d⁰ ferromagnetism in Li-doped ZnO compounds

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ABSTRACT

Recently, d⁰ ferromagnetic oxide materials have been demonstrated as one of the promising materials for spintronics devices. Herein, we have studied the crystal structure, micro-structure, and magnetic properties of Li-doped ZnO, i.e., Zn₁₋ₓLiₓO (x = 0, 0.02, 0.04, and 0.06) compounds, prepared by the solid-state reaction route method. These compounds were found to crystallize in single phase of the hexagonal wurtzite structure with average crystallite size of 35–50 nm, as confirmed from the XRD patterns. The incorporation of Li in ZnO leads to decrease in the lattice parameters and cell volume. The SEM images reveal uniform sample morphology with average grains size of the order of 50–70 nm. The measurements of magnetic properties show that the un-doped ZnO exhibits diamagnetic behavior, whereas Li-doping in ZnO induces room-temperature ferromagnetism along with ultra-soft coercivity (~ 50 Oe), and with a maximum saturation magnetization of 0.10 emu/gm for x = 0.02 sample. The magnetic irreversibility behavior between zero-field-cooled and field-cooled magnetization vs. temperature data indicates the co-existence of spin-glass phase in all Li-doped compounds.

1 Introduction

Over the last decade, a great deal of research was carried out to develop suitable ferromagnetic oxides with Curie temperature at or beyond room temperature for their possible application in spintronic devices. It is important to mention that the ferromagnetic ordering in transition metal (TM)-doped oxide materials was reported extensively and it was well documented [1–8]. But there is a debate about whether the observed magnetism is an extrinsic effect arising from impurity phases, or it is an intrinsic property [9]. Recently, doping with non-magnetic element (e.g., Li, Na, K, Mg, Ag, etc.) in the non-magnetic host oxides, such as ZnO, TiO₂, SnO₂, ZrO₂, and so on, has gained a lot of attention because of their possible application in spintronics. In particular, it was emphasized that ferromagnetism can be observed in such non-magnetic element-doped oxide materials and it was referred to as d⁰ or intrinsic ferromagnetism. There exist a variety of calculations that predict ferromagnetism in such d⁰ ferromagnetic oxide semiconductors, but the origin of observed ferromagnetism is still not completely understood, despite many efforts in the past two decades were made. In most of the cases, it was credited to
originate from oxygen vacancies/defects. In a model, proposed by Bouzerar et al., it was suggested that the difficulties of controlling the defects can be overcome by doping non-magnetic element in oxides, which could lead to ferromagnetism (FM) with high Curie temperature [10]. Peng et al. [11] suggested out that the optimum hole concentration is essential to induce room-temperature ferromagnetism (RTFM) in such d0 ferromagnetic oxide. Moreover, it was suggested that when the size of three dimensions is reduced to the order of nanometer, it exhibits promising properties that are quite different from their related bulk materials [12, 13]. Thus, d0 ferromagnetic materials were projected as an alternative pathway to TM-doped oxide materials and expected to lead clean materials for spintronics applications. Indeed, many ab-initio studies have predicted d0 ferromagnetism in oxides, such as SnO2, TiO2, and ZrO2 doped with different non-magnetic elements [14–25], and it was observed experimentally as well [26–35]. Recently, our group has observed d0 ferromagnetism in K-doped SnO2 [29], Li-doped SnO2 [30], K-doped rutile TiO2 [31], and Ag-doped monoclinic ZrO2 compounds [35].

