CONDESSED MATTER PHYSICS | RESEARCH ARTICLE

Structural, optical, electrical, and magnetic properties of Zn\textsubscript{0.7}Mn\textsubscript{x}Ni\textsubscript{0.3−x}O nanoparticles synthesized by sol–gel technique

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Abstract: The structural, morphology, optical, electrical, and magnetic properties of Zn\textsubscript{0.7}Mn\textsubscript{x}Ni\textsubscript{0.3−x}O (x = 0.05, 0.1, 0.15, 0.2) nanoparticles synthesized by sol–gel technique have been systematically investigated by using X-ray diffractometer (XRD), scanning electron microscope (SEM), UV–vis-NIR spectrophotometer, impedance analyzer, and vibration sample magnetometer (VSM). XRD patterns reveal that all samples have hexagonal wurtzite structure along with secondary phases such as NiO and ZnMnO₃. The average crystalline size increases with the increase in the Mn concentration in the host matrix. Diffuse reflectance studies (DRS) show an increment in optical band gap with increasing Mn content. AC conductivity of present samples has been studied as a function of frequency (100 Hz–10 MHz) of the applied AC signal in the temperature range of 323–463 K. The results showed that AC conductivity increases with an increase in frequency and temperature. The frequency exponent shows that small polaron conduction mechanism is the most favorable for all samples. The value of AC conductivity is observed to decrease with an increase in the Mn dopant concentration in the Zn\textsubscript{0.7}Mn\textsubscript{x}Ni\textsubscript{0.3−x}O system. At room temperature, Mn-modified Ni–ZnO nanoparticles are dilute magnetic semiconductors (DMSs), which exhibit magnetic, magneto-electronic, and magneto-optical properties, and hence suitable for spintronics devices.

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In present research paper, the structural, optical, electrical, and magnetic properties of Ni and Mn (Transition metals)-modified ZnO nanoparticles (synthesized by sol–gel technique) have been discussed in detail, being dilute magnetic semiconductors, these materials may be suitable for spintronics devices.

PUBLIC INTEREST STATEMENT

Mn-modified Ni–ZnO nanoparticles are dilute magnetic semiconductors (DMSs), which exhibit magnetic, magneto-electronic, and magneto-optical properties, and hence suitable for spintronics devices. So, keeping in view these technological applications, the influence of different Mn concentrations on structural, optical, electrical, and magnetic studies has been investigated in the present work.
magnetic characterization of the samples indicates the presence of both paramagnetic and ferromagnetic behavior. Magnetic saturation decreases with the increase in the Mn concentration in the host lattice.

Subjects: Condensed Matter Physics; Microscopy; Semiconductors

Keywords: X-ray diffraction; diffuse reflectance studies; AC conductivity; magnetic studies

