CHAPTER VI

STRUCTURAL, OPTICAL AND ELECTRICAL PROPERTIES OF ZnO-ZnS NANOCOMPOSITES

6.1 Introduction

Over the past decades, the terms nanomaterial and nanocomposite signify fresh and exhilarating fields in materials science. The domain of nanocomposite materials has had the attention, insight, and close analysis of scientists and engineers in recent years. Such materials have actually been used for centuries and have always existed in nature. A nanocomposite is defined as a combined material where at least one of the dimensions, of one of its elements is on the nanometre size scale [1]. Nanocomposites have drawn the fascination and attention of many researchers due to their budding applications in biomedical [2], catalytic [3], separation [4], chemical sensing [5], fuel cells [6], capacitors [7], micro fabrication [8], tribological [9], resonant coupling [10], high flux gas transport [11] and many more.

Wide band gap semiconductor materials have attracted enormous attention due to their size-dependent properties and appreciable technological applications. Among the metal oxide nanostructures ZnO and ZnS are alluring materials because of their unique properties; their high surface area to volume ratio, their energy band gap of 3.37eV and 3.54eV respectively.

The nanostructured ZnO is a multifunctional material with promising features for a variety of practical applications, such as piezoelectric transducers [12], optical waveguides, surface acoustic wave devices, varistors, phosphors, transparent conducting oxides, functional spin-electronic devices, field emitters[13, 14] and UV-light emitters or detectors [15–19]. Surface modification of a semiconductor with semiconductors of wider band-gap has been recognized as one of the most adequate and intriguing methods to improve the luminescence properties [20]. The properties of ZnO could be significantly improved by encapsulating it with some other nanoparticles having wide bandgap nanoparticles [21].

ZnS is non-toxic semiconductor with a wide and direct band gap which can be observed naturally in two phases, first is the zinc blend structure with a cubic phase, and second is the wurtzite structure with a hexagonal phase [22]. The band-gap energy of the bulk cubic
zinc oxide is 3.54eV and has established wide research interest because of its potential applications in various fields such as nonlinear optical devices, displays, sensors, infrared windows, and lasers [23–25].

The photoconductive properties determine how effective and responding the material will be; that is, the higher the mobility, the faster the response and the faster the material can be optically scanned. It is assumed that the study of the photoconductive properties will lead to more efficient materials. In addition, it would be of practical importance to develop materials sensitive over a broad region of wavelengths [26]. Photoconductivity is an optical and electrical phenomenon in which materials become more electrically conductive due to the absorption of electromagnetic radiation such as visible light, ultraviolet light, infrared light, or gamma radiations. It is due to the effect of increasing electrical conductivity $\Phi$ in a solid because of light absorption. When the so-called internal photo effect takes place, the energy absorbed enables the transition of activator electrons into the conduction band and the charge exchange of traps with holes being created in the valence band. In the process, the number of charge carriers in the crystal lattice increases and as a result, the conductivity is enhanced.

In this chapter, we have synthesized zinc oxide-zinc sulphide nanocomposite by hydrothermal method and the optical, structural and photoconductivity properties have been investigated. Photoconductive materials usually exhibit an increase in conductivity under illumination which is a direct result of the absorption of photon energy greater than the band-gap of the material. This phenomenon is known as positive photoconductivity. Most of the semiconductors are found to exhibit positive photoconductivity [27–34].

6.2 Materials

Zinc Nitrate ($\text{Zn (NO}_3\text{)}_2.6\text{H}_2\text{O}$), Sodium Hydroxide (NaOH), Zinc sulfate heptahydrate ($\text{ZnSO}_4.7\text{H}_2\text{O}$), Sodium Sulphide ($\text{Na}_2\text{S}$), were used as the starting materials. All the chemicals were purchased from Merck, were of analytical grade and used as received. Double distilled water was used during the experimental process.

