Structural, Morphological Properties of ZnO Nano Powders

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Abstract

Nano-powders of zinc oxide (ZnO) have been prepared by high energy ball-milling for various spans of mill time (1-18 h). The crystal structure, surface morphology of the prepared ZnO nano powder were characterized by X-ray diffraction and scanning electron microscope.

Keywords: zinc oxide ZnO; ball-milling, crystal structure, surface morphology.

1. INTRODUCTION:

In the current materials science research, great emphasis has been placed on the nano materials in order to study the unique physical properties.

The surface-to-bulk ratio for the nano materials is much greater than that for coarse materials, in nano materials, a large fraction of the atoms is present at the surface, and hence, the surface properties become paramount. Another important factor associated with the depth of charge region and the surface space is affected by the relation between gas adsorption and the particle size. These features of nano particles make the materials particularly good for their applications as gas sensors [1].

2. Experimental Procedure:

2.1. Preparation of ZnO Nano-powders

ZnO nano powders were prepared by high energy ball milling (HEBM) method. ZnO powders were milled by (Activator 2S, Russia) system during time from 1 to 18 h with step at 3 h. The milling conditions and the material compositions are summarized in Table.1

Table 1. Milling conditions in the high energy ball mill.

Raw materials	Materials of jars & balls	Ball to powder weight ratio	Volumes of jars	Ball diameters	Jar speed	Disc speed
ZnO	WC	1:20	80[ml]	10[mm]	320 [rpm]	640[rpm]

3. Results and Dissection :

3.1. Structural properties for prepared nano powders:

The X-ray diffraction patterns of ZnO nano powders were carried out using a STOE transmission Stadi-P diffractometer with monochromatic Cu $K_{\alpha 1}$ radiation (λ = 1:5406 Å). The wavelength was selected using an incident-beam curved-crystal germanium Ge(111) monochromator with a linear position-sensitive detector (PSD). The patterns were scanned over the angular range 20-50°(2θ) with a step length of the PSD of 0.5°(2θ) and a counting time of 30s per step.

The X-ray diffraction patterns for ZnO raw material and milled powders are shown in Figure 1. The X-ray patterns indicate that ZnO crystalize in Triclinic crystal structure with space group ($P6_3mc$) and cell parameters (a =b = 3.2494 A°, c= 5.2038 A°), angles between each pair of unit cell axes ($\alpha = \beta = 90°$, γ= 120°) [5].

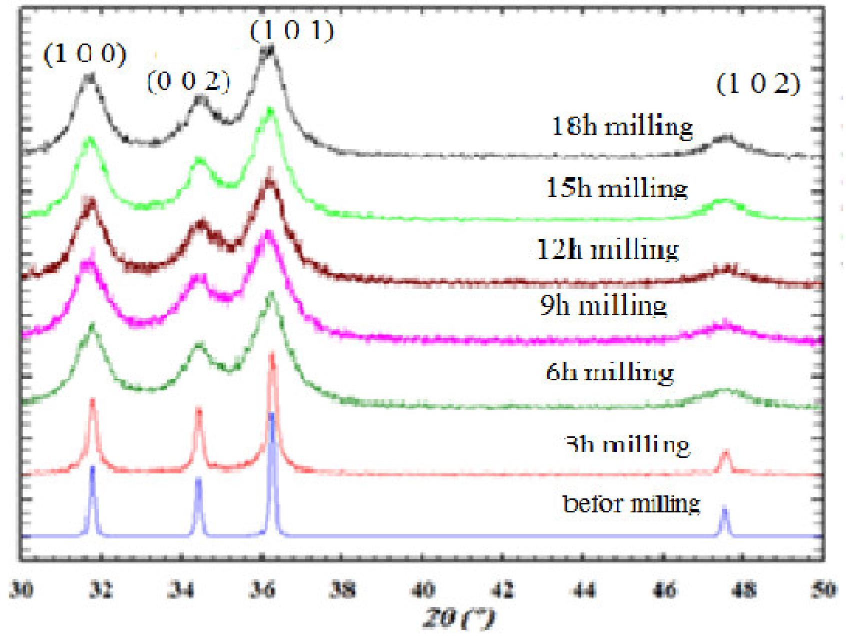


Figure 1. X- ray diffraction patterns at selected milling times for the ZnO sample milled at 320/640 rpm in comparison with raw material of ZnO

