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Tailoring of electrical properties in nanostructured (NiO)_{0.25}(Fe_{2}O_{3})_{0.75} composite and compound for sensing applications

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Abstract

Compound and composite of the (NiO)_{0.25}(Fe_{2}O_{3})_{0.75} nanoparticles have been prepared by without water and surfactants (WOWS), Sol-gel method. Synthesized nanoparticles are calcined at 500 °C/2 h and pellets are sintered at 550 °C/1 h. The compound and composite showed cubic lattice and rhombohedral crystallographic lattices respectively. Morphological analysis is done by Scanning electron microscope and Transmission electron microscope. UV characterization was done to calculate band gap energies for both samples. DC electrical conductivity as a function temperature from 373.15 to 703.15 K was done by two-probe method. DC conductivity has been explained by hopping mechanism. Activation energies were calculated from Arrhenius plots. AC electrical properties namely; dielectric constant, dielectric loss tangent, ac conductivity and impedance were measured as a function of frequency (20 Hz–3 MHz). AC electrical properties were explained by Maxwell-Wegner model, Koop’s theory and Johnsher’s power law. Composite of (NiO)_{0.25}(Fe_{2}O_{3})_{0.75} showed enhanced properties as compared to compound.

1. Introduction

Oxides with composition AB_{2}O_{4} (A and B are metal cations) are categorized as ferrites. These oxides are classified on the basis of magnetic properties as hard and soft ferrites. Based on distribution of cations in the spinel structure they are also classified into normal spinel and inverse spinel structured ferrites. They possess distinctive magnetic, electronic and optical properties [1]. Nickel ferrites are soft magnetic materials with cubic inverse spinel structure. They have applications in various electronic, magnetic and high frequency devices from past few decades and their most attractive is in gas sensing [1, 2] in environmental protection, explosive gases, combustible gases and to control fermentation in the food industry. Gas detection depends on various properties of material like optical, electrical, calorimetric etc and spinel ferrites possess all above mentioned characteristics and are superior as compared to the other materials like carbonaceous allotropes, carbon polymer composites, conducting polymers etc used as gas sensors [1–3]. Among inverse spinel ferrites, the most suitable ferrite used for gas sensor is nickel ferrite and is used for the detection of various gases like chlorine, acetone, NOx, CO, NH_{3}, H_{2} and H_{2}O [4–10].

It is well known that the particle size and shape play a major role in tuning their electrochemical properties of materials and specially oxides (Ferrites). Hence, efforts have been made to develop materials with different morphologies [4]. In this endeavour following methods have reported: hydrothermal [11, 12], combustion [13, 14], coprecipitation [15–17], sol-gel [18, 19] micro-emulsion [20, 21], chemical [22], microwave [23, 24], nonconventional mechano-chemical route [25] etc.

The dopants are known to improve the properties of spinal ferrites by changing their micro-structure and morphology, activation energy, electronic structure or band gap of the metal oxides [26]. In some cases, dopants
create defects by generating oxygen vacancy or by forming solid solutions. These defects may enhance the gas sensing properties. Moradmand et al. [27] synthesized nickel ferrite nanoparticles doped with magnesium by co-precipitation method and reported that saturation magnetization decreased and coercivity (Hc) decreased as a function of Mg concentration. Chavan et al. [28] reported that at low concentration Mg ions occupied A site whereas at higher concentration Mg ions occupied B site. However, much less attention has been paid to understand the dielectric properties of Mg doped nickel ferrite. Sabikoglu et al. [29] have reported Nd substitution in nickel ferrite results a low saturation magnetization 36–51 (Am^2/kg) and the negligible coercivity 0.14–84 (mT) values indicating the soft magnetic behaviour. Kargar et al. [30] studied magnetic properties of Cu substituted nickel ferrites and noted that maximum value of saturation magnetization at x = 0.3 of the order of 17–21 emu g^-1 and coercivity values increased as a function of Cu concentration (Hc = 150–170 Oe). The dielectric analysis shows that NPs has a high dielectric constant at x = 0.2 concentration and then reduced with respect to annealing temperature and concentration. The magnetization curves demonstrate soft magnetic nature where, the saturation and remnant magnetization increase and then coercivity decreases due to increase of annealing temperature and concentration. The effect of Li doping on the nickel ferrite nanoparticles are also reported [31]. They noted that dielectric constant and coercivity values decreased and saturation magnetization increased as a function of annealing temperature and Li concentration. The Ca doped NiFe_2O_4 has been reported [32] to increase values of lattice constant and the crystallite size as function of doping. The value of saturation magnetization increased doping and up to then 02 Ca and then decreased.

