Optimal temperature and Mn content for enhancing optical properties and inducing room temperature ferromagnetism of Mn doped In_2O_3 nanocubes

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Abstract

In this strategy, chemical vapor deposition technique is utilized to synthesize undoped and Mn doped In_2O_3 nanostructures. Temperature effects on oxygen content, doping level and the associated morphological, structural, optical and magnetic properties have been investigated. The surface morphology examinations showed the ability of manganese ions to catalyze the nanoflakes of undoped In_2O_3 to grow into nanocubes. An appropriate proposal for the growth mechanism has been elucidated. X-ray diffraction proved that Mn doping enhanced the growth of In_2O_3 along different crystallographic planes with preferred growth of the (222) plane. The strain and dislocation density were decreased while grain sizes were increased with increasing Mn content. The optical analysis showed reduction in transmission that was related to oxygen vacancies in undoped In_2O_3 and phase separation in doped samples. The optical band gap values were decreased from 3.82 to 3.71 eV with increasing Mn content whereas the refractive index values were increased. The magnetic response elucidated the optimal temperature and Mn content for inducing the room temperature ferromagnetism.

Keywords Mn doped In_2O_3 · CVD · Nanoflakes · Nanocubes · Oxygen vacancies · Ferromagnetism · Optical properties

1 Introduction

In recent years, the researcher's interest in synthesizing diluted magnetic semiconductors (DMS) have been increased. DMS are new class of materials that join the properties of both magnetic materials and semiconductors ones. The initial studies on tri-element diluted semiconductors showed limitation due to their low Curie temperature and high raw-material cost. The introduction of transition elements into a compatible semiconductor recently became the most prevailing setting for the fabrication of room temperature ferromagnetism (RTFM) [1]. As one of these systems,

Mn doped In_2O_3 have been attracted the researcher's interest, because Mn brings new physics by the introduction of localized spins [2]. Several techniques have been developed in order to obtain controllable shape, structure and dimension of DMS [3]. Unluckily, the low solubility of transition elements form clusters and heterogeneous regions that hinder the realization of RTFM [4]. On contrary, the chemical vapor deposition technique (CVD) is marked by the preparation of homogeneous alloys with high quality and performance [5, 6]. Also it allows the direct synthesis utilizing pure oxides, metals or mixed oxides/metals as precursors avoiding reactions of poisonous and potentially expensive starting materials. Via controlling the parameters of syntheses (such as the amount of doping materials, growth temperature, percentage and type of reactive and carrier gases, thickness and type of the catalyst, type and location of the substrates) different types of morphologies can be precisely obtained [7–9].

The origin of RTFM of Mn doped In_2O_3 has been interpreted by various models [10–21]. For instance, the theoretical s-d microscopic model and the green's function theory attributed the RTFM to the surface oxygen vacancies plus the disparity of ionic radii of both dopant and host [22].

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Fig. 1 The temperature profile of the tube furnace

Wang et al. [23] suggested that oxygen vacancies play mediated role in making coupling between Mn ions. Okazaki et al. [24] attributed the room temperature ferromagnetism in Mn and Fe co-doped In_2O_3 to the coexistence of Mn^{2+} and Mn^{3+} ions.

The optical properties of In_2O_3 nanostructures have found to be sensitive to Mn doping ratio [25]. Anand et al. [26] reported a red shift in the optical band gap and high photoluminescence intensity up on Mn doping in In_2O_3 nanoparticles. Sun et al. [27] found that by tailoring the optical band gap of the prepared Mn doped In_2O_3 thin films, the photoelectrochemical (PEC) activities are promoted and the PEC water splitting efficiency is enhanced. Amiri et al. [28] used the first-principles calculations that are based on the density functional theory, to calculate the optical spectra of Mn doped In_2O_3 in the energy range 0–40 eV. They found that the refractive index increased with Mn doping.