6.3 Experimental

The two base materials, pure zinc oxide (ZnO) and pure zinc sulfide (ZnS) are taken in the powdered form. These proportions are ground properly in order to get a homogeneous mixture. The mixture is placed in a crucible and annealed in a muffle furnace at 500 °C for
3hrs. The heated material is then cooled down to room temperature and ground once again in order to get microcrystalline structures. Nanocomposites of ZnO-ZnS with molar ratios [25:75], [50:50] and [75:25] were prepared by this procedure.

6.4 Characterization Techniques

After drying, the synthesized samples were characterized by several techniques. The X-ray power diffraction (XRD) analysis conducted by the Siefert 3003T/T X-Ray diffractometer with CuKα (λ = 1.5406 Å) radiation in the 2θ range 20 to 70 ° was used to identify the structural phase composition. The sample morphology was examined by using a FEI Quanta FEG 200 Scanning Electron Microscope equipped with an Energy Dispersive X-Ray Analyser (EDAX) detector. Optical absorption spectra of the powdered samples were recorded using the double beam Perkin Elmer (model Lambda 35) spectrophotometer in the range 190-1100 nm and their band gaps were calculated. Using Fourier Transform Infrared Spectrometer, FTIR–Perkin Elmer model Spectrum RX 1, the spectrum was recorded in the range of 400-4000 cm⁻¹. The electrical and photoconductivity properties of the samples were measured by the HIOKI 3532-50 LCR Hitester and Keithley electrometer.

6.5 Results and Discussion

6.5.1 X-Ray Diffraction Analysis

The XRD patterns of the ZnO-ZnS nanocomposites with different molar ratios were recorded in the range 20 to 80 ° using a Siefert 3003T/T X-Ray diffractometer with filtered CuKα radiation of wavelength λ=1.5406 Å. Figure 6.1 shows the XRD pattern for ZnO-ZnS nanocomposite annealed at 500 °C. For the nanocomposite, all the diffraction peaks can be indexed to the hexagonal wurtzite ZnO and the cubic ZnS (Figure 6.1), which is well consistent with the JCPDS file nos. 89-1397 and 05-0566, respectively. No other crystalline impurities are detected. The peak structure remains almost the same for the ZnO-ZnS (25:75) ratio. Following further increase in the ZnO ratio, the intensity is observed to increase which is gradually observed in the spectrums of ZnO-ZnS (50:50) ratio to ZnO-ZnS (75:25). The XRD peak positions of the ZnO-ZnS nanocomposites are in very good agreement with those of the bulk ZnO and ZnS crystal. Furthermore, it is be noted that the full width at half-maximum (FWHM) of the diffraction peaks of ZnO and ZnS crystal is very broad, which indicates a decrease in the crystalline size of ZnO and ZnS. The average size of the ZnO and ZnS crystals is calculated according to Scherrer formula.
\[ d = \frac{k \lambda}{\beta \cos \theta} \]  

(1)

\( k \) is the shape factor equal to 0.89, \( \lambda \) is the X-ray wavelength for CuK\( \alpha \) radiation (1.5406 Å), Similarly, the average size of the bare ZnO and ZnS nanoparticles are 46.23 nm, 23.06 nm respectively. The lattice parameters of the 3 different molar ratios are tabulated below [Table 6.1]

![XRD pattern of the nanocomposite of ZnO-ZnS for the different molar ratios](image)

