PAPER • OPEN ACCESS

# Microstructural studies of doped PEG Ag/$TiO_2$ thin film

To cite this article: Kamrosni Abdul Razak *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **701** 012004

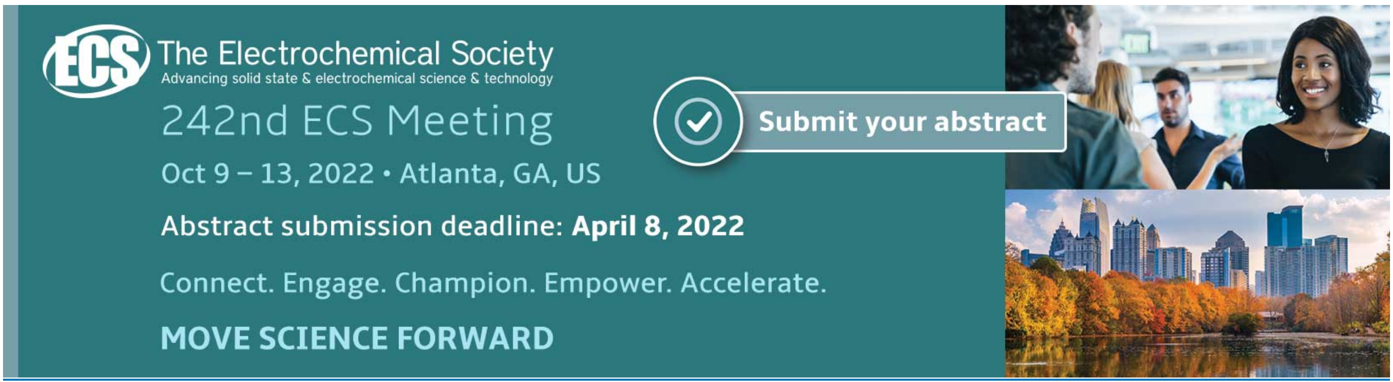
View the article online for updates and enhancements.

You may also like

- Design 5.0 µm Gap Aluminium Interdigitated Electrode for Sensitive pH Detection
M.N. Afnan Uda, Asral Bahari Jambek, U. Hashim et al.

- Effects of Sodium Hydroxide Treatment on LLDPE/DS Composites: Tensile Properties and Morphology
Abduati Alnaid, N Z Noriman, Omar S Dahham et al.

- CdSe modified $TiO_2$ nanotube arrays with Ag nanoparticles as electron transfer channel and plasmonic photosensitizer for enhanced photoelectrochemical water splitting
Enzhou Liu, Peng Xue, Jia Jia et al.




This content was downloaded from IP address 103.86.131.118 on 15/03/2022 at 06:34

IOP Conf. Series: Materials Science and Engineering **701** (2019) 012004 doi:10.1088/1757-899X/701/1/012004

# Microstructural studies of doped PEG Ag/$TiO_2$ thin film

*Kamrosni* Abdul Razak[1,2*], *Dewi Suriyani* Che Halin[1], *Azliza* Azani[1], *Mohd Mustafa Al Bakri* Abdullah[1], *Mohd Arif Anuar* Mohd Salleh[1], *Norsuria* Mahmed[1], *Varistha* Chobpattana[3], and *Ayu Wazira* Azhari [1,4]

[1]Center of Excellence Geopolymer & Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis, (UniMAP), 02600 Jalan Kangar-Arau, Perlis,
[2]Center for Diploma Studies, Universiti Malaysia Perlis, Unicity Alam Campus, Perlis,Malaysia
[3]Department of Materials and Metallurgical Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi (RMUTT), Thailand
[4]Water Research Group (WAREG), School of Environmental Engineering, Universiti Malaysia Perlis 02600 Arau, Perlis, Malaysia.

**Abstract.** Ag/$TiO_2$ thin film was prepared by the sol-gel method through the hydrolysis of titanium tetraisopropoxide and silver nitrate solution. Various amount of PEG was doped into the solution preparation to study the effect on crystalline state and microstructural of the prepared thin films. Spin coating method was used to get uniform film on ITO glass substrate followed by annealing process for 1 hour. The obtained thin films were analysed using XRD test, SEM and AFM. Results showed that all the prepared thin films are in anatase $TiO_2$ Increasing the PEG amount into the solution could increase the thickness and surface roughness of the obtained thin films.

