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FabricationofThin FilmUsing Modified Physical Vapor Deposition (PVD) Module

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Abstract
This paper explains the fabrication of thin film using modified Physical Vapor Deposition (PVD) Module. Physical Vapor Deposition (PVD) is a variety of vacuum deposition and is a general term used to describe any of a variety of methods to deposit thin films by the condensation of a vaporized form of the material onto various surfaces. The surface morphology of various such as Titanium Dioxide and Aluminum thin film has been studied. The Titanium Dioxide and Aluminum thin film has been fabricated on Silicon (Si) substrate using modified Physical Vapor Deposition (PVD) module system. The process started with the establishment of process flow, process modules, and process parameters. Two modules were developed. The characteristics prior to the thin film fabrication namely surface morphology, metal thickness characterization and V-I characteristic were recorded. The samples were characterized by Optical Microscope, Atomic Force Microscope (AFM),X-ray diffraction (XRD) and I - V characterization. The result and data were analyzed and applied in the fabrication of thin film using various materials. The thin film fabrication process used Titanium Dioxide (TiO_2) nanopowder and Aluminum (Al_2O_3) nanopowder for the coating process. The result for each processes are presented in this paper.
***Keywords*:**Aluminum Oxide, Titanium Dioxide, OpticalMicroscope, AFM, SEM andXRD.

I. INTRODUCTION

Semiconductor device fabrication is the process used to create chips, the integrated circuits that are present in everyday electrical and electronic devices. It is a multiple-step sequence of photographic and chemical processing steps during which electronic circuits are gradually created on a wafer made of pure semiconducting material. Silicon is the most commonly used semiconductor material today, along with various compound semiconductors.

Thin films are thin material layers ranging from fractions of a nanometer to several micrometers in thickness. Electronic semiconductor devices and optical coatings are the main applications benefiting from thin film construction. Work is being done to fabricate thin films with chemical such as titanium oxide for making a semiconductor and zinc oxide for application such as gas sensor, piezoelectricity material and many researches in solar cell.

In particular, the use of such coatings on cutting tools can extend the life of these items by several orders of magnitude. Research is being done on a new class of thin film inorganic oxide materials, called amorphous heavy-metal cation multi component oxide, which could be used to make transparent transistors that are inexpensive, stable, and environmentally benign. Thin-films are applied to surfaces using one of many techniques of thin-film deposition.

Basically, devices are fabricated on silicon and function as semiconductor because of the unique properties of this material. There are four main reasons for the selection of silicon as the primary semiconductor material:

i. Abundance of silicon
ii. Higher melting temperature for wider processing range
iii. Wider temperature range of operation
iv. Natural growth of silicon dioxide

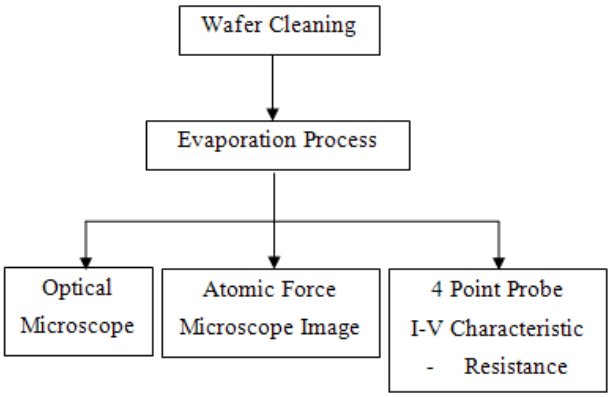
Silicon is the second most abundant element on earth and makes up about 25% of the earth's crust. If processed properly, silicon can be refined into sample quantities of the very pure form necessary for semiconductor fabrication which leads to lower costs. Silicon's melting temperature of 1412°C is much higher than the melting temperature for germanium which is 937°C. This higher melting temperature permits silicon to withstand high-temperature processing. Another advantage to using silicon is that a semiconductor device made

from silicon can function over a wider temperature range than germanium, which increases the semiconductor's application and reliability.

