# Influence of thickness on the physical properties of nanostructured TiO<sub>2</sub> thin films for nitrogen dioxide gas sensor

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TiO<sub>2</sub> thin films were deposited utilizing chemical vapor deposition. XRD data indicate that all the films were polycrystalline with a predominat plane along (121). As the dislocation density and strain parameter reduce from (91.15 to 64.13), (31.97 to 27.76 For film thicknesses of 250 nm and 350 nm, respectively. the grain size increases from 10.45 nm to 12.48 nm with an increase in thickness from 250 to 350 nm. AFM images revealed that average particle sizes were 80.1 nm to 24.8 nm as thickness increased, whereas surface roughness averages decreased as film thickness increased. SEM images reveal thin film surfaces (TiO<sub>2</sub>) (250, 300, 350 nm), and reduced grain size indicates finer particles. With increasing thickness, it is discovered that the band gap and the film's transmittance both decreases. It was found that as film thickness increases, so do absorption and absorption coefficient. The direct band gap dropped from 3.28 to 3.15 eV with the increase in thickness. The calculated extinction coefficient of the films with different thicknesses was reduced from 0.58 to 0.55. The gas sensor tested with  $NO_2$  exhibited increased resistance at 300 ppm, indicating high sensitivity. TiO<sub>2</sub> film with a thickness of 350 nm showed the highest resistance. Sensitivity decreased with higher thicknesses (250, 300, and 350 nm) for NO<sub>2</sub> gas, with reductions observed: 33.4% to 10.9% (100 ppm), 35.4% to 12.5% (200 ppm), and 37.2% to 14.8% (300 ppm).

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Keywords: TiO2, Thin film, Chemical vapor deposition, Optical, Topographical

# 1. Introduction

 $TiO_2$  has been the focus of intense research in academia and industry because it is nontoxic, affordable, highly photoactive, and simple to synthesize and handle.  $TiO_2$  films are suitable for storage capacitors, protective coatings, and optical components due to their high electrochemical characteristics, dielectric constant, and transparency [1]. Most studies concentrated on nanoscale  $TiO_2$  [2] to increase photocatalytic activity and optical absorption. Due to their high refractive index, several industrial uses, like  $TiO_2$  films, are widely used in optical coatings [3,4]. The band gap differs for each material, measuring 3.32 eV for anatase, 3.03 eV for rutile, and 2.96 eV for brucite

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[3]. Due to its intriguing electrical [5,] optical [6,] magnetic [7], and catalytic uses, the TiO<sub>2</sub> nanostructure has received a lot of attention recently in energy conversion [8] biomedical applications [9] functionalized hybrid materials [10] and nanocomposites [11] due to its semiconductivity and UV-induced photoelectrical and photochemical activity. TiO<sub>2</sub> nanostructures have a variety of applications, including TiO<sub>2</sub>/polymer nanocomposites [12], self-cleaning coatings [13], photocatalysis, chemical sensors [14], (D.S.S.C.) [15], and P.E.C. [16]. TiO<sub>2</sub> thin films can be created using various methods, including plasma spray. [17], RMS [18, 19], sol–gel [20, 21], PLD [22], solvothermal method [23], SP [24], ion beam sputtering [25]. thermal evaporation [26] and CVD [27, 28]. The Vacuum Thermal Evaporation Method effectively deposits titanium dioxide on various surfaces under various technological circumstances. This is expected to provide multiple structural and microscopic properties and diverse surface morphologies of the nanostructures. This study has investigated various aspects of TiO<sub>2</sub> thin films produced by CVD, encompassing their structural, morphological, and optical properties and their sensor characteristics. The influence of different thicknesses was also elucidated.

