Available online at www.scholarsresearchlibrary.com

Scholars Research Library

Archives of Physics Research, 2011, 2 (4):121-127
(http://scholarsresearchlibrary.com/archive.html)

Scholars Research Library

ISSN : 0976-0970
CODEN (USA): APRRC7

Studies on Structural, Optical and Mechanical properties of TiO2 thin film

Uma Ullas Pradhan* and Naveen Kumar S K†

** Department of Electronics, Mount Carmel College, Bangalore*
† Department of Electronics, Mysore University, Hassan

ABSTRACT

Titanium dioxide films were deposited on glass substrates by Spin Coating process. The thin film samples were characterized for its structural, optical and mechanical properties using SEM, EDAX, UV-Vis Spectrophotometer, X-ray Diffraction (XRD) and micro hardness unit. Optical constants such as k and α were studied and discussed. The variation of thickness of the thin film with number of times of deposition was found to be linear. The optical band gap energy was estimated to be 3.61 eV. Work hardening index was found to be 0.53.

Keywords: SEM, EDAX, Eg, k, α, Hardening index.

INTRODUCTION

The fabrication of Titanium dioxide thin films is a highly researched topic as the material exhibits a lot of important optical and electrical properties [1]. Titanium dioxide films have attracted attention for use in fabricating capacitors in manufacturing microelectronic devices due to their high dielectric constants [2]. To achieve good optical and electrical properties, it is necessary to carefully select the process of fabrication. Titanium dioxide films can be fabricated using different techniques such as thermal evaporation, rf-sputtering, chemical vapour deposition, laser ablation, Spray pyrolysis, Spin coating [2]... it becomes difficult to choose the method of preparation. The sol-gel technique (spin coating, dip coating) has distinct advantages over the other methods as the structural properties of the films can be carefully controlled due to the usage of the liquid precursors.

In this paper, we present a study of the structural, optical and mechanical properties of Titanium dioxide films fabricated using a low cost Spin coating technique.

MATERIALS AND METHODS

Spin coating

Titanium isopropoxide 10 ml/100 ml [3] was dissolved in ethyl alcohol. 2.5 ml of Hydro chloric acid was added as a catalyst. The solution after stirring for 1hour is left in an air tight container for 12 hours. 2 – 3 drops of sol were put on the glass substrate and spin coated. Films of different thickness were prepared and dried in a hot air oven at 60^0 for 6 hours. The films were annealed at 500^0 for 1 hour to make it crystalline.

RESULTS AND DISCUSSION

Optical properties

The prepared titanium dioxide films were characterized for their optical properties. The optical transmission spectra of annealed samples were recorded as shown in figure1 using a UV-VIS spectrophotometer (UV-1700 Pharma Spec, SHIMADZU).

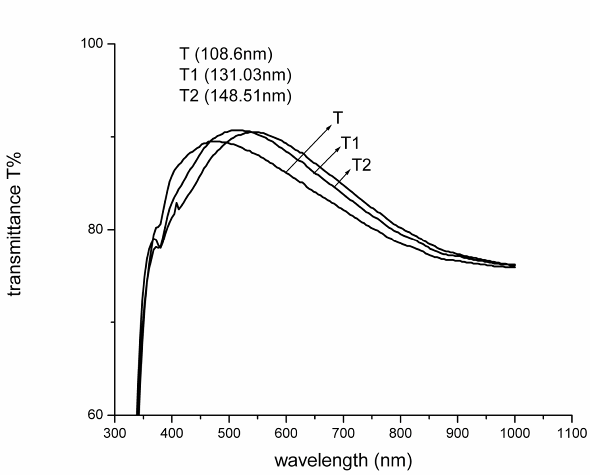

Figure 1. Transmittance spectra of TiO_2 films of different TiO_2 films annealed at 500^0 for 1hour.

The films showed maximum transmittance of about 90.82% at around 515-560 nm, with reference to blank glass substrate and showed a shift in the peak for varying thickness of the film. The relatively high transmittance of the film indicates low surface roughness and good homogeneity [5].