It is found that, a major disadvantage of most ceramic listed above is their high in cost. This factor has led to a situation of limited use commercially rather for research purpose only. In general, commercial ceramic are made from starting ceramic powder which will later been shaped to form desired ceramic. The starting powders are often made by energy – intensity processes or by other expensive methods adapted to produce high purity starting powders. Higher degree of purity will cause decrease in impurity content which will alter the total performance of the ceramic. The powders are then used to fabricate thin films on substrate.

In this paper, the $Al_2O_3$and TiO_2thin film was fabricated on the silicon (Si) substrate. Then, the morphology of the Al_2O_3 and TiO_2 thin film after evaporationprocess on the surface of the thin film is studied. Thesurface on the sampleare been characterized by Digital Camera, Optical Microscope (OM), Atomic Force Microscope (AFM), Scanning Electron Microscope (SEM), Energy Dispersive Spectrometer (EDS),I–Vcharacterization and X-ray diffraction (XRD). Influence of the evaporation process to the physical and chemical properties of Al_2O_3 and TiO_2 thin film is discussed.

II. METHODOLOGY



Evaporation Process

A. Formation of thin film process

First, aSilicon (Si) substrate wafer is cleaned with the Buffered Oxide-Etch (BOE) is used to remove organic and inorganic contaminants residue, respectively. It is usually followed byDeionized water (DI). The substrate was placed in anevaporation chamber which contains mixture of Titanium Dioxide (TiO_2)nanopowder and graphite (CaC_6) and mixture of Alumina (Al_2O_3)nanopowder and graphite (CaC_6) as a material source in tungsten boat. The chamber was pump up to 10^{-5} Torr using a high vacuum pumping system. It will consist of 45 minutes of waiting. Then, the evaporation process can be started by increasing the voltage of the tungsten boatabout 10% from its maximum range for 1 minute (tungsten – dark red), then up to 20% about 45 seconds (tungsten – red) and then to 30% for about 7 minute (tungsten – bright orange). The current range when we operate the PVD module is 73A until 83A for the maximum power. The colorof the tungsten will change according to the value of voltage supplied. After 7 minute at 30% of the maximum voltage source, the toggle is turn back to 0% and let to be cooled for 5 minute before the chamber is opened.

B. Measurement of material

To continuethe experiment, the compound of material must be measured to get a good result. For this experiment the compound of material were as follows:

Alumina (Al_2O_3)	Titanium Dioxide (TiO_2)
1 spoon of spatula = 50.4 mg	1 spoon of spatula = 96.2 mg

C. The experimental parameters

Various times

a). Titanium Dioxide (TiO_2)

	PARAMETER				
	1	2	3	4	5
Evaporation time(minutes)	0	1	5	10	15
Quantity of material (1 spatula)	1	1	1	1	1

b). Alumina (Al_2O_3)

	PARAMETER				
	1	2	3	4	5
Evaporation time(minutes)	0	1	5	10	15
Quantity of material (1 spatula)	1	1	1	1	1

D. Thin film evaporation process

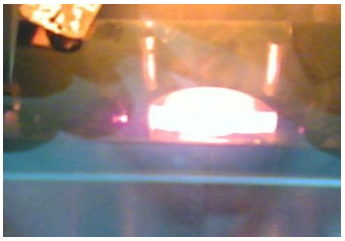


Figure 1: Evaporation process using PVD module

TheTiO_2 and Al_2O_3 thin film deposited on Si substrate is now already done. The system is located in *Microelectronic Laboratory* of FKEE, UTHM. The substrate was cut into several pieces of sample in order to provide thefundamental thickness (TiO_2 and Al_2O_3thin film). Every sample is characterized using Digital Camera, Optical Microscope, Atomic Force Microscope (AFM), Scanning Electron Microscope (SEM), Energy Dispersive Spectrometer (EDS), I – V characterization and X- ray diffraction (XRD).

