

Composition of TiO₂ Using Sol Gel Approach and Analysis under Different Characterization Methods

Suman Malik¹, Rajesh Kumar Malik², Rajesh Khanna³, Ajay Kumar Jaiswal⁴

¹Student, N.C College of Engineering Israna, Panipat

²Associate Professor, N.C College of Engineering Israna, Panipat

³Professor, Thapar University Patiala

⁴Lect, N.C College of Engineering Israna, Panipat

Abstract

This paper is presented to perform the extensive study and experimentation of TiO₂. The experimentation is performed for the composition of the TiO₂ solution under different cycles and the temperature values. The experimentation of the work is performed using sol gel approach. Later on the characterization on the solution is performed using XRD pattern. The analysis of the work is performed at different temperature and the pressure values.

Keywords: TiO₂, Sol-Gel, XRD, Characterization

I. Introduction

TiO₂ is one of the most constituent naturally available mineral that is obtained from the metal oxides by performing the transformation over it. TiO₂ provides the solution on many environmental problems so that the treatment and purification of water and air protection of the environment. The application area of TiO₂ includes the preparation of catalysts so that the film coating to overcome the disadvantages of industrial applications. Lot of work is defined in the TiO₂ thin coating under the activity analysis. The characterization of TiO₂ is defined under the surface hydroxyl content, film thickness and microstructure so that the activity of TiO₂ films.

TiO₂ has been used in different forms under the off-white analysis that depends on the physical characterization in the form of affecting light. TiO₂ is taken as the nano scale analysis under the dimensional constraints and also recognized as the valuable material under the white pigment in different materials such as in textile, rubber, plastic etc. The material based properties of the TiO₂ are analyzed under different field of anti-reflection coating in the thin film coating of optical devices. The effective properties of the TiO₂ includes the insolubility in water, hydrochloric acid, dilute sulfuric acid and the organic solvents. The solvents includes the hydrofluoric acid and the sulfuric acid. It is very important material for the solar cell for hydrogen and electric energy production, used as the photocatalyst in different applications.

Sol-gel is the effective processor to make the glass and ceramic material. In includes, the sol-gel process under the transition for liquid sol in the gel phase. It also include the work for different states

like solid as well as liquid under the material properties. It is defined as the chemical process at low temperature so that the inorganic oxide production will be performed. The process is defined under multiple component oxide and the crystalline amorphous form. Sol-gel also provide the substrate deposition at low as well as high temperatures. It is been used as the generation of fabricate ceramic in differ shapes and forms.

The presented work is the characterization and deposition of TiO₂ using sol-gel approach. The works include the experiment based analysis under different doping and characterization approaches. In this section, the basic review of the TiO₂ its characteristics is defined. A short note on the sol-gel approach is also defined in this section. In section II, the work done by the earlier researchers using sol-gel approach and the outcomes from the experimentation is discussed. In section III, the experiment is defined under the characterization approaches using sol-gel. In section IV, the results obtained from the work are discussed. In section V, the conclusion obtained from the work is presented.

II. TiO₂ using Sol-Gel

The study of deposition and characterization of titanium dioxide is done by different authors using different approaches as well by under different annealing parameters. Some of the work done by earlier authors is discussed in this section. In year 2011, S.L. Lin has defined the analysis on the effect of sol-gel approach for the transformation of titanium dioxide under CO₂ and the laser annealing. Author presented the sol-gel solution as the mixture of tetraisopropyl orthotitanate(TTIP), acetylacetone,

distilled water and alcohol at different molar ratio and coating for crystallization of titanium oxide. Author defined the phase and microstructure transformation of titanium dioxide under the X-ray diffraction pattern. The sol-gel approach is combined with CO₂ to obtain the cost optimization and phase control in room temperature [1]. Another work on the fabrication and property of titanium dioxide using sol-gel and CO₂ laser annealing by C.K.Chung in year 2012. Author used the CO₂ based sol-gel approach to analyze the phase transformation and the hydrophilic property analysis of TiO₂ thin film. Author defined the XRD pattern to analyze and control the sol-gel solution. Author performed the transformation at high temperature of 350°C and 800°C. Author provided the fabrication of hydrophilic and wide PL TO₂ to under the metric based analysis [2]. In year 2013, Valentina Smirnova has defined a water purification process using Titanium Dioxide. Author presented the work on drinking water analysis under heavy metal soluble. Author defined the synthesized by chemical and electrochemical method. Author defined the material crystalline phases to obtain the amorphous structure stabilization. Author presented the study under water resistance so that the titanium dioxide and generate after purification [3].

