

# **Morphology and Optical Properties of TiO<sub>2</sub> Thin Films Acquired by Sol-gel Spin Coating Method Using Titanium (IV) Butoxide**

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## **Abstract**

Titanium dioxide (TiO<sub>2</sub>) is widely recognised for its excellent electronic properties, particularly in gas-sensing applications. However, its large optical band gap limits electron transitions between the valence and conduction bands, restricting its full potential. This study investigates the fabrication of TiO<sub>2</sub> thin films using the sol-gel spin-coating method, with varying concentrations of titanium (IV) butoxide: 1.0 mL, 1.5 mL, and 2.0 mL. The primary objective is to optimise the concentration and analyse its impact on the films' morphological, topography and optical properties. To achieve this, the films were characterised using Field Emission Scanning Electron Microscopy (FE-SEM) and Atomic Force Microscopy (AFM) to evaluate surface morphology, cross-sectional structure, grain size, and roughness. The optimised concentration of 2.0 mL titanium (IV) butoxide produced films with a uniform coating, enhanced by solution ageing and controlled annealing temperature. FE-SEM images revealed a combination of nano-sized and micro-sized particles, with an increase in grain size correlating to pile-up at grain boundaries and an increase in roughness (3.914 nm). Additionally, UV spectroscopy analysis showed a reduced optical band gap of 3.20 eV and high transmittance. These results suggest that TiO<sub>2</sub> thin films, with their promising electronic and optical properties, hold significant potential for applications in gas sensors and solar cells.

**Keywords:** Optical Band-gap, Roughness, Sol-gel, Spin Coating, Titanium (IV) Butoxide

## **1.0 Introduction**

Titanium dioxide (TiO<sub>2</sub>), commonly known as titania, is a transition metal oxide that has garnered significant attention in various scientific and industrial fields. Due to its exceptional optical, electrical, and chemical properties, TiO<sub>2</sub> is widely used in applications such as photovoltaics [1], photocatalysis [2], gas sensors [3], and optoelectronic devices [4]. As a

semiconductor with a large optical band gap (~3.2 eV for anatase and ~3.0 eV for rutile), TiO<sub>2</sub> exhibits high chemical stability, strong adsorption capability, non-toxicity, and biocompatibility, making it a versatile material for both environmental and technological applications [5].

One of the key challenges in TiO<sub>2</sub>-based applications is optimising its structural, morphological, and optical properties to enhance performance. The fabrication technique and precursor concentrations play a crucial role in determining these characteristics. Among the various deposition techniques available, the sol-gel spin-coating method stands out due to its simplicity, cost-effectiveness, and ability to produce highly uniform and well-controlled thin films. This study explores the influence of varying concentrations of Titanium (IV) butoxide precursors on the morphological and optical properties of TiO<sub>2</sub> thin films synthesised via the sol-gel spin-coating method [6].

TiO<sub>2</sub> exists in three primary polymorphs: anatase, rutile, and brookite, each possessing distinct structural and electronic properties. The anatase phase, characterised by a tetragonal crystal structure, is the most commonly used form for photocatalytic and gas-sensing applications due to its superior charge transport and electron mobility. Rutile, another tetragonal polymorph, exhibits higher thermal stability and is widely used in optical coatings and pigment applications. The brookite phase, with its orthorhombic structure, is less explored due to the difficulty in obtaining it in a pure form [7].

The sol-gel spin-coating method provides a flexible approach to synthesising TiO<sub>2</sub> thin films with controlled thickness, porosity, and crystallinity. This technique allows for precise manipulation of synthesis parameters, such as precursor concentration [8], annealing temperature [9], and ageing time [10], which directly affect the film's morphology and optical properties [11]. However, despite extensive research on TiO<sub>2</sub> thin films, limited studies have systematically investigated the influence of precursor concentration on the microstructural evolution and band gap tuning of TiO<sub>2</sub> films [12].

The morphology properties of the synthesised TiO<sub>2</sub> are characterised using techniques such as FE-SEM and AFM. FE-SEM stands for Field Emission Scanning Electron Microscope. This tool utilises the structure and shape of a thin film with an imaging magnification of up to 500,000 times. FE-SEM can also be used to measure the thickness of a sample via the cross-sectional method. FE-SEM is capable of providing high-quality imaging solutions in nanotechnology applications [13]. An AFM is a device used to capture high-resolution photographs of the surface topography of samples by applying extremely low forces [14]. The atomic resolution is achieved by utilising an optical lever to detect the interaction forces between the surface and a sharp tip positioned on a cantilever. This method allows for an exceptionally high level of precision. The force magnitude between the probe and sample is determined by two factors: the spring constant of the cantilever and the distance between the probe and the sample surface. The output image of the sample structure may be acquired from a variety of surfaces [15].

