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Electrochromic Properties of Sol-Gel Deposited Electrochromic TiO_2 Thin Films

Abdelrahman Hamed Ebrahem Abdelhamed[1], Kah-Yoong Chan[1,*], Benedict Wen-Cheun Au[1,2], Gregory Soon How Thien[1], Pei-Ling Low[1], Yew-Keong Sin[1], Chu-Liang Lee[1], Wai-Leong Pang[1]

[1] Centre for Advanced Devices and Systems, Faculty of Engineering, Multimedia University, Persiaran Multimedia, 63100 Cyberjaya, Selangor, Malaysia
[2] Sri Desa International Secondary School, Taman Desa, Kuala Lumpur 58100, Malaysia

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ABSTRACT

Electrochromic (EC) smart windows are a type of glass window that can change from transparent to darker colour shades when a small voltage is applied. Titanium dioxide (TiO_2) can be utilised as an EC material for EC smart windows. Although the TiO_2 sol-gel spin-coating method is commonly used, the effect of the number of TiO_2 layers was not reported. Thus, this paper investigates the effect of the number of TiO_2 layers. The increasing number of TiO_2 film layers demonstrated a noticeable increase in the thin films 'anodic and cathodic diffusion coefficient, particularly with 11 TiO_2 layers. Additionally, the colouration and bleaching time was revealed to have a low correlation as the number of layers increased. Nonetheless, the lower number of TiO_2 layers resulted in lower colouring transmittance. Comparatively, the colouration efficiency for all films did not exhibit any significant change. Hence this study on the effect of the TiO_2 layering technique can open a new pathway in understanding the EC properties of TiO_2-based EC devices.

1. Introduction

Electrochromism is a reversible phenomenon in which a material known as an electrochromic (EC) material in an electrolyte solution changes its colour when an electrical voltage is applied across the layer [1]. Thus, the applied bias voltage causes the simultaneous electron and cation (Li^+, H^+, and Na^+) injections to change the colour of the EC materials. Furthermore, this phenomenon is a reversible process in which the colour can be alternately changed depending on the polarity of the applied voltage [2]. Currently, EC materials are utilised in several applications, particularly for manufacturing EC smart windows. Based on the ability of electrochromic (EC) smart windows to convert between transparent and opaque states, they have garnered much interest in their possible use in sunroofs [3], displays [4], vehicle rear-view mirrors [5], and aircraft[6]. EC smart windows can

* *Corresponding author.*
E-mail address: kychan@mmu.edu.my

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also dramatically lower energy use by controlling the amount of solar heat and light entering buildings compared to traditional double-glazed windows [7,8].

Titanium dioxide (TiO_2) is a potential candidate for EC applications among EC materials [9–11]. When a TiO_2-based EC device is applied to a negative potential, the TiO_2thin films synthesised using certain methods can change colour from transparent to blue [12]. Hence, transparent TiO_2 thin films have been fabricated until now using multiple arrays of methods such as pulsed laser deposition [13], magnetron sputtering [14], spray pyrolysis [12,15], and sol-gel route [12,16,17] Furthermore, the fabrication of TiO_2 via the sol-gel route is a promising method due to its simplicity of fabrication and cost-effectiveness compared to other methods [18].

This paper investigated the TiO_2 film thickness, optical, and electrochromic properties with the varying number of deposited TiO_2 layers. To the best of the authors' knowledge, the investigations in the effect of the number of deposited TiO_2 layers for Properties via the sol-gel spin-coating method were poorly understood. Most available studies are for TiO_2 fabrication using different methods or a single TiO_2 layer-based EC device using the sol-gel spin-coating method. Therefore, TiO_2 thin films were fabricated via the sol-gel spin-coating method on indium tin oxide (ITO) coated glass. Different layers were deposited on ITO substrates to obtain TiO_2 thin film samples. Resultantly, thin film thickness, optical, and electrochromic characterisation were conducted on the samples to observe their correlation with the increasing number of layers. Based on the output of this study, this paper demonstrated insights into the effect of the number of TiO_2 thin films and their corresponding EC characteristics.

