Synthesis of Chemically Deposited TiO_2 Thin Film and its Characterization

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Abstract

Chemical bath deposition method (CBD) was used to successfully produce TiO_2 (Titanium dioxide) thin films at ambient temperature. The obtained sample was annealed and analysed using X-ray diffraction, SEM, EDS, UV-Vis spectroscopy, and photoluminescence to evaluate its structural, optical, morphological, and compositional properties (PL). In the prepared sample, XRD has shown the presence of a pure rutile phase. The nanorod-like morphology was visualised using a scanning electron microscope, and the presence of Ti and O atoms was confirmed using an EDS micrograph. The optical study revealed that the observed band gap of TiO_2 nanoparticle is 3.1eV.

Keywords: XRD, SEM, EDS, UV-Vis etc.

Introduction

Nano titanium dioxide powder has many good functions and features, such as stable properties, non-toxic, low cost and good at resisting chemical attack. It is also a nice photocatalyst, disinfector and antiseptic. Thus, the preparation and features of nanometer titanium dioxide powder has naturally been a research hotspot for a long time [1-6]. Titania (TiO_2) powder possess interesting optical, dielectric, and catalytic properties, which leads to industrial applications such as pigments, fillers, catalyst supports, and photo-catalysts [7–11]. It has been demonstrated that the final properties of this material depend to size, morphology and crystalline phase of the prepared TiO_2 nanopowder. In order to prepare of TiO_2 nanostructured material with significant properties several processes have been developed over the last decade and can be classified as liquid process sol–gel [12], solvothermal [13], hydrothermal [14], solid state processing routes mechanical alloying/milling [15-16], RF thermal plasma [17] and other routes such as laser ablation [18].

Different synthesizing methods were help to improve the crystalline size, morphology as well as its modifications along with doping for the various applications. But still a simple reliable, cost effective non-toxic and time-consuming synthesis method development is still ongoing by many researchers. Therefore, in this research work a simple reliable, cost-effective chemical bath deposition method was used to synthesize a pure TiO_2 thin film and further with the help of characterization its structural, morphological and optical properties were investigated.

Experimental

This work involves a synthesis of TiO_2 by the simple and inexpensive CBD method. At first, glass substrates were washed with lab reagent. Further treated with chromic acid and washed thoroughly with double distilled water. Afterward, 15ml TTIP solution, 15 ml 2N HCl and 30 ml Distilled water was taken in a 100 ml beaked and stirred well on a magnetic stirrer till clear solution was obtained. To this clear solution washed glass substrates were immersed vertically without any disturbance. After 24 hours the deposition of white product is obtained on glass substrate. Afterward, this deposited glass substrate was taken out of the bath and washed in distilled water by taking percussion that loosely bound film will not be disturbed while immersing into the distilled water for washing. Further, this thin film was dried in an air and subjected to the annealing at temperature 400ºC for 2 hours for particle growth and remove moisture. After cooling the pure TiO_2 was obtained on the glass substrate.

The formed thin film was applied for the characterization of XRD, SEM, EDS, UV-Vis and PL spectroscopic techniques to study its structural, morphological, optical properties.