Ultraviolet-visible spectroscopy was employed to get data on the transmittance of the liquid sample by utilising ultraviolet and visible light wavelengths. Further, it is also referred to as absorption spectroscopy in the ultraviolet-visible spectral region, for which the transitions take place from the excited state to the ground state, whereby the absorption will be measured. However, the value of the optical band gap was relatively similar compared to the reported research (3.2 eV) due to the effect of the sample material being calcined at 550°C [16]. Instead of having a large optical band gap value, the optical band gap was close to the value reported in the research. The band gap shows an improvement in the thin film's quality, making the band gap for the 2.0 mL titanium (IV) butoxide concentration precursor suitable for use as a sensing film [17].

Despite the significant advancements in the synthesis of TiO<sub>2</sub> thin films, there remains limited research exploring the influence of varying concentrations of Titanium (IV) butoxide precursor on the morphological and optical properties of TiO<sub>2</sub> films. Previous studies have primarily focused on a narrow range of precursor concentrations, often overlooking the systematic effects of concentration variation on the morphology characteristics and optical performance of TiO<sub>2</sub> films [18]. This research, therefore, fills an important gap by systematically investigating the impact of three distinct concentrations of Titanium (IV) butoxide (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres) on the morphology, topography and optical properties of TiO<sub>2</sub> thin films. Understanding these relationships can significantly enhance the tailored design of TiO<sub>2</sub> thin films for advanced applications in areas such as solar cells [19] and sensors [20]. The objective of this study is to comprehensively evaluate how different precursor concentrations influence the key properties of TiO<sub>2</sub> thin films, contributing valuable insights for the optimisation of their fabrication for various technological applications.

This study introduces a systematic approach to optimising TiO<sub>2</sub> thin films by varying Titanium (IV) butoxide precursor concentrations, an aspect often overlooked in previous research. The key novel contributions include: the first novelty, systematic investigation of precursor concentration: Unlike prior studies that focus on a single or limited range of precursor concentrations, this research systematically examines three distinct concentrations (1.0 mL, 1.5 mL, and 2.0 mL) and their direct effects on film morphology, topography and optical behaviour. The second novelty enhanced understanding of morphological changes: By employing FE-SEM and AFM characterisation techniques, this study provides a detailed analysis of surface morphology, grain size evolution, and roughness variations with increasing precursor concentration. The third novelty is Band Gap Optimisation for Improved Performance: The study explores how changes in precursor concentration influence the optical band gap, with a particular focus on its implications for electronic and optoelectronic applications.

## 2.0 Methodology

Figure 1 shows the flowchart of the experiment design. Initially, the substrate preparation involved cutting a glass sheet measuring 2.5 cm × 2.5 cm, which served as the substrate. The glass was subjected to a cleaning process using acetone in an ultrasonic bath for 5 minutes at a temperature of 50 degrees Celsius. The cleaning technique was necessary to eliminate any organic contamination present on the surface of the glass substrate. Subsequently, the substrate was washed with deionised water. Next, the substrate was dried thoroughly using nitrogen gas (N<sub>2</sub>) [21]. Then, the research commenced by producing an optimised TiO<sub>2</sub> thin film by the sol-gel process.

