

A study on the microstructural parameters of $Zn_{(1-x)}La_xZr_xO$ nanopowders by X-ray line broadening analysis

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In the present study, the pure and La-Zr co-doped ZnO nanoparticles were prepared by sol-gel technique using zinc acetate dehydrate ($Zn(Ac)_2 \cdot 2H_2O$), lanthanum nitrate hexahydrate ($La(NO_3)_3 \cdot 6H_2O$) and zirconium chloride ($ZrCl_4$) as precursor. The structure and morphology of the prepared nanoparticle samples were studied using X-ray diffraction and transmission electron microscopy measurements. X-ray diffraction results indicated that all the samples have crystalline wurtzite phase. TEM showed that powder was polycrystalline in nature with random distribution of the pure and La-Zr doped ZnO nanoparticles. We demonstrate strain-size evaluations for pure and doped ZnO nanoparticles from the x-ray line profile analysis. The microstructural effects of crystalline materials in terms of crystallite sizes and lattice strain on the peak broadening were investigated using Williamson-Hall (W-H) analysis and size- strain plot (SSP) method. The average crystallite size of $Zn_{(1-x)}La_xZr_xO$ nanoparticles estimated from the W-H analysis and SSP method varied as the doping concentration increased. The incorporation of Zr^{4+} ion in the place of Zn^{2+} caused an increase in the size of nanocrystals as compared to undoped ZnO. The average particle sizes of co-doped ZnO nanoparticles estimated from the USDM model is in good agreement with the TEM results.

Keywords: ZnO; X-ray diffraction; TEM; Williamson-Hall analysis; SSP model

1. Introduction

Nano-structured metal oxide semiconductors are gaining attention due to their wide band-gap and related properties. Among the semiconducting materials, zinc oxide (ZnO) is a promising candidate due to its excellent physical and chemical properties for a wide range of applications such as varistors, luminescence, electrostatic dissipative coatings, transparent UV protection films, chemical sensors, etc.¹⁻⁵. ZnO nanoparticles in both powder and film form can be synthesized using various methods such as chemical vapor deposition, chemical spray pyrolysis, sol-gel technique, and hydrothermal treatment⁶⁻¹⁰. ZnO is doped with different types of metallic ions in order to enhance the optical and conducting properties¹¹. In the recent times, transition metal-doped ZnO (e.g., La, Zr, ...) has been broadly researched and concentrated on luminescence properties, magnetic, optical and photocatalytic activity, sensor and memory applications¹²⁻²⁰. It is evident that the materials in a nanometer scale have a large surface area and surface energy of the system. Therefore, a simple relaxation (expansion or contraction) of the crystalline lattice may lead to stabilization of metastable nanostructure. The change in lattice parameter of metal-doped ZnO powders is dependent upon the ionic radius of doping ion, which can substitute the Zn ion in the lattice²¹. The ionic radius of the dopant ion is important factor, which can strongly influence the ability of the dopant to enter into oxides

crystal lattice. If the ionic radius of the doping metal ions matches those of the lattice metal ion in oxides, the doping metal ion will substitute itself for the lattice in the doping reactive process (substitutional mode). Whereas, the ions with the radius which are much bigger or smaller than that of metal ion in oxides cause crystal lattice distortion^{22,23}. In the doping reactive process it can either isomorphously substituted or interstitially introduced into the matrix of ZnO to produce oxygen vacancies which accelerate the nanocrystallite growth of wurtzite ZnO²⁴. In addition, La-Zr co-doped ZnO did not give any peak corresponding to ZrO₂ or La₂O₃, possibly demonstrating that La³⁺ and Zr⁴⁺ were dispersed uniformly onto ZnO nanoparticles as the form of small cluster La₂O₃ or ZrO₂. This is due to the formation of nanosize particles in the range undetectable by XRD. X-ray profile analysis is a simple and powerful tool to estimate the crystallite size and lattice strain²⁵. There are many analytical techniques to evaluate the microstructure properties of materials²⁶⁻²⁹. To our knowledge, from the point of view of the microstructural properties, comparing many reported nanostructures synthesised, little work has been carried out to evaluate the microstructure properties of La-Zr doped ZnO nanopowders. This study highlights the microstructure analysis of La-Zr doped ZnO nanoparticles. The present work highlights the structure and morphology of pure and co-doped ZnO nanoparticles by X-ray diffraction

