

# Effect of Heat Treatment on the Structural Properties of TiO<sub>2</sub> Films Produced by Sol-Gel Spin Coating Technique

M. Nebi<sup>1</sup> and D. Peker<sup>1</sup>

<sup>1</sup> Physics Department, Eskisehir Osmangazi University, Eskisehir, TURKEY

E-mail: mnebi@ogu.edu.tr

**Abstract.** Due to have superior properties as photocatalyst and have wide band gap, TiO<sub>2</sub> thin films often investigated by researchers and used by technological applications widely. In this study TiO<sub>2</sub> films were deposited on glass substrate by Sol-Gel Spin Coating Technic. TiO<sub>2</sub> films were deposited at different number of layer and then annealed at 400° C, 500° C, and 600° C in air. Effect of anneal temperature to structural properties were investigated by XRD analysis. It was observed by the light of XRD results that the structural properties of films had changed by anneal temperature.

## 1. Introduction

Titanium dioxide (TiO<sub>2</sub>) is a large band gap (i.e., 3.20 eV for the anatase crystalline phase) semiconductor extensively studied due to its unique combination of chemical and physical properties used to develop various environment and energy related applications [1-3].

TiO<sub>2</sub> materials have thermal and chemical stability, good mechanical hardness and high UV photoactivity. TiO<sub>2</sub> is transparent to visible light and has high refractive index making it suitable for the development of various optical thin film applications [1-4].

It has been shown as one of the most important materials for applications in semiconductor electrochemistry, as gas sensor, as antireflective coatings, in electrochromic devices and solar energy conversion [5, 6].

When exposed to UV radiation with photon energy greater than the band gap energy of the material, the TiO<sub>2</sub> surface becomes super-hydrophilic, self-cleaning, self-sterilizing and antifogging. The application range of TiO<sub>2</sub> can be extended by improving the properties of these materials through doping with transition metal ions (Fe, Mo, Ag, Co, W, etc.) [7-17].

In this study TiO<sub>2</sub> films were deposited on glass substrate by Sol-Gel Spin Coating Technic. TiO<sub>2</sub> films were deposited at different number of layer and then annealed at 400° C, 500° C, and 600° C in air. Effect of anneal temperature to structural properties were investigated by XRD analysis.



## 2. Experimental

It is important that producing thin films by practical and economic way. There are various methods that using to produce semiconductive thin films. One of them which has wide use is Sol-Gel Spin Coating Technique.

This chemical method bases on preparing a solution and drop solution on very fast (ex; 1500-4000 rpm) spinning surface. This method uses to deposit film on hard and rough surfaces.

### Advantages of Sol-Gel Spin Coating Technique:

- 1. No need to high temperature and vacuum
- 2. Easy to learn and use.
- 4. Deposited films are homogeneous at macro dimension.
- 5. Has easy control process
- 6. Can apply to surfaces which has different geometric shapes.
- 7. Energy conservation
- 8. Low cost and fast.

Spin Coating Method contains successive 4 steps;

- Dropping
- Spinning
- Drying
- Repeating same steps for multiple layers

Steps of Spin Coating Method are shown in Figure 2.1.