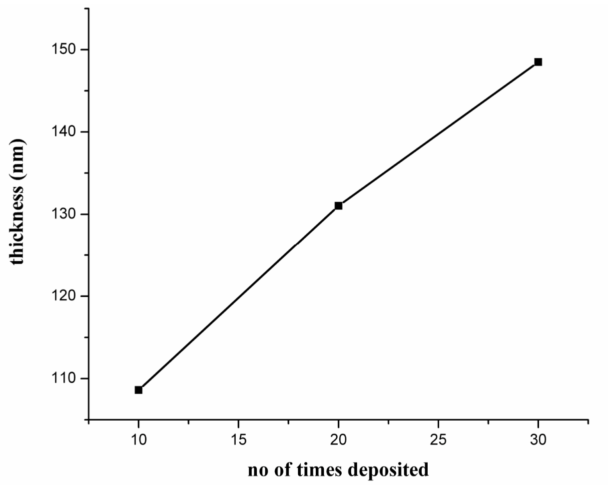

Figure 2. Thickness VS times of deposition of thickness annealed at 500^0 C for 1hour .

The thickness of the thin films were estimated using max-min method, using the formula

$$t = \lambda_1 \lambda_2 / 4n (\lambda_2 - \lambda_1)$$

Where't' is the thickness of the film, λ_1 and λ_2 are the wavelengths which corresponds to the maxima and minima of the transmittance spectra and 'n' is the refractive index to titanium dioxide. A graph of thickness of the film with number of time of deposition as in figure 2, shows a linear increase in the film thickness with increase in the number of times of deposition [9]. The thicknesses of the samples were found to be 108.6nm, 131.03nm and 148.51nm respectively for 10, 20 and 30 times of deposition.

A graph of $(\alpha h\nu)^2$ Vs (E = hν) was plotted to estimate the direct optical band gap [5, 7] of the thin films as shown in figure 3. The films T1 and T2, the as deposited and annealed films respectively, exhibited direct band energy of 3.61eV.

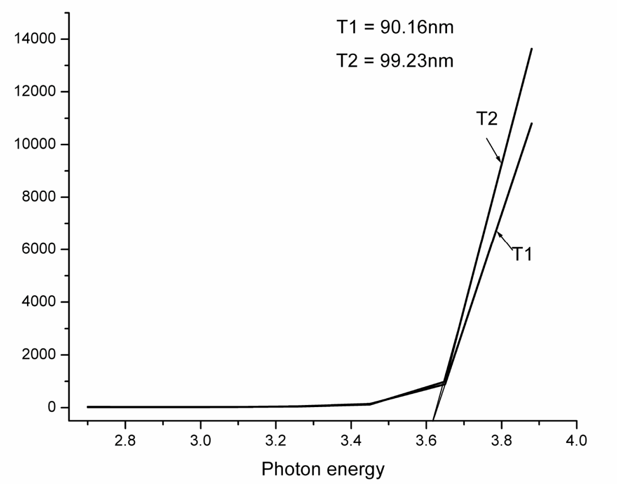

Figure 3. Variation of $(\alpha h\nu)^2$ with photon energy of the films annealed at 500^0 for 1hour.

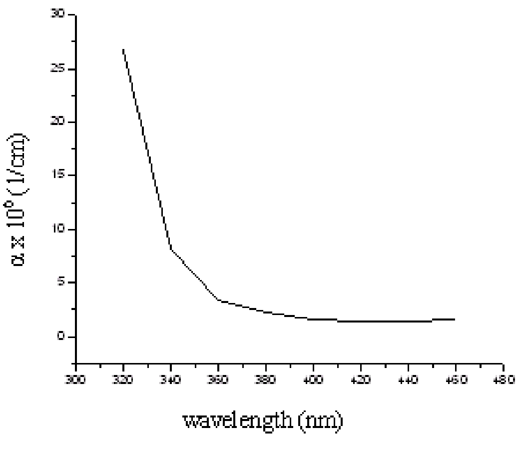

Figure 4. Variation of absorption coefficient with wavelength of the films.

Close to fundamental absorption, transmission characteristics can be converted to the absorption spectra as shown in figure 4, using the relation

$$\alpha = 2.303 t^{-1} \log (T^{-1})$$

Where '**α**' is the absorption coefficient of the film,'t' is the thickness and 'T' is the % transmission [7]. The value of '**α**' increase rapidly in the short wavelength range, this is due to the absorption of light, by the films. For this reason, most semi conducting and conducting oxides are not transparent for the visible light in the blue region.

