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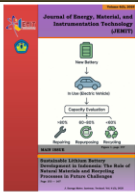

Effect of Layer Deposition and Thermal Treatment on the Structural, Optical, and Electrical Characteristics of Spin-Coated $CaTiO_3$ Thin Films

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Abstract

This study investigates the effect of coating repetition and annealing temperature on the structural, optical, and electrical characteristics of calcium titanate ($CaTiO_3$) thin films synthesized via a sol-gel spin coating method. The number of coatings (2, 4, 6, and 8 layers) and annealing temperatures (250°C, 350°C, and 450°C) were varied to optimize the characteristics. X-ray diffraction analysis indicated an improvement in crystallinity with increasing annealing temperature, as evident from the growing diffraction peaks. UV-Vis spectroscopy revealed a decrease in the optical band gap from 3.741 eV to 3.554 eV with increasing coating layers and thermal treatment, suggesting enhanced optical absorption. Current–voltage measurements using a two-point probe method generally showed linear conduction, indicative of ohmic behavior, with one sample exhibiting a mild nonlinear response at higher bias. The results confirm that process parameters significantly influence the functional properties of $CaTiO_3$ thin films. This fundamental study provides a foundation for employing $CaTiO_3$ as a functional layer in perovskite-based photovoltaic devices, paving the way for further device integration and optimization.

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Abstrak

Penelitian ini mengkaji pengaruh pengulangan pelapisan dan suhu annealing terhadap sifat struktural, optik, dan listrik dari film tipis kalsium titanat ($CaTiO_3$) yang disintesis melalui metode sol-gel spin coating. Jumlah pelapisan (2, 4, 6, dan 8 lapis) serta suhu annealing (250°C, 350°C, dan 450°C) divariasikan untuk mengoptimalkan karakteristik film. Analisis XRD menunjukkan peningkatan kristalinitas seiring naiknya suhu annealing, yang ditandai dengan menguatnya puncak-puncak difraksi. Spektroskopi UV-Vis menunjukkan penurunan nilai celah pita optik dari 3,741 eV menjadi 3,554 eV seiring bertambahnya jumlah pelapisan dan perlakuan termal, yang mengindikasikan peningkatan daya serap optik. Pengukuran arus-tegangan menggunakan metode probe dua titik secara umum menunjukkan konduksi linear yang menandakan sifat ohmik, dengan satu sampel memperlihatkan sedikit penyimpangan nonlinier pada tegangan tinggi. Hasil penelitian ini menegaskan bahwa parameter proses secara signifikan memengaruhi sifat fungsional film tipis $CaTiO_3$. Kajian dasar ini memberikan landasan bagi pemanfaatan $CaTiO_3$ sebagai lapisan fungsional dalam perangkat fotovoltaik berbasis perovskit, serta membuka peluang untuk integrasi dan optimasi perangkat lebih lanjut.

1. Introduction

The growing global demand for sustainable and environmentally friendly energy sources has intensified the search for alternatives to fossil fuels. Among these, solar energy stands out as one of the most abundant and promising renewable resources (Pourasl, et all., 2023). Solar energy harvesting is typically carried out using optoelectronic devices known as photovoltaic (PV) cells, which convert sunlight directly into electricity through the photovoltaic effect (Kadish & Guilard, 2018). Continuous advancements by researchers in the field of photovoltaics

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have led to the emergence of a new class of solar cells based on perovskite materials (Kim, et all., 2020). Calcium titanate ($CaTiO_3$) perovskite has gained significant attention due to its wide band gap value (Manjunath & Thimmanna, 2016), thermal stability (Fang, et all., 2025), and excellent electronic performance (Portia, 2020). These characteristics make it a promising candidate for optoelectronic devices, especially as a buffer or transport layer in perovskite solar cells (Patni, et all., 2022).