**Figure 6.1**: XRD pattern of the nanocomposite of ZnO-ZnS for the different molar ratios
XRD – Lattice Parameters

<table>
<thead>
<tr>
<th></th>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Structure</td>
<td>Hexagonal</td>
<td>Hexagonal</td>
<td>Hexagonal</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>a = b (Å)</td>
<td>3.253</td>
<td>3.242 ± 0.002</td>
<td>3.239 ± 0.016</td>
<td>3.258 ± 0.006</td>
</tr>
<tr>
<td>c(Å)</td>
<td>5.213</td>
<td>5.187 ± 0.004</td>
<td>5.218 ± 0.014</td>
<td>5.209 ± 0.005</td>
</tr>
<tr>
<td>V (Å³)</td>
<td>47.77</td>
<td>47.50</td>
<td>47.17</td>
<td>47.68</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Structure</td>
<td>Cubic</td>
<td>Cubic</td>
<td>Cubic</td>
<td>Cubic</td>
</tr>
<tr>
<td>a = b=c (Å)</td>
<td>5.406</td>
<td>5.386 ± 0.001</td>
<td>5.383 ± 0.002</td>
<td>5.398 ± 0.009</td>
</tr>
<tr>
<td>V (Å³)</td>
<td>157.99</td>
<td>156.22</td>
<td>157.94</td>
<td>156.76</td>
</tr>
</tbody>
</table>

(a) Calculated lattice parameters for ZnO and ZnS

<table>
<thead>
<tr>
<th>XRD Peaks of</th>
<th>Molar Ratio</th>
<th>2θ</th>
<th>hkl</th>
<th>β</th>
<th>d (nm)</th>
<th>ε (×10⁻³)</th>
<th>δ×10¹⁵ (lines/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnO</td>
<td>25:75</td>
<td>36.48</td>
<td>101</td>
<td>0.1959</td>
<td>44.61</td>
<td>2.6</td>
<td>1.039</td>
</tr>
<tr>
<td></td>
<td>50:50</td>
<td>36.37</td>
<td>101</td>
<td>0.1899</td>
<td>46.01</td>
<td>2.10</td>
<td>2.49</td>
</tr>
<tr>
<td></td>
<td>75:25</td>
<td>36.50</td>
<td>101</td>
<td>0.1807</td>
<td>48.07</td>
<td>2.33</td>
<td>2.641</td>
</tr>
<tr>
<td>ZnS</td>
<td>25:75</td>
<td>28.74</td>
<td>111</td>
<td>0.2842</td>
<td>24.61</td>
<td>1.04</td>
<td>1.30</td>
</tr>
<tr>
<td></td>
<td>50:50</td>
<td>28.80</td>
<td>111</td>
<td>0.4024</td>
<td>21.3</td>
<td>2.9</td>
<td>4.15</td>
</tr>
<tr>
<td></td>
<td>75:25</td>
<td>28.72</td>
<td>111</td>
<td>0.3681</td>
<td>23.28</td>
<td>1.10</td>
<td>1.405</td>
</tr>
</tbody>
</table>

(b) Crystallite size, average strain and dislocation density calculated from XRD for ZnO and ZnS

Table 6.1: Tabulation of the Lattice parameters and other calculated data from XRD analysis for the nanocomposite of ZnO-ZnS for different molar ratios.
Williamson and Hall plot calculations

<table>
<thead>
<tr>
<th>Ratio</th>
<th>Compound</th>
<th>Scherrer’s Method</th>
<th>W-H Method</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$D_{\text{avg}}$ (nm)</td>
<td>D (nm)</td>
</tr>
<tr>
<td>25:75</td>
<td>ZnO</td>
<td>44.61</td>
<td>40.61</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>24.61</td>
<td>21.75</td>
</tr>
<tr>
<td>50:50</td>
<td>ZnO</td>
<td>46.01</td>
<td>40.05</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>21.3</td>
<td>19.43</td>
</tr>
<tr>
<td>75:25</td>
<td>ZnO</td>
<td>48.07</td>
<td>39.05</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>23.28</td>
<td>22.21</td>
</tr>
</tbody>
</table>

Table 6.2: Comparison of the Geometric parameters obtained from the Scherrer formula and W-H plot from the nanocomposite of ZnO-ZnS for different molar ratios.

