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Synthesis of ZnO nanoflakes by the wet chemical method in the presence of Pb^{2+} alien cation and their structural and morphological properties

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ABSTRACT

Pb^{2+} assisted ZnO nanoflakes were synthesized by the wet chemical method. The structure and phase purity of the samples were identified as hexagonal wurtzite structure from X-ray diffraction (XRD). The effect of Pb^{2+} concentration on the morphology of ZnO nanostructures was investigated using a scanning electron microscope (SEM). The formation mechanism of ZnO nanoflakes is selective adsorption of cationic Pb^{2+} on certain crystallographic facet. The single crystalline quality of the nanoflakes was confirmed from high resolution transmission electron microscopy (HRTEM) image. The presence of Pb^{2+} additives with ZnO nanostructures were identified by energy dispersive spectroscopy (EDS) and X-ray fluorescence (XRF) spectroscopy. From EDS and XRD analysis, it is found that Pb^{2+} cations were not incorporated into the lattice and only were used as an additive for the synthesis of nanoflakes.

1. Introduction

Over the past decade, numerous efforts have been employed in controlling the sizes and shapes of inorganic nanocrystals which can determine their electrical and optical properties. ZnO is a wide band gap (3.37 eV) semiconductor having remarkable optical, electrical and optoelectronic properties. The properties of ZnO are strongly dependent on its structure, including the morphology and aspect ratio, as well as the size, orientation and density of the crystal [1], thus generating of immense interest in research. ZnO with different morphologies have been synthesized due to its potential applications such as optoelectronic devices, spintronics, gas sensors, solar cells, light emitting diodes and an antibacterial agent [2–5]. Various parameters like pH, concentration of precursor, reaction temperature, reaction time, solvent and the method employed will affect the morphology and size of the product [6–8]. Comparatively few reports are available on the effect of alien ions on the size and morphology of ZnO nanostructures.

In recent years, many research groups are focusing on the effect of inorganic ion additives on the morphology of semiconducting nanomaterials. For example, Dong et al. have reported the effect of alien cations (Pb^{2+}, Cu^{2+}, and Co^{2+}) on the morphology of ZnO nanostructures [9]. Pb-doped ZnO nanowires have been synthesized by the modified thermal evaporation method and the effect of Pb-doping on the morphology, structure and optical properties of ZnO nanowires has been reported [10]. Zhou et al. have reported the synthesis and properties of Pb-doped ZnO nanowires by the conventional vapor–liquid–solid (VLS) technique [11]. The ionic radius of Pb^{2+} (1.19 Å) is larger than that of Zn^{2+} (0.74 Å). Hence, detailed study is still required to identify the modifications in structure as well as in morphology as a result of Pb^{2+} incorporation into ZnO nanostructures. In the present work, we have investigated the effect of Pb^{2+} concentration over the morphology of ZnO nanostructures. Further, the structural, morphological and compositional properties have been studied by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS) measurements.

2. Experimental details

Synthesis of ZnO nanostructures: All the reagents used in the experiment were of analytical grade and procured from Sigma Aldrich. In the typical experiments, 0.25 M zinc acetate dehydrate $[Zn(CH_3COO)_2 \cdot H_2O]$ was dissolved in 50 mL of deionized water and 0.075 M sodium hydroxide (NaOH) was dissolved in 50 mL of deionized water. When NaOH solution was added dropwise into the above solution, white gel was formed. The gel was allowed to precipitate at room temperature for 5 h. The precipitate was

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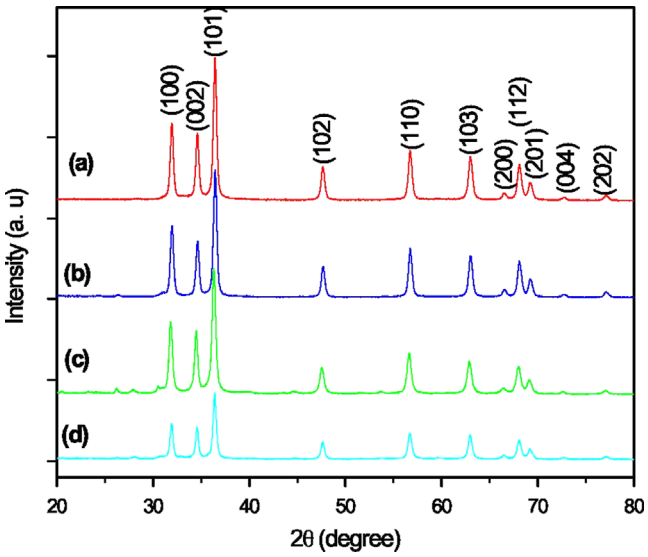

Fig. 1. XRD patterns of (a) Z1, (b) Z2, (c) Z3 and (d) Z4 nanostructures.

collected by centrifugation and washed using deionized water and ethanol and named as Z1. To study the morphology evolution of ZnO nanostructures, the content of Pb^{2+} was added at three different concentrations (0.0025, 0.005 and 0.0075 M) into the zinc acetate solution during the synthesis. The precursor taken for Pb^{2+} is lead acetate ($Pb(CH_3COO)_2$). Then the NaOH is added for the precipitation of $Zn(OH)_2$. Precipitate was subsequently washed three times using water and ethanol. The purpose of washing with ethanol is to remove the impurities in the resultant products. Finally, the product was dried in a furnace at 250 C° for 5 h. The obtained products with different Pb^{2+} concentrations were named as Z2 (Pb^{2+} 0.0025), Z3 (Pb^{2+} 0.0050) and Z4 (Pb^{2+} 0.0075).

