Characterization of low-temperature-grown ZnO nanoparticles: The effect of temperature on growth

F B Dejene
Department of Physics, Walter Sisulu University, Private Bag X-1, Mthatha, 5117, South Africa
E-mail: fdejene@wsu.ac.za and dejenemk@gmail.com

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Abstract
The growth temperature of semiconducting nanoparticles is well known to be an essential factor in determining their structure and quality of characteristics. To create high-quality ZnO nanostructures, it is critical to optimize the growth temperature. The sol-gel approach is used to render ZnO NPs at different growth temperatures in this report. The influence of growth temperature on the material properties of ZnO nanoparticles has been extensively investigated. The scanning electron microscopy (SEM) images show needle-like structures when synthesized at low temperatures (35 and 45 °C) that transformed into spherical particles as the temperature of the growing medium increased to 75 °C. X-ray diffraction (XRD) analysis displays an increase in crystallinity with annealing temperatures. The average crystallite sizes of ZnO nanoparticles rose with increasing growth temperature and varied from 28 to 34 nm. The XRD peaks positions shift slightly towards lower 2θ angles with the growth temperatures brought about by a change in lattice parameters. The Fourier transform infrared spectroscopy (FTIR) study revealed the presence of a Z–O bond at around 680 cm⁻¹. The intensity of the defect level emission (DLE) band decreased, as the growth temperature exceeded 35 °C. The photoluminescence (PL) study further demonstrated that the prepared nanoparticles had a strong emission peak at 546 nm. PL measurements confirm that with increasing growth temperature, the excitonic peak intensity increases, indicating that the quality of ZnO nanoparticles improves. The estimated bandgap changed from 3.31 to 3.24 eV with the growth temperature.

1. Introduction
Zinc oxide (ZnO) is a semiconductor compound with a large bandgap of 3.37 eV at room temperature. Its wurtzite structure has a hexagonal unit cell with two lattice constants ‘a’ = 0.32 and ‘c’ = 0.521 nm. It is nontoxicity and can retain ultraviolet (UV) radiations, which makes it an ecologically amicable oxide. It has attracted a lot of attention due to its extraordinary properties and various applications in transparent electronics, solar cells, photocatalysts, lasers, piezoelectric devices, photoelectrodes, antibacterial agents, gas sensors, supercapacitors, and electrostatic transducers [1–5]. Many semiconductors, such as ZnO, especially groups such as II–VI, III–V, and IV–VI-based materials, have a quantum confinement effect. Their material properties and the corresponding applications are documented to be strongly affected by the shape and size of the nanomaterials. As a result, much effort is put into adjusting various production parameters such as growth duration, growth temperature, precursor concentration, stabilizing capping agents, and solvents to monitor the size, shape, and aspect ratio of the semiconducting nanoparticles [6]. The surface morphology, particle size, and shape significantly influence the ZnO properties. As such, the nanoparticles have a unique characteristic of size-dependent physical and chemical properties due to their high surface area and nanoscale size. Developing ZnO nanostructures with improved properties for explicit application is critical to controlling the development process [7, 8]. The temperature at which ZnO nanostructures grow is a critical factor in determining their material properties. ZnO nanostructures’ morphology and aspect ratio are significant thermodynamic variables that influence the rate at which a crystal grows. Guo et al. [9] discussed the effect of growth temperature and...
concentration in collision theory. When two or more chemicals species react, the molecules have to collide with each other with sufficient energy to cause a reaction. Optimization of growth temperature is, therefore, a pivotal procedure to obtain high-quality ZnO nanostructures. Chemical vapor [10], electrochemical [11], sputter [12], and pulse laser [13] depositions are all viable synthesis options for forming ZnO nanostructures. The disadvantage of these processes is that they require extreme reaction conditions, like high temperature, precise gas fixation, inflexible gas stream rate, and strict process control. Paradoxically, the sol-gel setup is less demanding and less expensive with generally low development temperature; it permits the development of particles with enormous surface zone to volume proportion and the developmental boundaries are much effortlessly controlled [14]. That is the reason the sol-gel technique is a well-known strategy among scientists for developing ZnO nanostructures. Solvents assume a fundamental part in the response component during the combination interaction by giving a method for temperature control that decides the most elevated temperature at which reaction will occur [15]. Prior investigations have shown that particle development and coarsening are firmly reliant on dissolvable and forerunners through consistency, mass solvency, and surface energy. Accordingly, particle sizes are controlled by utilizing various solvents to achieve electrical, optical, and structural properties pertinent to explicit applications [16]. The polarity of the solvent was found to be the most important factor in the nucleation and growth of ZnO nanoparticles, as well as the form, scale, and aspect ratio of the ZnO NPs. ZnO nanopowders have been prepared to utilize the sol-gel technique because of the novel properties that give it a benefit over others [17]. Considering this, in the current work, we have embraced the synthesis of the ZnO NPs, at very low growth temperatures, by sol-gel method using ethanol as a solvent and completed an orderly examination of the impacts of the development temperatures on the underlying and iridescent properties of the ZnO nanoparticles.