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analysis (XRD) and transmission electron microscopy (TEM). From the modified Williamson- Hall procedure and the size-strain plot (SSP) method, we give more information on strain-stress and the energy density of pure and doped ZnO nanoparticles. A comparative evaluation of the mean particle size of pure and doped ZnO nanoparticles obtained from direct TEM measurements and from powder X-ray diffraction (XRD) peak broadening is also investigated.

2. Experimental Details

La-Zr co-doped ZnO nanoparticles were synthesized by a simple sol-gel route which its details reported elsewhere³⁰. We will discuss in the following the results of the structure analysis and the chemical composition measurements. The bulk sensitive X-ray diffraction (XRD) patterns were taken with Philips X'Pert diffractometer at room temperature using monochromatic Cu K α ($h\nu = 8042.55$ eV) excitation. Measurements were taken under beam acceleration conditions of 40 kV/35 mA. The surface sensitive transmission electron spectroscopy (TEM) measurements were obtained on a Philips, CM10 instrument with an accelerating voltage of 100 kV.

3. Results and Discussion

3.1. Methods of X-ray profile analysis

X-ray diffraction line profile analysis is one of the earliest methods which is conventionally used to study the physical and microstructural parameters of polycrystalline materials. Many new methods have been proposed to extract microstructural information from the XRD line profile. Fig. 1(a, b, c, d) shows the x-ray diffractogram of the Zn_(1-x)La_xZr_xO nanoparticles; $x=0,0.02,0.04,0.06$. A preferable growth along the {101}, {002}, {100}, {102}, {110} and {103} directions could be indexed as hexagonal wurtzite phase of ZnO as according to the JPCDS card number: 36-1451³¹. The calculated lattice parameters of the La-Zr co-doped ZnO nanoparticles are $a=b=3.2264$ Å⁰, $c=5.1739$ Å⁰ are closely well agreement with the reported values (JCPDS Card No. 01-089-0510). In addition, La-Zr

co-doped ZnO did not give any peak corresponding to ZrO₂ or La₂O₃, possibly demonstrating that La³⁺ and Zr⁴⁺ were dispersed uniformly onto ZnO nanoparticles. As it's evident from the table 1, the crystallite size of ZnO nanoparticles increases with increasing La-Zr ratio. The right graph in the Fig. 1 shows a negligible shift in (110) Bragg reflection for the samples with a different amount of La-Zr compared to the ZnO nanoparticles. This shift could be attributed to the strain in the lattice of compounds. Also, it is expected the replacement of some Zn²⁺ ions with the Zr⁴⁺ in each compound due to their different ionic radius. In order to find the effect of strain on the peak broadening, we use a modified equation and adopted the following techniques of line profile analysis to obtain microstructural information from the symmetrically broadened diffraction profiles.

3.1.1. Williamson-Hall Technique

In almost all cases X-ray diffraction profiles are influenced not only by crystallite size but also possibly by lattice strain and lattice defects. Williamson and Hall proposed a method for deconvoluting size and strain broadening by looking at the peak width as a function of 2θ . However it makes some very large assumptions along the way³²⁻³⁴. According to the Williamson-Hall (W-H) method the individual contribution to the line broadening of a Bragg reflection can be expressed as³⁵

$$\beta_{hkl} = \beta_D + \beta_\epsilon \quad (1)$$

$$\beta_{hkl} \cos\theta = (k\lambda / D) + 4\epsilon \sin\theta \quad (2)$$

Where β_{hkl} is the peak width at half-maximum intensity, β_D is due to the contribution of crystallite size, β_ϵ is the peak broadening due to the strain (ϵ) and D is the average crystallite size of a X-ray peak. In the Eq. 2 the strain was assumed to be uniform in all crystallographic direction implying a uniform deformation model ('UDM'). Fig. 2 shows the UDM analysis. The term ($\beta_{hkl} \cos\theta$) is plotted versus ($4\sin\theta$). The effective crystallite size can be estimated from the extrapolation on the plot and the slope of the fitted line represents the strain. Deviation from the straight line fit in Fig. 2 represents that an anisotropic approach such as

