Effect of Mg doping on the improvement of photoluminescence and magnetic properties of NiO nanoparticles

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Abstract
NiO nanoparticles doped with different concentration of Mg were synthesized by a low cost wet chemical method. XRD characterization indicated that the fcc structure of NiO is not affected with Mg doping. TEM analysis further reveals the Mg doping induced reduction in grain agglomeration along with the spherical shape transformation in NiO. Significant enhancement of UV emission in Mg doped NiO nanoparticles is evident from photoluminescence characterization. Magnetic characterization of the samples showed that all the samples show weak ferromagnetism along with the dominance of background antiferromagnetism at room temperature. The perseverance of background antiferromagnetic order is further probed through Raman characterization. All the samples show first order antiferromagnetic to weak ferromagnetic transition along with the presence of shape memory effect as evident by the negative slope in Arrott plot. The Ni0.9Mg0.1O sample with much enhanced UV emission and magnetization could find the usefulness in UV optoelectronic device, spintronics device etc.

1. Introduction

Now-a-day, transition metal doped oxide based diluted magnetic semiconductors (DMS) have appealed the attention of many researchers due to their potential applications in the field of spintronics, magneto-electronics and magneto-optoelectronics [1–4]. Most of the oxide DMS systems exhibit n-type electrical conductivity and possessing non-cubic crystal symmetry [5, 6]. It is worth mentioning here that one need to find DMS in cubic system with p-type conductivity to facilitate the integration of the same in advanced silicon based microelectronics [5, 7]. Among the transition metal oxides, nickel oxide (NiO) exhibits cubic crystal structure along with the p-type conductivity with a direct wide band gap in the range from 3.6 to 4.0 eV [8]. NiO shows type-II antiferromagnetic (AFM) order with highest Neel temperature 524 K [9–11]. When NiO is reduced to nano scale, it exhibits anomalous magnetic properties due to the size effect and enhanced nickel vacancy [5, 12]. For example: Bi et al [13] demonstrated the occurrence of ferromagnetic like order at low temperature for 5 nm sized spherical NiO particles due to missing bonds and lattice distortion while the same showed superparamagnetic order at 300 K. In contrast to this, Tadic et al [14] reported the room temperature ferromagnetic behavior for spherical NiO nanoparticle with same size due to their core–shell structure. It has been reflected in a review that the NiO nanoparticles evolve with the occurrence of different magnetic order like ferromagnetism, superparamagnetism, spin glass behaviour and core–shell like structure [15]. In contrast to the above cases, the spherical NiO nanoparticle (16 to 25 nm) showed AFM behaviour at 300 K [16]. Not only the size reduction but also doping has the significant impact on the magnetic properties of NiO.

Magnetic properties of NiO are shown to enhance by doping with different transition metal as well nonmagnetic ions. The substitution of nonmagnetic elements like Zn, Mg etc or other transition metals like Fe...
in place of Ni$^{2+}$ ion of NiO would disruption the AFM order of the host NiO and hence one should anticipate the appearance of different magnetic order. For example, NiO nanoparticle is shown to exhibit ferromagnetic order when doped with Fe [17-20]. On the other hand, the Mn doped NiO nanoparticle demonstrated either superparamagnetic [7] or ferromagnetic [17, 21, 22] order basically depending upon the synthesis method. It has been reported that the paramagnetic NiO evolves with superparamagnetic or ferromagnetic order for low or high Zn doping concentrations respectively [23]. In contrast to this observation, several studies indicated the emergence of ferromagnetic ordering at room temperature in Zn doped NiO [17, 24, 25]. NiO is also shown to exhibit room temperature ferromagnetic order when doped with Mg [26] and La [27] ions. It is therefore crucial to choose the synthesis method or dopant as these parameters finally influence the properties of the synthesized material.

In the present work, we synthesized Ni$_{1-x}$Mg$_x$O ($x = 0.01, 0.03, 0.05, 0.07, 0.1$) nanoparticles with an aim to study the role of Mg substitution on the photoluminescence and magnetic characteristics of NiO nanoparticles. Our study indicates the Ni$_{0.97}$Mg$_{0.03}$O nanoparticles exhibited enhanced UV emission characteristics along with improved magnetic order. Hence, this material could be find potential applications in UV optoelectronic device, spintronic devices etc.