The extinction coefficient '**k**' in relation to the wavelength [5] was plotted as shown in figure 5. In the visible region, the film samples exhibit low extinction coefficient. This factor is reduced for the annealed film in comparison with the as deposited film indicating the enhanced crystallization of the annealed film [7].

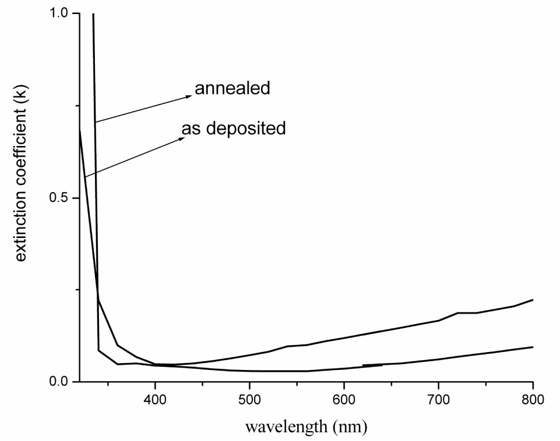

Figure 5. Variation of extinction coefficient with wavelength for the as deposited and annealed films.

Structural studies

The X-ray Diffraction pattern as shown in figure 6 was recorded using an X-ray diffractometer (-Hitachi) using Cu K_α radiation of wavelength λ = 0.15418 nm in the scan range 2θ = 20-80^o. The X-ray Diffraction of film indicates the amorphus state of the as deposited films.

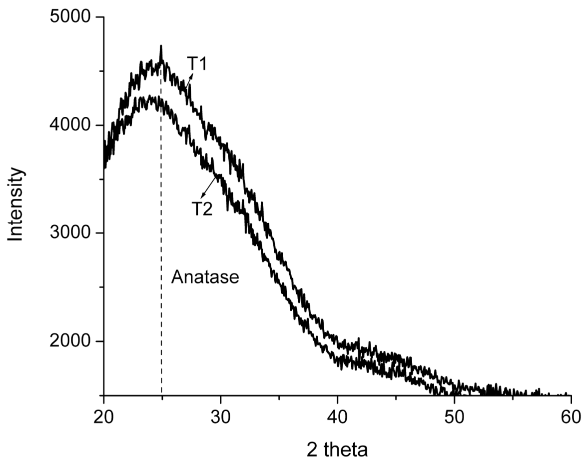

Figure 6. X-ray Diffraction pattern of TiO_2 thin films deposited on glass.

Figure7 shows the Scanning Electron Microscope (SEM) image of Titanium dioxide film prepared using spin coating technique. The SEM image reveals smooth crack free surface with nano grain sized films.

The chemical compositions of Titanium dioxide films were analysed using EDAX spectra as shown in figure 8. The EDAX spectra also show the chemical combination of the glass substrate on which the films were grown [10, 11].