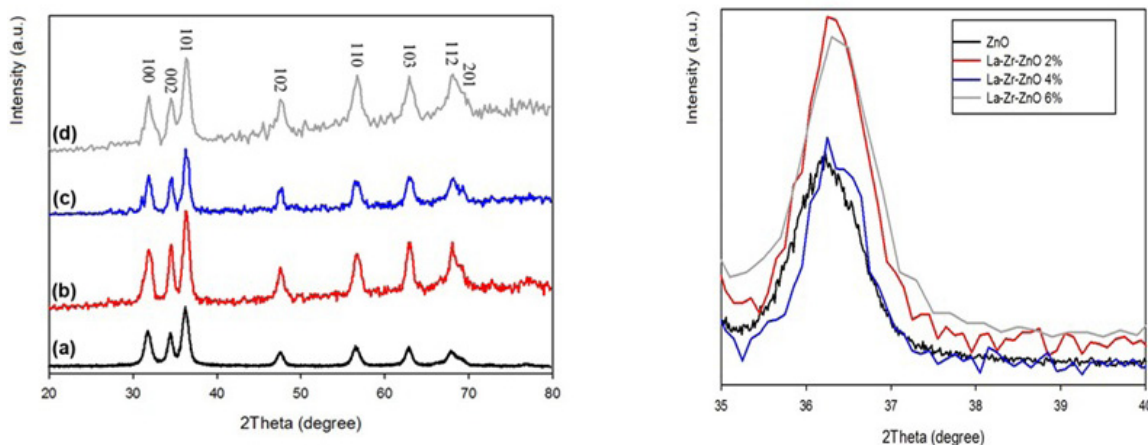


Fig.1. Powder X-ray diffraction data of (a) ZnO, (b) Zn_{0.98}La_{0.02}Zr_{0.02}O, (c) Zn_{0.96}La_{0.04}Zr_{0.04}O, and (d) Zn_{0.94}La_{0.06}Zr_{0.06}O nanoparticles. The right graph shows doping-induced (002) peak shift with La-Zr elements in the ZnO matrix.

Table 1. Microstructural parameters of Zn_(1-x)La_xZr_xO nanoparticles.

Ba Concentration (x)	Williamson - Hall Method									Size – Strain Plot Method				TEM	
	UDM			USDM			UEDDM			D (nm)	ε×10 ⁻³ (no unit)	σ (MPa)	u (Kj m ⁻³)	Std. Error	D (nm)
	D (nm)	ε×10 ⁻³ (no unit)	σ (MPa)	D (nm)	σ (MPa)	ε×10 ⁻³ (no unit)	D (nm)	u (Kj m ⁻³)	ε×10 ⁻³ (no unit)						
0	18.83	5.83	18.46	742.22	5.84	18.73	21.82	5.86	744.22	39.68	2.802	355.85	49.855	1.6032E-06	≈35[7]
0.02	22.48	6.23	22.31	801.56	6.31	22.55	25.19	6.29	798.83	47.17	1.529	194.18	14.845	1.7306E-06	-
0.04	33.83	5.26	25.93	582.47	4.59	29.11	15.06	4.87	618.49	53.57	3.654	484.06	92.249	3.2920E-06	-
0.06	59.09	10.24	52.77	1436.29	11.31	76.38	87.73	11.75	1492.25	67.57	1.838	233.43	21.453	7.1333E-07	50

