UNIVERSITI TEKNIKAL MALAYSIA MELAKA

THE INFLUENCE OF SOL VOLUME AND HEATING TEMPERATURE ON TiO_2 SOL-GEL COATING

This report is submitted in accordance with the requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours.

by

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This report is submitted to the Faculty of Mechanical and Manufacturing Engineering Technology of Universiti Teknikal Malaysia Melaka (UTeM) as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering Technology (Process and Technology) With Honours. The member of the supervisory is as follow.

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ABSTRAK

Kajian ini bertujuan untuk menyiasat pengaruh isipadu sol dan pemanasan suhu ke atas lapisan sol-gel Titanium Dioxide. Banyak kajian yang telah dijalankan terhadap TiO_2, namun kajian yang khusus terhadap Brookite amat jarang sekali. Brookite. Isipadu sol dihasilan melalui cara sol-gel dan kemudian dilapiskan di atas permukaan kaca mikrostuktur. Jumlah isipadu sol adalah berbeza. Setelah itu, kaca mikroskop yang telah dilapiskan dengan TiO_2 dipanaskan dengan suhu yang berbeza. Hasil lapisan filem TiO_2 bergantung terhadap cara ia dihasilkan dan parameter yang terlibat sepanjang proses. Lapisan filem nipis dianalisa mengunakan mesin XRD dan SEM untu melihat fasa dan morfologi sampel.Lapisan filem nipis yang dilapiskan dengan jumlah isipadu sol dan suhu pemanasan yang berbeza menunjukkan fasa dan morfologi yang berlainan, semakin banyak jumlah isipadu dilapiskan ke atas kaca mikrostuktur, ketebalan lapisan TiO_2 semakin tebal manakala perbezaan suhu memberi kesan terhadap struktur kristalografi, penghabluran filem nipin semakin baik apabila suhu ditingkatkan. Pada 2 pusingan, saiz bijirin meningkat apabila suhu panas ditingatkan 23.97 nm (200°C) kepada 47.98 nm (400°C). Pada 4 pusingan, lapisan filem juga meningkat apabila suhu panas ditingkatkan dari 249.0 nm kepada 680.92 nm. Pada 300°C, ketebalan filem meninglat apabila lapisan isipadu sol ditingkatkan dari 467.35 nm (4 lapisan) ke 337.10 nm (8 lapisan).

ABSTRACT

This study is to investigate the influence of sol volume and heating temperature on TiO_2 sol-gel coating. Many research had been carried out on TiO_2 but research that focuses on Brookite is rarely been done. The sol volume produced by using the sol-gel method and then the solutions deposited on the surface of microscope glass. The amount of sol volume is different. After all, microscope glass coated with TiO_2, undergo heat treatment at different temperature. The result of TiO_2 thin films depends on how it being prepared and a parameter that involved in the whole processes. Thin films sample analysed using XRD and SEM machine to see the phase and morphology of the sample. The thin films coated with different volume of sol and heated on different temperature shows a different morphology and phases, the more sol volume deposited on the glass, the thickness of coating are thicker while in term of temperature the structure of thin films crystallinity better at the higher temperature. At 2 spin, the grain size increased when heating temperature increased from 23.97 nm (200℃) to 47.98 nm (400℃). At 4 spin, thin film increasing when heating temperature increased from 3249.0 nm to 680.92 nm. At 300℃, film thickness increased when number of coating increased from 467.35 nm (4 coating) to 337.10 nm (8 coating).

DEDICATION

To my beloved mother and father.

Love.

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LIST OF ABBREVIATIONS

TTiP	Titanium Tetraisopropoxide
TiO_2	Titanium Dioxide
BDP	Bachelor Degree Project
XRD	X-ray Diffraction
SEM	Scanning Electron Microscope
Ti	Titanium
DI	Deionized Water
HCl	Hydrochloric Acid

CHAPTER 1

INTRODUCTION

In this chapter, it describes background of TiO_2, problem statement, objectives, scope of work and the important of study about this project.

