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The Effect of Oxygen Gas Flow Rate on TiO2 Thin Film Prepared by Double Zone CVD Technique

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Abstract: The research was conducted to determine the deposited TiO_2 thin film of titanium dioxide on a 210º C and 60º C glass substratum using a double zone Chemical Vapor Deposition (CVD) furnace. Through a different oxygen gas, the flow rate was varied in range of 0.6 ml ~ 1.2 ml. The effect of oxygen gas flow rate thin films was routinely tested at room temperature on titanium dioxide TiO_2 and will be annealed at a fixed time, which is 1 hour at 500º C. The crystal structure properties on TiO_2 was obtained using X-Ray Diffraction (XRD). In addition, the studies of surface morphology was performed using Atomic Force Microscope (AFM) and Field Effect Scanning Electron Microscopy (FE-SEM). The TiO_2 electrical properties were evaluated to obtain its resistivity and conductivity using a four-point probe. The characterization of TiO_2 thin film conducted more systematically and discussed in these research studies.

Keywords: Titanium Dioxide (TiO_2),Titanium Butoxide (IV), Chemical Reaction on CVD

1. Introduction

In the field of nanotechnology, titanium dioxide (TiO_2) is widely utilized material in coating technologies and optics [1]. TiO_2 has been widely investigated as a promising sensing material in the various thin film gas sensor. As a result of their outstanding properties: chemical stability, sensing properties, and electrical properties. In addition, it also has magnificent properties such as low toxic, low cost, and excellent chemical stability. In term of deposition methods, CVD is the most effective to produce high quality and wide-area graphene for a wide variety of applications [2]. In this work, TiO_2 thin film will be deposited onto thin indium oxide by using a coated glass substrate. Then, the thin film will be investigated using several characterization tools such as XRD, AFM, FE-SEM, and Four-point probe to reveal its structural, morphology, topography, and optical properties. On top of that, this project to study the TiO_2 properties with different reactive values of the gas flow rate [3]. Unlike a previous study, which uses a different precursor volume on the characterization of TiO2, where the substrate temperature set to the specific value. In comparison with this study, which uses different reactive values

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of the double zone on gas flow rate, a CVD technique where alumina boat and substratum temperatures is fixed. A photocatalytic activity on the TiO_2 thin film was also examined [4].

2. Materials and Methods

2.1 Titanium dioxide TiO_2

Nanostructured TiO_2 thin film had a high transmittance in refractive index, with chemical stability, excellent durability, and particularly in the visible region. It is a white solid inorganic substance that is inflammable, thermally stable, weak insoluble, and non-hazardous [4]. Therefore, TiO_2 has been received much attention in industry and academia in different fields for example, photocatalysis, solar energy, cosmetics, pharmaceuticals, and food coloring [5].

2.2 Application on TiO_2 thin film

Research of TiO_2 mostly focuses on the industries application in pigment and ceramic since it is effective in acting as a semiconductor photocatalyst. During deposition on the application film, the refractive index and colour become excellent for a dielectric mirror [6]. The most stable polymorph in condition temperatures might present the lowest free energy than either metastable anatase or brookite phases. Meanwhile, rutile consumes a tetragonal unit cell, with unit cell parameters: a = b = 4.584 å and c = 2.953 å. A titanium cations have a coordination number of six where is enclosed by an octahedron of six oxygen atoms, a brookite orthorhombic optional of TiO_2 [7].

The structure of TiO_2 is normally either anatase or rutile. These forms show a photocatalytic activity and has larger cell volume than others. Brookite strutures have eight groups per unit cell compared to four for anatase and two for rutile [8]. The brookite structure is composed of distorted octahedral by titanium ion at of the center and each six vertices oxygen where is octahedron will share three edges with attached than form an orthorhombic a structure [9-10].

2.3 Chemical vapour deposition (CVD)

The procedure in this research study can be flowing gasses of precursor in the furnace tube called as CVD. In this method, samples are deposited with specific material and temperature then annealed. There are hot surfaces on substrate and the thin film deposited on the substrate surface. The depositions of thin film on the surface during hot surfaces during chemical reactions.

2.4 CVD reaction on material

In this study deposition utilizing with titanium (IV) butoxide, a commercial material that react during the CVD process. The material is a colorless neutral liquid, although elderly samples are yellowish with a weak of alcohol- like odor and solvable in various organic solvents. In the investigative paper by An et. al. study the process of CVD and PVD on Tie6242S and Ti-555 titanium alloys, revealed evaluation performance of both processes. Study by Kuram et. al. is using PVD for materials TiCN, AlTiN, TiAlN, TiCN+TiN and AlTiN+TiN [11].

