

Surface Topology of Zinc Oxide Powder Prepared by High Temperature Sol Gel Combustion Method

Vaishali T. Salunke¹, P. B. Buchade², A. D. Shaligram³ and R. Y. Borse⁴

M. S. G. Arts, Science and Commerce College Malegaon Camp, Dist. Nasik, Maharashtra, India¹

Aabasaheb Garware College, Pune, India²

Department of Electronic Science, SPPU Pune, India³

M. J. M. Arts, Commerce, and Science College, Karanjali (Peth), Nasik⁴

Abstract: ZnO is most widely and most attractive element in today's research. ZnO powder is not available naturally. The ZnO powder was prepared by different methods. Here in this paper the preparation of powder was discussed. The powder was prepared by high temperature sol gel combustion method. Here in this study selected temperature was $500^{\circ}\text{C} \pm 10^{\circ}\text{C}$. to form the pure ZnO powder the synthesized powder was sintered at $650^{\circ}\text{C} \pm 10^{\circ}\text{C}$. After sintering the white ZnO powder was formed with amorphous in nature. This method was easy and high yield. The powder was characterized by using XRD and SEM. From XRD grain size was calculated. Using SEM the particle size, specific surface area, surface roughness and the surface morphology was studied.

Keywords: ZnO, combustion, sol gel, XRD, SEM

I. INTRODUCTION

Semiconducting oxides like ZnO materials have tremendously studied for various different applications such as gas sensors, solar cells, in photo catalytic materials, used in cosmetics like talcum powder, in medicine, as a immunity supplement and many more. ZnO has highly stable chemically, low cost, nontoxic and very good flexibility in fabrication. To synthesis the ZnO nanomaterial powder different methods like vapour deposition, oxidation, sputtering, and pulse laser deposition, sol gel, hydrothermal, co-precipitation, wet chemical etc. methods are used.[1] Each has their advantages and disadvantages. In this study high temperature solution combustion method was used to prepare the ZnO powder. Zinc nitrate hexahydrate as a source and dextrose as a decomposer was used as the basic precursor for zinc oxide powder preparation. The powder has good yield and easy to formation.

The wide bandgap of ZnO i.e. 3.4eV and having large binding energy of 60meV at room temperature is the main reason that ZnO can be used for wide applications. ZnO has chemically and thermally stable type f semiconducting material with hexagonal structure. ZnO microstructure/nanostructure having different shapes, size, structural and morphological characteristics are formed by using different synthesis methods. To stabilise these parameter controlling on synthesis method would be required. Synthesis of Nano rods, nano belts, and flower like structure are mostly focused by many researchers. [2,3]. The high temperature sol gel combustion method is easy to fabricate, require very less time means within 15 min. powder was synthesized. The structure of the powder is hexagonal in shape, and amorphous in nature.

II. EXPERIMENTAL

2.1 Synthesis of ZnO powder

Zinc oxide macro/nano structured powder was prepared high temperature sol gel combustion technique [4] as shown flowchart of preparation of powder in fig 1. The initial materials are Zinc nitrate and Dextrose. 20gm of zinc nitrate and 7.2 gm of dextrose were dissolved in pure water and beaker was placed on a hot plate for 20-25 minutes and wait for formation of the solution like a gel as solution dehydrates to form a deposition like a gel shown in fig 2(a). Then the beaker was placed in a preheated muffle furnace at $500^{\circ}\text{C} \pm 10^{\circ}\text{C}$ temperature as in fig 2(b). The solution boils, ignites with a flame and the entire reaction was completed within 10 minutes. The powder is amorphous in nature

fig 2(c). Then the powder was calcinated at $650^{\circ}\text{C} \pm 10^{\circ}\text{C}$.to get ZnO powder which is white in colour fig 2(d). The XRD pattern of this revels the formation of ZnO.