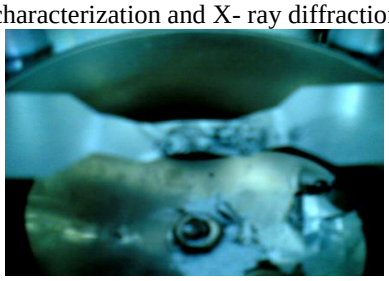


Figure 2: After evaporation process using tungsten boat

E. Sol-Gel

The sol-gel process is a wet-chemical technique such as chemical solution deposition widely used recently in the fields of materials science and electrical engineering. Such methods are used primarily for the fabrication of materials starting from a chemical solution which acts as the precursor for an integrated network (*gel*) of either discrete particles or network polymers. Thus, the sol evolves towards the formation of a gel-like diphasic system containing both a liquid phase and solid phase whose morphologies range from discrete particles to continuous polymer networks. Research had done using this method and comparing between this method and evaporation method. The result of the experiment shown that which one is the better method to fabricated a thin film.

F. Optical Microscope

The use of optical microscope is to magnify the sample up 30X, before having a rough look on surface of the sample. The sample is been placed on the tray and by varying the focus point of the lens, the optical image of each sample are recorded.

G. Atomic Force Microscope (AFM)

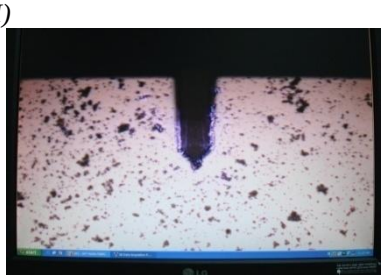


Figure 3: Scan surface morphology using XE-100 AFM

The AFM (PARK SYSTEM - XE-100) system is used to measures the sample surface using atomic force between tip and sample and also to observe the morphology of the TiO_2 and Al_2O_3 thin film after the evaporation process. The system is located in Microelectronic and Nanotechnology, Shamsuddin ResearchCentre (MiNT-SRC), UTHM. Sample is placed on the substrate holder and been inserted into the AFM. In this project, non- contact cantilever had been used. The non - contact cantilever had been choose because using this cantilever,sample will not damage and can overview the topology, amplitude and phase on the surface of the sample. After all the surface area of the sample is scan, we can observe the surface morphology, roughness and thickness on surface topology. All the result of the sample iscaptured.

H. Scanning Electron Microscope (SEM)

The SEM (JEOL JSM – 6380 LA) system is used to observe the surface morphology of the TiO_2 and Al_2O_3 thin film after the evaporation process. The system is located in *Makmal Sains Bahan* of FKMP, UTHM. Sample is placed on the substrate holder and been inserted into the SEM chamber before pumping up the chamber using turbo and rotary pump. The focus of image appear on the online video of the SEM is adjusted until clear image is produced. The sample images with the magnification of 100X are recorded.

I. Energy Dispersive Spectrometer (EDS)

The SEM is equipped with EDS system that allows the user to analyze the elements exists by marking the area in square, point or circle. In the present works, the rectangular type is chosen. EDS will verify and characterized the sample. The characterized result will appear in form of graph. Every sample will undergo 1 times of test at different areas and every result obtained is recorded.

J. X – Ray Diffraction (XRD)

The X- ray diffraction (XRD) is used to detect any elements on the surface material. The result in form of graph is compared with the pre-loaded library before assumption of material can be made. The data in form of graph is recorded.

K. I – V Characterization

The I-V characterization can be used to determine the resistivity of the sample in form of voltage versus current. Every data of the sample is recorded and been analyzed.

III. RESULT AND DISCUSSION

A. Digital Camera

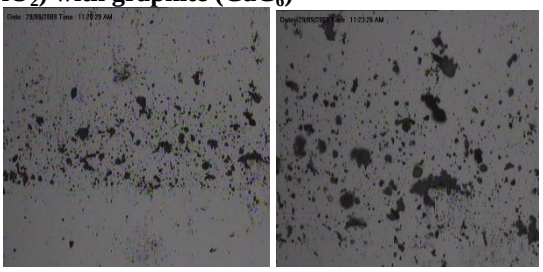
The Digital Camera is a camera used to capture photographs and stores the images digitally via an electronic image sensor. From the figure 4 below, the image of digital camera shown that there 2 different layer on sample surface. The clean side is the hiddenpart of the sample when evaporation process.It is because that part is hidden by the clip that used to holding the sample. Other part of sample shows a TitaniumDioxide or Alumina single layer. From general perception, it can prove that existence of thin film.