2.5 Characterisation of TiO2 thin film

There are various characterization techniques performed using XRD, AFM, FE-SEM, and Four-point probe. Therefore a set of measurement and material tool was identified, the research design, application of equipment, substrate preparation, measurement and characterization technique were discussed thoroughly. Hence, during a CVD preparation, the inside oxygen gas flow rate was an important aspect to be considered. Figure 3.1 illustrates the flowchart of the research process.

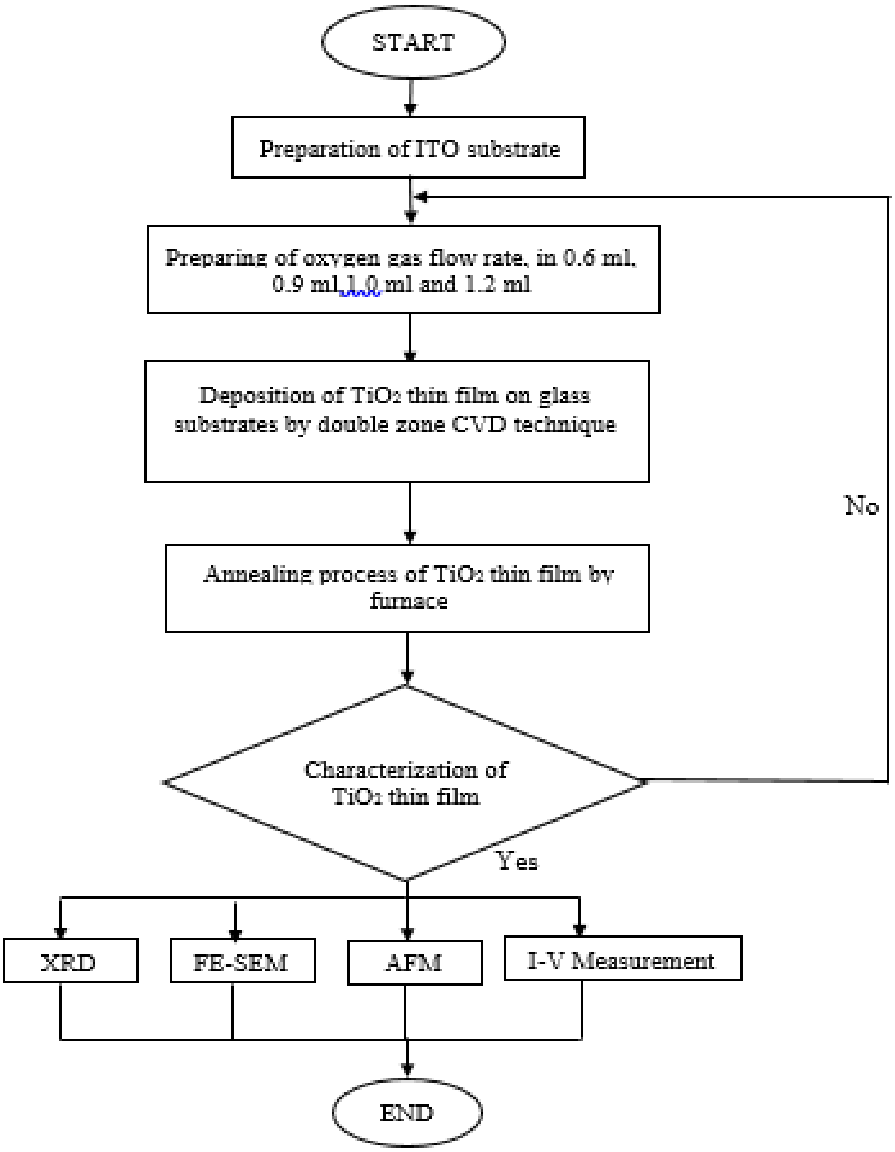

Figure 1: Process flow of research methodology

3. Results and Discussion

3.1 Results

Based on the results, the presence of titanium dioxide after using different oxygen gas flow rate of0.6 ml, 0.9 ml, 1.0 ml and 1.2 ml. Through different gas flow rate, the intensity varies based on the inclusion of gas flow value on this process. The XRD pattern of prepared TiO_2 thin film at different oxygen gas flow levels is shown in Figure 2 and the analysis is summarized in Table 1. The diffraction peaks were analyzed from the plotted graph according to the (101), (004), (200), (105), (211), and (204), and a crystal plane accomplished TiO_2 anatase. Results show that the highest peak of anatase is in 0.6 ml sample via the highest diffraction angle were assigned in (101) crystal plane. A complete width at half maximum (FWHM) is the width of a measured spectrum curve between specific points on the y-axis, half the full amplitude.

