



Study of Structural and Optical Properties of Thin Film Semiconductor Materials

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Abstract

Thin-film semiconductors are central to photovoltaic, sensing, display, photocatalytic and optoelectronic technologies because their crystalline structure, defect state and optical response can be tailored through deposition and post-deposition processing. This methodology paper presents a compact, reproducible framework for studying the structural and optical properties of semiconductor thin films, using sol-gel-derived ZnO as a representative model while relating the procedure to CdS and TiO₂ literature. The proposed workflow combines controlled substrate preparation, solution-based deposition, thermal annealing, X-ray diffraction (XRD), surface/morphological inspection and UV-Visible spectroscopy. Phase identification, preferred orientation, crystallite size, microstrain, transmittance, absorption coefficient and optical band gap are evaluated using established analytical relationships. Literature published no later than 2015 shows that film thickness and annealing conditions strongly modify crystallinity and optical behavior in ZnO, CdS and TiO₂ systems. Because no original experimental dataset was supplied for this paper, the Results section contains an explicitly labelled illustrative dataset designed to demonstrate calculations and expected trends rather than to claim new measurements. The methodology is suitable for undergraduate/postgraduate laboratory studies and can be adapted to other oxide and chalcogenide semiconductor films by changing precursor chemistry and thermal treatment conditions.

Introduction

Semiconductor thin films provide a practical route to controlling electrical and optical functions through material composition, film thickness, grain structure, defects and interfaces. Compared with bulk solids, thin films can be integrated directly on glass, silicon or conducting substrates and can be produced by vacuum techniques such as sputtering and evaporation or by low-cost chemical routes such as sol-gel coating, spray pyrolysis and chemical bath deposition. ZnO is a widely studied wide-band-gap oxide semiconductor, while CdS is an

important II-VI chalcogenide and TiO₂ is a stable oxide used in optical and photoactive systems. Studies before 2016 demonstrate that these materials can show high visible transparency and structure-dependent optical gaps when processed as thin films (Moualkia et al., 2009; Xu et al., 2011; Khan et al., 2015).

Two characterization families are especially important. XRD identifies crystalline phase and preferred orientation and allows peak-broadening analysis to estimate characteristic crystallite size and strain. The classic Williamson-Hall treatment separates size and

strain contributions to diffraction-line broadening (Williamson & Hall, 1953). Optical spectroscopy provides wavelength-dependent transmission and absorption; Tauc-type analysis is widely used to estimate the optical transition energy from the absorption edge (Tauc et al., 1966), while transmission-envelope methods can also support thickness and optical-constant estimation (Swanepoel, 1983). Together, structural and optical measurements permit a direct structure-property correlation.

The purpose of the present methodology is therefore to define a step-by-step experimental and analytical protocol for examining how processing affects structural order and optical response in thin-film semiconductors. A ZnO sol-gel/spin-coating experiment with controlled annealing temperature is used as the representative implementation because comparable procedures and trends are documented across several pre-2016 studies (Khan et al., 2011; Sengupta et al., 2011; Yadav et al., 2014; Shivaraj et al., 2015). The same characterization logic can be transferred to CdS, TiO₂, SnO₂ and related films.

Literature Review

Early analytical work established the quantitative basis used in modern thin-film characterization. Williamson and Hall (1953) showed that diffraction-line broadening can contain simultaneous contributions from finite coherent domain size and lattice strain. Tauc et al. (1966) connected optical absorption near the band edge with electronic transitions, providing the basis of the commonly used Tauc plot. Swanepoel (1983) later developed a transmission-spectrum method for determining thin-film thickness and optical constants on transparent substrates. These methods remain

useful because they transform routinely measured XRD and optical spectra into physically interpretable parameters.

Material-specific studies further show that deposition and thermal processing can substantially change the measured properties. Moualkia et al. (2009) prepared CdS films by chemical bath deposition and reported a preferentially oriented crystalline structure together with relatively high optical transmission. For ZnO, Xu et al. (2011) demonstrated that increasing sol-gel film thickness changed growth behavior and improved crystalline quality while visible transmittance remained high. Khan et al. (2011) reported highly oriented sol-gel ZnO with a hexagonal wurtzite structure and an optical gap near the expected wide-band-gap range. Sengupta et al. (2011) found that annealing increased ZnO crystallinity and grain size and altered surface roughness and optical transmission.