From review of foregoing literature shows that researchers have investigated either of a synthesis technique or doping to achieve enhanced electric and magnetic of NiFe_2O_4. In this report we have used a novel approach. We have somewhat disturbed chemical composition of NiFe_2O_4 compound and composite is formed by mixing NiO and Fe_2O_3 nanoparticles. Both materials are synthesized by without water and without surfactant method sol-gel method [33] and compared their structural, morphological, optical, DC and AC electrical properties. Results of this study are reported here.

2. Materials and methods

WOWS Sol-gel method is used to synthesize the nanoparticle [33]. For preparation of (NiO)_{0.25}(Fe_2O_3)_{0.75} compound, Ferric Nitrate nonahydrate and Nickel Nitrate hexahydrate are dissolved as precursors in Ethylene Glycol (molar ratio of 1:14). The homogenous solution is heated at 70 °C ± 5 °C until gel is formed. This gel is further heated until it burns, and powder was obtained. The calcination temperature of the nanoparticles was 500 °C ± 5 °C/2 hours and pellets were sintered at 550°C ± 5 °C/1 h. For the synthesis of (NiO)_{0.25}(Fe_2O_3)_{0.75} composite, Iron oxide (Fe_2O_3) and Nickel oxide (NiO) are individually synthesized. The synthesized powders of Fe_2O_3 and NiO are calcined separately at 500 °C ± 5 °C/2 h. The calcined powders of both oxides were mixed in 1:3 (NiO:Fe_2O_3) ratio by weight to form the composite. The composite pellets are sintered at 550°C ± 5 °C/1 h.

The monochromatic CuKα radiations from PAN Analytical, X’ Pert Pro equipment is used to record x-ray Diffraction Spectrum in the 2θ range 20°–80°. Scherrer equation $D = \frac{K\lambda}{\beta \cos \theta}$ was used to calculate the crystallite sizes [34]. Experimental density was determined by using formula: $\rho_{exp} = \frac{\rho_0}{V}$, where $\rho$- mass and V- volume of the pellet. Hitachi Transmission electron microscope and HITACHI SU-1500, Field Emission Scanning Electron Microscope is used to determine particle size, shape and morphology. UV absorption spectroscopy was done by using UV–vis spectrophotometer (SPECORD 200 Plus). The bandgap energies were calculate using the Tauc relation: $(a\nu^2)ν = A(\nu-E_g)$. Electrical properties are measured by Wayne Kerr Precision Component Analyzer; Model 6440B. Taking C—capacitance, $\infty$- permittivity of free space, d— thickness and A— area of cross-section of the sintered pellets; then the relation: $\varepsilon = Cd/\infty A$, may be used to calculate he dielectric constant. Similarly, denoting dielectric constant by $\varepsilon$; dielectric loss by tan $\delta$ and frequency by $f$ in hertz, the relation: $\sigma_{ac} = \varepsilon \tan \delta 2\pi f$ [35] is used to determine ac conductivity.

3. Results and discussion

3.1. Structural analysis

3.1.1. X-ray diffraction

X-ray Diffraction Spectrum is used to study the structural properties of both samples. Figure 1 shows the XRD spectrums of compound and composite samples. For compound, as prepared and heat-treated samples exhibited cubic crystal structure. Maximum intensity peak is detected at 2θ value of 35° and indexed as (311) in accordance with the JCPD card No. 01-086-2267. The values of full width and half maximum of highest
intensity peak (311) is used to calculate crystallite size, D by Scherrer equation \[4\]. The calculated crystallite size and lattice constants for compound are 65 nm and 8.27 + 3 Å, respectively.