Williamson and Hall proposed a method for deconvoluting size and strain broadening by looking at the peak width as a function of $2\theta$. The strain-induced broadening in powders due to crystal imperfection and distortion are related by the formula

$$\varepsilon = \frac{\beta}{4\tan\theta}$$

In particular, the W–H analysis is a simplified integral breadth method where strain-induced broadening arises from crystal imperfections and distortion in the lattice [35–37]. The W-H method does not follow a $1/\cos\theta$ dependency as in the Scherrer equation but instead varies with $\tan\theta$. This fundamental difference allows for a separation of reflection broadening when both microstructural causes - small crystallite size and microstrain - occur together [38]. The size and strain effects on peak widening are known from the above difference of $2\theta$. Size-induced and strain-induced broadenings are known by considering the peak width as a function of $2\theta$ [39]. From the plot of $\beta\cos\theta$ against $4\sin\theta$, a straight line with gradient and intercept $0.9/t$ is obtained. The strain and crystallite size are calculated from the slope and y-intercept of the fitted line respectively. Table 6.2 gives a correlation of data acquired from the Scherrer’s formula and the Williamson-Hall plot.
6.5.3 Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy

(a) ZnO - ZnS [25:75]

(b) ZnO - ZnS [50:50]
To study the morphology of the nanocomposites the SEM technique has been effectively employed. Figure 6.2(a) shows the magnified image of cluster-like morphology. The nanoparticles are seen to self-assemble to form complex hierarchical clustered structures. Figure 6.2(b) shows the SEM pictures which indicates that the sample is composed of large quantities of cube-like particles. It is also evident that the particles are attached to each other through the grain boundaries to form agglomerated particles. Figure 6.2(c) is the SEM image of the nanocomposite sample synthesized using the hydrothermal method. The morphology indicates a similar predominant cluster of flakes like morphologies.

The SEM micrographs of ZnO-ZnS nanocomposite were taken at different magnifications. In all three molar ratios it is observed that cluster-like morphology was evolved throughout, though they were oriented in different geometries.

Energy dispersive X-ray spectroscopy was employed to analyse the chemical composition. The spectra obtained during EDAX studies were used for carrying out the quantitative analysis of ZnO-ZnS nanocomposite. Figure 6.3 represents the histogram of the atomic percentage of the elements present at different molar ratios. From the histogram, it is observed that the atomic percentage of Zn and S are high in all three molar ratios accounting for the strong Zn peaks that were observed in the XRD spectra.
Figure 6.3: Histogram for the EDAX analysis (atomic weight %) for the nanocomposite of ZnO-ZnS for the three molar ratios

6.5.4 UV-Visible absorption study

The diffuse reflectance spectra were obtained using a UV-Vis. spectrophotometer. The ZnO-ZnS nanocomposites were subjected to ultraviolet-visible (UV-Vis) spectroscopy analysis and the spectrum were recorded in the wavelength range of 200 to 800 nm. The UV-Visible absorption spectrum of the samples are shown in Figure 6.4. All the three molar ratios have a strong absorption maximum below 400 nm. In the case of this nanocomposite, it is known that zinc oxide (ZnO) and zinc sulphide (ZnS) have their band gaps very close to each other (3.3 eV and 3.54eV) respectively. The absorption maximum of the samples is observed to be shifted to the lower.
Figure 6.4: UV-Visible absorption spectra for the nanocomposite of ZnO-ZnS for the three molar ratios

Table 6.3: Tabulation of the observed excitation wavelengths and the comparison between the bulk and calculated band gaps of the nanocomposite of ZnO-ZnS for the different molar ratios

<table>
<thead>
<tr>
<th>Sample</th>
<th>Standard Band gap (eV)</th>
<th>$\lambda_{ex}$ (nm)</th>
<th>Band gap (eV)</th>
<th>Band gap difference (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25:75</td>
<td>ZnO</td>
<td>3.37</td>
<td>335</td>
<td>3.71</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>3.54</td>
<td>335</td>
<td>3.71</td>
</tr>
<tr>
<td>50:50</td>
<td>ZnO</td>
<td>3.37</td>
<td>338</td>
<td>3.68</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>3.54</td>
<td>338</td>
<td>3.68</td>
</tr>
<tr>
<td>75:25</td>
<td>ZnO</td>
<td>3.37</td>
<td>333</td>
<td>3.73</td>
</tr>
<tr>
<td></td>
<td>ZnS</td>
<td>3.54</td>
<td>333</td>
<td>3.73</td>
</tr>
</tbody>
</table>
6.5.5 Fourier Transform Infrared Spectroscopy

Spectroscopic investigations were carried out for all the samples of the ZnO-ZnS nanocomposites. The spectrographs are presented in Figure 6.5 and the wavelengths assigned are presented in Table 6.4.

