Preparation of Titania Thin Films Using Sol-Gel Techniques

Abd Al-Abass Abd Al-Ameer Shakir Al-Hamadani, Khelfa Fadelshedhan Muselmawe

Abstract: ***Sol-gel technique have been used to prepare the anastane titania thin films and it is deposited on glass substrates. Under vigorous stirring, substrates are dipped into the solution. The films were dried at atmospheric temperature. Oley amine (OM) was used as a chemical agent within the synthesis part and it had been aggressively stirred with titanium tetraisopropoxide (TTIP). Samples of thin films are annealed under 550^{0}C for 15 hours. To study the properties of structure, X-Ray diffraction (XRD) analysis and Raman Spectroscopy has been taken place. Also by using Ultraviolet (UV) Spectroscopy, optical properties are analyzed for the samples. XRD results illustrated the amorphous structure and section of TiO2 and these results were additional confirmed by Raman spectrum analysis. Then ultraviolet light spectrographic analysis has been used for preparation of TiO2 lean films in association with the energy band three 3.26 and 3.22 eV, it is easy refers to make different in crystal structure for anatase titania. In addition, each samples have high transmission.***

Index Terms: Optical properties, Raman spectroscopy, Sol-Gel technique, Titania Nanoparticle, X-Ray diffraction design.

I. INTRODUCTION

The course of action for nanoparticles which has been done by different techniques, analysis of different procedure and its conditions strikingly impacts the nanoparticles properties. The features are listed as time taken and speed of reaction, rise of the temperature, predecessor, chemical reaction and composition, natural relationship of chemical reactions, transformation of phase, methods of preparation, and treatment of post heat for nanoparticles which impact on its state and size. Nanoparticles rely on all the above parameters by its determination and control. For instance, such parameters are accounted for proper treatment which performs better functionality [1]-[9]. For most recent two decades titanium dioxide standout as most often utilized semiconductor oxides which is an expensive material utilized in various applications of photovoltaic [1]-[14]. Because of it immense dielectric constant, Titania is utilized as dielectric gate which helps formation of field effect transistors [11]-[13]. Also, titania films have astounding properties, for instance, incredible strength, thermal stability is high and stability of chemical reaction is in the visible range [1]-[10], [15]-[18]

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In many applications, Titania has been inconceivably utilized [1]-[9]. Because of its potential applications, making of Titania has become very essential. Assortment of techniques are listed as sputtering, chemical vapors deposition, electron beam evaporation, spray paralysis, hydrothermal, sol-gel and dip-coating have been utilized for the preparation of titania films [17]. The places of titanium and oxygen components in the crystal structure of TiO2 shows three unique stages, such as anatase, rutile and brookite [1]-[11]. The chemical, electrical and physical properties at these stages make it efficient for few applications. The rutile and anatase stages have been analyzed in contrast with most parts of the brookite stage to define the difficulties encountered during the production of pure brookite stage. Normally, the brookite stages can be found at temperature lesser than 3500C and it is converted to anatase stage during calcination at 7000C, above this range it is changed to rutile stage. The main characteristics of anatase stages are high surface area, high electron mobility, low density and low dielectric constant. Because of these characteristics, it is widely used in several applications including solar cells and batteries [1]-[9].

The titania nanoparticle morphology was uniquely utilizing different concentration of titanium n-butoxide as a foreunner, numerous surfactant fixation, certain reaction temperature, ethanol and water as a solvants [2]. Other examination revealed different morphologies of titania nanocrystals utilizing surfactant, titanium tetrafluoride (TiF4) and titanium tetrachloride (TiCl4) as the titanium predecessors. Tetragonal bipyramidal anatase nano composite with TiF4, brookite nanorods and anatase nanoplate with TiCl4 were discovered as forerunners [6]. Also, the morphology of titania Nanoparticles utilizing nanoaqueous method with TiCl4 was identified as the predecessor. They showed dissimilar morphologies, for instance, nanorods, nanoparticles, nanowire and 3D Wulff structures for different concentration of forerunner and varying response time [4]. Because of its sensitivity issues due to humidity, under inert gas conditions drawback is present in TiCl4. For the preparation of titanium dioxide nanoparticles various techniques has been proposed such as solvo thermal methodology, sol-gel methodology chemical vapour deposition technique, electron beam evaporation method, hydrothermal methodology, pulsed laser technique [10], [12]-[14].

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Here, the sol-gel coating course was utilized because of its minimum cost, simple handing, control over stoichiometry and size of the crystallite, phase crystalline, chemical composition, morphology particle, temperature range of reaction, and time taken for ageing. It is low power utilization strategy which likewise gives better virtue, homogeneity, felicity and has capacity for covering the huge and complex zones [1], [15]. This experimentation intended to access the synthesis of nanostructured TiO2 thin films kept onto glass substrate with the help of sol-gel dip coating technique processed under room temperature and its properties are analyzed.

