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Synthesis of Barium Titanate as an Electroceramic Raw Materials

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ABSTRACT

Nowadays, most electronic and electrical parts of equipment in Thailand such as substrates, magnetics, capacitors, ferroelectric, piezoelectric, RAMs, DRAMs, FERAMs, loudspeakers and devices in ultrasonic cleaning are imported. Barium titanate powder is one of electroceramic raw materials. It is used to produce parts of electroceramic products. In this project, barium titanate was produced by hydrothermal synthesis and thermal treatment process. The mol ratios of titanium dioxide to barium hydroxide in the thermal treatment process were 0.5:1, 1:1, and 1:0.5 at 700, 1000 and 1200°C ,for 3 and 5 hours. Results of all experiments were analysed by x-ray diffraction (XRD), scanning electron microscope (SEM), particle size distribution and liquid pycnometer technique. A good condition for barium titanate synthesis is from the hydrothermal reaction at the weight ratio of 5:20, 90°C , and 72 hours. The obtained powders possessed high purity, perovskite structure and a density of 6.2520 g/cm^3. In addition, an average particle size at the accumulated particle size of 50 percent (d_{50}) is less than 1 micron. For the thermal treatment process, the appropriate condition is 0.5 mol titanium dioxide to 1 mol barium hydroxide at 700°C for 3 hours. An average particle size at d_{50} is larger than 5 microns and the density is 4.7100 g/cm^3 because its structure is composed of another composition.

Key words: barium titanate, perovskite structure, hydrothermal synthesis, thermal treatment process

INTRODUCTION

Panne *et al*.(1992) reported that Barium titanate ($BaTiO_3$) is an extensively studied and widely utilized perovskite-type electroceramics. There are two important crystalline phases of barium titanate being used in the microelectronics industry as reported in William (2002). The tetragonal phase of barium titanate is used in a broad array of electronic devices due to its ferroelectric properties, while the cubic form, although not ferroelectric, has a high dielectric constant that makes it suitable as capacitors. The main objective in barium titanate synthesis is to create smaller, more uniform particles to allow for finer ceramic layers to be used in multilayer capacitors, piezoelectric, capacitor and actuator (and, thus, achieve device miniaturization) without the loss of dielectric properties as reported in Kingery *et al*.(1992). Controlling the phase, composition homogeneity, particle size, density and mono-dispersity, microstructure, and the cost of particle production are important concerns in developing techniques for synthesizing barium titanate. In this research, two methods of barium titanate synthesis are studied. One method is thermal treatment process and the other is hydrothermal process. The hydrothermal process is based on Michael (2000).

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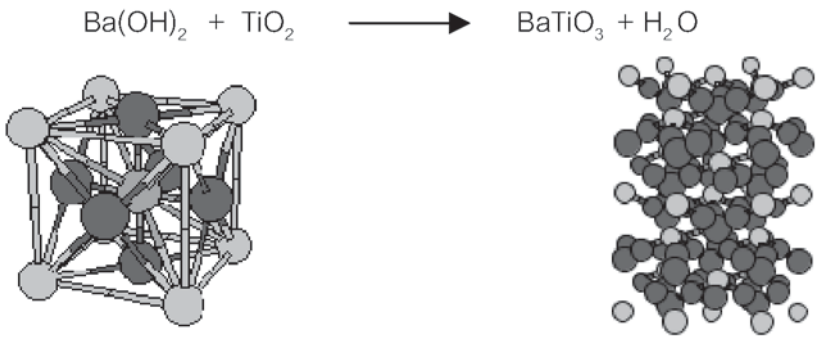

Figure 1 Perovskite structures in Ferroelectric materials.

MATERIALS AND METHODS

The synthesis of barium titanate as an electroceramic raw materials consisted of two methods. The principle of mol ratio mixing of two components are based on Ree and Dippel .(1992)., as follows :

Method 1 Thermal treatment process (Figure 2)

1. Mix titanium hydroxide and barium hydroxide at the mol ratios of 0.5:1, 1:1, and 1:0.5.
2. Pour the mixture from the first step into a ceramic crucible and heat in a furnace at temperatures of 700,1000 and 1200°C for 3 and 5 hrs.
3. Characterize the powder obtained from the second step by XRD, SEM, particle size analyzer, and liquid pycnometer technique.