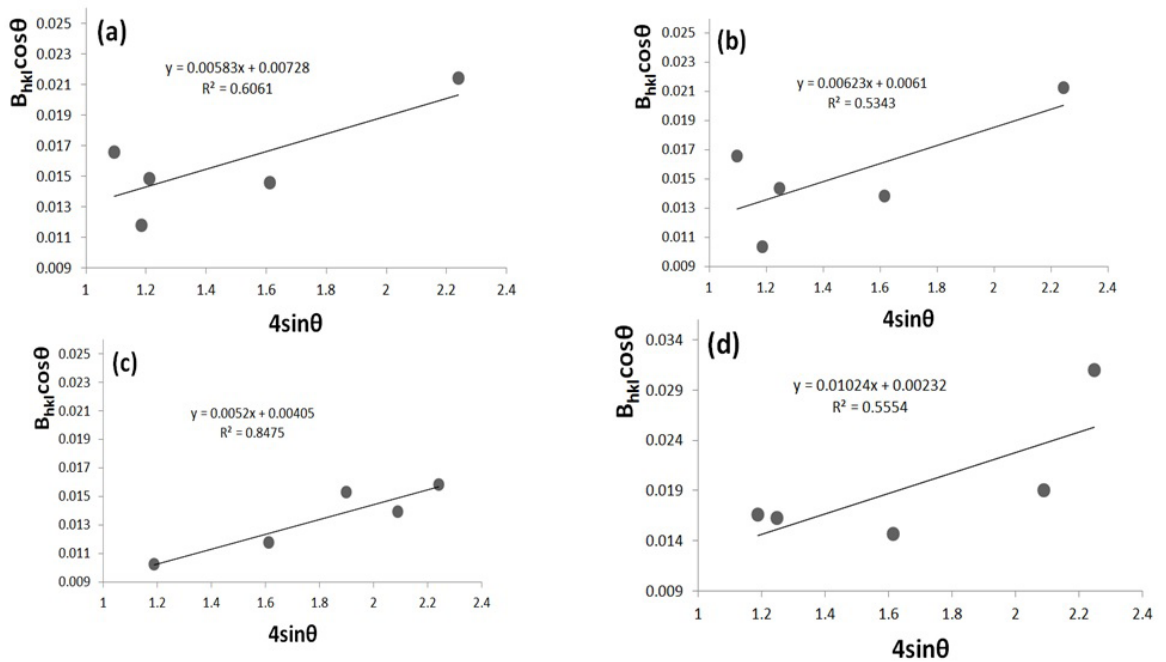


Fig. 2. W-H analysis of (a) ZnO, (b) Zn_{0.98}La_{0.02}Zr_{0.02}O, (c) Zn_{0.96}La_{0.04}Zr_{0.04}O, and (d) Zn_{0.94}La_{0.06}Zr_{0.06}O nanoparticles assuming UDM model.

uniform stress deformation model (USDM) and uniform deformation energy density model (UEDDM) should be adopted in the W-H approach. In the USDM, stress (σ) is related to the strain (ε) as σ=Y_{hkl} ε, where Y_{hkl} is young’s modulus and given by

$$Y_{hkl} = \frac{[h^2 + \frac{(h+2k)^2}{3} + \frac{(al)^2}{3}]^2}{s_{11}(h^2 + \frac{(h+2k)^2}{3}) + s_{33}(\frac{al}{c})^4 + (4s_{13} + s_{44})(h^2 + \frac{(h+2k)^2}{3})(al/c)^2} \quad (3)$$

For the hexagonal crystals, Where s₁₁, s₁₃, s₃₃, s₄₄ are the elastic compliances of ZnO with values of 7.858 x 10⁻¹², -2.206 x 10⁻¹², 6.940x10⁻¹², 23.57 x10⁻¹² m²N⁻¹, respectively³⁶. For La–Zr co-doped ZnO, Young’s modulus was calculated as ≈127 GPa. The modified form of W-H equation assuming USDM will be of the form

$$\beta_{hkl}Cos\theta = (k\lambda / D) + 4(\sigma Sin\theta) / Y_{hkl} \quad (4)$$