II. EXPERIMENT MATERIALS AND PREPARATION

Titanium isopropoxide (TIP, C12H28O4Ti), Oley amine (C18H37N), Acetone, C3H6O, Methanol, CH4O were utilized for the course of action taken to TiO2 thin films preparation. Titanium isopropoxide (TIP) was incrementally added to a mix of 20ml methanol and Oleyamine (OM). The molar proportion of Methanol/TIP/OM was looked with care for 9/1/0.1. the glass substrates were washed by consistent washing (acetone, methanol, distilled water). The solution was vigorously mixed and kept onto the glass substrates at 8000 rpm for 30s. In this way, the resultant films were toughened at 5500C for 15 hours [1], [12]. Separate verification at different periods of titania thin films was performed by Raman Spectroscopy and XRD. Prepared samples are analyzed in terms of optical highlight with the help of UV spectroscopy.

III. RESULTS & DISCUSSION

A. Design of X – Ray Diffraction

XRD design has been utilized for the stage recognition of deposit and strengthened at 550^0C nanocrystalline TiO_2 thin films onto glass substrate. Diffraction design of prepared films don't demonstrate any peak value because of low crystallinity which demonstrate that the film have amorphous nature shown in Fig. 1a and 1b. The peak values situated at 2μ of about 25.1, 48.3, 63.1 and 70.1 related to the Miller records of (101), (200), (204), and (116) planes exclusively. These peaks show the arrangement of pure anatase stage for the post toughened film at 550^0C shown in Fig. 1a and 1b. The nucleation and development of grains and increment in crystallinity are clearly looked for these films [16]. The consequences of XRD designs are correspond to the standard information of anatase period of TiO_2. Scherrer condition has been utilized to compute the crystallite size of titania thin films and results indicated 18-22nm particles sizes. Equation (1) gives the detailed calculation of average crystallite size.

$$D = 0.9\lambda/\beta\cos\theta \qquad (1)$$

Where, D - average crystallite size,

λ - X-ray radiation wavelength

β - full width at half-maximum height

θ - Bragg diffraction angle [8]

After the heat treatment, the size of the particle get incremented it could be attributable to the agglomeration and recrystallization of titania films at higher temperature [1], [8].

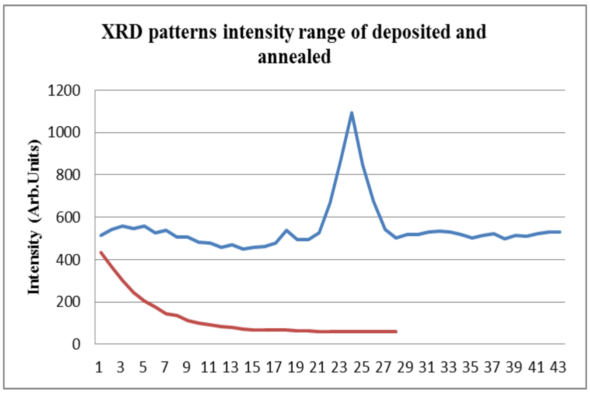

Fig. 1a. XRD patterns intensity range of deposited and annealed process

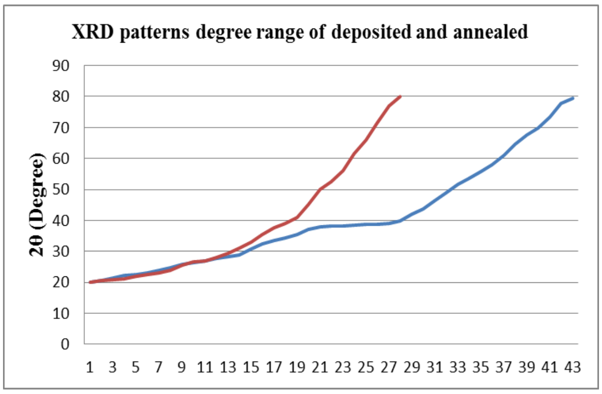

Fig. 1a. XRD patterns range of degree for deposited and annealed process.

B. Raman Spectroscopy

The aftereffects of XRD were affirmed utilizing Raman Spectroscopy. Fig. 2a and 2b demonstrate the Raman spectra recorded at room temperature with excitation wavelength 532 nm as the source in the scope of 100-800 cm -1 on a prepared and toughen titania thin films at 5500C. Fig. 2a and 2b demonstrates no peak, recommending the indistinct nature of prepared film. As it is outlined in the Fig., three powerful modes at 144, 458 and 561 (cm-1) were recognized as anatase period in terms of titania for tests at 5500C. The area of bands is in great concurrence with past reports for anatase stage and no rutile follows were seen in the examples. Subsequently, the consequences of XRD are complimented by the study of Raman spectroscopy which demonstrated the typical vibrational modes for anatase TiO2 [1].