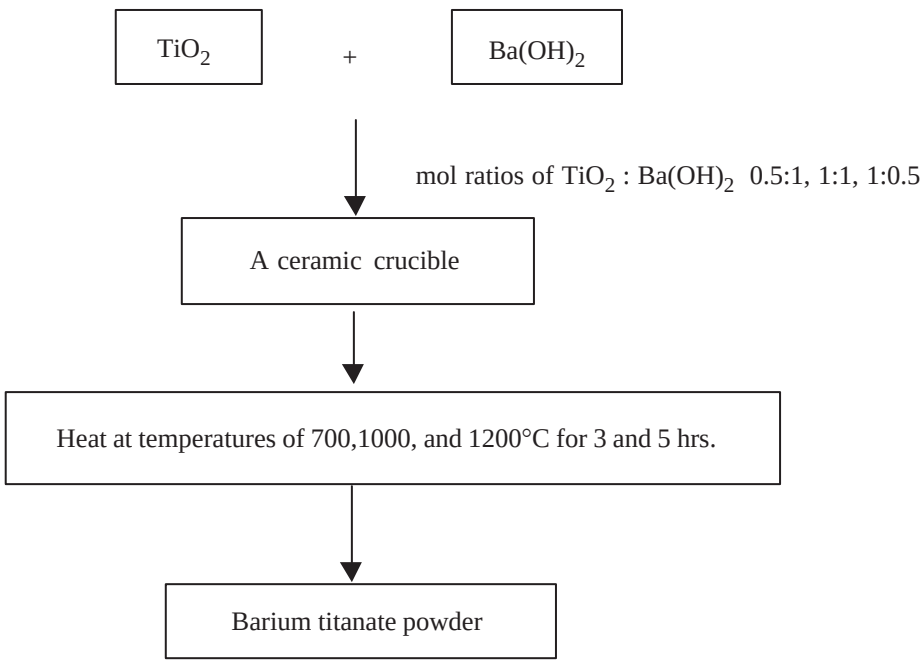

Figure 2 Flow chart of barium titanate synthesis by the thermal treatment process.

Method 2 Hydrothermal synthesis (Figure 3)

1. Mix 20 g. of barium hydroxide with 5 g. of titanium hydroxide(Ba:Ti equals to 1:1 mol ratio) in a Teflon bottle.
2. Add 30 ml. of distillate water into the mixture in the Teflon bottle, cover and shake vigorously.
3. Heat the mixture from the second step in an oven at 90°C for 24 and 72 hrs to activate the reaction.
4. Wash the precipitate from step 2 with 100 ml. of 1 molar of formic acid and filter.
5. Dry the filtered cake at 90°C for 24 hrs.
6. Characterize the powder as received by XRD, SEM, particle size analyzer, and liquid pycnometer technique.

RESULTS AND DISCUSSION

The suitable condition for barium titanate synthesis by thermal treatment process is 0.5 mol of titanium dioxide and 1 mol of barium hydroxide at 700°C for 3 hrs. The powder obtained has a density of 4.7100 g/cm^3 which is less than theoretical density value because of $Al_4Ti_2SiO_{12}$ mixed in perovskite phase.

Hydrothermal synthesis for barium titanate powder is a condition of 5 g. of titanium dioxide