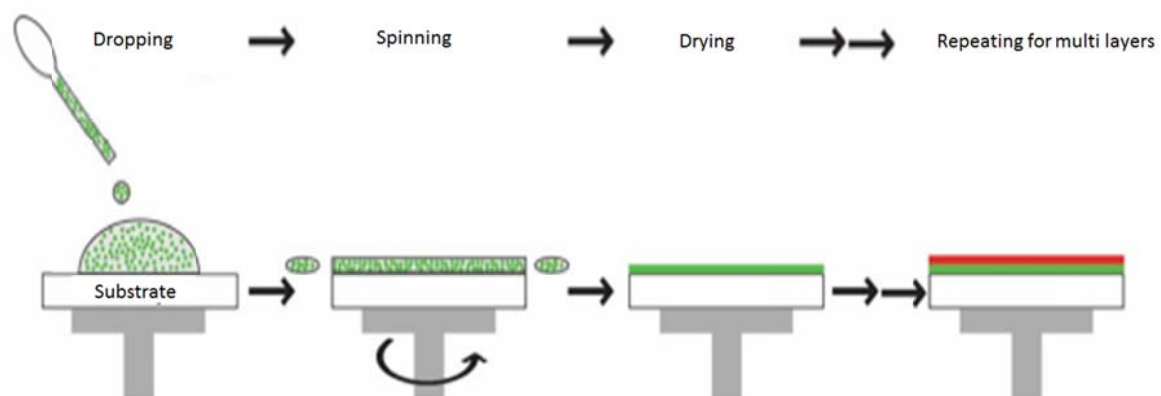


Figure 2.1. Schematic illustration of sol-gel spin coating method.

Prepared solution is drop on surface and surplus solution throw away from the sides of substrate by effect of spinning. At the same time, the film starts to deposit on substrate homogeneously. After substrates dried we can repeat this steps for tuning the film thickness of material.

### 2.1. Preparing Glass Substrates

In this study we used microscope glass for substrate. Microscope glasses cut as 1 cm square and then we applied following step for cleaning the glass substrates.

- Boil in a distilled water with little mass of detergent for 5 min.
- Wash with distilled water for 5 min.
- Wash with isopropyl alcohol water for 5 min.
- And then again wash with distilled water for 10 min.
- Dry at 70° C in a drying oven for an hour.

## 2.2. Preparing $\text{TiO}_2$ Solution

When 180 ml ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) stay mixing in a beher cup we added respectively 4 ml acetylacetone ( $\text{C}_5\text{H}_8\text{O}_2$ ), 2 ml distilled water and 14 ml titanium butoksit (Titanium (IV) n-butoxide, ( $\text{C}_{16}\text{H}_{36}\text{O}_4\text{Ti}$ )) After chemicals added solution awaited for 24 hour under atmospheric conditions.

$\text{TiO}_2$  films were deposited at different number of layer 1-3-5 and then annealed at  $400^\circ\text{C}$ ,  $500^\circ\text{C}$ , and  $600^\circ\text{C}$  in air. Then effect of anneal temperature to structural properties were investigated by XRD analysis.

## 3. Results

### 3.1. XRD ( X-Ray Diffractometer ) Analysis

The crystal structure and the preferred crystal orientation of  $\text{TiO}_2$  films were investigated by X-ray diffraction (XRD) measurements. XRD measurements were performed by Panalytical Emprayan X-ray diffractometer using  $\text{CuK}\alpha$  ( $\lambda=1.5405\text{ \AA}$ ) radiation in the  $2\theta$  range  $30^\circ - 80^\circ$  with a scanning speed of  $2^\circ/\text{min}$ . X-ray diffraction measurements were performed and the obtained results are shown in Figure 3.1 to 3.9.

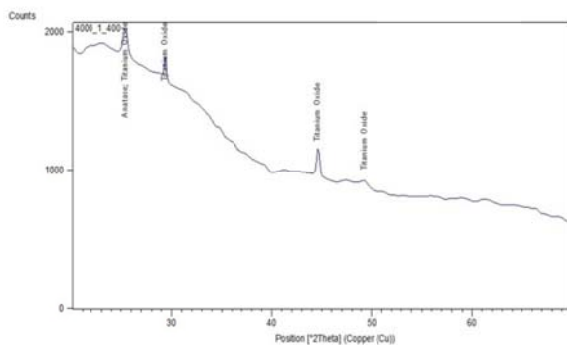


Figure 3.1. 4000 rpm, 1 layer, annealed at  $400^\circ\text{C}$ .

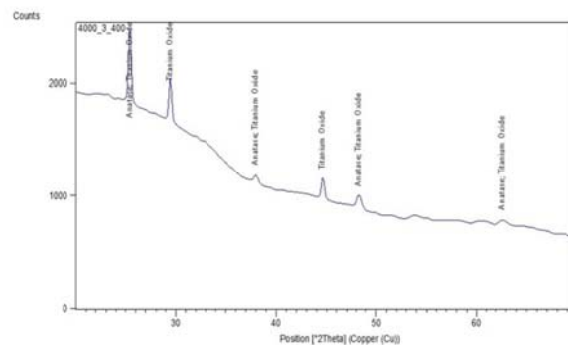


Figure 3.2. 4000 rpm, 3 layers, annealed at  $400^\circ\text{C}$ .

