

Effect of Deposition Time on the Structural and Electrical Properties of Self-Catalyzed Indium Tin Oxide Nanostructures

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ABSTRACT

This study examines self-catalyzed growth of indium tin oxide (ITO) nanostructures via a chemical vapor deposition (CVD) method under deposition times of 30, 70 and 110 min. Scanning Electron Microscopy (SEM) analysis showed a shift from agglomerated to worm-like nanostructures as deposition time increased, with nanowire diameters decreasing from 0.623 μm to 0.069 μm , indicating controlled growth. Using the Scherrer equation from XRD data, the crystallite size initially increased from 15.35 nm to 20.25 nm as deposition time increased from 30 to 70 min, indicating improved crystallinity, but slightly decreased to 18.06 nm at 110 min, indicating changes in crystallite development and lattice strain at longer deposition times. The lowest sheet resistance of 81.28 Ω/sq was observed for the 70 min sample indicating enhanced electrical conductivity compared to other samples. These findings highlight how deposition time influences the self-catalyzed growth of ITO nanostructures in achieving optimal structural and electrical properties specific for optoelectronic applications.

Keywords: Chemical vapor deposition; deposition time; indium tin oxide; self-catalyzed growth; structural properties

INTRODUCTION

Metal oxides have gained significant interest in recent years because of their high surface area and superior photoelectric characteristics [1,2,3]. Indium tin oxide (ITO) is one of the metal oxides generally known as transparent conductive oxide (TCO) are interesting materials due to their distinct physical, optical and electrical characteristics such as high transmission in the near infra-red and visible region [4,5]. The unique combination of electrical and optical properties such as high visible transmittance ($> 80\%$) [6], low

electrical resistivity [7,8] and wide band gap (> 3 eV) [3] provide high performance of ITO compared to other TCOs. These good properties of ITO have been remarkable to a selected potential application such as display touch screen and solar cells [9], functional glass technologies [10], gas sensor [11] and transparent antennas [12].

Previously, the growth of ITO has largely relied on catalysts such as nickel (Ni), gold (Au) and platinum (Pt) [13,14,15]. The presence of the catalyst on the growth may affect the properties and potential applications of the resulting nanostructures such as contamination and impurities [13]. In contrast, in self-catalytic growth, the material to be grown as nanostructure itself acts as a catalyst [13,16]. Moreover, some studies have explored the potential of self-catalytic growth of ITO nanostructures, which can offer advantages such as lower cost, improved performance and simplified synthesis processes [13].

There are various methods in the growth of ITO nanostructures such as chemical vapor deposition (CVD) [13,16,17], physical vapor deposition (PVD) [18], electron beam evaporation [19], magnetron sputtering [20] and DC magnetron sputtering [14]. Among the existing methods, CVD has been extensively employed for the growth of highly crystalline ITO nanostructures [13]. Recent studies have demonstrated the feasibility of self-catalyzed ITO nanostructure growth without the use of an external metal catalyst. Jung et al. [13] reported the growth evolution of self-catalytic ITO nanowires using thermal CVD, highlighting the role of catalyst droplet formation and oxygen flow during the growth process. More recently, Dhar et al. [16] demonstrated the synthesis of self-catalytic ITO nanorods by sputtering and investigating their structural and electrical characteristics for photovoltaic applications. These studies have established the feasibility of catalyst-free ITO nanostructure growth; however, the influence of prolonged deposition time on the simultaneous evolution of morphology, crystallite size, lattice strain and electrical properties under fixed thermal CVD conditions remains insufficiently explored.

In particular, the relationship between deposition time dependent structural evolution and electrical properties has not been systematically established. Therefore, the present work systematically investigates the effect of deposition time on self-catalyzed ITO nanostructures synthesized by chemical vapor deposition via thermal evaporation in a tube furnace. Deposition times of 30, 70 and 110 min were investigated under fixed deposition conditions to elucidate the evolution of the nanostructure morphology and crystallographic characteristics and their relationship with electrical performance. The novelty of this study lies in establishing a direct correlation between deposition time dependent morphological evolution, crystallite size, lattice strain, I-V behavior and sheet resistance, thereby establishing the relationship between deposition time and the resulting structural and electrical characteristics.