The XRD spectrum for sintered composite sample is shown in figure 1 (b) which showed two rhombohedral lattices for NiO and Fe₂O₃ composite pallet. The maximum intensity peaks were observed at 2\(\theta\) = 44° and 2\(\theta\) = 33° for NiO and Fe₂O₃ respectively. JCPD cards No. 00-044-1159 and 00-001-1053 were used to index the peaks for NiO and Fe₂O₃ respectively. The crystallite size determined for maximum intensity peaks were 87 nm and 67 nm for NiO and Fe₂O₃ Sintered pallets, respectively. The lattice constants for calcined NiO and Fe₂O₃ nanoparticles are \(a = b = 2.952 + 6\) Å, \(c = 7.247 + 2\) Å and \(a = b = 5.03 + 1\) Å, \(c = 3.713 + 3\) Å, respectively. In XRD pattern of composite no impurity peak was observed which shows that composite consist of phase pure components. The values of experimental density (\(\rho_{\text{exp}}\)) of sintered compound and composite samples are 2.71 g cm\(^{-3}\), and 2.35 g cm\(^{-3}\) correspondingly. The density of sample is higher for composite as compared to compound sintered pallet.

### 3.2. Morphological analysis

#### 3.2.1. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM)

SEM is used to study surface morphology of samples and shown in figures 2(a) and (b). The magnification of micrographs is 20,000 with the resolution value of 10 \(\mu\)m. Both micrographs show the agglomeration of nanoparticles due to magnetic nature of the materials \[36\]. To investigate morphology, further, Transmission Electron Microscopy (TEM) of the synthesized product was performed. Figures 3(a) and (b) present TEM images of compound and composite samples. Both samples show that nanoparticle are agglomerated \[36\]. The
size of nanoparticles was estimated using particle distribution histogram and was found to be 44 nm for compound and 74 nm for composite.

3.3. Optical studies

3.3.1. UV absorption spectroscopy

UV-Visible absorption spectroscopy for both samples was done over a spectral range of 200–800 nm of the electromagnetic spectrum. Plots of UV absorption are shown in figures 4(a) and (b) for compound and composite samples, respectively. In compound, the absorption spectrum displays an absorption band ranging from ∼300 nm to ∼450 nm while in composite a wide absorption band is observed between ∼300 nm to ∼700 nm. These absorption bands are formed due to d-d transition of Fe and Ni ions from valence band to the conduction band [37]. These plots show that composite materials absorb light more strongly as compared to compound. The bandgap energies for compound and composite samples are calculated using, Tauc relation: \((\alpha h \nu)^n = \beta (h \nu - E_g)\) [36]. Where, \(h \nu\) is incident photon energy, \(\beta\) is an energy independent constant, \(\alpha\) is absorption coefficient and \(E_g\) is the optical band gap energy of sample. Inset of figures 4(a) and (b) shows Tauc plots of compound and composite respectively. The calculated bandgap value from Tauc relation are \(E_g = 2.73\) eV and \(E_g = 2.31\) eV for compound and composite respectively [38]. The decrease in band gap energy...
and broad absorption of composite sample makes it more suitable to be used in optoelectronics devices as compared to compound sample [39].

3.4. Electrical analysis

3.4.1. DC measurements

DC conductivity of sintered compound and composite samples are shown in figure 5(a). Both samples show negative temperature coefficients of resistance i.e. dc conductivity increases with increase in temperature. The conduction in these materials is explained by hopping mechanism. The conductivity of compound sample remains almost constant up to 513.15 K. In this case, below 513.15 K, electrons do not have enough energy to cross the potential barrier between two ions. Thereafter, it increases sharply due to hopping of electrons from one site to the another. Hence the rate of successful hopping increases with increase in the temperature [40]. The electrons gained enough thermal energy to overcome the potential barrier between two sites. This indicates that sample behave like semiconductor above 513.15 K. In ferrites, mainly the hopping occurs between Fe$_{2}^{2+}$ and Fe$_{3}^{3+}$ ions. In this case, both n- and p-types conduction is occurring in the sample. The n-type conduction takes place due to Fe ions whereas Ni ions are responsible for p-type conduction.