2. Methodology

2.1 Sol-Gel Preparation

The TiO_2 thin film sol-gel was prepared using titanium isopropoxide (TTIP) as a precursor, diethanolamine (DEA) as a stabiliser and ethanol as a solvent, with a ratio of 3:1:20, respectively. Subsequently, all the chemicals were mixed into a solution and aged for a few days.

2.2 Thin Film Fabrication

ITO-coated glasses were used as the substrates for TiO_2 thin film fabrication. Before spin coating, the ITO substrates were cleaned in an ultrasonic bath using acetone, isopropyl alcohol, and deionised water (DI-Water) at 60℃ for 10 mins. The ITO substrates were then dried with a nitrogen spray gun to remove the excess water and were further dried in the oven at 60℃ for 30 mins. Subsequently, the TiO_2 sol-gel solution was deposited on the cleaned ITO substrates by spin-coating at 3000 rpm for 30 s. The ITO substrates were heated on a hotplate at 100℃ for 3 mins. The sol-gel deposition, spin coating, and hotplate heating cycle were repeated on different substrates to get samples with varying layers (1, 3, 5, 7, 9, and 11 layers) (see Table 1). Finally, the TiO_2 substrates were post-annealed at 400℃ in a furnace for 5 mins. In this study, the samples were defined based on the number of layers deposited on the ITO substrates, which were T1, T3, T5, T7, T9, and T11. A simple flow process for the fabrication is illustrated in Figure 1.

Table 1
Summary of the sample name used in this study, which corresponds to the number of TiO_2 layers deposited on the substrates

Sample Name	Definition
T1	One round of TiO_2 sol-gel deposition (1 Layer)
T3	Three rounds of TiO_2 sol-gel deposition (3 Layers)
T5	Five rounds of TiO_2 sol-gel deposition (5 Layers)
T7	Seven rounds of TiO_2 sol-gel deposition (7 Layers)
T9	Nine rounds of TiO_2 sol-gel deposition (9 Layers)
T11	Eleven rounds of TiO_2 sol-gel deposition (11 Layers)

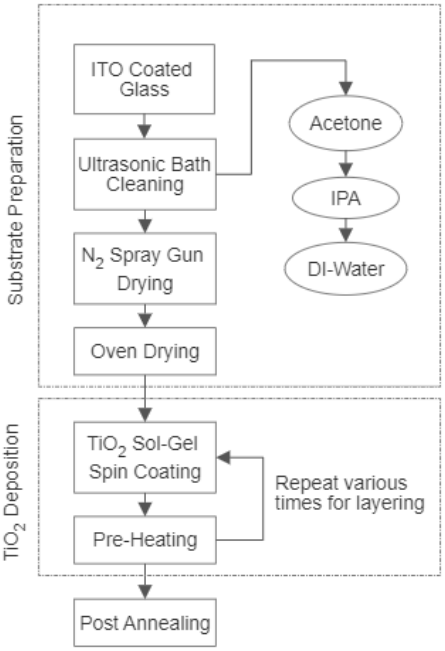

Fig. 1. Schematic diagram representing the flow process of TiO_2 thin film fabrication

2.3 Characterisation

The film thickness for the fabricated TiO_2 thin films was measured using a step profilometer. Meanwhile, the optical properties for TiO_2 thin film samples were performed using a UV-Vis spectrometer to measure the transmittance of the TiO_2 thin film layer only by excluding the substrate part (ITO and glass layers) during the measurement process. The electrochemical properties were measured by applying cyclic voltammetry (CV) and chronoamperometry (CA) with an Autolab Potentiostat in a three-electrode system setup, which was used to control the working electrode's potential in the three-electrode setup. The setup included TiO_2 substrates as the working electrode, platinum (Pt) sheet as the counter electrode, and silver metal in silver chloride (Ag/AgCl) as the reference electrode. All the electrodes were submerged in 1 M of lithium perchlorate in propylene carbonate ($LiClO_4$:PC) electrolyte solution.