METHODS

Indium (III) oxide (In_2O_3 , 99.99% trace metal basis) and tin (IV) oxide (SnO_2 , $\geq 99.99\%$ trace metal basis) powders were used for crystalline growth of the indium tin oxide (ITO) nanostructures. The samples were deposited on p-type silicon (Si) (111) substrates via chemical vapor deposition (CVD) in a horizontal tube furnace. The powders were mechanically mixed at a 1:1 weight ratio, placed in an alumina boat and positioned in the

furnace hot zone. A Si substrate, serving as the deposition surface, was prepared using standard RCA I and RCA II cleaning procedures [21]. RCA I involved immersion in $\text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{HCl}$ (6:1:1) for 5 min to remove metallic and inorganic contaminants, followed by RCA II in $\text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{NH}_4\text{OH}$ (5:1:1) for 5 min to eliminate organic residues. Native SiO_2 was removed by immersion in $\text{H}_2\text{O}:\text{HF}$ (10:1) for 5 min, with deionized (DI) water rinses between each step. The tube furnace was evacuated to $\sim 2 \times 10^{-2}$ Torr and maintained for 10 min before introducing argon (Ar) as a carrier gas. The furnace temperature was increased at a controlled heating rate of $60^\circ\text{C}/\text{min}$ to the target deposition temperature of 1100°C . The deposition performed at 3 Torr for 30, 70, or 110 min. After deposition, the furnace was cooled naturally to room temperature and the films were collected for characterization.

Scanning electron microscopy (SEM, Tescan Vega 3) was used to analyze the surface morphology of ITO thin film, providing comprehensive imaging of the structures' size, shape and distribution. The crystal structure of the ITO thin film was investigated using XRD Rigaku model equipment. The crystallite size (D) was determined using Debye-Scherrer equation (1):

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where λ is X-ray wavelength (1.5406 Å), β is Full Width at Half Maximum (FWHM) of XRD peak in radian and θ is XRD peak position, one half of 2θ . Moreover, lattice strain (ε) was determined using tangent equation (2):

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (2)$$

The electrical properties of the ITO nanostructures were characterized using a four-point probe configuration connected to a Keithley 236 SMU model. The four-point probe consists of four equally spaced and collinear probes positioned in contact with the surface of the ITO sample. During the measurement, a known DC current was applied through the two outer probes, while the resulting voltage drop was measured between the two inner probes. The sheet resistance (R_s) of the ITO nanostructures was calculated from the measured voltage and applied current using Equation (3):

$$R_s = \frac{\pi}{\ln 2} \frac{V}{I} \quad (3)$$

where R_s is the sheet resistance (Ω/sq), V is the voltage measured between the two inner probes (V) and I is the current applied through the two outer probes (A).

RESULTS AND DISCUSSION

Figure 1 shows the SEM images of various surface morphologies of ITO nanostructures grown at different times. A highly dense agglomerated nanostructure consisting of large grains with size $\sim 1.717 \mu\text{m}$ for the samples presents on the substrate prepared at 30 min deposition time (Figure 1(a)). As shown in Figure 1(b), at higher deposition time (70 min), the morphology changes to worm-like nanostructures, accompanied by smaller particle sizes $\sim 1.012 \mu\text{m}$ with a diameter of worm-like tail is $\sim 0.143 \mu\text{m}$. Furthermore, the ITO grains significantly decreased to $\sim 0.238 \mu\text{m}$ and the sharper worm-like tail of