The influence of post-deposition heat treatment is repeatedly observed. Yadav et al. (2014) studied sol-gel ZnO films annealed at different temperatures and documented systematic changes in structural, optical and electrical behavior. Shivaraj et al. (2015) compared dip- and spin-coated ZnO and found annealing-temperature-dependent changes in crystalline size, roughness and morphology. A similar structure-optical linkage occurs in TiO₂: Khan et al. (2015) reported improved crystallinity with annealing and a reduction in optical band gap as the annealing temperature increased. Collectively, these findings justify an experimental design in which deposition variables are held constant while annealing temperature is treated as a controlled independent variable.

Table 1. Selected pre-2016 studies informing the methodology

Reference	Material	Preparation	Methodological relevance
Moualkia et al. (2009)	CdS	Chemical bath	XRD + UV-Vis; crystalline orientation and high transmission.
Xu et al. (2011)	ZnO	Sol-gel	Thickness affected crystal growth; visible transmission remained high.
Sengupta et al. (2011)	ZnO	Sol-gel	Annealing increased crystallinity/grain size and changed optical response.
Yadav et al. (2014)	ZnO	Sol-gel spin	Annealing systematically modified structural and optical properties.
Shivaraj et al. (2015)	ZnO	Dip/spin coating	Annealing influenced crystalline size, roughness and morphology.

Khan et al. (2015)	TiO ₂	E-beam + anneal	Higher annealing improved crystallinity and reduced optical gap.
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Methodology

1. Research Design and Materials

A controlled laboratory design is proposed in which ZnO thin films are deposited on microscope-glass or fused-silica substrates by sol-gel spin coating and then annealed at three temperatures (for example 400, 450 and 500 °C). Annealing temperature is the independent variable; precursor concentration, spin speed, coating cycles, preheating time and substrate type are held constant. This design follows the logic of pre-2016 ZnO studies while keeping the experiment simple enough for reproducible structural-optical comparison (Sengupta et al., 2011; Yadav et al., 2014; Shivaraj et al., 2015). Analytical-grade zinc acetate dihydrate is used as the Zn source, ethanol or isopropanol as solvent and monoethanolamine/diethanolamine as stabilizer. Deionized water, acetone and ethanol are used for substrate cleaning.

2. Thin-Film Preparation

Step 1 - Substrate cleaning: glass substrates are washed with detergent and deionized water, ultrasonically cleaned in acetone and ethanol for approximately 10 min each, rinsed and dried. Step 2 - Sol preparation: a 0.3-0.5 M zinc acetate solution is prepared in the selected alcohol solvent; stabilizer is added gradually at approximately equimolar ratio to zinc precursor while stirring at 60-70 °C until a clear solution forms. The sol is aged before coating. Step 3 - Coating: 0.1-0.2 mL of sol is dispensed onto the substrate and spun at approximately 2500-3500 rpm for 20-30 s. Step 4 - Intermediate drying: every deposited layer is preheated near 150-250 °C to remove solvent and organics. The coating/drying cycle is repeated to obtain the target thickness. Step 5 - Final annealing: replicate films are annealed in air at the selected temperatures for 1 h and cooled naturally. Sol-gel procedures of this general type were used successfully for ZnO films by Xu et al. (2011) and Khan et al. (2011).

Table 2. Proposed experimental control matrix

Sample	Annealing	Time	Spin speed	Coating cycles	Purpose
Film S1	400 °C	1 h	Constant	Constant	Baseline annealed condition
Film S2	450 °C	1 h	Constant	Constant	Intermediate condition
Film S3	500 °C	1 h	Constant	Constant	Higher-temperature condition

The matrix isolates the thermal treatment as the principal variable. If the investigation instead aims to study thickness, the number of coating cycles can be varied while annealing temperature is fixed. Only one primary process variable should be changed at a time unless a factorial design is intentionally used, because

simultaneous changes in thickness, spin speed and annealing can make the origin of XRD and optical differences ambiguous. Each sample should be coded before measurement so that the same specimen can be tracked across thickness, XRD, microscopy and UV-Visible measurements.



Figure 1. Proposed workflow for preparation, structural characterization and optical analysis of semiconductor thin films.

3. Structural Characterization and Calculations

XRD patterns are recorded over a suitable 2θ range (for example 20-80°) using Cu K α

radiation ($\lambda = 1.5406 \text{ \AA}$). Phase identification is performed by comparing peak positions with standard diffraction data. For wurtzite ZnO, special attention is given to the (002) reflection

near 34.4° , because strong intensity at this peak indicates preferred c-axis orientation frequently observed in sol-gel ZnO films (Xu et al., 2011; Sengupta et al., 2011). The interplanar spacing is calculated from Bragg's law, $n\lambda = 2d \sin\theta$. A first estimate of crystallite size is obtained from the Scherrer expression $D = K\lambda/(\beta \cos\theta)$, where β is the corrected full width at half maximum in radians and K is normally taken near 0.9. When several peaks are measurable, size and microstrain are better separated using the Williamson-Hall relation $\beta \cos\theta = K\lambda/D + 4\epsilon \sin\theta$ (Williamson & Hall, 1953). Surface morphology may be examined by SEM or AFM to compare grain appearance and roughness with diffraction-derived trends.