The electrochromic properties for the TiO_2 thin film transmittances were measured from the 300 to 900 nm wavelength range using the UV-Vis spectrometer while deploying the three-electrode system setup as the UV-Vis light passes through the sample that is submerged in the $LiClO_4$:PC

electrolyte, and the original, coloured and bleached state transmittances were obtained from values at 630 nm wavelength. The CV measurements were performed to investigate the anodic peak current (I_{pa}), cathodic peak current (I_{pc}), and diffusion coefficient (D) for intercalation and deintercalation of the Li^+ ions under applied voltage from -1.5 to 1.5 V at a scan rate of 0.1 V s^{-1}. Lastly, the colouring and bleaching times of the TiO_2 thin films were obtained from the CA results. To plot the CA graph for the TiO_2 sample, first, a constant voltage of -1.5 V is applied for a duration of 60 s, and the current is measured at each 0.1 s time interval during this time. Immediately after that, the voltage is reversed, and a constant voltage of 1.5 V is applied for a duration of 120 s, and the current is measured for each 0.1 s time interval during this time. The raw data is then retrieved as the measured current vs time duration while the constant voltages were applied (-1.5 and 1.5 V), and plotted using plotting software (Figure 5).

3. Results

3.1 Thin Film Thickness

Based on the surface profilometer, the TiO_2 film thickness measurements for all samples produced similar thicknesses in the range of 100 nm. Hence, this could be attributed to the incomplete pre-heating process between each spin-coating process. When adding the sol-gel solution to prepare the second TiO_2 layer, the first layer may have dissolved in the ethanol medium, and the subsequent TiO_2 layers were not created. As a result, the formation of a highly crystalline TiO2 layer was not efficiently formed.

3.2 Optical Properties

The UV-Vis transmittance spectra for TiO_2 thin film (Figure 2) revealed that the transmittance for all samples at 630 nm wavelength was around 80% except for T9, which produced a slight decrease of approximately 75%. Nevertheless, all layers exhibited an absorption edge at 320 nm, confirmed with values reported in the literature for TiO_2thin films [19].

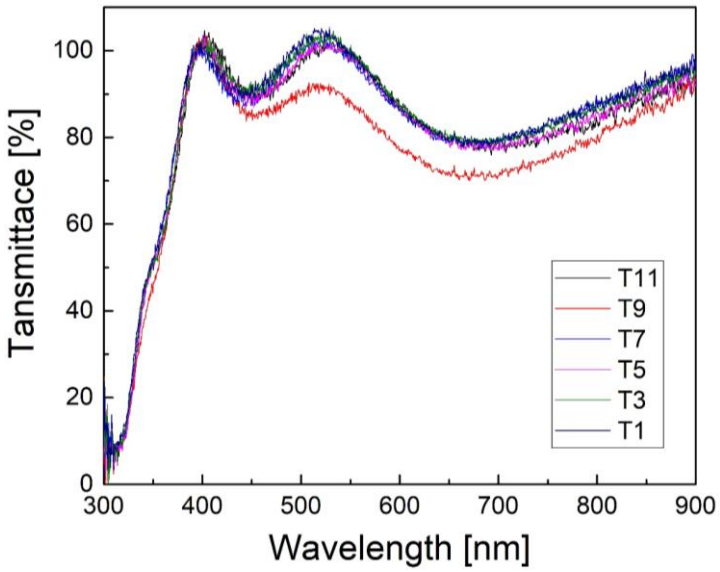

Fig. 2. The transmittance spectra for the various number of TiO_2 layers

3.3 Electrochromic Properties

3.3.1 Transmittance

The transmittance of the fabricated layers was measured for the different electrochromic states of the TiO_2thin films in the three-electrode system setup: original (before any measurements), colouring (by applying -1.5 V), and bleaching (by applying 1.5 V). Figure 3 demonstrates the transmittance for all samples from T1 to T11. Thus, it can be observed that all layers have a similar transmittance value in the original and bleaching states. In contrast, in the colouring state, the transmittance increases as the number of layers increases.

The transmittance at 630 nm for different states was tabulated in Table 2. All layers exhibited a similar transmittance in the original state in the 92 to 82% range. In the bleaching state, the transmittance ranges from 76 to 91%, with the highest and lowest values observed in T3 and T5, respectively. Meanwhile, the transmittance ranges from 74 to 60% was displayed in the coloured state, with the lowest and highest transmittance observed in T3 and T7, respectively.