2. Experimental methods

ZnO NPs were prepared using zinc acetate dihydrate (Zn(CH₃COO)₂·2H₂O) aqueous solutions and sodium hydroxide (NaOH). Zn(CH₃COO)₂·2H₂O was dissolved in ethanol to a concentration of 0.2 M in a three-necked glass flask and was heated, under constant stirring, to different growth temperatures (room temperature (RT), 35, 45, 55, 65, and 75 °C). After accomplishing an ideal synthesis temperature, a 0.8 M solution of NaOH was slowly dripped into the glass flask containing Zn(CH₃COO)₂·2H₂O aqueous solution for 60 min. In this methodology, the selected reaction temperature was kept consistent. When an alkaline aqueous solution was dropped into a Zn(CH₃COO)₂·2H₂O solution, the suspension was consolidated for two hours at constant temperature and left to rest until a gel-like solution developed. The composite mixture was separated and washed with deionized water several times. The product was allowed to dry at room temperature before being baked for 60 min at 60 °C. The dried ZnO powders were then annealed in a furnace at 600 °C for 2 h before characterizations. The effect of development temperature on the material properties of the prepared ZnO nanoparticles was investigated using a variety of characterization techniques. X-ray diffraction (XRD) was used to determine the crystallite size, lattice constants, and crystallographic structure of ZnO NPs. The XRD data were collected using a Philips model Bruker D8 Advance X-ray diffractometer equipped with Cu Kα irradiation (λ = 1.5406 Å), scanned at the Bragg angle 2θ ranging from 20° to 80° with a 0.02° step. In order to assess whether the generated ZnO nanoparticles are amorphous or crystalline, a graph of reflection intensities is plotted against the 2θ. A Shimadzu model SX–550 super scan Scanning Electron Microscope (SEM) was used to investigate the surface morphology. A Cary Eclipse spectrophotometer, model LS–55, with a built-in 150W xenon flash lamp as the excitation source and a grating to pick a suitable excitation wavelength was used to analyze photoluminescence (PL). The optical properties were acquired using a Perkin Elmer Lambda 950 UV–vis photo spectrometer (UV–vis). Lastly, the absorbance property of ZnO was studied using a Perkin Elmer Spectrum 100 Fourier transformed infrared spectroscopy (FTIR), and thermal gravimetric analysis was measured using a Perkin Elmer STA 6000 simultaneous thermal analyzer (TGA).

3. Results and discussion

3.1 Structural and compositional analysis

3.1.1 XRD analysis

The XRD patterns of ZnO particles deposited at various growth temperatures are shown in figure 1(a). The hexagonal structure of ZnO is produced, as shown by XRD spectra, which match the standard card JCPDS No. 79–0208. The crystallite sizes of ZnO NPs were determined using Scherrer’s formula [18]. With increased growth temperature up to 65 °C (figure 1(b)), the diffraction peak broadening of the most intense peak decreases while relative strength increases, confirming improved crystal quality [19].