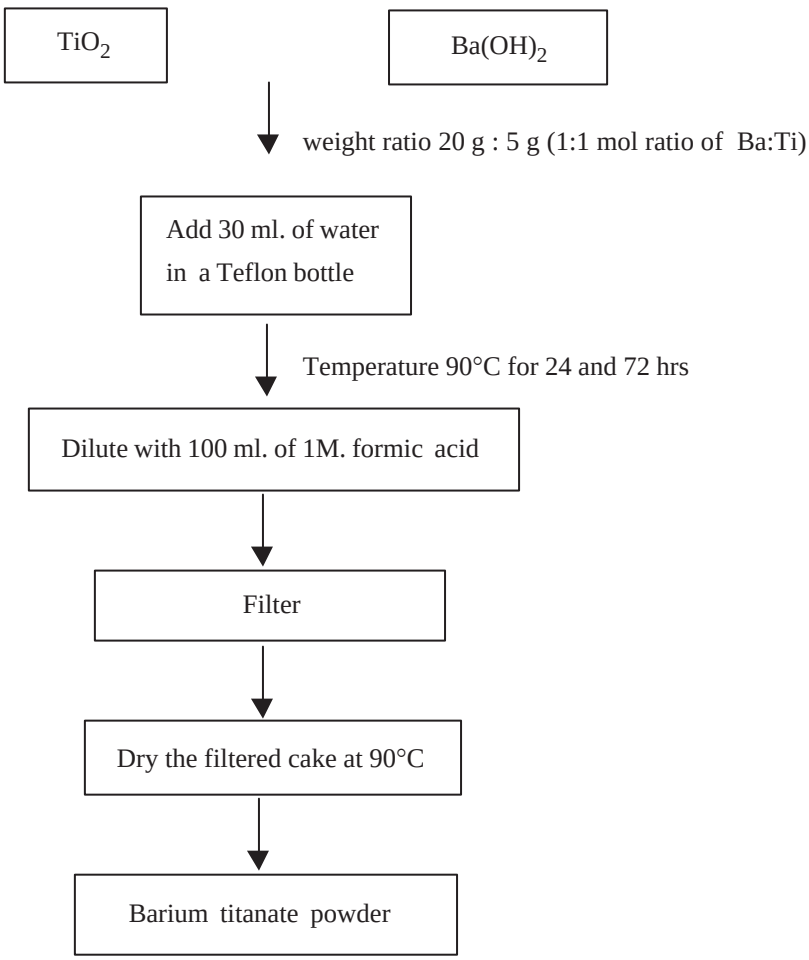

Figure 3 Flow chart of barium titanate synthesis by the hydrothermal synthesis.

mixed with 20 g. of barium hydroxide (mol ratio 1:1), dried at 90°C for 72 hrs. Barium titanate powder has characteristic close to the theory. The powder has less aggregation and better defined microspherical shape (solid sphere with smooth surface) than that of thermal treatment process. Figure 4 shows crystalline phase compositions, most of them are perovskite structure, as Hung and Riman.(1998). The average particle size is smaller than 1 micron, as shown in Figure 5. The microstructure of barium titanate powder, as-prepared by SEM is shows in Figure 6. In this method, 100 ml. of 1 M. of formic acid was added to reduce the barium carbonate impurity level in the barium titanate suspension, the procedure was slightly modified from the work of Eckert *et al*.(1996). Formic acid was added immediately during the suspension was still hot. The suspension was allowed to stand for 15 min at room temperature; then it was centrifuged and washed with deionized water twice, and was removed by pipette. The remaining white paste was air dried to form a cake, and dried in an oven overnight. The other hydrothermal synthesis condition is to use 5 g. of titanium dioxide to 20 g. of barium hydroxide, dry at 90°C for 24 hrs., but the crystalline phase compositions are composed of the perovskite, cubic and tetragonal. The density was reduced to 3.1191 g/cm^3

CONCLUSION

Hydrothermal synthesis is a suitable method to make barium titanate from 5 g. of titanium dioxide of 20 g. of barium hydroxide (Ti:Ba equals 1:1) at 90°C for 72 hrs. Barium titanate powder obtained is filtered with formic acid during hydrothermal process to purify. The powder obtained has monodispersed-nanocrystalline structure, high density, no aggregation and high purity.