2. Experimental

Ni$_{1-x}$Mg$_x$O ($x = 0.01, 0.03, 0.05, 0.07, 0.1$) samples were prepared by chemical route using nickel nitrate [Ni(NO$_3$)$_2$.6H$_2$O] and magnesium nitrate [Mg(NO$_3$)$_2$.6H$_2$O] as initial precursor following the procedure described elsewhere [25]. The prepared mixture solutions with different concentrations of Mg were dried with the help of hot plate. Finally, the samples are calcined at 300 °C for 1h and subsequently at 500 °C for 1h to get the Mg doped NiO nanoparticles in powder form.

The structural and microstructural modification of Mg doped NiO was probed through the Bruker X-ray diffractometer (D8 Advance) operated with Cu-K$_α$ radiation ($λ = 1.54$ Å). The microstructural along with morphological feature of the samples was studied using the transmission electron microscope (TEM) (JEOL JEM-2010 (UHR)) operated at 200 kV. Raman spectroscopic characterization of the samples was done using a Jobin Yvon Horibra Raman spectrometer (LABRAM-HR). The emission features of the samples along with the presence of defect or vacancy in the samples were probed through photoluminescence (PL) spectra using Cary Eclipse instrument (No: EL08083851). The evolution of magnetic characteristics of Mg doped NiO nanoparticles were probed through a quantum design PPMS based vibrating sample magnetometer (Model No: 6700).

3. Results and discussion

Figure 1 showed the x-ray diffraction (XRD) pattern of Ni$_{1-x}$Mg$_x$O ($x = 0.01, 0.03, 0.05, 0.07, 0.1$) nanoparticles. All the samples revealed the fcc structure of NiO as per the standard JCPDS card (number: 78-0643) without forming any impurity phase. The XRD data of all the samples are refined with Rietveld refinement using FullProf [28] to obtain lattice parameter and Ni–O bond length. Both the parameters slowly increase with increasing of Mg doping concentration in NiO (figure 2). Here, the data of pure NiO sample [25] is considered to compare with the data of Mg doped NiO nanoparticles. The increase of lattice parameter and Ni-O bond length of NiO with Mg doping (figure 2) could be due to the substitution of Mg$^{2+}$ ions (0.72 Å) with slightly larger Shannon ionic radii than that of Ni$^{2+}$ (0.69 Å) ions [29, 30]. Similar type of observation has also been reported earlier [26, 31]. We also calculated the crystallite size ($D = \frac{Kλ}{β \cos θ}$) of Mg doped NiO nanoparticles from XRD line width using Debye–Scherrer relation [32] (table 1). In order to get better information on size along with the morphology of the nanoparticles, the TEM characterization on representative Ni$_{1-x}$Mg$_x$O nanoparticles with $x = 0.03$ and 0.05 under taken (figure 3). Like the case of Zn doping [25], the agglomeration is also reduced along with the spherical shape transformation in Mg doped NiO (figures 3(A) and (B)). The calculated average particle sizes for Ni$_{0.97}$Mg$_{0.03}$O and Ni$_{0.95}$Mg$_{0.05}$O nanoparticles are 27.38 ± 1.03 nm and 23.08 ± 0.73 nm respectively. The particle size calculated from TEM image is in accordance with the size calculated from XRD data (table 1).

Figure 4 showed the Raman spectra of Ni$_{1-x}$Mg$_x$O nanoparticles measured at room temperature. All the peaks appeared in the spectra are matched well with reported literature on NiO sample [33–36]. The absence of TO + LO mode for all the samples could be an indication of nanocrystalline nature of the samples [25, 37]. Since NiO is shown to evolve with different magnetic ordering at the expense of AFM ordering, one should expect the signature of the same in Raman spectra. However, the appearance of 2M mode (at ∼1547 cm$^{-1}$) in the spectra confirms the persistence background AFM super exchange interaction via Ni$^{2+}$–O$^{2-}$–Ni$^{2+}$ in all the samples [38]. The existence of disorder in the samples viz. interstitial oxygen or nickel vacancies, surface effects
and imperfection is also confirmed due to the existence of first order peaks (TO and LO) in the Raman spectra [2,25]. In fact, the intensity of 1TO and 2TO mode significantly diminishes at highest Mg doping (10%) concentration. Also, the intensity of 2LO and 2M mode progressively decreased beyond 1% Mg concentration. This result suggested that not only the vibrational properties but also the AFM super exchange interaction in NiO nanoparticles influenced strongly up on Mg doping. The modification in vibrational properties will in turn

![Figure 1. Rietveld refinement of XRD pattern of Ni$_{1-x}$Mg$_x$O nanoparticles with different Mg dopant concentrations ($x = 0.01, 0.03, 0.05, 0.07, 0.1$) using Fullprof software.](image1)

