Synthesis of Tungsten Oxide (WO3) Film on Glass Substrate using Aqueous Based Solution Spray Deposition Method

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Synthesis of Tungsten Oxide (WO_3) Film on Glass Substrate using Aqueous Based Solution Spray Deposition Method

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Abstract. A Tungsten oxide (WO_3) film on top of a glass substrate had been successfully constructed. The immobilization of tungsten oxide was performed using spray-deposition method. The precursor volume was varied as 5 mL, 10 mL and 25 mL. Characterization using x-ray diffraction (XRD) and scanning electron microscopy (SEM) yield that precursor volume influence the quality of the film. It was found that the good uniform film was obtained using precursor volume 5 mL and 10 mL. The crack which was resulted from non-uniformity of the film appear when the precursor film was 25 mL.

Introduction

Tungsten oxide (WO_3) is an n-type metal-oxide semiconductor with energy gap of about 2.7 – 2.8 eV [1,2]. Hence, this type of semiconductor is very sensitive to the visible ligh irradiation. The intrinsic conductivity originates from its non-stoichiometric composition cause to a donor level formed by oxygen vacancy defect in the lattice. The tungsten compound can exist in many forms because that the tungsten has many oxidation states, i.e., 2, 3, 4, 5 and 6. The typical forms of tungsten oxides are tungsten (VI) oxide (WO_3, lemon yellow appearance) and tungsten (IV) oxide (WO_2, brown and blue appearance) [3]. Due to its distinctive photocatalytic, photochromic and electrochromic properties, WO_3 is widely used in environmentally friendly and renewable energy applications, such as solar-powered water splitting, smart windows, gas-sensing, virus deactivation and organic pollutan or dye photodegradation [4 – 6]. Additionally, since WO_3 shows better photoabsorption in visible-light irradiation, this makes WO_3 can be activated either indoor using domestic visible light source or outdoor condition under the irradiation of sunlight.

The powder method is usually used in pollutant degradation [6]. In this method, the powder of WO_3 is directly distributed into the pollutant. However, this step leads to the contamination of water by the powder of WO_3 which needs a further separation process [6]. This side effect can be minimized by the immobilization WO_3 powder on top of a substrate. The WO_3 molecules are tied up with a substrate which prevents the WO_3 powder dispersed into the water. In this present study, we report the properties of WO_3 film which is resulted from the immobilization of WO_3 on top of a glass substrate by using spray-deposition method.

Experimental

Immobilisation of Tungsten Oxide on top of the glass substrate

The 3.081 grams of Ammonium (para) tungstate hydrate (Sigma Aldrich, Singapore) were dissolved in the 100 mL aquadest. Those solution were heated and stirred on the hot plate stirrer at 40°C for around 20 minutes. The process resulted in an aqueous based transparent solution of 0.01 M. Then, WO_3 was immobilised by growing a film from the raw materials on a glass substrate. The process to obtain WO_3 film was started by putting a glass substrate on top of a temperature

controllable hot plate at 150°C. Then, the prepared precursor in a spray equipment was sprayed with the dimension 2.5 cm x 5 cm directly on top of a glass substrate, as it is illustrated in Fig. 1. In the next step, the formed film was cooled down to the room temperature. The last step was annealing process, where the coated glass was heated in the furnace at 600°C for two hours. The whole process was performed with the volume of the precursor was varied as 5 mL, 10 mL and 25 mL.

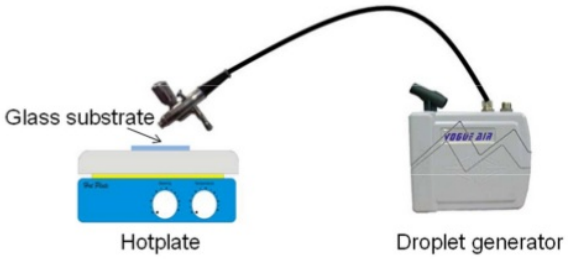

Figure 1. The spray deposition equipment

Film Characterization

The WO_3 film was characterized using a scanning electron microscope (SEM, JEOL, Tokyo, Japan) and an X-ray diffractometer (XRD, Phillips, Expert pro, USA). The XRD measurement was performed using radiation from the target of Cu Kα (λ=0.154 nm) with the voltage and the current were set at 40 kV and 30 mA.

Result and Discussion

The WO_3 films with the volume of the precursor were varied as 5 mL, 10 mL and 25 mL are illustrated in Fig. 2. It is clearly seen that the volume of the precursor influence transparency of the films. Transparency of the films decrease as the volume of the precursor is increased. The increase of the precursor's volume result in the increase of the thickness and the density of the film which then reduce the possibility of light to pass through the film.

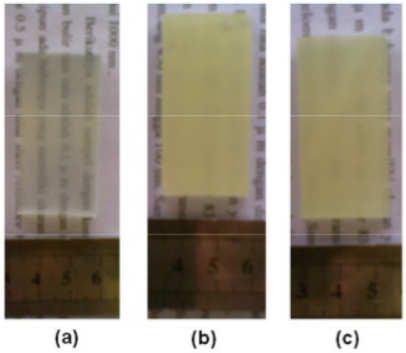

Figure 2. WO_3 film synthesized at different volume of precursor, (a) 5, (b) 10 and (c) 25 mL

Crystal structure of WO_3 film.

