Drastic improvement in magnetization of CdO nanoparticles by Fe doping

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Abstract Hydrothermal route was used in the preparation of Fe-doped CdO nanoparticles with 3, 5, 7 and 10 wt% and their structural and magnetic properties were investigated. Structural characterization using powder X-ray diffraction confirmed the single phase cubic symmetry of these nanoparticles. Particle size decreased monotonically with increasing the dopant ion concentration. The microstructure of the synthesized samples was studied by a high-resolution transmission electron microscopy (HRTEM) which confirmed the nanocrystalline nature of the samples. A vibrating sample magnetometer provided the hysteresis curves of all samples. Pure CdO reveals ferromagnetic behavior while antiferromagnetization is observed for all Fe-doped samples. Large coercivity as well as large area for the sample $x = 0.05$ is achieved.

Keywords DMS · Fe-doped CdO · Coercivity · Spin valve

Introduction

Diluted magnetic semiconductors (DMSs) are defined as nonmagnetic semiconductors in which a small fraction of host cations is replaced by transition metal (TM) or rare-earth (RE) ions, with both spin and charge degrees of freedom in a single material (Ohno 1998; Pearton et al.

2007). In these materials, the presence of magnetic ions such as $3d$ (TM) and/or $4f$ (RE) ions leads to an exchange interaction between itinerary sp band electrons or holes and the d -electron spins localized at the magnetic ions, leads to versatile magnetic field-induced functionalities (Furdyna 1988). Recently, DMSs have fascinated researchers because of their potential applications in spin-based multifunctional electronic devices including spin field-effect transistors, optoelectronics, field emission devices, gas sensors, ultraviolet absorbers, quantum computer, and nonvolatile memory devices (Moezzi et al. 2012; Chu et al. 2009; Zhang et al. 2009; Liu et al. 2009; Neal et al. 2006; Huang et al. 2009).

Among all the oxide DMSs, the transition metal (TM)-doped CdO has been considered as a promising one because the host material CdO is an n -type semiconductor material with a direct band gap in the range of ~ 2.2 – 2.7 eV and a strong excitonic binding energy at room temperature. Furthermore, CdO nanoparticles have interesting properties such as low electrical resistivity in the range of $\sim 10^{-2}$ – 10^{-4} Ω cm and high transmission in the visible region (Waghulade et al. 2007; Lewis and Paine 2000; Yan et al. 2001; Carballeda-Galicia et al. 2000). CdO and TM-doped CdO are valuable for a wide range of optoelectronic applications such as transparent conducting oxide, photo transistors, photo diodes, solar cells, transparent electrodes, and gas sensors (Aydin et al. 2012; Zhao et al. 2002; Daza et al. 2001). Hence, TM ions such as Mn, Co, Ni, Cu, and Fe have been tried as dopants for CdO, with the objective of modify some of their properties (Jin et al. 2001; Ahmad et al. 2013, 2014; Dakhel 2014). Particularly, iron has been recognized as one of the most efficient doping element to improve and tune the structural, electrical and magnetic properties of CdO nanoparticles due to its unique chemical stability. Moreover, doping of

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CdO with Fe^{2+} ions of smaller size than that of Cd^{2+} [ionic radii of Cd^{2+} and Fe^{2+} are 0.95 and 0.78 Å, respectively (Shannon 1976)] could create ferromagnetic properties at room temperature.

The properties of nanoparticles are sensitive to the conditions of their preparation. Variety of synthesis routes for the preparation of ultrafine oxide nanoparticles including: sol–gel (Li et al. 2014), hydrothermal (Moulahi and Sediri 2014), microwave irradiation (Rajesh et al. 2015), conventional precipitation (Rahmati et al. 2014; Esmat et al. 2017), and sonochemical methods (Ranjbar and Morsali 2011) have been investigated. Among these, the hydrothermal method is considered as a one of the best methods for synthesizing high crystalline advanced materials with control in the nanostructure and surface properties. Nevertheless, very few reports on the magnetic behavior of Fe-doped CdO, synthesized using hydrothermal method have been studied. The aim of the present work is to explore and tailor the magnetic properties of Fe-doped CdO nanoparticles synthesized by hydrothermal method and to investigate the influence of doping ratio on the ferromagnetic properties to use CdO nanoparticles in various DMS applications.