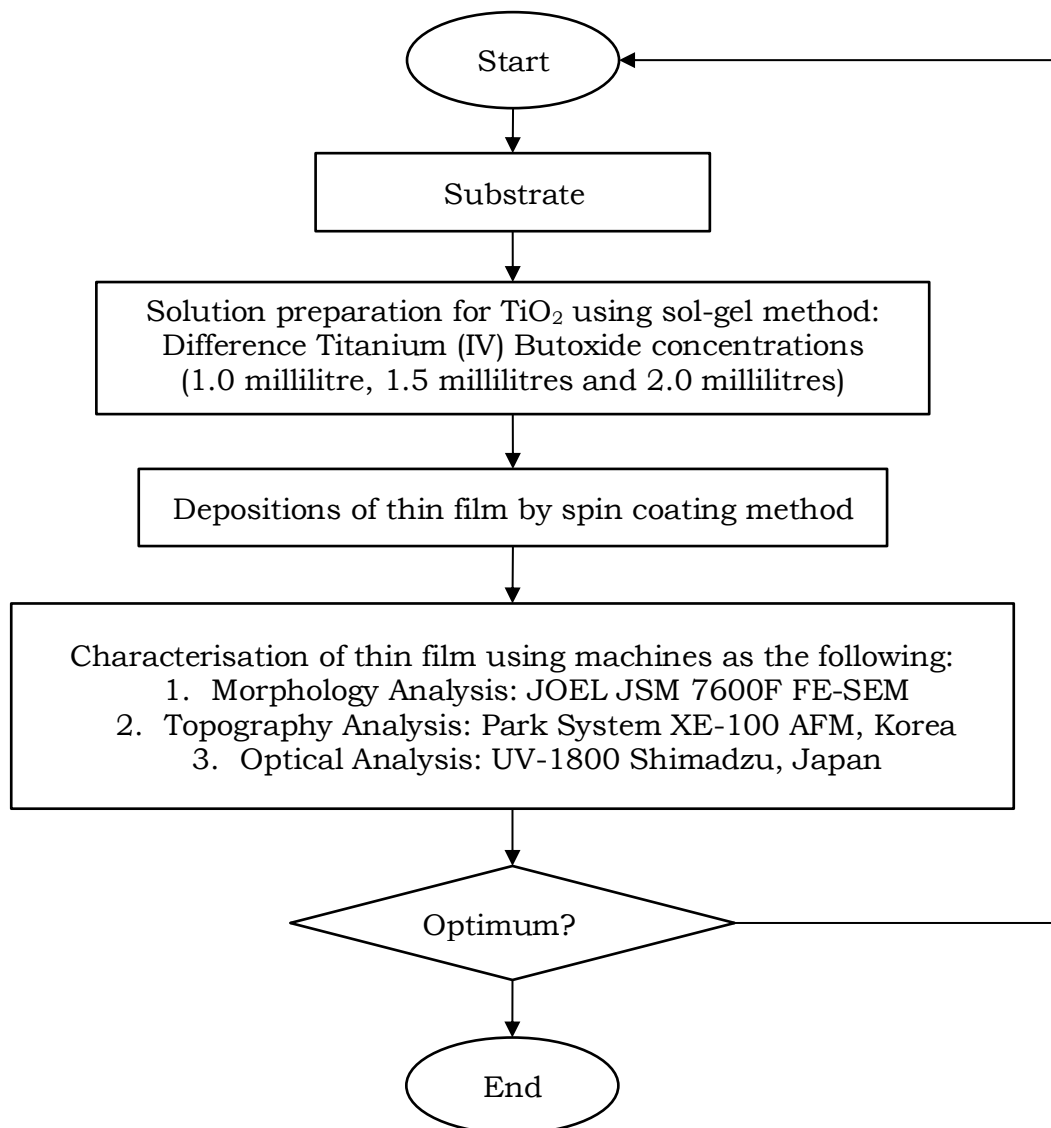


Figure 1: Flowchart of the experiment design

The first precursor parameter consisted of titanium (IV) butoxide concentration [Ti(OC<sub>4</sub>H<sub>9</sub>)<sub>4</sub>; Sigma-Aldrich, 97%] in volumes of 1.0 millilitres, 1.5 millilitres, and 2.0 millilitres. This parameter was chosen because the

weight of concentration was sequenced from 1.0 mL, 1.5 mL, and 2.0 mL. This sample was more uniform than others. Further, the annealing temperature was 500 °C. The pure TiO<sub>2</sub> solutions were mixed with other materials. The materials used in the experiment included 10 mL of ethanol (C<sub>2</sub>H<sub>5</sub>OH) as a solvent, 5 mL of glacial acetic acid (CH<sub>3</sub>CO<sub>2</sub>H) and 1 drop of Triton X-100 (Sigma-Aldrich) as a stabiliser to prevent precipitation in the solution. Additionally, a molar ratio of hydrochloric acid (HCl) to deionised water (10 mL:10 mL) was used, along with 3 mL of deionised water as a source of oxygen (O). The mixture consists of Titanium (IV) butoxide combined with 10 millilitres of ethanol, 5 mL of acetic acid catalysts, 1 drop of Triton X-100, and a molar ratio of hydrochloric acid (HCl) to deionised water of 10 mL:10 mL. These solutions were stirred at 40 °C for 30 minutes. The heating process was needed to halt the carbon chain and to increase the reaction between molecules.

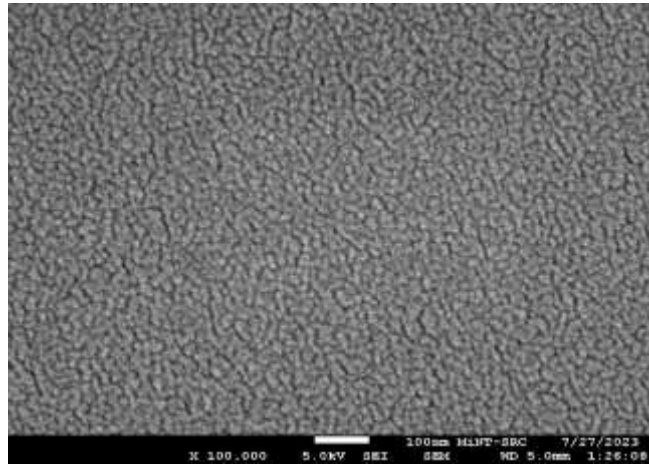
Subsequently, the solution underwent continuous stirring for 3 hours (referred to as the ageing phase) employing sol-gel synthesis under normal environmental circumstances. This resulted in the formation of a clear solution that solidified into a gel at room temperature. Then, the sol was applied onto the glass substrate using spin coating at a speed of 3000 rpm for 30 seconds, resulting in the deposition of five layers of a homogenous film. The TiO<sub>2</sub> solution was applied to the substrates by dropping it up to 10 times. The layers generated after spin-coating were subjected to preheating at a temperature of 100 °C for 5 minutes. The annealing process was conducted at a temperature of 500°C for 1 hour to enhance the crystallisation of all the layers [22]. The machines used were JOEL JSM 7600F Field Emission Scanning Electron Microscopy (FE-SEM), Park System XE-100 Atomic Force Microscopy (AFM) and Ultraviolet (UV)-1800 Shimadzu. FESEM was used to show the uniformity of the morphology and the accuracy of the thickness of the thin film. AFM was used to identify the highest surface roughness and the lowest grain size. Ultraviolet (UV)-1800 Shimadzu was used to identify the highest transmittance and the lowest optical band gap of the thin film.