Figure 4: Thin film of the Mixture ofTitanium Dioxideand graphite.

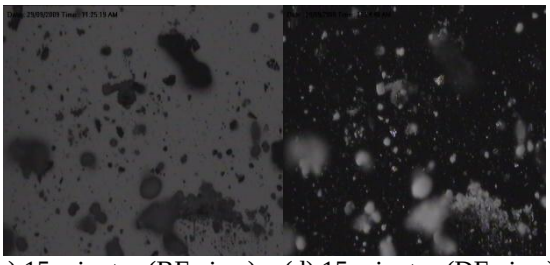
B. Optical microscope test

The Optical Microscope is use to observe the surface area after the evaporation process. The Optical Microscope is connected with the computer and use IMAPS Version. 4.0 Professional Edition software.Figure 2 shows the Optical Microscope images of the TiO_2 and Al_2O_3 thin film after the evaporation processat various times.The images clearly show that the surface morphology changed after evaporation process at specific time. Figure 2(a), 2(b),2(c) and 2(d) show that the images have a different surface morphology. Therefore, it is interesting to study the evaporation process using the tungsten boat.The image that shows are like below:

Mixture Titanium Dioxide (TiO_2) with graphite (CaC_6)



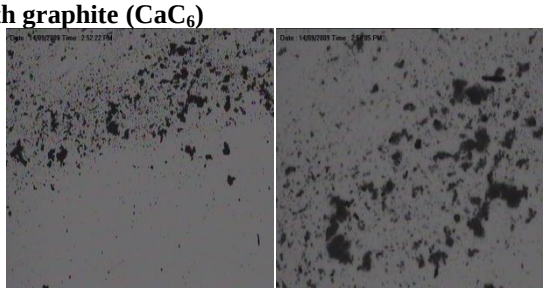
(a) 1minute (b) 5minutes



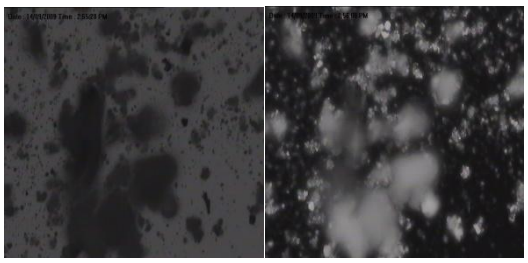
(c) 15 minutes (BF view) (d) 15 minutes (DF view)

Figure 5: The surface morphology of Titanium Dioxide (TiO_2) thin film. a) TiO_2 thin film after 1 minutes theevaporation process. b) 5 minutes. c) 15 minutes (BF view). d) 15 minutes (DF view).

Mixture Alumina (Al_2O_3) with graphite (CaC_6)



(a) 1 minute (b) 5minutes

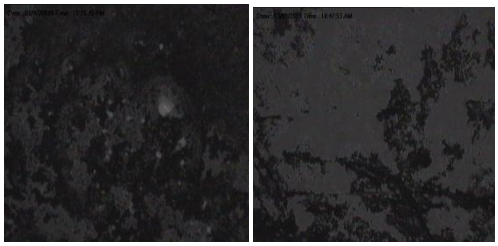


(c) 15 minutes (BF view) (d) 15 minutes (DF view)

Figure 6: The surface morphology of Alumina (Al_2O_3) thin film. a) Al_2O_3thin film after 1 minutes theevaporation process. b) 5 minutes. c) 15 minutes (BF view). d) 15 minutes (DF view).

