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Influence of calcination temperature on structural, optical, dielectric properties of nano zirconium oxide

C. Rajababu Chintaparty*

Department of Materials Science and Nano technology, Yogi Vemana University, Kadapa 516003, India

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ABSTRACT

Zirconium Oxide (zirconia) nanoparticles have been prepared through hydrothermal method at different calcinatation temperatures. The structural properties of zirconia powders were characterized by X-ray diffraction (XRD) technique. The sample prepared at 400 °C shown the mixed phase with co-existence of cubic and monoclinic phases and the samples prepared at 600 °C, 800 °C and 1100 °C shown the monoclinic phase. Further, surface morphology and particle size of the sample calcined at 400 °C was confirmed by transmission electron microscopy (TEM). The band gap is estimated from UV–vis spectra of zirconia particles .The frequency dependence of dielectric constant and loss were investigated at room temperature. Result showed, dielectric constant decreases with frequency.

1. Introduction

Ceramic oxides play an important role in the field of dielectric and electric applications for many years due to its unique properties such as high thermal stability, high dielectric constant, low electrical conductivity and high ionic conductivity [1,2]. In recent years most of researchers focused on alternative material to silicon based dielectric devices for enhancement of storage density in dynamic random access memory, fabrication of very large scale integrated circuit, gate dielectrics, prevent chemical corrosion, oxidation in metals and to sense oxygen [3–5]. In the view of above, zirconium oxide (ZrO_2) is one of the promising candidate having its high dielectric constant, low toxicity, higher band gap and ecofriendly ness when compare to other ceramic oxides [6–8].

The properties of zirconia based devices were strongly depending on crystal phase and crystallite size. It is, well known that; pure ZrO_2 exhibits three polymorphs namely cubic, tetragonal and monoclinic. The stable form at room temperature is the monoclinic phase, which is known to be transforming to tetragonal phase at 1170 °C and transform to cubic phase at 2370 °C [9]. To enhance performance of nano scale devices and high surface area catalysts, so it is essential to understand the relation between phase transformations with crystallite size affects the properties of nano zirconium oxide [5,9–11]. Zirconium oxide nano particles are prepared by several methods such as hydrothermal method, Sol–Gel method, Co-Precipitation method etc [12–17].

Hydrothermal technique as it is an ideal technique for synthesis of ceramic oxides with high purity, controlled stoichiometry, narrow particle size distribution, controlled morphology, microstructure and high crystalinity [18,19]. The aim of this article is to characterize nano crystalline zirconium oxide synthesized by hydrothermal method. The effect of calcinatation temperature on the structural, optical and dielectric properties has been studied.

2. Experimental details

The starting materials namely zirconium oxochloride, lithium hydroxide were used without further purification. Initially, 1 M zirconium oxochloride and 4 M lithium hydroxide aqueous solutions were prepared separately. In the next step $LiOH{\cdot}H_2O$ solution was slowly added to zirconium oxochloride solution using hot plate magnetic stirrer to obtain homogeneous solution of pH value 12. This homogeneous solution was transferred in to an autoclave and is loaded in to an oven. The oven was maintained at 110 °C for 14 h. After cooling to room temperature the autoclave was taken out from the oven. The precipitate was filtered, washed with distilled water and ethanol to reduce the agglomeration. The obtained powder was dried in an oven at 80 °C and then calcined at 200, 400, 600, 800 and 1100 °C for 2 h.

The structural analysis of the above prepared powders is characterized by X-ray diffraction (Model Rigaku ultima III). The surface morphology and size of the powder samples are determined by TEM (Model Tecnai-12). The optical properties were analyzed from

* Corresponding author. Tel.: +91 9949677407.
E-mail address: baburaja46@gmail.com