### **3.0 Results and Discussion**

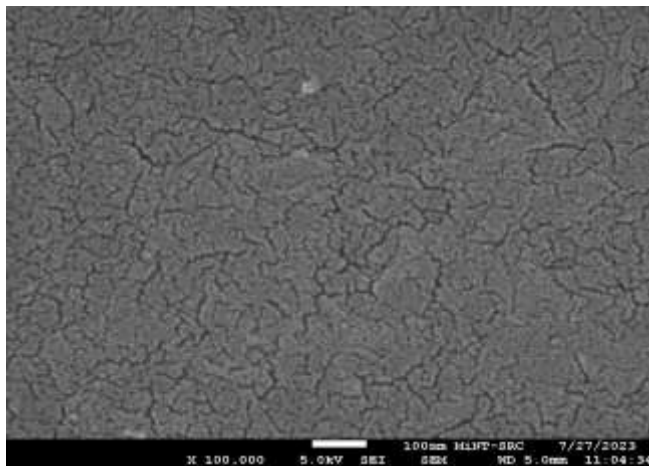
#### **3.1 Morphology Analysis**

The surface morphological characteristics of TiO<sub>2</sub> thin film were examined using Field Emission Scanning Electron Microscopy (FE-SEM) for varying concentrations of titanium (IV) butoxide precursor (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres). Figure 2 shows the surface morphology image of titanium (IV) butoxide film for different concentrations of precursors. Based on the outcome, the uniformity of 2.0 millilitres of titanium (IV) butoxide is superior to that of 1.0 millilitres and 1.5 millilitres of titanium (IV) butoxide. This uniformity might be due to the dissolved solution while ageing and the annealing temperature. The FE-SEM micrographs reveal a combination of nano-sized and micro-sized particles in the film formations [23].

