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# Effect of annealing and O<sub>2</sub> pressure on structural and optical properties of pulsed laser deposited TiO<sub>2</sub> thin films

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#### ABSTRACT

In this paper, effect of annealing and  $O_2$  pressure on the structural and optical properties of pulsed laser deposited thin films of TiO<sub>2</sub> is reported. XRD, FTIR spectra and SEM images confirm that at high annealing temperatures, the rutile phase and crystalline quality of thin films increases. Higher pressure of  $O_2$  during deposition improves the rutile phase and favors the rod like growth of TiO<sub>2</sub> thin film. The red shift in photoluminescence (PL) spectra of TiO<sub>2</sub> thin films with annealing temperature is reported. Contact angle measurement data for the thin films reveals the hydrophobic nature of the films. The very low reflectivity ( $\sim$ 10%) reported in this paper may be promising for anti-reflection coating applications of pulsed laser deposited TiO<sub>2</sub> thin films.

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#### 1. Introduction

TiO<sub>2</sub> is an important n-type wide band gap II–VI semiconductor with high refractive index and high dielectric constant. TiO<sub>2</sub> thin films have drawn a great deal of attention in recent years due to their wide application in solar cells, photo catalyst and self cleaning windows [1–3]. TiO<sub>2</sub> thin films have found applications in multilayer optical filter [4], anti-reflection coating [5] and metal-insulator–semiconductor (MIS) devices [6]. Room temperature grown TiO<sub>2</sub> thin films can have three structural forms anatase (tetragonal), rutile (tetragonal) and brookite (orthorhombic). Annealing at temperatures between 300 °C and 800 °C initiates a transition from primary anatase to the rutile phase [7,8].

Deposition of TiO<sub>2</sub> in anatase as well as rutile phase has been reported very well in the literature [5,7,9]. Among all the reported techniques, pulsed laser deposition (PLD) provides the precise manipulation of the properties of thin films of TiO<sub>2</sub> by controlling the deposition parameters [9–14]. Normally, TiO<sub>2</sub> thin films deposited at room temperature are amorphous with the anatase phase which gets converted to rutile phase after annealing [15] as former being thermodynamically unstable. Ambient oxygen pressure and post-deposition annealing are the most important

parameters that affect the phase formation, morphology and microstructures and optical properties of pulsed laser deposited TiO<sub>2</sub> thin films. Xin et al. [16] discussed the effect of oxygen pressure on the structural properties of Co-doped TiO<sub>2</sub> films and Long et al. [17] have reported effect of substrate temperature and ambient pressure on the microstructure of TiO<sub>2</sub> films. However, the effect of ambient pressure and post-annealing is not well documented in the literature for room temperature deposited TiO<sub>2</sub> thin films using PLD.

In the present paper, we have reported pulsed laser deposition of high quality  ${\rm TiO_2}$  thin films at room temperature in oxygen ambient using high purity  ${\rm TiO_2}$  pellet. The effect of ambient pressure  ${\rm (O_2)}$  and post-deposition annealing over structural, optical properties and the contact angle with water of pulsed laser deposited  ${\rm TiO_2}$  thin films is presented.

#### 2. Experimental setup

The experimental setup used to deposit the  $TiO_2$  thin films is shown in Fig. 1. The second harmonic of Q switched Nd:YAG laser (Model—Quanta systems-HYL101, 400 mJ/pulse in fundamental with 8 ns pulse duration and 10 Hz repetition rate) is focused on to the rutile  $TiO_2$  of high purity (sintered at  $1100\,^{\circ}$ C for 3 h and at  $1200\,^{\circ}$ C for 18 h) target with a lens of focal length of 35 cm. The target was mounted inside the vacuum chamber through a motorized vacuum feed through and continuously moved in order to avoid piercing with the repeated shots of laser. The chamber was initially evacuated to a base pressure of  $10^{-6}$  mbar and then filled with  $O_2$  gas in the pressure range of  $10^{-1}$  mbar to  $10^{-3}$  mbar. When the high power laser was focused on to the  $TiO_2$  target in the ambient of  $O_2$  gas, plasma containing neutral

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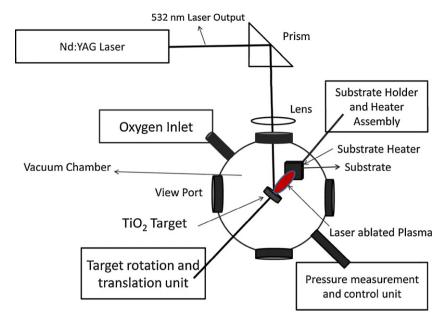


Fig. 1. Experimental setup.

and excited species of titanium and oxygen expanded and undergoes through the proper dynamics and finally thin films of  ${\rm TiO_2}$  were deposited on ultra sonically cleaned, polished glass substrates placed parallel to and 3 cm apart from the target at room temperature as shown in Fig. 1. Deposition time was kept fixed for 30 min for all the results reported in this paper. After deposition,  ${\rm TiO_2}$  thin films were annealed in high temperature furnace for 6 h at different temperatures in the

range of 400–650 °C in air. These films of TiO<sub>2</sub> were scanned with SEM (LEO-1430vp) for surface morphology and XRD (SEIFERT 3003) for the crystal structure. Optical characterizations were performed with UV–vis spectrophotometer (Cary 100 Varian), photoluminescence (Thermo-spectronic Aminco Bowman Series 2) and FTIR (Horriba Jobin Yuvon). Contact angle measurement of TiO<sub>2</sub> films annealed at different temperature was performed by the pendant drop technique using Phoenix

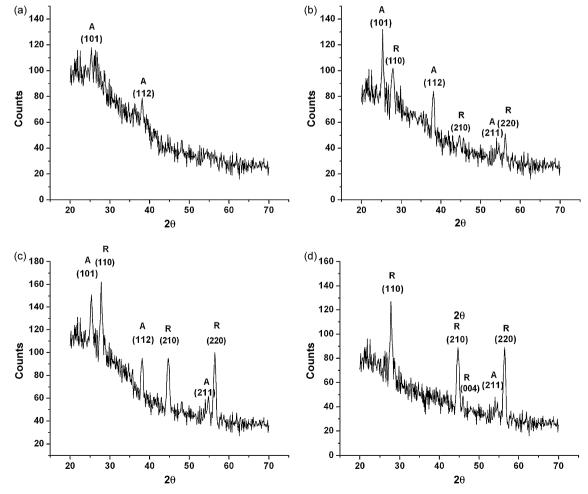


Fig. 2. XRD pattern for TiO<sub>2</sub> thin films deposited at 10<sup>-1</sup> mbar O<sub>2</sub> pressure at room temperature; post-annealed at (a) as-deposited (b) 400 °C (c) 500 °C and (d) 650 °C.

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