The USDM plots are shown in the Fig. 3 and the microstructural results are listed in Table 1. In the modified W–H equation based on a uniform deformation energy density model (UEDDM), the young modulus and strain are connected to the deformation energy density ‘u’ by u=ε²/2Y_{hkl} and the Eq. 4. can be modified according the energy and strain relation as

$$\beta_{hkl}Cos\theta = (k\lambda / D) + \left(4 \sin \theta (2u / Y_{hkl})^{1/2} \right) \quad (5)$$

From the plots of β_{hkl} Cosθ versus 4 sin θ(2u/Y_{hkl})^{1/2}, the anisotropic energy density (u) and the crystallite size can be estimated from the slope and the y-intercept of fitted line, respectively (see Fig. 4). The USDM results are collected in table 1. As can be seen from the table 1, the crystallite sizes calculated from various models are approximately same, which indicate that the inclusion of strain in various W–H models has a very small effect on the average crystallite size of ZnO nanoparticles.

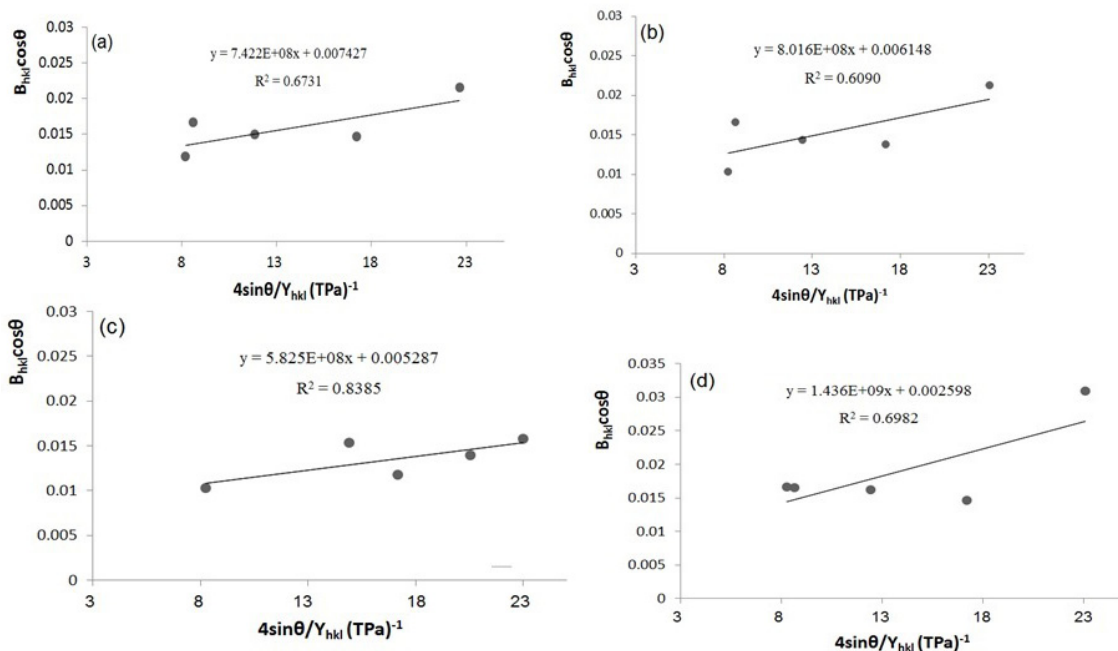


Fig. 3. The modified form of W-H analysis assuming USDM for (a) ZnO, (b) Zn_{0.98}La_{0.02}Zr_{0.02}O, (c) Zn_{0.96}La_{0.04}Zr_{0.04}O, and (d) Zn_{0.94}La_{0.06}Zr_{0.06}O nanoparticles.