In Year 2012, Ella Gale has defined a filamentary extension under the application of Titanium Dioxide using sol-gel approach. Author presented the work under two operational modes so that the bulk memristance for the whole volume analysis. Author defined the filamentary analysis under the conducting filament analysis. Another work on the electrons conductance was defined by author for different devices. Author defined the filament analysis at low resistance titanium dioxide and presented the comparative experimental results for filamentary memristance[4]. Another work on the deposition behaviour and the analysis of Titanium Dioxide was performance by Fitrah Rabani in year 2013. Author presented the study during the electrophoretic deposition. The main research was defined on the deposition behaviour of Titanium dioxide so that the nanoparticles during electrophoretic deposition will be performed. Author presented the research on the effect of pH for the deposition behaviour of Titanium Dioxide using electrophoretic deposition. Author presented the study under different behaviours of nanoparticles[5]. In same year, Fallah Schojaie has defined the experimental evaluation on the dielectric breakdown of voltage under the transformer mixed with Titanium dioxide for nanoparticles. Author measure the breakdownvoltage of transformer under the oil-based nanofluids with IEC standard. The results shows the strength and surface composition of TiO₂

under the transformer oil to enhance the dielectric strength of the surface composition [6]. Another work in same year was proposed by Valeri Mladenov for the synthesis of PSPICE model of TiO₂. Author presented the equivalent circuit deposition under the current voltage relationship [7]. A work on the TiO₂ under different coating techniques with SiO₂ layer was proposed by M.Rezaei. Author presented the work under the soda lime glass preating with the spin coating approach so that the ferric nitrate calcinations will be characterized [8]. Yongxiang Li presented a sol-gel approach work on TiO₂ for photovoltaic cell derivation. Author defined the characterization TiO₂ under spin coding and blading techniques. Author analyzes the cell performance analysis under the structure formation and the heating process analysis[9]. Author defined the structural and morphological study of TiO₂ for Supekar A.K. in year 2013. Author defined the prepared film under the pre-annealing and post-annealing. Author presented the structural property analysis optical property analysis of Titanium Oxide films[10]. S.Kment has defined the characterization of transition of metal-doped and undoped of TiO₂. Author presented the behavioural analysis under the photocatalytic and photo-induced electrochemical properties[11]. E. Haimi has defined the structural and optical property based analysis on Titanium dioxide thin film by using sol-gel dip coating. Author defined the optical characteristics analysis for reactive aqueous sol-gel process [12].

III. Experimental

In this section, the experimentation about the deposition and the characterization of TiO₂ films is defined under sol-gel method. To prepare the TiO₂ film a mixture that includes 17.02ml of Tetrabutylorthotitanate and 4.8ml of diethanolamine were dissolved in 67.28ml ethanol. This solution is then stirred under the room temperature for 2 hours. Later in this solution, .9ml of water and 10ml of ethanol is included in continuous drops under the stirring process. The obtained solution from the work is then placed at room temperature for 2 hours to identify analyze the hydrolysis reaction. This solution performed the chemical composition in defined Ti:C₂H₅:OH:H₂O:NH at ratio 1:26:5:1:1. This substrate of thin film is also taken for the soda-lime glass plates. The coating on this thin film substrate is then performed under the dipping ambient atmosphere. This coated substrate with gel films were heated at different temperature value. The adjustment to the thickness is performed with each cycle of applied heat.