1. Introduction
Semiconducting nanomaterials have attracted great scientific attention due to their unique properties for different nanotechnological potential applications (Jagadish & Pearton, 2006). ZnO (II–VI semiconductor)-based nanomaterials have recently attracted attention due to their wide direct band gap (3.37 eV) and large exciton binding energy (60 meV) for various applications in areas such as gas sensors, solar cells, laser diodes, ultraviolet (UV) luminescence devices, transparent thin transistors, biosensors, spintronics, and antibiotic materials (Al-Hardan, Abdullah, & Aziz, 2009; Nanto et al., 1986; Navale, Ravi, Mulla, Gosavi, & Kulkarni, 2007). Transition metals (TMs) such as Ni, Fe, Mn, Co, Cr, V, etc. are doped into ZnO to form dilute magnetic semiconductors (DMSs), which exhibit magnetic, magneto-electronic, and magneto-optical properties suitable for spintronics devices (Ohno, 1998). The addition of TM dopant in ZnO induces dramatic changes in structural, thermal, optical, electrical, and magnetic properties. The conductivity and optical properties depend on the amount of doping in ZnO. The electrical conductivity is influenced by the impurity atoms, defects, and grain boundaries which change the electric potential of the lattice (Karthik, Pandian, & Jaya, 2010). The band gap engineering using various dopants like Mn, Ni, Co, Cr has been reported (Bhat & Deepak, 2005; Fukumura, Jin, Ohtomo, Koimuma, & Kawasaki, 1999). DMS (TM–Mn-doped ZnO) exhibits room temperature ferromagnetism as has been observed by several researchers (Bhat & Deepak, 2005; Viswanatha et al., 2004). The formation of secondary phases in TM-doped ZnO significantly influences the structural, optical, transport, and magnetic properties of ZnO (Bhargava, Sharma, Kumar, Pandey, & Kumar, 2010). For Mn-doped ZnO nanoparticles many researchers have reported different magnetic behavior such as ferromagnetism (Jung et al., 2002), spin glass behavior (Fukumura et al., 2001), and paramagnetism (Tiwari et al., 2002). Nirmala, Smitha, and Anukalian (2011) have studied the optical and electrical properties of (Mn, Co) co-doped ZnO nanoparticles synthesized by DC thermal plasma method. The significant enhancement of optical and magnetic properties of (Cu, Cr) co-doped ZnO nanoparticles has been reported (Reddy, Murali, Poornaprakash, Vijayalakshmi, & Reddy, 2012). TM-doped ZnO nanoparticles were synthesized by spin coating (Schwartz, Norberg, Nguyen, Parker, & Gamelin, 2003), sol–gel (El Ghoul, Barthou, Saadoun, & El Mir, 2010), and spray pyrolysis (Lee, Jeong, Cho, & Park, 2002). There are no reports on (Mn, Ni) co-doped ZnO nanoparticles. The substitution of Mn2+ ions in Ni-modified ZnO nanoparticles leads to the modification of structural, optical, electrical, and magnetic properties. In the present study, Zn0.7Mn0.3Ni0.3−xO (x = 0.05, 0.1, 0.15, 0.2) nanoparticles have been synthesized by sol–gel technique. The influence of different Mn concentrations on structural, optical, electrical, and magnetic studies has been investigated in the present work.

2. Experimental details

2.1. Synthesis
The various compositions of the system, Zn0.7Mn0.3Ni0.3−xO (x = 0.05, 0.1, 0.15, 0.2), were prepared in the form of polycrystalline nanoparticles powder by sol–gel technique. The zinc acetate (Zn(CH3COO)2.2H2O), nickel acetate (Ni(CH3COO)2.4H2O), and manganese acetate (Mn(CH3COO)2.4H2O) of analar grade were dissolved in distilled water in proper stoichiometric ratio separately and the resulting solutions were mixed and stirred to ensure homogenous mixing. The precipitates were filtered and washed with distilled water and subsequently with ethanol and...
acetone to remove impurities. The precipitates were dried at 200°C in an electric muffle furnace for one hour and subsequently grinded in mortar and pestle to make fine powder. The calcination of all samples was performed by heating them at 700°C in electric muffle furnace for 1 h each.

2.2. Characterization techniques
Structural analysis of the samples was performed by using Rigaku Table-Top X-ray diffractometer with CuKα radiation (λ = 1.54 Å) at 40 KV and 100 mA with scanning rate of 2°/min. The microstructure of samples was characterized by using scanning electron microscope (Carl Zeiss Supra 55). Diffuse reflectance of annealed nanoparticles has recorded using double beam UV–vis-NIR spectrophotometer (Shimadzu Model MPC-3100) in the range of 200–800 nm. Conductivity measurements were carried out using Novo control Technologies GmbH & Co. KG in frequency range of 100 Hz–10 MHz and temperature ranging from 323 to 463 K. The magnetic measurements were carried out at room temperature using vibration sample magnetometer (VSM – Microsense).

