

Characterization of TiO₂ Thin Films Prepared By SOL–GEL Technique

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Abstract: *Stable TiO₂ sols were prepared using sol–gel processes with titanium IV isopropoxide and Isopropyl alcohol respectively. TiO₂ thin films were synthesized by chemical bath deposition method. Crystallization and phase transitions of thin films were studied by X-ray diffraction. The thin films obtained were homogeneous and transparent in the visible range. The effect of the changing the dipping time was absorbed.*

Keywords: TiO₂, Thin film, sol–gel, CBD.

1. Introduction

Among the inorganic oxides, TiO₂ is a well known material due to its broad application area. Crystalline TiO₂ exists in three phases; brookite (orthorhombic), anatase (tetragonal with $c/a \approx 1$) and rutile (tetragonal with $c/a \approx 1$). TiO₂ is transparent in the visible range and has a wide optical band gap. This, together with its chemical and thermal stability, makes TiO₂ an excellent candidate for optical coatings [1]. Titanium dioxide is also utilized for gas sensing[2]. TiO₂ coatings with high photoactivity [3] are attractive for industrial applications such as water and air purification by photocatalysis, self-cleaning or anti-fogging materials (due to its superhydrophobicity) and solar energy conversion[4],[5]. Although for most of these applications anatase is favoured, rutile TiO₂ is commonly used in the paint industry as a white pigment in paints, as it scatters light more efficiently, is more stable and more durable than anatase pigments[6]. In order to prepare TiO₂ fine powders and films, a wide range of techniques have been used such as hydrothermal methods[7],[8], electron beam evaporation[9],[10], DC and RF magnetron sputtering[11], solvothermal synthesis and sol–gel methods[1],[2],[12].

Among these techniques the sol–gel method offers several advantages: fine powders and films with high homogeneity can be prepared using simple equipment, the thickness and the porosity of the films can be easily controlled by changing the concentration of the sol and large surface areas can be coated by spin or dip coating.

When precursors with strong reactivity towards water (like titanium alkoxides) are used, the hydrolysis reaction can result in precipitation. The problem can be overcome using non-aqueous sol–gel techniques with titanium chloride[1],[13] or titanium alkoxides[14],[15] as a precursor. In case of the reactions using TiCl₄ as a precursor, TiO₂ forms under release of a large amount of HCl gas. Because HCl gas is toxic and chlorine impurities often remain in the final oxide material[16], we preferred titanium isopropoxide as precursor.

In order to increase the stability of the solutions from titanium alkoxide precursors, it is not sufficient to switch to a

non-aqueous sol–gel route. Indeed a chelating agent is still needed in a non-aqueous sol–gel route to obtain highly stable sols [12], [14], [15].

In this study we make an investigation of sol–gel technique which was found in literature. Crystallization and phase transformation for both types of samples are determined on thin films. The films prepared can be used as optical coating. The influences of the viscosity, substrate type, spin speed and heating temperature on film properties can also be sought.

2. Experimental

Titanium dioxide thin films were prepared using sol–gel techniques. sols were synthesized using titanium IV isopropoxide (Ti(OCH(CH₃)₂)₄ (sigma-aldrich co., 97%) as precursor and isopropylalcohol [1] (CH₃)₂CHOH(Nice chemicals) as solvent. In order to begin hydrolysis via an esterification reaction, glacial acetic acid (Alfa Aesar, 99%) was used. To prepare a stable precursor solution, first (Ti(OCH(CH₃)₂)₄ and (CH₃)₂CHOH were mixed. The molar ratio between (Ti(OCH(CH₃)₂)₄ and (CH₃)₂CHOH was 1:3. This mixture was stirred for 30 min at room temperature. Acetic acid was slowly added into the alkoxide solution under stirring for 30 min at room temperature. A molar ratio between acetic acid and (Ti(OCH(CH₃)₂)₄ of 1:3 was utilized. A white precipitate will be formed. Finally Methanol (1:8) was added to get a moonstone color precipitate. Let this precipitate settle for 60 min.

A SLG was dipped inside this sol and remain for 30 min. and one more SLG for 60 min. Finally both the SLG's are annealed at 500 degree celcius.