Using optical modulation (OM) results, the values were measured for all samples using Eq. **Error! Reference source not found.**) and summarised in Table 2 as follows

$$OM = T_b - T_c \quad (1)$$

where *OM* is the optical modulation, T_b is the transmittance in the bleached state, and T_c is the transmittance in the coloured state. Therefore, from the OM results, the values increased with increasing layers until the T3 sample. Subsequently, the OM values decreased until the T7 sample. In contrast, the OM value suddenly increased again from T9 to T11 samples. Based on the results, the OM value for T3 was depicted as the most efficient sample, whereas T7 exhibited the least promising one. Overall, the transmittance results demonstrated that the transmittance values in the original and bleached states were similar. In contrast, in the coloured state, the transmittance increases with the number of layers.

Table 2
Summary of the transmittance states at 630 nm for the EC-TiO_2 samples

Sample Name	Transmittance, T (%)			Optical Modulation, OM (%)
	Original	Bleached, T_b	Coloured, T_c	
T11	89	89	70	19
T9	85	84	69	15
T7	84	84	74	10
T5	82	76	63	13
T3	92	91	60	31
T1	90	88	63	25

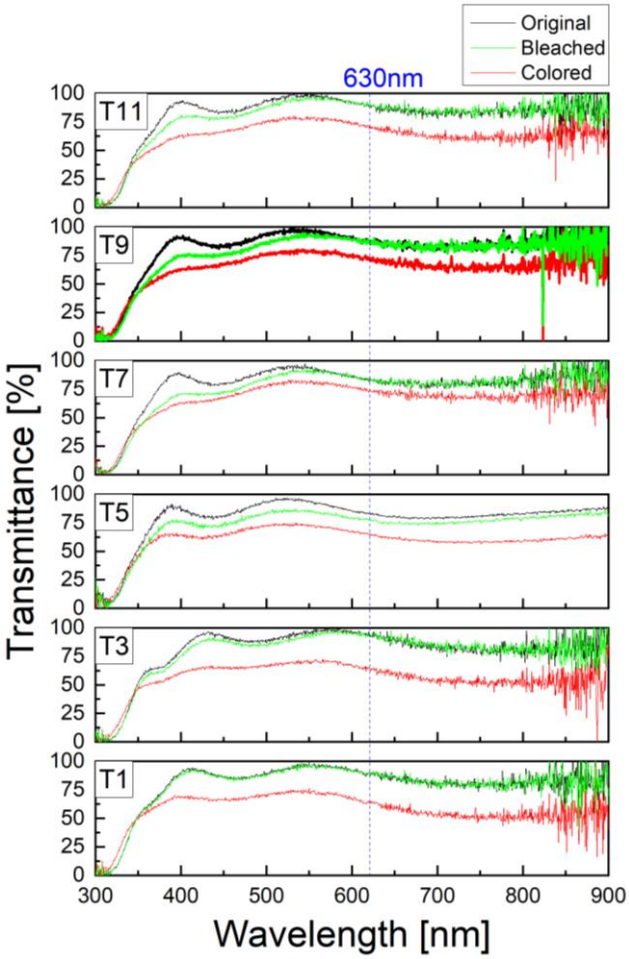

Fig. 3. The transmittance spectra of the EC-TiO_2 in original, bleached, and coloured states

3.3.2 Cyclic voltammetry

The CV graphs for the TiO_2 films with a different number of layers are presented in Figure 4. Moreover, the cathodic and anodic peak characteristics of the CV curves indicated the insertion and extraction of ions. Based on the CV graphs, the curves were like other reported sol-gel fabricated TiO_2 films [20]. Hence, the anodic and cathodic current peaks generally increased as the number of TiO_2 thin film layers increased.

The cathodic peak current increased from -0.35 to -1.41 mA, while the anodic peak current increased from 0.20 to 0.69 mA. The rising active mass deposited on the substrates was successfully demonstrated through the rising cathodic and anodic peak currents. Thus, this implies that the number of layers in the TiO_2 films influenced the number of charges inserted and extracted. From the CV curves, the *D* values (cm^2 s^{-1}) for the inserted and extracted ions during the anodic and cathodic cycles were calculated using the Randles-Sevcik Eq.**Error! Reference source not found.**) [21] shown below

$$I_p = (2.687 \times 10^5) n^{3/2}.A.C(D.v)^{1/2} \quad (2)$$

where I_p is the peak current in amperes, *n* is the number of electrons in the redox cycle, *A* is the area of the TiO_2thin film in cm^2, *C* is the molar concentration of the electrolyte, and *v* is the scan rate used

for the CV measurement in V s^{-1}. Based on the CV curves, the peak current for anodic and cathodic cycles was obtained with the number of electrons and molar concentration were 1, TiO_2 thin film dimension area of 2.5 x 1.5 cm, and a scan rate of 0.1 V s^{-1}.

