Synthesis of the Ultrafine $BaTiO_3$ powder by Hydrothermal Process

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Abstract

The $BaTiO_3$ fine powder was prepared by hydrothermal method using titanium tetrahydoxide ($Ti(OH)_4$) and barium dihydroxide ($Ba(OH)_2 \cdot 8H_2O$) as raw materials. The fine powder was obtained at temperatures as low as 160 to 185 ℃. The properties of the $BaTiO_3$ powder were studied as a function of various parameters (reaction temperature, reaction time, Ba/Ti=ratio, etc). The average particle size of the $BaTiO_3$ increased with increasing reaction temperature. After hydrothermal treatment at 170 ℃ for 8 h, the average particle size of the $BaTiO_3$ powder was about 30 nm and the particle size distribution was narrow.

1. Introduction

There are many advantages in the hydrothermal preparation of multinary oxides in the form of fine powders. The moderate temperatures employed during this technique not only reduce the energy costs but also enhance the reactivity of the products. High purity, single-phased oxides can be obtained at relatively faster rates under elevated water vapour pressures and temperatures with minimum pollution.

This technique can produce fine, high purity, stoichiometric particles of single and multicomponent metal oxides [1]. Some precipitated hydroxides subjected to prolonged boiling under atmospheric pressure in their mother liquor or hydrothermally treated under enhanced pressure at elevated temperatures transform to fine-grained oxides of narrow particle size distribution. It has been demonstrated that such powders are composed of much softer agglomerates and sinter much better than those prepared by calcination decomposition of the same oxides [2]. These powders could be sintered at low temperature without calcination and milling steps [3].

The objectives of this study were to prepare ultrafine $BaTiO_3$ by hydrothermal conditions.

2. Experimental Procedure

The $BaTiO_3$ fine powder was prepared by hydrothermal method using titanium tetrahydoxide ($Ti(OH)_4$) and barium dihydroxide ($Ba(OH)_2 \cdot 8H_2O$) as raw materials. The process for preparing $BaTiO_3$ by hydrothermal treatment is schematically illustrated in Fig. 1. The titania sol was obtained by dissolving titanium tetraisopropoxide, $Ti(OiPr)_4$, in isopropanol followed by hydrolysis with excess water. Mixed solution was prepared by dissolving barium dihydroxide in water and by adding titania sol to the solution under reflux condition.

The resulting suspension was placed in a 1000 ml stainless steel pressure vessel. The vessel was then heated to the desired temperature at a rate of 5 ℃/min. During heating, the autogenous pressure gradually increased to 1 MPa and was usually maintained below 5 MPa during the holding period. The reaction products were washed at least two times by repeated cycles of centrifugation and redispersion in methanol. The recovered powders were analyzed for phase composition using X-ray diffraction (Phillips, PW 1825/00) over the 2 theta range from 10-70° at rate of 2.5° /min. The morphology of the synthesized particles was observed using scanning electron microscopy (SEM, Hitachi S-4200).

3. Results and discussion

The hydrothermal reaction proceed chemical reactions and crystallization at practical speeds. At the same time, the hydrothermal reaction promotes uniformly of particle size and chemical composition. As a result, it is possible to obtain fine powders with desirable particle size and good morphology, sharp size distribution, good crystallinity and homogeneous chemical composition. These powder have outstanding dispersability, packability, and sinterability, and so should be useful in improving the quality of devices made from them [4].

Fig. 2 shows the scanning electron micrographs of the synthesized $BaTiO_3$ powders. All the $BaTiO_3$ powders were synthesized at 150 to 180 ℃. Hydrothermal synthesis of the $BaTiO_3$ somewhat nearly spherical and ultrafine particles which was on the order of about 20 to 100 nm in size. The reaction temperature has an effect on the size of the $BaTiO_3$ particles synthesized in solution. The temperature had a great effect on the grain size of the products and the agglomeration among grains.