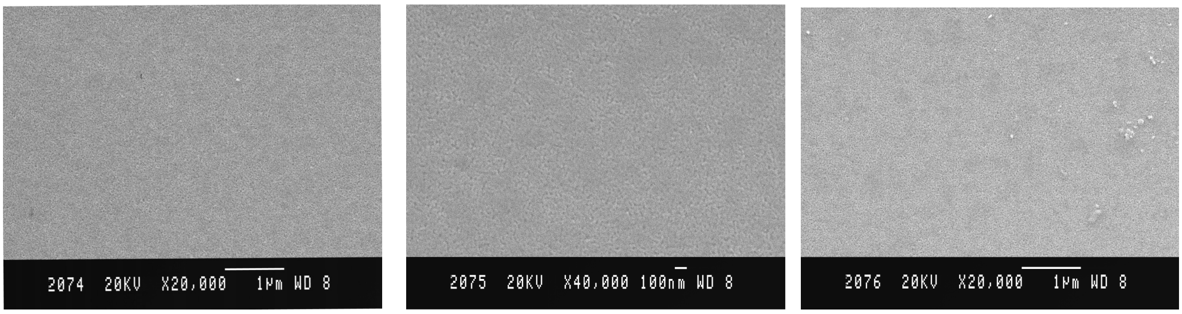

Figure 7. SEM images of TiO2 thin films prepared using spin coating technique

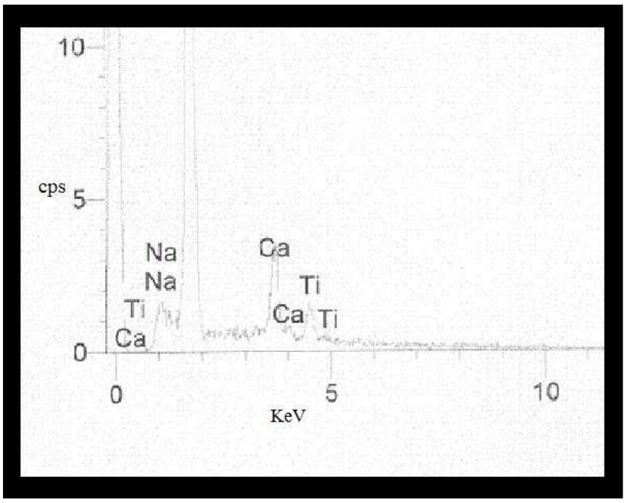

Figure 8. ***EDAX spectra.***

Microhardness

Microhardness measurements are commonly carried out to determine the mechanical strength of the materials. Micro hardness measurements were carried out using Type M hardness tester fitted with a Vicker's diamond pyramidal indenter. The films were indentated at different sites for the load of 25gm to 500gm for 5 second. The micro hardness was calculated using

$$\mathbf{H = 1.8544\ p/\ (d)^2\ Kg/mm^2}$$

Where 'p' is the applied load in gm and 'd' is the average diagonal length of the Vickers impression in mm after unloading. The diagonal lengths were readily calculated from the measurements taken from the main and vernier scale attached to the instrument.

Figure 9 shows the variation of micro hardness H_{mv} with load in grams for as - deposited film. The non linear variation in hardness implies the presence of imperfections (such as impurities, dislocations etc…) and voids. The Mayer's formula gives the relation between the load and the size of the indentation as $\mathbf{p = ad^n}$ Where 'p' is the load in Kg, 'd' is the diameter of the recovered indentation in mm, 'a' and 'n' the constants for given materials. The Mayer index number 'n' gives the value of work hardening index. Nano scale / nano grain sized samples were always characterized by Mayer index or work hardening index. The lower the value of work hardening index, the better will be the hardness of the nano grain sized materials [12, 13].

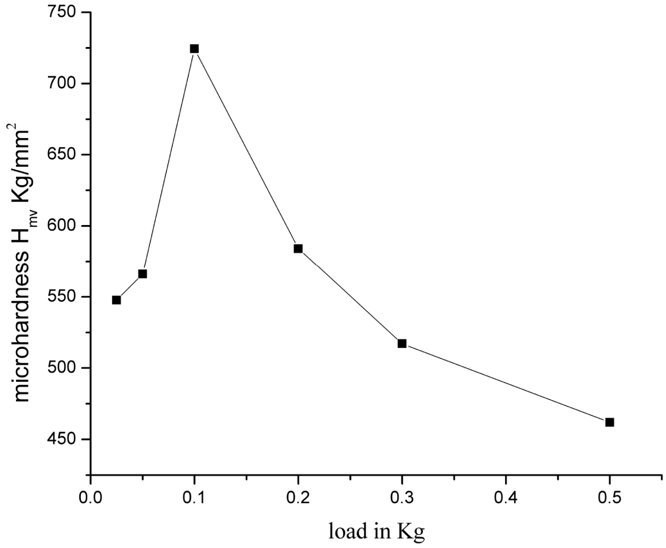

Figure 9. Variation of microhardness of the film with respect to the applied load.

The log-log plot between 'd' and 'p' of figure10 yields almost a straight line graph. The slop determines the value of work hardening index 'n'. For the as deposited titanium dioxide film, 'n' from the graph is found to be 0.53. The lower the value of work hardening index, the better will be the hardness of the nano grain sized material. For hard materials, the value of 'n' range between 1 – 1.6 and is more than 1.6 for soft materials.

