PREPARATION OF HIGH-PURITY AND NANOMETER-SCALE BARIUM TITANATE POWDERS①

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ABSTRACT By using H_2TiO_3 and $Ba(NO_3)_2$ as the raw materials, and $(NH_4)_2CO_3$ as the precipitant, high purity(> 99.7%) and superfine(< 1 μm) $BaTiO_3$ powders were obtained by co-precipitation method. Then the nanometer-scale $BaTiO_3$ powders were prepared with the mechanophysical solid effect equipment. The equipment of the present process is characterized by simplicity, convenience and low capital cost. Hence it is suitable for industrial production.

Key words barium titanate powder co-precipitation mechanophysical solid effect high purity nanometer-scale

1 INTRODUCTION

As an important dielectric ceramic material, $BaTiO_3$ is widely used in fabrication of thermal resistors, polylaminate ceramic capacitors and electric-light devices[1]. It is an important research task to obtain high-purity, superfine and uniformly distributed $BaTiO_3$ powders. The traditional method is to grind and calcine the mixture of oxides or salts of Ba/Ti, i. e. solid synthesis. In recent years, $BaTiO_3$ powders are obtained mostly by means of low temperature synthesis[2-7]. There are many ways to manufacture nanometer $BaTiO_3$ powders[8-10]. In order to obtain high purity and superfine $BaTiO_3$ powders, the co-precipitation method is used in the present work. Then the nanometer $BaTiO_3$ powders are prepared by the mechanophysical solid effect[11]. The present process is characterized by low capital cost, high-quality product which can meet the needs for manufacturing high-performance dielectric ceramic cells, in addition, the equipment is simple, convenient, low capital outlay. Hence it is suitable for industrial production.

2 EXPERIMENTAL

2.1 Preparation of $BaTiO_3$ powders

By using the raw materials of H_2TiO_3, $Ba(NO_3)_2$, $(NH_4)_2CO_3$ and $NH_3 \cdot H_2O$, $BaTiO_3$ powders were prepared by means of co-precipitation. After purification and heating, the H_2TiO_3 was turned into colloid which was fully mixed with $Ba(NO_3)_2$ aqueous solution, and then was mixed with $(NH_4)_2CO_3$ aqueous solution. By adding $NH_3 \cdot H_2O$ to adjust the pH value of the mixture, the co-precipitation reaction occurred. And finally after filtration, washing, drying and calcination, $BaTiO_3$ powders were obtained.

2.2 Fabrication of nanometer $BaTiO_3$ powders

By feeding the former $BaTiO_3$ powders into the mechanophysical solid effects equipment and controlling the technology parameters and processing time, the nanometer-scale powders were obtained. The microstructures of the samples regularly taken from the argon-filled glovebox were examined.

2.3 Examination of $BaTiO_3$ powders

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The $BaTiO_3$ powders obtained by means of co-precipitation were examined by X-ray diffraction, emission spectrum and scanning electron microscope (SEM), the nanometer Ba_2TiO_3 powders prepared with mechanophysical solid effect was observed by transmission electron microscope(TEM).

3 RESULTS AND DISCUSSION

X-ray diffraction analysis (Fig. 1) shows that the main crystal phases of $BaTiO_3$ powders obtained by co-precipitation method exist the (200), (020) and (002) diffraction double-peaks when 2θ is about 45°, which can be defined as tetragonal structure without other impure phases. The results of emission spectrum are presented in Table 1, which indicates that the content of the impurities in product $BaTiO_3$ is very low, the purity of the $BaTiO_3$ powders is > 99.7%.

SEM analysis (Fig. 2) indicates that the $BaTiO_3$ particles are narrowly distributed in size, the average diameter of the powder is below 1 μm. TEM photograph of nanometer $BaTiO_3$ samples treated by wet method for 8 h is shown in Fig. 3, which indicates that the figure of the grain is nearly spherical, and the average size is about 30 nm. TEM analysis also shows the particle size of $BaTiO_3$ powder prepared by mechanophysical solid effect is superfine. The grain size and figure of the samples treated by dry process for 24 h are the same as those of the samples treated by wet method for 8 h. If the processing time is prolonged, the grain size will get smaller.

4 CONCLUSIONS

(1) High purity (> 99.7%) and superfine (< 1 μm) tetragonal $BaTiO_3$ powders can be obtained by means of co-precipitaion.