Table 3 summarises the *D* values calculated in both cycles. The anodic and cathodic diffusion coefficients were observed to increase by increasing the number of layers. The highest diffusion coefficient values were produced with T11, with a value of 7.09 x 10^{-18} cm^2 s^{-1} (anodic) and 6.09 x 10^{-18} cm^2 s^{-1} (cathodic). Alternatively, the lowest diffusion coefficient values were reported with T5, with a value of 2.74 x10^{-17} and 4.21 x10^{-17} cm^2 s^{-1} for anodic and cathodic peaks, respectively.

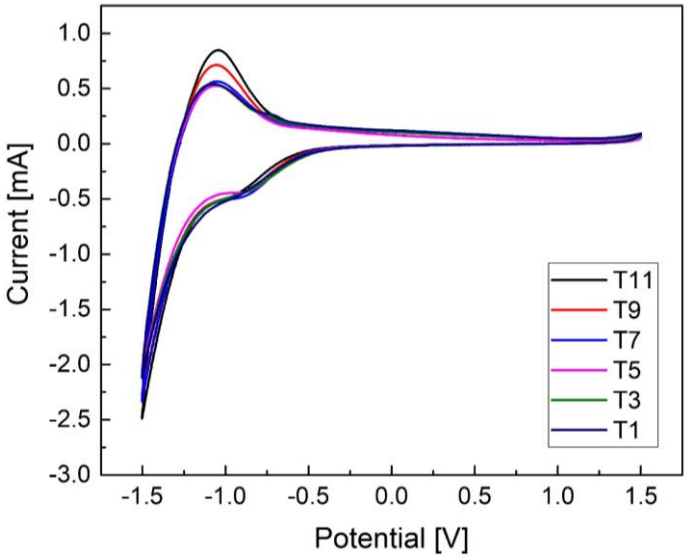

Fig. 4. Graph indicating the CV curves for EC- TiO_2 samples

Table 3
Summary of the EC parameters obtained for the EC-TiO_2 samples

Sample Name	T1	T3	T5	T7	T9	T11
Film Layers	1	3	5	7	9	11
Thickness (nm)	~100	~100	~100	~100	~100	~100
Anodic Diffusion Coefficient, D_a (10^{-18} cm^2 s^{-1})	2.89	2.92	2.74	3.15	5.0	7.09
Cathodic Diffusion Coefficient, D_c (10^{-17} cm^2 s^{-1})	4.43	4.36	4.21	5.35	5.16	6.09
Colouring Time, t_c (s)	18	16	24.4	16.5	11.9	14
Bleaching Time, t_b (s)	5.9	4.8	1.3	4.9	4	5.8
Colouration Transmittance, T_c (%)	63	60	63	74	69	70
Bleaching Transmittance, T_b (%)	88	91	76	84	84	89
Optical Modulation, *OM* (%)	25	31	13	10	15	19
Colouration Efficiency, *CE* (cm^2 C^{-1})	0.97	1.38	0.44	0.43	0.87	1.32

3.3.3 Chronoamperometry

Based on the increased TiO_2 layers, the CA measurements (see Figure 5) were utilised to understand the EC properties. It was used to calculate the switching time for the TiO_2 bleaching and colouring states using the current evolution of the CA graph. Switching times for all samples reported in Table 3 were obtained, as demonstrated in Figure 6.