X-ray pattern of ZnO powder oxide before milling shows high intensity of Bragg peaks due to the micro powder oxide, with advance milling X-ray patterns show a gradual broadening and simultaneous decreasing in intensity; while that the position of the peaks have no changes and no new peaks appear just only low peaks disappearance which mean that the crystal structure of ZnO powder oxide doesn't change during the milling but the average of crystal size of ZnO powder is changing considerably.

the average dimension of the size of the crystals was measured by depending on the x-ray diffraction spectra for each peak using the Scherrer's Equation (diameter D) [6].

$$D = \frac{K \times \lambda}{\beta \times \cos\theta}$$

To measure the average dimension of crystal size by using Scherrer's Equation must identify the Integral Breath Estimated (β)

Table 2 :The Integral Breath Estimated for peaks of ZnO raw powders (β_0) and milling for various times (β1-18)

2θ°	β_0 [rad.]	β_3 [rad.]	β_6 [rad.]	β_9 [rad.]	β_{12}[rad.]	β_{15}[rad.]	β_{18}[rad.]
31.77	0.00291	0.0050	0.0113	0.0104	0.0131	0.0108	0.0116
34.42	0.00250	0.0032	0.0052	0.0064	0.0066	0.0063	0.0043
36.25	0.00305	0.0046	0.0104	0.0096	0.0073	0.0079	0.006
47.54	0.00323	0.0049	0.0063	0.0093	0.0102	0.0163	0.0110

Table 3: the values of the average dimension of the crystals size of ZnO raw powders (D_0) and milling for various times (D_{1-18}).

2θ°	D_0[nm]	D_3[nm]	D_6[nm]	D_9[nm]	D_{12}[nm]	D_{15}[nm]	D_{18}[nm]
31.77	55	32.04	14.13	15.399	12.23	14.83	13.8
34.42	64.31	49.90	30.77	25.203	24.4	25.60	37.51
36.25	53.15	35.24	15.68	16.881	22.35	20.51	27
47.54	52.12	34.36	40.8	18.098	16.52	10.33	15.3

The following figure shows the change in the average dimension of the crystals size of the zinc oxide powders calculated from the Scherrer's Equation for the first four levels of the crystalline (100), (002), (101), (102) As a function of milling time.

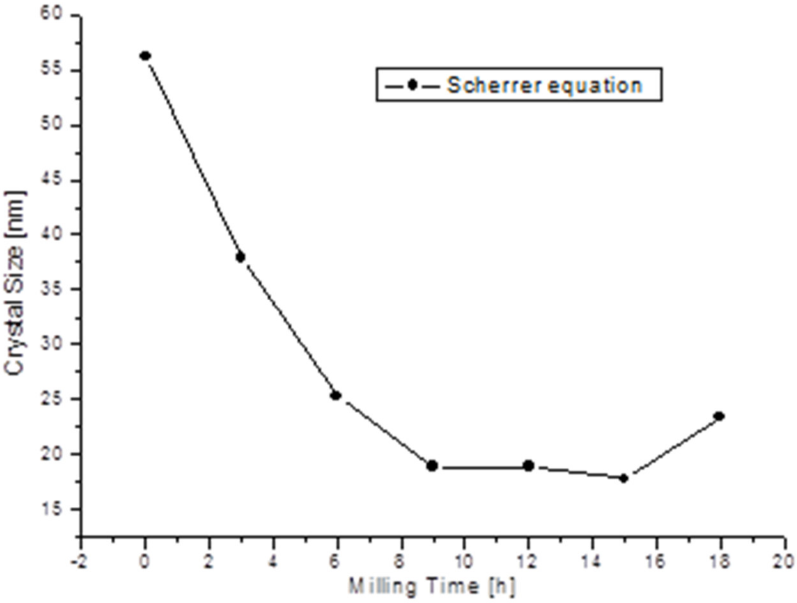
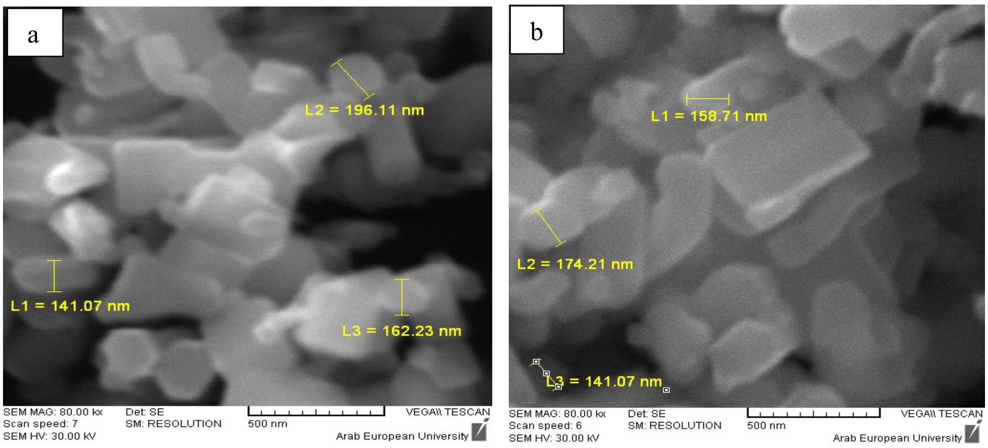