In recent years, various synthesis methods have been explored to fabricate high-quality $CaTiO_3$ thin films, including pulsed laser deposition (Wang, et all., 2019), sputtering (Ikeuchi, et all., 2014), chemical vapor deposition (Sato, et. All., 2007), and sol–gel techniques (Torimtubun, et all., 2018). Among them, the sol–gel spin coating method has emerged as a low-cost, scalable, and controllable approach for producing uniform oxide thin films on conductive or transparent substrates (Aslam, et all., 2021). Despite its advantages, challenges remain in achieving optimal film crystallinity, uniformity, and electronic performance through spin coating, especially when processing conditions such as coating repetition and annealing temperature are not well optimized (Abbas, et all., 2020). Previous studies have mostly focused on either structural or optical properties of $CaTiO_3$ films, often using a single set of process parameters. There is limited systematic research exploring how the number of coating layers and thermal treatment simultaneously affect the structural, optical, and electrical behavior of $CaTiO_3$ thin films. Additionally, only a few studies report their electrical conduction characteristics, leaving a knowledge gap in understanding their potential as functional layers in photovoltaic devices (Wendari, et all., 2024).

This study addresses the need for a comprehensive analysis of $CaTiO_3$ thin films synthesized by the sol–gel spin coating method with controlled variations in the number of coatings and annealing temperatures. The research aims to evaluate the crystallinity, band gap energy, and electrical conduction behavior of the resulting films (Huang, et all., 2017). The hypothesis underlying this work is that increasing thermal energy and layer deposition would enhance film quality, reduce band gap, and improve conductivity (Hossain, et all., 2017). By understanding these effects, this study contributes to optimizing $CaTiO_3$ thin films for future use in perovskite-based optoelectronic applications (Sahoo, et all., 2018).

2. Research Methods

This research employed an experimental design to synthesize and characterize $CaTiO_3$ thin films using the sol–gel spin coating method. The process involved two main variables: the number of coating repetitions (2, 4, 6, and 8 layers) and the annealing temperature (250°C, 350°C, and 450°C). **Figure 1** illustrates the synthesize process of $CatiO_3$. The precursor solution was prepared by dissolving calcium chloride dihydrate ($CaCl_2{\cdot}2H_2O$) and titanium (IV) isopropoxide ($Ti[OCH(CH_3)_2]_4$) in absolute methanol with the addition of diethanolamine (DEA) as a stabilizer. The solution was stirred at room temperature for 60 minutes until homogeneous. The resulting sol was aged 24 hours before deposition.

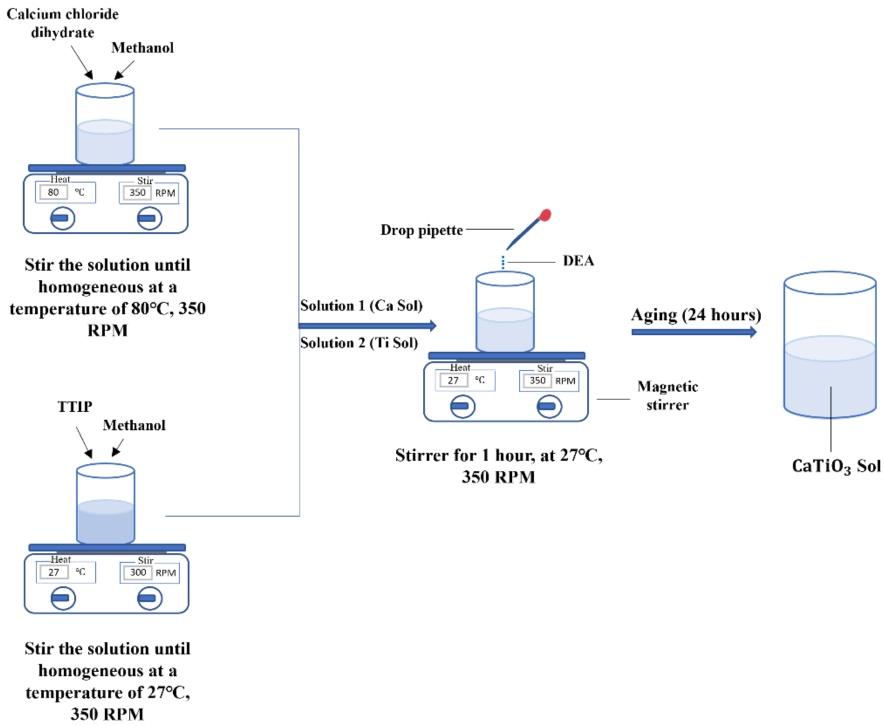

Figure 1. Schematic diagram of synthesizing $CaTiO_3$ sol

Figure 2 shows film deposition process that was carried out on indium tin oxide (ITO)-coated glass substrates. Before coating, the substrates were ultrasonically cleaned with ethanol and aquadest. The spin coating process was performed at 2000 rpm for 30 seconds for each layer, followed by drying on a hot plate at 100°C for 5 minutes after each coating step. After reaching the desired number of layers, the films were annealed at the target temperature (250°C, 350°C, or 450°C) for 1 hour in ambient air.