## 1 Introduction

Titanium dioxide known as titanium (IV) dioxide or titania has a molecular weight of 79.87 g/mol which is a natural titanium oxide with $TiO_2$ chemical formula. Titanium dioxide is a substance commonly used as white pigment due to its remarkable properties such as high purity, brightness, and non-toxicity. It is also known for its excellent chemical stability, biologically non-toxic and low cost [1]. It is an important inorganic functional material with good physical properties, making it ideal for thin film applications. $TiO_2$ thin films are one of the most important oxides due to their attractive chemical, electrical and optical properties [2-4]. $TiO_2$ has been studied by many researchers because of its many applications in various industries [5-7]. Due to its high corrosion resistance and chemical

Corresponding author:dewisuriyani@unimap.edu.my

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

stability, excellent optical transparency near infrared and visible areas, it provides a high refractive index which makes it useful for anti-reflection coating in optical devices [8].

Additives are used to enhance the performance and simultaneously improve the coating of the thin film. Additives can be in many types of materials or chemicals. The additive was found to improve the surface area whereby increase the surface area resulting the great efficiency of prepared thin films. Polyethylene glycol (PEG) is made up of various sizes and functional groups that are synthetic polymers [9]. These attractive additives are also known as polymer additives which have several advantages over the resulting coating such as adding active surface area with porous surface structure due to pore formation and reducing energy gap in $TiO_2$ [10]. Porous films have been obtained by using PEG as a chelating agent [11]. In this research, we focused on the effect of the addition of PEG into Ag/$TiO_2$ thin films.

## 2 Materials and Preparation of Ag/$Tio_2$ Thin Films

Titanium (IV) isopropoxide 97% (TTIP) and silver nitrate ($AgNO_3$) powder were obtained from Sigma Aldrich. . Propan-2-ol was purchased from QReC Chemicals and acetic acid (99.5%) and methylene blue were obtained from Daejung Reagent Chemicals and respectively. All the chemicals were analytical reagents and were used as received without further purification.

The Ag/$TiO_2$ thin film without PEG was prepared as in previous study [12]. Then, for the Ag/$TiO_2$ thin film with PEG, a different amount of PEG (0.05g, 0.10g, 0.15g and 0.20g) was added into the solution after the 25 min sol stirring. The resultant alkoxide solution was kept stirring at room temperature until a clear sol produced, without any precipitation. ITO coated glass (15mm×15mm×0.5mm) were used as the substrates for the deposition of films.

The prepared Ag/$TiO_2$ thin films were characterised structurally through XRD and morphologically by SEM. The phase composition of the synthesized thin film were determined by using an X-ray diffraction (XRD) over a 2θ range 10°- 65° using Cu Kα (λ=1.5046). Analysis of the XRD patterns was carried out using Diffract Eva Software. Surface morphology and surface roughness of prepared the thin films were observed via SEM and AFM.

## 3 Results and Discussions

### 3.1 Phase and Crystallite Size Analysis

X-ray diffraction analysis of Ag/$TiO_2$ thin films with different amount of PEG deposited on ITO substrate is shown in **Fig. 1**. All the films present five strong diffraction peaks located at 21.51◦, 30.57◦, 35.40◦, 51.04◦and 60.70◦. These peaks are attributed to the ITO substrates, $SiO_2$ and $In_2O_3$ peaks. As can be seen, a small peaks at 25.30$^o$ which are assigned to $TiO_2$ anatase (101) plane, referring to JCPDS JCPDS PDF-021-1272. No diffraction peak of silver is detected which can be explained by the dominance of the ITO peaks in the spectrum with their high intensities, making it difficult to detect silver existing in low amounts [13]. A small peak located at 33.66$^o$ is assigned to silver oxides, $Ag_2O$ (100) plane, according to JCPDS No 01-072-2108. However, the X-ray diffraction pattern for the doped PEG Ag/$TiO_2$ thin film showed the intensity of the diffraction lines is gradually decrease with the increasing amount of PEG. The addition of the PEG 2000 in the Ag/$TiO_2$ influenced on the unit cell parameters of anatase [14]. The intensity of the diffraction peak decreases and the amplitude of the diffraction peak decreases, indicating that $TiO_2$ crystalline size is decreasing, consistent with studies conducted by previous researchers [15-17].

