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Synthesis and characterization of TiO₂ doped SnO₂ thin film prepared by sol-gel method

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Abstract. In this work, preparation of titanium dioxide doped with tin oxide, SnO₂/TiO₂ thin films deposited onto silicon wafer via sol-gel method. Different amount of SnO₂ was added (5 ml, 10ml and 15 ml) into parent solution. The obtained films were annealed at different temperature which is 400°C, 500°C and 600°C for 1 hour. Morphological and surface topography of the SnO₂ doped TiO₂ thin films were studied using Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM). The annealed films shows non-uniform crack due to the mismatch of coefficient of thermal expansion (CTE) between SnO₂/TiO₂ thin films and silicon wafer.

1 Introduction

Nowadays, there are numerous application of titanium dioxide (TiO₂) that benefits the society such as cosmetics, foods, restraining and sterilizing virus, electrical devices and photocatalytic oxidation (PCO) under ultraviolet (UV) and visible light also known as a pollution control technology [1, 2]. Self-cleaning activity will occur when TiO₂ is being exposed under light source which will display a photocatalytically induced superhydrophilicity properties. The hydrophobic surface of the substrate will transform to hydrophilic surface after coated with TiO₂ where it will cause a uniform water film. This will prevent the adhesion of organic or inorganic components on the surface. Therefore, the cleanliness can be retained. TiO₂ thin film can be coated on the surfaces of various substrates such as metals, textiles, ceramics, fibres and glass that will exhibits self-sterilisation and self-cleaning properties when it is exposed to the light source.

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When TiO₂ is doped with metal oxide such as SnO₂, it will improve the efficiency of TiO₂ photocatalyst. By doping SnO₂ into TiO₂ thin films, hydrophilicity and photocatalytic activity of composite thin films would be improved due to the reduction of TiO₂ particle growth rate [3-6].

In this work, TiO₂ doped with SnO₂ thin films coated on silicon substrate were fabricated using a sol-gel dip coating technique. The thin films were annealed at 400°C, 500°C and 600 °C for 1 hour with the heating rate of 5 °C/min. X-ray Diffractometer (XRD) was used to study the phase composition of SnO₂ doped TiO₂ thin films while Scanning Electron Microscope (SEM) and Atomic force microscope (AFM) was used to observe the morphology and the surface topography of SnO₂ doped TiO₂ thin films, respectively.

2 Methodology

2.1 Materials

The main raw materials used to produce TiO₂ sol in these studies are titanium (IV) isopropoxide (TTIP), Ti(OC₃H₇)₄, isopropanol, C₃H₈O and acetic acid, CH₃COOH. Tin-tetrachloride penta-hydrate (SnCl₄.5H₂O) powder was used as the source of SnO₂ solution. The silicon wafer was used as a substrate.

2.2 Methods

Firstly, 0.5 ml titanium (IV) isopropoxide (TTIP) and 10 ml isopropanol, C₃H₈O were stirred at room temperature for 20 minutes using magnetic stirrer. Then, 2.5ml of acetic acid was added into the solution while stirring to speed up the chemical reaction. SnO₂ solution was produced by adding 50ml of distilled water and 50 ml of ethylene glycol into 3.51g tin-tetrachloride penta-hydrate (SnCl₄.5H₂O) powder. The solution was dissolved for 10 minutes to make a 100 ml precursor mixture. The TiO₂ solution was mixed together with 5ml, 10ml and 15ml of SnO₂ respectively. The mixture was vigorously stirred for another 30 minutes at room temperature. The substrate used is silicon wafer (1 cm x 1 cm). The silicon wafers were washed in acetone using ultrasonic cleaner for 30 minutes. The TiO₂/SnO₂ sol were deposited on silicon wafer by using spin coating at 1500rpm for 30s. The coated samples were dried at 40°C for 10 minutes. After that, the coated samples were annealed in muffle furnace for 1 hour with heating rate of 5°C/min at 400°C, 500°C and 600°C. The TiO₂ doped with SnO₂ thin films were prepared by sol-gel method according to the amount of SnO₂ doping. In this study, the characterization of TiO₂ thin films was investigated by using Scanning Electron Microscope (SEM) and Atomic Force Microscopes (AFM).

3 Results and Discussions

Fig. 1 shows the XRD pattern of SnO₂ doped TiO₂ thin films. It can be seen that the diffraction pattern shows dual-phase of anatase and rutile with no existence of SnO₂. The samples were annealed at three different temperatures which are, 400°C, 500°C and 600°C, respectively. The angle 2θ shifts towards lower angles with the increase of annealing temperature from 400 °C to 600 °C.