Zinc oxide (ZnO) has long been used for many applications such as piezoelectric transducers, varistors, and transparent conducting films, and so on. Interestingly, d0 ferromagnetism was observed in undoped ZnO thin films also, wherein a successive transition from ferromagnetism to paramagnetism and diamagnetism as a function of film thickness was observed [36, 37]. The ferromagnetic order in ZnO thin film was believed to be originated from defects induced [36, 37]. However, the ZnO compound prepared in bulk form was found to exhibit diamagnetic behavior [38, 39]. In addition to undoped ZnO compound, the possibility of d0 ferromagnetism in ZnO was explored by doping with various non-magnetic elements, such as Ag-doped ZnO [38], Li-doped-ZnO [39–43], alkali metal-doped ZnO [44], Cu-doped ZnO [45, 46], and Mg-doped ZnO [47]. Particularly, the magnetic properties of Li-doped ZnO [39–44] were studied by a few research groups by preparing them in different forms of materials using various methods. Lin et al. [40] reported that Li-doped ZnO thin films exhibit paramagnetic behavior, whereas ferromagnetic behavior was observed in Li-doped ZnO, prepared in the form of nanorods [39], thin film [41], and nanoparticles using the sol–gel technique [42] and using chemical precipitation method [43]. Chawla et al. [39] observed that Li-doped ZnO nanorods exhibit d0 ferromagnetism with high Curie temperature and it was proposed that the substitutional Li dopant induces magnetic moments on neighboring oxygen atoms whereas few other groups [41–43] pointed out that complex defects are responsible for controlling and tuning the observed d0 ferromagnetism in Li-doped ZnO compounds. The maximum saturation magnetization in these compounds was observed to be 0.055 emu/gm. Thus, Li-doped ZnO compounds have led to different results on magnetism and a strong dependency of magnetic properties on the preparation method and form of the materials was observed.

Although Li-doped ZnO compounds were studied by a few other research groups, the observed d0 ferromagnetism was never seen in defect-free bulk materials and in most of the cases, it was claimed to be originated from defects in the samples. In a quest of observing the magnetic property in defect-free Li-doped ZnO compounds, we have synthesized these compounds in bulk form at equilibrium conditions using the solid-state route method. As both Zn ions (with ionic radii of 0.74 Å) and Li-ions (with ionic radii of 0.60 Å) have similar ionic radii, there is less probability of having defect in the final prepared materials. Moreover, the solid-state route method has the obvious advantage of eliminating the possibility of clustering/defects which is a serious drawback in the materials prepared in thin-film form or prepared by chemical route. We have demonstrated that these Li-doped compounds exhibit room-temperature bulk ferromagnetism.

2 Experimental details

2.1 Sample preparation

The Li-doped ZnO samples, i.e., Zn1-xLixO (x = 0, 0.02, 0.04, and 0.06) were prepared by solid-state reaction route, using high-purity starting materials of ZnO (Sigma Aldrich, purity-99.999% trace metals basis) and Li2CO3 (Sigma Aldrich, purity-99.997% trace metals basis). As per the chemical analyses report provided by supplier, the trace of magnetic impurities in the starting materials was found to be less than 2 ppm. The essential amount of the starting compounds (ZnO and Li2CO3) was weighed using an electronic balance and they were grinded using a
mortar and pestle under acetone for a proper mixing and homogeneity. After grinding the starting materials, the pre-sintering of the samples in powder form was carried out in air at 100 °C, 200 °C, and 300 °C for 10 h at each temperature. The final annealing of these samples in pallet form was done in the air at 500 °C for 20 h and these samples were used for the study of crystal structure and magnetic properties.

2.2 Measurement of crystal structure and micro-structure

The crystal structure and phase purity of these prepared samples were studied by recording powder X-ray diffraction (XRD) patterns at room temperature using a Philips diffractometer operated in 0–2θ Bragg-Brentano goniometer geometry and by employing CuKα (1.541874 Å) incident radiation beam. Data collections were carried out over a scan range of 20 = 20° to angle 20 = 90° with a step of 0.02°. Microstructural study was carried out by recording microstructural images using a ZEISS-ultra-plus scanning electron microscope (SEM). The elemental analysis was done with BRUKER energy-dispersive spectrometer (EDS) attached in situ with SEM.

2.3 Measurement of magnetic properties

The measurements of the temperature variation of magnetization (M-T) under zero-field-cooled (ZFC) and field-cooled (FC) conditions were carried out under an applied field of 500 Oe and for a temperature range of 3–300 K using commercial SQUID magnetometer (Quantum Design MPMS XL). The field dependence of magnetization (M-H) curves was measured in the field range of ±5 T at 3 and 300 K. The magnetic measurements were performed with utmost care and handling of the samples was done with plastic tweezers.