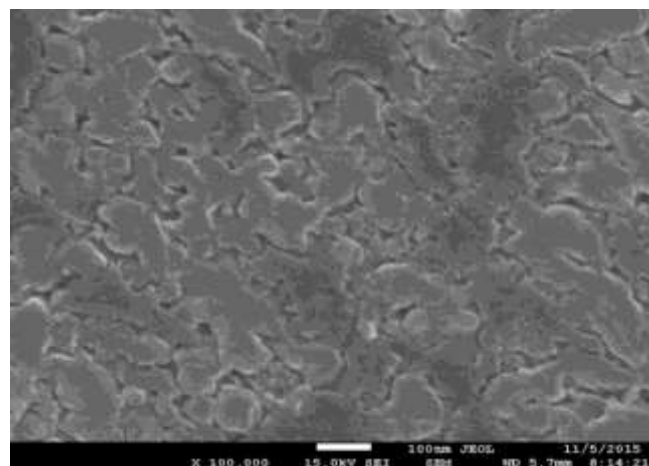




(a) 1.0 millilitre titanium (IV) butoxide



(b) 1.5 millilitres titanium (IV) butoxide

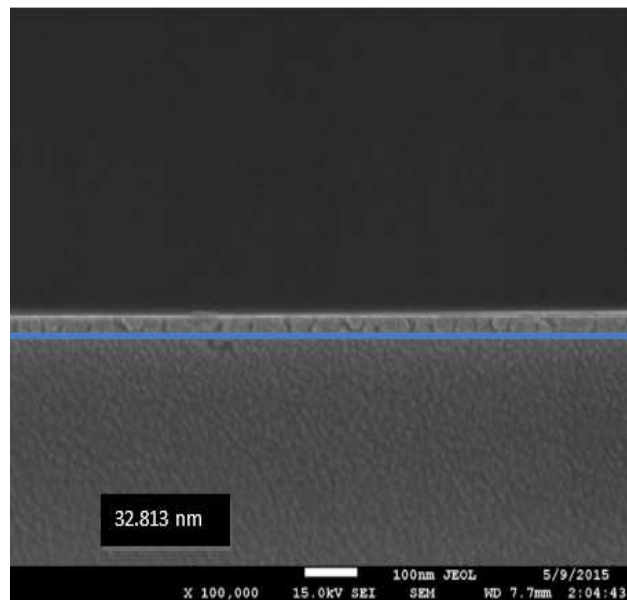


(c) 2.0 millilitres titanium (IV) butoxide

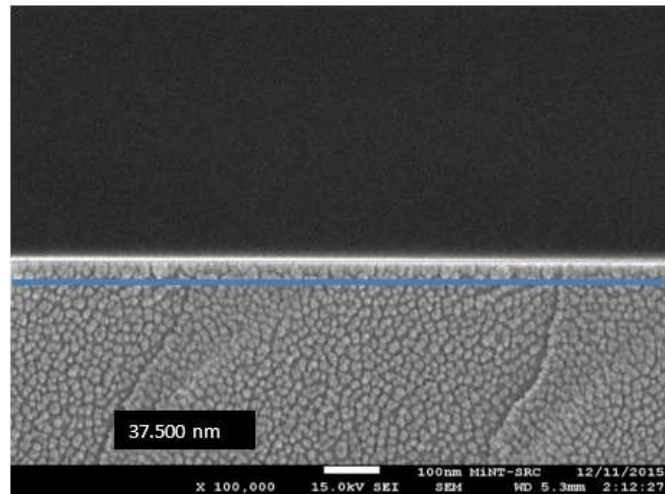
Figure 2: The surface morphology of a TiO<sub>2</sub> thin film was examined using a Field Emission Scanning Electron Microscope (FE-SEM) for various concentrations of titanium (IV) butoxide precursor (a) 1.0 millilitre of titanium (IV) butoxide (b) 1.5 millilitres of titanium (IV) butoxide (c) 2.0 millilitres of titanium (IV) butoxide

According to Radhia et al., the surface morphology was more uniform compared to the reported research (low solution concentration) due to a smooth surface and the increased titanium (IV) butoxide concentration. Instead of surface morphology being uniform, it can be enhanced with other methods to obtain different nanostructures [23].

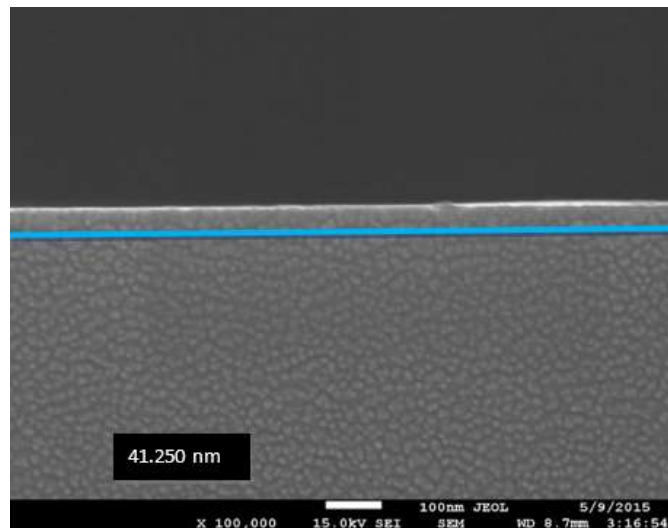
Figure 3 depicts the cross-sectional perspective of TiO<sub>2</sub> thin films that have undergone annealing at a temperature of 500°C. The measured thicknesses were 32.813 nm, 37.500 nm and 41.250 nm by FE-SEM for samples deposited using 1.0 millilitre, 1.5 millilitres, and 2.0 millilitres of titanium (IV) butoxide concentration precursor, respectively. The result showed an increment in thickness with the concentration of titanium (IV) butoxide. The thickness of the TiO<sub>2</sub> thin film correlated positively with the concentration of the titanium (IV) butoxide precursor. The relationship between thickness and titanium (IV) butoxide content is readily evident. However, the thickness value was low compared with the reported research (0.4 µm) due to the increased titanium (IV) butoxide concentration. Isrihetty et al. found that the TiO<sub>2</sub> particles were applied onto the substrate and served as the primary components of the final TiO<sub>2</sub> thin film. The TiO<sub>2</sub> thin film displayed a sleek structure at low concentrations of beginning titanium (IV) butoxide and a grainy structure at higher concentrations of initial titanium (IV) butoxide. A one-step procedure can be used to prepare a TiO<sub>2</sub> thin film with controllable morphologies by altering the initial precursor concentration in the solution.



(a) 1.0 millilitre titanium (IV) butoxide



(b) 1.5 millilitres titanium (IV) butoxide



(c) 2.0 millilitres titanium (IV) butoxide

Figure 3: The cross-sectional view of a TiO<sub>2</sub> thin film obtained using a Field Emission Scanning Electron Microscope (FE-SEM). The film was prepared with varying concentrations of titanium (IV) butoxide. Preceding or preliminary. 1.0 millilitre of titanium (IV) butoxide (b) 1.5 millilitres of titanium (IV) butoxide (c) 2.0 millilitres of titanium (IV) butoxide

### 3.2 Topography Analysis

The films produced with varying concentrations of titanium (IV) butoxide were examined using AFM surface image analysis, as depicted in Figure 4. By conducting this measurement, one can acquire supplementary data, including grain size, surface roughness, and a three-dimensional (3D) visualisation of the films.