![FTIR spectra](image)

**Figure 6.5 FTIR spectra for the nanocomposite of ZnO-ZnS for different molar ratios**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Wavenumber (cm(^{-1}))</th>
<th>Assignments</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnO:ZnS [25:75]</td>
<td>522.23</td>
<td>Zn-O stretching vibration</td>
</tr>
<tr>
<td></td>
<td>660.12</td>
<td>Zinc and sulphide Stretching vibration</td>
</tr>
<tr>
<td></td>
<td>1117.30</td>
<td>C-O Stretching vibration</td>
</tr>
<tr>
<td>Wavenumber (cm⁻¹)</td>
<td>Description</td>
<td></td>
</tr>
<tr>
<td>------------------</td>
<td>-----------------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td>3410</td>
<td>Zinc hydroxyl groups</td>
<td></td>
</tr>
<tr>
<td>448.18</td>
<td>Zn strong absorption</td>
<td></td>
</tr>
<tr>
<td>677.32</td>
<td>Zn-S bond</td>
<td></td>
</tr>
<tr>
<td>876.46</td>
<td>Vibrational frequency of ZnO</td>
<td></td>
</tr>
<tr>
<td>1115.06</td>
<td>C-C Stretching vibration</td>
<td></td>
</tr>
<tr>
<td>1631.18</td>
<td>presence of –OH residues</td>
<td></td>
</tr>
<tr>
<td>3407.67</td>
<td>Existence of hydroxyl groups on the surface of the sample</td>
<td></td>
</tr>
<tr>
<td>505.77</td>
<td>Zn-O (associated with oxygen deficiency or oxygen vacancy defect complex in ZnO)</td>
<td></td>
</tr>
<tr>
<td>611.30</td>
<td>Zn-S bond</td>
<td></td>
</tr>
<tr>
<td>1110.49</td>
<td>C-O Stretching vibration</td>
<td></td>
</tr>
<tr>
<td>1617.50</td>
<td>Due to deformation vibrations of H₂O molecules</td>
<td></td>
</tr>
<tr>
<td>3370.45</td>
<td>Broad band corresponding to the stretching mode of the OH group contributed by the water content in the sample. [40-47]</td>
<td></td>
</tr>
</tbody>
</table>

Table 6.4: Tabulation of the observance for the wavenumbers from the FTIR spectra for the nanocomposite of ZnO-ZnS for different molar ratios

6.5.6 Dielectric Studies

The dielectric properties of materials are primarily determined by their polarization at a certain frequency. For multicomponent systems, when free charge carriers drift through the material, space charges build up at the interfaces of the elements owing to the mismatch of the conductivities and dielectric constants of the materials at the interfaces [48]. The dielectric behaviour is mainly attributed to the contributions from the ionic, electronic, space-charge and dipolar polarizations. The dielectric constants of the ZnO-ZnS nanocomposite
pellets were measured using HIOKI 3532 LCR Hi TESTER in the frequency range from 50 Hz to 5 MHz. The as-prepared samples were first pelletized and then loaded between the two electrodes placed in a sealed chamber and analysed. It is clear from the Figure 6.6 that the dielectric constant decreases with the increase in the frequency of the applied voltage. And also the a.c. conductivity increases with the increase in the frequency of the applied voltage. It is shown experimentally that for each sample, the dielectric loss is very high at very low frequencies, decreases significantly with increasing frequency and reaches a constant value at high frequencies.