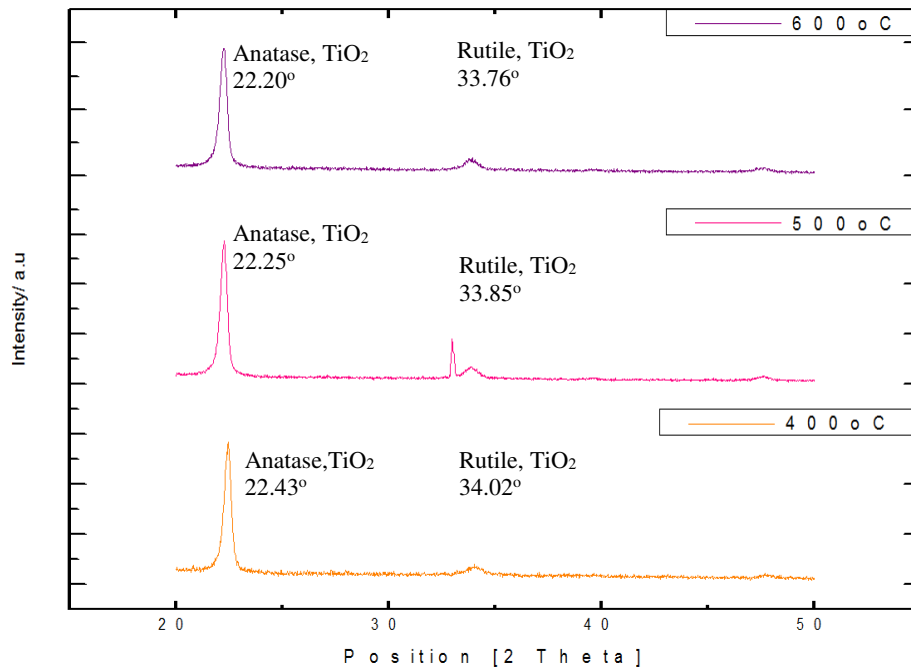


Fig. 1. XRD pattern of SnO₂ doped TiO₂ thin films which was annealed at 400°C, 500°C and 600°C.

Fig. 2 and Fig. 3 show the micrographs of SnO₂/TiO₂ thin films that were coated on silicon wafer with different amount of SnO₂ added and annealed in muffle furnace at 400°C, 500°C and 600°C for 1 hour. The micrographs show a non-uniform films with large flaky and large cracks film. It might due to the surface tension between the film and the air during the drying process [7]. It can be seen that as the annealing temperature increases, the distance between cracks become smaller. The higher the annealing temperature, the least cracks formed. To prevent crack from forming when annealed at high temperature, more layers of coating were deposited to ensure the coverage of substrate become better and formed a thicker film. It is well agree with the previous work by Abdul Razak et al [8]. From Fig. 2, it was observed that 5 ml SnO₂ doped with TiO₂ annealed at 400°C has the most agglomerated region as compared to the samples annealed at 500°C and 600°C. Meanwhile, in Fig. 3 shows the micrograph of 10 ml SnO₂ doped with TiO₂ annealed at 500°C has the most agglomerated region as compared to the samples annealed at 400°C and 600°C. The micrographs show a Volmer-Weber growth. Volmer-Weber growth of SnO₂ doped TiO₂ thin films includes nucleation of 3-D islands, growth, impingement and coalescence of islands [4].

The surface topography of SnO₂/TiO₂ thin films was investigated by Atomic Force Microscopes (AFM). Typical topographical images of the surface of SnO₂/TiO₂ coatings are represented in 3D images. Fig. 4 shows the surface topography of 5ml SnO₂ doped TiO₂ thin films at different annealing temperature of 400°C, 500°C and 600°C respectively. Fig. 5 shows the surface topography of 10ml SnO₂ doped TiO₂ thin films at different annealing temperature 400°C, 500°C and 600°C respectively.

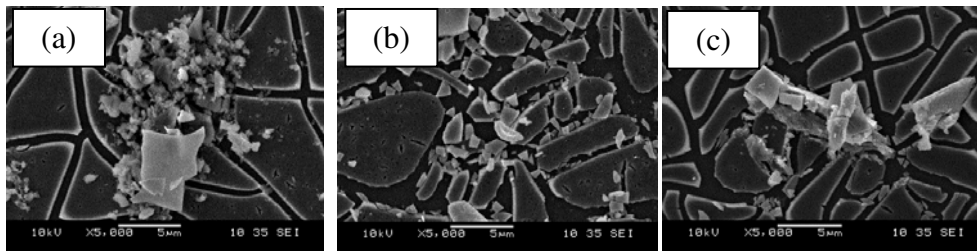


Fig. 2. SEM image of the surface morphology for 5 ml SnO₂ doped with TiO₂ thin films at temperature of (a) 400°C, (b) 500°C and (c) 600°C.