The decline of the FWHM with growth temperature could be because of coalescences of grains at higher temperatures prompting an expansion in the crystallite size. According to Nanda et al [20], Ostwald ripening
allows grain size to increase by merging smaller grains to form larger ones due to the potential difference in energy between tiny and enormous grains, which can occur via solid-state diffusion. The crystallite size of the ZnO NPs is given by:

\[ D = \frac{0.9 \lambda}{\beta \cos \theta_B} \]  

(1)

where \( \lambda \), \( \theta \) and \( \beta \) are the x-ray wavelength (1.540 56 Å), Bragg’s diffraction angle, and full width at half maximum of the ZnO (101) diffraction peak, respectively. As shown in figure 1 (c) and table 1, the crystallite size of ZnO NPs changed from 28 to 34 nm as the growth temperature rise from 35 to 75 °C. Figure 1 (d) shows that the intensity of the most intense peak increase with the growth temperature up to 65 °C and reduces after that, signifying an improvement in crystallinity of the ZnO nanoparticles.

Additionally, figure 1 (d) shows that the maximum peak position of relative intensities of the XRD spectra shifts slightly to a lower 2\( \theta \) angle with increasing growth temperature; this is evidenced by peak (101) location...
shift from 36.00 to 36.25 for growth temperatures 35 to 75 °C, respectively. This implies that a slight variation in the lattice parameters occurs as growth temperature increases, attributable to a change in the stress in the ZnO nanoparticles. Table 1 shows how crystallite sizes, lattice parameters, ratios of lattice constant c/a, positional parameters L, and Zn–O bond lengths (L) vary with changes in growth temperatures. In the ZnO hexagonal structure [21], the lattice constants \( a' \), and \( c' \) of ZnO NPs were determined using the relation below;

\[
\frac{1}{d_{(hkl)}^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c'^2}
\]

where \( d \) is the vertical distance between planes defined by the Miller indices \( h, k, \) and \( l \) and \( a, c \) is the lattice parameters. The expansion of lattice due to increased growth temperature is responsible for the increase in lattice parameters \( a' \) and \( c' \) as displayed in table 1 [22]. The change in lattice constants \( a' \) and crystallite size as a function of growth temperature is shown in figure 1(c). Singh et al. [23] observed that the lattice constant and crystallite size grew in lockstep with growth temperature. The length of the Zn–O bond, \( L \), was determined using the equation [24];

\[
L = \sqrt{\left(\frac{a^2}{3} + \left(\frac{1}{2} - u\right)^2 c^2\right)}
\]

where \( u \) is the wurtzite structure’s positional parameter and is a measure as to how far each atom is displaced from the next along the ‘c’ axis and is defined by;

\[
u = \frac{a^2}{3c^2} + 0.25
\]

Equations below [25] are used to quantify the cell volume and number of atoms per unit cell for hexagonal ZnO NPs;

\[
V = \frac{3\sqrt{3}}{2} a' c
\]

\[
n = \frac{4\pi}{3V} \left(\frac{D}{2}\right)^2
\]

where \( D \) is the crystallite size, and \( a \) and \( c \) are lattice parameters (nm).

Table 1 displays the bond lengths, cell volume, and number of atoms per unit cell of ZnO NPs determined for different growth temperatures. Continuous increases in bond lengths with an increase in growth temperatures are observed. The estimated average bond length is 19.88 nm, which is slightly longer than the recorded value of 19.77 nm for the bulk ZnO [26]. As a result, the bond length calculated corresponds to the Zn–O bond length in the unit cell. Table 1 also shows how the c/a ratios decrease as \( u \) values rise, resulting in a distortion of tetrahedral angles that keep the four tetrahedral distances of ZnO nearly constant [27]. The average c/a ratio of the lattice parameters is 1.603, which is marginally lower than for the bulk ZnO (1.604). Because of a slight change in \( 2\theta \) as a consequence of a rise in growth temperature, the inter-planar spacing increased.