6.5.6.1 Dielectric Constant for the nanocomposite of ZnO-ZnS

![Graph showing dielectric constant vs log f]

a) Dielectric constant of ZnO-ZnS (25:75)
b) Dielectric constant of ZnO-ZnS (50:50)

c) Dielectric constant of ZnO-ZnS (75:25)

Figure 6.6: Dielectric Constant plots for the nanocomposite of ZnO-ZnS for different molar ratios
6.5.6.2 Dielectric Loss for the nanocomposite of ZnO-ZnS

a) Dielectric constant of ZnO-ZnS (25:75)

b) Dielectric constant of ZnO-ZnS (50:50)
c) Dielectric constant of ZnO-ZnS (75:25)

Figure 6.7: Dielectric Loss plots for the nanocomposite of ZnO-ZnS for different molar ratios

6.5.6.3 A.C. Conductivity analysis for the nanocomposite of ZnO-ZnS

a) A.C. Conductivity of ZnO-ZnS (25:75)
b) A.C. Conductivity of ZnO-ZnS (50:50)

c) A.C. Conductivity of ZnO-ZnS (75:25)

Figure 6.8: A.C. Conductivity plots for the nanocomposite of ZnO-ZnS for different molar ratios
6.5.7 Photoconductivity

Photoconductivity and dark conductivity behavior of ZnO-ZnS nanocomposite in visible light was studied and is represented in Figure 6.9. The dependence of photocurrent and dark current on several parameters such as changing field, intensity of illumination, wavelength and rise and decay time response is known. In this chapter, the variation of the dark and photo currents with applied voltage at different temperatures have been studied extensively. The photo conductivity under white halogen lamp illumination and dark conductivity were measured with the help of Keithley 6514 System Electrometer. The value of the photocurrent (photopeak) is seen to increase with increasing temperature. When the temperature is low, the variation in photoconductivity is very small and approaches a constant value for all compositions. Photoconductivity is found to be larger than dark conductivity in the entire range of temperature. It is experimentally proved from the plot that the photocurrent is greater than dark current suggesting that ZnO-ZnS nanocomposites at all the three molar ratios exhibit positive photoconductivity. The super linear behavior suggests that additional charge carriers are injected into the sample from one of the electrodes [49].

![Photoconductivity of ZnO-ZnS (25:75)](image)

a) Photoconductivity of ZnO-ZnS (25:75)
b) Photoconductivity of ZnO-ZnS (50:50)

c) Photoconductivity of ZnO-ZnS (75:25)

Figure 6.9: Variation of current with applied voltage under darkness and white halogen lamp illumination for ZnO-ZnS nanocomposite.
6.6 Conclusion

In summary, ZnO-ZnS nanocomposites are successfully synthesized by the hydrothermal method for three different molar ratios. The variation in the properties of the nanocomposites due to change in the molar ratios were investigated. The powder XRD results demonstrate that the samples have peaks of both zinc oxide with a hexagonal and zinc sulphide with cubic structure with good crystallinity. Using the XRD data the crystalline size was determined by the Williamson-Hall method and with the Scherrer’s formula. In addition, the dislocation density and lattice strain were calculated for all the samples. The SEM analysis revealed the morphological features of the nanocomposites whereas the EDAX studies confirmed the presence of the elements. From the optical absorption spectra, the values of the band gaps of ZnO and ZnS were observed to exhibit a blue shift which may be attributed to the transition of electrons, mediated by oxygen vacancies. FTIR revealed the presence of all the characteristic functional groups of the nanocomposites ZnO-ZnS. For all the samples, the photoconductivity is found to increase linearly with the applied voltage at all temperatures. This behavior suggests that additional charge carriers are injected into the sample from one of the electrodes. The dielectric analysis revealed the change in the dielectric parameters and AC conductivity of the nanocomposites in response to the applied voltage with increase in frequency at different temperatures.

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