#### 2. Experimental

Ti was vaporized on glass slides using an Edwards vacuum thermal evaporation system at ambient temperature and low pressure (10<sup>-6</sup> or 10<sup>-5</sup>) to prevent interaction between the vapour and the atmosphere. The material vapor on the cooled substrate produced thin films (Ti) by placing the material in Tungsten and Molbidnyom boats, which were then heated to cause it to evaporate. Evaporation-produced films are comparatively pure, making them attractive from a theoretical perspective. TiO<sub>2</sub> thin films were annealed at 500 °C (Ti thin films oxidation temperature). To detect the crystallographic phase, use XRD (Rigaku RINT2000). CuK<sub>a</sub> radiation, recoded from  $20^{\circ}$  to  $80^{\circ}$ of 2 at a scan rate of 2° min<sup>-1</sup> was used to measure the XRD patterns at a glancing angle of 3°. AFM was used to examine how the samples' thickness and surface morphology evolved (Nanoscope IV, Veeco Instrument Inc.). The samples underwent surface morphology analysis through scanning electron microscopy (SEM, EVO40-LEO). Measuring optical characteristics using UV-VIS spectrophotometer from the SPECTROMOM 195D, the optical properties of the newly created TiO<sub>2</sub> were examined. The produced films' transmittance and absorption spectra are between 300 and 900 nm in wavelength. A popular way to assess gas sensitivity is to look at the alteration in film resistance upon irradiation. For testing, samples were placed inside a cylinder that measured 15 cm in height and 7.5 cm in radius.

#### 3. Results and discussions

Figure 1 presents the XRD patterns for TiO<sub>2</sub> thin films of various thicknesses produced via chemical vapor deposition at temperatures of 250, 300, and 350°C. The XRD analysis revealed a polycrystalline structure for TiO<sub>2</sub> with the Rutile phase, as per the American Standard for Testing Materials (ASTM) cards. Figure 1 indicates that the predominant growth pattern occurred at the plane, with peaks appearing at 20 values of 25.46°, 30.74°, 49.84°, and 64.31°, corresponding to the (120), (121), (132), and (203) planes, respectively. These findings align with previous investigations [31], confirming the polycrystalline structure of the films and corroborating the results with the reference value from JCPDS card No (29.1360). Table 1 compares the measured 20 values of the produced samples with the standard values found on the ASTM cards, showing remarkable similarity. Scherer's Eq. was employed to get the samples' average grain size (*D*) via [32-34]:

$$D = \frac{k\lambda}{\beta \cos\theta} \tag{1}$$

where k=0.9,  $\lambda$  is wavelength of the incident X-ray,  $\theta$  is Bragg angle, and  $\beta$  is FWHM of the (200) plane orientation diffraction peak. The predicted grain size for thin films of TiO<sub>2</sub> with a thickness of

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250 nm was 10.45 nm, but after 300 and 350 nm thicknesses, the grain size increased to 11.44 nm and 12.48 nm, respectively. The density of nucleation centres decreased with increasing film thickness, resulting in fewer centres growing and larger grains.

Using Scherer's formula, the samples' average *D* was calculated using X-ray line broadening analysis. [35].

The dislocation density ( $\delta$ ) was obtained via the equation [36, 37]:

$$\varepsilon = \frac{\beta \cos\theta}{4} \tag{2}$$

 $\delta$  decreases from (91.15 to 64.13).

The strain (%) in thin films was estimated using equation [38, 39]:

$$\delta = \frac{1}{D^2} \tag{3}$$

The strain (%) decreases from 31.97 to 27.76 as the thickness of the thin films increases. The obtained structural parameters (Pst) are provided in Table 1. It can be observed that both ( $\delta$ ) and ( $\epsilon$ ) increase as film thickness increases [40]. Fig. 2 illustrates the variation of full width at (FWHM), (D), and other parameters concerning various thicknesses.



Fig. 1. XRD peaks of entended films.

Table 1. D,  $E_g$  and  $P_{st}$  of grown films.

Thickness (nm)	2 θ	(hkl)	FWHM	Eg(eV)	D	$\delta$ (× 10 <sup>14</sup> )	Strain
	(°)	Plane	(°)		(nm)	(lines/m <sup>2</sup> )	$(\times 10^{-4})$
250	30.74	121	0.76	3.28	10.45	91.15	31.97
300	30.71	121	0.72	3.22	11.44	76.32	30.29
350	30.67	121	0.66	3.15	12.48	64.13	27.76



Fig. 2. Pst of entended films.

Atomic force microscopy (AFM) in many modes was used. The measurements examined the surface, crystal growth pattern, grain sizes, and surface roughness( $R_a$ ). The surface morphology of TiO<sub>2</sub> thin films discussed appears in Figure (3). The average particle  $P_{av}$  size reduces as the thickness increases, with values of  $P_{av}$  variable ranging from (80.1 -24.8 nm) to (250) nm and (350) nm, respectively. This may be because larger clusters are produced when two or more grains combine [41]. These findings are consistent with those of other research [42]. Root mean square (rms) was observed in the area of 6.32 nm to 2.72 nm with the thicknesses (250) nm and (350) nm, respectively, in the AFM picture, while  $R_a$  showed the same trend dropped from 7.28 nm to 3.67 nm. AFM parameters  $P_{AFM}$  are listed in Table 2.