### 3.1.2 Fourier transform infrared spectroscopy

The absorbance properties of ZnO NPs processed at various growth temperatures were analyzed by FTIR, as shown in figure 2, to deduce the form and existence of bonds present in ZnO NPs. The hydroxyl (–OH) groups are described by the absorbance peaks of about 3,400 cm\(^{-1}\) in the defined profile on the wavenumber axis, which indicate incomplete removal of organic solvent. At a wave number of about 1,500 cm\(^{-1}\), the absorbance of the carbon group (carbonyl carbon –C=O) induces the next absorption maxima. For all samples, the stretching vibrations of Zn–O bonding in octahedral configurations (Zn–O) at around 680 cm\(^{-1}\) have been attributed to fragile bands [28–31]. The spectra for the samples generally show minimal% absorbance (between 3 and 9%), implying great purity, which is verified by XRD data. However, because the band’s intensity increases with growth temperature, more organic solvents are seen at high growth temperatures, indicating more impurity than at lower growth temperatures. Figure 2 illustrates the highest % transmittance at all wavenumbers to be for samples grown at room temperature. The trend of percentage (%) transmittance of ZnO NPs, generally declines with the growth temperature up to 65 °C. A deeper look at the wavenumber about 700 cm\(^{-1}\) reveals that the absorption band at 680 cm\(^{-1}\) is slightly shifted with growing temperature, implying that the quantity of organic impurity affects the O–Zn–O link in the ZnO structure.

### 3.2 Thermal gravimetric analysis

Thermogravimetric analysis (TGA) is an analytical technique that monitors the weight change that occurs as a sample is heated at a constant pace to assess a material’s thermal stability and the fraction of volatile components. The TGA graph of ZnO nanoparticle growth at different temperatures is shown in figure 3. TGA curves for ZnO nanoparticles with varying growth temperatures displayed a different temperature gradient. Since the...
samples were made with ethanol as a solvent, there are traces of ethanol in the products. Peaks observed around 57 °C represent the loss of mass due to the evaporation of ethanol. The decrease of mass observed at 170 °C could be due to decompositions of Zn(OH)$_2$, which liberate water molecules. Due to the evaporation of the crystallization water, a further 5% loss of mass was observed at about 600 °C. These findings were in line with previous research [31]. When water molecules are removed, crystallization occurs. More sample residue was observed due to a gradual drop in % weight at high temperatures (75, 65, and 55 °C), resulting in a 3.5% weight loss of ZnO NPs. However, at low temperatures (RT, 35, and 45 °C), a slightly steeper drop in % weight was observed, leading to a 6% loss in weight.

3.3 Surface morphological analysis
SEM spectra, figures 4(a)–(c), show increased ZnO NPs sizes with increasing growth temperature. The SEM analysis verified that the morphology of ZnO nanoparticles was modified from needle-like to spherical as the growth temperature increased from 35 to 75 °C. Other researchers [32] also observed similar results. The formation of ZnO nanostructures is regulated by diffusion. With increasing growth temperature, the diffusion of
adatoms on the nanoparticle surface becomes smoother and faster. Furthermore, it should be remembered that at low temperatures, the ad atoms’ mobility values are also low, resulting in a low working diffusion solution. Accordingly, more adatoms will diffuse to the tips at a higher temperature, which is a thermodynamically favorable location, instead of staying on the side surface of nanoparticles [33]. Spherical particles are formed at higher growth temperatures (figures 4(b) and (c)); while at lower temperatures figure 4(a) needle-like rods particles were observed. Mukherjee et al used a simple chemical approach to make Ag-doped ZnO nanoparticles at a low temperature (65°C) by adjusting the quantity of Ag [34]. Control over the size and form of the NPs was accomplished during the synthesis phase. Shape deformation of the NPs from the sphere for undoped samples to the rod for doped samples is discovered, which is significant. The kinetic energy for the formation of different morphologies of ZnO is known to be determined by growth temperature, which is a significant experimental parameter. As a result, before using ZnO in various applications, it should be considered for the creation of various ZnO morphologies. Yuan et al [35] investigated the effects of various oxidation temperatures on ZnO growth morphologies and microstructure. It was observed that for oxidation below the melting point of Zn, ZnO has bicrystalline nanowire morphology, single-crystalline morphology between the melting and boiling points of Zn, and tetrapod morphology above the boiling point of Zn. Lim et al [36] reported on the production of ZnO nano-coalesced microstructures with various morphologies via thermal evaporation at 500, 600, and 700 °C, without the need for a metal catalyst or a pre-deposited ZnO seed layer. With raising the synthesis temperature, a temperature-driven morphological evolution of the zinc oxide nano-coalesced microstructures was observed, culminating in the change of quasi-mountain chain-like to pyramidal textured zinc oxide.