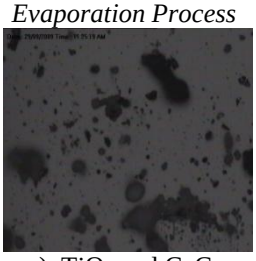
Comparison Sol-Gel and Evaporation Process

Sol- Gel



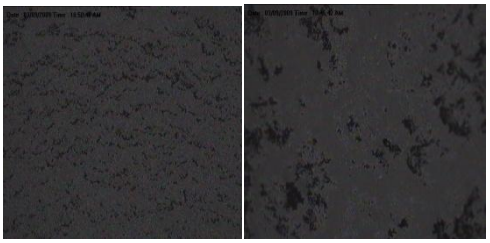
a). TiO_2 and CH_3OH b). TiO_2 and C_2H_5OH

Evaporation Process



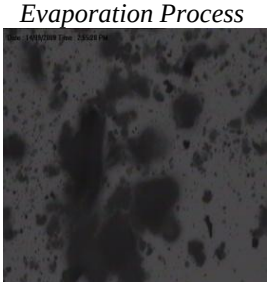
c). TiO_2 and CaC_6

Sol- Gel



a). Al_2O_3 and CH_3OH b). Al_2O_3 and C_2H_5OH

Evaporation Process



c). Al_2O_3 and CaC_6

Keywords– Titanium Dioxide (TiO2), Methanol (CH_3OH)Alumina (Al_2O_3), Ethanol (C_2H_5OH), Graphite (CaC_6)

a. Scanning Electron Microscope Test

Figures4a and 4b shows the scanning electron microscope images of the TiO_2 and Al_2O_3thin film before and after the evaporation processes. From theimages, the evolution of the sample was nearly the same with the optical microscope images. The growth of the rough image of the sample was observed clearly with the sample 15minutes.

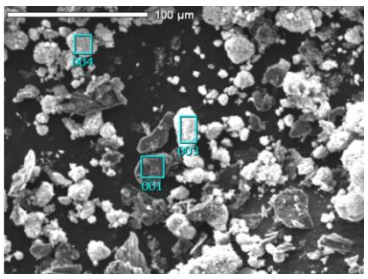


Figure 7a: Surface morphology of TiO_2 thin film after theevaporation process.

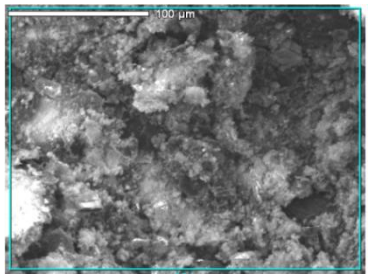


Figure 7b: Surface morphology of Al_2O_3 thin film after theevaporation process.

b. Atomic Force Microscope Test

By using the AFM, we determine the surface morphology in a particular region of thin film. The XEI software with the help of AFM unit is used for this analysis. For example, the topography on the surface ofthin film is been analyzed and shown in the Figures8 and 9. Figure 5a shows that the element of $TiO_2$and graphite were observed on the topography of thin film.

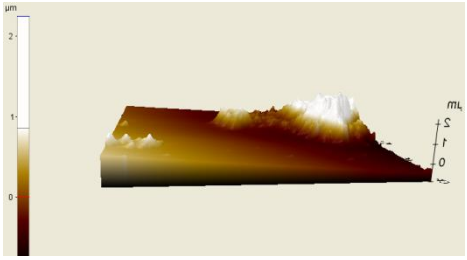


Figure 8a: 3D-image of surface morphology of TiO_2 thin film after theevaporation process using AFM.

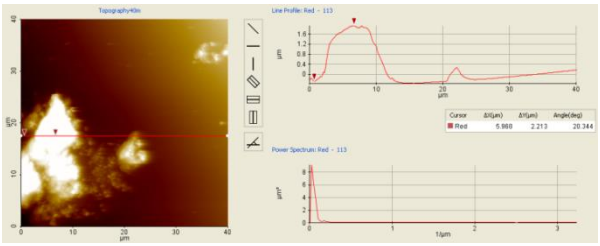


Figure 8b: Thickness of TiO_2 thin film after the evaporation process using AFM. The cursor pair has shown the change of X-axis of the surface selected.