The smaller FWHM reveals TiO_2 thin film has higher crystallinity because smaller FWHM will create sharper peaks. Therefore, a study of 0.6 ml has shown the highest crystallinity when the oxygen gas flow rate would increase the diffraction angle at (101) and relative strength. Therefore it was noted that the thin-film crystal orientation changed under different range oxygen gas flow rate in condition of 0.6 ml.

Table 1: The crystalline size with different range gas flow rate (ml)

The oxygen gas flow rate (ml)	FWHM (2θ)	Diffraction angle at (101) peak (degree)	Relative intensity at (101) (%)
0.6	0.3149	25.356	45.8
0.9	0.3149	25.331	25.4
1.0	0.2362	25.214	11.18
1.2	0.3247	25.325	23.4

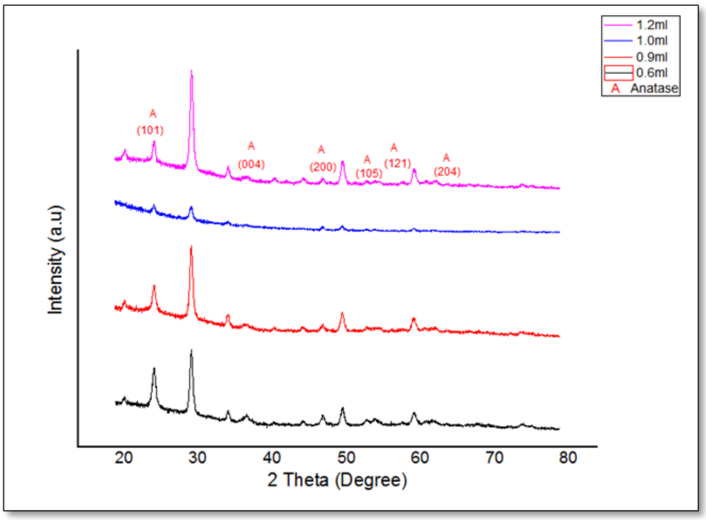

Figure 2: Pattern of XRD TiO_2 on 0.6 ml, 0.9 ml, 1.0 ml and 1.2 ml gas flow rate

3.2 Topography analysis on AFM result

Result shown in Table 4.2 shows the grain size (nm) and average roughness, Ra (nm2). By increasing gas flow rate from 0.6 ml to 1.0 ml, the average roughness is increased from the sample. In short, 1.0 ml sample had the highest average roughness that indicate the lowest uniformity. However, the sample with 0.6 ml gas flow rate has the lowest roughness, thus has the highest uniformity.

Generally a high roughness has benefit in solar cell because it can improve the efficiency by capturing more light [20]. Thus, in a high gas flow rate, the surface on TiO_2 thin film becomes more rough due to existence of large grain size. Moreover, the movement electron can be improved by one grain to another. This can be seen since grain size increases when the gas flow rate is increasing. The different gas flow rate of 1.0 ml with higher roughness that is suitable for solar cell application.

3.3 Topography analysis on AFM result

The morphology TiO_2 thin film was obtained through an FESEM analysis. Based on the result of FESEM, when oxygen gas flow rate increases from 0.6 ml to 1.2 ml, nanoparticles grain size and grain area of TiO_2 was increased. The 0.6 ml sample has the smallest grain size, and the grain size starting increase in 0.9 ml sample. Increasing in grain size was due to agglomeration, since more solvent particles are formed.