3 Results and discussion

To understand the crystal structure and phase purity of all prepared Li-doped ZnO samples, X-ray diffraction (XRD) patterns were recorded at room temperature and it is presented in Fig. 1. Un-doped ZnO and Li-doped ZnO samples were found to crystallize in single-phase form with hexagonal ZnO wurtzite structure (space group P63/mc). The XRD data indicate that no secondary phase is present in the compounds within the instrumental limit of the X-ray diffractometer. In order to further ascertain the phase purity and to estimate the lattice parameters ‘a,’ ‘b,’ ‘c,’ and cell volume (V), the recorded XRD patterns were refined with the help of the Fullprof program software by employing the Rietveld refinement technique [48]. The background coefficient parameters, peak shape parameters (α, β, γ, u, v, w), and lattice parameters (a, b, c) were refined during Rietveld refinement. The XRD patterns for all samples could be refined by using the P63/mc space group. The XRD patterns along with Rietveld refinement for ZnO and 6% Li-doped ZnO samples are shown in Fig. 2. Here, the experimental data are shown as open circles and the calculated intensities are shown as solid lines. The bottom line represents the difference between measured and calculated intensities. It is seen that the experimental XRD data match nicely with the Rietveld software calculated XRD data. The refinement of the XRD data indicates that the lattice parameters for the un-doped ZnO compound are found to be a = b = 3.2508 Å, c = 5.2076 Å, and V = 47.66 Å³, and these values are comparable with those reported by other groups [39]. The incorporation of Li atoms into ZnO lattice leads to small decrease in the values of lattice parameters (only in the fourth decimal place), and cell volume up to x = 0.04, followed by an increase for x = 0.06 sample (presented as Fig. 3). The decrease in the lattice parameters can be understood in terms of the
Incorporation of Li-dopant with relatively smaller ionic radii of 0.60 Å, in Zn ions with ionic radii of 0.74 Å. The further increase in lattice parameters is likely due to the distribution of Li to substitutional and interstitial site. However, it is very difficult to comment about the percentage of distribution of Li ions at the substitutional or interstitial sites. The crystallite size ($S_C$) was calculated from the FWHM (full width at half maximum) data obtained from the refinement and using the Debye–Scherrer formula. The crystallite size of un-doped ZnO is estimated to be 35 nm, whereas $S_C$ for 2, 4, and 6% Li-doped ZnO samples is found to be 37, 49, and 38 nm, respectively.

Further, from the microstructural study with SEM, it is observed that the morphology of the samples is quite uniform and nanometric grains have been formed, as demonstrated in Fig. 4a and b for 2 and 4% Li-doped ZnO samples, respectively. The grain size was determined from the analyses of the grain size distribution with software integrated with SEM and; by fitting the distribution of grain size with a Gaussian function. One representative fitting is shown for 4% Li-doped ZnO sample in Fig. 4c and the grain size is found to be the order of 50–70 nm. In order to ascertain if any other unwanted ferromagnetic impurities (such as Fe or Co) have not crept into the final prepared samples, the elemental analysis of the samples has been carried out by recording EDS spectra. One typical EDS spectrum of 4% Li-doped ZnO sample is shown in Fig. 4d. Since Li is very light element, it was beyond the instrumental limit of the EDS to detect the distribution of Li. But the EDS analyses confirm that there is no unwanted magnetic impurity (such as Fe or Co) in the final prepared samples. Usually, the peak position for Fe and Co is at 6.4 and 6.9 keV, respectively, in EDS spectrum, which is absent in these samples.

Followed by the study of crystal structure and micro-structure, the magnetic properties of these prepared samples were studied. As a first step, the magnetic properties of all the starting compounds viz. ZnO and Li$_2$CO$_3$ have been checked and they clearly exhibit diamagnetic behavior (not shown). The measurements of magnetization versus

![Fig. 2 XRD patterns along with Rietveld refinement for a ZnO, b 6% Li-doped ZnO samples. The circles represent experimental points and solid line represents Rietveld refined data. The dotted lines show the difference between experimental and refined data.](a) (b)

