

Chapter 8

Synthesis and Characterization of Hexagonal Shaped Nanocrystalline Zinc Oxide Powders

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ABSTRACT

Nanocrystalline ZnO powders are synthesized using a quick, simple and inexpensive combustion method. Glycine and zinc nitrate, used as fuel and oxidant/cation sources respectively, were mixed together at room temperature to form a slurry or gel. A series of experiments were performed to synthesize nanoparticles for different fuel to oxidant ratios. The rate of heating and stirring greatly influenced the combustion process. The as-synthesized powder was heat treated at elevated temperatures for varying time to remove adsorbed impurities from the surface of the ZnO nanoparticles. Both the as-synthesized and heat treated powders were characterized using a variety of analytical techniques. The hexagonal wurtzite phase of the ZnO powder was revealed from x-ray diffraction measurements. A decrease in the x-ray diffraction density (D_x) and the bulk density (D) resulted in an increase in percentage porosity (%P) of as-synthesized and heat treated ZnO powders. The range of fuel to oxidant ratios used in the present work indicates that they had a strong influence on the grain size of ZnO powders. The average grain size estimated by using the Scherrer formula indicated an approximate value of 6-21 nm for the as-synthesized and heat treated samples.

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1. INTRODUCTION

Materials at the micrometer scale generally show physical and chemical properties similar to that of the bulk material. However at the nanoscale materials can exhibit the physical and chemical properties which are different from that of the bulk form. Improved sinterability, higher catalytic activity and other remarkable properties are features of the nanoscale due to their large surface area, nanocrystallite size and different surface properties such as surface defects (Hwang & Wu, 2004). Zinc oxide (ZnO) is a piezoelectric, dielectric, transparent, II–VI compound semiconductor with a direct band gap of about 3.37 eV at room temperature and a large exciton binding energy of about 60 meV, which is considerably larger than the thermal energy at room temperature (26 meV). As a consequence of these properties, zinc oxide is used extensively in various applications, such as transparent conductors, photonic devices, surface acoustic devices, solar cell windows, and gas sensors. ZnO is an environmentally friendly material, which is advantageous especially for bio-applications, such as bio-imaging and cancer detection (Kim & Manzoor, 2006; Chen, Gao, Ruan, & Shi, 2005; Vorob'ev, 2005; Umar, Rahman, Kim, & Hahn, 2007; Wama, Utiyama, Plashnitsa, & Miura, 2007; Wu, Tok, Boey, Zeng, & Zhang, 2006). Furthermore, ZnO is the hardest of the II-VI semiconductors due to the higher melting point (2248 K) and a number of experiments confirmed that ZnO is very resistive to high energy radiation, making it an attractive candidate for space applications. Due to its exceptional physical and chemical qualities, it can also be applied extensively to information technology, bio technology and environmental technology and next generation technologies (Hughes, 2006; Kwon, Kim, Lim, & Shim, 2002). ZnO normally forms hexagonal wurtzite crystal structure (6mm point group symmetry) with $a=3.25 \text{ \AA}$ and $c=5.12 \text{ \AA}$. The zinc atoms are tetrahedrally coordinated to four oxygen atoms. The oxygen anions occupy

the octahedral sites. The structure is non-centrosymmetric, which gives rise to polarization and piezoelectric properties (Lee, 2006).

Various kinds of methods for synthesis of crystalline zinc oxide powders have been reported such as conventional solid state process, co-precipitation, sol-gel, combustion hydrolysis, Pechini (polymerized complex method) and simple combustion. Combustion synthesis is an important technique for the synthesis and processing of advanced ceramics, catalysts, composites, alloys, intermetallics and nanomaterials (Sousa, Sagadaes, Morelli, & Kiminami, 1999; Sagadaes, 2009). Combustion synthesis processes are characterized by high-temperatures, fast heating rates and short reaction times. These features make combustion synthesis an attractive method for the manufacture of technologically useful materials at lower costs compared to conventional ceramic processes. Other advantages of combustion synthesis include the use of simple equipment, formation of high-purity products, stabilization of metastable phases and formation of almost any size and shape (Patil, Aruna, & Mimani, 2002).

The materials selected for this experimental work were Zinc nitrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] and glycine [$\text{NH}_2\text{CH}_2\text{COOH}$] having 99.9% purity are used to synthesize nanocrystalline ZnO powder, through a chemical reaction expresses as below:



where Ψ is the glycine to zinc nitrate molar ratio. In this experimental work, glycine is used as fuel [inexpensive and having more negative $\Delta H_{\text{combustion}}$ (-3.24 k Cal/g) than urea (-2.98 k Cal/g) and citric acid (-2.76 k Cal/g)], whereas zinc nitrate performs dual roles as a zinc source and the oxidant¹. Combustion technique involves an exothermic decomposition of a fuel–oxidant precursor which results in either a finely divided powder of the required phase or semi-decomposed precursor having a considerable carbonaceous

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