X-Ray Diffraction

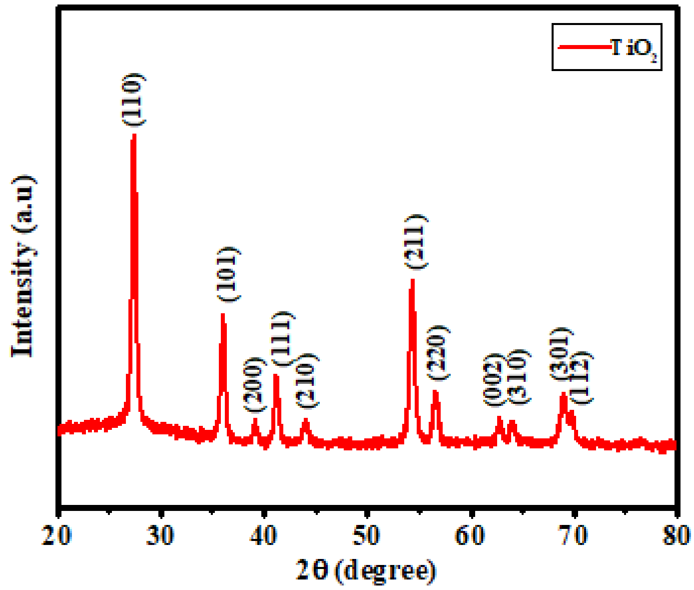

Figure 1. Powder X-ray diffraction patterns of TiO_2

Fig. 1 shows the XRD (X-ray diffraction) pattern of the TiO_2 obtained by chemical bath deposition (CBD) method, calcined at 400 °C for 2 hr. We can observe for the calcination of 400 °C. The synthesized sample was characterized by a Philips automated X-ray diffractometer (PW- 3710) equipped with a crystal monochromator employing Cu–Ka radiation of wavelength 1.5406 A°. The diffracting angle (2h) is varied between 20° and 80° and the recorded XRD patterns for the thin films are shown in Figure 1. The formation of rutile phase was formed with the intense of their peaks. The diffraction peaks showed considerable enlargement, thereby indicating the presence of nanoparticles of TiO_2 powder. The diffraction peaks at 2θ = 27.42°, 36.04°, 39.16°, 41.30°, 43.92°, 54.36°, 56.67°, 62.91°, 64.06°, 68.98°, 69.71° are corresponds to (110), (101), (200), (111), (210), (211), (220), (002), (310), (301), (112) plans respectively. No any extra peak was found corresponding to rutile phase. There is no secondary phases present, thus indicating that high purity of the product with a rutile structure. The rutile phase of the synthesized TiO2 nanoparticle was confirmed by matching the with the [JCPDS file No. 21-1276] [19]. The crystalline was calculated by using Scherrer formula (1)

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad \text{.....................} \ (1)$$

Where, β is the half width of diffraction peak measured in radian. And the calculated crystalline size of the TiO_2 is 32 nm.

SEM and EDS

Scanning Electron Microscopy (SEM) micrographs of TiO_2 films are shown in Fig. 2(a-d).