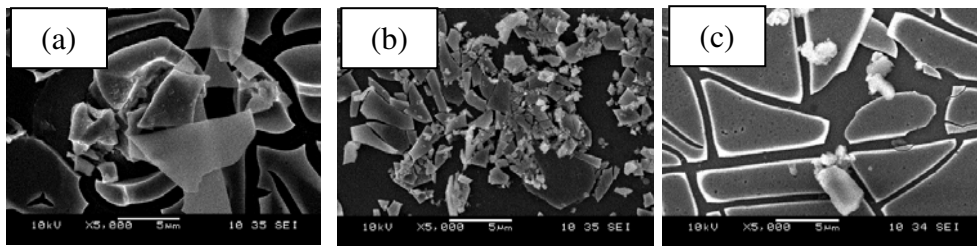
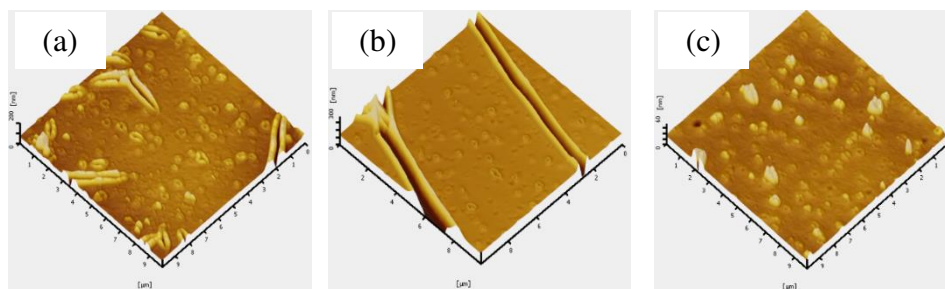


Fig. 3. SEM image of the surface morphology for 10 ml SnO₂ doped with TiO₂ thin film at temperature of (a) 400°C, (b) 500°C and (c) 600°C.

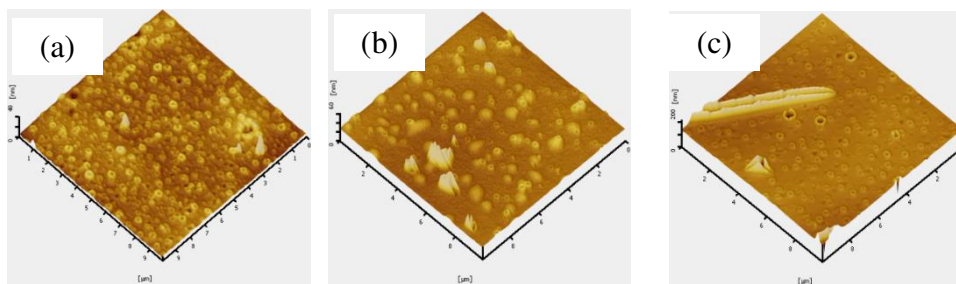


RMS=1.155nm

RMS=3.677nm

RMS=3.796nm

Fig. 4. The surface topography and 3D images of 5ml SnO₂ doped TiO₂ thin films at different annealing temperature of (a) 400°C, (b) 500°C and (c) 600°C.



RMS=1.570nm

RMS=2.995nm

RMS=9.937nm

Fig. 5. The surface topography and 3D images of 10 ml SnO₂ doped TiO₂ thin films at different annealing temperature of (a) 400°C, (b) 500°C and (c) 600°C.

It can be observed that the sample which was annealed at 600°C produce the highest value of roughness (RMS) while the sample annealed at 400°C produced the lowest value of roughness (RMS). This showed that the surface roughness (RMS) of the samples

increased with the annealing temperature. It is well agreed that the presences of SnO₂ influenced the RMS value. As the amount of SnO₂ added increased, the RMS value also increased. This revealed that the RMS value was affected by annealing temperature and the amount of SnO₂ added into the parent solution.

4 Conclusion

In this study, SnO₂ doped TiO₂ thin films were prepared by sol-gel method. The XRD pattern shows the phase composition of SnO₂ doped TiO₂ thin films were dual-phase of anatase and rutile with no existence of SnO₂. The morphological characteristics of SnO₂ doped TiO₂ thin films have non-uniform cracks, agglomerate and the formation of Volmer-Weber growth. When the annealing temperature and the amount of SnO₂ increased, the surface roughness (RMS) value increased.

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