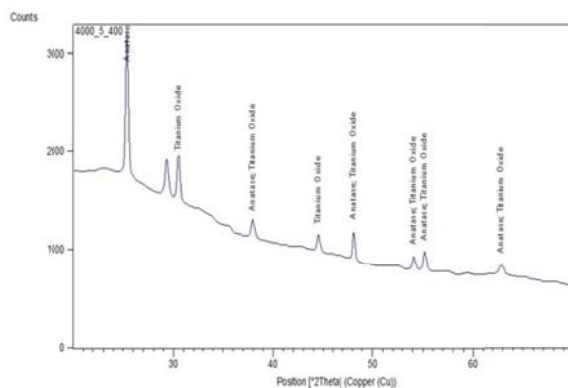


Figure 3.3. 4000 rpm, 5 layers, annealed at  $400^\circ\text{C}$ .

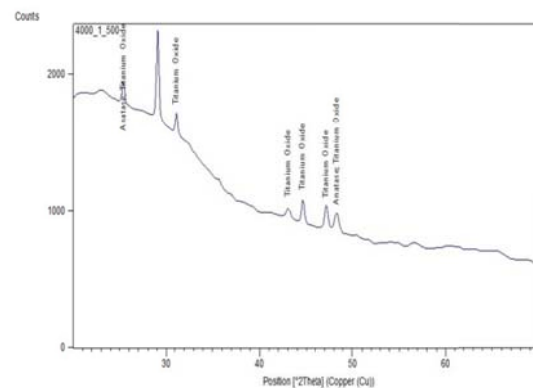


Figure 3.4. 4000 rpm, 1 layer, annealed at  $500^\circ\text{C}$ .

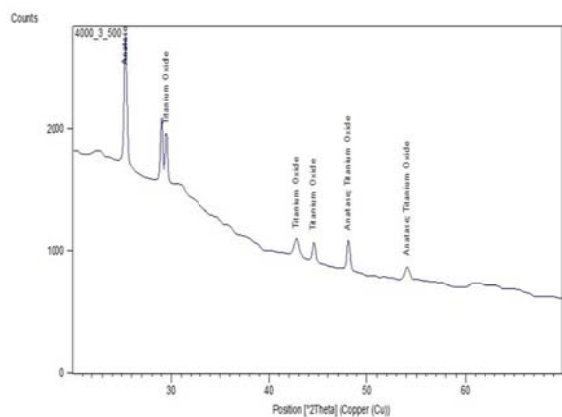


Figure 3.5. 4000 rpm, 3 layers, annealed at 500° C.

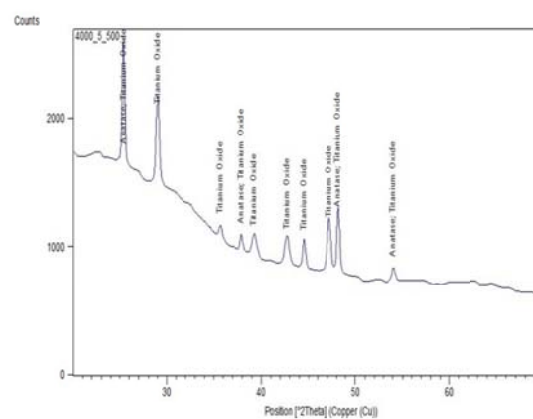


Figure 3.6. 4000 rpm, 5 layers, annealed at 500° C.