3.4 Photoluminescence analysis
Photoluminescence study assists with discovering the primary imperfections in nanoparticles. Defect density and crystallite size have an effect on the PL intensity and peak location of ZnO NPs. Figure 5(a) shows the room temperature PL excitation spectra. A significant change in PL intensity with growth temperatures is displayed. Figure 5(a) depicts a shift to the higher excitation wavelength of peak 381 nm for growth temperature up to 55 °C as the growth temperature increases.

![Figure 4. SEM micrographs of ZnO nanoparticles synthesized at (a) 35 °C, (b) 55 °C and (c) 75 °C.](image)
However, the excitation peaks at larger wavelengths shifted from 375 to 382 nm with increased growth temperature. Figure 5(b) depicts the dependence of PL emission spectra at room temperature for ZnO NPs prepared at various growth temperatures. The spectra display emissions centered at 391 and 546 nm (when probed at 381 nm) representing UV and visible emissions, respectively, with varied relative intensity and exact location of the emission peaks. The UV luminescence peak correlates to the near band-edge emission. The recombination of excitons is generally responsible for it. In contrast, the green emission peak is due to a deep level emission or trap-state emission corresponding to the singly ionized oxygen vacancy in the ZnO (oxygen-related defects) resulting from the recombination of the photons generated holes with the single ionized charged state of this defect. The excitonic peak emission is attributed to the band edge emission, while the green emission at around 546 nm is a tribute to the defect level emission (DLE). Using electron paramagnetic resonance, multiple, alternative contributions to the sources of green luminescence at 554 nm and 504 nm were proposed by Vlasenko et al [37], Ahn et al [38], and Cao et al [39]. One of their suggested contributions was also found in our results and matches their findings very well. The transition from \( Z_{\text{in}} \) to \( V_{\text{o}} \) is thought to be responsible for the
component at 554 nm, while the transition from conduction band (CB) to V_{o} is thought to be responsible for the component at 504 nm. The excitonic peak intensity rises with growth temperature, while the visible emission peak intensity rises with growth temperature until it exceeds 35 °C, after which it decreases. Figure 5(c) shows a steady increase in excitonic peak intensity until 65 °C, then a general decrease in visible emission peak intensities after 35 °C, showing a clear reduction in defect density as growth temperature rises and implying an improvement in crystallinity as well. This increase in UV luminescence intensity could be owing to slightly larger crystallite sizes as the synthesis temperature rises, resulting in higher radiative recombination. The smaller surface-to-volume ratio of smaller nanoparticles, which favour more increased defects and surface recombination, is credited with the decrease in DLE emission intensity [40]. The dissociation of donor-bound excitons into free excitons and neutral donors is also thought to be the cause of the increase in UV emissions at higher growth temperatures. This can make it more likely for free excitons to recombine, resulting in more UV emission intensity [41]. Surface defect passivation is another possible mechanism for lower DLE emission and enhanced excitonic emission as samples are manufactured at higher growth temperatures. The UV: DLE emission intensity ratio is directly related to the crystal quality and surface states which are the two major factors that determine the optical properties of ZnO nanoparticles. It also dictates the possible applications of ZnO nanoparticles for UV sensors applications. Figure 5(d) demonstrates how the ratio of relative intensities of the green to UV emission is reduced with the increasing growth temperature, contrary to the report by Kim et al [42], who indicated that the ratio generally reduces with the growth temperatures. In this case, the ratio increases with the growth temperature from 25 to 35 °C and reduces thereafter until the growth temperature of 75 °C. Figure 5(e) shows the variation of emission wavelengths of both UV/DLE emissions. The emission peak positions are both affected by growth temperatures. The peaks shift somewhat to longer wavelengths (about 392/547 nm) at high growth temperatures compared to 389/545 nm for lower growth temperatures. The redshift in the visible emission and UV emission with increasing crystallite size closely follows the redshift in the band edge emission, indicating that they are related. The increase in crystallite size and the resulting shifts in tensile stress due to lattice distortion are thought to be the cause of the shift in band edge emission. Since the lattice reorganization is related to the electron-hole density, the shift will be enhanced substantially. Therefore, the photoluminescence study revealed here reasons that the kind of emission from the ZnO NPs, i.e., regardless of whether UV (or) violet, is dictated by the growth temperature of the precursor solution.