The current study aimed to examine the influence of Mn content on tuning the morphological, optical and magnetic properties of In_2O_3 nanostructure using CVD technique. The challenge here lies in determining the range of temperatures and Mn content that achieve the room temperature ferromagnetism. Also, proving that the synthesis of DMS at relatively high temperature do not disturb the magnetic dipoles. The confirmation of the oxygen deficient for inducing room temperature ferromagnetisms has been also investigated.

2 Experimental procedures

Thin films of undoped and Mn doped In_2O_3 nanostructures were synthesized using chemical vapor deposition technique. A horizontal tube furnace of inner diameter 5.5 cm and length of 100 cm was used for samples preparation. The deposition were achieved on Au-coated (~ 15 – 20 nm) Si (100) and quartz substrates that are previously cut into $1 \times 1 \text{ cm}^2$ and cleaned via ultrasonic equipment. Mixtures

of In + In_2O_3 granules with ratios of 1:1 wt.% were used as precursor for the In_2O_3 nanostructures growth at temperature of 1000 °C. The Mn doped In_2O_3 nanostructures were synthesized using mixtures of In + In_2O_3 + MnO_2 granules with ratios of 2:2:1 wt.% at temperatures of 1000 °C (Mn-1000) and 1030 °C (Mn-1030) (Aldrich chemicals; 99.98%). The suitable quantities were loaded in an alumina boat at the center of the tubular furnace and heated with a rate of 9° per minute, while the substrates were located along 15 cm distance after the boat. Figure 1 shows the temperature gradient through which the tested films are placed. Gas mixture of Ar and O_2 was introduced into the system with flow rates of 200 sccm and 15 sccm in series, where argon was allowed to flow from the start of the experiment while oxygen flow was synchronized with the synthesis stage. The time of synthesis was 3 h for each group after that the furnace was cooled naturally to room temperature. A typical white–yellow precipitate was carefully collected for examination.

The crystal structure was examined using X-ray diffraction (XRD). The patterns were achieved by Bruker D8 ADVANCE diffractometer. The diffractometer utilizes Cu-K_α radiation of wavelength $\lambda = 1.54056 \text{ \AA}$. The morphology of the prepared thin films was observed by field emission scanning electron microscope (SEM) (model Quanta 250 FEG). The elemental ratios were evaluated by Energy Dispersive Analyses of X-ray (EDAX) unit attached to SEM. The thicknesses were estimated utilizing a Form Talysurf 50 profilometer. The reflectance (R) and transmittance (T) spectra were measured at normal incidence using Jasco V-670 UV–Vis–NIR spectrophotometer over the wavelength range from 200 to 2500 nm. The vibrating sample magnetometer (mode 7410 series VSM, Lake Shore cryotronics, Westerville, OH, USA) was utilized to test the magnetic response of the samples under the application of magnetic field. The measurements were done at room temperature.

Fig. 2 XRD of **a** undoped In_2O_3 , **b** Mn-1000 and **c** Mn-1030 thin films

3 Results and discussion

3.1 Structural, morphological and compositional examinations

The X-ray diffraction patterns of the films are depicted in Fig. 2a–c. Clearly, all diffracted peaks are basically indexed to single phase of cubic In_2O_3 (COD: 2310009) with the domination growth of the (222) planes that is more clear in Mn-1030. No prominent change in diffracted peaks of Mn-1000 (Fig. 2b) compared with the undoped In_2O_3 (Fig. 2a) that confirms the good solubility of Mn ions within the indium oxide crystal lattice. In addition, there is a possibility that the Mn ions may segregate to the amorphous parts at grain boundaries [29, 30]. The separate phases of In (COD: 9012377), Mn (COD: 1539039) and MnO_2 (COD: 9011409) appeared in Mn-1030 (Fig. 2c), as a result of exceeding the limit of solubility in In_2O_3 crystal lattice. Also, the competitive growth of the overcrowded vapor species in the ambient of the reaction may delay the crystal growth [31]. The small lower shift in the diffracted peaks of 0.3° in case of Mn-1000 is due to the lattice expansion by the incorporation of Mn^{+2} ions (ionic radius $0.83 \text{ \AA}$) at

the expense of In^{+3} ions (ionic radius $0.8 \text{ \AA}$) [32]. In case of Mn-1030 (Fig. 2c), the shift in 2θ does not exceed 0.02 referring to the existence of different oxidation states other than Mn^{+2} ions in the In_2O_3 crystal matrix.