The sharp diffraction peaks consistant with the well defined and crystallized particles shown Fig. 3. The crystal structure of the synthesized $BaTiO_3$ particle is cubic. Thus, the hydrothermal method used here led to ultrafine spherical particles which may be useful applications for various field.

4. Conclusions

The ultrafine and nearly spherical of synthesized $BaTiO_3$ powder can be prepared by hydrothermal conditions.

After hydrothermal treatment at 180 ℃ for 8 h, the average particle diameter of the synthesized $BaTiO_3$ was about 100 nm. The average particle diameter of the synthesized $BaTiO_3$ increased with increasing reaction temperature and time.

The results of this study show that it is possible to control the size of the synthesized $BaTiO_3$ particles hydrothermal process such as reaction temperatures and time are carefully controlled.

References

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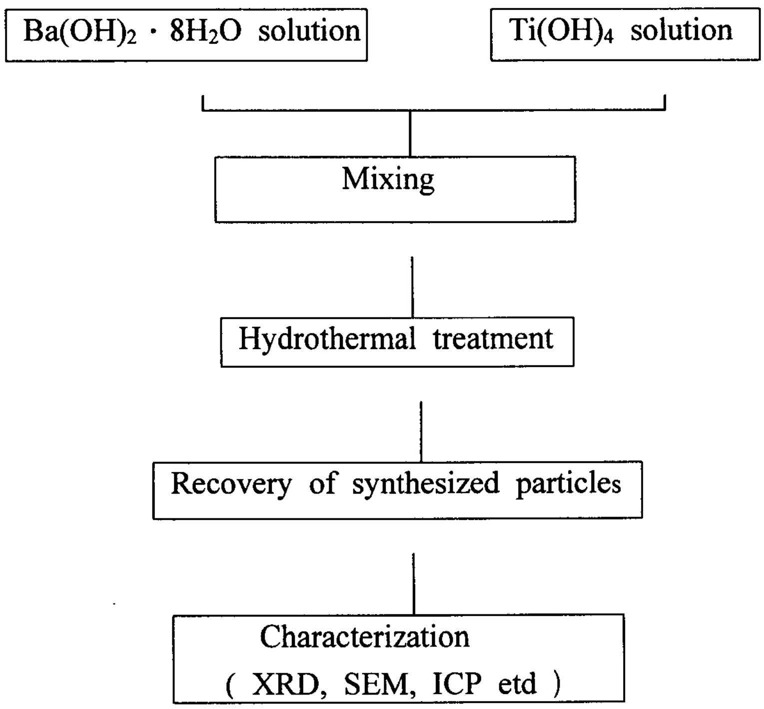
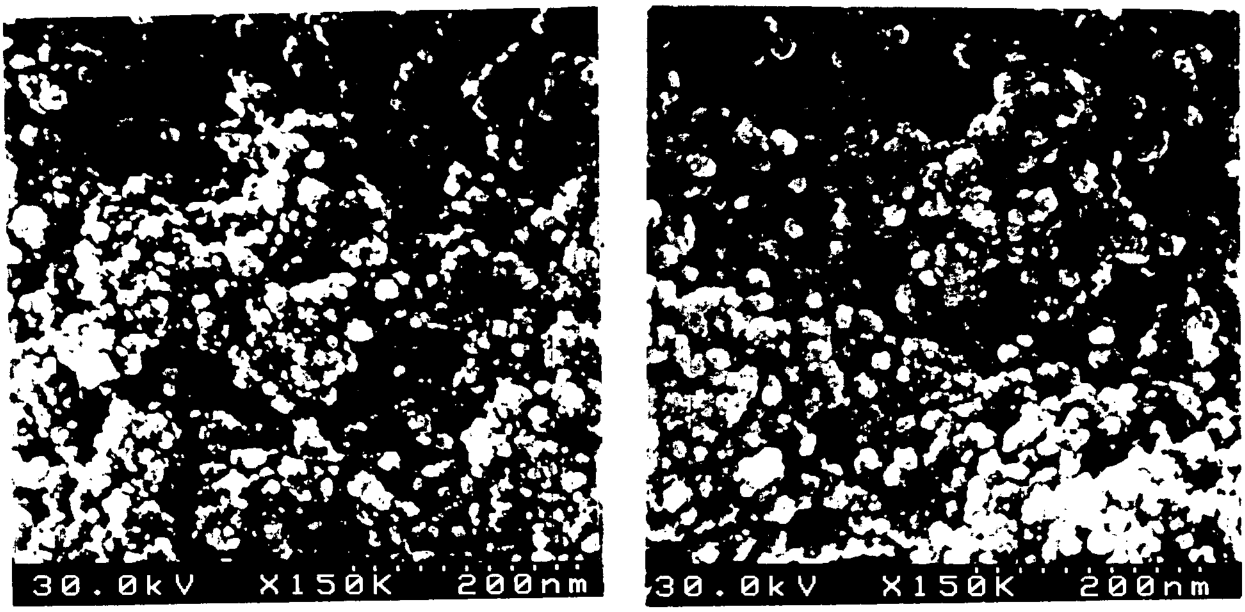


