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ANNEALING EFFECT AND STRUCTURAL CHARACTERIZATION OF TiO_2 THIN FILM PREPARED BY e-BEAM EVAPORATION METHOD

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ABSTRACT

TiO_2 is a cheap, non-toxic and one of the most efficient semiconductor photo catalysts for extensive environmental applications because of its strong oxidizing power and corrosion resistance. Transparent titanium dioxide (TiO_2) thin films were prepared by e-beam evaporation technique on a well-cleaned glass substrate. The pressure and deposition rate were 3.0×10^{-6} m.bar and 3 nm/s respectively. The structural characteristic of the film were analysed using GIXRD and Raman spectroscopy. The surface morphology of the film were analysed using Atomic Force Microscopy (AFM) and XRR. GIXRD studies show the as-deposited films to be amorphous and an increase in crystalline of the film was observed with increase in annealing temperature. The anatase phase of the TiO_2 film was confirmed from the GIXRD and Raman data.

Keywords : Anatase, Thinfilm, GIXRD, XRR, Raman spectroscopy, AFM.

I INTRODUCTION

Titanium Dioxide (TiO_2) is reported to have unique and important properties by various researchers. It finds applications in photocatalysis, for optical coating, as gas sensors, humidity sensors, in transparent electronic and as an dielectric material [1-8]. The optical, structural and electrical properties are found to depend on the fabrication technique used and growth conditions. There are numerous techniques available to fabricate TiO_2 thin films like electron beam evaporation, sputtering, pulse laser deposition, sol gel and spin coating. [9 – 15].In present work, we report on the effect of annealing temperature on the structure and surface of the as prepared TiO_2 thin films prepared by e-beam evaporation method.

II EXPERIMENTAL

The films under study have been evaporated by e-gun at 8kW in a high vacuum chamber at a pressure of 3.0×10^{-6} m.bar . Commercial materials of TiO_2 (palate) were used as original materials. The TiO_2 (99.99%) powder was compressed into a pellet form by applying pressure about 7.5 tons/cm^2 (747 MPa) using a hydraulic press and annealed at 500 ^{0}C for 10 hours. The substrate used were normal glass plate. The deposition rate and substrate temperature were monitored by a quartz crystal oscillator and thermocouple placed on the substrate holder. The thickness of the prepared film was ~80 nm. Post- annealing of thin films were done in a muffle furnace in a normal air atmosphere at 500 ^{0}C for 1 hour The crystallinity of the films was analysed using Grazing Incident X-Ray diffraction (GIXRD) using CuKα radiation.

III RESULT AND DISCUSSION

3.1 GIXRD and XRR Analysis

Normal XRD to analyse the structure of the fabricated films was not possible due to small thickness of the film. GIXRD patterns of the as deposited films are shown in fig. 1. GIXRD patterns of the films without annealing shows amorphous nature. The amorphous nature of the as deposited films is due to the slow surface diffusion of the particles with low energy. The GIXRD patterns of the post annealed films shows the transformation of the films from amorphous to a crystalline structure. The post annealed films of TiO_2 exhibits the anatase phase with preferential growth along (101) plane. The obtained pattern matches with JCPDS data card (21-1272). The crystalline size estimated using Debye Scherrer’s formula shows 37 nm for the (101) plane.

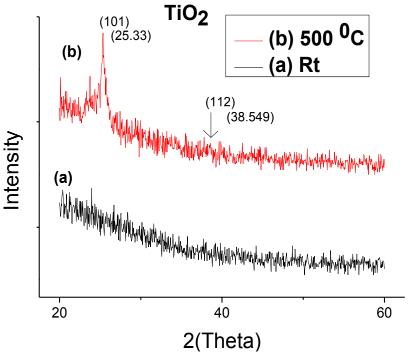

Fig. 1 : GIXRD data of the TiO_2 films.

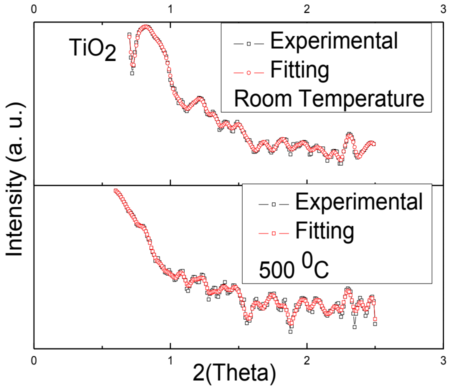

Fig. 2 (a): XRR data of the TiO_2 thin film prepared by e-beam technique.

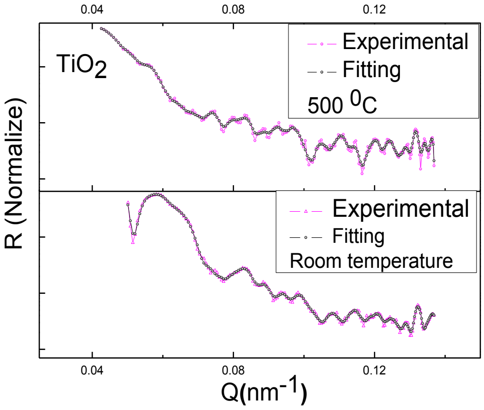

Fig. 2 (b): XRR fitting of the TiO_2 thin film prepared by e-beam technique

Since technological applications of thin films require films of definite thickness and surface morphology we used the XRR technique [16] for studying the surface property of the prepared film. From figure 2(a) and 2(b) it is clearly observed that annealed films shows a more smooth surface as compared to those which were not without annealed.