C. Optical Properties

The absorption edges are seen to be 380 and 385 nm for as deposited and annealed samples at 550^0C respectively.

There is a red shift in the absorption wavelength which is related to the development in particle size after heat treatment [1], [8]. The energy band gap (E_g) of the films was evaluated from the immediate change of electrons from the highest point of the valance band to the base of the conduction band. The estimated energy band gap was 3.26eV for as deposited sample, from that point on lessened to 3.22eV for 550^0C annealed TiO_2 fims. The reduction in the energy band gap after expanding the calcination temperature could be because of increment in particle size and phase transformation from amorphous to anatase [1], [12]. The transmittance (%) spectra of the as deposited and toughened films at 550^0C were around 86 and 77 % respectively. The adjustment in transmitted is because of the expansion in surface scattering which is related to the surface harshness [1], [8]. The absorption coefficients (α) of the films were evaluated from the transmission spectra (T) in the noticeable region utilizing the accompanying experimental articulation [16], [18].

$$\alpha = 1/t \ln (1/T) \qquad (2)$$

Where, T - maximum transmittance and
t – film thickness

The estimated absorption coefficients at a wavelength of 500nm were 96.7 * 106 m^{-1} and 94.6 * 106 m^{-1} for as deposited and annealed titania thin film at 550^0C which conspicuously show a reduction trend after heat treatment.

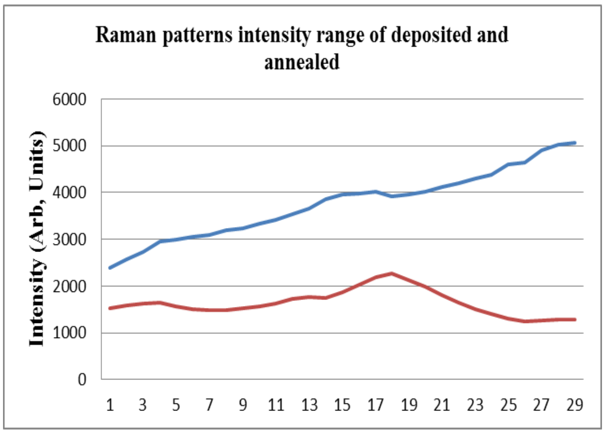

Fig. 2a. Raman patterns intensity range of deposited and annealed process.

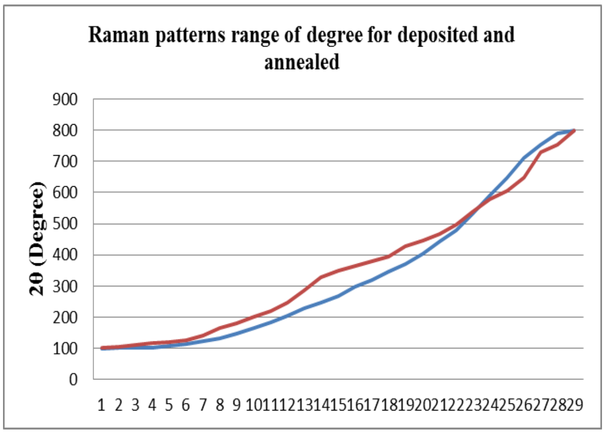

Fig. 2b. Raman patterns range of degrees for deposited and annealed process.

Incident photon energy (hv) and absorption coefficient (α) relationship is given by

$$\alpha hv = A(hv - E_g)^n \qquad (3)$$

Where, A – Value of constant,
h – Constant of Planck's
v – Incident radiation frequency
E_g – Material band gap and
n = 1, for direct gap and
n = 4, for indirect allowed transition.

By using above relation, optical energy band gap (E_g) has been calculated. The same values as obtained using the relation (2) have been achieved by applying expression (3). The band gap values have been also evaluated [17], [18].

IV. CONCLUSION

Here we have effectively utilized sol-gel coating technique and designed nanostructured titania films. The properties of TiO_2 films were inspected during the impact of treatment of post heat. Aftereffects of XRD affirmed that the indistinct nature for as synthesized and anatase stage for sintered sample at 550^0C. These outcomes were additionally affirmed that by using Raman spectroscopy. The properties of TiO_2 thin films are impacted by the process of post annealing. Crystallite size was extended and the optical examination uncovered the edge of absorption and decrease in the essential band gap and transmission spectra after heat treatment. The past was credited with adjustment in titania thin films crystal structures and the later was attributable to either change in transformation of phase and size of the particle.

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