The preferred orientation can be determined by calculating the relative texture coefficient, $\text{TC}(hkl)$, known as Harris analysis [33] given by the equation:

$$\text{TC}(h_i k_i l_i) = \left(\frac{I(h_i k_i l_i)}{I_r(h_i k_i l_i)} \right) \left(\frac{1}{N} \sum_{i=1}^N \frac{I(h_i k_i l_i)}{I_r(h_i k_i l_i)} \right)^{-1} \quad (1)$$

where $I(h_i k_i l_i)$ refers to the intensity of the diffracted peaks of the sample, $I_r(h_i k_i l_i)$ refers to the intensity of the standard powder sample (COD cards) and N is the number of diffraction peaks. If $\text{TC}(h_i k_i l_i)$ is more than 1, the plane is said to be preferred oriented. The estimated texture coefficient are presented in Table 1 that indicate the preferred orientation of the plane (222).

The crystallite size (C), dislocation density (δ) and strain (ϵ) are depicted in Table 2 using the equations:

$$C = \frac{0.9\lambda}{\beta \cos \theta_c} \quad (2)$$

$$\delta = \frac{1}{C^2} \quad (3)$$

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (4)$$

where β refers to the FWHM and λ refers to the wavelength of X-ray. It is noticeable that both strain and dislocation density are decreased while grain sizes are increased with increasing Mn content.

The surface morphology in Fig. 3 shows uniform distribution over the whole film surface. In case of undoped In_2O_3 , at 1000°C (Fig. 3a, b), entangled nanoflakes with terraced nanopyramids are grown perpendicular to the substrate. The research results of Herrera et al. [32] showed the inability of synthesizing thin films of In_2O_3 below 1200°C using CVD technique. The incorporation of Mn in the structure catalyzed the nanoflakes to grow into nanocubes (Fig. 3c, d). The same result is reported in ref [34]. The elongated growth of indium oxide cannot be justified due to the small difference in surface energy along different crystallographic planes [32]. Increasing the deposition temperature to 1030°C (Fig. 3e, f) stimulated the cubes to grow in a direction perpendicular to substrates. Noteworthy, the growth temperature, the flow rate of the carrier gas, the duration of reaction and the nature of the substrate are number of factors that have great influence on different properties of the product.

An explanation of the growth mechanism of nanostructures thin films can be summarized as following: (i)

Table 1 X-ray diffraction intensities and relative texture coefficients

(hkl)	I_r	Pure In_2O_3		Mn-1000		Mn-1030	
		I	$\text{TC}(hkl)$	I	$\text{TC}(hkl)$	I	$\text{TC}(hkl)$
Δ (211)	111	98	1.15	95	0.97	96	1.39
O (111)	19					18	1.52
$\square$ (210)	999					49	0.08
* (110)	457					40	0.14
Δ (222)	1000	1000	1.31	1000	1.13	1000	1.60
Δ (400)	293	234	1.04	234	0.90	235	1.29
Δ (411)	45					36	1.28
Δ (332)	35					29	1.33
Δ (431)	46	34	0.97	33	0.81	34	1.185
Δ (440)	375	232	0.81	240	0.72	152	0.65
Δ (622)	261	145	0.73	158	0.68	89	0.55

The symbols Δ , O, $\square$ and * referee to In_2O_3 , Mn, MnO_2 and In phases

Table 2 Precursors, crystallite size (C), dislocation density (δ) and strain (ϵ) of Mn doped In_2O_3

Sample	Precursors	Crystallite size (nm)	Dislocation density (lines/nm ²)	Strain
Pure In_2O_3	In + In_2O_3 (1:1 wt.%)	30	1.1×10^{-3}	1.2×10^{-3}
Mn-1000	In + In_2O_3 + Mn (2:2:1 wt.%)	35	8.2×10^{-4}	9.9×10^{-4}
Mn-1030	In + In_2O_3 + Mn (2:2:1 wt.%)	42	5.6×10^{-4}	9.3×10^{-4}