Fig. 3. AFM informations.

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Thickness (nm)	Pav	R <sub>a</sub> (nm)	rms
	nm		(nm)
250	80.1	7.28	6.32
300	31.9	6.45	4.50
350	24.8	3.67	2.72

SEM images in Fig. 4 (A,B,C) provide visual insights into the surface morphology of  $(TiO_2)$  thin films with different thicknesses (250, 300, and 350 nm). The observed decrease in grain size indicates a trend toward smaller particles as the thickness increases. Additionally, the deposited films exhibit a compact structure, suggesting effective film deposition and adhesion to the substrate. The slight variations in surface morphology between the 300 and 350 nm thickness  $TiO_2$  films compared to the 250 nm thickness  $TiO_2$  films may stem from differences in lattice structure and the formation of defects during deposition. These variations can influence chemical adsorption, nucleation, and subsequent growth processes, leading to observable differences in surface morphology.



Fig. 4. The SEM TiO<sub>2</sub> thickness (A) 250 nm (B) 300 nm, (C) 350 nm.

As can be observed from Fig. (5), which is a plot of absorbance against wavelength, the absorbance that corresponded to greater thickness (350 nm) was low in the short wavelength region of the VIS, swiftly declined to lower values in the long wavelength portion of the VIS, and then gradually decreased to the NIR region. However, the thinnest film (250 nm) had a larger absorbance than the other films, steadily dropping from the visible spectra short wavelength region to the NIR region. With increasing TiO<sub>2</sub> film thickness, the absorbance of the films decreased, which may be attributable to a higher film density and/or a thicker precursor [43]. It was widely researched how varying TiO<sub>2</sub> film thicknesses affected optical characteristics.

The transmittance (T) of these films was calculated using the formula that is displayed in Fig. 6, from absorbance (A) data: [44]:

$$A = 2 - \log 10 \, (\% \, \mathrm{T}) \tag{4}$$

Figure (6) depicts grown films' T spectra. According to this Figure,  $TiO_2$  films have a high visible T of up to 73.6% for 250 nm, slightly decreasing to 70.5.4% for 350 nm films due to light losses and increasing film absorption. Transmittance is inversely related to thickness, meaning that as thickness grows, transmittance reduces [45], though only marginally. The phenomenon is

explained by two factors: film crystalline nature throughout the coating area, which is the result of identical oxidation and an enhancement of the lattice structure, which increases absorptance and decreases transmittance, and an increase in atom density with increasing thickness, which increases the number of incident atom collisions [46]. This outcome is dependent on numerous other works. [47,48]. Within the visible range, TiO<sub>2</sub> coatings are highly transparent. It is well known that basic light absorption and free-carrier absorption are the reasons behind the abrupt drop inT of TiO<sub>2</sub> films in UV-IR areas.



Fig. 6. T of deposit films.

The absorption coefficient ( $\alpha$ ) is calculated from the experimental optical absorption spectra using the relation [49]:

$$\alpha = (2.303 \times A)/t \tag{5}$$

where (t) is film thickness. The fluctuation of the incident photon energy is seen in Figure 7. According to the graph,  $\alpha$  rises as photon energy(h $\nu$ ) rises for the thin films under investigation. The values are larger than 10<sup>4</sup> cm<sup>-1</sup>, supporting the earlier reports that the likelihood of direct electronic transitions is relatively high [50]. Using Tauc's relation, the direct optical band gap E<sub>g</sub> of deposited TiO<sub>2</sub> thin films was calculated. [51]:

$$(\alpha hv) = A \left( hv - E_q \right)^{1/2} \tag{6}$$

where A is a proportional coefficient. Figure 8 displays the plots of  $(h\upsilon)^2$  vs. h $\upsilon$ . With increasing thickness, it can be shown that  $E_g$  values for TiO<sub>2</sub> thin films decrease. The calculated Eg values fall between 3.28 and 3.15 eV; the drop in Eg value may be attributed to the thin film's crystallinity. This narrowing of the energy gap may result from the thin film's thickening, which caused donor levels to form  $E_g$  close to the conduction band. Consequently, low-energy photons will be absorbed. [52].

![](_page_7_Figure_3.jpeg)

Fig. 8. Eg of grown films.