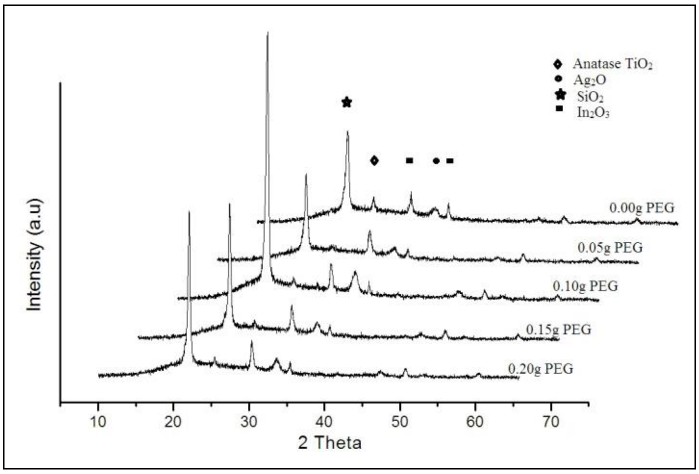



**Fig. 1**: XRD pattern of Ag/$TiO_2$ thin films with different amounts of polyethylene glycol

As can be seen, the intensity of the diffraction peak decreases and the apex aperture decreases, indicating that $TiO_2$ crystalline size decreases, consistent with a study conducted by previous researchers [15-17]. The graph in **Fig. 2** shows the crystalline size distribution of Ag/$TiO_2$ thin films produced with different PEG amounts. The crystalline size is calculated from the Debye-Scherer formula using the XRD extension. Overall, the $TiO_2$ crystalline size decreased with the addition of PEG additives. According to [16], the presence of PEG in the Ag/$TiO_2$ thin film layer reduced the size of the crystals, but did not reduce the amount of $TiO_2$ anatase crystals that were an important factor in the photocatalytic process. The Ag/$TiO_2$ thin film crystals were reduced from 40.39 nm to 29.78 nm, 23.88 nm, 19.62 nm and 23.63 nm with the addition of PEG weights of 0.05g, 0.10 g, 0.15 g and 0.20 g, respectively. The smallest crystals recorded were 0.15 g of PEG, 19.62 nm, which is approximately 105% of the Ag/$TiO_2$ thin film structure that was not added to PEG, 40.39 nm.

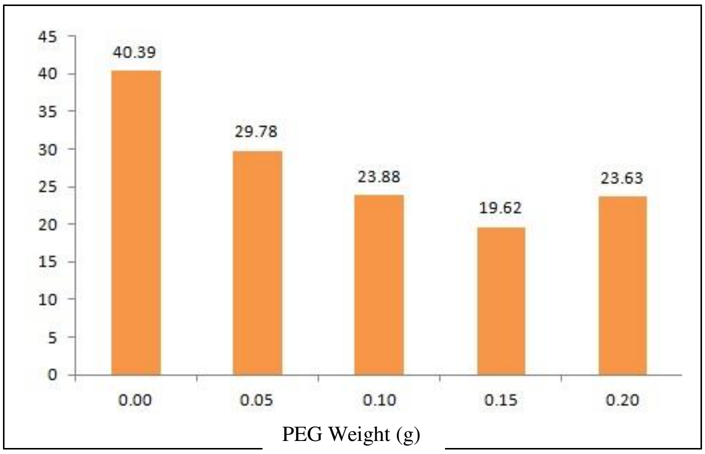



**Fig. 2**: Crystallite size distribution of $Ag/TiO_2$ thin films with different amounts of polyethylene glycol

According to Petrik et al. [18] during the $Ag/TiO_2$ thin film synthesis process, the PEG molecules are absorbed externally into the -Ti-O-Ti- oligomers and form a hydrogen bond. The subsequent formation and polymerization process consists of the formation of an oligomer/PEG composite, in which PEG determines the size of the crystals formed.