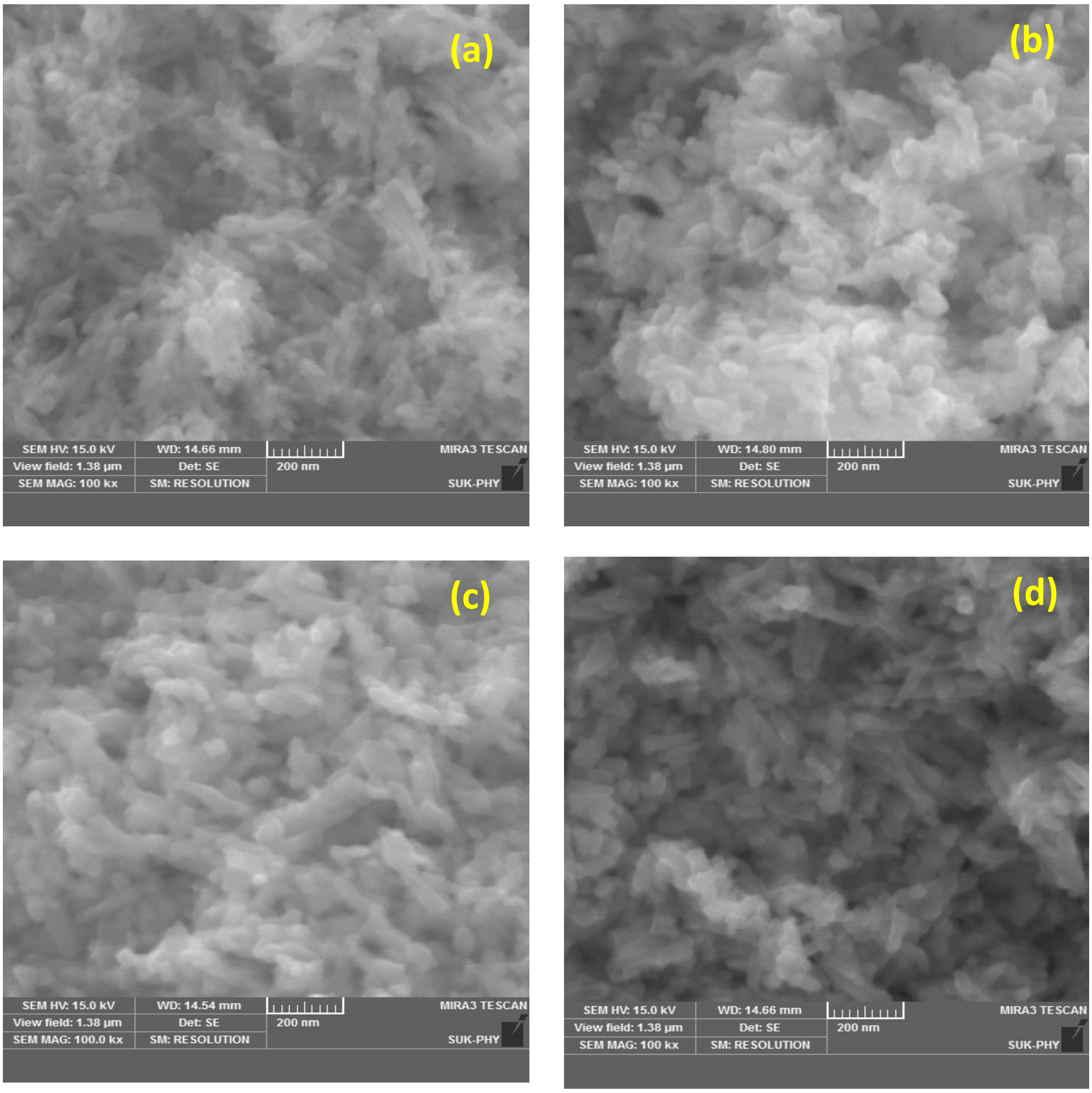

Figure 2. (a-d) Scanning electron microscope image of TiO_2

The morphology of the annealed samples was measured by Field- Emission Scanning Electron Microscopy (FESEM, JSM-7001F, JEOL). Scanning electron microscope images of pure TiO_2 shows interconnected nanorod like morphology.

Elemental composition is confirmed by Energy Dispersive X-ray Spectroscopy (EDS). From Figure 3 and Table 1 show that atomic weight % corresponds to titanium and oxygen. Titanium and oxygen are the major constituents of the sample. These constituents show their intensity according to their percentage present in the film as shown in figure 3. So it is clears that the sample doesn't have any impurity.