Characterization: In order to identify the phase purity and crystalline structure of synthesized ZnO nanostructures, the XRD patterns were recorded with a powder X-ray diffractometer (X'Pert PRO MPD, Panalytical, Cu-Kα radiation, $\lambda = 0.154178$ nm). The morphology and chemical composition of the samples were inspected using a field emission scanning electron microscope (FESEM) and energy dispersive X-ray spectroscopy (EDS) measurements, respectively by a Quanta 200 FEG scanning electron microscope. Transmission electron microscopy (TEM) and high resolution transmission electron

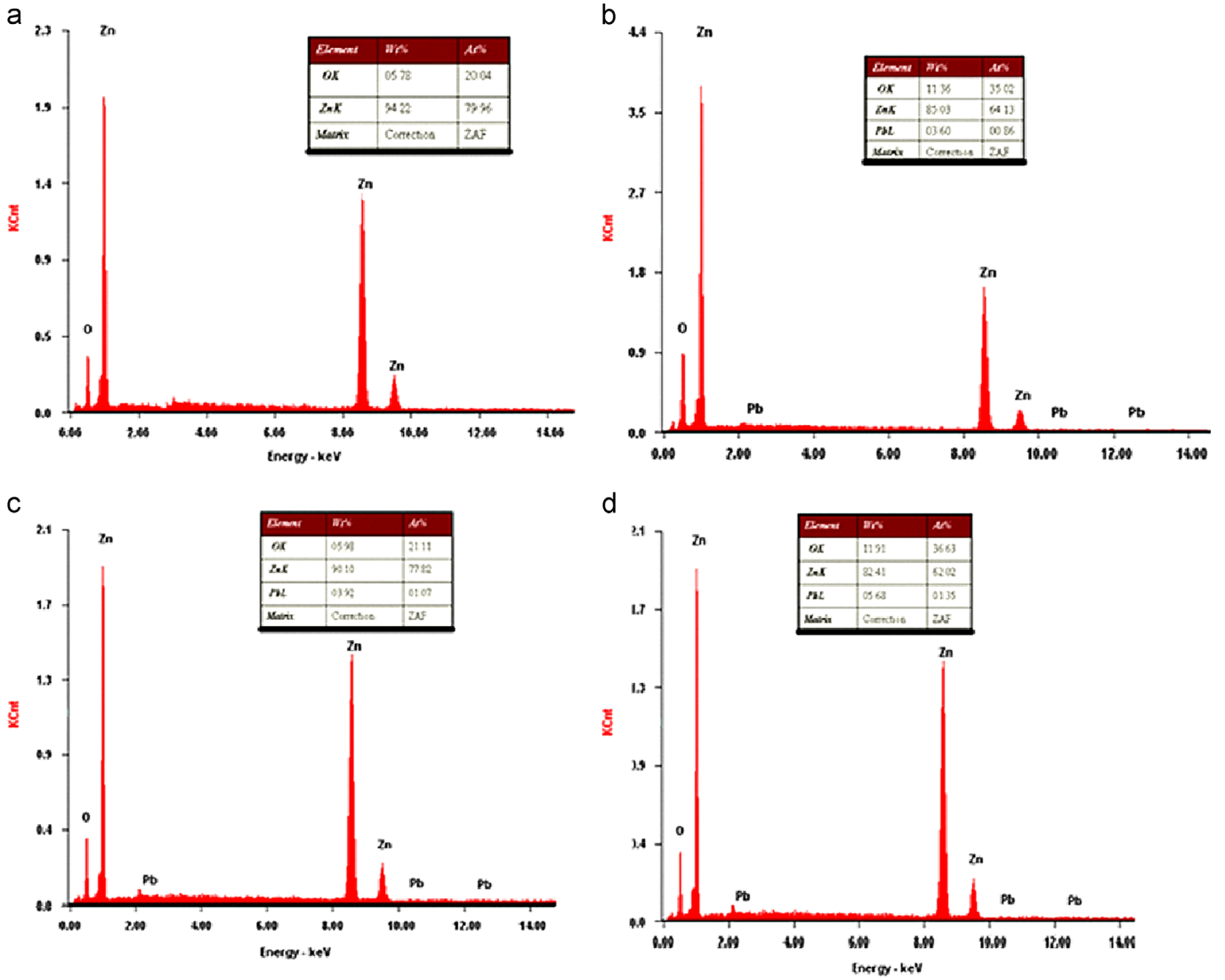

Fig. 2. EDS spectra of (a) Z1, (b) Z2, (c) Z3 and (d) Z4 nanostructures.