Methods

Materials

All chemicals used for the preparation of CdO and Fe-doped CdO were of analytical grade and used without any further purification. They include cadmium acetate [$\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, 99.0%], iron sulfate [$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, > 99.0%] and sodium hydroxide (NaOH, 99.99%).

Fe-doped CdO nanoparticle synthesis

For the synthesis of $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00, 0.03, 0.05, 0.07$, and 0.1) nanoparticles, cadmium acetate [$\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, 99.0%] and iron sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, > 99.0%) were prepared separately in double distilled water and then mixed together according to the required amounts of the corresponding chemical reagents under magnetic stirring at room temperature. To this solution, 2 M of sodium hydroxide (NaOH, 99.99%) was added dropwise. The solution was stirred for 2 h at room temperature. The mixture was transferred into a 100 mL Teflon-lined stainless steel autoclave. The hydrothermal synthesis was heated at 160 °C and maintained for 10 h in an electric oven and then cooled down to room temperature. Resulting white precipitate was washed thoroughly using double distilled water and ethanol to remove the impurities adsorbed on the surface. The hydroxide, thus

formed was dried at 80 °C and grinded into powder. Finally, the powder was annealed at 500 °C for 3 h and cooled down to room temperature.

Characterization

The crystalline structure of the prepared samples was examined by X-ray diffractometer (PANalytical Empyrean) equipped with graphite monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 1.54056$ Å, 40 kV, 35 mA). The samples were scanned in the angular range from 20° to 80° (2θ) with scanning rate 0.005°/s and step size 0.02°. SEM-Joel 5410 attached with EDS unit, Oxford (INCAPentaFETx3) was used in the determination of the nominal weight ratios and the elemental compositions. High-resolution transmission electron microscopy (HRTEM, JEM-2100) was used for microstructure and particle shape observation. Magnetic properties of the formed powders were investigated at room temperature using a vibrating sample magnetometer (VSM, Lakeshore 7304) with an applied field from − 20,000 to 20,000 Oe.

Results and discussion

It is well known that CdO adopts the cubic structure. One aimed to examine the influence of Fe dopant on the CdO crystal structure using XRD. Figure 1 represents the XRD patterns of the investigated samples. The data pointed to good crystallinity as strong reflections appeared from the planes located at $2\theta = 33.08^\circ, 38.38^\circ, 55.34^\circ, 65.96^\circ$ and 69.32° and indexed to the planes (111), (200), (220), (311) and (222). The charts are compared and indexed according

Fig. 1 X-ray diffraction patterns of as-synthesized undoped and various contents of Fe-doped CdO nanopowder

to the standard ICDD 04-006-0781. The samples are very homogenous in a single phase of cubic symmetry with space group Fm3m. Here, Fe^{2+} obviously replaced the Cd^{2+} ions at the octahedral coordination. The lattice parameter was calculated from the following equation based on cubic symmetry and reported in Table 1:

$$a = d\sqrt{h^2 + k^2 + l^2}, \quad (1)$$

where d is the interplanar distance and (hkl) are the miller indices as indexed from ICDD cards.

The theoretical density was computed from $[D_x = ZM/NV]$, where Z is the number of molecules per unit cell, M is the molecular weight, N is the Avogadro's number and V is the unit cell volume. The crystal size was calculated from the corrected broadening of the most intense peak in Debye–Scherrer equation (Zhou and Guo 1999). The general trend of the data in Table 1 is the decrease in the crystallite size with increasing Fe content from 50 nm at $x = 0.0$ to 30 nm at $x = 0.1$. This was attributed to the difference between the ionic radii of both Cd^{2+} (0.95 Å-6f) and Fe^{2+} (0.78 Å-6f). This is also reflected as the general behavior of the change in (a) vs Fe content (x) (Fig. 5). The theoretical density was lowered by increasing the Fe content owing to difference in the atomic weight of both Cd (122.41 amu) and Fe (55.85 amu) which could not be compensated by the unit cell volume shrinkage.

EDS spectrum showed the elemental composition of the $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ nanostructures. It also indicates the successful doping of Fe in the CdO matrix.

The EDS spectra of $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ nanostructures are shown in Fig. 2. The EDS spectra contain the signals corresponding to Cd, O and Fe which confirm the presence of all key elements (Cd, O and Fe) in the nanostructures. The quantity of Fe doping estimated by the EDS measurements was found to be in good agreement with actual concentration of Fe doping in the samples. The quantitative analysis of all the samples confirms the oxygen-rich stoichiometry by showing the dominance of oxygen over cadmium.