Table 1 shows the characteristics of the titanium (IV) butoxide thin film at different precursor concentrations. An increase in grain size leads to a pile-up at grain boundaries. Nevertheless, the grain size was abruptly increased



because of diffusion occurring as expected, resulting in grain boundary grooving and an excess of material on the surface adjacent to the groove at the boundaries. However, the roughness of the thin film is increasing from 2.112 nm (1.0 millilitre) to 3.194 nm (2.0 millilitres) due to the increment of arrangement atoms in the solution [22].

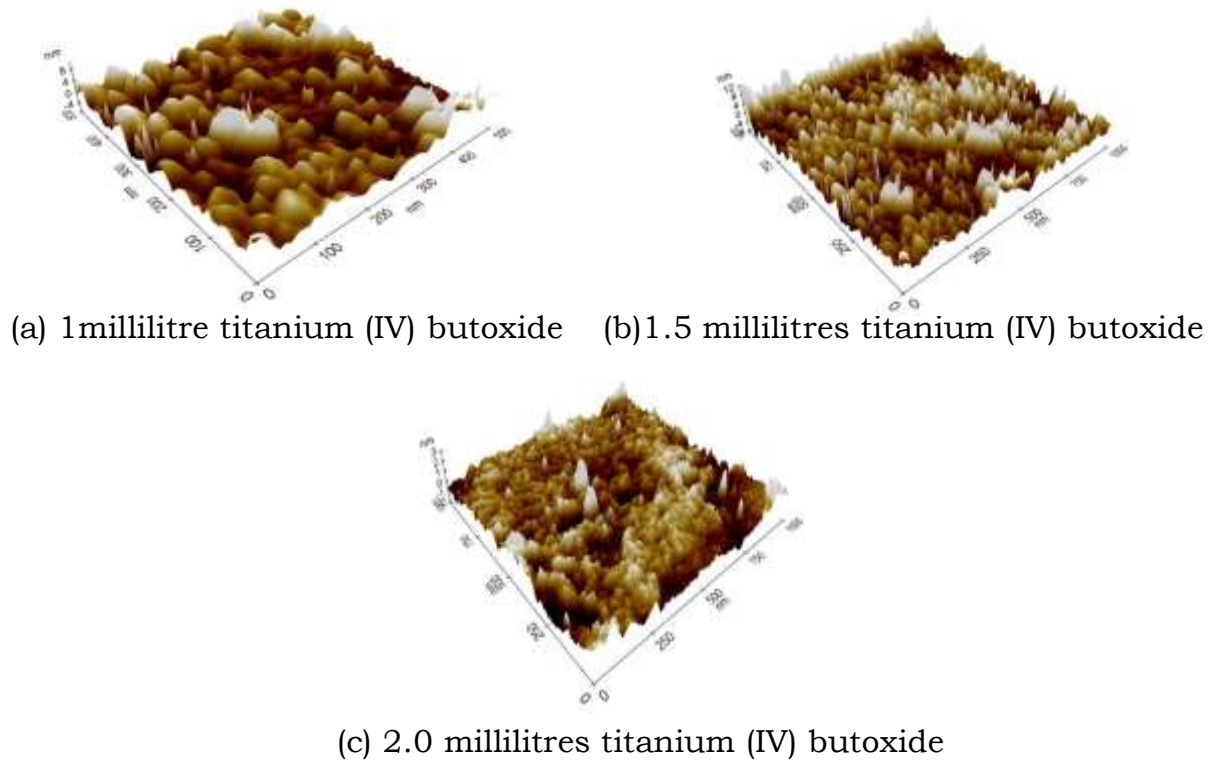


Figure 4: The surface topography of a TiO<sub>2</sub> thin film was examined using Atomic Force Microscopy (AFM) for various concentrations of titanium (IV) butoxide precursor. (a) 1.0 millilitre of titanium (IV) butoxide, (b) 1.5 millilitres of titanium (IV) butoxide (c) 2.0 millilitres of titanium (IV) butoxide

Table 1: The thickness, grain size, and roughness of a TiO<sub>2</sub> thin film were measured using varied concentrations of titanium (IV) butoxide precursor (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres)

Titanium Butoxide con. (mL)	Thickness (nm)	Grain Size (nm)	Roughness (nm)
1.0	32.813	40.085	2.112
1.5	37.500	40.664	2.312
2.0	41.250	44.084	3.914

The AFM images demonstrate a correlation between the concentration of titanium (IV) butoxide precursor and the observed alterations in topography. According to Isrihetty et al., the factors affecting the increase in roughness were the accumulation of nanoparticles and the creation of high roughness width when the titanium (IV) butoxide concentration precursor increased. The bright spots in the AFM images at 2.0 mL show the formation of the smaller

nanoparticles, while black spots correspond to the etched area by oxygen. Therefore, 2 mL of titanium (IV) butoxide precursor is the best precursor to be used as a sensing film. The film is quite uniform; the increased uniformity caused the roughness to increase (roughness = 3.914) compared with the reported research (roughness = 1.690) [22]. Instead of the roughness value being smaller, the roughness was approximately close to the reported research.