After the films were formed, it was characterized using X-ray diffraction (XRD) to determine the structure of the WO_3 crystal. The pattern of the diffraction of immobilised WO_3 is presented in Fig. 3. The grown film wass identified using JCPDS No.43-1035 and No.36-0101. The patterns show that the less precursor volume result in the increase of the quality of the film which is confirmed by the increase in the intensity of the dominant peaks. In our argument, 5mL precursor is adequate to cover glass perfectly by the WO_3. However, the increase in precursor volume (for

example into 25 mL) tend to make the film become inhomogen which result in the appearance of the crack. This crack cause the film become porous and decrease the film density that decrease the diffraction intensity at the preferential orientation. It was confirmed by the result of the film morphology which is illustrated in Fig. 4. It will be discussed later in the next sub section.

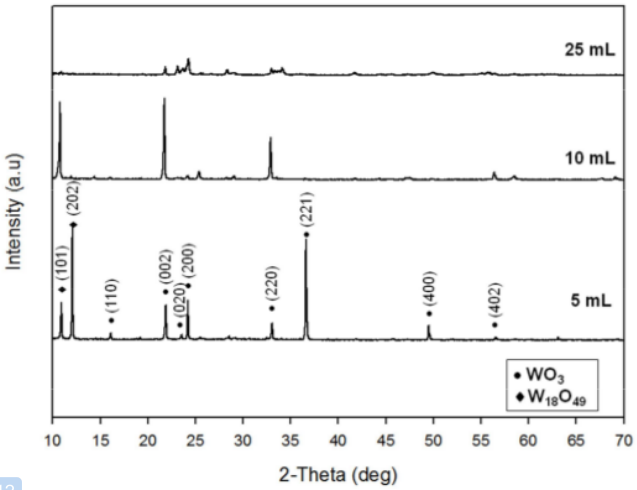

Figure 3. XRD patterns of WO_3 film synthesized at different volume of precursor.

In Fig. 3, we can also identify the peaks which are formed from the other phase of WO_3. This phase is $W_{18}O_{49}$ which can be identified using JCPDS No.36-0101 in the crystal orientation of (101) and (202) which related to the diffraction angle of $2\theta = 10.93^{o}$ and 12.07^{o}, respectively. The other phase is probably contributed from the annealing process at 600°C for two hours where the crystal did not achieve pure WO_3 yet.

The data of diffraction peak and FWHM (full width at half maximum) from the XRD characterization could also be used to calculate crystallite size by using Scherrer equation (Eq. 1). Using the dominant diffraction peak ($2\theta = 21.8°$) for each variation of precursor volume (5 mL, 10 mL, 25 mL). The result is presented in table 1. The increase of precursor volume tends to increase the crystallite size.

$$\tau = \frac{K\lambda}{B\cos\theta} \quad (1)$$

Where, τ is the mean size of the ordered (crystalline) domains. K is a dimensionless shape factor. B is FWHM. θ is Bragg/diffraction angle.

Table 1. The crystallite size of WO_3 calculated using Scherrer equation.

Volume of precursor (mL)	2θ (°)	θ (°)	FWHM (°)	FWHM (rad)	Crystallite size (nm)
5	21.8924	10.9462	0.1181	0.0020601	69.9017
10	21.7461	10.8731	0.1181	0.0020601	69.0308
25	21.8441	10.9221	0.0984	0.0017165	82.4784

Morphology of the WO_3 film

The study of the surface morphology of the films was performed by using SEM. The SEM characterisation was studied using 500 times and 5000 times magnification. The images of the surface morphology are illustrated in Fig. 4 both for low and high magnifications. It can be easily

seen that the profile of the surface are relatively flat and homogeneous for the low precursor's volume (5 mL and 10 mL). However, for the precursor volume of 25 mL, the profile is inhomogeneous which shows the porous structure in the layer surface. This symptom is showing the sign of crack. This crack is visible in insert of Fig. 4 which is obtain using 5000 magnification. This images are showing the trend that the increase of precursor volume tend to rise the possibility of the crack.

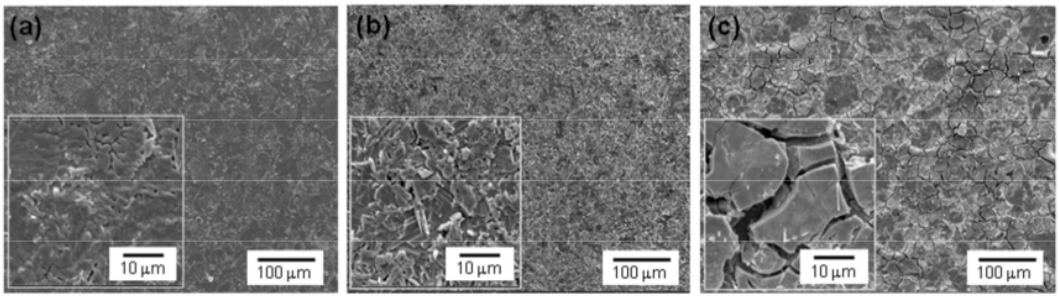

Figure 4. SEM images of WO_3 film at different volume of precursor, (a) 5, (b) 10 and (c) 25 mL.

Conclusion

Using aqueous based spray-deposition method with the annealing temperature 600°C, the WO_3 film had been successfully constructed. The precursor volume influence the quality of the film. The characterization shows that the film with the good uniformity was obtained with the precursor volume not more than 10 mL. When the precursor volume is 25 mL, the film become non-uniform and the crack was formed. This mentioned results may find potential application in photocatalyst.

Acknowledgments

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