### 3.2 Morphological Observations

The surface morphology of $Ag/TiO_2$ thin films with different amounts of PEG was observed under SEM as shown in **Fig. 3**. All the prepared $Ag/TiO_2$ thin films are uniform and evenly distributed in uneven shape and porous structure. From **Fig. 3**(b), $Ag/TiO_2$ thin film with the addition of 0.05g of PEG shows a thin film with a series of a small, coarse and loose crystalline structures compared to the non-PEG thin film structure (**Fig. 3**(a)), with a larger and thicker structure, in line with the crystalline size obtained from XRD analysis. The PEG 2000 additives in the $Ag/TiO_2$ solvents act as a dispersion agents, where the low $TiO_2$ particle aggregation can be explained by the presence of a steric barrier. PEG additives ensure that the soluble particles are separated in solution due to the dispersion of polymers as long-chain molecules [17]. However, no significant changes were observed in the surface morphology of the $Ag/TiO_2$ thin films when the amount of PEG was increased from 0.10g to 0.15 and 0.20g as shown in **Fig. 3** (c) to **Fig. 3** (e). Structural changes on the surface of the resulting thin films were due to the thermal decomposition of PEG during the annealing process which led to the formation of coarse and porous structures. A similar situation has been reported by previous researchers [13].

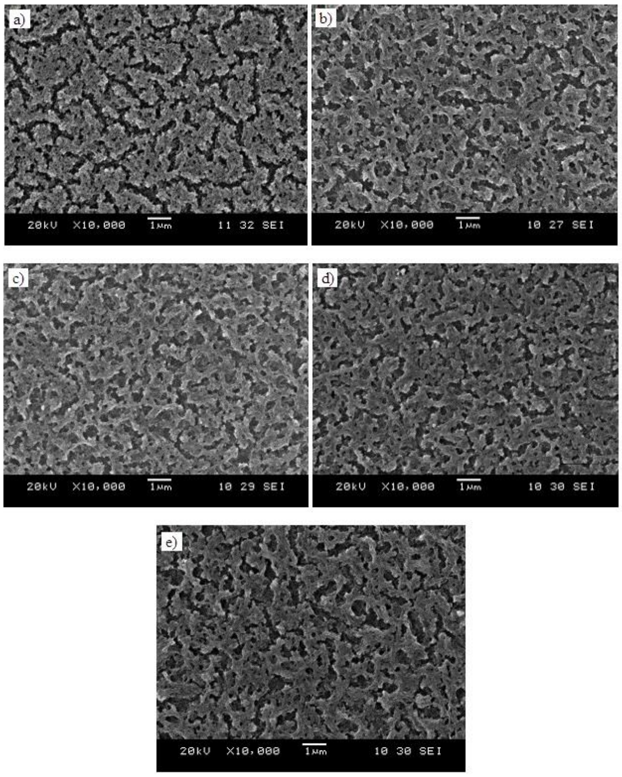



**Fig. 3** : SEM image of $Ag/TiO_2$ thin films with different amounts of polyethylene glycol

In order to investigate the effect of PEG addition on the morphology and surface roughness of the resulting $Ag/TiO_2$ thin films, AFM analysis was performed. **Fig. 4** shows the topography and 3D views of the surface structure of the thin films produced, while **Table 1** shows the surface roughness of $Ag/TO_2$ thin films with different PEG amounts. It can be clearly seen that the structure of the $Ag/TiO_2$ thin film without the addition of PEG is smooth, uniform and covers the entire surface of the substrate, **Fig. 4** (a). The maximum height of this film was 14.98nm with an average roughness of 1.81nm. When 0.05g of PEG was added, the resulting film structure changed to a coarse, non-uniform form, as shown in **Fig. 4** (b). The maximum height of this film was 36.29nm with the average roughness slightly increase to 5.88nm.

Furthermore, when the added PEG weight is increased to 0.10g, 0.15g and 0.20g, the resulting $Ag/TiO_2$ thin film structures becomes rough and porous. The $Ag/TiO_2$ films thickness were also increased with the maximum layer thickness recorded at 229.67 nm, 279.49 nm and 300.80 nm. The root mean square roughness of the film also increased with increasing PEG weight to 39.5nm, 44.71nm and 49.37nm. The differences in the surface morphology of the resulting films can be explained by the formation of pores within the $Ag/TiO_2$ film layer due to PEG thermal decomposition during the annealing process [13]. When more amount of PEG added to the sol solution, the viscosity of the sol increased. The solution attached to the substrate increase and make the resulting thin film layer thicker. Tiwari et al., [19] in their study also found that the $TiO_2$ thin film coated with PEG was thicker than the non-PEG coated film.