X-ray diffraction (XRD, Bruker D8-Discovery) measurements were employed to acquire information about the crystal structure of the coated layers. Crystallization kinetics were studied in detail via in situ X-ray diffraction (XRD) (Bruker D8-Discovery with Vantec linear Detector and homemade heating stage). The phase transition of the samples was followed on the basis of dipping time of 30 min and 60 min respectively. The obtained X-ray diffraction

patterns were compared with the anatase (ICDD file No. 21-1272) and rutile (ICDD file No. 21-1276) reference patterns.

3. Crystallization and Phase Transition of Thin Films

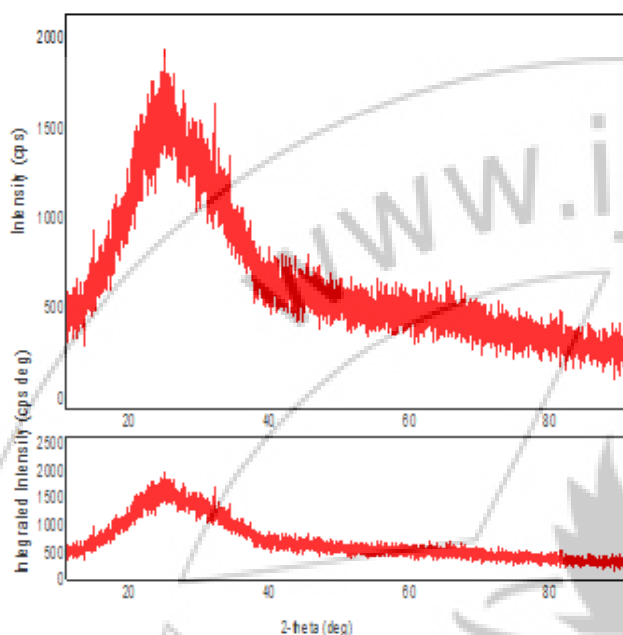


Figure 1: shows XRD spectra of TiO₂ thin films Prepared By sol gel method after CBD of 30 min , annealed at 320 to 500 °C. in hot air oven.. The XRD spectrum of the film indicates that there is formation of anatase TiO₂ nanoparticles.

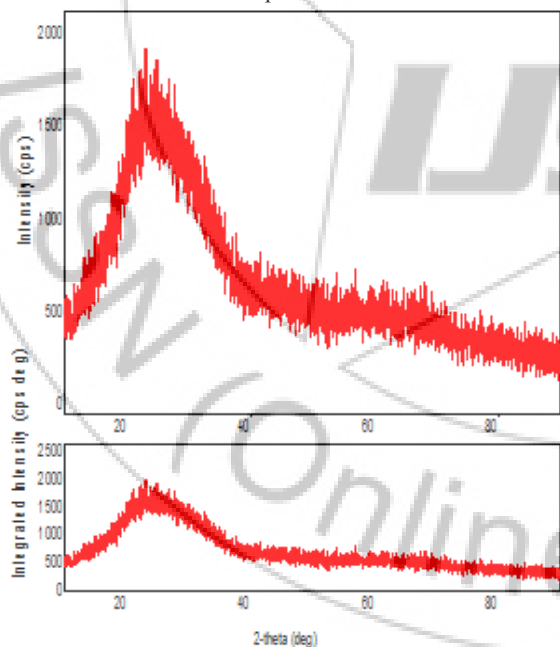


Figure 2: shows XRD spectra of TiO₂ thin films prepared By sol gel method after CBD of 60 min , annealed at 320 to 500 °C. in hot air oven.. The XRD spectrum of the film indicates that there is formation of anatase TiO₂ nanoparticles.

When the XRD results of thin films are compared, it is clear that the anatase phase remains stable to higher timings in thin films. Thus the thin films remain amorphous in nature.

4. Conclusion

In the present work, TiO₂ bulk powder as well as TiO₂ thin films on SLG were obtained using sol-gel technique. The films were homogeneous and their thickness was less than 250nm by CBD method. By changing the dipping time, films with several thicknesses and refractive indices were obtained. The crystallization of the films was similar on both the films and amorphous Coating in glass substrates. TiO₂ thin films synthesized by sol-gel techniques may be utilized as a protection layer for moisture sensitive particles such as sulfides.

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Author Profile

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