1.1. Background of TiO_2

Titanium oxide, TiO_2 is referred to as Titania occurring chemical compound of Ti. TiO_2 is widely used in various application all over the world such as in industry and consumer uses. Titania has gained popularity among researcher and scholar as it have a great properties like self-cleaning, improve the efficiency of electrolytic, paint and coating applications. Anatase, Rutile and Brookite are types of crystalline modification of Titanium Oxide. Each of them have different crystal structure.

The most studied phase of TiO_2 among researcher was rutile and anatase because it is easier and less complicated to synthesis rutile and anatase compared to brookite. But since the unique properties owned by the brookite and its potential to go more further in thin film coating application had been discovered, researcher start to pay attention more to brookite. Brookite can exist together with anatase or rutile depend on the parameter setting or the way it synthesis.

TiO_2 have unique physical, chemical and electro-optic properties that make it more interesting to be study on thin film application. TiO_2 also can be deposited on different types of substrate like glass, tiles, window and etc. A substrate that coated with TiO_2 will exhibit photocatalytic and a very high affinity for water when expose on ultra-violet (UV) light.

For producing TiO_2, technique such as sol-gel, chemical vapor deposition (CVD) and spray pyrolysis can be used but sol-gel method is a common method that researcher always use. This is because sol-gel method have more advantage comparing other method such as capability to have a sintering low temperature, provide protection performance by producing thick coating and able to produce high purity products. The properties of TiO_2 depend upon the sample preparation conditions, crystal section, expanse, size distribution, porosity, and therefore the presence of extra compounds like metal particles that are accustomed to enhance the chemical action response.

The parameter that will involve in this paper is volume of sol-gel TiO_2 and heating temperature. This two are the most crucial factors that will affect the result of thin film coating. By looking at volume, as a general idea different amount volume of sol-gel TiO_2 deposited on the substrate will effect the thickness of the thin coating layer while the heating temperature will affect the crystallinity of the surface. These both factors are important to look after if we want to have a very good result from the experiment and achieve our objectives.

To analyse the morphological and phase of TiO_2, this act can be done by analysing TiO_2 under X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Raman spectroscopy and Transmission Electron Microscopy (TEM) analyser. All analyser can be used to see through and more detail on TiO_2 surface such as grain size, the crystallinity, morphology and phases.

This study will be focus on the influence of TiO_2 sol-gel volume and heating temperature on TiO_2 coating via spin coating. The morphology and phase of TiO_2 will be studied by using XRD and SEM analyser.

1.2. Problem Statement

The properties of TiO_2 coating depend on the parameter in sol-gel method, substrate cleaning, heat treatment and other factors such as concentration of the fluids. All factors and parameter is very important in determining the nucleation and the growth of crystal phase that will affect the final properties of TiO_2. For titanium dioxide, it has 3 main phase which are anatase, rutile and brookite. In this study, the main focus is brookite, the least known of TiO2 because of its difficultness to produce.

There are several deposition methods and technique to produce TiO_2 thin film which is hydrothermal synthesis, chemical vapor deposition, metal organic vapor deposition, pulsed laser deposition and many more. But in this study, the deposition technique that will be use is on sol-gel method. The main principle for producing TiO_2 is the procedure and raw materials involved in some of these preparation techniques will affect the morphology of TiO_2. Temperature is one of the important factor that will affect the morphological of TiO2. For an example, (Muaz et al., 2016) stated that the AFM topology images of as-deposited and annealed TiO2 thin films at 573 and 773 K were taken in a scan area of 1μm x 1μm, the surface topology of the as-deposited revealed that the films surface is looks rather smooth, compact, bulky and not uniform is found and when the annealing temperature was increased to 573 K, some column-like grains were seen while the films annealed at 773 K, a more uniform surface morphology was observed, however, as the annealing temperature increased the grains tend to agglomerate. In this research, different temperature will be use after TiO_2 coating were deposited on the substrate.