$\sim 0.069 \mu\text{m}$ was seen in deposition time of 110 min with the as illustrated in Figure 1(c). The growth of ITO nanostructures is clearly present, increasing the worm-like at 70 min and 110 min, as shown in the inset of Figures 1(b) and 1(c), respectively. Extended deposition time of indium tin oxide nanostructures led to a notable increase in the formation of nanoparticles. This phenomenon suggests that prolonged exposure to growth conditions allowed for enhanced nucleation events or aggregation processes, thereby promoting the formation of additional nanoparticles alongside the primary nanowire structures [10]. These nuclei can grow into nanoparticles before or alongside the growth of nanowires. Over time, nanoparticles may aggregate due to attractive forces between them, such as Van der Waals forces or electrostatic interactions [22]. This aggregation process is more likely to occur with prolonged exposure to growth conditions. The kinetics of the deposition process may favor the formation of nanoparticles under certain conditions, such as high precursor concentrations or specific growth temperatures.

Longer deposition times increase the likelihood of reaching conditions conducive to nanoparticle formation. The presence of these nanoparticles may influence the overall morphology and properties of the ITO nanostructures, potentially impacting their applications in various technological fields. It should also be noted that no catalysts were used during the growth of ITO nanostructures deposition. This relationship emphasizes how important it is to regulate the amount and spatial arrangement of nanostructures created by self-catalyzed by varying the deposition period [13]. The observed morphological evolution from agglomerated structures to more pronounced worm-like features with increasing deposition time is consistent with a proposed self-catalytic vapor-liquid-solid (VLS) growth mechanism reported for ITO nanostructures [13]. Under a VLS-type growth pathway, vaporized In- and Sn-containing species may undergo nucleation and form transient liquid-like regions that facilitate the subsequent growth of ITO nanostructures [6]. With increasing deposition time, the prolonged supply of vapor phase species may promote continued growth and morphological evolution, resulting in the more pronounced worm-like features observed at 70 and 110 min. The sustained availability of adatoms during prolonged deposition may further contribute to the development of these elongated morphologies. Similar VLS-type growth behavior has been reported in ITO and other oxide nanostructures [13,16].

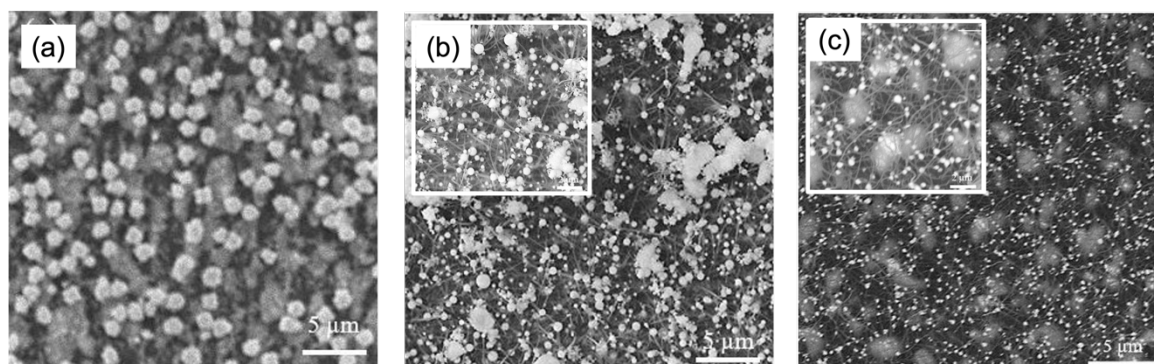


Figure 1. SEM images the growth of the indium tin oxide nanostructures by chemical vapor deposition with varying deposition times of (a) 30 min, (b) 70 min and (c) 110 min