3.5 Optical reflectance analysis

The diffuse reflectance of the ZnO nanoparticles was obtained using the UV–Visible spectra. It is known that the absorbance or reflectance spectra can be used to probe the crystallite molecular orbital and provide information concerning size, particle concentrations, and composition [38]. The optical reflectance spectra of ZnO nanoparticles grown at various temperatures are shown in figure 6(a). The absorption in the visible region changes with growth temperature. The percentage absorption of excitation energy reduces from 80% to just above 70% with an increase in growth temperatures, while an opposite effect was observed in the UV region. The intrinsic bandgap absorption of ZnO caused by electron transitions from the valence band to the conduction band induces a sharp band edge at 385 nm, as shown in figure 6(a). With decreasing particle size, the onset of the absorption band edge was blue-shifted, confirming the size-dependent absorption properties of ZnO NPs. In a semiconductor with a direct bandgap, the bandgap energies of ZnO NPs were estimated using the Kubeka–Munk remission function [43]. As the growth temperature increased, the approximate bandgap decreased from 3.31 to 3.24 eV, as shown in figure 6(b). Tensile strain causes the bandgap for ZnO to decrease, while compressive strain causes the bandgap to increase, according to Liao et al [44]. UV measurements depict a shift in absorption edge confirming the changes in particle sizes with varying growth temperatures. A wide bandgap is anticipated in a compacted lattice due to increased repulsion between the oxygen 2p and zinc 4s bands. However, the projected bandgap is smaller than that of bulk ZnO. (3.37 eV). The density of surface defects in undoped ZnO can cause a bandgap reduction that varies depending on the synthesis method [45]. The non-uniformity in particle sizes may be due to the difference in the slope of different energy curves, as confirmed by SEM analysis.

4. Conclusions

The sol-gel process was used to successfully prepare ZnO NPs with hexagonal wurtzite structures. The effect of growth temperature on the structure, crystallite size, bandgap, and PL were investigated. As the growth temperature rises from RT to 75 °C crystallite sizes increased correspondingly. SEM results also showed increased ZnO NPs sizes with an increase in growth temperatures. The excitonic emission observed from PL measurement showed an index of good crystal quality. Increased growth temperature significantly quenched the green emission, and the peak positions and intensities are both affected by growth temperatures. The estimated bandgap reduced from 3.31 to 3.24 eV with the rise in the growth temperature due to tensile strain, while
compressive strain increases the bandgap. However, the bandgap from the analysis of the reflectance curve was observed to be lower than that of the bulk ZnO (3.37 eV). The bandgap reduction may be due to surface defects density of undoped ZnO. FTIR measurement illustrated the highest % transmittance at all wavenumbers to be at room temperature. The trend of % transmittance of ZnO NPs, generally declined with the rise in growth temperature.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors. https://www.wsu.ac.za

ORCID iDs

F B Dejene @ https://orcid.org/0000-0002-4474-199X

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Figure 6. (a) The reflectance spectra of ZnO nanoparticles prepared at various temperatures, and (b) Graph to determine the bandgap energy of ZnO NPs prepared at various temperatures.
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