The extinction coefficient (k) was expressed as [40]:

$$k = \frac{\alpha\lambda}{4\pi} \tag{8}$$

where  $\lambda$  is the wavelength of the incident ray. The fluctuation of (K) for films is shown in Figure (9). Due to their previous relationship, which shows extinction coefficient decreases as thickness increases, extinction coefficient exhibits the same behavior as absorption coefficient ( $\alpha$ ). The decrease in k value might be connected to the film's crystallinity. [53]. The refractive index (n) is measured via the relation [20]:

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \tag{9}$$

where ((R) is the reflectance. The fluctuation of (n) for films is shown in Figure (10). n show 3.06 and 2.95 for  $\lambda$  between 300 and 900 nm, n value decreased with increasing thickness. The increase in packing density and the altered crystalline structure may be responsible for the drop brought on by the acceleration of crystalline growth. [54].

![](_page_8_Figure_3.jpeg)

Fig. 9. k of grown films.

![](_page_8_Figure_5.jpeg)

Fig. 10. n for grown films.

The sensitivity of the sensor, can be computed as [55]:

$$Sensitivity = \frac{\Delta R}{R_g} = \left| \frac{R_g - R_a}{R_g} \right| \times 100 \%$$
 (10)

The gas sensor, consisting of porous silicon and a thin film of  $TiO_2$  on glass, was exposed to  $NO_2$  at a content of 300 ppm. The connection between resistance and time for TiO2 films at 300 ppm with varying thicknesses (250, 300, and 350 nm) is shown in Fig.11.

The sensor was operated at a temperature of 200 °C. Upon exposure to NO<sub>2</sub> molecules, an oxidation process is initiated on the surface of the TiO<sub>2</sub> film. This process causes certain  $O^{2+}$  ions to release bonded electrons to the surface, leading to electron drift back to the conduction band [56]. Consequently, resistance increases, and a more pronounced potential barrier is observed [57]. Furthermore, it was noted that the TiO<sub>2</sub> film with a thickness of 350 nm exhibited the highest resistance.

The sensitivity curves depicted in Figure (12) showcase how the thickness of TiO<sub>2</sub> films (250, 300, and 350 nm) affects their response to NO<sub>2</sub>. Sensitivity is greatly influenced by the charge carrier recombination mechanism, with a noticeable decrease as the thickness increases. Across the range of thicknesses (250, 300, and 350) nm, sensitivity dropped from 33.4 % to 10.9 % (100 ppm), 35.4 % to 12.5 % (200 ppm), and 37.2 % to 14.8 % (300 ppm) [58]. This diminishing sensitivity can be attributed to decreased surface-to-volume ratio as the TiO<sub>2</sub> film thickness increases, resulting in fewer active sites available for gas interaction.

![](_page_9_Figure_2.jpeg)

Fig. 11. Resistance via time of TiO<sub>2</sub> of grown films at 300 ppm NO<sub>2</sub> in at the optimum working temperature.

![](_page_9_Figure_4.jpeg)

Fig. 13. Sensitivity (S) as a result of operating time for  $TiO_2$  thin films with different thicknesses (250, 300, and 350) nm.

## 4. Conclusion

Chemical vapor deposition was employed to be set  $TiO_2$  films on to glass slide. Atomic force microscopy, UV-VIS-NIR spectrophotometer, and an X-ray diffractometer were each used to analyze optical transmittance and the crystal structure, respectively. The deposited films had a thickness of around (250, 300, and 350) nm and were transparent. According to the XRD data, each sample had a polycrystalline structure. The grain size increases as thicknesses from (250 to 350) nm increase. The strain parameter drops from (31.97 to 27.76) and the dislocation density drops from (91.15 to 64.13), with increase in film thicknesses.

The AFM image revealed that  $P_{av}$  size was observed in the domain of 80.1 nm to 24.8 nm. SEM images display that the grain size of thin films (TiO<sub>2</sub>) indicates finer particles. Transmittance, n, and k decreased with increasing thicknesses, whereas absorbance and  $\alpha$  increased with film thickness. E<sub>g</sub> is in the range of (3.28 - 3.15) eV, according to the absorption spectra. The NO<sub>2</sub> gas sensor exhibited heightened resistance at 250 ppm, indicating sensitivity. TiO<sub>2</sub> films with a thickness of 350 nm demonstrated the maximum resistance. Sensitivity decreased with higher thicknesses (250, 300 and 350) nm for NO<sub>2</sub> gas.

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