3. Results and discussion
3.1. Structural and morphological studies
Typical X-ray diffractometer (XRD) patterns of Zn$_{0.7}$Mn$_{x}$Ni$_{0.3-x}$O ($x = 0.05, 0.1, 0.15, 0.2$) samples calcined at 700°C is presented in Figure 1. The diffraction pattern shows the segregation of secondary phases namely cubic NiO (JCPDS No. 04-0835) and face-centered ZnMnO$_3$ structure (JCPDS No. 19-1461) besides the major hexagonal wurtzite structure corresponding to ZnO (standard data JCPDS No. 36-1451) (Toloman et al., 2013; Tong et al., 2010). The average crystallite size (grain size) has been calculated from the full width at half maxima (FWHM) of the diffraction peaks using the Debye–Scherrer equation (Elilarassi & Chandrasekaran, 2012)

$$D = k\lambda / (\beta \cos \theta)$$

where $D$ is the average crystalline size, $k$ is the shape factor, $\lambda$ is the wavelength of X-ray radiation, $\beta$ is the FWHM, and $\theta$ is the Bragg’s angle. The most intense peak (101) in the XRD pattern has been used to calculate the average crystalline size and it is listed in Table 1. The average crystallite size is observed to increase with an increase in the Mn content in the host matrix. The peak (101) shifts towards lower 2θ angle with an increase in $x$. The increase in Mn content causes lattice disorder and strain induced by incorporation of Mn$^{2+}$ ions in the host matrix. So there is slight variation in lattice parameters (Jayakumar et al., 2007; Omri et al., 2013).

The scanning electron microscope (SEM) micrograph of Zn$_{0.7}$Mn$_{x}$Ni$_{0.3-x}$O ($x = 0.05, 0.1, 0.15, 0.2$) samples is more or less similar. Figure 2 shows the typical SEM micrograph for Zn$_{0.7}$Mn$_{0.2}$Ni$_{0.1}$O ($x = 0.1$) sample. The micrograph reveals that there is heterogeneity at the surface and particles.
agglomeration. The particles are almost spherical and uniform in size. The average particle sizes estimated from SEM micrograph are found out to be in the range 40–50 nm and the values are listed in Table 1. The SEM micrographs, therefore, indicate the formation of nanoparticles.

### 3.2. Diffuse reflectance studies (DRS)

Diffuse reflectance spectra of nanoparticles Zn_{0.7}Mn_{x}Ni_{0.3–x}O (x = 0.05, 0.1, 0.15, 0.2) were recorded in the spectral range 200–800 nm. The acquired diffuse reflectance spectra has been converted to Kubelka–Munk function (F(R)), which is proportional to the absorption coefficient (α) (Torrent & Barron, 2002).

\[ \alpha = A F(R) \]
\[ F(R) = \frac{(1 - R)^2}{2R} \]
\[ \text{Diffuse reflectance } R = \frac{R_{\text{sample}}}{R_{\text{standard}}} \]

where \( R_{\text{sample}} \) and \( R_{\text{standard}} \) represent reflectance of sample and standard reference (BaSO₄).

The absorption coefficient (α) near band edge as a function of photon energy (hv) is expressed by Tauc relation (Suwanboon, Amornpitoksuk, Haidoux, & Tedenac, 2008):

\[ \alpha hv = B (hv - E_{\text{opt}})^p \]

#### Table 1. Compositional dependence of average crystalline size (D), SEM (D), optical band gap (Eopt), activation energy of grain boundaries (Egb), grains (Eg), coercive field (Hc), remanent magnetization (Mr), and saturation magnetization (Ms)

| Sample  | D (nm) | SEM D (nm) | Eopt (eV) | Egb (eV) | Eg (eV) | Hc (Oe) | Mr (emu) | Ms (emu) |
|---------|--------|------------|-----------|----------|---------|---------|---------|---------|
| ZnO     | 32.30  | 41         | 3.20      | 0.52     | 0.46    | 0.00    | 0.000010 | 0.002   |
| x = 0.0 | 29.61  | 40         | 3.14      | 0.64     | 0.41    | 69.64   | 0.000175 | 0.002   |
| x = 0.05| 32.32  | 43         | 2.74      | 0.40     | 0.27    | 39.36   | 0.000180 | 0.042   |
| x = 0.1 | 35.53  | 45         | 2.83      | 0.47     | 0.05    | 22.99   | 0.000080 | 0.036   |
| x = 0.15| 32.30  | 42         | 2.97      | 0.48     | 0.16    | 48.16   | 0.000139 | 0.011   |
| x = 0.2 | 39.47  | 47         | 3.04      | 0.77     | –       | 37.22   | 0.000233 | 0.022   |

