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Effect of Sintering Temperature and Soaking Time on the Physical and Dielectric Properties of $BaTiO_3$ for Different Ba/Ti Ratios

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Abstract

Recent research has examined the improvement of physical and dielectric properties of $BaTiO_3$ ceramic material by small addition of excess TiO_2 or $BaCO_3$. The prepared samples sintered at different temperatures and varying soaking time. The results show that increasing the sintering temperature within 1350°C and soaking time of 10 hrs gives better electrical and physical properties, which indicate the reaction is complete at higher temperature and period.

Keywords: sintering, soaking, barium titanate.

Introduction

Piezoelectricity is one property of a group of dielectric materials called ferroelectrics. These materials are characterized by a domain structure that can be modified by an electric field. The piezoelectric effect manifests as a spontaneous potential difference across the opposite faces of a volume of material when under an applied stress. This potential is proportional directly to the mechanical stress applied to it. The inverse is also true, i.e., application of an electric field causes strain in a volume of piezoelectric material [1].

The first piezoelectric material discovered was Calcium Titanate, which has the perovskite structure. A traditional commercial example is Barium titanate, which is commonly used in capacitors and transducers due to its high permittivity [2]. The reaction between $BaCO_3$ and TiO_2 proceeds via several intermediate stages of which formation of $BaTiO_4$ is the most distinct. The grain size of $BaTiO_3$ ceramic is controlled by inhibition the grain growth by using an excess amount of TiO_2 as a second phase which behaves as inhibitor [3]. The stoichiometric powders can be obtained by adjusting the Ba/Ti ratio, where the higher Ba/Ti ratio leads to the incorporation of excess Barium in the powder [4]. It is expected that optimum sintering behavior and electric properties are obtained, with stoichiometry range 0.995 - 1.00 [5].

Experimental work

The piezoceramic material barium titanate ($BaTiO_3$) was prepared by the reaction of a suitable grade of $BaCO_3$ with TiO_2. These raw materials are weighed out in four different ratios (1.03/1, 1/1, 1/1.03, 1/1.003) according to the stoichiometric equation below:

$$BaCO_3 + TiO_2 \rightarrow BaTiO_3 + CO_2 \qquad (1)$$

For each run the raw materials mixed and milled in an agate ball mill for 8 hrs and then calcined the result powder at 1200°C for 8 hrs.

The calcined powder milled and sieved in an 38 □m sieve, then weighed a proper amount and press it after mixing with PVA binder, in a rod with 1 cm diameter using 2.5 ton load. These samples then sintered in air at different temperatures (1200, 1300, 1350°C) for different periods (2, 4, 6, 8, 10 hrs). The bulk density measured and the true porosity is calculated by the relation:

$$\rho_{bulk} = \rho_{H_2O}\left[\frac{dry\ mass}{impregnated\ mass - immersed\ mass}\right] \qquad (2)$$

Since the density of water equal to 1 g/cm^3. then:

$$T.P\% = \left[1 - \frac{bulk\ density}{true\ density} \times 100\right] \qquad (3)$$

The true density for $BaTiO_3$ is 6.02 g.cm-3 [5] and the relative dielectric constant (K) may be measured by using LCR meter for the electroded samples:

$$K = \frac{c.d}{\varepsilon_o A} \qquad (3)$$

Where c is the capacitance of the prepared samples (in Farad), d is thickness of the sample (in m), εo is the permittivity of vacuum = 8.85*10-12 $C^2/N.m^2$, and A is the cross-sectional area of samples (m).

Results and Discussion

Bulk density

Results shows that the sintering temperature and soaking time has a remarkable influence on the bulk density of the prepared $BaTiO_3$ for the two Ba/Ti ratio's as shown in Fig. 1, and Fig. 3. Above 1300°C sintering causes discontinuous grain growth by increased diffusion in the liquid phase. A number of different compounds are possible with either excess of TiO_2 or $BaCO_3$. Under the condition of excess titania, a liquid phase is formed to enhance sintering and this has been reported experimentally in Fig. 1 and Fig. 3 [3].

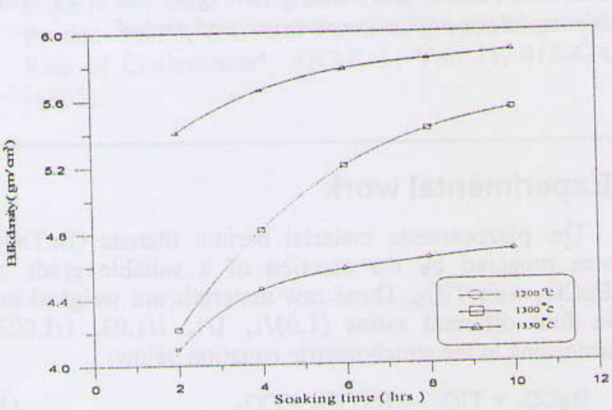

Fig. 1 Bulk density of $BaTiO_3$ samples sintered at different temperatures with Ba/Ti = 0.997

