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Microstructural Parameters of TiO_2 Thin Films by SPD Technique

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Abstract : Titanium dioxide thin films have been deposited onto glass substrate by spray pyrolysis technique at different substrate temperature(Ts =300^{0}C to 400^{0}C) and annealed at 500^{0}C for 1hr.The annealed films were characterised by x-ray diffraction,scanning electron microscopy and uv-vis spectroscopy in order to identify their structure,phase,morphology compositionand optical properties respectively.The XRD profiles showed that films were polycrystalline,anatase type and oriented predominantly to the (101) plane.The optical properties of the films were studied using uv-vis spectrophotometer and from that energy band gap were evaluated.
Keywords: TiO_2,Thin Films, XRD,SEM,EDAX,UV-VIS.

Introduction

During the past decade titanium dioxide,(TiO_2-titania) has emerged as an important material for applications such as transparent electrical conductors[1], photovoltaics[2], water purification media[3], self-cleaning surfaces[4] and gas sensors[5]. For these applications thin films are used commonly owing to the advantages of easy preparation, flexibility of use and low cost in comparison to bulk materials.There are several effective methods for the synthesis of Titaniafilms including sputtering[6], laser ablation[7],electrophoreic deposition and anodic oxidation[8],sol-gel[9],screen printing[10],dip coating[11], gel oxidation[8],spray pyrolysis[12],and spin coating 13]. Spray pyrolysis is one of the most widely used methods for the fabrication of titania thin films owing to rapid growth rate,large sample size capacity and mass production capability,it tends to have a low precursor product yield rate during deposition. Titanium di oxide could crystallise in three different phases, anatase (tetragonal), rutile(tetragonal) and brookite (orthorhombic). In particular, rutile TiO_2 is the most stable phase and can be obtained in most crystal growth processes.The rutile form is thermodynamically stable at all temperatures. On the other hand anatase TiO_2 is less dense and less stable when compared to rutile phase. By applying thermal treatments it is possible to transform metastable anatase to rutile.The present study is focussed on microstructural properties of titanium dioxide thin films prepared by micro controller based spray pyrolysis technique using precursor solution of Titanium (iv) butaoxideandhave been investigated.

Experimental Details

Titanium(iv) butaoxide (Aldrich)mixed with hydrochloric acid was used as the precursor and sprayed onto the hot glass substrate by the micro controller based spray pyrolysis set up.The other deposition parameters like solution flow rate, carrier gas pressure and nozzle to substrate distance were initially set and the spray was started.The crystal structural study of film was examined by XPERT PRO diffractometerusing CuKα radiation. The scanning angle 2θ was varied in the range of 10^0-80^0. The SEM photograph was taken with scanning electron microscope to study the surface morphology.The spectral data was used to determine the type of optical transition and the band gap present in the sample.

Results and discussion.

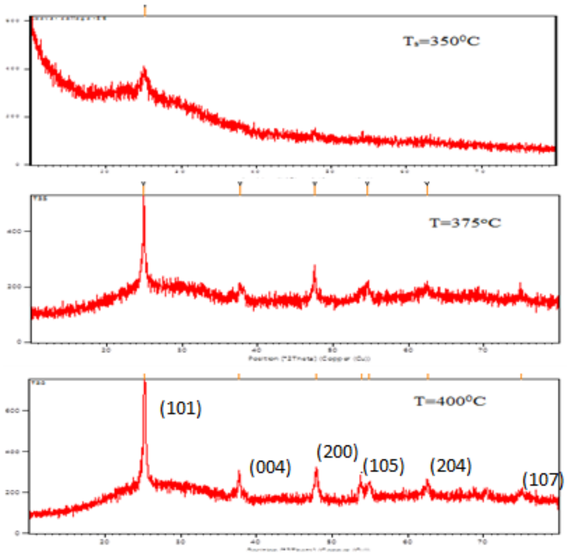

Figure 1XRD Spectra for the annealed TiO_2 films obtained at different substrate temperatures

The XRD profiles of TiO_2 thin films deposited at different substrate temperatures and annealed at 500^0 c is shown in fig1. The annealed films are polycrystalline in nature and oriented along A(101) A(004), A(200),A(105),A(211) planes,A(101) being the predominant peak.The films exhibited tetragonal structure and the peak intensities are in agreement with JCPDS data (21-1272). The intensity of the diffraction peaks gradually increases with increase in substrate temperature and the observed peaks were in agreement with reported values. The crystallite size is evaluated from the FWHM of the (101) plane using Scherrer's formula

$$D = \frac{k\lambda}{\beta cos\theta}$$

Where k=0.94 is the shape factor, λ is the x-ray wavelength of Cu K_α radiation,θ is the Braggs angle and β is full width at half maximum of the peaks.The micro strain is calculated using the relation.

$$\varepsilon = \frac{\beta cos\theta}{4}$$

The value of dislocation density is calculated using the relation.

$$\delta = \frac{1}{D2}$$

The effect of substrate temperature on the microstructural parameters of TiO_2 thin films are summarised in table 1From the table the crystallite size of TiO_2 thin films can be tuned between 21nm to 47nm.