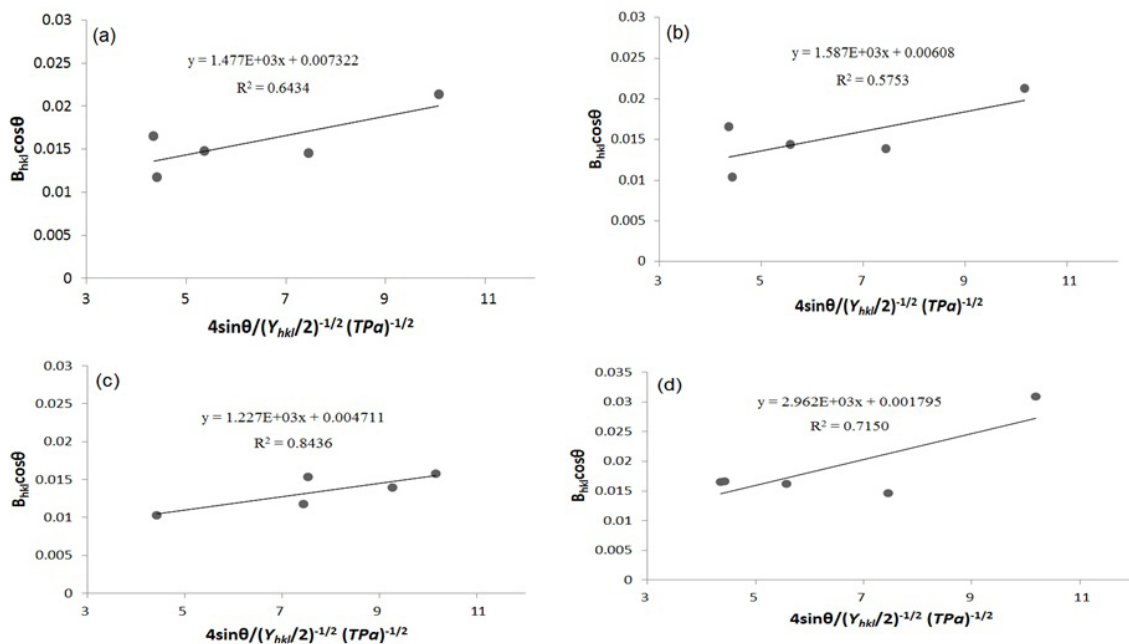


Fig. 4. The modified form of W-H analysis assuming UEDM for (a) ZnO, (b) Zn_{0.98}La_{0.02}Zr_{0.02}O, (c) Zn_{0.96}La_{0.04}Zr_{0.04}O, and (d) Zn_{0.94}La_{0.06}Zr_{0.06}O nanoparticles.

3.1.2. Size- Strain plot method

Size-strain plot method is another procedure to obtain the size-strain parameters. This method is constructed according to the following relation:

$$(d_{hkl} \beta_{hkl} \cos \theta)^2 = \frac{k}{D} (d_{hkl}^2 \beta_{hkl} \cos \theta) + \left(\frac{\epsilon}{2}\right)^2 \tag{6}$$

Where d_{hkl} is the lattice distance between the $\langle hkl \rangle$ planes and k is a constant and depends on the shape of the particles (for spherical particles $k = \frac{3}{4}$ ³⁷. Plot of $(d_{hkl}^2 \beta_{hkl} \cos \theta)^2$ versus $(d_{hkl}^2 \beta_{hkl} \cos \theta)^2$ were constructed for the all Bragg reflection of La–Zr co-doped ZnO nanoparticles (see Fig. 5). In this method less importance is given to data from reflections at high angles and the crystallite size distribution is