The composite sample showed constant conductivity up to 473.15 K, above that it shows sharp increase in dc conductivity. Up to 473.15 K the intra-grain defects are dominant due to which electron hopping reduced. Afterwards the dc conductivity increases sharply. The increase in conductivity is directly related to the rate of hopping. The rate of successful hopping increases due to the thermal energy provided to the sample. The electron hopping occurs between Fe$_{2}^{2+}$ and Fe$_{3}^{3+}$ ions while the hopping of hole occurs between Ni$_{2}^{2+}$ and Ni$_{3}^{3+}$ ions [40–42]. The high magnitude of dc conductivity of composite is due to excessive charge accumulation on the multiple interfaces present in the sample. This excessive charge accumulation increases the rate of successful hopping. The increase in the density (of composite) is also one of the factors which supports higher magnitude of dc conductivity in composite as compared to compound. Further, to calculate Activation energies ($E_a$) of the both samples, Arrhenius plots were drawn with the help of dc conductivity data as shown in figure 5(b) [43]. The values of activation energy for compound and composite are $E_a = 0.418(5)$ eV and $E_a = 0.368(3)$ eV, respectively. The composite sample showed enhanced dc conductivity as compared to compound sample.

3.4.2. AC measurements

The dielectric loss ($\tan \delta$), ac conductivity ($\sigma_{ac}$) and dielectric constant ($\varepsilon$) are measured in the frequency range of 20 Hz to 3 MHz. Plot of dielectric loss is shown in figure 6(a). Composite samples showed low losses as compared to the compound samples. The high losses in low frequency region are the result of different polarization mechanisms taking place in sample due to applied field. The charge particles (dipoles) follow the applied frequency and consume energy due to which dielectric loss increases. But when the applied frequency increases, they are unable to follow the field and stop consuming energy consequently the dielectric loss decreases. The relaxation peak observed in the low frequency region indicates the presence of interfacial polarization. The relaxation peak in composite is shifted towards high frequency side and its magnitude is also reduced which indicates the strong polarization mechanism present in composite sample [44, 45].
The dielectric constant of both samples is shown in figure 6 (b). Dielectric constant decreases as frequency is increased. This is explained by Koop’s model [45]. This model postulates that such materials are composed of grains which behave as conductor and grain boundaries which behave as low conductor or insulator and material behaves as inhomogeneous medium. The dielectric behaviour of this type of materials is explained based on Maxwell—Wagner model [46]. In the low frequency region, the polarization effect is dominant which is indicated by high magnitude of dielectric constant. As electric field is imposed on the sample, electronic exchange between Fe$^{2+}$ and Fe$^{3+}$ ions follow applied field which originate the space charge polarization. Afterwards at higher frequencies the charge particles are unable to follow the applied field and the polarization dies out. As a result, the magnitude of dielectric constant decreased. The high magnitude of dielectric constant of composite as compared to compound is due to multiple interfaces present in it which supports the polarization mechanism. Stronger the polarizations higher is the dielectric constant. Also, dielectric constant is directly proportional to the electron hopping (dc conductivity) that’s why the dielectric constant of composite is higher than that of compound.