The XRD pattern of ITO nanostructures grown at 30, 70 and 110 min deposition times are illustrated in Figure 2. The nanostructures consisted of cubic In_2O_3 (JCPDS No. 00-006-0416) and hexagonal InSn_4 (JCPDS No. 00-048-1547). At 30 min, the (222) peak of cubic In_2O_3 at $2\theta = 30.6^\circ$ is dominant but relatively broad, indicating smaller crystallite domains. As deposition time increase to 70 min, newer peak of cubic In_2O_3 present at $2\theta = 21.5^\circ, 35.5^\circ, 51.0^\circ$ and 60.7° which correspond to (211), (400), (440) and (622) orientation. Moreover, the tetragonal SnO_4 (JCPDS No. 01-077-0450) start to form at $2\theta = 26.5^\circ, 33.7^\circ, 37.9^\circ$ and 51.7° indicating the crystallographic planes (110), (101), (200) and (211), respectively. The appearance of additional diffraction peaks at 70 min indicates the development or increased detectability of additional crystalline phases or orientations with increasing deposition time. This change may be associated with differences in nucleation and crystallite development during prolonged deposition. This structural improvement is reflected in SEM where worm-like nanostructures with smaller grains and well-defined tails are observed, pointing to a more controlled and uniform nanostructure formation. This worm-like morphology is consistent with the XRD results, where longer deposition times produce higher peak intensities and larger crystallite sizes. The development of more elongated nanostructures coincides with the sharper and more defined diffraction peaks observed at 70 min, suggesting a relationship between the morphological evolution and the crystallographic characteristics of the ITO nanostructures. Therefore, the evolution from large grain to longer worm-like structures in the SEM images is consistent with the changes in crystallographic characteristics observed in the XRD patterns [23,24]. Further increasing the deposition time up to 110 min the cubic In_2O_3 at $2\theta = 30.6^\circ, 44.9^\circ$ and 45.6° become sharper and more other peak start to diminished. The observed changes in diffraction peaks with increasing deposition time may be associated with changes in crystallite development and preferred orientation during prolonged deposition [25]. Therefore, it is worth noting that the emergence or disappearance of specific diffraction planes suggests a dynamic evolution of the nanostructures over the deposition period, characterized by intricate crystalline interactions and structural transformations.

The FWHM, crystallite size and lattice strain are tabulated in Table 1. As deposition time increased from 30 to 70 min, the crystallite size of ITO nanostructures changed from 15.35 nm to 20.25 nm, followed by a slight reduction to 18.06 nm at 110 min. The increase in crystallite size up to 70 min, accompanied by the lowest lattice strain value of 3.51×10^{-3} , suggests improved crystallographic characteristics at this deposition time. This trend is also supported by the narrowing of the diffraction peak (FWHM = 0.4439° at 70 min), which indicates larger coherent diffracting domains. However, a further increase in deposition time to 110 min resulted in a decrease in crystallite size and an increase in lattice strain ($\epsilon = 4.40 \times 10^{-3}$), along with a broader FWHM (0.4975°). The reduction in crystallite size and increase in calculated lattice strain at 110 min may indicate increased structural disorder or residual strain associated with prolonged deposition. This behavior may be related to changes in nucleation, crystallite coalescence and strain development during extended deposition, despite the more developed worm-like morphology observed in the SEM images. The observed variation in FWHM, crystallite size and calculated lattice strain is consistent with changes in the crystallographic characteristics of the ITO nanostructures with deposition time [19]. The larger crystallite size and lower calculated lattice strain at 70 min suggest a more

favorable crystallographic condition among the investigated deposition times. At 110 min, the reduction in crystallite size and increase in calculated lattice strain may be associated with changes in crystallite development and strain evolution during prolonged deposition.