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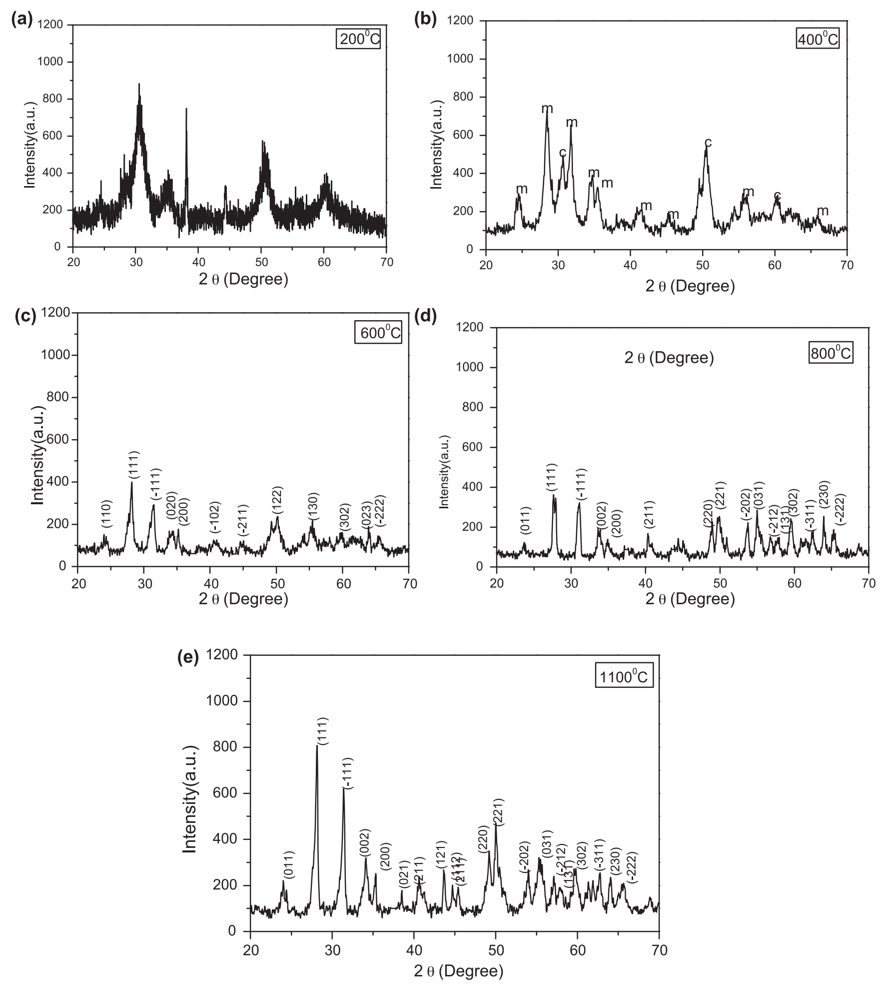

Fig. 1. XRD patterns of zirconium oxide calcined at (a) 200 °C, (b) 400 °C, (c) 600 °C, (d) 800 °C and (e) 1100 °C.

UV–vis spectrophotometer (Model: UV-3092). The dielectric characterization is measured by low frequency impedance analyzer (model HIOKI 3532-50).

3. Results and discussion

3.1. Structural and morphology studies

The X-ray diffraction pattern obtained for ZrO_2 nano particles calcined at 200 °C, 400 °C, 600 °C, 800 °C and 1100 °C are shown Fig. 1. From Fig. 1, it is observed that there are some peaks related to cubic phase and also unknown peaks also detected for the sample calcined at 200 °C. It represents is not completely crystallization process of ZrO_2. It should require higher calcination temperature needed for completely crystallization process. Upon increasing calcination temperature 200 °C to 400 °C only those peaks which are related to the co-existence of monoclinic and cubic phase of zirconia and for the sample calcined at 600, 800 and 1100 °C showed single monoclinic phase of zirconia. The average crystallite size of prepared samples was 11, 9, 14 and 21 nm, respectively. The morphology and the size of particle sample prepared at 400 °C further checked with TEM studies (Fig. 2). The crystallite size observed by TEM is in good agreement with result estimated by X-ray diffraction. From these results we can say that transformation of meta stable phases to the monoclinic occurs mainly due to decrease in surface energy. Hence particle size analysis finds a place in deciding and transformation of crystalline phase. There are numerous studies which showed such transformation from meta stable state to its original monoclinic phase. At higher calcination temperature, the crystallites formed are larger size, which can attribute to the thermally promoted crystallite growth [20–23].

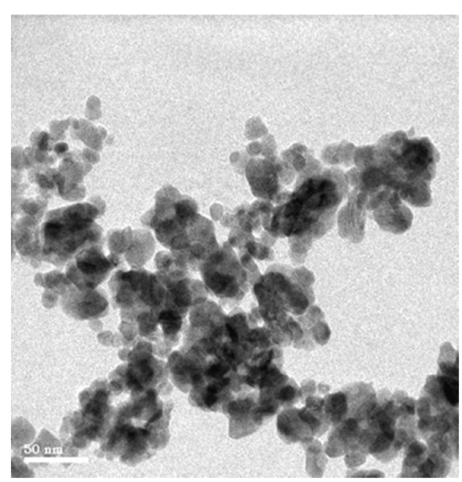

Fig. 2. TEM image of zirconia nano particles calcined at 400 °C.

3.2. Optical studies

The UV Visible absorption spectra ranging from 200 nm to 800 nm wavelength was taken for the samples prepared nano