Sintering and Grain Growth in Nanocrystalline ZnO Particles

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Abstract: **Nano crystalline ZnO particles were synthesized using a wet chemical synthesis route. The synthesized particles were pressed and sintered at higher temperature (at 700^{o}C for 2 h) in order to study the effect of sintering on grain growth in these nano particles. The as dried and the sintered particles were characterized by X-ray diffraction and FESEM. A noticeable change in the structural and morphological parameters was observed between as dried and sintered particles. The X-ray diffraction patterns showed highly intense peaks for sintered particles as compared with the as dried ones showing increase in crystallite size after sintering. FESEM images also confirmed the grain growth due to sintering. So it can be said that ZnO nano particles of varying size as desired can be synthesized using this low cost wet chemical synthesis technique for its potential application in various fields.**

Keywords: ***ZnO nano particles; Wet chemical synthesis route; Crystallite size; Sintering; Grain growth***

I. INTRODUCTION

ZnO is an *II-VI* compound semiconductor having a stable wurtzite structure of lattice constants a = 3.25 Å and c = 5.2 Å; their ratio c/a ~ 1.60 is close to the value for an ideal hexagonal cell c/a = 1.633 [1]. It has a direct and wide band gap (3.37 eV) in the near-UV spectral region [2], and a large free-exciton binding energy of 60 meV (sufficiently larger than the thermal energy at room temperature (26 meV)) [2], which makes the excitonic emission possible at or even above room temperature [3]. The above physical properties of ZnO has attracted intensive research efforts in numerous applications, such as semiconductor devices [4-5], transparent conductors [6], solar cell [7], varistors [8], liquid-crystal displays [9], spintronic applications [10], gas sensors [11], etc. Due to the above cited applications and hence the motivation of device miniaturization, large effort has been focused on the synthesis, characterization and device applications of ZnO nanomaterials. These nanostructures of ZnO, which can be deposited even at/ near RT, have been successfully synthesized via a variety of methods including sol-gel route, wet chemical synthesis technique, spray pyrolysis, and so on [12-13]. During synthesis various factors like concentration of precursor materials, annealing, nature of substrate and so on affects the properties of the ZnO material.

In this paper ZnO nanoparticles have been synthesized using a wet chemical synthesis route. The grown particles were sintered at higher temperature and the variation in physical properties of the sintered particles as compared with the as dried particles was investigated.

II. EXPERIMENTAL PROCEDURE

Nanocrystalline zinc oxide particles (0.025M) were prepared using a wet chemical synthesis route using zinc acetate [Zn $(CH_3CO_2)_2{\cdot}2H_2O$] as precursor material. The precursor materials were dissolved in 2-methoxyethanol through continuous stirring. Mono-ethanolamine (MEA) was added drop by drop into the precursor solution in 1:1 volume ratio through continuous stirring. The obtained solution was heated at 80 °C and cooled to form a gel. The gel was dried in a vacuum oven kept at 80 °C overnight to form powder. The powder so obtained, termed 'as dried particles', was crushed in a mortar pestle and sintered at 700 °C for 2 h in air.

The crystalline structure of the particles was determined by X-ray diffraction (Rigaku Ultima III Xray diffractometer) using Cu Kα radiation. The microstructure was characterized by field emission scanning electron microscope (FESEM) (CARL ZEISS SUPRA-40).

III. RESULTS AND DISCUSSION

A. Structural Characterization

Fig. 1 (a) and (b) shows the X-ray diffraction pattern of as dried and sintered ZnO nano particles.

