Research on Preparation of Nano-Barium Titanate and Dielectric Property of Barium Titanate Ceramic Capacitor

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ABSTRACT

A mild alcohol-thermal synthesis of nano-BaTiO$_3$ from H$_2$TiO$_3$ and Ba(OH)$_2$ at an Atmospheric Press was systematically studied and the optimized preparation conditions were obtained. When the reaction is carried out at a temperature of 79°C, an atmospheric press for 6h, the moral ratio of Ba(OH)$_2$: H$_2$TiO$_3$=1.0, pH=11, the cubic crystal structure of nano-BaTiO$_3$ was obtained. The dielectric ceramic was fabricated with nano-BaTiO$_3$. By comparison with ordinary ceramic, the ceramic with nano-BaTiO$_3$ from alcohol-thermal method could lower sintering temperature from 1300°C to 1150°C, change Courie point to 70°C, make dielectric constant 20000.

Keywords: nanometer powder, Barium titanate, solvent-thermal method, dielectric property

1 INTRODUCTION

Barium titanate is increasing importance in the preparation of dielectric capacitors or transducers due to its high dielectric constants, ferroelectric properties, piezoelectric properties and positive temperature coefficient (PTC) effect. Barium titanate exhibits unique features that strongly differ from those of bulk phase, such as switch of Curie temperature and increase in dielectric features that strongly differ from those of bulk phase, such as switch of Curie temperature and increase in dielectric constant, and BaTiO$_3$ can be used to prepare nano-BaTiO$_3$ powder. By comparison with ordinary ceramic, the ceramic with nano-BaTiO$_3$ from alcohol-thermal method could lower sintering temperature from 1300°C to 1150°C, change Courie point to 70°C, make dielectric constant 20000.

2 INTRODUCTION

H$_2$TiO$_3$ was scattered in alcohol at room temperature (marked A); Ba(OH)$_2$ was scattered in alcohol in a closed bottle at 79°C (marked B). Aqueous ammonia and A were gradually added to B respectively, which were stirred and refluxed for some time at 79°C. The suspension were cooled to room temperature, separated by centrifugation, washed with the acid, distilled water and alcohol, dried at ambient temperature. Thus the BaTiO$_3$ powders were gained.

BaTiO$_3$ powders were characterized with TEM (JEM-2010 model, JEOL Corporation, Japan) and X-ray diffraction (XRD) model D/MAX-ⅡⅡⅡ, Tokyo, Japan. BaTiO$_3$ Capacitor was characterized with SEM (SEM-5900LV, JEOL Corporation) and the capacitance at different temperature was tested by LCR apparatus (Tianjin No. 6 radio Factory, China).

3 RESULTS AND DISCUSSION

3.1 The Effect of Ba/Ti Molar Ratio

When the reaction is carried out at pH=1, 79°C, 6h, the effect of Ba/Ti molar ratio in the precursors on the products is shown in Fig.1.

It can be seen from Fig.1 that with the increase Ba/Ti molar ratio in the precursors, the Ba/Ti molar ratio in the powders all the while increased. The XRD patterns of the sample prepared by the different moral ratio of Ba/Ti in reactants at 79°C for 6h were showed in Fig.2.
The reactants molar ratio of Ba/Ti is affected by the constitute of products. To obtain pure BaTiO$_3$, it is necessary to control reactants molar ratio of Ba to Ti, that is the optimum Ba/Ti moral ratio in the precursors is 1.0.

The reaction process abides by the following steps:

\[
\begin{align*}
H_2TiO_3 + Ba(OH)_2 & \rightarrow BaTiO_3 + 2 H_2O \\
H_2TiO_3 + BaTiO_3 & \rightarrow BaTi_2O_5 + H_2O
\end{align*}
\]

3.2 The Effect of Reaction Time on Products

Table 1 showed the effect of reaction time on products. It is clearly seen that Ba/Ti molar ratio and the content of BaTiO$_3$ in the products increased with the increase of reaction time from table 1. The increase extent was not large after 6h, which indicated the reaction can complete when the reaction is 6h.

<table>
<thead>
<tr>
<th>Time(h)</th>
<th>Ti/Ba in product</th>
<th>BaTiO$_3$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.32</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>0.48</td>
<td>30.8</td>
</tr>
<tr>
<td>5</td>
<td>0.98</td>
<td>80.4</td>
</tr>
<tr>
<td>6</td>
<td>1.01</td>
<td>92.1</td>
</tr>
<tr>
<td>10</td>
<td>1.02</td>
<td>93.5</td>
</tr>
</tbody>
</table>

(reactants moral ratio of Ba / Ti:1.0, temperature:79°C)

Table 1: The effect of reaction time on powder

3.3 TEM Analysis

Fig.2.a is the XRD patterns of sample from reactants moral ratio of Ba to Ti: 1.0, it is clear that all diffraction peaks can be indexed to the cubic structure of BaTiO$_3$. Fig.2.b is the XRD patterns of sample from reactants moral ratio of Ba to Ti: 0.8, there are peaks of BaTi$_2$O$_5$ (monclinic) and BaTiO$_3$ (cubic), which suggest the reactants molar ratio of Ba to Ti affected the constitute of products. To obtain pure BaTiO$_3$, it is necessary to control reactants moral ratio of Ba to Ti, that is the optimum Ba/Ti moral ratio in the precursors is 1.0.

The reaction process abides by the following steps:

\[
\begin{align*}
H_2TiO_3 + Ba(OH)_2 & \rightarrow BaTiO_3 + 2 H_2O \\
H_2TiO_3 + BaTiO_3 & \rightarrow BaTi_2O_5 + H_2O
\end{align*}
\]
TEM photos showed that the BaTiO$_3$ nanoparticles were square in shape, the size was about 50nm. Particle size distribution was in narrow range, and no agglomerates were observed.

### 3.4 SEM Analysis

The micrograph of BaTiO$_3$ ceramic at different sintering temperature are showed in figure 4.

![SEM micrograph of BaTiO$_3$ ceramic](image)

1150°C

1200°C

1250°C

Figure 4: SEM micrograph of BaTiO$_3$ ceramic

It can be seen that the diameter of BaTiO$_3$ in ceramic increased with increasing temperature, which conform with the relationship between sintering temperature and the diameter.

SEM photos showed that the particle size in BaTiO$_3$ ceramic sintered at 1150°C was about 200nm, Particle size distribution was in narrow range.

### 3.5 Dielectric Constant of BaTiO$_3$ Capacitor

![Dielectric constant vs Temperature](image)

Figure 5 Temperature dependence of dielectric constant of BaTiO$_3$ capacitors

The relationship between temperature and dielectric constant is nonlinear, which indicated the ceramic sintered at 1150°C from nano-BaTiO$_3$ is ferroelectric ceramic. By comparison with ordinary BaTiO$_3$ ceramic capacitor, the ceramic capacitor fabricated by nano-BaTiO$_3$ could not only lower sintering temperature from 1300°C to 1150°C, but also change Courier point from 130 to 70°C, improve dielectric constant from 6000 to 20000, which can satisfy the development of microdevices.

### 4 CONCLUSION

When Ba/Ti moral ratio in the precursors is 1.0 and the reaction time is 6h, the reaction of synthesis pure BaTiO$_3$ has completed. one step synthesis BaTiO$_3$ particles without sintering are cubic structure and the size is 50nm. The ceramic capacitor fabricated by this kind of BaTiO$_3$ is ferroelectric ceramic, sintering temperature is 1150°C, Courier point is 70°C, dielectric constant at Curie point is 20000.

### REFERENCE


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