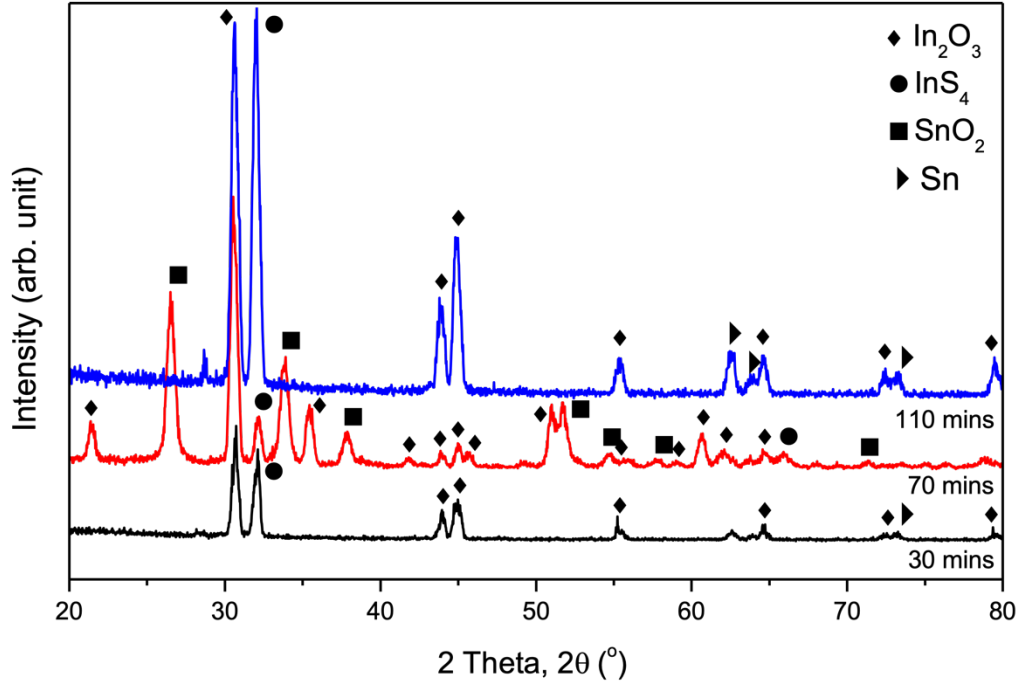


Figure 2. XRD pattern of ITO nanostructures grown at different deposition time

Table 1. WHM, crystallite size, D (nm) and lattice strain (ϵ) of ITO thin film at various deposition times 30, 70 and 110 min

Deposition Time (min)	FWHM (°)	Crystallite Size, D (nm)	Lattice Strain, ϵ ($\times 10^{-3}$)
30	0.5854	15.35	4.98
70	0.4439	20.25	3.51
110	0.4975	18.06	4.40

The I-V characteristics of the self-catalyzed ITO nanostructures grown at deposition times of 30, 70 and 110 min are shown in Figures 3(a), 3(b) and 3(c). For the samples deposited at 30 min and 70 min, the I-V curves display a nearly linear relationship between current and voltage with a slope 21.88 and 17.93 Ω , respectively, indicative predominantly ohmic behavior. The lower slope observed for the 70 min sample is consistent with its lower electrical resistance. The relatively linear I-V response suggests stable electrical conduction under the measurement conditions [26]. In contrast, the I-V curve of the 110 min sample exhibits pronounced non-linear behavior, with a much higher slope of 4114.40 Ω and deviations from the approximately linear response observed for the 30 and 70 min samples (Figure 3(c)). The curve exhibits multiple slopes and irregular regions, which may be associated with changes in the electrical transport

behavior resulting from the morphological and crystallographic evolution during prolonged deposition [27].

