



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**EFFECT OF ELECTRICAL GROUNDING OF SUBSTRATE ON  
THE MAGNETRON SPUTTERING OF TITANIUM FILM**

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia  
Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering  
(Engineering Materials) (Hons.)

by

**MOHD NABIL FIKRI BIN HANIF**

**B050910187**

**880207085039**

**FACULTY OF MANUFACTURING ENGINEERING**

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# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

## BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: Effect of Electrical Grounding of Substrate on the Magnetron Sputtering of Titanium Film

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering B.ENG. (HONS) (Engineering Materials). The member of the supervisory is as follow:

.....

(Principal Supervisor)

.....

(Co-Supervisor)

## **ABSTRACT**

Property of thin film depends on the voltage bias that applied to the depositing substrate during the D.C. magnetron sputtering process. . This shows ionized atoms or molecules are involved in deposition of film to the substrate. The supposed accumulation of charges (ions) on the surface of substrate may affect the deposition of the incoming ionized atom or molecules because like-charges exert repulsive electric force. Improvement of electrical grounding of substrate used for the deposition of Ti film may help to reduce the charge accumulation on the surface of substrate. The reduction of the accumulated charges on the substrate may decrease the repulsion effect on incoming ionized sputtered atoms and increases the deposition rate of sputtered atoms on the substrate. The effect on the Ti film may be exhibited through the microstructure and phase composition of the film. Electrical grounding condition of depositing substrate during Ti film deposition will be varied using different: (1) Design of substrate holder, (2) substrate material, and (3) coating thickness linking substrate with substrate holder. Obtained Ti films were characterized using were characterized with optical microscopy, SEM, and electrical resistivity measurement.

## ABSTRAK

Sifat-sifat filem nipis bergantung kepada kadar voltan yang dikenakan ke atas substrat deposit semasa proses pemercikan DC dilakukan. Ia menunjukkan atom terion atau molekul berlaku dalam pemendapan filem substrat. Pengumpulan caj (ion) pada kebiasaannya terjadi di permukaan substrat boleh menjeaskan pemendapan atom terion atau molekul yang masuk kerana perubahan yang daya elektrik yang dikenakan ditolak.

Peningkatan asas elektrik substrat digunakan untuk pemendapan Ti filem boleh membantu untuk mengurangkan pengumpulan caj di permukaan substrat. Pengurangan caj terkumpul pada substrat boleh mengurangkan kesan penolakan atom yang terion pada pemercikan atom dan meningkatkan kadar pemendapan pemercikan atom pada substrat. Kesan ke atas filem Ti itu boleh dipamerkan melalui mikrostruktur dan fasa komposisi filem. Keadaan asas elektrik pemegang substrat semasa Ti filem pemendapan akan diubah yang digunakan berbeza iaitu: (1) Bentuk pemegang substrat, (2) Bahan-bahan substrat, dan (3) Ketebalan lapisan substrat yang dihubungkan dengan pemegang substrat. Merujuk kepada Ti filem, pengujian sifat-sifat Ti filem dilakukan dengan menggunakan mikroskop optik, Scanning Electron Microscopy (SEM), dan pengukuran rintangan elektrik ke atas substrate.

## **DEDICATION**

“Special to my parents, my family and all my friends that helps me in finishing this research”.

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In the Name of Allah, the Most Gracious, the Most Merciful.

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## SIGN AND SYMBOLS

A	-	Ampere
AC	-	Alternating Current
Ag	-	Argentum
Al	-	aluminium
Ar	-	Argon
Cu	-	Copper
CVD	-	Chemical Vapor Deposition
eV	-	Electron Valence
Eq	-	Equation
G cm <sup>-3</sup>	-	Gram per centimetre cubic
Hz	-	Hertz
K	-	Kelvin
Kg	-	kilograms
KJ/Mol	-	kilo Joule per mol
KM	-	Kilometer
kV	-	kilo volt
Kw	-	Kilowatts
L	-	Liter
mA	-	miliampere
max	-	Maximum
mbar	-	millibar
MHz	-	Megahertz
mins	-	Minutes
mm	-	millimeter
Mn	-	Manganese
Mpa	-	Mega Pascal
m/s	-	meter per second