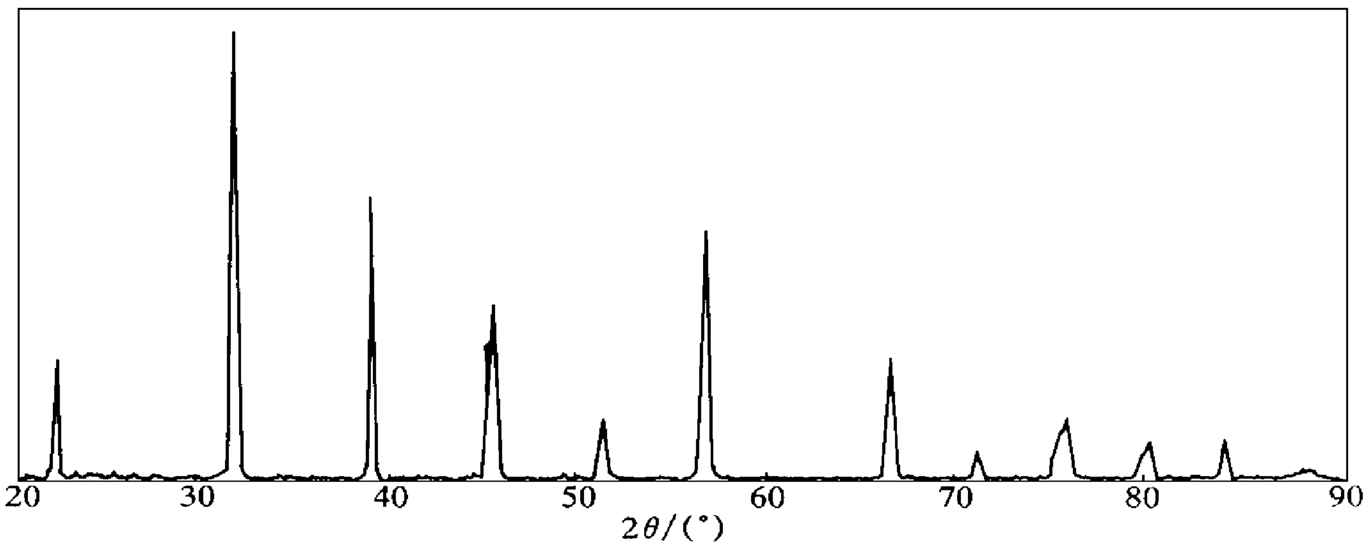

Fig. 1 X-ray diffraction pattern of $BaTiO_3$ powders obtained by means of co-precipitation

Table 1 Results of emission spectrum analysis of $BaTiO_3$ powders prepared by co-precipitation method

Element	Si	Pb	Sn	Sb	Fe	Mn	V	W
Content /%	< 0.003	≤0.001	< 0.001	< 0.005	< 0.002	0.005	0.0001	< 0.001
Element	Bi	Cr	Ni	Co	Mg	Cu	Al	
Content /%	< 0.001	< 0.001	< 0.001	< 0.005	< 0.001	< 0.0002	< 0.001	

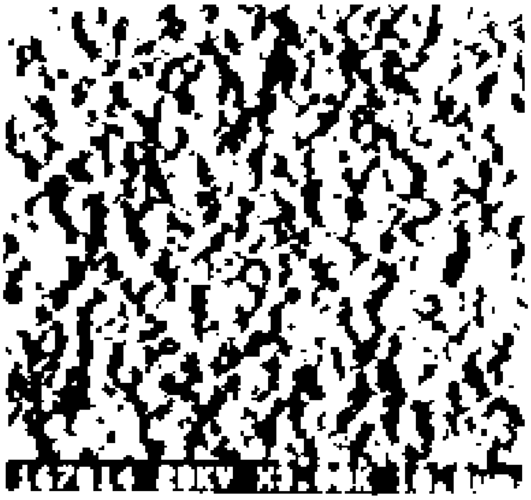

Fig. 2 SEM photograph of $BaTiO_3$ powders obtained by means of co-precipitation

Fig. 3 TEM photograph of nanometer $BaTiO_3$ powders (× 60 000)

(2) The former $BaTiO_3$ powders can be further refined in mechanophysical solid effects equipment to obtain nanometer $BaTiO_3$ powders.

(3) The process is characterized by low capital cost, simple equipment, hence it is suitable for industrial production.

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