3.2 Raman Spectroscopy

The scans were taken in the range of 100 to 200 cm^{-1}. The main peak of anatase phase of TiO_2 is observed near about 144 cm^{-1}. The Raman spectroscopy of the samples confirms the anatase phase of TiO_2 as determined by GIXRD analysis.

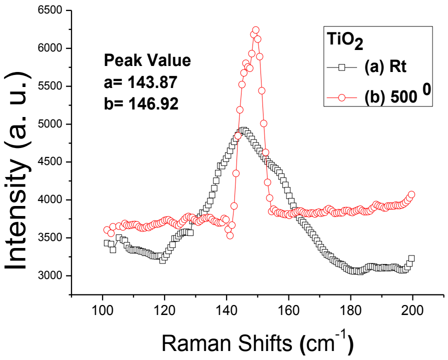

Fig. 3, Raman spectra of TiO_2 thin film prepared by e-beam technique.

Obtained Raman spectra shows a broad spectra for the non-annealed film and a narrow peak is observed in the annealed film. A small shift is also observed in the peak for the annealed film which indicates change in the structural parameters of the film. The film which was annealed shows a Raman peak at 143.87 cm^{-1} and the annealed film shows a Raman peak at 146.92 cm^{-1}.

3.3 Atomic Force Microscopy

The AFM images for the films are shown in Fig.4(a) and 4(b). Both images shows roughness at the surface, but more homogeneity is seen in the case of annealed film. The 2D images for both films shows the spherical shape of the particles but the particle size is found to be small and of uniformly spherical shape for the aneealed film.. The particle size calculated from the AFM data is ~115 nm for the annealed films. AFM images obtained on different regions of the samples showed that the films exhibit a homogeneous globular structure. The entire film surface is formed by small grains of the deposited material. The AFM result suggests that the surface morphology of the film depends on annealing temperature of the film.

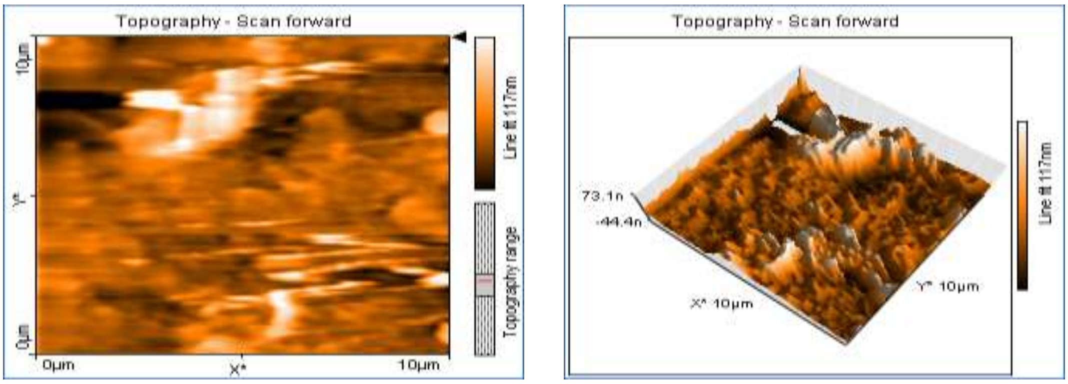

Fig. 4 (a), AFM data of the TiO_2 films (without annealed)

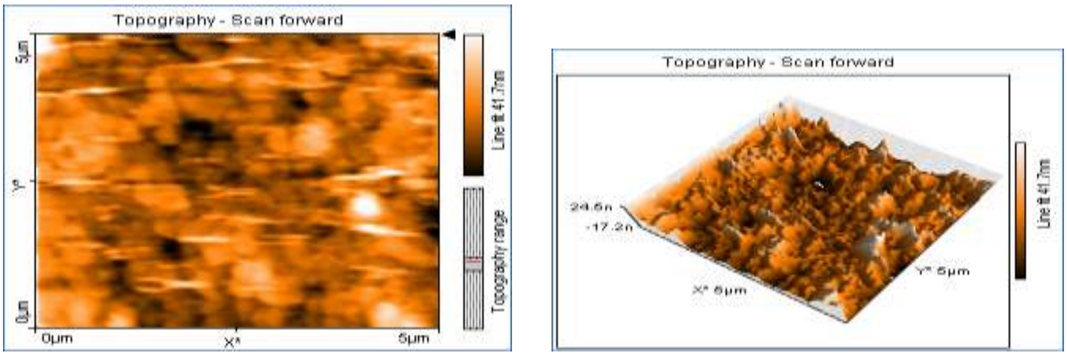

Fig. 4 (b), AFM data of the TiO_2 films annealed at 500^0C

III CONCLUSION

Thin films are smooth and highly transparent. Thin films shows the amorphous nature at room temperature (without annealed film) and post annealed film shows that the crystallinity of the films increases for the anatase phase of TiO_2. XRR study also suggests that the post annealed films has surface morphology which is more smooth as compared to without annealed films. Raman data suggests that the anatase phase is present in the prepared TiO_2 film. AFM study suggests that the smoothness of the film increases with increase in calcination temperature.

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