Fig. 1. Preparative procedure for the preparation of the synthesized $BaTiO_3$ particles by hydrothermal process.



(a) (b)

Fig. 2. SEM micrographs of the $BaTiO_3$ particles were synthesized by hydrothermal treatment as a function of reaction temperature at (a) 170 ^{0}C and b) 180 ^{0}C for 8 h.

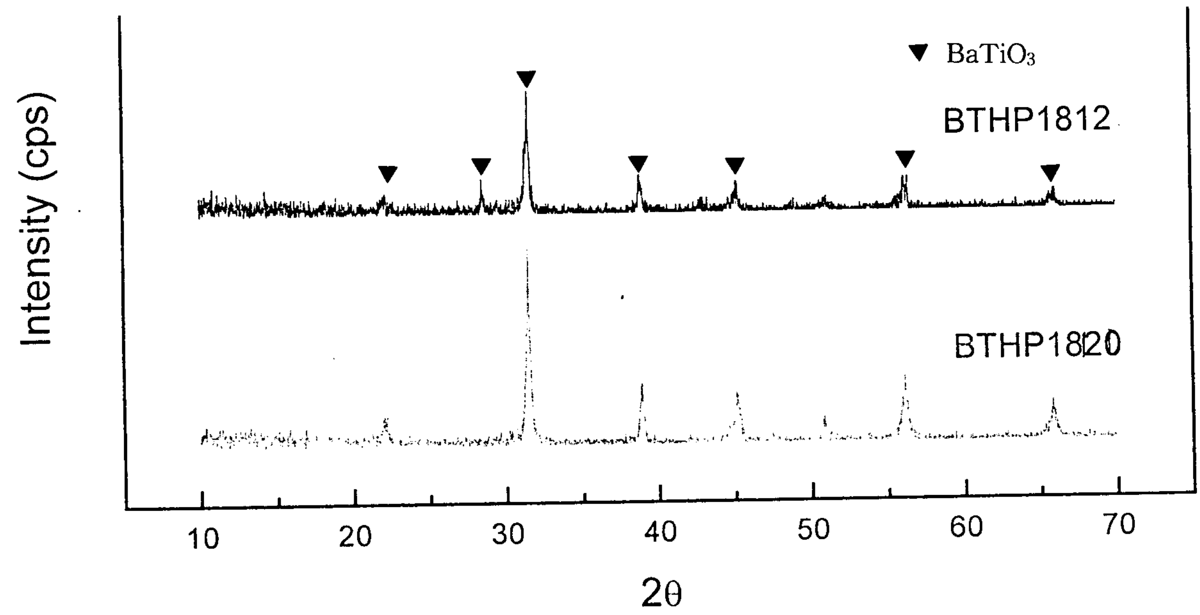


Fig. 3. X-ray diffraction pattern of the $BaTiO_3$ particles synthesized by hydrothermal treatment : a) 160, b) 170 ^{0}C and c) 180 ^{0}C.