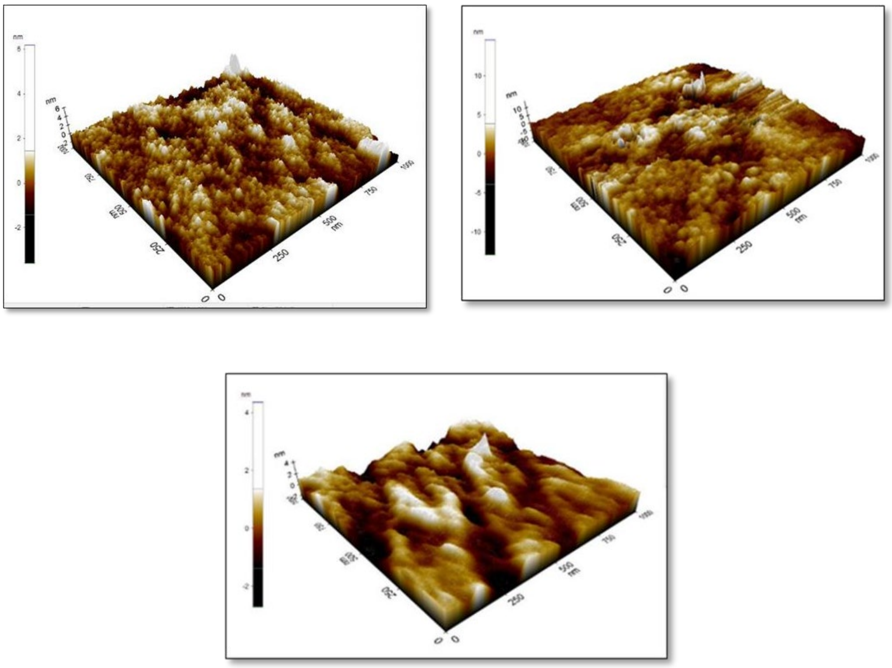

Figure 3: The AFM images of TiO2 thin films for various oxygen gas flow rate

Table 2: The crystalline size with different range gas flow rate (ml)

The oxygen gas flow rate (ml)	Grain size (nm)	Grain size (nm^2)	Average roughness, Ra (nm)
0.6	0.055	0.023	0.399
0.9	0.034	0.004	0.541
1.0	1.411	0.996	1.268

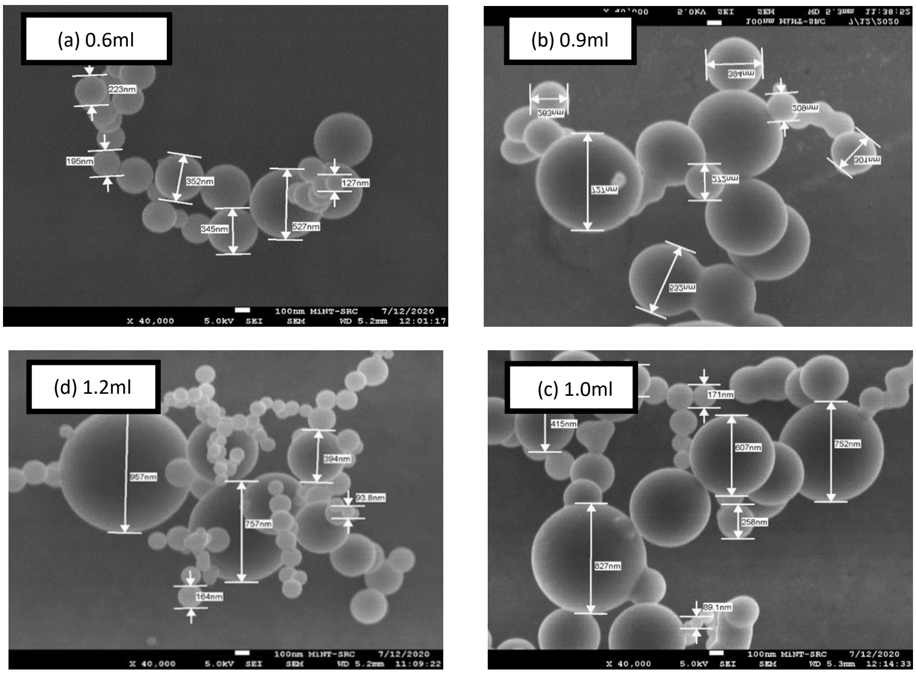

Figure 4: Nanoparticle grain size with different gas flow rate at (a) 0.6 ml (b) .0.9 ml (c) 1.0 ml (d) 1.2 ml on FE-SEM image

Table 3: The result of grain size and grain area nanoparticle of TiO_2 thin film

Gas flow rate (ml)	Grain size (nm)	Grain area (nm^2)
0.6	127 - 527	$1.970 \times 10^4 – 4.629 \times 10^5$
0.9	263 - 727	$3.047 \times 10^4 – 5.120 \times 10^5$
1.0	171 – 827	$4.047 \times 10^4 – 7.216 \times 10^5$
1.2	164 - 957	$5.78^5 \times 10^5 – 8.659 \times 10^5$

3.4 Electrical analysis on four point probe result

The conductivity and resistivity of TiO_2 thin film can be obtained using a four-point probe. Table 4 shows that the average resistivity varied a lot, the 0.6 ml sample had the lowest average resistivity or the highest conductivity. The 0.9 ml sample has the highest resistivity or the lowest conductivity. Besides, in 1.0 ml sample has the lowest resistivity or the highest conductivity. However, when the gas flow rate in 1.2 ml, the result shows that it has the lowest resistivity or the highest conductivity. A high conductivity indicated that the thin film has good crystalline structure as electron tend to move from one grain to another easily. Larger grain size also lead to high conductivity and low resistivity, which are important for solar cell applications.