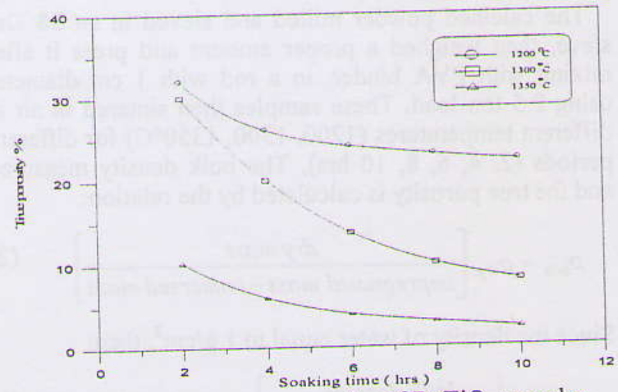

Fig. 2 Percentage true porosity of $BaTiO_3$ samples sintered at different temperatures with Ba/Ti = 0.997

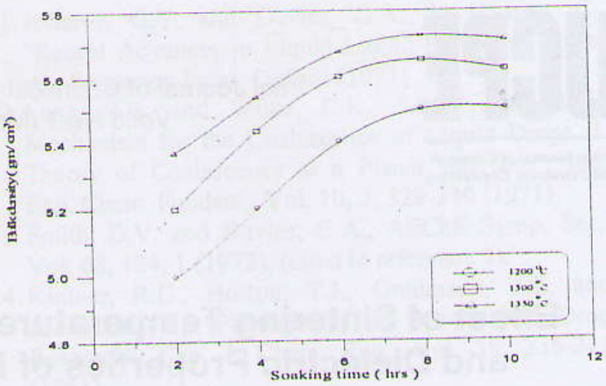

Fig. 3 Bulk density of $BaTiO_3$ samples sintered at different temperatures with Ba/Ti = 1.03

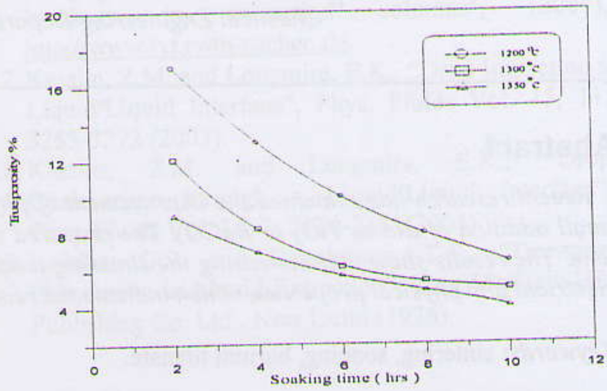

Fig. 4 Percentage true porosity of $BaTiO_3$ samples sintered at different temperatures with Ba/Ti = 1.03

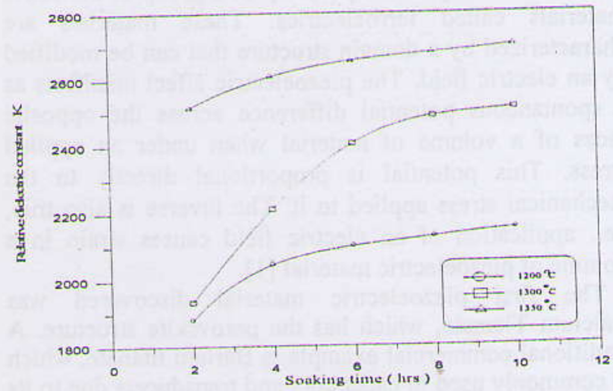

Fig. 5 Dielectric constant (measured at 10 KHz) as a function soaking time for $BaTiO_3$ with Ba/Ti = 0.997

True porosity

Porosity for the sintered $BaTiO_3$ with different sintering temperature and soaking time for the two cases of Ba/Ti (0.997, 1.03) was calculated in the percentage law using true density of $BaTiO_3$ (6.02 gm/cm^3) and plotted in Fig. 2 and Fig. 4.

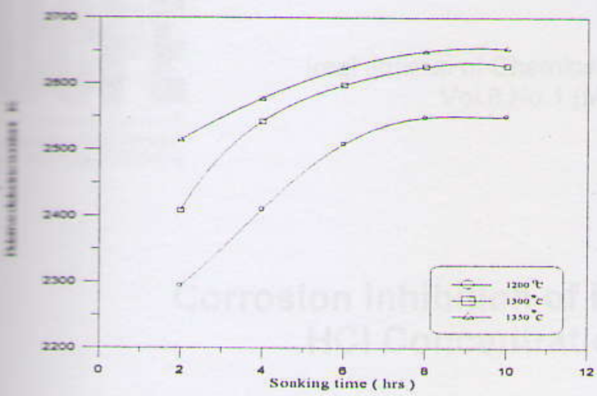

Fig 6 Dielectric constant (measured at 10 KHz) as a function soaking time for BaTiO3 with Ba/Ti = 1.03

Dielectric constant

Dielectric constant of $BaTiO_3$ is extremely sensitive to Ba/Ti ratio, sintering temperature, and soaking period as shown in Fig. 5 and Fig. 6, in which the dielectric constant increased as the sintering temperature increased, it reaches a maximum value at 1350°C at 8-10 hrs, and it differs slightly after this temperature.

Conclusions

1. Sintering below 1350°C is advised to prevent production of reaction phases. However, high temperature is required to improve the sintered density by thermal activation.
2. The physical and dielectric properties influence by the Ba/Ti ratio, sintering temperature and the soaking time.
3. Increasing TiO_2 in a small proportion effect largely the physical and dielectric properties of the resulted piece.
4. Increasing $BaCO_3$ in a quantity larger than that of TiO_2 also effect the physical and dielectric properties of $BaTiO_3$. But the small addition of TiO_2 effect is more effective than that of larger amount of $BaCO_3$.

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