The amount of volume of sol-gel TiO_2 also will help to improve the properties of TiO_2 thin film coating because a perfect amount of TiO_2 that coated on the substrate will give better result of coating application because it will effect the viscosity and the concentration of the solution while spin coating were spinning. So this study will investigate the most suitable amount to produce better thin film coating. Moreover, there is not much research has been done on the effect of amount of sol-gel TiO_2 via spin coating.

Hence, heating temperature and the volume of sol-gel TiO_2 will be the main subject of this project and will be focusing on its morphology and phases of thin films that produced via spin-coating sol-gel method.

1.3. Objectives

I. To investigate the effect of sol volume on morphology and phase of brookite thin film deposited in understanding the thickness of thin film, and

II. To investigate the effect of heating temperature of brookite thin film in term of crystal phase, grain size and surface morphology of thin film deposited in understanding the material properties.

1.4. Scope of Work

The scope of this research focus on the effect of sol volume and heating temperature of TiO_2 on substrate by using Spin-Coater. The analysis of TiO_2 thin film properties derived from different volume and temperature on substrate. The image of the TiO_2 microscope will be characterized using:

I. X-Ray Diffraction (XRD), and

II. Scanning Electron Microscopy (SEM).

1.5. Importance of the Study

The importance of this study is to investigate how much volume of TiO_2 coated on the substrate during spin-coating process can affect the morphology and phases of TiO_2. The amount of TiO_2 sol-gel drop on substrate will be different in producing the thin films. Besides that, the effect of heating temperature on the TiO_2 morphology and phases also will be investigate while conducting the experiment.

CHAPTER 2

LITERATURE REVIEW

In chapter 2, introduction about thin coating will be included. The properties, application, sol-gel method, and spin coating also will be explained in this chapter based on the reading on past study and research. The parameter involved in this study, volume of TiO_2 sol-gel and temperature also included to get more understanding about the parameters.

2.1. Introduction

Thin film coating is a layer of a material that have minimal thickness ranging from nanometer to micrometer. It is a deposition of a liquid on the surface of substrate. The deposition can be done in many way like stated by (Schmiedova et al., 2015), gas phase methods (PVD and CVD) and liquid phase ones (dip-, spin- or spray-coating, doctor blade spreading, roller coating etc.), generally, the wet coating techniques constitute a simpler alternative to the vacuum processes requiring sophisticated instrumentation and are therefore favored for large-scale manufacturing. The purpose of thin film coating development is because to self-clean the glass and maintain the surface uniformity.

Thin film has numerous advantage such as simple fabrication, require small amount of material, flexible and non-breakable. Other than that, thin film also can be applied on different type of surface like glass, battery, mirror, tiles and etc. Thin film coating deposition depend on the various factors that involved while producing such as choosing the right material, technique use to synthesis the TiO2 and parameter controlled in the experiment.

In this study, TiO_2 thin film we be synthesis by using sol-gel methods and the focus will be on the amount of volume of TiO_2 sol-gel and the heat treatment temperature. After that, the morphology and the phase of the thin film will be analyse using XRD and SEM analysis.

2.1.1. Properties of TiO_2

For properties, Titanium Dioxide has a low density, weight and modulus. In thin film application, hydrophilic nature on the surface had been discovered in photocatalytic and keep evolving as self-cleaning.