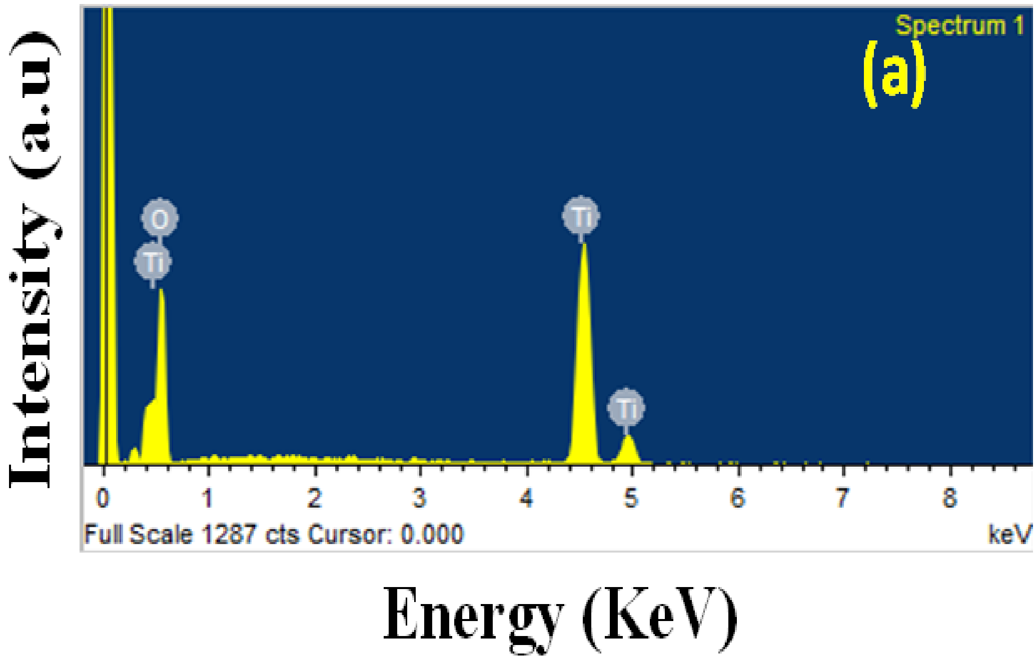

Figure 3. EDS graph of TiO_2

Table 1 Elemental analysis of TiO_2

Elements	**Atomic %**
	TiO_2
O	55.97
Ti	44.03
Total	100

UV-Visible spectroscopy

The optical absorbance of the sample recorded in the spectral region 200 to 800 nm at room temperature is shown in Figure 4. The figure showing a plot of wavelength vs absorbance. It is evident from Figure 3 that, Maximum absorption observed in the UV region at 293 nm.

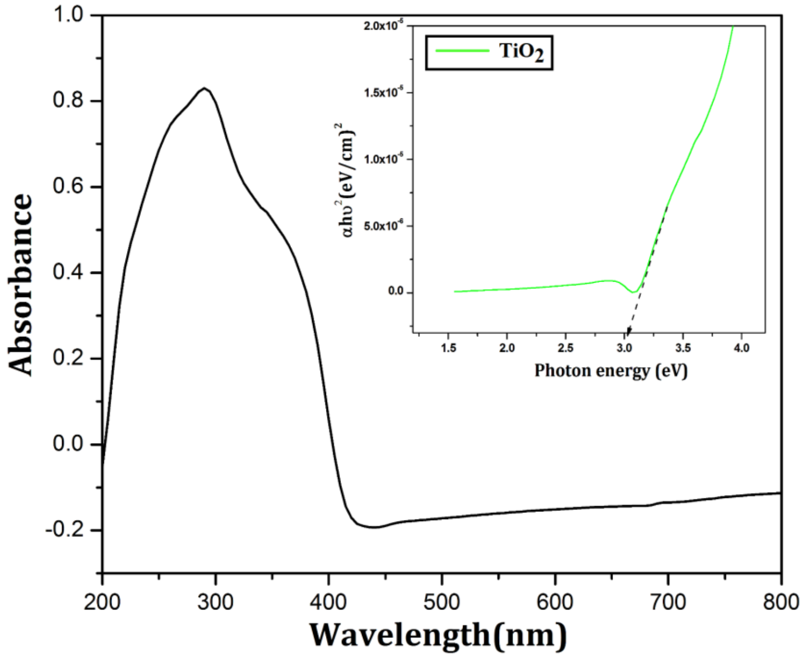

Figure 4. UV-Visible spectra of TiO_2

The inset showing an optical plot of $(\alpha h\nu)^2$ against (hν) plot, extrapolated to the point a = 0. The intercept of the tangent to the plot on the x-axis gives a good approximation of the bandgap energy for direct bandgap material. An optical study estimated the bandgap energy for pure TiO_2 is 3.1 eV which agrees with already reported values [19].

Conclusion

The chemical bath deposition method (CBD) was used to successfully produce TiO_2 (Titanium dioxide) thin films at room temperature. X-ray diffraction study revealed a rutile structure of synthesized TiO_2 thin film. SEM study depicted the nanorod like morphology whereas EDS showed good agreement of the elements with high purity of Ti and O. The crystallite size of the sample is found to be 32 nm. The optical analysis found that the band gap of TiO_2 nanoparticles is 3.1 eV using UV-Vis spectroscopy.

Conflict of interest: *There is no any conflict of interest.*

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