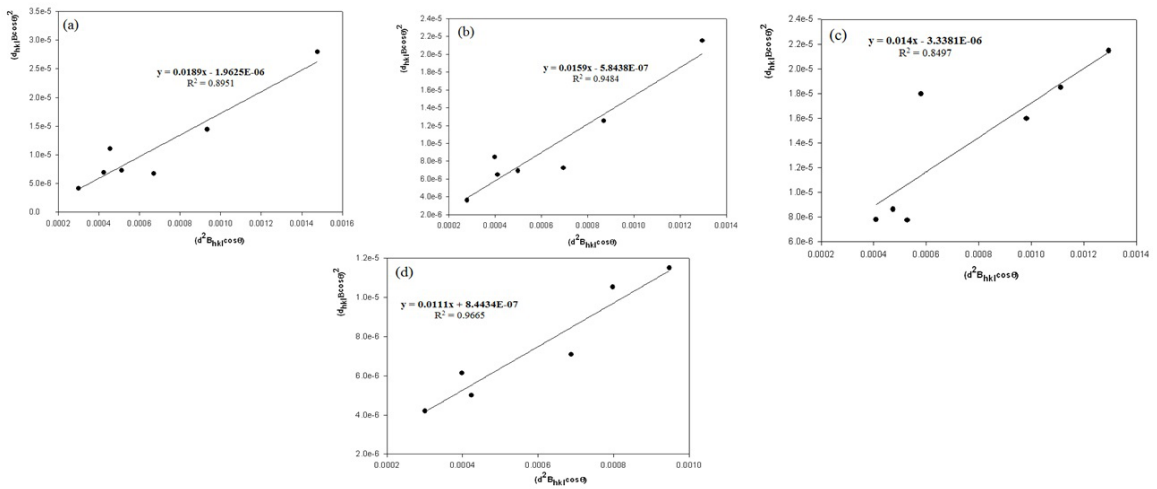


Fig. 5. The SSP plot of a) ZnO, (b) Zn_{0.98}La_{0.02}Zr_{0.02}O, (c) Zn_{0.96}La_{0.04}Zr_{0.04}O, and (d) Zn_{0.94}La_{0.06}Zr_{0.06}O nanoparticles.

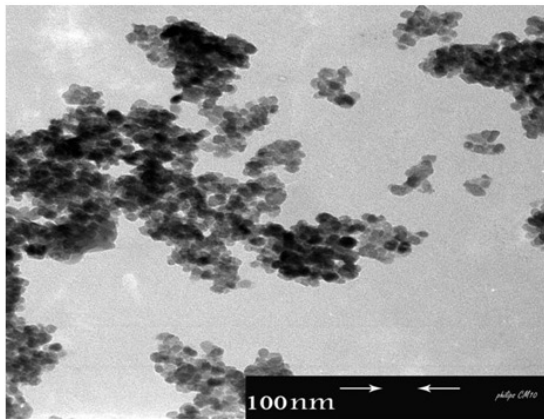


Fig. 6. TEM image and the particle size distribution of Zn_{0.94}La_{0.06}Zr_{0.06} nanoparticles.

described by a Lorentzian function and the strain by a Gaussian function³⁸. The strain is given from the root of the y-intercept and slop gives the particle size. The results attained from the SSP models are summarized in table 1. It was observed from the table 1 that the crystallite size of the La–Zr co-doped ZnO nanoparticles increased with increase in the La–Zr concentration. The polycrystalline show a larger value of ϵ indicating more strain on the lattice. The larger strain induced by the internal stress could lead to peak broadening.

3.2. TEM Method

The size and shape of the La–Zr co-doped ZnO nanoparticles could be best examined by TEM measurements. TEM image for the pure ZnO nanoparticles was obtained according to the method described elsewhere³⁰ about 35 nm. Fig. 6 displays a TEM image and particle size distribution of the Zn_{0.94}La_{0.06}Zr_{0.06} nanoparticles. As it's clear from the particle size distribution, the width of the nanoparticles

varies from 25 to 65nm with an average particle size of 50 nm. The average size obtained from the TEM analysis is in good agreement with the results of the USDM model.

4. Conclusions

The pure and La-Zr co-doped ZnO nanoparticles prepared by a simple sol-gel method were characterized by powder X-ray diffraction (XRD) and TEM measurement. XRD analysis shows that the prepared samples are in hexagonal wurtzite phase and free of any other Zr-La phase after doping. The size and strain contributions to line broadening were studied using the X-ray peak broadening analysis by the Williamson–Hall method and the size-strain plot (SSP) method. The crystallite sizes calculated from various models are approximately same, which indicate that the inclusion of strain has a very small effect on the average crystallite size of ZnO nanoparticles. The average particle size obtained from the TEM results is in good agreement with the results of the USDM model.

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