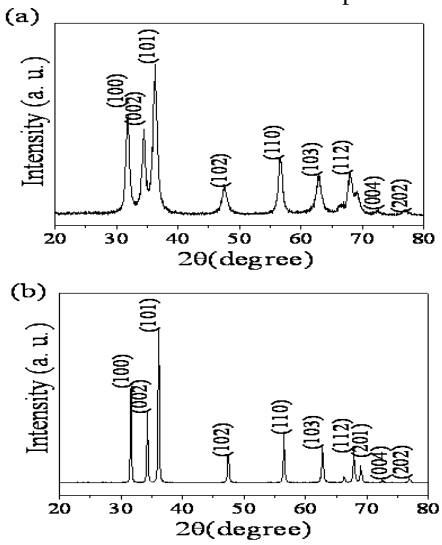

Fig. 1: XRD pattern of (a) as dried and (b) sintered ZnO nanoparticles

As reflected from Fig. 1 (a) and (b) both the as dried and sintered particles show polycrystalline hexagonal ZnO structures with prominent peaks at 31.72°, 34.28°, 36.20°, 47.50°, 56.46°, 62.85°, 67.76°, 72.45° and 76.71° corresponding to (100), (002), (101), (102), (110), (103), (112), (004) and (202) planes respectively. However, the intensity of peaks for sintered particles is stronger and sharper compared to as dried particles. Hence the FWHM of the sintered particles is smaller than that of as dried particles, which indicates the improvement in crystalline quality of the particles [14]. Some more peaks such as (201) corresponding to 69.04° are prominent in the sintered particles which may be attributed to the requirement of higher energy for these planes to be developed unlike (002) plane in ZnO which has lowest free energy [15]. The average crystallite size is found to increase in a large extent which can also be seen from the FESEM micrographs discussed in the following section.

B. Micro- structural Characterization

Fig. 2 (a) and (b) shows the scanning electron micrograph of the as dried and sintered ZnO nano particles.

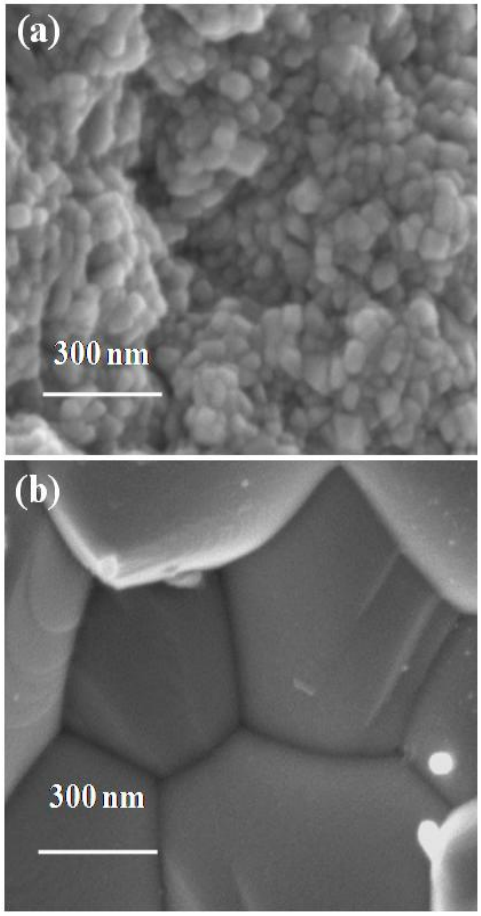

Fig. 2: FESEM micrographs of (a) as dried and (b) sintered ZnO nanoparticles

As observed from the Fig. 2 (a) the as dried particles are very small in size with disorder arrangement. The average grain size as estimated from the micrograph is ~50 nm. On sintering, as shown in Fig. 2 (b) individual grains grow much larger with estimated average grain size ~300 nm. This observation is in well agreement with the decrease of FWHM as observed in XRD pattern. The microstructure becomes denser with decrease in grain boundary density and formation of large and well defined grains. This is in agreement with the literature [15-16]. As reported by Wang et al. [15] at higher annealing temperature the activation energy of the atoms is sufficient to occupy their correct sites in the crystal structure. In our study it is also demonstrated that after sintering the individual grains acquire higher energy, redistributed themselves and joined together into large and well oriented grains.

CONCLUSION

In summary, nanocrystalline ZnO particles have been synthesized using a wet chemical synthesis route. The synthesized particles were sintered at temperature 700°C for 2 h. XRD pattern of the particles showed polycrystalline behavior with lower FWHM and hence larger crystallite size for sintered particles as compared to the as dried ones. FESEM images revealed the grain growth due to sintering. The obtained results suggested the tailoring of size of the ZnO nano particles as desired for its versatile applications in various fields.

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