Table 4: The average sheet resistance, resistivity, and conductivity of TiO_2 thin film

Rate oxygen gas flow (ml)	Thickness (μm)	Average sheet resistance, Rs (Ω/sq)	Average resistivity, ρ (Ω cm)	Average conductivity,σ (S/cm)
0.6	0.674	175.8029	0.0105	95.238
0.9	0.720	156.5341	1.5653	0.6388
1.0	0.824	215.0640	0.0215	46.511
1.2	0.909	781.6620	0.0781	12.8040

4. Conclusion

In summary, this work successfully synthesized on the effect of various oxygen gas flow rate on TiO_2 thin film on ITO substrate using a technique double zone CVD technique. In this work, the parameter of annealing time 500°C in 1 hour and the concentration on a precursor substrate on titanium (IV) butoxide 1.5 ml is fixed. Results show that changes in oxygen gas flow rate affect the structural, topographical morphological, and electrical properties of TiO_2 thin films. It is found that the resistivity increased as the thickness increased, and thus the conductivity decreased. The effect of resistivity and conductivity can be observed from the various range sample on oxygen gas flow rate. Furthermore, the effect of different oxygen gas flow rate on the properties of the thin film TiO_2 was studied successfully. In it concluded that increasing oxygen gas flow rate significantly affected the structural, topography, morphology and electrical properties of TiO_2 thin film prepared using the CVD technique.

References

[1] P. Piszczek, Ż. Muchewicz, A. Radtke, M. Gryglas, H. Dahm, and H. Różycki, "CVD of TiO_2 and TiO_2/Ag antimicrobial layers: Deposition from the hexanuclear μ-oxo Ti(IV) complex as a precursor, and characterization tool," Surface and Coatings Technology, vol. 222, pp. 38–43, 2013

[2] A. K. Singh, V. Chaudhary, A. K. Singh, and S. Sinha, "Effect of TiO_2 nanoparticles on electrical properties of chemical vapor deposition grown single layer graphene," Synthetic Metals, vol. 256, pp. 116–155, 2018

[3] N. Rausch and E. P. Burte, "ChemInform Abstract: Thin TiO2 Films Prepared by Low Pressure Chemical Vapor Deposition" ChemInform, vol. 24, no. 14, 2010

[4] A. Patil, D. N. Suryawanshi, I. G. Pathan, and D. G. Patil, "Effect of variation of precursor concentration on structural, microstructural, optical and gas sensing properties of nanocrystalline TiO_2 thin films prepared by spray pyrolysis techniques," Bull. Mater. Sci., ,vol. 36, no. 7, pp. 1153–1160, 2013

[5] Z. Zarhri, M. Á. A. Cardos, Y. Ziat, M. Hammi, O. E. Rhazouani, J. C. C. Argüello, and D. A. Avellaneda, "Synthesis, structural and crystal size effect on the optical properties of sprayed TiO2 thin films: Experiment and DFT TB-mbj," Journal of Alloys and Compounds, pp. 153–156, 2019

[6] K. Nakata and A. Fujishima, "TiO2 photocatalysis: Design and applications," J. Photochem. Photobiol. C Photochem. Rev., vol. 13, no. 3, pp. 169–189, 2012

[7] S. Mokhtar, M. Ahmad, C. Soon, N. Nafarizal, A. Faridah, A. Suriani, M. Mamat, M. Shimomura, and K. Murakami, "Fabrication and characterization of rutile-phased titanium dioxide (TiO2) nanorods array with various reaction times using one step hydrothermal method," Optik, vol. 154, pp. 510–515, 2018

[8] Janzeer, Yasmeen, "Experimental Surface Modification of Titanium and titanium alloys to enhance bone healing", pp.10–13, 2013

[9] N. S. Allen, N. Mahdjoub, V. Vishnyakov, P. J. Kelly, and R. J. Kriek, "The effect of crystalline phase (anatase, brookite and rutile) and size on the photocatalytic activity of calcined polymorphic titanium dioxide (TiO2)", Polymer Degradation and Stability, vol. 150, pp. 31–36, 2018

[10] M. Gardon and J. M. Guilemany, "Milestones in Functional Titanium Dioxide Thermal Spray Coatings: A Review", Journal of Thermal Spray Technology, ,vol. 23, no. 4, pp. 577–595, Aug 2014

[11] M. K. Singh and M. S. Mehata, "Phase-dependent optical and photocatalytic performance of synthesized titanium dioxide (TiO2) nanoparticles", Optik, , vol. 193, pp. 163–167, 2019