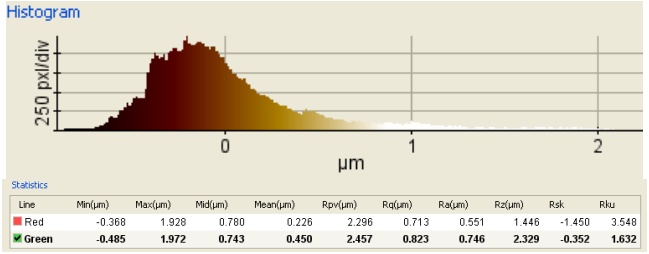


Figure 8c: Roughness of TiO_2 thin film after the evaporation process using AFM. The cursor pair has shown the change of Y-axis at the surface selected

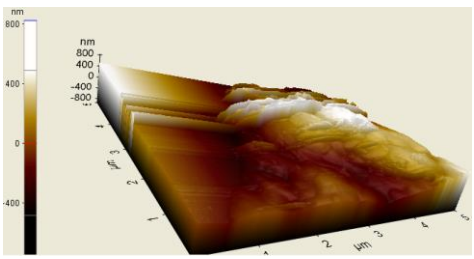


Figure 9a: 3D-image of surface morphology of Al_2O_3 thin film after the evaporation process using AFM.

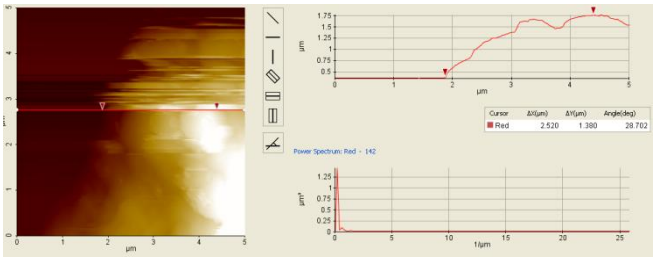


Figure 9b: Thickness of Al_2O_3 thin film after the evaporation process using AFM. The cursorpair has shown the change of X-axis of the surface selected.

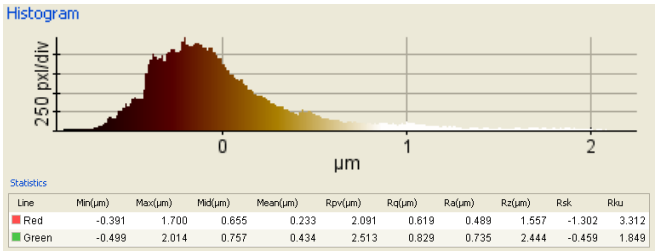
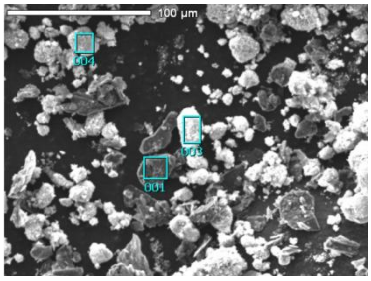


Figure 9c: Roughness of Al_2O_3 thin film after the evaporation process using AFM. The cursor pair has shown the change of Y-axis at the surface selected

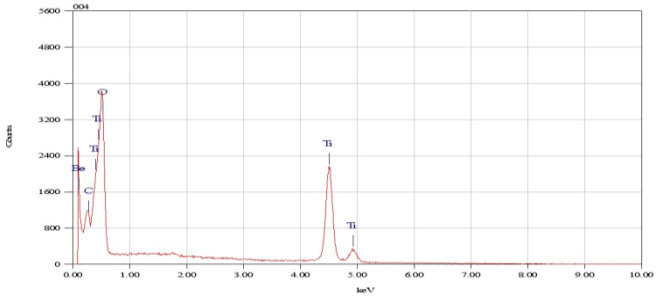
C. Energy Dispersive Spectrometer Test

By using the EDS, we determine the composition of element in a particular region of thin film. The software Analysis Station with the help of SEM unit is used for this analysis. For example, the area from the sample TiO_2 and Al_2O_3 for 15 minutes are been analyzed and shown in the Figures7 and 8. Figure 8 shows that the element of oxygen and titaniumdioxide were observed on the surface of thin film.



a)

Figure 10a): The area selected of sample 15 minutes is analysis using EDS. The resultant analysis (graph) is shown in figure 10b).