![Fig. 3 Variation of crystal structure parameters a, c, and cell volume (V), along with error bar of Zn$_{1-x}$Li$_x$O ($x = 0, 0.02, 0.04$ and $0.06$) compounds](a) (b) (c)
temperature (M-T) and field variation of magnetization (M-H) curves (measured at 3 and 300 K) of undoped ZnO compound indicate a clear diamagnetic behavior, as demonstrated in Fig. 5. Similar diamagnetic behavior in the ZnO compound was reported by other groups also [38, 39]; but this is unlike other reports on ZnO thin film, where ferromagnetism was observed for the undoped ZnO compound due to defects [36, 37]. The measurement of zero-field-cooled (ZFC) M-T curves (Fig. 6) for all Li-doped ZnO compounds indicate that there is a very slow dependency of magnetization with temperature throughout the measured temperature range and there was no ferromagnetic transition up to 300 K, indicating that the transition temperature of all Li-doped ZnO compounds is well above the room temperature. Moreover, an upturn/peak in ZFC M-T curves was observed for 2 and 4% Li-doped ZnO samples at 65 K and 55 K, respectively. Here, the characteristic temperature was estimated from fitting the peak with a Gaussian function. We did not observe this feature for 6% Li-doped ZnO sample. This observed feature could originate from small antiferromagnetic phase embedded in the dominant ferromagnetic matrix or due to some disorder in the lattice. The measurement of field-cooled (FC) M-T data (as shown in Fig. 6) indicates a magnetic irreversibility behavior between ZFC and FC curves for 2, 4, and 6% Li-doped ZnO compounds. The magnetic irreversibility behavior of these samples starts out from 300 K itself and a continuous increase in the value of FC magnetization on lowering the temperature is observed. Moreover, a prominent drastic upturn in the FC magnetization curve, below the characteristic temperature, is observed for 2 and 4% Li-doped ZnO samples. Such magnetic irreversibility between ZFC and FC M-T curves may be attributed to the presence of magnetic spin-glass phase, possibly
arising due to the competing interactions or due to the presence of some disorder in the lattice.

Thus, the limit of lithium doping that introduces the spin-glass behavior in the system is $x = 0.06$, as the system with 6% of Li in ZnO makes the ZFC magnetization curve goes flat. When spin-glass behavior dominates, they eventually cancel out any local magnetic moments and gives rise a flat curve. Usually, such spin-glass or cluster-glass phenomena are commonly observed in magnetically inhomogeneous phase. These effects are normally associated with the competition between two exchange interactions of opposite sign (ferromagnetic versus antiferromagnetic) that frustrates the long-range order [49, 50] and the bifurcation of zero-field-cooled and field-cooled curves of temperature variation of magnetization is generally observed [49, 50]. A similar kind behavior, i.e., presence of magnetic spin-glass phase, was observed in Li-doped ZnO nanoparticles prepared by chemical sol–gel technique [42].

To get a further understanding of the magnetic properties of these samples, we have measured field dependence of magnetization in the field range of ±5 T at 3 K and 300 K for all Li-doped ZnO compounds and they are presented in Fig. 7. The M-H curves measured at 3 and 300 K show typical ferromagnetic hysteresis loop for all Li-doped ZnO compounds. The M-H curves measured at 300 K exhibit ultra-soft coercivity, whereas the M-H curves measured at 3 K exhibit enhanced coercivity (as shown in the inset of Fig. 7b). The coercivity was found to be 50, 25, and 48 Oe for 2, 4, and 6% Li-doped samples, respectively. These observations indicate that all Li-doped compounds exhibit soft ferromagnetic properties. The value of saturation magnetization ($M_s$) at 300 K (as shown in Fig. 8) is observed to be 0.025, 0.008, and 0.002 emu/gm for 2, 4, and 6% Li-doped samples, respectively, which is found to be enhanced at 3 K. The value of $M_s$ at 3 K is found to be 0.098, 0.019, and 0.004 emu/gm for 2, 4, and 6% Li-doped samples, respectively. Thus, at both the measured temperatures, the value of $M_s$ decreases with the increases of Li concentration. Chawla et al. [39] reported the saturation magnetization value of 0.055 emu/gm for 2% Li-doped ZnO, which was the largest $M_s$ value, attributed to substitutional Li. However, Awan et al. [42] have demonstrated that the saturation magnetization of 0.055 emu/gm is due to interstitial lithium. Our measured values (0.098 emu/gm) appear to be higher in comparison to all previously reported values.