Fig. 3 SEM images with low and high magnifications for **a, b** undoped In_2O_3 , **c, d** Mn-1000 and **e, f** Mn-1030 thin films

Table 3 Elemental ratios (at.%) and optical band gap (E_g) of Mn doped In_2O_3

Sample	Elemental ratios (at.%)				E_g (eV)
	In	Mn	O	Composition	
In_2O_3	42.11	—	57.89	$\text{In}_2\text{O}_{2.75}$	3.82
Mn-1000	40.01	4.23	54.71	$\text{In}_2\text{Mn}_{0.15}\text{O}_{2.73}$	3.77
Mn-1030	38.9	7.02	54.08	$\text{In}_2\text{Mn}_{0.26}\text{O}_{2.78}$	3.71

vaporization of solid species (ii) vapor transportation by carrier gases, (iii) species deposition on substrates surfaces, finally (iv) nucleation and growth on different substrates. The utilization of Au catalyst is in order to protect the volatile species from being submerged [35]. With deep insight, one can see random nucleation then nucleation and growth that are governed by the surface energies [36]. In this context, indium and indium oxide are dissociated into various species that are transported by carrier gases to different substrates. The low formation energy of In_2O_3 ($\Delta_f H_m^0 = -925.8 \text{ kJ mole}^{-1}$) facilitated the growth of In_2O_3 into nanoflakes then terraced nanopyramid. On the other hand, the high formation energy of MnO_2 ($\Delta_f H_m^0 = 42 \pm 1.6 \text{ kJ mole}^{-1}$) helped the existence of Mn ions as doping element instead of being as separate phase [37]. The substitute of In^{+3} by Mn^{+2} creates charge imbalance that may be returned by somehow. At 1000 °C, the difference of electro negativities of In (1.78) and Mn (1.55) ions is not sufficient to restore the charge balance. Increasing temperature to 1030 °C will increase the charge motility, hence approaching charge symmetry [32], see EDAX results in Table 3. As a result of solubility limit Mn ions in In_2O_3 crystal lattice, a separate phase of MnO_2 are created at temperature of 1030 °C.

The EDAX analysis is used to quantify the elemental ratios in the composition. Generally, the data recorded in Table 3 shows over stoichiometric of indium due to oxygen deficient in all the synthesized films, in consistent with that previously reported data [38, 39]. The checkup on Mn-1000 and Mn-1030 indicated the presence of Mn atoms with approximate ratios of 4.23 at.% and 7.02 at.%, respectively, that are faring from the nominated values. The reason may arise from the low vapor pressure of Mn ions. The same result is recorded in ref [40]. Wongsaprom et al. [41] attributed the low Mn doping in In_2O_3 to the substitution of In by Mn ions in the In_2O_3 matrix. Berardan et al. [42] found that the maximum solubility limit of Mn^{3+} ions in In_2O_3 do not exceed 7.5 at.% using sintering process in air. In this regard, when In^{+3} is replaced by Mn^{+2} ions, oxygen vacancies are created in case of Mn-1000 and reduced in case of Mn-1030 in terms of temperature increase [32] and the favor of charge neutrality by Mn^{+3} ions.

Fig. 4 Transmission and reflection of undoped and Mn doped In_2O_3 films **Fig. 5** $(\alpha h\nu)^2$ versus $h\nu$ for undoped and Mn doped In_2O_3 films

3.2 Optical properties

The behavior of light when fall on the undoped and Mn doped In_2O_3 thin films is depicted in Fig. 4. The transmission of the undoped In_2O_3 is not very high as expected which is further reduced in Mn-1000 and Mn-1030. This is mainly originated from light scattering by oxygen vacancies in case of In_2O_3 and to phase separation and electronic polarization in cases of Mn-1000 and Mn-1030 [37]. The reflectance exhibits an opposite behavior to transmittance.