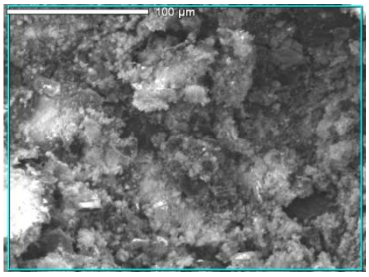


b)

Element		(keV)	mass%	Error%	At%	Compound	mass%	Cation	K
N K	*	0.392	1.30	0.46	2.48			0.7816	
O K		0.525	4.35	0.16	7.29			2.9638	
Al K		1.486	4.53	0.08	4.50			4.6748	
Si K		1.739	89.81	0.09	85.72			91.5798	
Total			100.00		100.00				

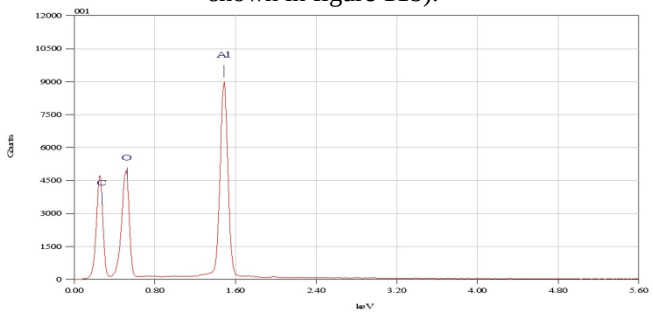
c)

Figure 10c: The resultant result of sample 15 minutes at area 003 is shown. b) The resultant graph with label of related element. c) The resultant element analysis in form of percentage is shown.



a)

Figure 11a): The area selected of sample 15 minutes is analysis using EDS. The resultant analysis (graph) is shown in figure 11b).



b)

Element	(keV)	mass%	Error%	At%	Compound	mass%	Cation	K
C K	0.277	38.86	0.10	51.59			33.3515	
O K	0.525	30.30	0.23	30.19			55.4143	
Al K	1.486	30.84	0.22	18.22			67.9474	
Total		100.00		100.00				

c)

Figure 11c: The resultant result of sample 15 minutes at area 003 is shown. b) The resultant graph with label of related element. c) The resultant element analysis in form of percentage is shown.

a. X – Ray DiffractionTest

The elements compose in the TiO_2 thin film after the evaporation process was also evaluated using XRD analyses. The result is shown in Fig. 6. The peak position is consists of TiO_2 composition.

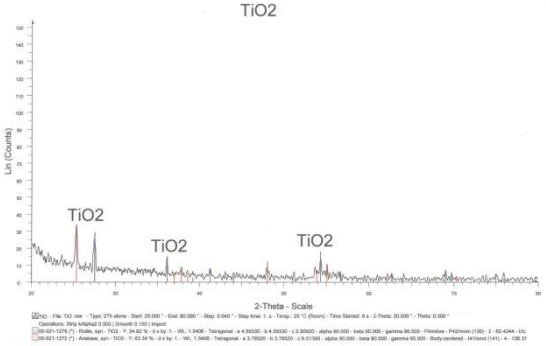


Figure 12:XRD analyses of TiO_2 thin film after the evaporation process.

Figure 12shows that the peak of TiO_2. The result wasconsistent with the scanning electron and microscope atomic force microscope images shown in Figs. 4 and 5.

b. I – V Characterization test

Finally, the I-V characteristic of each samples were evaluated used four point probe. From the I-V characteristic, the resistivity of the TiO_2 and Al_3O_2 thin film after the evaporation processes at various times were calculated.One important indirect measure of coating technique is the sheet resistivity. Sheet resistivity (ρ) is a quantity which combines all the material resitivity with some (but not all) of the geometrical factors and has unit Ω/(ohms persquare).The formula of sheet resistivity and bulk resistivity is shown below:-

$$\text{Sheet resistivity, } \rho = \frac{\pi}{\ln 2} \times \frac{V}{I} \qquad (1)$$

$= 4.523 \left(\frac{V}{I}\right)$

The results were summarized in Table 2.

