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Original research article

Electrochemically synthesized 1D and 3D hybrid Fe^{3+} doped ZnSe dandelions for photoelectrochemical cell application

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ABSTRACT

In the present study, Fe^{3+} doped ZnSe thin films are deposited using the galvanostatic mode of electrodeposition. Further, deposited thin films are characterized using X-ray diffraction study, X-ray photoelectron spectroscopy and Raman spectroscopy for their confirmation. After that morphological study is carried using field emission scanning electron microscopy, topological study is carried using atomic force microscopy (AFM). Optical properties are studied using optical absorbance and photoluminescence. The electrochemical properties are studied using electrochemical impedance spectroscopy. The most important part is study of photoelectrochemical properties. In the present study, 0.75% Fe^{3+} doped ZnSe thin film shows the relatively better photoelectrochemical cell performance than the other deposited thin films due to dandelion like nature. The observed short circuit current is 155 μA and open circuit voltage is 487 mV. The calculated Fill factor and efficiency are 0.35 and 0.17%, respectively.

1. Introduction

The construction of functional molecular alloys with well defined shapes and structures is of great interest because of a variety of applications. 1D nano materials are also very useful in multiple applications [1–6]. Recently, one dimensional (1D) and three dimensional (3D) semiconductor nanostructures have received much attention for potential applications in photonics, optoelectronics, field emission, catalysis, energy conversion, and sensing. Also they have unique electronic, optical, chemical, and mechanical properties [2–4]. The dandelions are the product of rods and spheres. The electrodeposition is promising method for developing nanorods [7]. It is because; in electrodeposition constant current density has an attractive approach for free growth of nanorods along the c axis under the weak electric field [8]. Electrodeposition is one of the most widely accepted techniques for the inexpensive and well-organized growth of the films in aqueous medium, and numerous reports are available on the deposition of ZnSe thin films by this technique [9,10].

The ZnSe is wide band gap (2.7 eV) II-IV semiconducting material. Also Fe doped ZnSe is diluted magnetic semiconductor, which is obtained by doping Fe into the lattice of ZnSe matrix. The magnetic, electronic and magneto-optic properties of transition metal doped ZnSe alloys have been studied by a number of researchers [11,12]. Lv et al. [13] reported the ZnSe

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nanorods via a surfactant soft-template method. Das et al [14] reported ZnSe nanorods by chemical reduction method. Wang et al reported ZnO nanorod arrays with dandelion-like morphology [15]. Also in our previous report, we have synthesized Fe^{2+} doped ZnSe nanostructures using potentiostatic mode of electrodeposition [16], the Fe^{3+} doped ZnSe nanoparticals using potentiostatic mode of electrodeposition [17], and the Fe^{2+} doped ZnSe nanostructures using galvanostatic mode of electrodeposition [18]. Also, effect of high energy electron irradiation on Fe^{2+} doped ZnSe nanorods and their modified properties are explained [19]. Also, using galvanostatic mode of electrodeposition, ZnSe and Fe doped ZnSe [20,21] thin films are deposited and work has been published. But, Fe^{3+} doped ZnSe nanostructures using galavanostic mode of electrodeposition and its properties are not mentioned.

By observing our previous work and literature [16–21], in the present work, efforts are focused on synthesis of Fe^{3+} doped ZnSe thin films using galvanostatic mode of electrodeposition and their corresponding, morphological, optical, structural, photoelectrochemical and electrochemical properties. Also, observed properties of present work are compared with our previous work. So, to the best of our knowledge and after observing the literature, electrosynthesized Fe^{3+} doped ZnSe dandelion using galvanostatic mode of electrodeposition is not observed. In the present study, we have discussed these results with our previous work on the basis of electrodeposition mode and Fe^{2+} and Fe^{3+} doping.