4. Optical Characterization and Calculations

UV-Visible transmission spectra are measured from approximately 300 to 900 nm using an uncoated substrate as the reference. Average visible transmittance is calculated over a fixed wavelength window such as 400-700 nm. If film thickness t is known, the absorption coefficient can be approximated from transmission by $\alpha = (1/t) \ln(1/T)$ when reflection and interference are small; more rigorous analysis can include reflectance or transmission-envelope corrections. Swanepoel's method provides a classical route for deriving thickness and optical constants from interference envelopes when suitable fringes are present (Swanepoel, 1983). The optical gap is estimated from the Tauc relation $(\alpha h\nu)^n = A(h\nu - E_g)$. For a direct allowed transition, the commonly used plot is $(\alpha h\nu)^2$ versus $h\nu$; linear extrapolation to the

energy axis gives E_g (Tauc et al., 1966). The chosen transition exponent should be justified for the material being studied.

5. Quality Control and Data Analysis

At least three films per condition should be prepared where resources permit. Instrument settings, baseline/reference measurements and sample orientation should remain constant. XRD peak positions, FWHM values and derived parameters are tabulated with units; UV-Vis spectra should be collected using identical wavelength resolution and slit settings. Results are summarized as mean $\pm$ standard deviation for repeated samples. One-way ANOVA can be used to test whether annealing temperature causes statistically significant changes in crystallite size, transmittance or band gap, provided replicate measurements are available. Structural and optical variables are finally compared using correlation analysis or trend plots.

Results

No raw experimental measurements were provided with this request. Therefore, the numerical values in Table 3 are an illustrative dataset created solely to demonstrate how the proposed methodology would be reported; they are not presented as new experimental observations. The directions of change are consistent with pre-2016 literature showing that suitable annealing can sharpen ZnO diffraction peaks, increase characteristic crystalline dimensions and modify optical absorption/transmission (Sengupta et al., 2011; Yadav et al., 2014; Shivaraj et al., 2015).

Table 3. Illustrative structural and optical result format for annealed ZnO thin films

Sample	Anneal (°C)	2 θ (002) (°)	FWHM (°)	D (nm)	Strain ($\times 10^{-3}$)	Tvis (%)	E _g (eV)
S1	400	34.36	0.42	20.0	2.8	84	3.31
S2	450	34.40	0.34	24.8	2.2	87	3.28
S3	500	34.43	0.28	30.1	1.8	89	3.25

In the illustrative analysis, increasing annealing temperature causes the (002) peak to narrow, corresponding to an increase in Scherrer crystallite size from approximately 20 to 30 nm and a reduction in calculated strain. Such behavior is interpreted as improved crystallinity and defect relaxation during thermal treatment, in agreement with the qualitative trend reported for annealed ZnO thin films by Sengupta et al. (2011) and Yadav et al. (2014). Shivaraj et al. (2015) similarly showed that annealing influences crystalline size and morphology in

dip- and spin-coated films. XRD interpretation should, however, distinguish coherent-domain size from larger SEM/AFM grain dimensions because the two are not necessarily identical.

The illustrative optical results show a high visible transmittance of 84-89%, together with a modest change in band gap from 3.31 to 3.25 eV. High transparency is consistent with many ZnO sol-gel reports, including Xu et al. (2011), while band-edge changes can reflect crystallinity, strain, defect concentration, carrier effects and

thickness. The magnitude and even the direction of a band-gap shift can vary with material and processing. For example, Khan et al. (2015) reported a decrease in TiO₂ optical gap with increased annealing temperature, emphasizing that structural evolution and phase composition must be considered when interpreting optical trends. CdS results likewise depend on bath conditions, deposition time and film structure (Moualkia et al., 2009).

A complete interpretation should compare more than one property at a time. If XRD peaks become narrower while the optical absorption edge becomes sharper, the combined evidence supports improved structural order. If transmission falls substantially after annealing, the cause should not automatically be assigned to band-gap change; increased thickness, roughness, scattering, secondary phases or surface contamination should first be excluded. Likewise, a Scherrer size should be reported as a coherent crystallite dimension rather than as an exact particle diameter. Repeating thickness measurements at several positions and correcting XRD peak width for instrumental broadening reduce common sources of error. These checks are important because small differences in FWHM or optical-edge extrapolation can produce apparently meaningful numerical changes that are actually within experimental uncertainty.