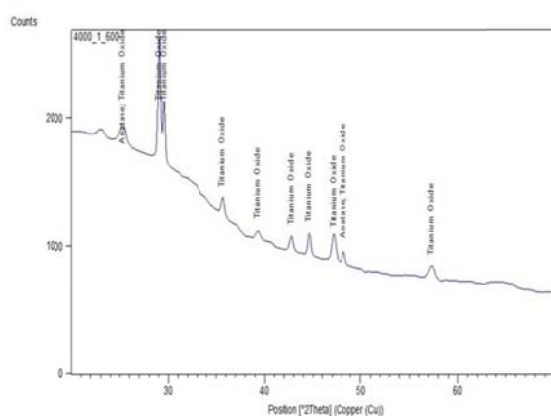


Figure 3.7. 4000 rpm, 1 layer, annealed at 600° C.

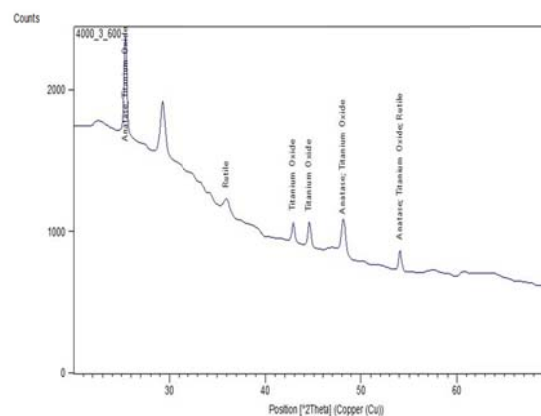


Figure 3.8. 4000 rpm, 3 layers, annealed at 600° C.

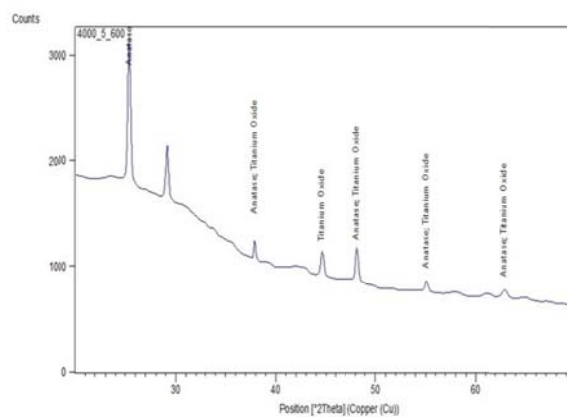


Figure 3.9. 4000 rpm, 5 layers, annealed at 600° C.

#### 4. Conclusions

Effect of heat treatment on the structural properties of TiO<sub>2</sub> films produced by Sol-Gel Spin Coating Technique has been examined. It was observed by the light of XRD results that the structural properties of films, had changed by anneal temperature and number of layers. X-ray diffraction studies revealed that the crystalline structure of films which were annealed at 600° C in air are relatively better than the others.

#### References

- [1] Fujishima A *et al* 2008 Surface Science Reports **63** 515
- [2] Fujishima A *et al* 2000 Journal of Photochemistry and Photobiology C: Photochemistry Reviews **1** 1
- [3] Manole A V *et al* 2013 Ceramics International **39** 4771
- [4] Karunakaran B *et al* 2005 Physica B **369** 129
- [5] Gratzel M and Kalyanasundaram K 1998 Coordination Chemistry Reviews **77** 347
- [6] Subramanian M *et al* 2008 Thin Solid Films **516** 3776
- [7] Thompson T L and Yates J T 2006 Surface Chemical Reviews **106** 4428
- [8] Kubacka A *et al* 2009 Catalysis Today **143** 286
- [9] Subasri R *et al* 2010 Materials Chemistry and Physics **124** 63
- [10] Castro A L *et al* 2009 Journal of Solid State Chemistry **182** 1838
- [11] Furubayashi Y *et al* 2006 Thin Solid Films **496** 157
- [12] Hitosugi T *et al* 2008 Thin Solid Films **516** 5750
- [13] Maghanga C M *et al* 2009 Thin Solid Films **518** 1254
- [14] Sato Y *et al* 2008 Thin Solid Films **516** 5758
- [15] Hung K H *et al* 2011 Journal of Alloys and Compounds **509** 10190
- [16] Zhao L *et al* 2010 Journal of Sol–Gel Science and Technology **53** 475
- [17] Liu J X *et al* 2011 Applied Surface Science **257** 10156