STRUCTURAL, MORPHOLOGICAL STUDIES OF CU DOPED BaTiO₃ CERAMICS

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Abstract
An array of Ba₁ₓCuₓTiO₃ (X = 0.2, 0.4, 0.6, 0.8) nanoparticles were manufactured by virtue of hydrothermal technique. The diffraction analyzing substantiates the cubic perovskite structure of the barium copper titanate ceramics. The average crystallite-size (Dc) was commence to be of 21 nm employing the Scherrer method. The high concentration of copper addition (x = 0.8) manifests that the crystal architecture undergoes transformation from cubic to mixed phases (cubic and copper titanate). The surface morphology was investigated using field emission scanning (FESEM). The results cater the well defined grains with uniform grain structure. The average grain size is found to be varying between 57 to 181 nm.

Keywords: Structure; Ceramic Titanate; Nanoparticles; Field Emission Scanning Electron Microscopy, Hydrothermal Method; Diffraction; Morphology.

1. INTRODUCTION

Nanoparticle synthesis techniques have got more importance than the bulk synthesis technique owing to their potential applications in biomedical sciences, drug delivery systems, sensors, nanofibers, carbon nanotubes, quantum dots, photocatalysis, dielectric, ferroelectric and piezoelectric properties [1]. Several researchers prepared the BT nanoparticles via distinct methods such as hydrothermal [2], sol-gel, micro-emulsion, polymeric precursor and microwave heating. All the above stated techniques focussed on the structural, morphological, Raman & IR-spectra, UV-visible spectra, dielectric and ferroelectric properties. More recently Selvarajan et al. [3] prepared the piezolectric BT nanoparticles and proposed that it can be used as an active biosensor for the biomolecular detection via the conventional-solid state reaction method. In addition to these, Singh et al.[4] experimentally showed that the perovskite BT thinfilm is an effective material for LPG sensor. Therefore, in the current investigation, the authors focussed to elucidate the structural, morphological and optical properties of BT microspheres.

2. Experimental Procedure

In order to synthesize the barium copper titanate nanoparticles the starting materials were chosen as Ba(NO₃)₂, Cu(NO₃)₂3H₂O and TiO₂. These precursors were mixed together after taking their stoichiometric ratio. The whole mixed precursors were transfer to glass beaker. Furthermore distilled water is added to the precursors in the ratio of 1:4 (mixed precursors (gm): distilled water (ml)) and the resultant solution is kept on a magnetic stirrer. This stirring rate of 500 rpm is maintained in order to stir the solution. Later NaOH solution is slowly added and the pH value reached to 11.3. Furthermore this solution is transferred to 300 ml Teflon bowl inserted in an autoclave. The sealed autoclave is kept in a hot-air oven at an operating temperature of 130°C/6 hrs. After completion of the reaction the autoclave was slowly cooled to room temperature. The final BaCuTiO₃ nano-particles were removed from the Teflon lined autoclave and washed with acetone and distilled water for 10 to 12 times until the pH is reduced to 7. The final BCT new particles were removed and characterized for various characterized techniques such as X-ray diffraction method field emission scanning.

3. Results and Discussions

3.1 XRD Analysis

The diffraction pattern of BCT nanoparticles is depicted in Fig.1. It can be seen that the BCT nanoparticles (X=0.2 to 0.8) exhibit the cubic perovskite structure pertaining the reflection planes of (100), (110), (111), (002), (200), (210), (211), (202), (003), (301), (311), (100). These reflection planes are in good agreement with JCPDS data of file number: 52-0626. Among these planes the (110) plane revealed the maximum intensity for all the composition. A small secondary peak (*) is observed for X=0.2 composition this secondary peak is related to CuTiO₃ phase. With increase of Cu-content in the BaTiO₃ system the number of CuTiO₃ secondary phases starts increasing. At higher concentration of Cu-content (X=0.8) larger number of CuTiO₃ (CT) secondary phases are noticed. Hence it is an established fact that there is a change of transition from cubic to mixed phases (cubic and CT phases). The average crystallite size ‘Dp’ is evaluated with the help of average full-width at half-maxima (FWHM) of reflection planes using the Debye-Scherrer equation [5]
\[ D_R = \frac{k\lambda}{\beta \cos\theta} \]  

(1)

where \( \beta \) is full width half maxima (FWHM) is wave length of CuK\(\alpha \) radiation (0.1542 nm) and \( \theta \) is diffraction angle and ‘K’ is a numerical constant which is equal to 0.9 for a spherical atom. The results are repeated in Table.1. It is clear from the table that the average crystal size is decreasing from 49 to 9 nm for \( x=0.2 \) to 0.6 and further it starts increasing \( x=0.8 \) composition. In addition, the lattice constants (a) are calculated after finding the inter-planer spacing (d) and miller indices (hkl) by using the following formula [6]:

\[ a = d (h^2 + k^2 + l^2)^{1/2} \]  

(2)

3.2 Surface morphology

The surface morphology of BCT nano-particles is analyzed by FESEM. The FE-SEM photographs of BCT nano-particles are showed in Fig. 2. The grain size (\( G_a \)) is determined using linear intercept method [7]:

\[ G_a = \frac{3L}{2MN} \]  

(4)

where ‘L’ is the line length, ‘N’ is the number of grains intercepting the test line’ M’ is the magnification. The average grain size is found to be varying between 57 to 181 nm.

4. Conclusions

The Ba\(_{1-x}\)Cu\(_{x}\)TiO\(_3\) (\( x = 0.2, 0.4, 0.6, 0.8 \)) nanoparticles were prepared by hydrothermal technique for the first time. The diffraction pattern revealed that there is transformation from BCT cubic phases to mixed phases of copper titanate and BCT. The average grain size is observed to be altering between 57 to 181 nm.

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