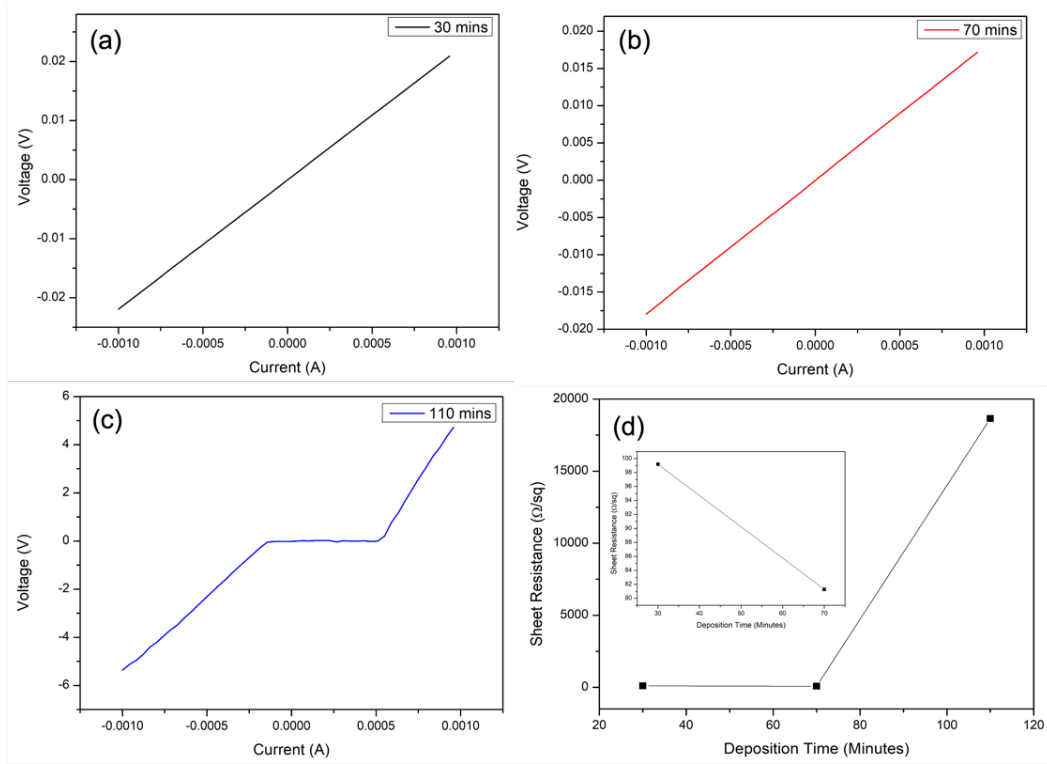


Figure 3. I-V characteristics of the self-catalyzed ITO nanostructures grown at deposition times of (a) 30, (b) 70, (c) 110 min and (d) sheet resistance of ITO nanostructures deposited at different deposition times

The variation in sheet resistance shown in Figure 3(d) further supports the observed electrical behavior. The lowest sheet resistance of $81.28 \Omega/\text{sq}$ was recorded for the 70 min sample, which also exhibited the largest calculated crystallite size and lowest calculated lattice strain among the investigated samples. At 30 min, the sheet resistance was slightly higher at $99.18 \Omega/\text{sq}$. This may be associated with the early stage of nanostructure development, as the SEM observations show a predominantly agglomerated morphology rather than the more developed worm-like features observed at 70 min. Conversely, the sheet resistance increased sharply to $18,650.36 \Omega/\text{sq}$ at 110 min. This substantial increase may be associated with the morphological and crystallographic changes that occur during prolonged deposition, as reflected by the reduced calculated crystallite size and increased lattice strain compared with the 70 min sample. These results demonstrate a clear relationship between deposition time, morphological evolution, crystallographic characteristics and electrical performance of the self-catalyzed ITO nanostructures [5,28].

CONCLUSIONS

In this study, self-catalyzed ITO nanostructures were successfully grown using chemical vapor deposition at different deposition times. The results demonstrate that deposition

time plays a crucial role in governing morphology, crystallinity and electrical transport properties. SEM revealed a transition from agglomerated grains at 30 min to well-defined worm-like structures at 70 min, with further surface refinement at 110 min. Moreover, XRD analysis confirmed that crystallite size increased with deposition time up to 70 min, accompanied by a reduction in calculated lattice strain, suggesting improved crystallographic characteristics. Electrical measurements further revealed that the 70 min deposition time sample exhibited the lowest sheet resistance (81.28 Ω/sq) and stable ohmic behavior, correlating with its crystallographic characteristics and surface morphology. Among the deposition times investigated, 70 min produced the most favorable combination of morphological, crystallographic and electrical characteristics. These findings provide useful insights into tailoring deposition time for the fabrication of ITO-based transparent conducting electrodes and optoelectronic devices.

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AUTHOR CONTRIBUTION STATEMENT

Nurul Fateha Abdul Halim: writing original draft, data collection and analysis, visualization and figure preparation. Azianty Saroni: supervision, manuscript review and editing and research coordination. All authors have read and approved the final version of the manuscript.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

DATA AVAILABILITY

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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