Figure 2. Size of ZnO nano crystal of ball-milled composites versus milling time.

Figure 2 shows the rapid decreasing of the average of the crystal size for ZnO powders to nano powders (from about 52 nm in raw material to about 17 nm after 9h milling) then become stable about (17nm) from (9-15h) We can say that the milling with HEBM produces nano crystallite This is done to obtain large numbers of nano dislocations and accompanying a stress fields, where the crystal lattice constants are changing then the periodic length in crystal lattice is reduced and the crystal size decreased. This is a agree with the broadening and the decreasing of intensity of X-ray diffraction peaks taken after milling to a specific time, and agree with the scientific papers [4,7]. So we can explain cause of stable the curve from (9-15h) the crystallites begin absorb stresses and pressures and this time that electrical forces between particles equal their weight forces.

3.2. Morphological properties for ZnO nano powders:

Scanning electron microscope SEM (TESCAN VIGA II XMU) was used for surface morphology study, figure 3 shows the SEM images of the nano powders of ZnO milled at different times:



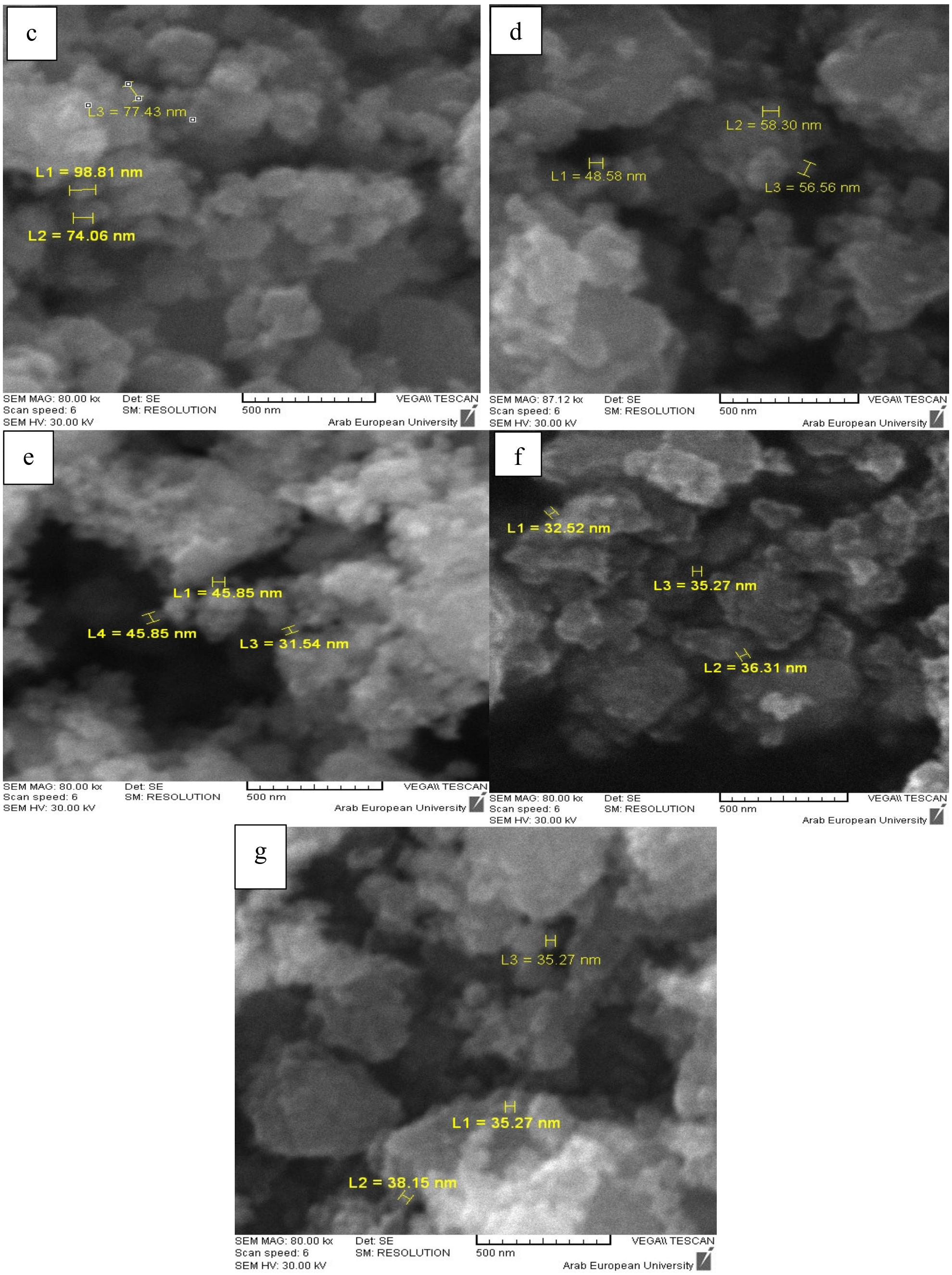


Figure 3. SEM images of ZnO powders during various periods milling: (a) 0 h, (b) 3 h, (c) 6 h, (d) 9 h, (e) 12 h, (f) 15 h, (g) 18 h.

As we see the powders consist of ZnO particles of various sizes ranging from about 166.47 nm to 34.7 nm tended to increase (about 36.23 nm) with increasing milling time. Considering the particle sizes shown in Figure 3 and the crystallite sizes calculated from X-ray patterns (Figure 2), most of the particles are agglomerates consisting of two or more crystallites.