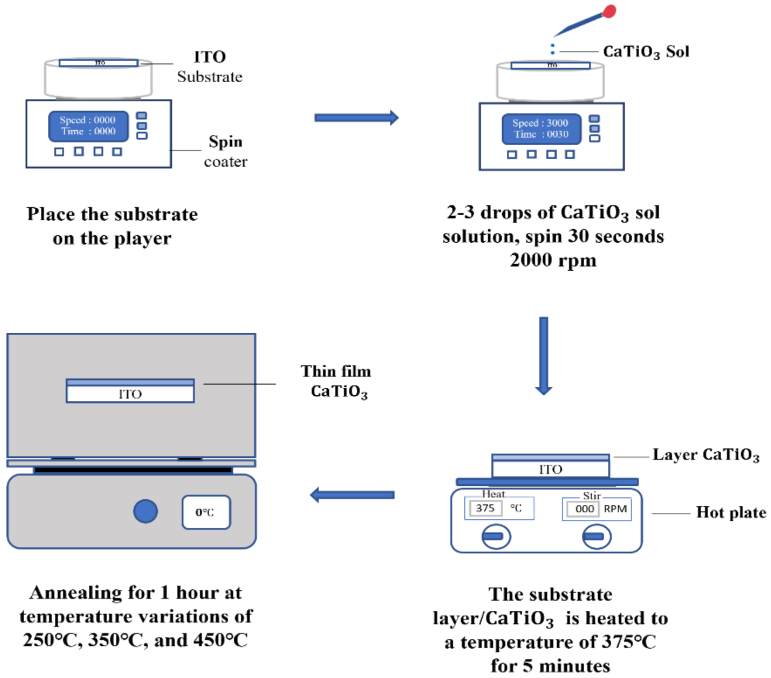

Figure 2. Schematic diagram of film deposition process

The structural properties of the films were analyzed using X-ray diffraction (XRD) with Cu-Kα radiation (λ = 1.5406 Å). Optical properties were characterized using UV-Vis spectroscopy to determine the absorbance spectra and calculate the optical band gap using Tauc plot analysis. Electrical properties were measured using a two-point probe method with a source meter to obtain the current–voltage (I–V) characteristics. All measurements were conducted at room temperature.

3. Results and Discussions

To optimize the fabrication of $CaTiO_3$ thin films via the spin coating technique, three critical process parameters were systematically evaluated: droplet volume, spin speed, and preheating temperature. Among the tested droplet volumes, dispensing three drops of precursor solution resulted in the most uniform and continuous film, avoiding the excessive thinness observed with two drops and the localized accumulation seen with four drops. Spin speed significantly influenced film distribution, where 3000 rpm produced the most homogeneous surface coverage, while 1000 rpm led to excessively thick and uneven films. A speed of 2000 rpm yielded an intermediate thickness with acceptable uniformity, making it a practical compromise between coverage and material economy.

Preheating temperature also played a crucial role in determining the film's crystallinity and mechanical integrity. A preheating temperature of 350°C was found to promote optimal crystallization without introducing thermal stress. In contrast, films preheated at 250°C exhibited incomplete phase formation, while those treated at 450°C showed a tendency to crack, likely due to rapid solvent evaporation and thermal mismatch. Taken together, the combination of three droplets, a spin speed of 2000 rpm, and a preheating temperature of 350°C emerged as the most effective parameter set for producing high-quality $CaTiO_3$ thin films with desirable structural and morphological characteristics. The following figures (Fig. 3 & 4) show the resulting thin films deposited on the ITO substrates with varying conditions based on the number of coating repetitions and annealing temperature.