HRTEM micrographs are presented in Fig. 3a–e. The pure CdO sample displayed platelet shape of homogenous

size and tolerable distribution as the powder was prepared without surfactant as shown in Fig. 3a. The particle size distribution is generally dependent on the type and/or the ratio of surfactant. Another important issue is that small-sized particles are electrostatically attracted to each other, which is our case. By doping CdO with Fe ($x = 0.03$) the shape is kept unchanged while agglomeration started to appear owing to magnetostatic interactions between the nanoparticles (Fig. 3b). The size is reduced from 50 nm for $x = 0.0$ to 41 nm at $x = 0.03$. At $x = 0.05$, the micrograph illustrated some elongated fiber-like shape on the boundaries of the platelets. This will be reflected in the magnetic properties. The perfect crystalline phase is clarified from the lattice fringes and the observed interplanar spacing as clear from the inset of Fig. 3c. The appearance of a dislocation in the magnified micrograph is clearly zoomed. This will consequently be reflected in the measured magnetic properties. Selected area electron diffraction assures the crystalline nature of CdO with perfect symmetry as agreed with XRD findings that the nano CdO either undoped or Fe-doped samples crystallized in cubic symmetry with space group Fm-3m. The elongated fibers may result from the anisotropic growth of CdO crystals at this doping ratio. With further increasing Fe content to attain $x = 0.07$, the platelet shape predominates again. When $x = 0.10$, the ratio is large enough to cause high agglomeration owing to the magnetic nature of the Fe itself. Selected area electron diffraction assumed polycrystalline nature as well as the ordered crystalline phase despite the small particle size.

Figure 4 illustrates the room temperature M–H loop of the investigated DMS nanopowder. The magnetic parameters are depicted from the M–H loop and listed in Table 2. The anisotropy constant value was calculated from the following equation (Kambale et al. 2009):

$$H_c = \frac{0.96K}{M_s}. \quad (2)$$

The pure CdO nanoparticles, $x = 0.0$, reveal a clear hysteresis loop with a remarkable area and early saturation. This is an evidence of a weak ferromagnetic order despite

Table 1 Values of the crystallite size, lattice parameter (a), volume (V), and theoretical density (D_x) for $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00, 0.03, 0.05, 0.07$ and 0.10) nanostructures

Sample	Crystallite size (nm)	Lattice parameter (Å)	Volume (Å ³)	D_x (g/cm ³)
$x = 0.00$	50	4.6937	103.41	8.247
$x = 0.03$	41	4.6931	103.36	8.142
$x = 0.05$	41	4.6981	103.69	8.043
$x = 0.07$	34	4.6836	102.74	8.044
$x = 0.10$	30	4.6989	103.75	7.858

Fig. 2 EDS spectra with elemental concentration of undoped and Fe-doped CdO nanopowder [$\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00, 0.03, 0.05, 0.07$, and 0.1)]

the diamagnetic nature of the CdO itself. This agrees well with the reported ferromagnetic behavior of CdO (Bououdina et al. 2015). The difference here is annealing atmosphere where our system is annealed in air. Here the situation could be a consequence of the existence of some oxygen vacancies ordered in such a way to result in a weak long range ferromagnetic order with a very small value of remanence but with a considerable coercivity value and a pronounced area. The calculated squareness ratio is 0.12 and is correlated to magnetostatic interactions between nanoparticles. The asymmetry of the hysteresis loop around the H -axis is certainly due to an exchange bias phenomenon (Arman and El-Dek 2017; Azab et al. 2016).

This room temperature exchange bias seen and recorded here for the first time in nonmagnetic nanoparticle system is certainly amazing which needs further studies at low temperatures. This phenomenon is related to the surface driven change with respect to the bulk of the CdO itself. One could expect that oxygen vacancies are ordered in such a way that surface magnetic configuration is quite different than that of the bulk. Another plausible reason is that the core spins configuration differs much more than the surface ones (exchange bias) as reported in Table 2. We expected that surface magnetization is larger than bulk one which completely disagrees with the reported literature for nanomaterials (Mansour et al. 2016). Figure 4b represents

