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Short communication

Synthesis of bismuth oxide nanoparticles using bismuth nitrate and urea

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Abstract

A mixture of bismuth nitrate and urea is taken in 1:5 molar ratio and heated on hot water bath. A precipitate is formed on evaporation of the water which on decomposition at 400 °C produces nanocrystalline Bi_2O_3 particles. Formation of nano crystallites of Bi_2O_3 is confirmed by X-ray diffraction (XRD) study. Transmission electron microscopy (TEM) investigations revealed for these powders an average particle size of 50 nm.

Keywords: A. Calcination; B. Electron microscopy; B. X-ray methods

1. Introduction

Bismuth oxide as a component finds use in wide applications in varistors, catalyst and gas sensors. Bismuth oxide (Bi_2O_3)-based compounds are much better solid electrolytes than well-known stabilized zirconia, because the face-centred cubic (fcc) Bi_2O_3 exhibits the highest ion conductivity of all oxide ion conductors [1–3]. Bi_2O_3 is also used as an additive in paints. Furthermore the total oxidation of isobutene is enhanced on bismuth containing tin catalysts. Typically Bi_2O_3 is prepared via the oxidation of bismuth metal at 800 °C via thermal decomposition of carbonates or hydroxides produced by the addition of alkali-metal hydroxides to bismuth salt solution [4–6]. These powders on calcinations yield fine particles of Bi_2O_3. Flame spray pyrolysis [7] is also used to produce nano-sized Bi_2O_3 particles. The properties of ceramics are greatly affected by the characteristics of the powder, such as particle size, morphology, purity and chemical composition. Using chemical methods, e.g. co-precipitation, sol–gel, hydrothermal and colloid emulsion technique have been confirmed to efficiently control the morphology and chemical composition of prepared powders. Here, we report a simple urea nitrate process of for the preparation of nanocrystalline Bi_2O_3. This method is commonly used for preparation of various oxides [8–11] and not yet reported for the preparation of Bi_2O_3 ceramics. Urea is used as a fuel, precipitating agent and as a resin former with formaldehyde. When urea is used along with nitrate salt of a cation and heated at 400 °C, the exothermic reaction between nitrate (oxidant reactant) and urea (fuel) leads to formation of corresponding nanocrystalline oxides. The main advantage is that necessary heat for synthesis is obtained directly from the reaction [8,9]. In the case of homogeneous precipitation, urea acts as a precipitant. Since urea decomposed around 100 °C to produce carbon dioxide and ammonia thereby increasing the pH of the solution at which metal cation precipitation takes place [10]. In yet another method [11] urea forms polymeric resin with formaldehyde along with reactants and on the decomposition of the resin at higher temperatures, the final product is formed.

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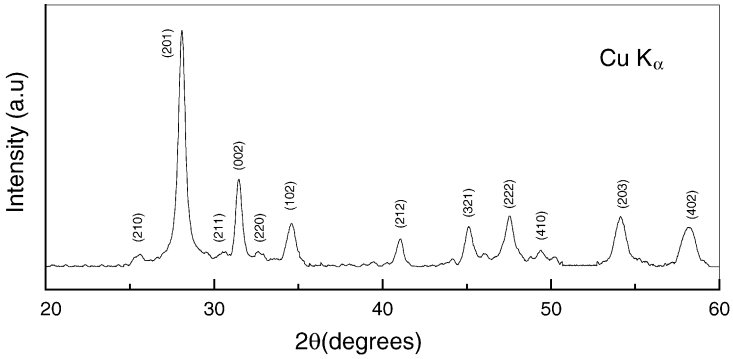

Fig. 1. XRD of Bi_2O_3 powders calcined at 400 °C.

2. Experimental

Bismuth nitrate and urea used for the preparation of Bi_2O_3 are of AR grade. A known quantity of (4 g) $Bi(NO_3)\cdot 5H_2O$ is dissolved in nitric acid (50 ml) and mixed with urea in a 1:5 molar ratio and heated on a water bath. Since there was no precipitation during mixing, the pH of the solution was not varied. On heating on a water bath at 373 K a white precipitate was formed after evaporation of water. Subsequently, the precipitate is decomposed at various temperatures ranging from 400 to 700 °C. The powders initially started to swell and filled the beaker producing a foamy precursor. This foam consists of homogeneous flakes of very small particle size. Various techniques such as XRD (Philips PW 1710 Diffractometer) and BET surface area measurements (Nova 1200 instrument) were employed to characterize these powders. For lattice parameter and interplanar distance (d) calculation, the samples were scanned in the 2θ range of 10–80° for a period of 5 s in the step scan mode. Silicon was used as an internal standard. Least squares method was employed to determine the lattice parameters. The TEM picture was recorded with JEOL model 1200 EX instrument at the accelerating voltage of 100 kV. The fine powders were dispersed in amyl acetate on a carbon coated TEM copper grid.

3. Results and discussion

The mixture of urea and bismuth nitrate solution forms a precipitate on heating on a water bath which on decomposition at 400 °C yields nano particles of bismuth oxide. During calcinations process, a white fluffy mass (foam-like) is formed which occupies large volumes of the furnace. The urea decomposes into ammonia and carbon dioxide. Fig. 1 shows the XRD for the samples heated at 400 °C. The sample showed the crystalline pattern and the observed d-lines match the reported values for the β-Bi_2O_3 phase (JCPDS no. 27–50). The calculated lattice parameters by least square fit are $a = 7.732$ Å and $c = 5.618$ Å. The surface area of these powders was found to be 100 m^2/g. The average particle size of these powders is found to be 50 nm (not shown). The crystallite size measurements were also carried out using the Scherrer equation, $D = k\lambda/\beta \cos\theta$ where D is the crystallite size, k is a constant (=0.9 assuming that the particles are spherical), λ is the wavelength of the X-ray radiation, β is the line width (obtained after correction for the instrumental broadening) and θ is the angle of diffraction. The particle size obtained from XRD data is 100 nm.

4. Conclusions

A simple urea–nitrate process was elucidated to prepare bismuth oxide nanoparticles. The average size of these particles ranges from 50 to 80 nm.

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