Figure 2. Scanning electron micrograph (SEM) of Zn_{0.7}Mn_{x}Ni_{0.3–x}O (x = 0.1) sample.
where $E_{opt}$ is the optical band gap and $B$ is a constant. The parameter $\alpha$ in the Tauc’s equation, when substituted with $F(R)$, becomes:

$$(F(R)\hbar \nu) = B(\hbar \nu - E_{opt})^p$$

(6)

Exponent $p$ depends on the type of transition and it may have values $1/2$, $2$, $3/2$, and $3$ corresponding to the allowed direct, allowed indirect, forbidden direct, and forbidden indirect transitions, respectively (Punia et al., 2011). Tauc’s plot has been plotted for $p = 1/2$, $2$, $3/2$, and $3$, but plot with $p = 1/2$ gives best fit. The value of direct band gap is determined by extrapolating the straight line portion of $(F(R)\hbar \nu)^2$ vs. $\hbar \nu$ plot at $(F(R)\hbar \nu)^2 = 0$ axis (Kortüm, 1969) as shown in Figure 3 and the values are listed in Table 1. Optical band gap increases with the increase in the Mn concentration and decrease in NiO in the host matrix. Similar trend is observed by other researchers (Cheng & Chien, 2003; Fukumura et al., 1999). The observed blue shift behavior or increase in band gap cannot be assigned to quantum size effects (Cheng & Chien, 2003; Fukumura et al., 1999). This behavior may be due to band continuum and orbital hybridization between Mn atom and host band (Cheng & Chien, 2003; Fukumura et al., 1999).

### 3.3. AC conductivity

The AC conductivity $\sigma'$ derived from the complex impedance data using the relation

$$\sigma' = \omega \varepsilon_0 \varepsilon''$$

(7)

where $\omega$ is the angular frequency of the applied field, $\varepsilon_0$ is the permittivity of free space, and $\varepsilon''$ is the imaginary part of dielectric constant. The frequency-dependent conductivity spectra for all samples were studied in temperature range of $323 \leq T \leq 463$ K. The conductivity vs. frequency spectra for all samples at temperature 323 K are illustrated in Figure 4. The spectra may be divided into three regions. According to the jump relaxation model (Funke, 1986, 1992), in region I, (low frequency), the electric field cannot perturb the hopping conduction mechanism of charged particles. The conductance is approximately equal to the DC value ($\sigma_{dc}$) and the conduction mechanism is the same as that for DC conduction, i.e. hopping of charged particles from one localized site to another. The conductivity begins to increase nonlinearly after the frequency exceeds the critical frequency $f_c$, in dispersive region II. It may be due to the fact that with the increase in frequency, capacitor admittance becomes numerically larger than the resistor admittance. With further increase in frequency (region III), conductivity becomes proportional to frequency resulting in nearly constant loss (NCL) (León et al., 2001). In most materials, the NCL regime dominates AC conductivity at high frequency. The curves are thus flat in low frequency region as the conductivity values approximately correspond to $\sigma_{dc}$. With the increase in frequency, the conductivity spectra become more dispersive. The frequency conductivity spectra exhibit multiple step-like increments which is a characteristic of a potential profile with multiple activation energies (Barsoukov & Macdonald, 2005).
Generally AC conductivity behavior is analyzed using the Jonscher’s power law (Punia, Kundu, Murugavel, & Kishore, 2012),

\[ \sigma'(\omega) = \sigma_{dc} + \left( \frac{\omega}{\omega_H} \right)^n \]  