Table 1: Sheet resistivity calculated from the I-V characteristic of TiO_2thin film samples.

Sample TiO_2	Voltage (V)	Current (A)	Gradient (V/ I)	Sheet resistivity (Ω/□)
TiO_2	26.4	6.408 e^{-10}	4.11 e^{10}	1.86 e^{11}
Al_2O_3	26.4	9.55 e^{-10}	2.76 e^{10}	1.25 e^{11}
Silicon Wafer	26.4	1.295 e^{-9}	2.05 e^{10}	9.27 e^{10}

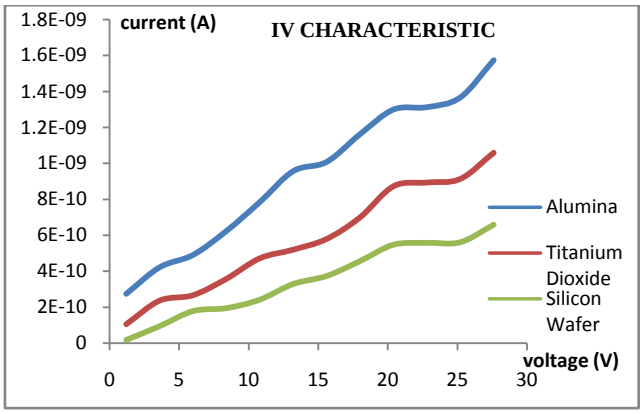


Figure 13: I-V Characteristic

Using four point probes, the I-V Characteristic of different material is showing in figure 1. From this graph, the wafer with no evaporation process is above and then followed by the evaporation of Al_2O_3 and $Ti0_2$.

This I-V characteristic graph show that 3 lines which represents the substances used to fabricated a thin film. The substances that had been used are such as Alumina, Titanium Dioxide and Silicon that does not contain any types of material. From the graph, show that Alumina have the high current flow rate compare to Titanium Dioxide Silicon. Refer to the graph, a table for the calculation of sheet resistivity and axial gradient graph has been build.

Table 2: Sheet resistivity at various time

Time (minutes)	**Resistance, R = (V/I)**	**Sheet resistivity, ρ = Ω/□**
0	$4.58x10^{10}$	$2.07x10^{11}$
1	$4.16x10^{10}$	$1.88x10^{11}$
5	$3.55x10^{10}$	$1.61x10^{11}$

From the table above, it is shown that when the time is increase, the resistance is decrease that means the sheet resistivity also decrease. When the sheet resistivity is decrease, the conductivity is increase.

Table 1show that the sheet resistivity of TiO_2 and Al_2O_3 thin film. The sheet resistivity of the TiO_2 and Al_2O_3 thin film evaporate for 15 minutes shows that the value is becoming closer to the sheet resistivity of the Si wafer before the evaporation of TiO_2 and Al_2O_3. This may be due the deposition of TiO_2 and Al_2O_3thin film during the evaporation process.

IV. CONCLUSION

After the experiment, we can conclude that the TiO_2 and Al_3O_2thin filmwhich was evaporate in the modified physical vapor deposition module system shows that there is a component of TiO_2 and Al_2O_3exist on the surface of the thin film. This was proven from the Optical Microscope, SEM, XRD, EDS andAFM results. The surface morphology of TiO_2 and Al_2O_3 thin film was changed when the material added and mixture with CaC_6 in the evaporation process. The thin film also shows that, when the mixture of TiO_2 nanopowder and mixture of Al_2O_3nanopowder added the thickness of the thin film layer also increased.

V. SUGGESTION

The research on the particular method can be expand for further analysis using several other advance equipment such as Atomic Force Microscope (AFM) and Field Emission Scanning Electron Microscope (FE – SEM) to understand the phenomena happened in atomic scale. Also, it is suggested to havefabricated moresamplesusing other material such as zinc oxide and etc and to be tested using a more efficient equipment to understand better the reaction of thin film towards evaporation effect.

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