Fig. 3 High-resolution TEM images of $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ nanoparticles for **a** $x = 0.00$, **b** $x = 0.03$, **c** $x = 0.05$, **d** $x = 0.07$ and **e** $x = 0.10$. The insets of **c** show high-resolution TEM image and selected area electron diffraction pattern for the $\text{Cd}_{0.95}\text{Fe}_{0.05}\text{O}$ nanoparticles. The electron diffraction pattern corresponds to the cubic structure

the (M–H) loop for the sample with $x = 0.03$ as a separate loop. It is clearly seen that butterfly shape hysteresis is obtained with lack of saturation even at large field values (20,000 Oe). Another striking feature is the low value of M_s (0.31 emu/g) which points to weak ferromagnetic component. The most probable description of this situation is canted antiferromagnetic character accompanied with weak ferromagnetism. The common character is the asymmetric

loop around the origin. This could be due to an exchange bias phenomenon where the positive H_c value is unequal to the negative one as added in Table 2. By doping CdO with 0.03 Fe, the resultant loop lacked saturation with an anti-ferromagnetic character rather than typical ferromagnetic one. This could be due to the antiferromagnetic superexchange interaction between Fe^{2+} ions located at the equivalent crystallographic lattice sites. Increasing Fe

Fig. 4 **a** Room temperature M–H hysteresis loops for $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00, 0.03, 0.05, 0.07$ and 0.10) nanostructures. The inset shows M–H loop for the $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00$) nanostructure. **b** Room temperature M–H hysteresis loops for $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.03$) nanostructures

Table 2 Magnetic parameters for $\text{Cd}_{1-x}\text{Fe}_x\text{O}$ ($x = 0.00, 0.03, 0.05, 0.07$ and 0.10) nanostructures

Sample	M_s (emu/g)	M_r (emu/g)	H_c (Oe)	H_c (Oe)		SQR = M_r/M_s	Area (erg/g)	K (erg/g)
				Positive	Negative			
$x = 0$	0.12	0.016	96.48	140.65	– 52.302	0.13	112.03	12.41
$x = 0.03$	0.31	0.044	114.27	109.10	– 119.44	0.14	433.98	37.34
$x = 0.05$	1.16	0.413	852.81	916.97	– 788.64	0.36	5289.7	1031.01
$x = 0.07$	0.56	0.067	83.67	81.856	– 85.493	0.12	367.15	49.21
$x = 0.1$	0.92	0.093	86.09	93.345	– 78.827	0.10	207.73	82.41

content up to 0.05 drastic changes were noted, namely, (1) the largest value of both M_s and H_c , (2) the colossal compositional changes of M_s and H_c were calculated from

$$\frac{\Delta M_s}{M_s(x=0)} = \frac{M_s(x=0.05) - M_s(x=0)}{M_s(x=0)} \times 100 = 886\% \quad (3)$$

and

$$\frac{\Delta H_c}{H_c(x=0)} = \frac{H_c(x=0.05) - H_c(x=0)}{H_c(x=0)} \times 100 = 784\%, \quad (4)$$

(3) enlargement of the loop area reaching 5289 erg/g, and (4) the exchange bias field increased. All these findings are not only correlated to the Fe doping ratio but also to some changes in the particle shape. From a closer look to the HRTEM Fig. 3c, the nanoparticles appear to have two different shapes; platelets and fibers. This observation leads to the assumption of the contribution of shape anisotropy to the total one, as clear from the values of K in Table 2. The dislocation observed in HRTEM in Fig. 3c for $x = 0.05$

Fig. 5 Variations of magnetization and lattice parameter as a function of Fe doping concentration

likely behaves as a pinning layer preventing demagnetization. Accordingly, this is an evidence of large coercivity for this sample as a special case. The existence of some defects that could result from the smaller ionic radius of

Table 3 Comparative report on the values of crystallite size, saturation, remanent magnetization and coercivity for pure and Fe-doped CdO