NaCl	-	Sodium Chloride
Ni	-	Nikel
nm	-	Nanomicron
N <sub>2</sub>	-	Nitrogen
O <sub>2</sub>	-	Oxigen
Pa	-	Pascal
Psi	-	Pounds Per Square Inch
PVD	-	Physical Vapor Deposition
RF	-	Radio Frequency
Sdn. Bhd	-	Sendirian Berhad
SEM	-	Scanning Electron Microscope
T	-	Temperature
Ta	-	Tantalum
Ti	-	Titanium
TiO <sub>2</sub>	-	Titanium Dioxide
T <sub>s</sub>	-	Substrate Temperature
T/T <sub>m</sub>	-	Thorton Model unit
UTeM	-	Universiti Teknikal Malaysia Melaka
V	-	Volt
V <sub>s</sub>	-	Voltage Bias
Wt %	-	Weight Percentages
XRD	-	X-Ray Diffraction
Zn	-	Zinc
ZnO	-	Zinc Oxide
°C	-	Degree Celsius
%	-	Percentage
$\rho$	-	Density
nm	-	nanometer



# CHAPTER 1

## INTRODUCTION

Chapter 1 will give a brief explanation about this project, starting with the background of the project title, “Effect of electrical grounding of substrate on the magnetron sputtering of titanium film”. This chapter will also discuss about the problem statement, the objective and the scope and limitation of this project.

### 1.1 Background

Nowadays, the technique of deposition of thin film by physical vapor deposition (PVD) has been used widespread in many industrial sectors. This technique is used for a variety of field coating application such as metalworking industry, biomedical application and optical or electrical component. High hardness, wear and corrosion resistance, low friction, and specific optical or electrical properties and decorative color and often complex combination of properties are the reasons about increasing demand in the industry. Magnetron sputtering for thin film process is one of the technology that developed by PVD. It's the one of choice for deposition of a wide range of industrially important coating. Theoretically, this process allows deposition of metal, alloy, ceramic, and polymer thin film into any substrate material. Sputtering, also known as sputter etching, is used for patterning semiconductor wafers, for cleaning surfaces, for micromachining, depth profiling, and a number of applications which require careful, microscopic erosion of a surface. Sputter deposition is used for film deposition on semiconductor wafers, on magnetic media and head surfaces, for coating tools and cutting surfaces for wear resistance (this includes, by the way, such tools as shaving razors), for reflective coatings on window glass, for coating the insides of plastic bags and the

surfaces of automobile parts, and a number of other wide ranging applications. Sputtering is usually practiced by means of plasmas which generate charged particles which can be accelerated towards a surface electrically. Sputtering is simply the process of erosion of that surface by the energetic particles, a sort of atomistic sandblasting. Sputter deposition is nothing more than the accumulation of these atoms which are blasted off the surface onto a nearby sample. For the most part, this chapter deals with sputtering and sputter deposition with a slant towards semiconductor processing. However, the discussion is wide enough that other areas are introduced from time to time as needed.

## **1.2 Problem Statement**

The Ti film fabricated by many techniques such as reactive sputtering in PVD, sol-gel coating, atomic layer deposition and etc. the microstructure, crystallographic texture, state of stress and chemical compound are influenced by particle bombardment of the growing film, deposition temperature, and composition of elements in the coating material. However there are other parameters that may contribute to the characteristic of the Ti thin film.

Electrical grounding of the substrate during deposition of the Ti film may help to reduce the charge accumulation on the surface of substrate. These suggest that electrical grounding of the substrate may affect the deposition of Ti film. Its effect may be exhibited through the microstructure and phase composition of the film. Property of thin film depends on the voltage bias that applied to the substrate. When voltage is increased on thin film shows the higher hardness and compressive stress value on microstructure. This shows ionized atoms or molecules are involved in deposition of film to the substrate. The supposed accumulation of charges (ions) on the surface of substrate may affect the deposition of the incoming ionized atom or molecules then this may affect the microstructure and phase composition of the deposited thin film.