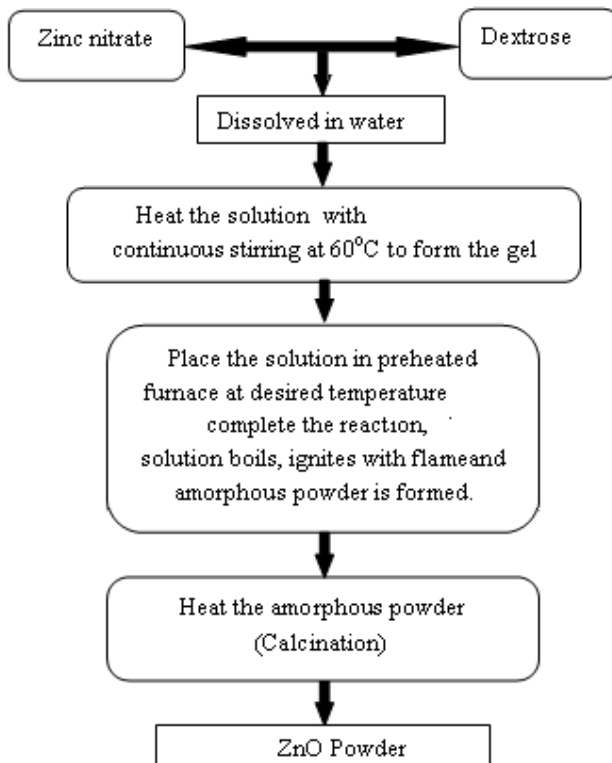


Figure 1: Schematic diagram of synthesis of ZnO powder

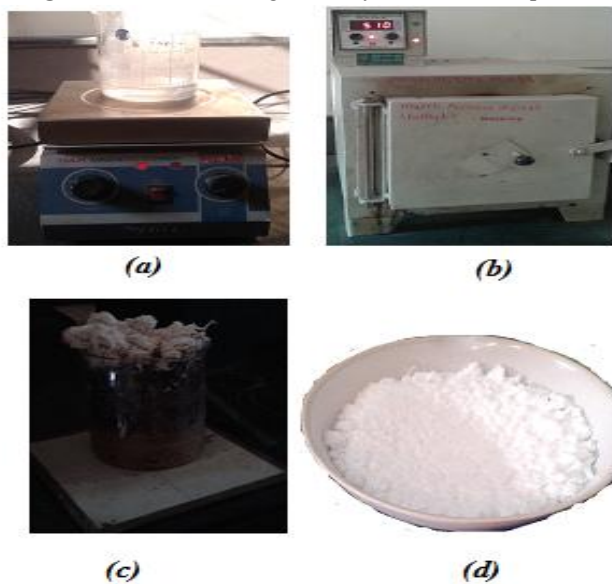


Figure 2: steps of preparation of ZnO powder

The basic principle of the method is the decomposition of an oxidizer in the presence of a fuel. The synthesized powder was then characterized using XRD and SEM

III. RESULT

3.1 XRD of Synthesized Powder

To analysed the powder extracted from the procedure the XRD(X-ray : Cu / 40 kV / 40 mA Goniometer : Ultima IV, Scintillation counter, Rigaku diffractometer was used. The peaks of the XRD were observed between 20° and 80°(2θ) range using CuKα radiation. From the XRD peaks in fig 3 the synthesized powder was polycrystalline in nature with a hexagonal wurtzite type crystal structure and the growth orientation in c-axis at (101) plane. The experimental (observed from X-ray data) *d* values are compared with the standard data [JCPDS/ASTM data files (JCPDS 36- 1451)] [5][7]. Thepeaks (100), (002), (101), (102), (110), (103) and (112) shows the hexagonal ZnO crystal structure similar to Joseph et al. [6].

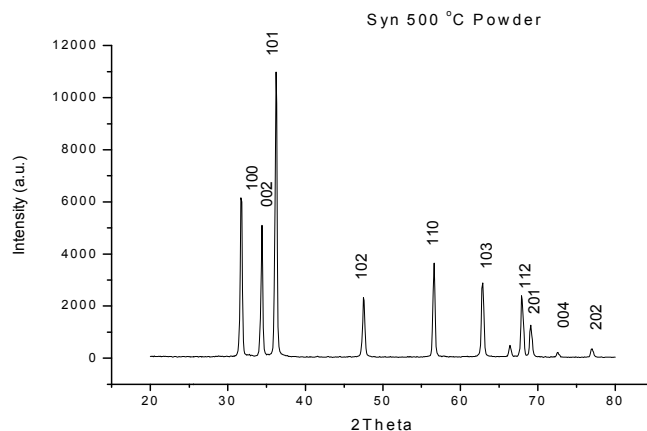


Figure 3: XRD patterns of synthesised ZnO powder

The crystallite size was determined using Scherrer's formula

$$D = \frac{0.94\lambda}{\beta \cos\theta}$$

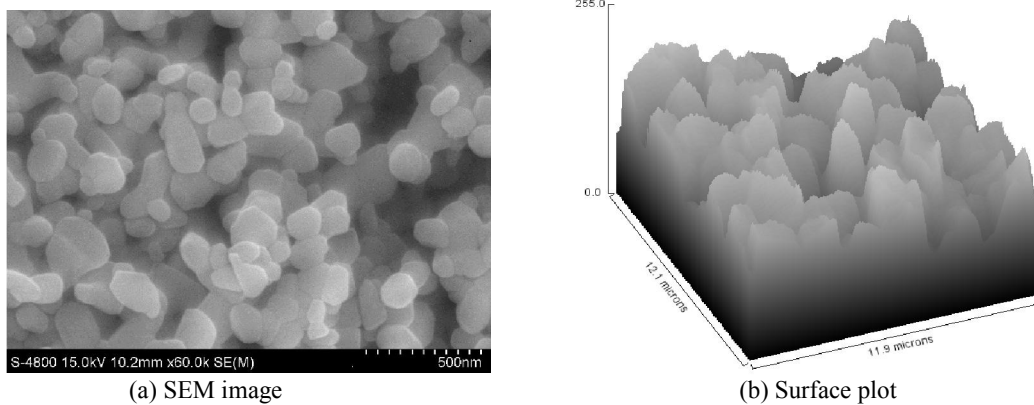
Where where, β = Full angular width of diffraction peak at the at half maxima peak intensity.

λ = wavelength of X-radiation.

The average crystalline size was 27.91nm.

3.2 SEM analysis of Synthesized Powder

Fig 4 (a) shows the scanning electron microscopy of ZnO powder. Morphology of ZnO particles was observed by scanning electron microscope, [SEM-Model JOEL 6300(LA) Germany].



(a) SEM image

(b) Surface plot

Figure 4: (a) SEM image of ZnO synthesized powder

The particles are agglomerated. Particles are circular in nature. Mean area of the particle is $7.038E4$ in nm^2 and the diameter of the particle is around 300nm i.e. radius of the particle is approximately 150nm using SEM. Fig 4(b) shows the surface plot of the powder plotted using imageJ software. From this plot the surface roughness and particles are under and overlay one on another particle is clearly seen. Also the voids or porosity in particles arrangement is clearly seen so this powder can be used for gas and humidity sensing.

V. CONCLUSION

The high temperature sol gel combustion method was the easy, time saving, cost effective to prepare the ZnO nano powder. The prepared powder is polycrystalline in nature and the gap or porosity in nature.

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