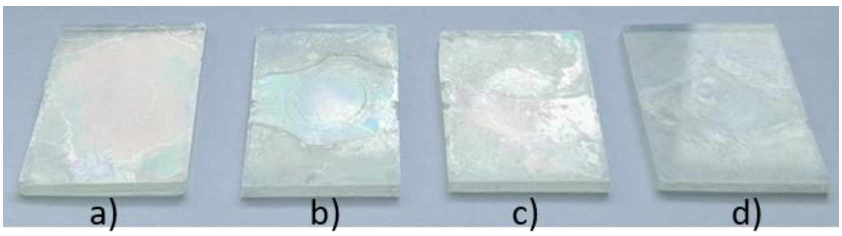

Figure 3. Surface appearance of $CaTiO_3$ thin films deposited with varying numbers of coating layers: a) 2 layers; b) 4 layers; c) 6 layers; and d) 8 layers.

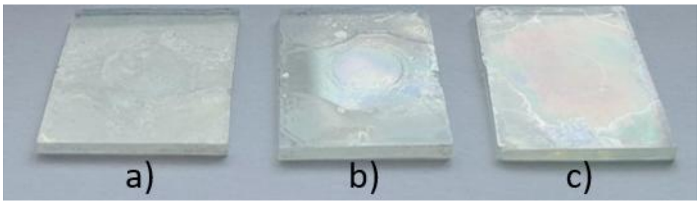

Figure 4. Surface appearance of $CaTiO_3$ thin films deposited with varying annealing temperatures: a) 250°C, b) 350°C, and c) 450°C.

The surface appearance of $CaTiO_3$ thin films varied noticeably with both the number of coating layers and the annealing temperature. As shown in **Figure 3**, increasing the number of coatings resulted in changes in film coverage and the appearance of interference patterns, indicating progressive alterations in thickness and surface uniformity. The sample with four layers appeared most uniform, while excessive coating led to localized accumulation and reduced optical clarity. Similarly, **Figure 4** illustrates that films annealed at higher temperatures exhibited more prominent interference colors, with the sample annealed at 350°C showing the most defined and uniform appearance. These visual differences reflect the influence of thermal energy on crystallization and stress distribution within the film.

3.1 Structural Properties (XRD)

The X-ray diffraction (XRD) was plotted to observe the diffraction pattern of the material, using *Origin Pro 9.0* for graphical representation as shown in **Figure 5**.

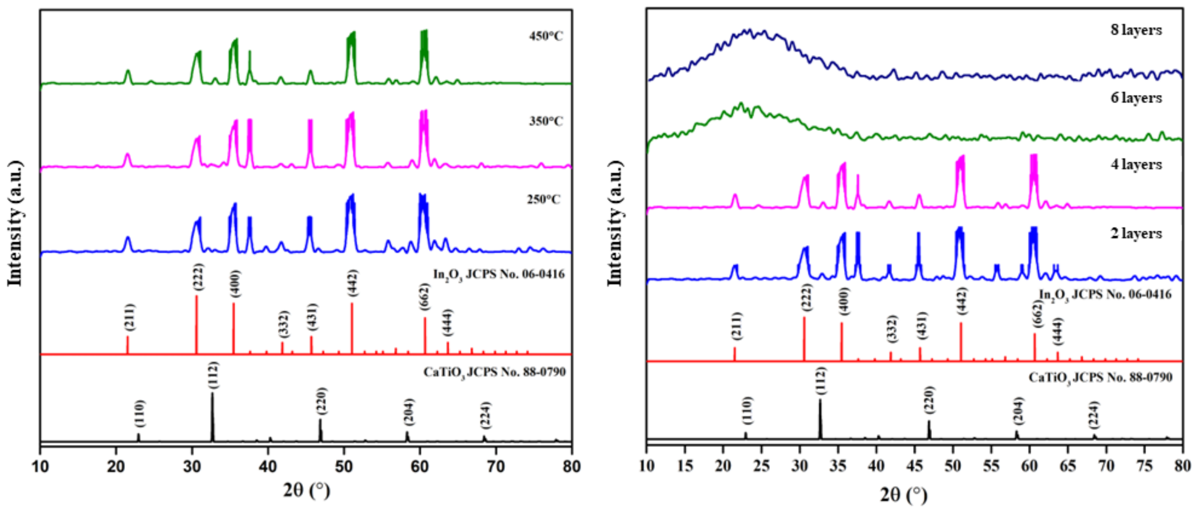

Figure 5. Diffraction peaks of $CaTiO_3$ thin films deposited with varying annealing temperatures (left) and varying numbers of coating layers (right)