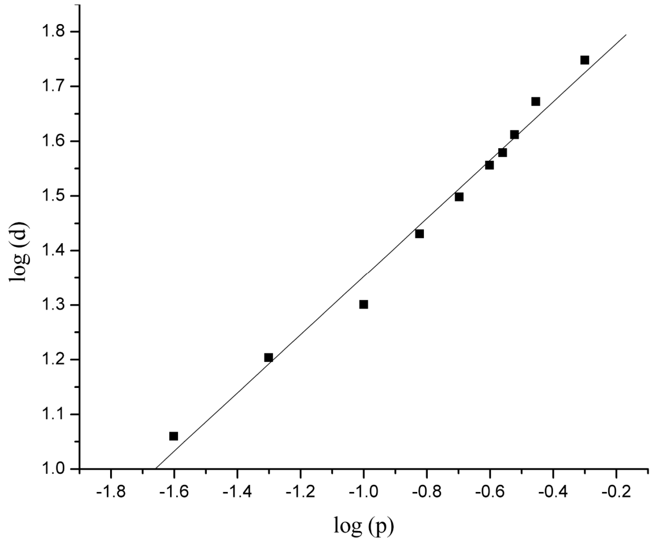

Figure 10. Graph of log 'd' versus log 'p'.

CONCLUSION

Titanium dioxide thin films have been prepared using spin coating technique. X-ray Diffraction patterns have shown formation of non-crystalline as- deposited film. The thicknesses of the annealed films and optical constants (α, k) have been found from the transmission spectrum. The optical band gap energy has been found to of the range 3.61eV. SEM image showed a smooth, crack free surface and EDAX confirmed the composition of titanium dioxide. The micro hardness test showed that the film had a work hardening index number of 0.53 which estimates the hardness of the material.

Acknowledgements
The authors gratefully acknowledge the University Grants Commission (UGC), New Delhi, the management of Mount Carmel College for their constant encouragement and providing necessary facilities to carry out this work.

REFERENCES

[1] Karunagaran B, Rajendra Kumar R T, Viswanathan C, Mangalaraja D, Sa. Narayandass K and Mohan Rao G, **2003** *Cryst. Res. Technol*. **38**, No. 9, 773- 778
[2] Marius D. Stamate, **2000** *Thin solid films* **372** 246 - 249
[3] Narasimha Rao K.; Vishwas M.; Kumar Sharma, Sudhir; Arjuna Gowda, K. V., Edited by Kruschwitz, Jennifer D. T.; Ellison, Michael J.**2008** *Proceedings of the SPIE*, Volume **7067**, pp. 70670F-70670F-8 .
[4] Borse P H.; Kankate, Laxman S.; Dassenoy, Fabrice; Vogel, Walter; Urban, Joachim; Kulkarni, Sulabha K, *Journal of Materials Science: Materials in Electronics*, volume 13, issue 9, 553-559
[5] Hemissi M, Amardjia-Adnani H, 2007 *Digest journal of nanomaterials and biostructures* Vol. **2**, No. 4, December, p. 299-305
[6] Jaroslaw Domaradzki, Agnieszka Borkowska, Danuta Kaczmarek, Eugeniusz L. Procio'w, 2005 *Optica Applicata*, Vol**. XXXV**. No. 3.
[7] Hasan M. M, Haseeb A. s. M. A, Saidur R, and Masjuki H H, **2008** *International journal of chemical and biomolecular engineering* 1;2 © www.waset.org Spring
[8] zhongchun Wang, Ulf Helmersson, Per-Olov Kall, **2002** *Thin solid films* 405 50-54
[9] Lili Hu, Toshinobu Yoko, Hiromotsu Kozuka and Sumio Sakka, **1992** *Thin solid films*, 219 18-23
[10] Yu-Lan Lin, Ting-Jie Wang*)*, **2002** *Yong Jin*, *Elsevier*, *Powder Technology* 123. 194–198
[11] Cătălina ITICESCU, Geta CÂRÂCa, Olga MITOŞERIU and Thomas LAMPKT **2008** *Revue Roumaine de Chimie*, *53*(1), 43–47
[12] Tokuda K, Mitashita K and Ubukata T, ISSP **2003** *The Seventh International Symposium on Sputtering and Plasma Processes*, 96-99
[13] Chih-JenWang, Chi-Yuen Huang, **2008** *Materials Science and Engineering* A 492 306–310