Conclusion and Discussion

This paper defines a concise methodology for examining structural and optical properties of semiconductor thin films using controlled deposition, thermal treatment, XRD and UV-Visible spectroscopy. The most important methodological principle is that processing conditions must be linked to measurable valuation.

structural parameters before optical changes are interpreted. XRD provides evidence of phase, preferred orientation, peak width, crystallite size and strain, while optical spectra quantify transmission, absorption behavior and band-edge energy. The foundational methods of Williamson and Hall (1953), Tauc et al. (1966) and Swanepoel (1983) give a transparent analytical basis for these calculations. Pre-2016 literature confirms that the proposed approach is applicable across different semiconductor families. ZnO studies demonstrate sensitivity to film thickness and annealing (Xu et al., 2011; Sengupta et al., 2011; Yadav et al., 2014; Shivaraj et al., 2015), while CdS and TiO₂ studies show comparable links between fabrication conditions, crystallinity and optical response (Moualkia et al., 2009; Khan et al., 2015). For an actual research submission, the illustrative values in Table 3 should be replaced with measured spectra and calculated data from the researcher's own samples. With that substitution, the proposed workflow can support a defensible 6–7-page experimental methodology paper without overstating results. The main limitation of the proposed paper is that a single representative ZnO process cannot capture every chemistry used for semiconductor films. CdS chemical-bath growth, TiO₂ sol-gel processing and vacuum-deposited films require different precursor, atmosphere and temperature constraints. Nevertheless, the characterization sequence remains broadly transferable: establish deposition history and thickness, identify phase and microstructure, quantify optical spectra, calculate physically justified parameters and then relate these outputs to processing. This modular structure is useful for comparative studies because material-specific preparation can be changed without changing the logic of structural and optical e

References

- Williamson, G. K., & Hall, W. H. (1953). X-ray line broadening from fcc aluminium and wolfram. *Acta Metallurgica*, 1(1), 22-31. DOI: 10.1016/0001-6160(53)90006-6.
- Tauc, J., Grigorovici, R., & Vancu, A. (1966). Optical properties and electronic structure of amorphous germanium. *physica status solidi* (b), 15(2), 627-637. DOI: 10.1002/pssb.19660150224.
- Swanepoel, R. (1983). Determination of the thickness and optical constants of amorphous silicon. *Journal of Physics E: Scientific Instruments*, 16(12), 1214-1222. DOI: 10.1088/0022-3735/16/12/023.
- Moualkia, H., Hariech, S., & Aida, M. S. (2009). Structural and optical properties of CdS thin films grown by chemical bath deposition. *Thin Solid Films*, 518(4), 1259-1262. DOI: 10.1016/j.tsf.2009.04.067.
- Xu, L., Li, X., Chen, Y., & Xu, F. (2011). Structural and optical properties of ZnO thin films prepared by sol-gel method with different thickness. *Applied Surface Science*,

257(9), 4031-4037. DOI:
10.1016/j.apsusc.2010.11.170.

Khan, Z. R., Khan, M. S., Zulfequar, M., & Khan, M. S. (2011). Optical and structural properties of ZnO thin films fabricated by sol-gel method. *Materials Sciences and Applications*, 2, 340-345. DOI: 10.4236/msa.2011.25044.

Sengupta, J., Sahoo, R. K., Bardhan, K. K., & Mukherjee, C. D. (2011). Influence of annealing temperature on the structural, topographical and optical properties of sol-gel derived ZnO thin films. *Materials Letters*, 65(17-18), 2572-2574. DOI: 10.1016/j.matlet.2011.06.021.

Yadav, A. B., Pandey, A., & Jit, S. (2014). Effects of annealing temperature on the structural, optical, and electrical properties of

ZnO thin films grown on n-Si<100> substrates by the sol-gel spin coating method. *Acta Metallurgica Sinica (English Letters)*, 27(4), 682-688. DOI: 10.1007/s40195-014-0097-4.

Shivaraj, B. W., Narasimha Murthy, H. N., Krishna, M., & Satyanarayana, B. S. (2015). Effect of annealing temperature on structural and optical properties of dip and spin coated ZnO thin films. *Procedia Materials Science*, 10, 292-300. DOI: 10.1016/j.mspro.2015.06.053.

Khan, A. F., Mehmood, M., Durrani, S. K., Ali, M. L., & Rahim, N. A. (2015). Structural and optoelectronic properties of nanostructured TiO₂ thin films with annealing. *Materials Science in Semiconductor Processing*, 29, 161-169. DOI: 10.1016/j.mssp.2014.02.009.