Preparation and characterization ZnO nanoparticles and study of morphology at high temperature

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Abstract

In context of this paper we prepare high purity powder ZnO nanostructures by chemical method at low temperature solution and study the effect off annealing at high temperature, ZnO nanoparticles have been successfully synthesized by chemical method at $0C^{o}$ solution. In this method, suddenly reaction is occurred between zinc acetate solution and sodium hydroxide solution at $0C^{o}$, annealing temperature of powder product surfactant plays an important role in morphological changes. The nanostructures have been characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM), differential scanning calorimetry(DSC) and UV-visible .analysis Effect of annealing temperatures on the morphology , structure and optical properties is discussed.

Key words
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تحضير و دراسة خواص المركب النانوي اوكسيد الزنك ودراسة خواصة السطحيه عند درجات حرارة عالية

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الخلاصة

في سياق هذا البحث تم تحضير حبيبات بإحجام النانومتر لمادة أكسيد الزنك الشبه الموصلة وبنقاوة عالية وتم استخدام الطريقة الكيميائية في التحضير عند درجات الحرارة المنخفضة (الصفر المئوي) ويكون إضافة محلول خلات الزنك إلى محلول هيدروكسيد الصوديوم بشكل سريع وفجائي . وتم دراسة الخواص التركيبية لهذه المادة بعد ان تم تعريضها لدرجات الحرارة العالية ودراسة التغيرات التركيبية بواسطة طيف الحيود للأشعة السينية وكذلك باستخدام المكروسكوب الالكتروني ودرست نقاوة المسحوق المحضر باستخدام جهاز التحليل الحراري التفاضلي .وكذلك دراسةخواص الامتصاص البصري باستخدام جهاز امتصاص الأشعة فوق البنفسجية .

Interdiction

ZnO nanoparticles, as an n-type semiconductor with a wide band gap II-VI compound semiconductor, have a stable wurtzite structure with lattice spacing a= 0.325 nm and c= 0.525nm [1], it have been attracting much attention in research for their potential application in optoelectronic devices, UV lasers, solar cells, gas sensors [2], hydrogen storage devices, optical waveguides [3], light emitting diodes and catalysts .using ZnO as an active channel in invisible thin film transistors [4]. ZnO nanoparticles have a large excitation binding energy (60 MeV) [5], to produced nanoparticles large number of methods, We can used like magnetic liquids, metal polymer nanocomposite, semiconductors, colloidal systems [6], thermal evaporation of oxide powder [7]. In this article we report a simple chemical method to synthesis

ZnO nanoparticles and study the change of morphology and grain size in different temperature annealing .Among control of the particle shape is one main concern for nanostructured material synthesis because electrical and optical properties of nanomaterials depend on both size and shape of the particles. Therefore, it is desired to synthesize nanomaterial in a controllable shape and size by a simple approach. Among the various chemicals methods to prepare ZnO, these methods depended on attraction same numbers of ions in solution. But it was still difficult to control the size and shape of the nanoparticles. Recently one step and multistep synthesis methods of metal and oxide nanoparticals are presented including both bottom –up and top-down procedures, these methods called polyol process and developed in our laboratory, is a soft chemistry method which allows to prepare oxide or metal nanoparticales with controlled size and shape [8, 9,10,11].

Experimental Details

1- Chemicals

Materials used in this experiment are analytical grade without further purification. zinc acetate dehydrate $(CH_3COO)2Zn.2H_2O$ (ANALAR HOPKIN and WILLAMS ESSEX ENGLAND) and sodium hydroxide NaOH (KLAUS ENGLERT EMCLAB GERMANY) and 2- propanol alcohol, preparation ZnO nanoparticles at low temperature chemical reaction without any capping agent and the chemical reaction involved is as follows:

$(CH_3COO)2Zn.2H_2O + NaOH \longrightarrow ZnO + (CH_3COO).Na$

In typical procedure, 0.2 M of zinc acetate dehydrate was dissolved in 100 ml 2-propanol alcohol and stirred with heating 50°C - 70°C for 60 minutes .The solution was cooled in 0°C and continues stirred for 60 minutes. 0.2 M sodium hydroxide solution was prepared using stirred with heating at 60°C for 90 munities. This solution adds suddenly to zinc acetate solution at 0ºC. The mixture was placed in water path. The white solid produced was subsequently separate by centrifuge washed with 2-propanol alcohol and dried in air at room temperature.