Figure 6 (c) shows the ac conductivity of both samples. The ac conductivity increases with the increase in frequency and is explained by Jump relaxation model [47] and Jonscher’s power law [48]. The ac conductivity is due to hopping of charged particles from one site to another. As discussed earlier, in these samples, the hopping occurs between Fe$^{2+}$ and Fe$^{3+}$ ions and Ni$^{2+}$ and Ni$^{3+}$ ions [44]. Two regions are observed in ac conductivity plots. These regions are classified as frequency independent and frequency dependent regions. At frequencies below 10 kHz (which may be called frequency independent region), the ac conductivity is almost constant because charged particles do not have enough energy to hop [49]. Thereafter, the ac conductivity increased due to increase in successful hopping of charged particles. Dominant interfacial effect in composite is responsible for
the high values for the ac conductivity in composite as compared to compound samples. Over all composite material showed improved electrical properties as compared to compound material.

3.4.3. Impedance spectroscopy

The impedance ($Z$) in the frequency range of 20 Hz to 3 MHz for the compound and composite samples, is measured and shown in figure 7(a). Impedance shows decreasing trend with increase in applied frequency for both samples. The high magnitude of $Z$ at low frequencies is due to interfacial polarization. This phenomenon follows the Maxwell Wagner model as explained in previous section in detail [46]. The composite shows low magnitude of impedance as compared to compound. This may be due to high values of dc conductivity of composite samples. Impedance of compound sharply decreases after 1 kHz which indicates that interfacial polarization is low at that frequency. But in case of composite the $Z$ decreases gradually, which indicates strong polarization mechanism in composite due to (strong) interfacial effect. For further analysis real, $Z'$ and imaginary, $Z''$ parts of the impedance are calculated. With the help of $Z'$ and $Z''$, Cole-Cole plots and respective equivalent circuits for both samples as shown in figure 7(b). Impedance decreased with increase in frequency showing opposite trend to the ac conductivity. Semi-circular arcs are seen in graphs which represent the relaxation phenomenon due to bulk and grain boundary resistance. One large semicircle appeared in case of compound which indicates the domination of bulk resistance in the sample. This arc is represented by a parallel RC circuit. RC circuit shows parallel combination of grains-resistance and grains-capacitance [50, 50]. While in composite the depressed semi-circular arc appears, which showed that impedance of composite is less than that of the compound. In composite sample along with major arc another small arc appears which represent the grain boundary resistance. Normally this small arc appears at higher temperature but in composite it has appeared at room temperature [52]. The composite plot is represented by equivalent circuit consist of two parallel RC circuits in series. This RC circuit shows parallel combination of grain-resistance and grains-capacitance placed in series with similar circuit of grain-boundary resistance and grain-capacitance. These results confirmed that composite have better conductivity than the compound sample.

4. Conclusions

Phase pure ($\text{NiO}_{0.25}(\text{Fe}_2\text{O}_3)_{0.75}$) compound and composite are synthesized by simplified sol-gel method. Compound nanoparticles showed cubic crystal lattice whereas composite sample showed two rhombohedral lattices i.e. for NiO and Fe$_2$O$_3$ nanoparticles. Materials exhibited agglomerated spherical particles in nanometre range. The average grain size for compound and composite nanoparticles was 44 and 74 nm, respectively. The calculated bandgap value for compound is 2.73 eV and composite sample is 2.31 eV. The decrease in band gap energy and broad absorption of composite sample makes it more suitable to be used in optoelectronics devices as compared to compound sample. DC conductivity showed negative temperature coefficients of resistance for both samples. Composite sample showed semiconductor like behaviour above 500 °C. The activation energy for compound and composite samples are 0.418(5) and 0.368(3) eV, respectively. The composite sample showed enhanced dc conductivity as compared to compound sample. The dielectric loss tangent, dielectric constant and impedance decreased as a function of frequency and ac conductivity increased with increase in frequency. At
20 Hz the dielectric loss tangent of composite is four times less than that of compound. The maximum dielectric constant was observed for composite sample at 20 Hz. Maximum ac conductivity was found to be 0.0005 S m⁻¹ at 3 MHz for composite. Cole-Cole plots revealed the dominant grain boundary resistance in composite at low frequency region. Composite showed improved dielectric constant, dc conductivity, ac conductivity and low losses as compared to that of the compound. Thus, it is concluded that composite samples may be used in devices like sensors.

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