2. Experimental details

The Fe^{3+} doped ZnSe dandelions were synthesized using the analytical grade reagents. It contains, zinc nitrate $Zn(NO_3)_2{\cdot}6H_2O$, selenium dioxide (SeO_2), ferric nitrate nonahydrate $Fe(N_3O_9)$, triethanolamine $N(CH_2{\cdot}CH_2{\cdot}OH)_3$ (TEA), sulphuric acid 98% (H_2SO_4), acetone. For electrodeposition, potentiostat, tin doped indium oxide (ITO) substrates were used. In the present work, synthesis of Fe^{3+} doped ZnSe thin films using the galvanostatic mode of electrodeposition is discussed. These thin films were deposited at 338 K in aqueous alkaline bath. The electrodeposition bath consisted 0.25 M zinc nitrate $Zn(NO_3)_2.6H_2O$, 1 mM selenium dioxide (SeO_2), 0.25 M ferric nitrate nonahydrate $Fe(N_3O_9)$ used for Fe^{3+} doping. By adding sulphuric acid (H_2SO_4) solution, the pH has been maintained ~2.5. In order to dope Fe^{3+} in ZnSe, 0.5%, 0.75% and 1% of ferric nitrate are selected and the deposition is carried out for 40 min. For the deposition, ITO substrates are used. Electrodeposition of ZnSe and Fe^{3+} doped ZnSe thin films were carried out using the galvanostatic mode at a constant current density 70 $\mu A/cm^2$. After the deposition of 0, 0.5, 0.75 and 1% Fe^{3+} doped ZnSe thin films were named as sample 1, sample 2, sample 3 and sample 4 respectively.

3. Results and discussion

3.1. X-ray diffraction (XRD) study

Fig. 1 shows the XRD patterns of sample 1, 2, 3 and 4. The recognized d-spacing characters are in favorable with JCPDS Card No-01-089-2940 and JCPDS Card No-00-037-1463. The XRD analysis conceded that thin films are polycrystalline with hexagonal as well as cubic structure. The peaks are noticed at an angle 25.24, 29.78, 37.69 and 45.29° which applied to hexagonal structure (JCPDS Card No - 01-089-2940) and it displays the planes (100), (101), (102) and (110) respectively. The peaks are observed at an angle 27.25° and 45.23° which belong to cubic structure (JCPDS Card No 00-037^{-1}463) [22] which shows the planes (111) and (220) respectively. The Δ and δ represents the peak belongs to ITO and $ZnSeO_3$, respectively revealed in Fig. 1. The crystallite size has been calculated using the Scherrer's formula [23–25]. The observed crystallite sizes for sample 1, 2, 3 and 4 are 61, 40, 32 and 42 nm, respectively. The ionic radii of Zn (0.74 Å) is more than Fe (0.64 Å). When Zn^{2+} atoms are replaced by Fe^{3+} there is change in intensity as well small change in peak position. Also, small change in intensity is responsible for change in FWHM of observed peaks and resultant change in crystallite size and this is also a reason for decrease in crystallite size [16,17].

3.2. X-ray photoelectron spectroscopic study (XPS)

The chemical properties of electrodeposited sample 1 and sample 3 were investigated by XPS. Figs. 2 and 3 are XPS spectra of sample 1 and sample 3, respectively. All the XPS peaks in Fig. 2(a) properly indicate ZnSe. XPS consists of different peaks consequent to different electronic levels of Zn and Se in sample 1. Fig. 2(b) and (c) show the narrow scan for Zn and Se, respectively. Similarly Fig. 3 represents the XPS scan for sample 3; Fig. 3(a) shows the survey scan for sample 3. Fig. 3(b)–(d) show high resolution XPS spectra obtained for Zn, Se and Fe, respectively. Figs. 2(b) and Fig. 3(b) show the characteristic peaks in the Zn (2p) spectra for sample 1 and sample 3. It is signifying the presence of element Zn in both samples. Nevertheless, to decide accurately the features of the double peaks of Zn ($2p_{3/2}$) and Zn($2p_{1/2}$) are disintegrated by using Voigt curve fitting function. These two disintegrated peaks of the samples 1 are positioned at the binding energy of 1022.03 eV and 1045.04 eV, for Zn ($2P_{3/2}$) and Zn ($2P_{1/2}$), respectively. Similarly, two disintegrated peaks of the samples 3 are placed at binding energy of 1021.79 and 1044.92 eV, for Zn ($2p_{3/2}$) and Zn ($2p_{1/2}$), respectively. These peak locations agree well with literature [26] for Zn ($2p_{3/2}$) and Zn ($2p_{1/2}$). The observed $\Delta\upsilon$ for Zn in ZnSe is 23.01 eV and the observed $\Delta\upsilon$ for Zn in Fe^{3+} doped ZnSe is 23.13 eV. The change in $\Delta\upsilon$ in sample 3 is indicating that, Fe^{3+} successfully doped in sample 3. Figs. 2(c) and Fig. 3(c) show enclosed double peak characteristic in the Se (3d) spectrum for both samples which promote the presence of element Se in both samples. A strong Se (3d) peak superposed on subordinate peak is observed for both the samples. The disintegration of