The primary diffraction peaks correspond to specific Bragg angles (2θ), where each peak represents constructive interference from crystallographic planes within the material (Callister & Rethwisch, 2016). Bragg's law, shown in Equation (1), is used to calculate the interplanar spacing (d_{hkl}) in crystalline materials.

$$d_{hkl} = \frac{n\lambda}{2\sin\theta} \tag{1}$$

where d_{hkl} is the spacing between adjacent crystal planes, λ is the wavelength of the X-ray radiation, n is the diffraction order, and θ is the angle of diffraction (in radians). In addition to interplanar spacing, the average crystallite size was estimated using the *Debye–Scherrer* equation (eq. 2), which relates the broadening of the diffraction peaks to the size of coherent scattering domains within the sample.

$$D = \frac{0{,}9\,\lambda}{\beta\cos\theta} \tag{2}$$

where D is the average crystallite size, 0,9 is the shape factor for spherical crystallites, β is the full width at half maximum (FWHM) of the diffraction peak (in radians), λ is the wavelength of the X-ray radiation, and θ is the angle of diffraction (in radians).

The XRD patterns revealed variations in crystallinity with different processing conditions. As shown in **Figure 5** (left panel), the diffraction peaks corresponding to $CaTiO_3$, those indexed to the (112) and (204) planes, became more pronounced as the annealing temperature increased from 250°C to 450°C. This indicates that thermal energy enhances atomic mobility and crystal growth during annealing. In addition to the thermal treatment, the number of deposited layers—which directly influences film thickness—also affected the crystallinity and phase visibility of $CaTiO_3$ films, as shown in **Figure 5** (right panel). Samples with 2 and 4 coating layers exhibited clearer diffraction peaks, while those with 6 and 8 layers showed reduced peak intensity and increased broadening. This trend suggests that thicker films may introduce additional microstructural complexity, such as increased grain boundary density, internal stress, or surface roughness, which can contribute to partial peak suppression or X-ray scattering losses.

Table1. XRD analysis results for selected $CaTiO_3$ thin films.

Sample variations	hkl	2θ (°)	FWHM (°)	D (nm)	Microstrain
2 layers	112	32.88	0.837	9.901	0.0124
4 layers	112	33.00	1.009	8.215	0.0149
6 layers	-	-	-	-	-
8 layers	-	-	-	-	-
250℃	204	58.85	1.360	6.709	0.0105
350℃	204	58.96	0.898	10.166	0.0069
450℃	112	33.00	1.009	8.215	0.0149

The resulting diffraction patterns were then analyzed to calculate the key crystallographic parameters as shown in **Table 1**. The sample with 2 coating layers exhibited a narrower FWHM (0.837°), resulting in a larger crystallite size of 9.901 nm and lower microstrain (0.0124). Increasing the number of layers to 4 caused the FWHM to broaden (1.009°), indicating reduced crystallite size (8.215 nm) and slightly higher microstrain (0.0149). In contrast, the 6- and 8-layers samples don't show diffraction peaks, and hence not included in the calculations. This suggests that additional coating may introduce more internal lattice distortion or stacking disorder, which hinders long-range crystallization and promotes strain accumulation. These findings support the earlier qualitative observation that excessive layering may reduce structural coherence. For the temperature variation, the film annealed at 250°C shows the broadest peak (FWHM = 1.360°), corresponding to the smallest crystallite size (6.709 nm) and relatively higher microstrain (0.0105). Upon annealing at 350°C, the FWHM significantly narrows to 0.898°, reflecting a marked increase in crystallite size (10.166 nm) and the lowest microstrain value (0.0069), indicating enhanced crystallization and reduced defect density. However, further annealing at 450°C does not yield additional improvement; the FWHM increases again to 1.009°, reducing crystallite size to 8.215 nm with a microstrain rise to 0.0149—similar to the 4-layer sample. This behavior may be due to over-annealing, which can lead to grain coalescence, stress relaxation anomalies, or even surface cracking, as hinted in prior visual and diffraction results.