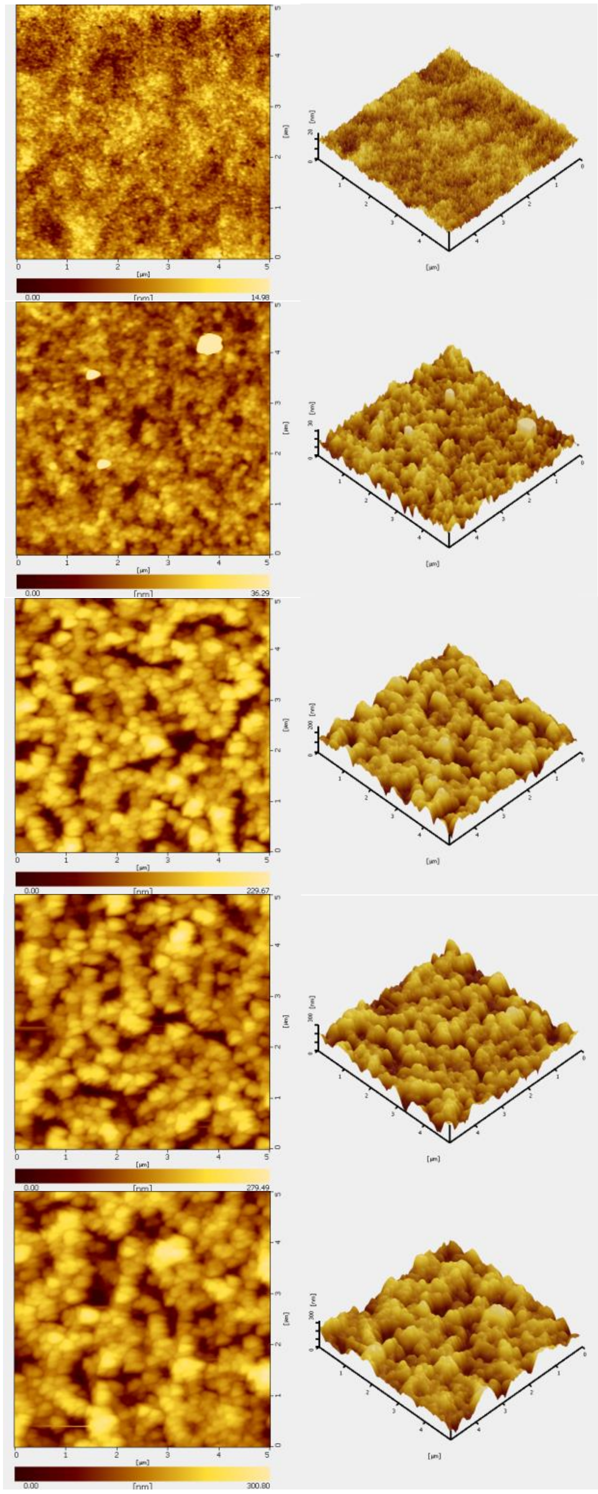



**Fig. 4** : Surface morphology of $Ag/TiO_2$ thin films with different amounts of polyethylene glycol

**Table 1** : Surface roughness of Ag/TO$_2$ thin film with different amount of polyethylene glycol

| Ag/TiO$_2$ thin film | PEG weight (g) | Surface roughness, Ra (nm) | Surface roughness, RMS (nm) |
|---|---|---|---|
| (a) | 0.00 | 1.38 | 1.81 |
| (b) | 0.05 | 3.60 | 5.88 |
| (c) | 0.10 | 30.71 | 39.5 |
| (d) | 0.15 | 35.55 | 44.71 |
| (e) | 0.20 | 38.78 | 49.37 |

## 4 Conclusion

This work observes the effect of various PEG amounts on morphological and optical properties of Ag/TiO$_2$ thin films. The XRD pattern showed the presence of anatase TiO$_2$ and Ag$_2$O phases. The Ag/TiO$_2$ thin films shows a rough and porous surface morphology. The surface roughness of the film increased with the increasing of PEG amounts.