A) Characterization Process

Once the Titanium Dioxide thin film solution is obtained. The characterization on the TiO₂ thin film is performed under XRD. The radiation analysis on the crystallization of thin film was performed under the Cu radiation analysis. The cauterization is performed at different voltage and the current value with acceleration. The crystallization behavior is later analyzed under the behavior of the machine as well as the physical characteristics of the TiO₂ is been observed. The observation is performed using the electron microscopy with 20kV acceleration voltage. TiO₂ film is also performed under the spectrophotometer values at different wavelength. This analysis is performed using TiO₂ films by XRD in ultrahigh vacuum chambers. The pressure analysis is also performed in these chapter at high energy constraint and different Xray sources. The high resolution based survey spectra is applied to perform the analysis. The X-ray photoelectrons were referenced so that the adventitious hydrocarbon is obtained as the sample surface.

The measurement analysis is the final stage of the TiO₂ film is performed under the temperature and the pressure constraint. The high pressure mercury lamp can be used as the major light source. The TiO₂ face is placed under the irradiated along the direction. The intensity analysis is performed under the irradiance analysis with different measuring values. The peak value analysis for the wavelength is also performed on the TiO₂ solution. During this process, the pressure is applied with air. The photo catalytic decolorization of methyl is defined as the reaction process and the kinetic process.

IV. Results and Discussion

In this section, the results of the TiO₂ characterization under the XRD pattern and the sol-gel process. The XRD patterns of the synthesized in different conditions. It shows the FTO substrate under the solution TiO₂. The diffraction is performed on the samples at XRD patterns. The analysis is performed for different samples. TiO₂ film is prepared by at the high temperature under the dipping, hitting and the absorption process. The wavelength range analysis is performed at TiO₂ films for 5 and 10 cycles. This process is kept under the short term heat treatment and then the crystallization is performed relatively. The transmittance of the TiO₂ is decreased with each cycle.

The DTA curve of the TiO₂ gel is shown in figure 1. Here the peak temperature value start at temperature 150C and analysis is performed with increasing temperature with each cycle. The relation between the thickness and the coating cycle is shown in this figure.

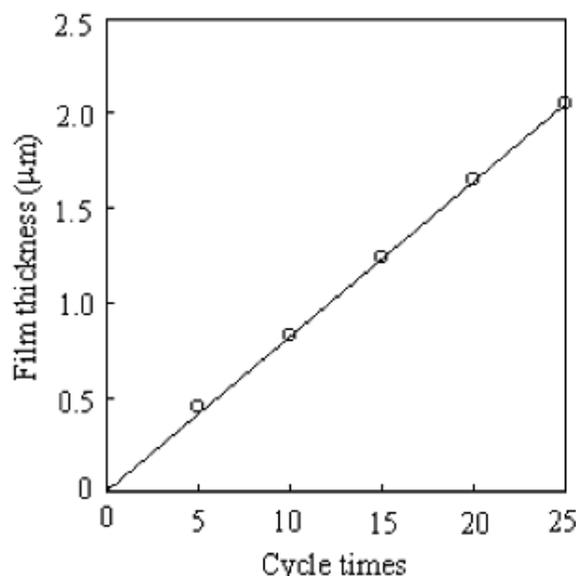


Figure 1 : Doping Cycle Vs. Thickness

The figure shows that as the thickness of the material is increased, the number of required cycle also increased. XRD patterns of the TiO₂ gel films are coated on the

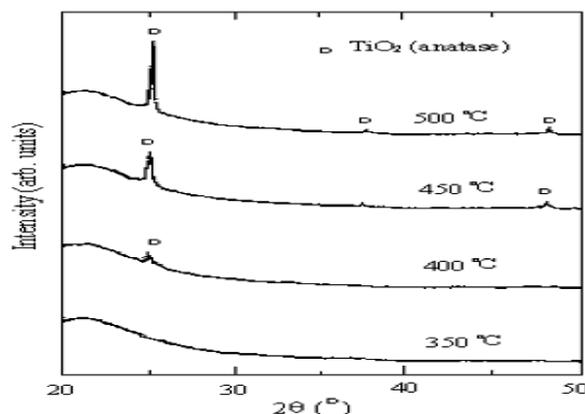


Figure 2 : XRD Intensity Analysis at different Temperature

Soda-lime glass substrates by performing the coating cycles and the apply the heat treatment at different temperature values for 1 hour. The XRD patterns intensity at different temperature values is shown in figure 2.