Calcium titanate ($CaTiO_3$) adopts a perovskite-type structure with the general chemical formula ABX_3 and is known to undergo several temperature-dependent phase transitions. At room temperature, $CaTiO_3$ crystallizes in an orthorhombic structure with the space group *Pbnm* and refined lattice parameters of a = 5.3789 Å, b = 5.4361 Å, and c = 7.6388 Å, as reported by Ali and Yashima (2005). These values serve as a reliable structural reference for confirming phase purity in synthesized thin films. In the context of thin film synthesis, the processing temperatures applied in this study (≤450°C) remain well within the orthorhombic phase stability window, thus the detected diffraction peaks are consistent with the orthorhombic *Pbnm* structure.

3.2 Optical Properties (UV-Vis Spectrophotometry)

Optical properties of the $CaTiO_3$ thin films were carried out using UV-Vis spectrophotometry in the wavelength range of 200 to 500 nm. This measurement aimed to determine the absorbance behavior and estimate the optical band gap of the films utilizing the following equation:

$$(\alpha h v)^2 = A\left(h v - E_g\right) \quad (3)$$

The recorded spectra provided a plot of absorbance as a function of wavelength, as depicted in **Figure 6**. It shows strong absorption in the ultraviolet range (250–325 nm), which is consistent with the wide band gap nature of $CaTiO_3$. The left panel shows that films annealed at 250°C exhibit the highest absorbance intensity, peaking sharply near 290–310 nm, which is characteristic of strong UV absorption in wide band gap perovskite oxides like $CaTiO_3$. As the annealing temperature increases to 350°C, the absorbance peak becomes broader and slightly reduced in magnitude, indicating microstructural evolution and changes in film density or surface morphology. At 450°C, the overall absorbance further decreases, and the spectrum flattens—suggesting possible grain coarsening, densification, or the formation of microcracks or voids that reduce effective light interaction. The subsequent decline in optical absorbance with increasing annealing temperature reflects a typical trade-off between enhanced crystallinity and the onset of thermally induced morphological degradation.

Meanwhile, increasing the number of layers, as depicted in **Figure 6** (right panel), gradually reduces the absorbance intensity across the spectrum. This trend shows that in sol–gel spin-coated systems, excessive layering without sufficient annealing may lead to poor film adhesion, increased surface roughness, light scattering, or partial delamination. Furthermore, the red shift of the absorbance edge with increasing layers implies a narrowing of the optical band gap, consistent with enhanced packing density and possible electronic structure modification. The films with 4 layers offer a balance between absorbance strength and smooth spectral features, suggesting an optimal condition for uniform film formation.

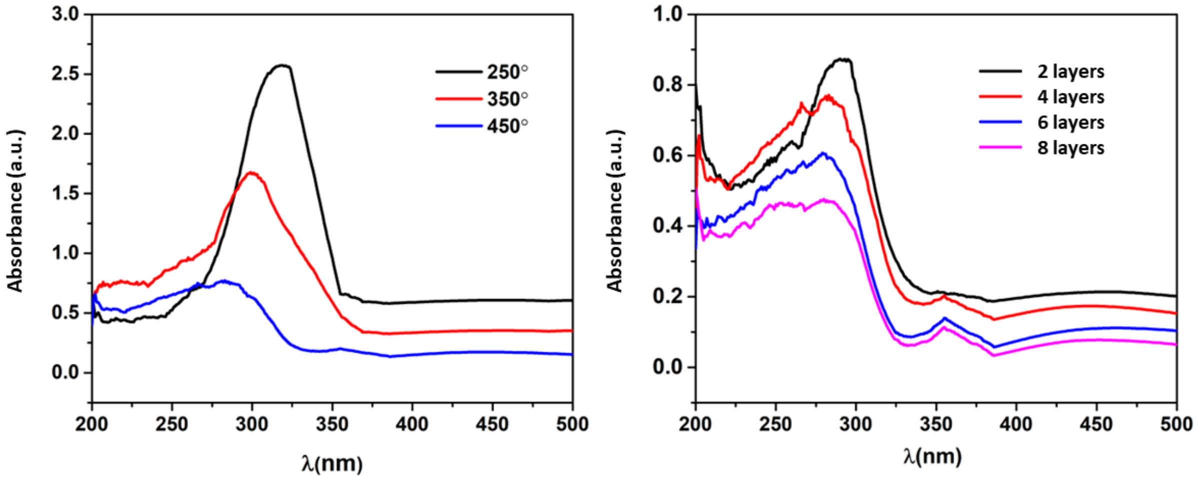

Figure 6. UV-Vis absorbance spectra of $CaTiO_3$ thin films with varying annealing temperature (left) and number of coating layers (right).