2- Annealing

The white powder produced divided in to two part, the first part (sample a) put in R.F furnace at 120 ºC for about one hour. The second part (sample b) put at 400°C for three hour. Films have been prepared by using layer by layer method on glass substrates. This samples have been annealed at (120, 400) ºC.

3- Characterization

Fig.1. shows the X-ray diffraction (XRD) patterns of ZnO nanoparticles with different morphologies. The two samples (a,b) structures have similar XRD patterns, except for relative peak intensities, due to their random orientation. Both of XRD patterns can be indexed as the pure hexagonal wurtzite ZnO structure with (100) plane. The Lattice parameter has been calculated for two samples and found a=2.82351°A for sample(b) and =4.08113 °A for sample (a). The values lattice constant sample(b) is consistent with previously reported data sheet XRD,because no diffraction peaks were observed from other impurities in the XRD patterns, we can be conclude that the pure hexagonal-phase with wurtzite structure with unit cell parameters were synthesized through this fast and simple chemicals method at 0°C, The average practical size can be estimated from the extrapolation of the plot shown in figure 2. Sample (a) it can be that changes in lattice parameter for sample (a) because of impurity in this sample lead to observe the diffraction peaks in the XRD patterns [14].

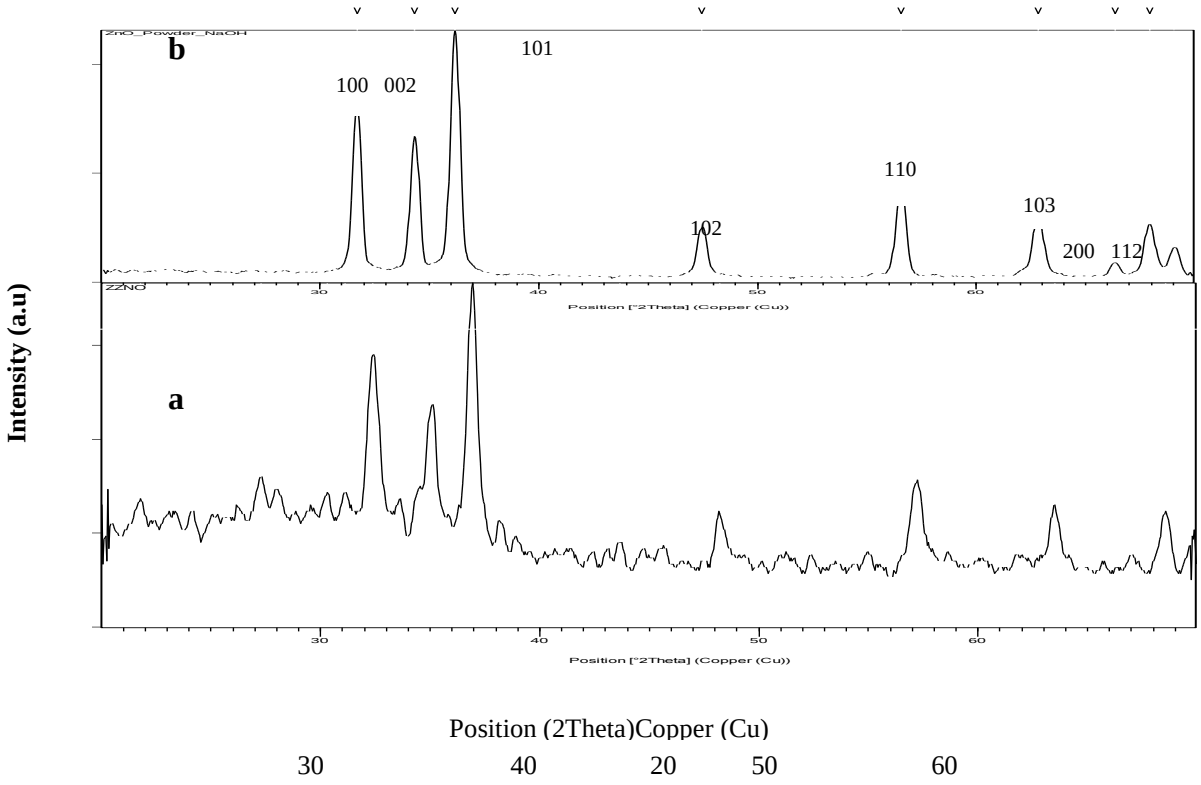
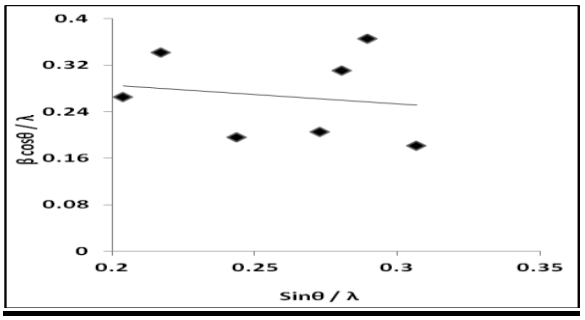