The author would like to acknowledge the support from the Fundamental Research Grant Scheme (FRGS) under a grant number of FRGS/1/2017/TK07/UNIMAP/02/6 from the Ministry of Education Malaysia and Tin Solder Technology Research Grant (TSTRG) under grant number of 9002-00082 from Tin Industry (Research and Development) Board. The authors wish to thank the Center of Excellence Geopolymer & Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis, UniMAP for their partial support.

## References

1. Y. Liang, S. Sun, T. Deng, H. Ding, W. Chen, & Y. Chen, Materials, **11**(3), 1–12 (2018)
2. A. Marzec, M. Radecka, W. Maziarz, A. Kusior and Z. Pedzich, Journal of the European Ceramic Society **36** 2981- 2989 (2016)
3. A. Lewkowicz, A. Synak, B. Grobelna, P. Bojarski, R. Bogdanowicz, J. Karczewski, K Szczodrowski and M. Behrendt , Optical Materials **36** 1739 – 1744 (2014)
4. N. Arconada, Y. Castro, A. Duran, S. Suarez, R. Portela, J. M. Coronado and B. Sanchez Applied Catalysis B: Environmental **86** 1-7 (2009)
5. M. Kumar, M. Kumar and D. Kumar, *Microelectronic Engineering* **87** 447–450 (2010)
6. A. Azani, D. S. C. Halin, K. A. Razak, M. M. A. B. Abdullah, M.A.A. Mohd Salleh, N. Mahmed , M. M. Ramli, S. Sepeai, IOP Conf. Series: Materials Science and Engineering **551** (2019)
7. A. Azani, D. S. C. Halin, K. A. Razak, M. M. A. B. Abdullah, M.A.A. Mohd Salleh, N. Mahmed , M. M. Ramli, S. Sepeai, V. Choptabbana, AIP Conference Proceedings **2129**, 020062 (2019)
8. A. Elfanaoui, E. Elhamri, L. Boulkaddat, A. Ihlal, K. Bouabid, L. Laanab, A. Taleb and X. Portier, International Journal of Hydrogen Energy **36** 4130-4133 (2010)

9. Z. Karimi, L. Karimi and H. Shokrollahi, *Materials Science & Engineering C*, *33*(5), 2465–2475 (2013)
10. A. R. Nurhamizah, M. R. Zulkifli and J. M. Juoi, *www.scientific.net/KEM* **694** 160–164 (2016)
11. K. A. Razak, D. S. C. Halin, M. M. A. B. Abdullah, M.A.A. Mohd Salleh, N. Mahmed and N.S. Danial. Solid State Phenomena, ISSN: 1662-9779, Vol. **280**, pp 26-30 (2018)
12. K. A. Razak, D. S. C. Halin, A. Azani, M. M. A. B. Abdullah, M.A.A. Mohd Salleh, N. Mahmed , M. M. Ramli, S. Sepeai, IOP Conf. Series: Materials Science and Engineering **551** (2019) 012098
13. D. Guitoume, S. Achour, N. Sobti, M. Boudissa, N. Souami, Y. Messaoudi, Optik 154 182–191 (2018)
14. V. Vetrivel, *UGC Sponsored National Seminar on Emerging Trends in Plasma Technology and Its Applications* (2014)
15. Calderon Moreno, J.M., Popa, M., Ivanescu, S. et al. Met. Mater. Int. **20**,I1,pp 177–187 (2014)
16. J. C. Morales Mejia, L. Angeles, R. Almanza, Computational Water, Energy, and Environmental Engineering, 2014, **3**, 36-40 (2014)
17. H. S. Bhojya Naik, P. N. Prashanth Kumar, K. N. Harish and R. Vishwanath, European Journal of Applied Engineering and Scientific Research, **2** (2):1-7 (2013)
18. I. Petrik., E. Frolova, A. Turchin, N. Smirnova, and A. Eremenko, Research on Chemical Intermediates, (2019)
19. D. Tiwari, C. Lalhriatpuia, Lalhmunsiama, S. M. Lee and S. H. Kong, Applied Surface Science **353** 275–283 (2015)