

XRD studies on hydrothermally synthesised BaTiO₃ from TiO₂–Ba(OH)₂–NH₃ system

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Abstract

A detailed work on synthesis of mono-phasic barium titanate (BT) using TiO₂ powder in Ba(OH)₂–NH₃ medium at moderate temperature has been reported. Influence of various preparation parameters such as reaction temperature, reaction time, ammonia concentration and [Ba/Ti] ratio on the formation of barium titanate was studied. The progress of reaction for formation of BT was monitored by analyzing the X-Ray diffraction data obtained under different processing conditions. To quantify the percentage of reacted titania, calibration curves were generated from synthetic mixtures of titania and pure barium titanate. Mono-phasic tetragonal barium titanate having mean crystallite diameter of 27 nm along (101) plane was formed when the reaction was carried out for 1 h at 130 °C while keeping the initial [Ba/Ti] ratio as 1.64 and ammonia concentration as 10 M.

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1. Introduction

Barium titanate, especially the tetragonal phase, has excellent ferroelectric properties, which makes it the most important compound used in the composition of broad range of microelectronic devices. BaTiO₃ also possesses very good piezoelectric and dielectric properties which make it suitable for multilayer ceramic capacitors (MLCC), dynamic random access memories (DRAM), IR sensors, transducers, etc. [1–3]. There are many methods for synthesizing high pure, homogeneous, reactive fine BaTiO₃ powders, such as high temperature ceramic route [4,5], co-precipitation [6,7], sol–gel method [8,9], hydrothermal method [10,11] and co-precipitation in combination with inverse micro-emulsion [12]. The overriding goal in barium titanate processing is to create smaller, more uniform particles to allow for finer ceramic layers to be used in MLCCs. This leads to device

miniaturization without loss of dielectric properties, using fast and low temperature synthesis mechanisms. Hydrothermal processing is one of the most important techniques that can produce either large single crystals or fine sub-micron ceramic powders, depending on the apparatus configuration and the material system [13]. In addition, this method possesses the potential to produce crystalline powders with controlled particle size, controlled stoichiometry and in some cases, controlled particle shape [14–16].

Lu et al. [11] prepared barium titanate (BaTiO₃:BT) nano-crystals by hydrothermal method in the presence of polyoxyethylene sorbitan mono-oleate as a polymeric phase modifier at 230 °C at a pH of 13.5 and varying the reaction time from 0.5 to 2 h. Xu et al. [17] also prepared barium titanate powder, with an average particle size of 80 nm, by hydrothermal method at a temperature of 240 °C. In this case the reactants used were barium and titanium chlorides. Hu et al. [18] prepared mono-dispersed micro spheres of nano-crystalline barium titanate by initially synthesizing titania micro spheres and then reacting these spheres with barium hydroxide

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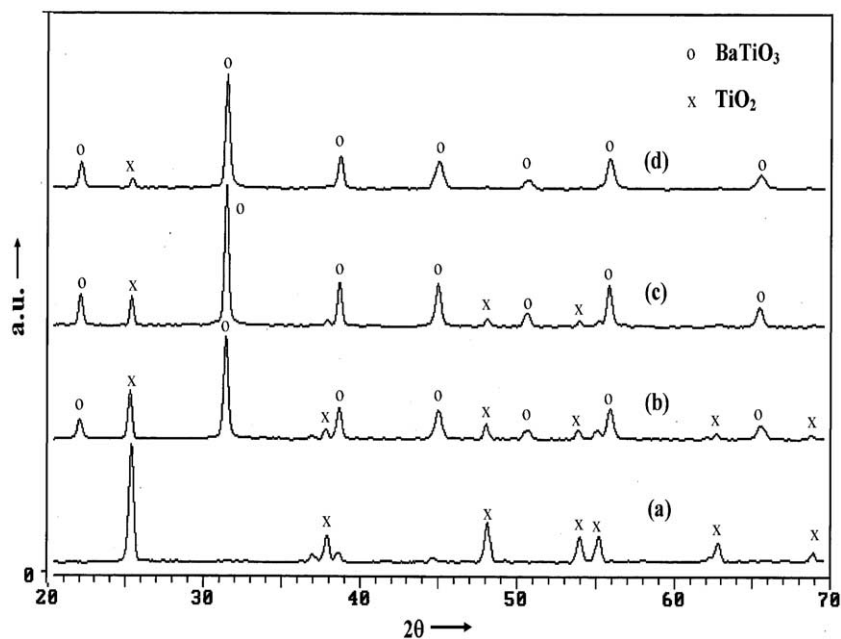