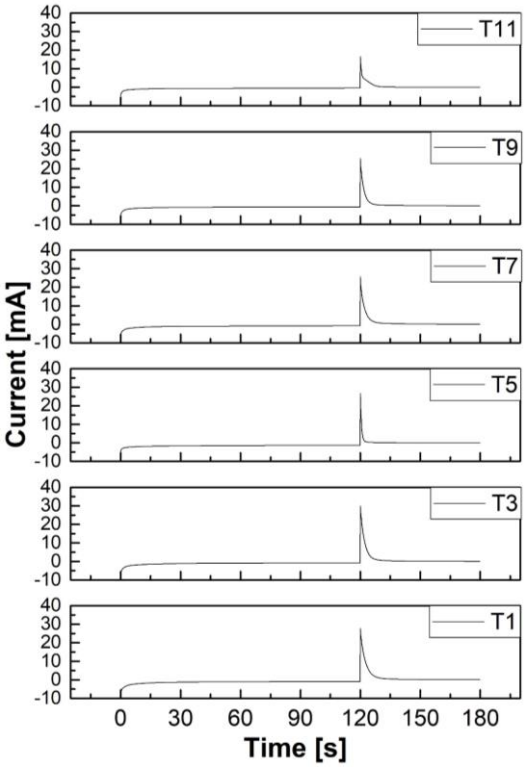

Fig. 5. Graph indicating the CA curves for EC- TiO_2 samples

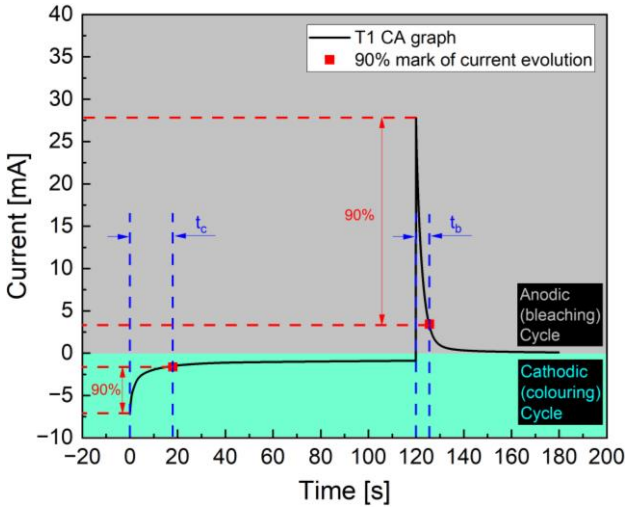

Fig. 6. Graph indicating the switching time for the T1 sample

By applying negative (-1.5 V) and positive (1.5 V) voltage bias, the TiO_2 colouring (t_c) and bleaching (t_b) time during the CA measurement were successfully demonstrated. From Table 3, the lowest and highest t_c values were concluded in T9 and T5, with 11.9 and 24.4 s, respectively. On the contrary, the lowest and highest t_b values were observed in T5 and T1, with a value of 1.3 and 5.9 s, respectively. To elucidate more, as the TiO_2 thin films with different numbers of layers were prepared using sol-gel spin-coating, this can result in differences in the microstructure and morphology of the films. These differences in film properties can affect electron transport and ion diffusion, affecting the colouration and bleaching times. Therefore, the observed differences in t_b and t_c values can be

attributed to the variation in film properties resulting from the differences in the number of layers used for each sample.

4. Conclusion

In conclusion, the EC-TiO_2 thin films with different numbers of layers were fabricated on ITO-coated glasses using a sol-gel spin-coated method. The TiO_2 film thickness for all layers was approximately 100 nm, which was affected by the dissolution process. Regarding optical properties, the optical transmittance of the TiO_2 films was as high as 90 to 100% around the 550 nm wavelength. From CV measurements, the diffusion coefficient increased with the increasing number of layers, and T11 revealed the highest values. The colouration and bleaching time obtained from CA measurements did not show a clear trend correlated to the increased number of layers. Nevertheless, it was observed that the lowest colouration and bleaching times were found in T9 and T5, respectively. Conversely, the transmittance in the bleached and coloured state was successfully demonstrated in T3, with an efficient colouration efficiency. Through this study, the initial study on the effect of the TiO_2 layering technique can aid in understanding the development of EC-TiO_2 devices.

Further investigation is needed for TiO_2 thin film layering using the sol-gel spin-coating method to determine the effect of layer increasing on its optical and electrochromic properties. Changing one of the parameters in the TiO_2 fabrication process is suggested, for example, changing the post-annealing or pre-heating temperatures and observing its influence on the TiO_2thin film performance.

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