Figure 4 shown the average particle size that taken by SEM as a function for milling time

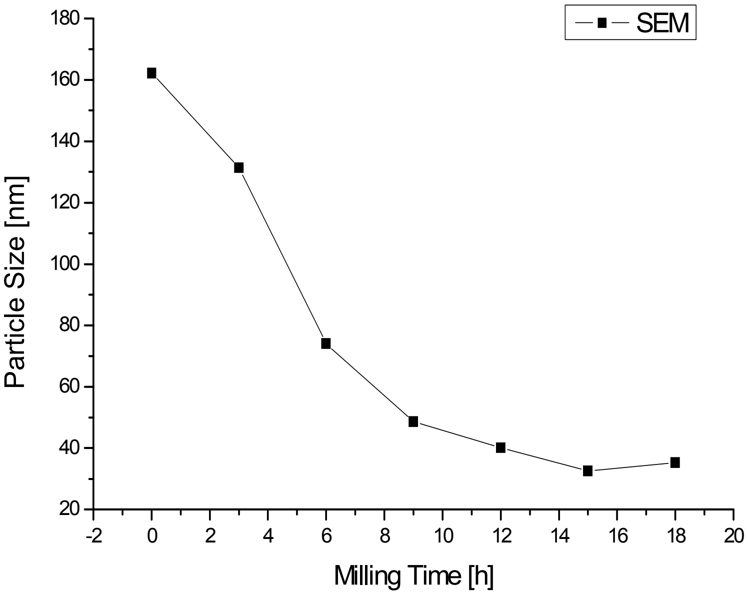


Figure 4. Size of ZnO nano particles of ball-milled composites versus milling time

We note from the figure 4 a gradual decreasing of the particle size with increasing milling time until 15 h then as increasing milling time as particle size, which is explained as: at the beginning of milling, particles respond, and disintegrate into softer particles, coupled with electrically charged surfaces are opposite because of direct cracking for these particles which exposure to stresses and pressures and the forces of cut affecting on areas of weakness in the granular structure, when arrived at specific nano scale dimensions the electrical forces between the nano particles become greater than the forces of their weights, because of the ability materials to conformation these particles are clustered together and formed larger than the previous one so we see it by SEM like particle one [8].

4. CONCLUSION

We can say that the milling with HEBM produces nano crystallite This is done to obtain large numbers of nano dislocations and accompanying a stress fields, where the crystal lattice constants are changing then the periodic length in crystal lattice is reduced and the crystal size decreased. As well as the SEM images showed a gradual decreasing of the particle size with increasing milling time until 15 h then as increasing milling time as particle size.

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