Now, let us discuss the observed d$^0$ ferromagnetism in the studied Li-doped ZnO compounds. From the crystal structure study, it is observed that the prepared compounds have been formed in pure
single phase and the incorporation of Li atoms into ZnO lattice leads to very small changes in the lattice parameters (only in the fourth decimal place). The magnetic properties, measured from M-T and M-H curves, indicate that un-doped ZnO compound exhibit diamagnetic behavior, whereas the incorporation of Li into ZnO lattice provoked ferromagnetic behavior with a transition temperature much beyond room temperature. The value of saturation magnetization was found to decrease with the increase in Li concentration. It should be noted that we have not observed any secondary phase from the crystal structure and thus the observed d^0 ferromagnetism is intrinsic in nature. In the recent theoretical model of Ref. [10], it was suggested that three physical parameters are essential to explain induced d^0 ferromagnetism in the case of a direct non-magnetic cationic substitution in oxide materials: (i) the position of the vacancy (defect) induced impurity level, (ii) the density of carrier per defect/vacancy, and (ii) the electron–electron interaction strength. Moreover, it was pointed out that the optimum window of the density of carrier is very important to tune room-temperature ferromagnetism in the non-magnetic element (e.g., Li, Na, K) substitution-induced magnetism in oxides [10, 11]. The incorporation of Li into ZnO will create holes because of its lower valence than Zn and they can form additional band inside the forbidden gap region. The electrons after getting excited from the valance band, try to combine with the holes in this addtional band first, followed by recombining with the holes in the conduction band and it is likely to reduce the bandgap and thus, favoring the holes mediated ferromagnetism. Beyond the optimum value of holes concentration, the magnetic ordering vanishes as magnetic couplings are destroyed by Ruderman–Kittel–Kasuya–Yosida (RKKY)-type oscillations or antiferromagnetic super-exchange. It was further emphasized by this model that when the on-site potential controlling the position of impurity level is too small, antiferromagnetic nearest neighbor couplings exist, whereas

![Fig. 7](image-url) Field variation of magnetization for Zn_{1-x}Li_{x}O (x = 0.02, 0.04, and 0.06) compounds measured at (a) 300 K and (b) 3 K. The inset in figure (b) indicates the coercivity of Li-doped ZnO compounds at 3 K.

![Fig. 8](image-url) The variation of saturation magnetization (M_s) with Li concentration, measured at 3 and 300 K for Zn_{1-x}Li_{x}O (x = 0.02, 0.04, and 0.06) compounds.
the coupling becomes ferromagnetic with the increase of the on-site potential strength. However, with a further increase of this potential, some couplings become antiferromagnetic again. Our experimental results on Li-doped ZnO compounds may fall in line with the predictions of this theory. Our experimental observations show that Li incorporation in ZnO lattice leads to d0 ferromagnetic ordering at room temperature. With the increase of Li-doping, a greater number of holes are created and thus it may also create Ruderman–Kittel–Kasuya–Yosida-type oscillations in the couplings, leading to the appearance of spin-glass phase, as observed from magnetic irreversibility behavior between ZFC and FC M-T curves. Moreover, RKKY-type oscillations in the couplings gradually reduce the magnetic ordering, resulting a decrease of the saturation magnetization with the increase of Li concentration, as observed from the M-H measurement.

4 Conclusion

To conclude, we have studied the crystal structure, micro-structure, and magnetic properties of Li-doped ZnO compounds, i.e., Zn1-xLi xO (x = 0, 0.02, 0.04, and 0.06), prepared by solid-state reaction route method. From the study of crystal structure by X-ray diffraction (XRD) patterns, it is evident that the prepared materials have been formed in single phase of the hexagonal wurtzite structure. The refinement of the XRD patterns suggests that there is small decrease in the lattice parameters up to 4% of Li incorporation in ZnO, followed by a decrease for 6% of Li. The average crystallite size (S C) was found to be in the range of 35–50 nm. The microstructural study by scanning electron microscope reveals the uniform morphology of the grains in the range of 50–70 nm in size. The energy-dispersive spectrum indicates that no unwanted ferromagnetic impurities have crept into the final prepared samples. The measurement of the temperature (T) variation of magnetization (M) with SQUID magnetometer indicates that un-doped ZnO exhibits diamagnetic property but all Li-doped compounds exhibit room-temperature ferromagnetism. The ZFC and FC M-T curves indicate a magnetic irreversibility for all Li-doped compounds, revealing the co-existence of the spin-glass phase in the system. The measurements of hysteresis curves (M-H) at 3 and 300 K indicate that Li-doped samples exhibit ferromagnetic loops with ultra-soft coercivity (~ 50 Oe) and the maximum saturation magnetization (0.10 emu/gm) was observed for x = 0.02 sample. The value of Ms decreases with an increase in Li concentration. Thus, from the results of M-T and M-H measurements, we can conclusively say that Li-doped compounds exhibit defect-free bulk ferromagnetism with a transition temperature much beyond room temperature and co-existence of low-temperature spin-glass phase.

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