![Figure 2. Variation of bond length (Ni-O) and lattice parameter of Ni$_{1-x}$Mg$_x$O ($x = 0$ to 0.1) nanoparticles with Mg doping concentration obtained from Rietveld refinement of XRD data. The data of pure NiO sample from reference [25] is given for comparison.](image2)
affect the lattice parameter and different bond length up on Mg doping in NiO [27]. The Raman study in fact supports the results obtained in XRD. Figure 5 showed the photoluminescence (PL) spectra of Mg doped NiO nanoparticles measured at room temperature. All the samples show bands in UV (at ~362, 376, 385 and 390 nm) as well as visible (at ~410, 438, 457 and 490 nm) region. The band at UV region is the characteristics feature of near band edge emission [39]. Among the different UV bands, the band present at 362 nm is recognized to the recombination of excitons [40] and its intensity is improved with increasing Mg doping concentration. This could suggest the substitution of Mg in NiO. The improvement of UV emission intensity in NiO with Mg doping could be due to the reduction in defect density and hence improvement in quality of the nanostructure [41–43]. The reduction in grain agglomeration along with spherical shape transformation as evident from TEM characterization further supports the Mg doping induced improvement in crystal quality which in turn regulated the UV emission.

Table 1. Size obtained from XRD and TEM for Mg doped NiO nanoparticles.

| Sample Name       | Size (nm) from XRD | Size (nm) from TEM |
|-------------------|--------------------|--------------------|
| NiO               | 28.92 [25]         | 28.02 ± 0.15 [25]  |
| Ni0.99Mg0.01O     | 26.90              |                    |
| Ni0.97Mg0.03O     | 25.56              | 27.38 ± 1.03       |
| Ni0.95Mg0.05O     | 25.29              | 23.08 ± 0.73       |
| Ni0.93Mg0.07O     | 26.78              |                    |
| Ni0.9Mg0.1O       | 25.80              |                    |

Figure 3. TEM image of (A) Ni0.97Mg0.03O and (B) Ni0.95Mg0.05O nanoparticles along with their size distribution (inset of respective micrograph).

Figure 4. Raman spectra of Ni1−xMgxO (x = 0.01, 0.03, 0.05, 0.07 and 0.1) nanoparticles.
intensity. The violet emission bands at 410 and 438 nm in the samples suggested the presence of electrons trapped at Ni interstitial to the valence band \[8\]. The blue emission band featured with low intensity at \(\sim 457\) and \(\sim 490\) nm could have ascribed to the recombination of electrons from Ni\(^{2+}\) vacancy to the holes in the valence band \[8, 27\].

Like our recently studied result on Zn doped NiO \[25\], Mg doping has the significant impact on the PL spectral intensity in UV region particularly at \(\sim 362\) nm. To quantify this, we calculated ratio of peak intensity at UV region (362 nm) to the same at visible region (490 nm) i.e. \(I_{UV}/I_{Vis}\). Figure 6 illustrates the CIE 1931 X-Y chromaticity diagram for Ni\(_{1-x}\)Mg\(_x\)O samples obtained using the software from [http://geocities.com/krjustin/gocie.html]. We use the PL data of NiO from reference \[25\] to obtain the CIE coordinate of NiO nanoparticles as (0.30, 0.47). This result indicated the color emitted by NiO is near green region. The CIE coordinate shifted to (0.15, 0.07) for 7% Mg doped NiO while the same remain at (0.15, 0.06) for all other Mg doped NiO. This is an indication of the downward shifting of emission to the blue region with increasing of Mg doping concentration in NiO.

Figure 5. Variation of Room temperature PL spectra of Mg doped NiO nanoparticles.

Figure 6. CIE 1931 X-Y chromaticity diagram for Mg doped NiO samples. CIE co-ordinate of NiO extracted from the data of reference \[25\] is given for comparison.
Figure 7 shows the temperature dependent magnetization (ZFC and FC) curve recorded at $H = 100$ Oe for (A) Ni$_{0.99}$Mg$_{0.01}$O, (B) Ni$_{0.97}$Mg$_{0.03}$O, (C) Ni$_{0.95}$Mg$_{0.05}$O, (D) Ni$_{0.93}$Mg$_{0.07}$O and (E) Ni$_{0.9}$Mg$_{0.1}$O nanoparticles.