(8)

where \( \sigma_{dc} \) is the DC conductivity, \( \omega_H \) is the crossover frequency separating DC regime (plateau region) from the dispersive conduction, and \( n \) is the frequency exponent that lies between 0 and 1. Equation 8 describes the conduction mechanism, with the first term in RHS is the DC conductivity due to band conduction and the second term shows the transport properties of polarons, ions, and electrons (Punia, Kundu, Dult, Murugavel, & Kishore, 2012). The AC conductivity in the entire frequency region cannot be accounted by Equation 8, rather two such formulae have been used in different frequency regions to explain the presence of two relaxation mechanisms (Zhang, 2005). The conductivity at low frequencies has almost a constant value assigned to DC conductivity for grain boundaries (\( \sigma_{gb} \)) and the frequency independent plateau in region II is due to the DC conductivity for grains (\( \sigma_g \)). The values of \( \sigma_{dc} \), \( \omega_H \), and \( n \) are obtained by the fitting of the frequency-dependent conductivity (\( \sigma'(\omega) \)) data measured experimentally at different temperatures with Equation 8 for different frequency regions. Figure 5 shows the experimental data fitted with Jonscher’s universal power law (Equation 8) in different frequency regions for \( \text{Zn}_x\text{Mn}_{0.05}\text{Ni}_{0.25}\text{O} \) sample.

The value of \( n \) for different frequency regions increases with the increase in temperature. Figure 6 illustrates the variation of exponent \( n \) with temperature at different compositions for low-frequency
dispersion region. As $n$ increases with increasing temperature in all studied samples, then the conduction process can be explained with the small polaron quantum mechanical tunneling theory (Punia, Kundu, Dult et al., 2012). If small polarons are formed, then according to tunneling model (Punia, Kundu, Dult et al., 2012) frequency exponent ($n$) becomes

$$n = 1 - \frac{4}{{\ln \left( {\frac{1}{\omega_0}} \right) - \frac{\sigma_0}{\kappa_0 T}}}$$

Equation 9 suggests that $n$ increases with an increase in temperature. In the present samples, small polaron conduction mechanism is predominant. Equation 8 shows that conductivity increases with the increase in frequency which is in good agreement with our results. The increase in conductivity with the frequency can be explained on the basis that charge carriers are transferred between different localized states and trapped charges are liberated with the help of pumping force of the applied frequency (Ahmed, Ateia, & El-Dek, 2003). The electron-hopping model explains the electrical conduction mechanism in which electron hopping between the lattice sites takes place. Consequently, the hopping frequency of charge carriers increases with the increase in frequency resulting in an increase in conductivity. Electrical conductivity increases with the increase in temperature due to the increase in drift mobility of the thermally activated charge carriers (Uitert, 1995).

Figure 6. Plot of frequency exponent $n$ with temperature for Zn$_{0.7}$Mn$_x$Ni$_{0.3}$O ($x = 0.05, 0.1, 0.15, 0.2$) samples at low frequency dispersion region.

The inverse temperature variation of DC conductivity for grain boundaries and grains follows the Arrhenius relation:

$$\sigma_{dc} = (T) = \sigma_0 \exp[-(E_A/kT)]$$

where $\sigma_0$ is the pre-exponential parameter that depends on the nature of semiconductor, $E_A$ denotes the thermal activation energy of electrical conduction, and $k$ is Boltzmann’s constant. The activation energies for grain boundaries ($E_{gb}$) and grains ($E_g$) are calculated from the slope of linear fit of $\log\sigma_T$ vs. $1,000/T$ plot shown in Figure 7((a) and (b)) and the obtained values are presented in Table 1 for all studied samples. Perusal of the data presented in Table 1 indicates that activation energy of grain boundaries and grains increases with an increase in the Mn concentration in Zn$_{0.7}$Mn$_x$Ni$_{0.3}$O nanoparticles, suggesting thereby decrease in conductivity (Wang, Thomas, & O’Brien, 2006). These grain boundaries act as an insulating layer between grains thus creating the interfacial/space polarization. Perusal of the data shown in Figure 4 indicates that conductivity decreases with the increase in the Mn concentration. This may be attributed due to the fact that dopant (Mn) introduces the defect ions in Zn$_{0.7}$Mn$_x$Ni$_{0.3}$O system. During sintering and cooling processes, the diffusion process occurs due to which these defects tend to segregate at grain boundaries. The increase in dopant concentration enhances the concentration of defect ions which ease to the formation of grain boundary defect barrier leading to the blockage of flow of charge carriers. So, the conductivity decreases with the increase in Mn concentration and the decrease in Ni concentration in Zn$_{0.7}$Mn$_x$Ni$_{0.3}$O system.
3.4. Magnetic studies

Room temperature magnetization of Zn$_{0.7}$Mn$_x$Ni$_{0.3-x}$O ($x = 0.05, 0.1, 0.15, 0.2$) samples was measured as a function of applied magnetic field in the range of $-15,000$ to $+15,000$ G using VSM. The magnetization curves are shown in Figure 8 and the values of coercive field ($H_c$), remanent magnetization ($M_r$), and saturation magnetization ($M_s$) for all samples are listed in Table 1. The magnetization curves presented a small kink at around an origin indicating that the samples have small ferromagnetic component along with significant paramagnetic component. Similar results of co-existence of ferromagnetism and paramagnetism have been observed by other researchers (Ahn, Park, Shim, & Kim, 2004; Ohno, Munekata, Penney, Molnar, & Chang, 1992; Sharma et al., 2003). The paramagnetic behavior may be due to the spins of isolated Mn$^{2+}$ ions (Jayakumar et al., 2007). There are several factors that can induce ferromagnetism in Zn$_{0.7}$Mn$_x$Ni$_{0.3-x}$O ($x = 0.05, 0.1, 0.15, 0.2$) system such as oxygen vacancies, lattice defects, and presence of secondary phases (Sharma et al., 2003). In the present samples, ferromagnetic behavior (Figure 8, inset for $x = 0.2$) may be due oxygen vacancies which increases with the increase in the Mn ion concentration. Oxygen vacancies are responsible for ferromagnetism in Mn-doped ZnO samples at room temperature (Gao et al., 2013). The saturation magnetization of the samples tends to decrease as the concentration of Mn increases and the Ni concentration decreases, which suggests the partial and reduced incorporation of Mn$^{2+}$ ions in Zn$_{0.7}$Mn$_x$Ni$_{0.3-x}$O lattice and values are listed in Table 1. This decrease in magnetization may be due to the interaction among Mn magnetic moments and anti-ferromagnetic exchange increases with the increase in the Mn concentration (Wang et al., 2004).
4. Conclusions

Zn_{0.7-x}MnxNi_{x+0.3}O (x = 0.05, 0.1, 0.15, 0.2) nanoparticles have been synthesized by the sol-gel technique. The XRD patterns indicate that these samples have secondary phases NiO and ZnMnO\textsubscript{3} besides the major hexagonal wurtzite structure corresponding to ZnO. The average crystalline size was observed to increase with an increase in the Mn content. The average particle size estimated form SEM was found in the range 40–50 nm. Optical studies show that the optical band gap increases with the increase in the Mn concentration in the host matrix. AC conductivity increases with the increase in frequency and temperature. The hopping frequency of charge carriers increases with the increase in frequency and small polaron hopping conduction mechanism predominates in the studied samples. The value of AC conductivity decreases with the increase in the Mn concentration in ZnO lattice. It may be due to the enhancement of defect ions concentration. The magnetic measurements show that both paramagnetic and ferromagnetic behavior is present in studied samples. In the present samples, ferromagnetic behavior may be due to the formation of secondary phases such as ZnMnO\textsubscript{3}. The saturation magnetization of the samples tends to decrease as the concentration of Mn increases.

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