Fig. 1. XRD patterns of products obtained during heating (non-isothermal phase) (a) 90 °C (b) 100 °C (c) 115 °C (d) 130 °C.

solutions at 100 °C for 24 h. Attempts to prepare barium titanate in the temperature range of 75 to 100 °C required reaction time of more than 20 h [19]. In all the above cases and in almost all-hydrothermal synthesis, researchers have used alkali metal hydroxides namely KOH or

NaOH as mineraliser [20–22]. Use of aqueous ammonia solution in place of NaOH or KOH can be advantageous because: (a) ammonia does not have a tendency to get incorporated into the oxide matrix and (b) any residual ammonia entrained in BT powder can be easily driven off during drying the powder at 100 °C. Due to these advantages, a systematic study was carried out for synthesizing BT using TiO_2 in barium hydroxide–ammonia medium.

2. Experimental

A predetermined quantity of barium hydroxide octahydrate $[\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}]$ crystals (Merck, 98% pure) was added to a fixed quantity of titanium dioxide (TiO_2) (Merck, 99% pure) powder and transferred to a 2 L. PARR autoclave [Model No. 4542] having provisions for heating and sampling. A calculated quantity of 10 M

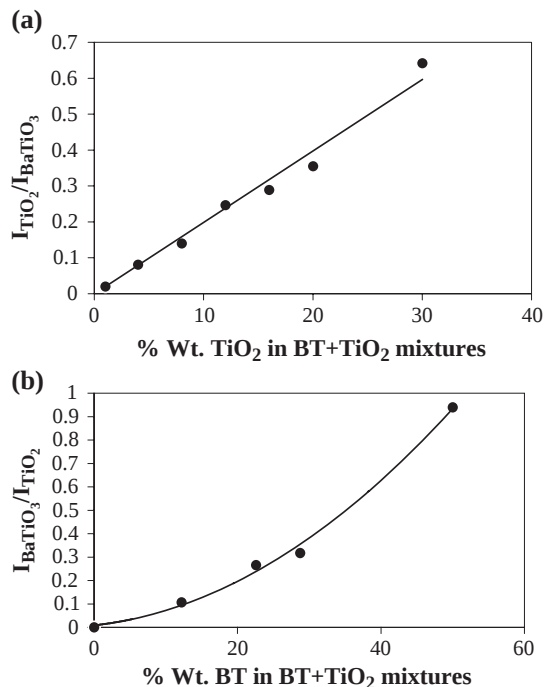


Fig. 2. (a) Calibration curve for percent weight of TiO_2 in BT– TiO_2 mixtures with 100% RI for plane (101) of BT. (b) Calibration curve for percent weight of BaTiO_3 in BT– TiO_2 mixtures with 100% RI for plane (101) of TiO_2 .

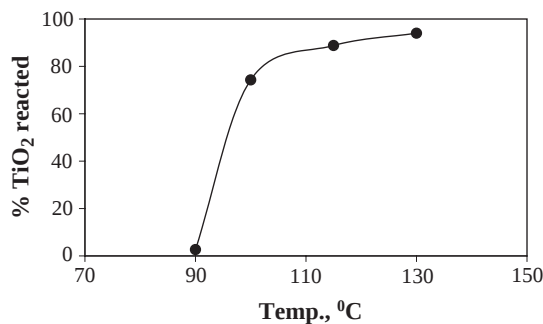


Fig. 3. Percentage of TiO_2 reacted during heating at different temperature (non-isothermal phase).

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