The optical band gap values of the $CaTiO_3$ thin films were estimated using the Tauc method, as shown in **Figure 7**. The results clearly indicate that annealing temperature and number of coating layers significantly influence the band gap energy of the films. On the left panel, a progressive widening of the band gap is observed from 3.418 eV at 250°C to 3.701 eV at 450°C, indicating a blue-shift in optical absorption. Similarly, the right panel shows that increasing the number of coating layers also results in a modest band gap widening, from 3.688 eV (2 layers) to 3.741 eV (8 layers). These band gap shifts confirm that both thermal treatment and deposition strategy play crucial roles in tuning the optical properties of sol–gel-derived $CaTiO_3$ thin films.

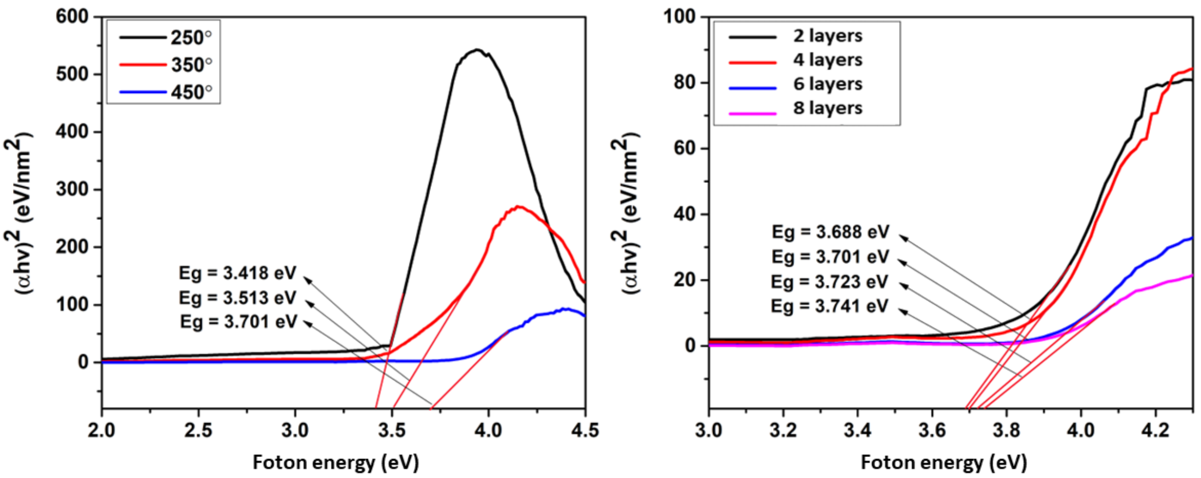

Figure 7. Tauc plot for $CaTiO_3$ thin films deposited at varying annealing temperatures (left) and different coating layers (right)

3.3 Electrical Properties (I–V Measurement)

The electrical characterization was performed using a two-point probe method to examine the conduction behavior of the films. The I–V characteristics presented in **Figure 8** show the electrical response of $CaTiO_3$ thin films under different annealing temperatures and coating repetitions. For all samples, the I–V plots display a predominantly linear relationship between current and voltage, indicative of ohmic behavior. This suggests that charge transport across the films is dominated by a constant resistance regime with minimal barrier formation at the contact interface. Notably, the film annealed at 450 °C exhibits slightly higher current response at a given voltage compared to those annealed at 250 °C and 350 °C.

Similarly, the effect of coating layers on the I–V response reveals a subtle trend in which films with a higher number of coatings tend to yield improved conductivity compared to 2-layer films, likely due to increased film thickness and better surface coverage. However, excessive layering such as in the 8-layer sample leads to a marginal decrease in current, potentially due to increased internal scattering or surface roughness, which hampers charge transport. The non-linear deviation observed at higher bias in some samples (especially the 4-layer film) may suggest the onset of space-charge-limited conduction (SCLC), a phenomenon where injected carriers exceed thermally generated carriers and dominate the transport mechanism—commonly found in wide band-gap semiconductors.

These results align well with the structural and optical characterizations, confirming that both annealing and deposition parameters significantly influence the electrical performance of $CaTiO_3$ thin films.