In order to solve the problem, this project will study either the electrical grounding effect towards the process. The study must be considered about testing the different type of thin film and different voltage apply to substrate holder.

## **1.3 Objectives**

The objectives to be achieved at the end of this project are as below:

- i. To synthesize the titanium thin film on the substrateS using Direct Current (DC) sputtering technique.
- ii. To study the effect of the electrical grounding of the substrate on the microstructure and phase composition of the deposited titanium dioxide thin film.
- iii. To compare the differences in the effect between all of the substrates

#### **1.4 Scope**

This project will determine the effect of electrical grounding on the substrate during deposition of Ti. Three main factors that influence the electrical grounding of the substrate has been identified and they are:

- i. Conductivity of substrate and coating material
- ii. Physical contact between substrate and holder
- iii. Coating thickness on a substrate - holder which form a conduction path between substrate and holder.

The titanium oxide is selected as a target in DC magnetron sputtering. The effect electrical grounding is identified by using three types of tester and they are the optical image by using a microscope, thickness and surface morphology changing by using Scanning Electron Microscope and the value of the resistivity effect of the film coated on substrates. After the result has shown, it will compare together to see either the result shows the grounding is one of critical parameters or wisely.

## **CHAPTER 2**

### **LITERATURE REVIEW**

This chapter will explain about all elements used for this report. First explanation will come to machine, machine process, material (specimen) and process sequences for magnetron sputtering in order to know the element used and identify the parameter appear toward the study. The studies also related about analyzing the structure of the specimen after sputtering process.

#### **2.1 Ti properties and applications**

Titanium (Ti) is a lustrous transition metal with a silvery-gray color, high strength and low density. It is a chemical element and the number of atomic is 22. To produce strong lightweight alloys, titanium usually can be alloyed with iron, aluminum, vanadium, and many more among others element. In coating, titanium is nearly as resistant to corrosion as platinum, and resists many salt solutions, acids and even chlorine gas. It is also not water soluble, save perhaps in concentrated acids. Titanium is nonmagnetic and has good heat-transfer properties. Its coefficient of thermal expansion is somewhat lower than that of steel and less than half that of aluminum. Titanium and its alloys have melting points higher than those of steels. Titanium has the ability to passively and thereby exhibits a high degree of immunity to attack by most mineral acids and chlorides.

The crystal structure of titanium at ambient temperature and pressure is close-packed hexagonal ( $\alpha$ ) with a  $c/a$  ratio of 1.587. Slip is possible on the pyramidal, prismatic and basal planes in the close-packed directions. At about 890°C, the titanium undergoes an allotropic transformation to a body-centered cubic  $\beta$  phase which remains stable to the melting temperature (Figure 1).