### **3.3 Optical Analysis**

In the optical properties, only transmittance was measured by different concentrations of titanium (IV) butoxide precursor (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres) were analysed using a UV-vis machine. Figure 5 shows the transmittance for the thin films with titanium (IV) butoxide of 1.0 mL, 1.5 mL, and 2.0 mL in the spectra region of 340-1000 nm. Glass substrates were utilised in this experiment to prevent the influence of the absorption edge of the substrate. The results show a slight decrease in transmittance with an increase in titanium (IV) butoxide concentration.

The optimum TiO<sub>2</sub> thin film spectra exhibited high visible transmittance ranging from 77.611% to 100.018%, for a titanium (IV) concentration of 2.0 mL. This high transmittance is attributed to the uniformity of the thin film, resulting in stable transmittance levels from the initial to the highest values. However, for 1.0 mL and 1.5 mL titanium (IV) butoxide solutions, the thin films were less uniform due to the non-homogeneous solution, resulting in slightly lower initial transmittance compared to the 2.0 mL solution. The thin film with 1.5 mL titanium (IV) butoxide exhibited transmittance ranging from 75.507 % to 96.945 %, showing the second-lowest initial transmittance. Similarly, the thin film with 1.0 mL titanium (IV) butoxide showed initial transmittance ranging from 71.759% to 88.545%, indicating the lowest transmittance due to XRD results affecting the crystallinity of the thin film, which less the amount of the anatase phase [22]. However, the transmittance value did not initially start at zero compared to the reported research, likely due to the effect of the glass substrate. According to Radhia et al., the TiO<sub>2</sub> thin films with high-quality transmittance exhibited different roughness conforming to interference fringes [23].

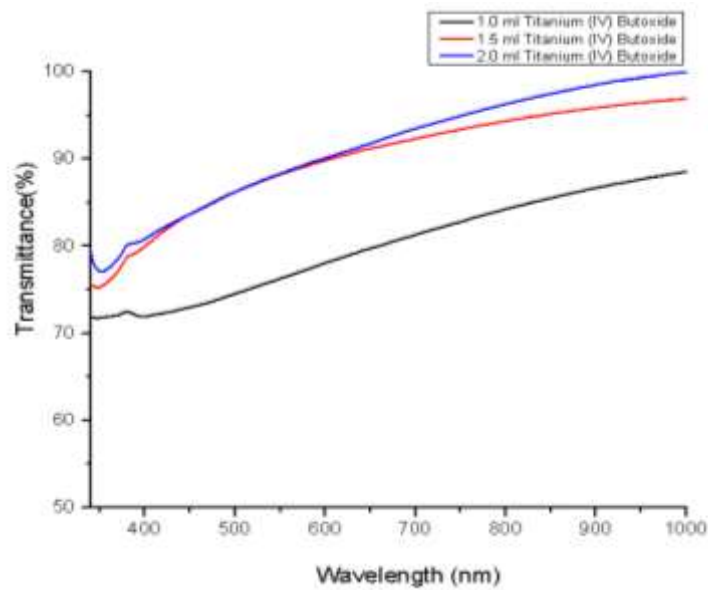


Figure 5: UV-Vis transmittance of TiO<sub>2</sub> thin film with different titanium (IV) butoxide concentrations precursor (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres)

The optical band gap of TiO<sub>2</sub> thin film for three different titanium (IV) butoxide concentration precursors is shown in Figure 6. The resulting band gap values were 3.40 eV, 3.30 eV, and 3.20 eV, which corresponded to titanium (IV) butoxide concentration precursors of 1.0 mL, 1.5 mL, and 2.0 mL, respectively. For the pure film with 1.0 mL titanium (IV) butoxide concentration, the band gap was 3.40 eV due to higher roughness, making it difficult for UV light to pass through the sample despite the decreased grain size.

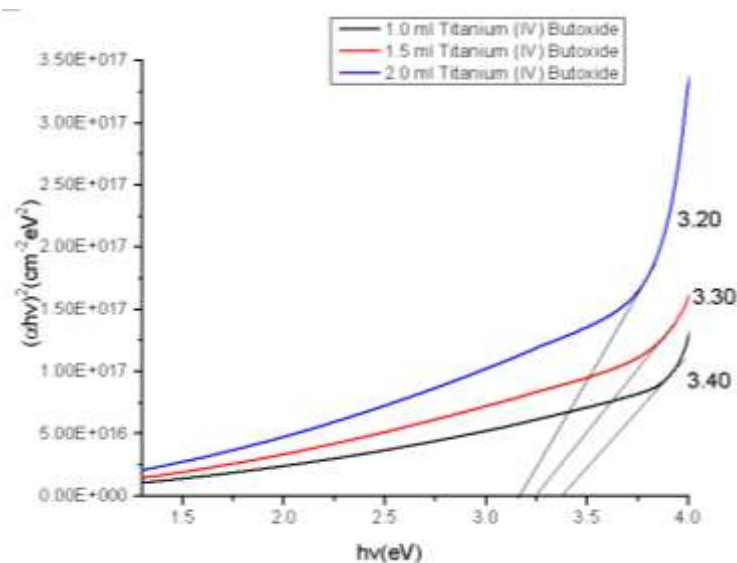


Figure 6: Optical band gap of TiO<sub>2</sub> thin film with different titanium (IV) butoxide concentrations precursor (1.0 millilitres, 1.5 millilitres, and 2.0 millilitres)