Fig 1: The XRD pattern of ZnO nanoparticales of samples a and b

Sample (a)



Sample(b)

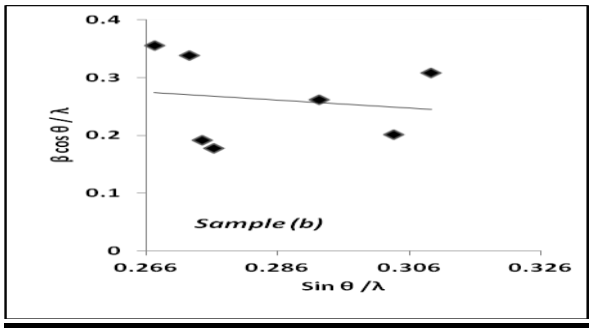


***Fig. 2: β cosθ /λ vs. Sinθ/λ of ZnO nanoparticales for samples a* and b**

For sample (b) it can be determined the value of purity ZnO nanoparticals (97.69 mol %) using thermal analyses Fig.3 by differential scanning calorimetry model (TA instrument Universal Analysis Q20) [12,13]. Crystallite size of the prepared sample was analyzed using XRD technique Sample were preferably orientated along different planes. The particle size of the ZnO powder sample was calculated using Debye- Scherrer formula [15] given by:

$\beta\cos\theta/\lambda=1/D+(\tau.\sin\theta)/\lambda$

where D is the average particle size of the crystallites [16], λ is the wavelength of CuKα line, ß is full width at high maximum (FWHM), θ is the diffraction angle. The particle size was determined for sample a & b and found to by 35nm and 37nm, respectively. The average lattice strain (E str) of the ZnO nanoparticles was calculated using Stokes – Wilson equation given by:

$E\ str= ß /4\tan\theta$

and The dislocation density (d) was also calculated from the relation [17].

$d = 15E /aD$

The value of average lattice strain and the dislocation density (lines/m) were listed in the Table (1).

Table (1):. Represent the structure for ZnO nanoparticales

	h k l	Lattice strain	Dislocation Density $*10^{-9}$	Lattice parameter a (A°)
Sample (a)	1 0 0	0.42	0.0651	2.763
	0 0 2	0.3112	0.111	1.278
	1 0 1	0.3318	0.13	1.088
Sample (b)	1 0 0	0.37	0.056	2.823
	0 0 2	0.358	0.1112	1.305
	1 0 1	0.3765	0.086	1.755