The UV-VIS spectra of Titanium Dioxide films were prepared and analysis is performed for different coating cycles. The consideration is taken for 1,5 and 10 coating cycles for the different wavelength that varies between 300 and 800nm. Here figure 3 is showing the results for the process for different cycles. Here (a) represents the results for 1 coating cycle at 500C for 1 hour. Here (b) represents the results of dipping and heating process for 5 cycles and (c) is showing the results for 10cycles.

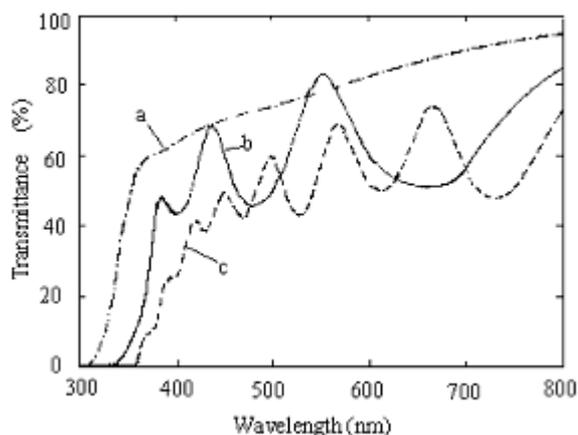


Figure 3 : %T analysis at different wavelength

The SEM photograph of the surface of TiO₂ for 10 coating cycles at 500C is shown in figure 4. The TiO₂ solution is placed at this temperature for 1 hr. The obtained results shows the granular microstructure of the particles.

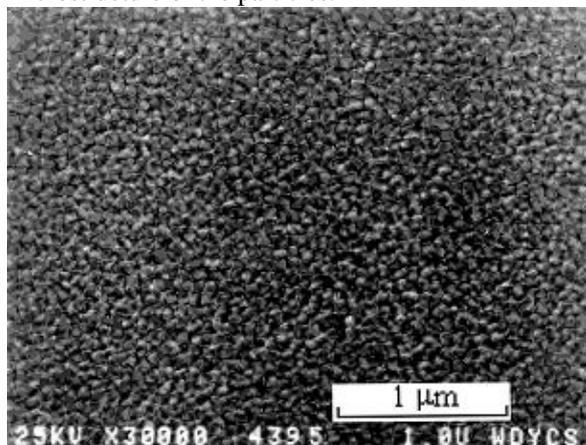
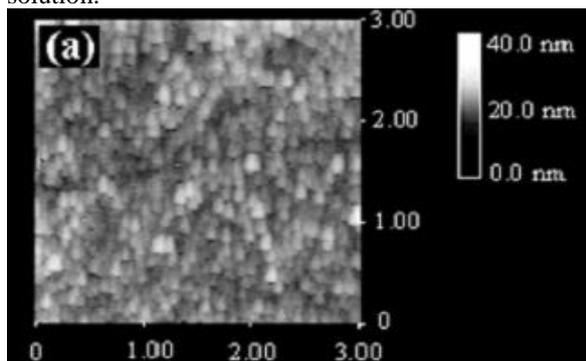
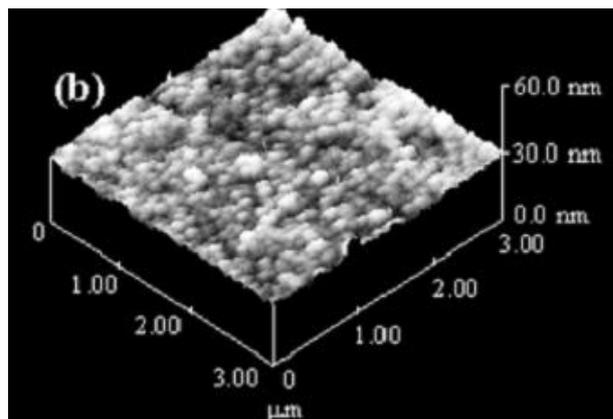