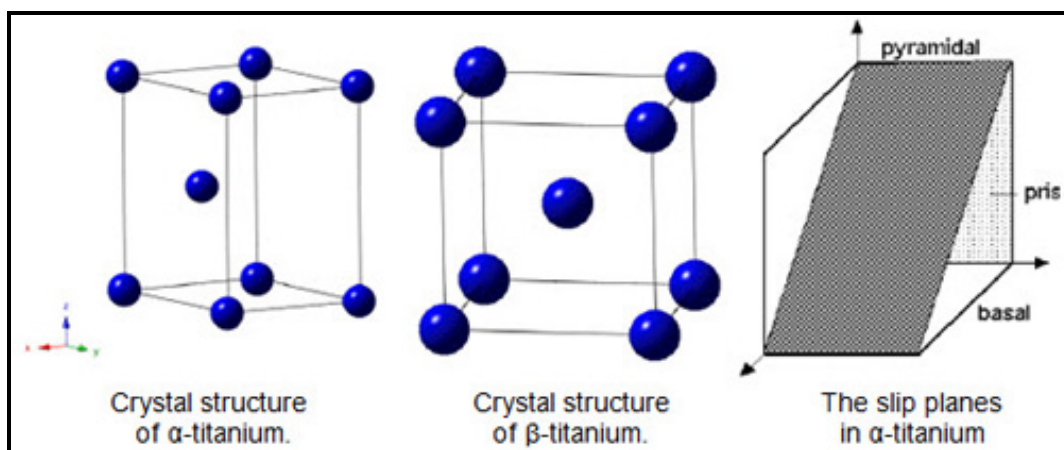


Figure 2.1: Crystal structures of Titanium

Titanium is a remarkable, very light metal with a silvery color and is the fourth most abundant element in the earth's crust. As a rule, all ignoble metals are extracted as a metal oxide. The lower the position of the standard potential of the metal in the nobility table, the more difficult it becomes to separate the metal from the bound oxygen. Iron has a potential of -0.44 volts and can be quite easily separated from the bound oxygen. This can be represented

by the reduction formula  $2\text{FeO} + \text{C} + e$

$\square 2\text{Fe} + \text{CO}_2$

furnace, and the “e” in the formula stands for the energy that needs to be added to make this reduction possible.

If one adapts this mechanism to titanium oxide, also called rutile, nothing happens, since titanium oxide must be treated very differently in order to separate these two strongly bound elements.

This process is as follows:

1. Rutile ( $\text{TiO}_2$ ) is chlorinated with cokes to produce titanium tetrachloride ( $\text{TiCl}_4$ ) and carbon dioxide ( $\text{CO}_2$ ). Titanium tetrachloride is a colorless liquid that remains to be refined.
2. Magnesium or sodium is added to the titanium tetrachloride in an inert environment.
3. This produces the chemical reaction  $\text{TiCl}_4 + 2\text{Mg}^{2+} \rightarrow \text{Ti} + 2\text{MgCl}_2$ . Finally, we obtain titanium sponge and magnesium chloride.
4. The titanium sponge is pressed into blocks and melted under inert gas to form ingots, to which alloy elements and scrap may be added. These ingots are then rolled to produce all kinds of half-finished products such as sheets or rods.

It will by now be clear that it takes a lot of energy to win titanium from its ore. This has to do with the fact that it takes a lot of energy to prepare magnesium or sodium. Therefore a large part of the price of titanium is determined by energy prices (Stepanov, 2011).

$\text{TiO}_2$  belongs to the family of transition metal oxides. There are four commonly known polymorphs of  $\text{TiO}_2$  found in nature: anatase (tetragonal), brookite (orthorhombic), rutile (tetragonal), and  $\text{TiO}_2$  (monoclinic). Besides these polymorphs, two additional high-pressure forms have been synthesized from the rutile phase (Stepanov, 2011).

In this review, only the crystal structures (Table 1) and properties of the rutile and anatase polymorphs are considered.

Table 2.1: Crystal Structure for Titanium Dioxide (TiO<sub>2</sub>) (Stepanov, 2011).

Propeties	Rutile	Anatase
Crystal structure	Tetragonal	Tetragonal
Lattice constant (Å)	$a = 4.5936$ $c = 2.9587$	$a = 3.784$ $c = 9.515$
Space Group	P42/mnm	I41/amd
Molecule (cell)	2	2
Volume/molecule (Å <sup>3</sup> )	31.2160	34.061
Density(g cm <sup>-3</sup> )	4.13	3.79
Ti-O bond length (Å)	1.949(4) 1.980(2)	1.937(4) 1.965(2)
O-Ti-O bond angle	81.2° 90.0°	77.7° 92.6°