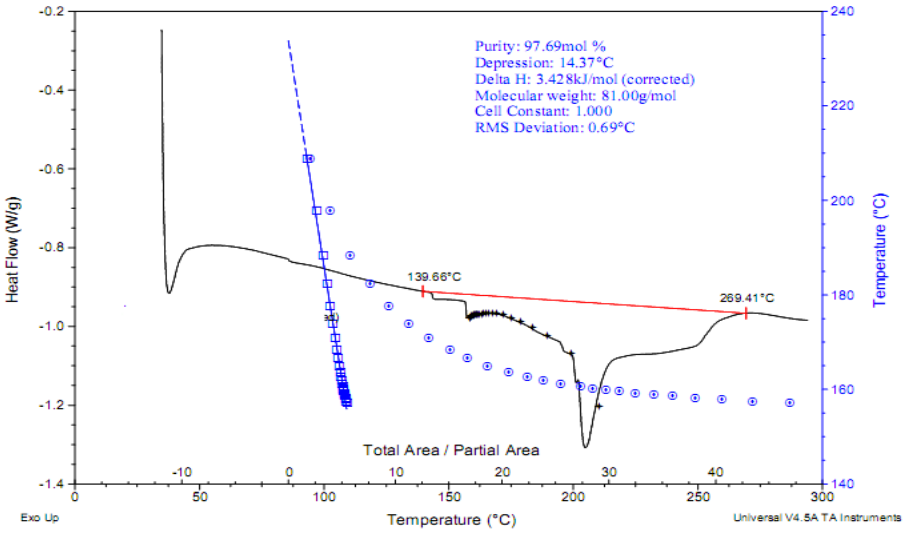


***Fig. 3: Differential scanning calorimetry of ZnO nanoparticales for* sample (b)**

SEM micrographs model (Inspect^{Tm} S50 with Image processor smart SCAN^{TM} scan strategy) for powders of the samples (a_1,a_2,b_1,b_2) and films (a_3,b_3) are shown in Fig.4 It is clear that ZnO powders show an homogeneities regarding particle size distribution, the films preparation by using layer by layer method on glass substrates micrograph we can see in homogeneities distribution practical's and big oxygen vacancies , that lead to a perpetration thin films in this method and high temperature annealing with long time its product increasing in practical's size and more defects like oxygen vacancies and zinc interstitials.