Sample	Crystallite size (nm)	Preparation method	M_s (emu/g)	M_r (emu/g)	H_c (Oe)	References
Pure CdO	50	Hydrothermal	0.12	0.016	96.48	Our study
Pure CdO	44.8	Thermal co-decomposition	0.0011	0.16	90	Dakhel et al. (2014a)
Pure CdO	44.8	Thermal co-decomposition	0.0011	0.16	90	Dakhel et al. (2014b)
Fe-doped CdO	36.5	Thermal co-decomposition	0.0029	0.19	13	Dakhel et al. (2014a)
$Cd_{1-x}Fe_xO$ ($x = 0.03$)	41	Hydrothermal	0.31	0.044	114.27	Our study
$Cd_{1-x}Fe_xO$ ($x = 0.05$)	41	Hydrothermal	1.16	0.413	852.81	Our study
$Cd_{1-x}Fe_xO$ ($x = 0.07$)	34	Hydrothermal	0.56	0.067	83.67	Our study
$Cd_{1-x}Fe_xO$ ($x = 0.1$)	30	Hydrothermal	0.92	0.093	86.09	Our study
Fe-doped CdO 0.25% Fe	33.7	Thermal co-decomposition	0.025	0.0001	60	Dakhel et al. (2014b)
Fe-doped CdO 0.50% Fe	37.2	Thermal co-decomposition	0.001	0.00005	35	Dakhel et al. (2014b)
Fe-doped CdO 0.75% Fe	31.6	Thermal co-decomposition	0.006	0.00065	45	Dakhel et al. (2014b)
Fe-doped CdO 1.0% Fe	34.8	Thermal co-decomposition	0.0055	0.00064	79	Dakhel et al. (2014b)
Fe-doped CdO 1.4% Fe	31.9	Thermal co-decomposition	0.0074	0.00015	12	Dakhel et al. (2014b)
Fe-doped CdO 1.8% Fe	33.8	Thermal co-decomposition	0.0115	0.00021	13	Dakhel et al. (2014b)

Fe^{2+} as compared with Cd^{2+} resulted in the formation of the so-called magnetic polarons. This in turn could represent a milestone with change of the magnetic properties of such weak system (parent compound).

Further increasing Fe content up to 0.07 decreased all magnetic parameters. Size dependent magnetic properties are certainly tuned by the coherence length as one of the fundamental magnetic lengths (anisotropy, crystalline, etc.). One of the essential incentives for the exploration of magnetic nanomaterials is the exciting variation of magnetic properties that happen when the particle size drops less than a definite extent. Alterations in the magnetization arise via activation over an energy barrier, and each physical mechanism accountable for an energy barrier has an associated length scale.

The major magnetic lengths for magnetic materials principally comprise the crystalline anisotropy length (l_K), the applied field length (l_H), and the magnetostatic length (l_S) (Leslie-Pelecky and Rieke 1996),

$$\begin{aligned}
 l_K &= \sqrt{J/K}, \\
 l_H &= \sqrt{2J/KM_S}, \\
 l_S &= \sqrt{J/2\pi M_S^2}.
 \end{aligned}
 \quad (5)$$

If there exist more than one type of barrier, the magnetic materials are dominated by the smallest characteristic length. These lengths are predominantly in the order of 1–100 nm (Leslie-Pelecky and Rieke 1996; Holz and Scherer 1994). Here, in this case, the most effective magnetic length is the crystalline anisotropy one which depends mainly on the anisotropy constant. The later increased extremely owing to the contribution of shape anisotropy. Accordingly, the sample with $x = 0.05$ possesses critical crystalline anisotropy length.

Decreasing the particle size and increasing Fe content up to 0.05 drastically changed the magnetic properties. A nice correlation is plotted in Fig. 5 when the Fe content is allowed to reach 0.1, i.e., $x = 0.1$, the magnetization increased again. One could argue that Fe doping is a successful way of spin injection into CdO to obtain DMS of small size with extraordinary magnetic properties. The obtained value of magnetization here is superior to those reported for Mn-doped CdO (Kumar et al. 2015), Co-doped CdO (Ahmad et al. 2014), Fe-doped CdS (Kaur and Verma 2015) and Fe-doped ZnO (Kumar et al. 2014). The colossal compositional variation of H_c as reflected from the anisotropy with the large value of room temperature coercivity recommend the use of this type of magnetic nanoparticles as pinning layer in spin valves demanding difficult and large demagnetizing field.

Table 3 is a comparative report on the magnetic properties of pure and Fe-doped CdO. Evidently, our results revealed improved magnetization as well as coercivity for all samples despite the small differences in crystallite size (Dakhel et al. 2014a, b).

Conclusion

Successful Fe-doped CdO nanoparticles were synthesized in single phase of cubic symmetry. Ferromagnetic character of the undoped sample is obtained. The magnetization is increased with increasing Fe ratio. Colossal compositional increment of H_c is reported especially at $x = 0.05$. This trend was correlated to anisotropic change and structural dislocations. These nanoparticles are recommended for further investigations and possible applications in spin valves as a pinning layer.

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