Table 1. Micro structural parameters of annealed TiO_2 thin films at different substrate temperatures.

Substrate Temperature(^{0}C)	Crystallite Size (nm)	Strain (€)	Dislocationdensity (δ) Lines/m^2	Structure
350	47nm	7.1	0.0064	Tetragonal
375	33nm	3.4	0.0025	Tetragonal
400	21nm	4.5	0.0021	Tetragonal

Figure 2shows the surface morphology of TiO_2 thin films deposited at 400^0C.It is clearly seen from the micrograph the film had a well surface smoothness and uniformity. A representative EDS spectrum of TiO_2 thin film deposited at 400c is shown in fig 3. It is seen that titanium and oxygen are present in near stoichiometric ratio.

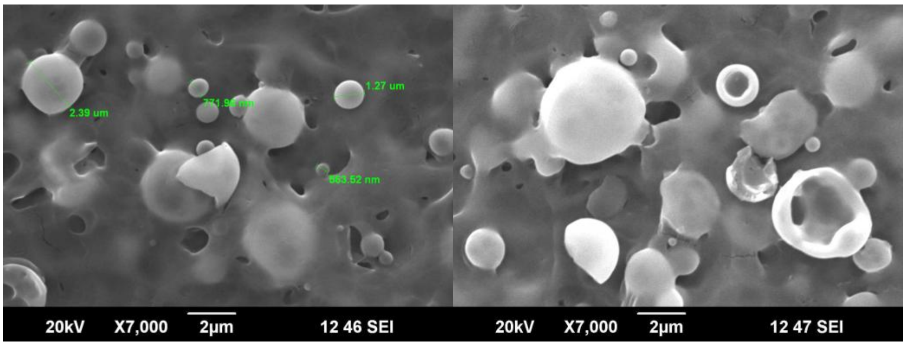

Figure 2 SEM Images of annealed TiO_2 films at different temperatures.

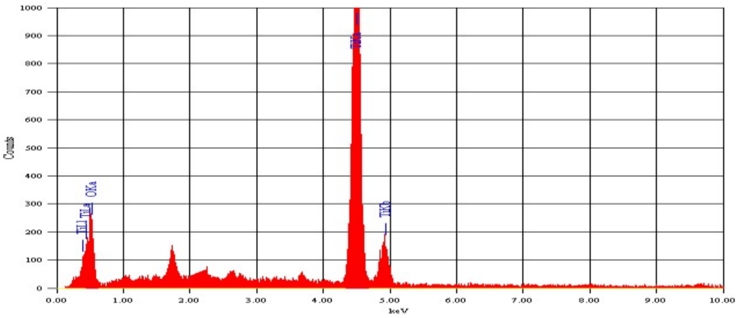

Figure 3 EDS spectrum for the annealed TiO_2 film prepared at $T_s = 400^0C$

The optical transmittance spectra of TiO_2films were taken using uv-vis spectrophotometer with wavelength range 300-900 nm. It was seen that average transmittance of films were transparent. The energy band gap of film was evaluated from the relation.

$(\alpha h\nu)^2=A(h\nu-E_g)$

where Ais the proportionality constant andE_g is the direct transition band gap.The band gap was found to be 3.2 eV and it agreed with previous results.

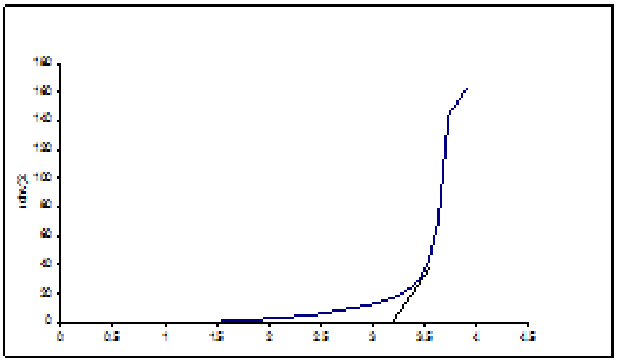

Figure 4$(\alpha h\nu)^2$ versus photon energy (hν) for annealed TiO2 film

Conclusion

The study of titanium di oxide thin films prepared by micro controller based spray pyrolysis technique using precursor solution of Titanium ivbutaoxide was carried out.Before annealing the films were amorphous and after annealing polycrystalline films were produced.The films were crystallised in the tetragonal form with preferred orientation along (101) plane.Optical studies showed direct band gap and the band gap energy was found to be 3.2eV and it agreed with previous results.

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