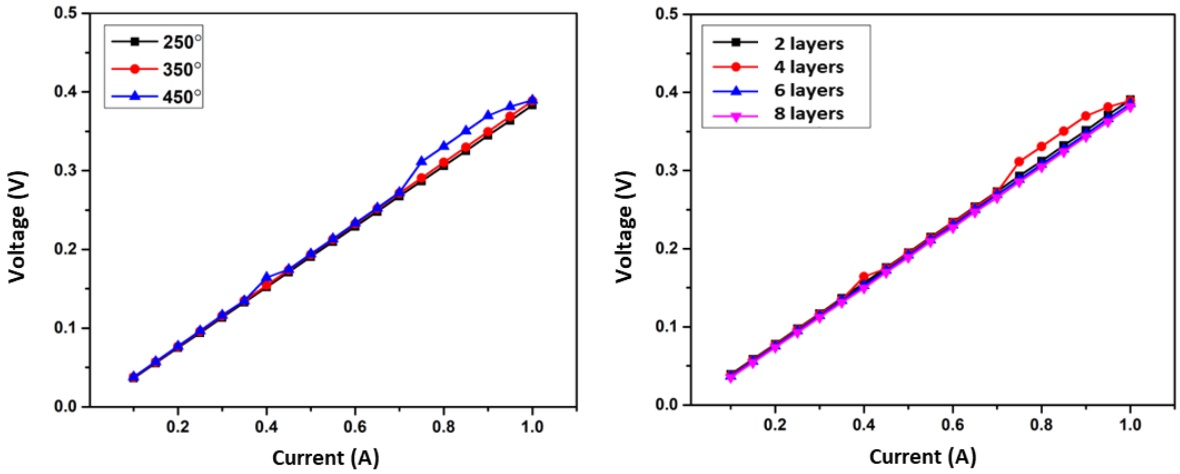

Figure 8. Current–voltage (I–V) characteristics of $CaTiO_3$ thin films as a function of annealing temperature (left) and coating layers (right)

3.4 Interpretation and Implications

The experimental findings suggest a considerable correlation between the spin-coating parameters—specifically the number of deposition layers and annealing temperature—and the resulting structural, optical, and electrical properties of $CaTiO_3$ thin films. XRD results indicated that higher annealing temperatures enhance the crystallinity of the films, as evidenced by the sharper and more intense diffraction peaks. This crystallinity improvement leads to reduced microstrain and increased crystallite size, particularly at 350 °C, which emerged as the optimal annealing condition. Additionally, UV-Vis absorption analysis showed that increased coating repetitions and higher thermal treatments improve optical absorption and slightly reduce the optical band gap, indicating a densification and improved packing of the film. These physical changes contribute to the observed variations in conductivity, where smoother, denser, and better-crystallized films—especially with 4 to 6 coating layers and 350–450 °C treatment—exhibited superior charge transport performance.

From a broader perspective, these findings underscore the tunability of $CaTiO_3$ thin film properties through straightforward modifications in fabrication protocols. The manifestation of near-ohmic I–V characteristics with occasional hints of space-charge-limited conduction reveals that $CaTiO_3$ films possess semiconducting traits suitable for optoelectronic applications, especially as buffer layers or interfacial materials in perovskite solar cells. The ability to manipulate band gap values and crystallinity through low-cost sol-gel spin-coating and thermal processing opens new possibilities for scalable and efficient device integration.

4. Conclusion

This study successfully demonstrated the influence of deposition layers and annealing temperature on the structural, optical, and electrical characteristics of $CaTiO_3$ thin films synthesized via the sol-gel spin-coating method. XRD analysis confirmed that annealing at 350 °C facilitate better crystallinity and reduced microstrain, while UV-Vis spectroscopy revealed a tunable optical band gap ranging from 3.741 eV to 3.554 eV depending on the number of layers and thermal treatment. Electrical measurements indicated predominantly ohmic behavior, with a slight nonlinear trend at higher voltages in certain samples, suggesting possible space-charge-limited conduction. Overall, the results indicate that the film quality and functionality can be optimized through simple adjustments to spin-coating parameters. These findings serve as a foundational step toward the integration of $CaTiO_3$ thin films in perovskite solar cell applications.

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