method has been employed for the deposition of dense films, porous films and for powder production. Even multilayered films can be easily prepared using this technique (Goran *et al.,* 2004).

Direct deposition of $SrTiO_3$ thin film on a silicon substrate by the Liquid Phase Deposition (LPD) method has been reported by Lee *et al*., (1992). The film was grown using an aqueous solution of strontium fluorotitanate which was obtained from a mixture of hexafluorotitanic acid and strontium nitrate. The Metal-Oxide-Semiconductor (MOS) device using the as-deposited thin film as a gate oxide demonstrated good characteristics of current-voltage and capacitance-voltage. However, the chemical composition of the film was nonstoichiometric, typically Sr/Ti/O = 1/10.9/16.5, which might have major effects on the dielectric properties of the annealed film. For example, a large shift of flat band voltage is generated by nonstoichiometry-derived oxygen vacancies, which act as electron donors and high leakage paths.

Wang *et al.,* (2006) studied the structural and electrical properties of $SrTiO_3$ thin films deposited on Si by rf magnetron sputtering at various substrate temperatures. They found that these properties were strongly dependent on the substrate temperatures. On the other hand, few studies exist for the optical properties of $SrTiO_3$ films deposited at various substrate temperatures.

Xu *et al.,* (2010) used a sol–gel technique to prepare polycrystalline $SrTiO_3$/$BaTiO_3$ multilayered film on Pt/Ti/SiO_2/Si substrate and compared the dielectric response with the uniform $BaTiO_3$ and $SrTiO_3$ films. They found that the dielectric constant of the polycrystalline $SrTiO_3$/$BaTiO_3$ multilayered films could reach the 400 – 600 range at 10 kHz while keeping the dielectric loss near that of the uniform films.

Grabner and Sihvonen (1987) was reported photoluminescence of single-crystal $SrTiO_3$, a broad and structureless visible emission band peaked around 500 nm under ultra-violet radiation at low temperature. Investigation on temperature dependence of the visible emission demonstrated that the visible emission band was quenched at temperatures above 110 K. Many experiments, such as radio luminescence, cathodoluminescence, and X-ray induced luminescence have been performed and not given uniform explanations for the mechanism of the visible emission. Using time resolved spectroscopy, Leonelli and Brebner (1989) proposed a model to describe the luminescence process where electrons form small polarons and holes interact with the polarons to produce selftrapped excitons (STEs), and the recombination of STEs results in the visible emission, either immediately, or after being trapped for a certain time by impurities and defects.

Kang *et al.*, (1999) showed that flame spray pyrolysis could be applied to the direct preparation of multi-component oxide phosphor particles without post-treatment. The $SrTiO_3$: Pr, Al phosphor particles prepared by general spray pyrolysis had poor luminescence characteristics because of hollow and porous morphology of particles. On the other hand, the $SrTiO_3$: Pr, Al prepared by flame spray pyrolysis had high luminescence characteristics because of high crystallinity, spherical shape, and dense morphology.

Jinfang *et al.*, (1995), presented a visible emission band excited by the 514.5 nm line of an Ar laser whose energy is much lower than that of the band gap has been separately observed in nanocrystalline $BaTiO_3$ and $SrTiO_3$ at room temperature. Its nature and origin are attributed to the recombination of self-trapped excitons. Enhancement of the efficiency and the blue shift of the visible emission with decreasing grain size are closely correlated with interfaces of nanocrystallites, d-surface states in the forbidden gap and distortion of TiO_6 octahedra.

The visible emission bands in the nanophase $SrTiO_3$ show that the properties of the electronic states are strongly related to particle size and temperature were observed by Zhang *et al.*, (2000). The visible emission band is ascribed to the recombination of the long-lived selftrapped exciton (STE) with the forceful binding energy which can be established in the nanostructured $SrTiO_3$ at room temperature. The excitation-wavelength dependence of the visible emission band indicates that there are a series of STE levels related to the intrinsic surface states and defect centers in the nanophase $SrTiO_3$.

Bao *et al.,* (2011) prepared $SrTiO_3$ thin films by the sol-gel method and studied the relationship between band gap energy and crystallinity of $SrTiO_3$ thin films. They found that the band gap energy was film thickness-dependent and gave a critical thickness of 200 nm. Above 200 nm, the film had band gap energy close to those of crystals or bulk materials, but below 200 nm, the value shifted largely.

1.2 Problem Statement

There is much interest in the areas of science and technology in perovskite type structured compounds, $SrTiO_3$. Many researchers have investigated and developed $SrTiO_3$ materials in particles or film forms using various techniques, parameters, and some properties of thin film have been reported. Most of them, studies on $SrTiO_3$ thin films doped with other material such as Pr and Al via spray pyrolysis method. Goran *et al.,* (2004) prepared $SrTiO_3$ thin films by the spray pyrolysis method and studied the optimal deposition parameters such as solution concentration, time and temperature of deposition, and flow rate of carrier gas, but no detailed related on energy gap information was given.

Although, the properties of $SrTiO_3$: Pr, Al, thin films such as luminescence and structural have been studies and attracted a number of researchers because of their wide-ranging industrial and technical applications, there are lack of knowledge studied on the undoped of $SrTiO_3$ prepared by spray pyrolysis method. In additional, the information of the influence of an annealing temperature on the properties of strontium titanate has not been studies at all. Therefore, an investigation of structural and band gap properties will be carried out and the results of this study are presented in this thesis.

1.3 Research Objectives

The objectives of this study are as follows:

a) To determine the influence of molar concentration and annealing temperature on the band gap energy of $SrTiO_3$ thin films

b) To determine the influence of annealing temperature on crystalline phase of the prepared $SrTiO_3$ thin films.

c) To determine the influence of annealing temperature on topography properties of $SrTiO_3$ thin films.

d) To determine the influence of annealing temperature on structure features of the prepared $SrTiO_3$ films.

e) To determine whether the prepared $SrTiO_3$ thin films will give luminescence properties and how annealling temperature will affect the luminescence properties.

1.4 Scope of study

The sample of strontium titanate, $SrTiO_3$ thin films have been prepared using spray pyrolysis technique. The thickness of the thin films deposited will be measure using surface profiler. The spectrum of transmission for optical properties measurement will be obtained by UV – visible spectrophotometer. The characteristics of $SrTiO_3$

structural and phase identification are determined using X- ray diffraction (XRD) methods. Fourier Transform Infra-red spectroscopy is used to characterize the structural feature and functional group of chemical bonding for each thin films sample. The surface morphology and surface roughness of the thin films will be measure using atomic force microscopy (AFM). The emission spectral of prepared thin films will be measure by Photoluminescence (PL) spectroscopy.

1.5 Significance of study

Strontium titanate, $SrTiO_3$, is a well-known perovskite material and its band gap energy is 3.75 eV. Also, $SrTiO_3$ is chemically and compositionally very stable. Because of these attractive advantages of $SrTiO_3$, it is considered as a good candidate for host material in low-voltage electron-excitation displays, such as FED (field emission display) and VFD (vacuum fluorescence display). For thin film applications, one of the key issues concerns the thin film morphology and particle size. Therefore, we applied the spray pyrolysis for producing thin film with fine particles (Samsudi Sakrani, 1987).

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