Table 2.1 Physical and Thermal Properties of Titanium Dioxide

Properties	Metric
Density	4.23 g/cm3
Molar Mass	79.9378 g/mol
Boiling Point	2,972° C
Melting Point	1,843° C

Table 2.2 Chemical Composition of TiO_2

Element	Content (%)
Titanium	59.93
Oxygen	40.55

Table 2.1 and Table 2.2 shows general physical and chemical properties of TiO_2. For phases in Titanium Dioxide which is Anatase, Rutile and Brookite, each have different properties. Titanium dioxide have a unique properties that make it more interesting to be study more especially in thin film application. In (Royal Society Of Chemistry, 2017) study stated that TiO_2 has high brightness and a very high refractive index enable the light passes through the crystal slowly and its path is substantially altered compared to air. Essalhi et al

(2016) also stated that Titanium dioxide is a white inorganic solid substance which is thermally stable, non-stable, non-flammable, poorly soluble, cheap non-toxic material that has very good semiconducting properties.

2.1.2. Application of TiO_2

In recent studies, Weng et al (2013) stated that TiO_2 films are known for their hydrophilic and photocatalytic characteristics that help in the purification, treatment of polluted water and hydrophilicity as the “self-cleaning” property of flat glass surfaces could mitigate the harm caused by dust. While (Educational et al., 2013) stated that the nanostructured materials made up of grain or clusters in the nanometric range exhibit new optical properties of interest in many area of application such as electroluminescent devices, chromogenic, optical and chemical sensors, modulators and display devices. Other than that, photocatalytic also one of the application of TiO_2, Patil et al (2015) stated that the photocatalytic ones have been more widely explored due to the increased concern over the protection of ecology and environment from several wastes arising from different kinds of industry.

Titanium dioxide is widely used as a white powder pigment because of it characteristic that have very high refractive index. By having a very high refractive index, that’s mean only small amount of pigment needed to achieve a white opaque coating. Resistance to discolouration under ultraviolet light helps plastic undergo discolouration while under sunlight, it also have a high refractive index that use in sunscreen to protect skin.

Photocatalytic have been attract the attention of researchers due to its oxidative and hydrolysis properties. While exposed under the sunlight, it becomes more hydrophilic and can be used as an anti-fogging coating and self-cleaning windows. Meher et al (2014) stated that the primary and important stage is breaking down of the organic contaminants through photocatalytic activity, it makes the glass surface super hydrophilic which reduces the water contact angle and in the super hydrophilic stage water forms a thin layer on glass and the dirt is washed away.

2.1.3. Brookite

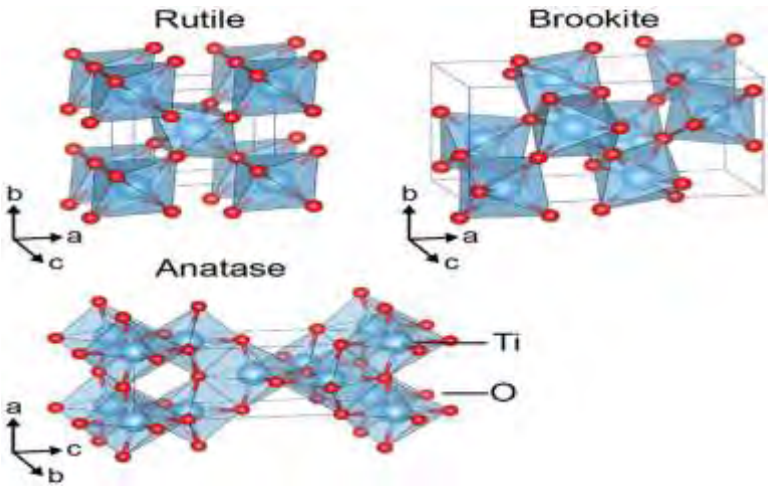

Figure 2.1 Crystal structures of TiO_2, (Haggerty et al., 2017)

Based on Figure 2.1, it shows three crystal structure of TiO_2 that is anatase (tetragonal), rutile (tetragonal) and brookite (orthorhombic). Each of the phase have different structure of crystallinity which means they have a different of properties. In this study, it will be focused on brookite.

Di Paola et al (2013) stated that brookite structure composed is octahedral, each with a titanium atom at its center and oxygen atoms at its corners, octahedral share edges and corners with each other to such an extent as to give the crystal the correct chemical