The direct band gap (E_g) are calculated from Tauc formula by taking the relation between $(\alpha h\nu)^2$ versus $h\nu$ where the intercepts with energy axis represents E_g values (Fig. 5) [43]. Table 3 summarized the E_g values that are decreased from 3.82 to 3.71 eV with increasing Mn content. Similar results are obtained in refs [19, 44–47]. This decrease can

Fig. 6 Refractive index **a** and extinction coefficient **b** as functions of wavelength for undoped and Mn doped In_2O_3

be ascribed to the SP-D exchange between the localized d-electrons of Mn cations and the In_2O_3 band electrons. This exchange interaction resulted in lowering conduction band and raising the valence band gap edges, therefore shrinking the optical band gap [46].

Figure 6 shows the changes in refractive index (n) with wavelengths that show anomalous dispersion in UV region and normal dispersion in visible and near IR region. The undoped In_2O_3 shows n values smaller than its counterpart using flux method [48]. The reason may arise from oxygen deficient as indicated in EDAX results [49]. The increase in refractive index for the doped thin films arise from the high density of Mn (7.43 g/cm^3) compared to that of In (7.31 g/cm^3). The extinction coefficient (k) of Mn-1000 and Mn-1030 shows steeper shoulder around 400 nm that is higher than other regions, due to electrons transitions to the impurity levels [50]. The opposite is observed in the undoped In_2O_3 with slight shift toward shorter wavelengths. Generally, various defects in the structure promotes different absorption types at different wavelengths besides the fundamental one.

3.3 Magnetic behavior

The magnetic response of undoped and Mn doped In_2O_3 at room temperature is depicted in Fig. 7a–c. Although diamagnetic behavior is observed in undoped In_2O_3

Fig. 7 The magnetic behavior at room temperature of undoped and Mn doped In_2O_3

[33] hysteresis loops are created in case of Mn-1000 and Mn-1030 at low magnetic field ($H = 116.75 \text{ G}$). The reduction in saturation magnetization (M_s) of Mn-1030 (0.006 emu/g) compared with Mn-1000 (0.14 emu/g) indicates that secondary phase in Mn-1030 is not responsible for inducing the room temperature ferromagnetism. The responsible is the trapped electrons by oxygen vacancies that are spin oriented under the application of magnetic field [1]. In Mn-1000 the spin orientations are in the same direction, while in Mn-1030 the tilt present in hysteresis loop with prominent decrease in saturation magnetization may arise from spiral domain wall movement clockwise and counterclockwise due to different Mn ions that are spun oppositely [51]. It is worth noting that the values of M_s obtained in Mn-1000 are greater than those values obtained by Zong et al. [21]. The disparity of coercivity and retentivity values of Mn-1000 (33.09 G and $8.99 \times 10^{-3} \text{ emu/g}$) compared with (43.12 G and $1.2 \times 10^{-3} \text{ emu/g}$) of Mn-1030 ensures the differences in their structures and morphologies. Although Mn-1030 has the lowest dislocation density, see Table 2, the anisotropic structure by phase separation represents an obstacle for domain walls movement and this is not recommended in practical applications. On the other hand, the higher retentivity and the lower coercivity of Mn-1000 are related to their homogenous structure and morphology

that facilitate the domain wall movement hence inducing their beneficial as soft ferromagnetism [51, 52]. Small coercive is beneficial for pulse transformer in the construction of its core [52].

4 Conclusion

Diluted magnetic semiconductors have been successfully prepared using CVD technique. The various analyses showed that although Mn doping has slight impact on the crystal structure, it has a significant effect on the magnetic, optical and morphological properties. The E_g values decreased from 3.82 to 3.71 eV with increasing Mn content. The n values, between 200 and 2000 nm, increased with increasing Mn content. The behavior of hysteresis loop and EDAX analysis was tabulated the optimal temperature and Mn content for inducing the room temperature ferromagnetisms to be 1000 °C and 4.23 at.%, respectively. The higher retentivity and the lower coercivity of Mn-1000 allow their use in pulse transformer.

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Declarations

Conflict of interest The authors declare that there are no conflict of interest either personally or financially.

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