Figure 4 : SEM results of TiO₂ for 10 coating cycles

The band-gap based analysis was performed to derive the absorption coefficient. The AFM(Atomic Force Microscopy) image in 2D and 3D form is shown in figure 3. These surface images are shown for undoped TiO₂. Figure is showing the roughness of the surface prepared from precursor solution.



5(a) : AFM 2D Image for undoped TiO₂



V. Conclusion

This paper is presenting the experimentation of the TiO₂ using the sol gel approach. The work includes the solution generation at different process cycles as well as different temperature values. The work is also performed for different XRD pattern based characterization.

ACKNOWLEDGMENTS

Authors are grateful to the AICTE, New Delhi for providing financial support under RPS Ref No. 8023/RID/RPS/78/11/12, N.C.C.E. (Israna) ECE department to carry out this work. Thanks are also due to the AIRF, JNU Delhi, SAI lab, Thapar University, Patiala for providing XRD and SEM facilities.

REFERENCES

- [1] S.L. Lin," The effect of sol-gel composition ratio on phase transformation of titanium dioxide under CO₂ laser annealing", Proceedings of the 2011 6th IEEE International Conference on Nano/Micro Engineered and Molecular Systems 978-1-61284-777-1/11 ©2011 IEEE
- [2] C.-K. Chung," Fabrication and property of titanium dioxide thin film using sol-gel method and CO₂ laser annealing", The Institution of Engineering and Technology 2012
- [3] Valentina Smirnova," The Use of Titanium Dioxide in the Process of Water Purification", 978-1-4673-1773-3/12©2013 IEEE
- [4] Ella Gale," Filamentary Extension of the Mem-Con Theory of Memristance and its Application to Titanium Dioxide Sol-Gel Memristors", 2012 IEEE International Conference on Electronics Design, Systems and Applications (ICEDSA) 978-1-4673-2163-1/12 ©2012 IEEE

- [5] Fitrah Rabani A. A., " Deposition Behaviour of Titanium Dioxide (TiO₂) Nanoparticles During Electrophoretic Deposition (EPD) Technique Effects of pH", 2013 IEEE Business Engineering and Industrial Applications Colloquium (BE IAC) 978-1-4673-5968-9/13 ©2013 IEEE
- [6] A Fallah-Shojaie, " Experimental Evaluation on the dielectric breakdown voltage of fresh and used Transformer Oil mixed with Titanium dioxide nanoparticles in the Gilan Electrical distribution company", 978-1-4673-5634-3/13 ©2013 IEEE
- [7] Valeri Mladenov, " Syntheses of a PSPICE Model of a Titanium-dioxide Memristor and Wien Memristor generator".
- [8] M. Rezaei Nasher, " Preparation and antifog behavior of Fe³⁺ -doped TiO₂ nanometer thin films on soda – lime glass pre-coated with a SiO₂ layer", Proceedings of the 4th International Conference on Nanostructures (ICNS4) 12-14 March, 2012
- [9] Yongxiang Li, " Titanium dioxide Plms for photovoltaic cells derived from a sol-gel process".
- [10] Supekar A.K., " Optical, Structural and Morphological Study of TiO₂ Thin film using Sol-gel Spin Coating Techniques", IOSR Journal of Engineering (IOSRJEN) e-ISSN: 2250-3021, p-ISSN: 2278-8719
- [11] S. Kment, " Notes on the photo-induced characteristics of transition metal-doped and undoped titanium dioxide thin films", Journal of Colloid and Interface Science 348 (2010) 198–205
- [12] E. Haimi, " Optical and structural properties of nanocrystalline anatase (TiO₂) thin films prepared by non-aqueous sol-gel dip-coating", Thin Solid Films 519 (2011) 5882–5886