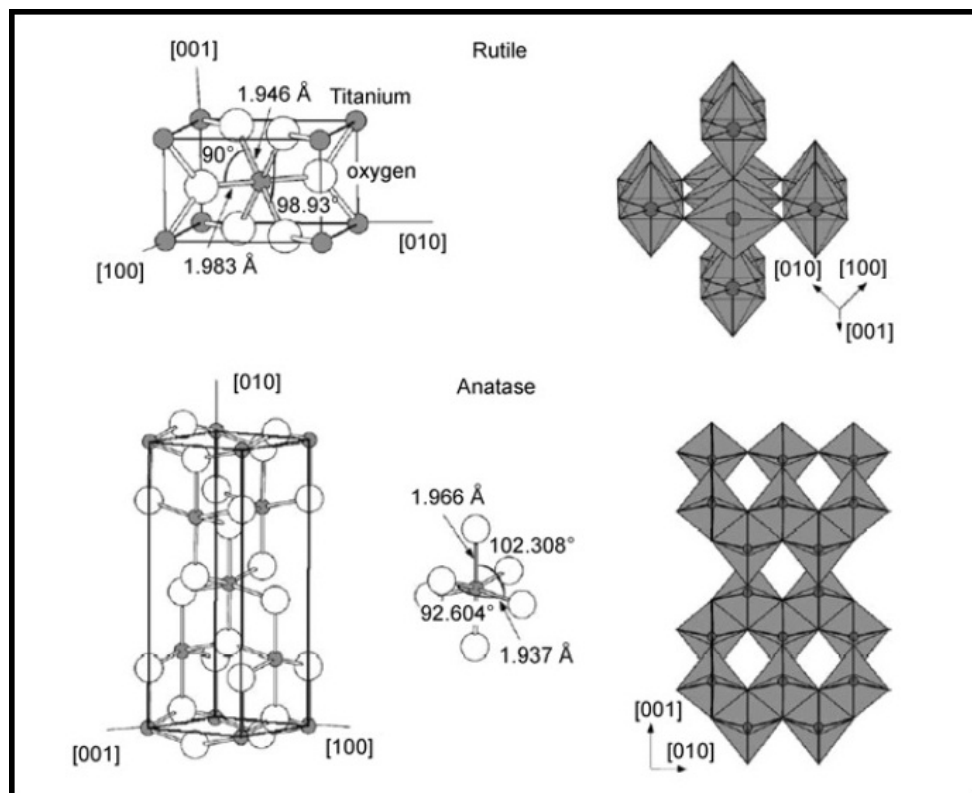


Figure 2.2: Crystal structure of rutile and anatase phases of TiO<sub>2</sub> (Stepanov, 2011).

Rutile  $\text{TiO}_2$  has a tetragonal structure and contains 6 atoms per unit cell. The  $\text{TiO}_6$  octahedron is slightly distorted. The rutile phase is stable at most temperatures and pressures up to 60 kbar, where  $\text{TiO}_2$  becomes the thermodynamically favorable phase. anatase structures transformed to the rutile phase after reaching a certain particle size, with the rutile phase becoming more stable than anatase for particle sizes greater than 14 nm. Once the rutile phase formed, it grew much faster than the anatase. The activity of the rutile phase as a photocatalyst is generally very poor. However, the rutile phase can be active or inactive, depending on its preparation conditions (Yingyot, 2008).

Anatase  $\text{TiO}_2$  also has a tetragonal structure but the distortion of the  $\text{TiO}_6$  octahedron is slightly larger for the anatase phase, as depicted in Figure 1. The anatase phase is more stable than the rutile at 0 K, but the energy difference between these two phases is small ( $\sim 2$  to 10 kJ/mol). The anatase structure is preferred over other polymorphs for solar cell applications because of its higher electron mobility, low dielectric constant and lower density. The increased photoreactivity is because of the slightly higher Fermi level, lower capacity to adsorb oxygen and higher degree of hydroxylation in the anatase phase. The reactivity of (001) facets is greater than that of (101) facets in an anatase crystal. Uniform anatase crystals containing 47% (001) facets using hydrofluoric acid as a morphology controlling agent (Stepanov, 2011).