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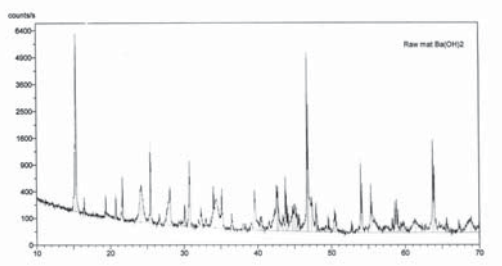
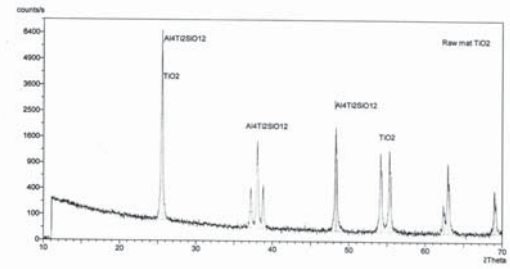
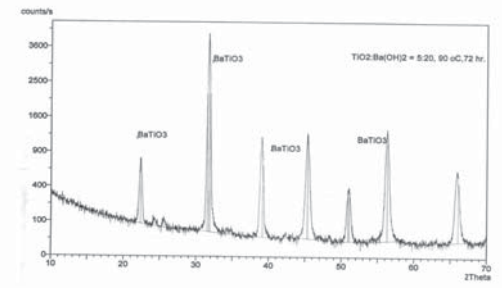


(a) X-ray diffraction of barium hydroxide.



(b) X-ray diffraction of titanium dioxide.



(c) X-ray diffraction of the mixture of titanium dioxide and barium hydroxide at a ratio of 5:20 at 90°C for 72 hrs.

Figure 4 Crystalline phase compositions.

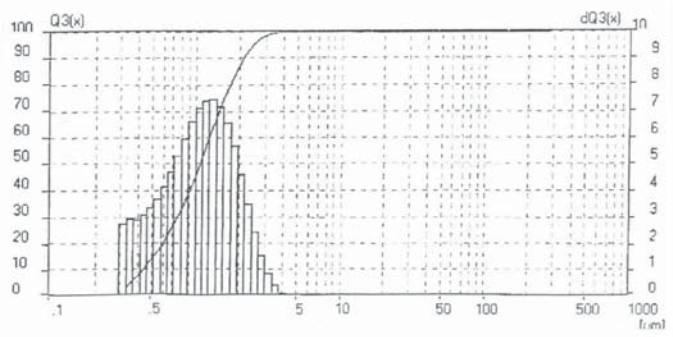


Figure 5 Particle size distribution of barium titanate (hydrothermal synthesis at 90°C for 72hrs.) by particle size analyzer.

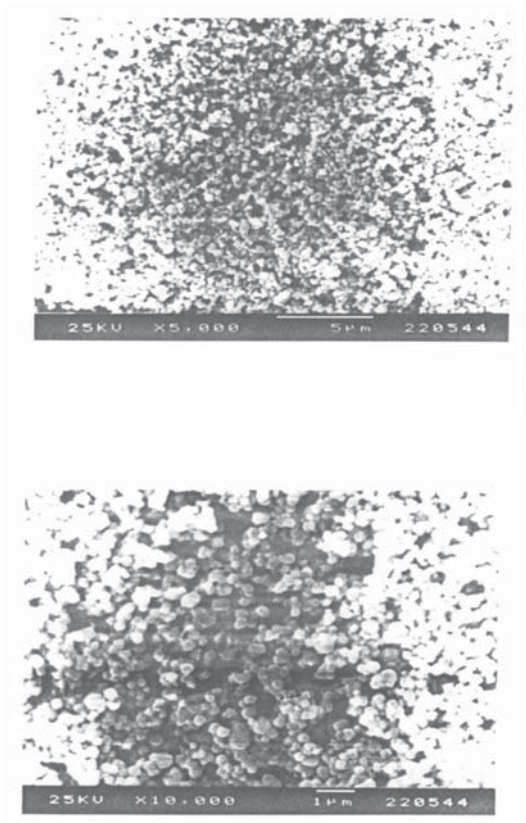


Figure 6 Particle size distribution of barium titanate (hydrothermal synthesis at 90°C, 72 hrs.) by SEM manification 5,000 and 10,000.

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