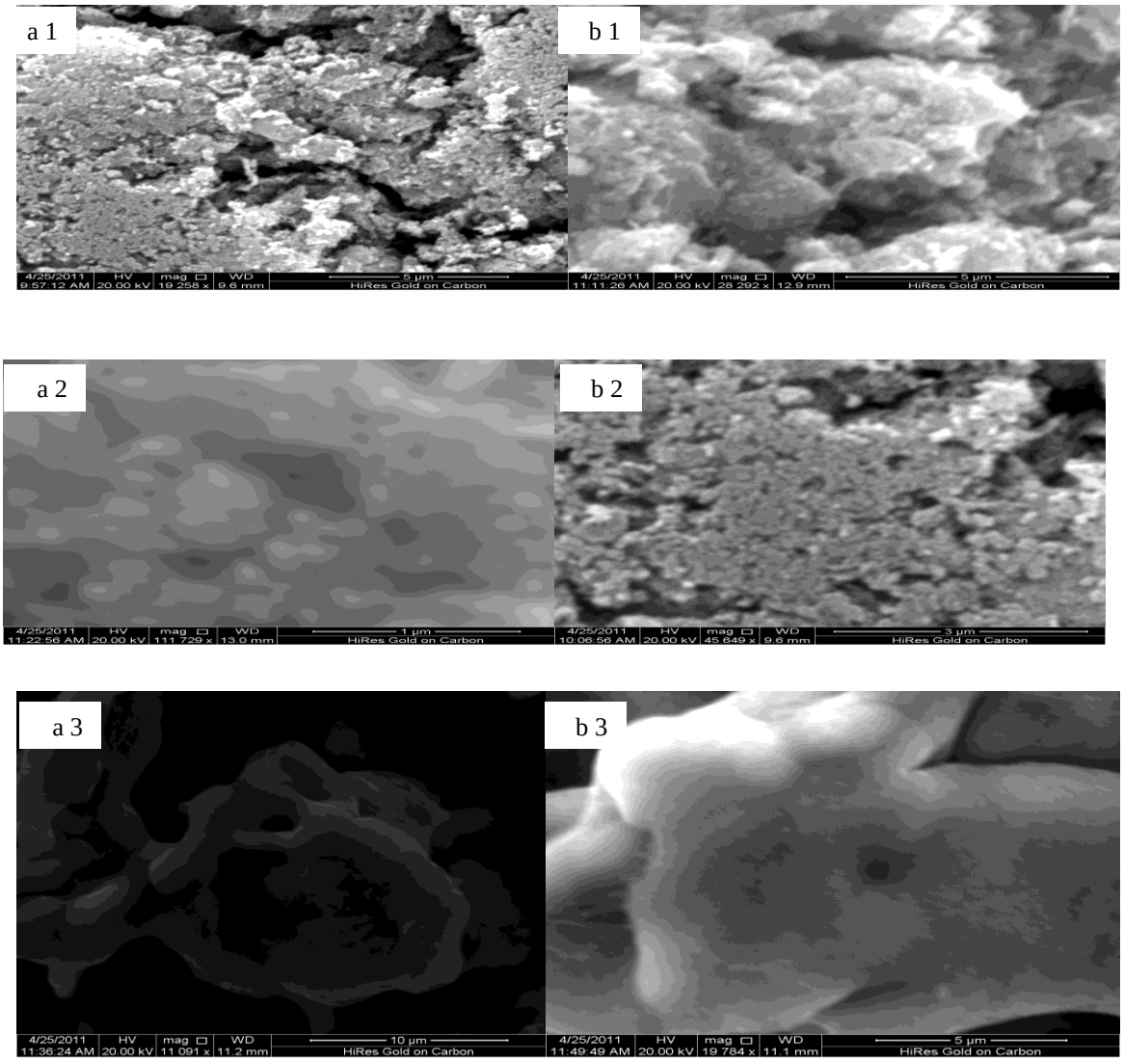


Fig. 4: Scanning electron microscope micrographs samples for powders (a_1,a_2,b_1,b_2) and films (a_3,b_3)

4-UV- Visible spectra

High crystalline qualities for ZnO, Fig.5 shows a dominated UV–vis absorption spectra of the prepared ZnO particles. A strong UV absorption is characteristic for all measured samples, which attains a plateau λ_{max} =364nm (E_g =3.3 ev). The crystal quality of the deposited ZnO is an important factor for the high UV absorption and found to be increase the crystal quality (less structural defects and impurities such as zinc interstitials) may enhance the intensity of UV[18]. In our case, increasing intensity, which indicates that the grown nanostructures are good in crystal quality, high purity and exhibiting a good optical property [19, 20].

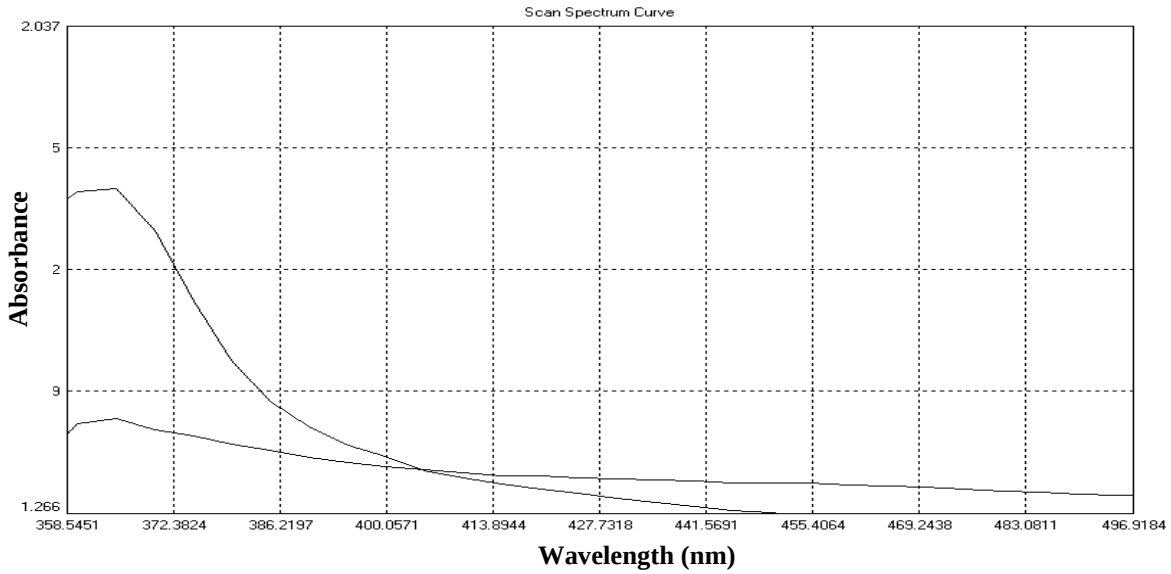


Fig. 5 : U-V visible spectra ZnO nanoparticales to samples a and b

Conclusions

High purity Wartizite ZnO nanoparticles had been prepared using chemical reaction between Zinc acetate and sodium hydroxide solutions. X-Ray diffraction (XRD) ,differential scanning calriomatry (DSC) ,scanning electron microscope (SEM) and U-V visible spectroscopy were used to characterize the structure, and optical properties, it is found that particles size increase and more defects like oxygen vacancies when preparation layer by layer thin films and high temperature annealing with long time .

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