Figure 7 shows the temperature dependent magnetization for Ni$_{1-x}$Mg$_x$O nanoparticles recorded under zero field cooled (ZFC) and field cooled (FC) condition at an applied field of 100 Oe. The continuous decrease in FC magnetization of Ni$_{1-x}$Mg$_x$O nanoparticles with increasing temperature indicating the weak or non-interacting nature of the particles [44]. As we move away from high temperature regime, the ZFC magnetization decreased slowly up to certain temperature samples beyond which the same increased sharply with further lowering the temperature due to the presence of weakly coupled or uncoupled metal ions in the system [45]. These characteristics features could be an indication of the existence of two magnetic phases i.e. (i) one is the low temperature dominated ferromagnetic phase and (ii) other is the high temperature dominated antiferromagnetic phase [25, 46, 47]. The ZFC and FC magnetization as discussed above is observed for all the samples except for 7% Mg doped NiO sample (figure 7(D)) in which both ZFC and FC magnetization showed continuous decrease with increasing temperature. Similar type of behaviour has also observed for 3% Zn doped NiO nanoparticles by Peck et al [48] and the authors suggested that the appearance of paramagnetic type behavior could be associated with the uncompensated spin of the core.
In order to understand the magnetic behavior further, we studied the variation of magnetization ($M$) with magnetic field ($H$) measured at 300 K (figure 8). All the samples showed perfect hysteresis loop along with the absence of saturation in $M$ even at high $H$ regime signifies the weak ferromagnetic phase embedded in the background AFM of NiO. Similar observation has also been reported for Zn doped NiO nanoparticles [25]. In fact, the presence of background AFM order is in agreement with our Raman study as well as the magnetization study under ZFC condition. To quantify the magnetization, we calculated remanence magnetization ($M_r$) for Ni$_{1-x}$Mg$_x$O nanoparticles from the M-H curve (figure 8). The variation of $M_r$ for Ni$_{1-x}$Mg$_x$O nanoparticles with Mg concentration is shown in figure 9. It is clear from our recent study that the NiO synthesized with similar procedure as used in the present study exhibits core–shell magnetic structure with antiferromagnetic core and ferromagnetic shell [25]. The dominating antiferromagnetic super-exchange interaction in NiO of core is determined by Ni–O–Ni linear chains with opposite spins along [111] direction [7]. Since $M_r$ value increases up to 3% Mg doping in NiO, we believe that more amount of Mg is substituted in core of NiO. As a result, the antiferromagnetic super–exchange interaction of the core breaks down which in turn enhances the ferromagnetic properties in NiO nanoparticles. The sequential decrease of the value of $M_r$ for 5 and 7% Mg doped NiO than that of pure NiO case could be due to the substitution of more amount of Mg on the surface of NiO.
NiO causing the suppression of magnetization. Similar type of result has also been reported for Zn doped NiO at certain Zn doping concentration [25]. The observation of highest value of $M_r$ for 10% Mg doped NiO samples could possibly be due substitution of more Mg in the core of NiO which resulted into the enhanced magnetization which is in accordance with the result of Zn doped NiO nanoparticles [25]. The appearance of low $M_r$ values of the Ni$_{1-x}$Mg$_x$O nanoparticles like the case of Zn doped NiO indicated the samples could find its usefulness in data storage applications [49]. While comparing the variation of $M_r$ and the IUV/IV is obtained for PL spectra (figure 9), we have seen that both the parameters vary identically with Mg doping concentration in NiO.

Since the Arrott ($H/M$ versus $M^2$) plot through some light on the type of magnetic transition along with application prospective of the sample [50], we therefore plot $H/M$ versus $M^2$ for Ni$_{1-x}$Mg$_x$O nanoparticles (figure 10). In our case, all the samples show first order antiferromagnetic to weak ferromagnetic transition [51] along with the presence of shape memory effect [52] due to appearance of negative slope at high field region. Hence, the samples could be useful for various applications like spintronic devices [5], magneto-electric random access memory [52] etc. PL characterization as well as Magnetization characterizations of the samples thus indicated that the 10% Mg doped NiO samples with enhanced luminescence and magnetization could be useful for UV optoelectronics as well as spintronic devices.

4. Conclusion

We report the improvement of photoluminescence and magnetic characteristics of Mg doped NiO nanoparticles. XRD and TEM studies indicated that the size of the NiO nanoparticles are not affected much up on Mg doping. Photoluminescence characterization indicated the Mg doping induced enhancement of UV emission in NiO nanoparticles. Magnetic characterization indicated the all the samples exhibit weak room temperature ferromagnetism along with the dominance of background antiferromagnetic order at high field regime. Our results combining the magnetization and optical studies indicated the 10% Mg doped NiO samples with enhanced luminescence and magnetization could be useful for UV optoelectronics as well as spintronic devices.

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