For the 2.0 mL titanium (IV) butoxide concentration precursor, the band gap was 3.20 eV because of the uniform thin film, allowing UV light to easily pass through and reducing carrier scattering at grain boundaries. The band gap for the 1.5 mL titanium (IV) butoxide concentration precursor, with the lowest roughness but less uniform thin film, was 3.30 eV, slightly higher than that of the 2.0 mL. However, the value of the optical band gap was relatively similar compared to the reported research (3.095 eV) due to the effect of the glass substrate. Instead of having a large optical band gap value, the optical band gap was fairly close to the value reported in the research. The decrease in the band gap indicates an improvement in the thin film's quality, making the band gap for the 2.0 mL titanium (IV) butoxide concentration precursor suitable for use as a sensing film. Therefore, the result agrees with the XRD result, indicating that the crystallite size increases with film thickness, contributing to further enhancement of order and localisation within the optical band gap due to a decrease in the optical band gap [24].

The starting transmittance for 2 mL titanium butoxide was due to the Ti atoms contributing to the roughness ( $R_a = 3.914$  nm) value and the intensity of the crystallite of the anatase peak, which could influence the scattering of light. The light scattering intensity increases as the substrate roughness increases. The FE-SEM micrographs reveal a combination of nano-sized and micro-sized particles in the film formations, which affects the light scattering properties.

#### **4. Conclusion**

This research aimed to investigate the effect of varying precursor concentrations of Titanium (IV) butoxide (1.0 mL, 1.5 mL, and 2.0 mL) on the morphological and optical properties of TiO<sub>2</sub> thin films fabricated via the sol-gel spin-coating method. The primary objective was to optimise the precursor concentration to achieve uniform, high-quality thin films with improved surface morphology, topography and optical characteristics suitable for applications in gas sensing and optoelectronic devices. The experimental results demonstrated that the TiO<sub>2</sub> thin film synthesised using a 2.0 mL concentration of Titanium (IV) butoxide exhibited the most uniform coating with well-distributed nano-sized grains, minimal defects, and improved surface roughness. FE-SEM analysis confirmed the formation of nano- and micro-sized particles, with increasing precursor concentration leading to grain growth and enhanced film uniformity. AFM characterisation revealed a roughness value of 3.914 nm, indicating a smoother and more homogeneous film surface compared to the lower-concentration samples. UV-Vis spectroscopy analysis showed that the optical band gap of the TiO<sub>2</sub> thin film fabricated with a 2.0 mL precursor concentration was reduced to 3.20 eV, which is advantageous for improving charge carrier mobility in optoelectronic applications. Additionally, the films exhibited high optical transmittance, making them suitable for integration into solar cells and transparent electronic devices. These findings validate the research hypothesis that optimising precursor concentration plays a critical role in tailoring TiO<sub>2</sub> thin

films for enhanced performance. The potential for commercialisation of these optimised TiO<sub>2</sub> thin films is significant, particularly in the development of high-performance gas sensors, solar cells, and optoelectronic devices. The ability to fine-tune the film morphology, topography, and optical properties using an easily scalable and cost-effective sol-gel spin-coating technique makes it highly attractive for industrial applications. Furthermore, the uniform and well-structured TiO<sub>2</sub> thin films could be incorporated into functional coatings for self-cleaning surfaces, anti-reflective coatings, and photocatalytic applications, expanding their commercial viability. While this study has successfully demonstrated the impact of precursor concentration on TiO<sub>2</sub> thin films, further research can be conducted to enhance their properties and applications: The first suggestion is doping and composite materials: investigating the incorporation of dopants such as noble metal nanoparticles (e.g., Au, Ag, Pt) or transition metal oxides to enhance gas sensing capabilities and charge transport properties. The second suggestion is p-n junction formation: Exploring the combination of TiO<sub>2</sub> thin films with p-type semiconductors to develop heterojunction-based devices for improved gas sensing and optoelectronic applications.

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### Author Contributions

**Nor Damsyik Mohd Said:** Conceptualisation, Methodology, Software, Writing – Original Draft, Writing – Reviewing & Editing;

**Mohd Zainizan Sahdan:** Data Curation, Validation, Supervision;

**Feri Adriyanto:** Validation, Writing – Reviewing & Editing.

### Conflicts of Interest

The manuscript has not been published elsewhere and is not under consideration by any other journals. All authors have approved the manuscript, agreed to its submission, and declare no conflicts of interest.

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