

PREPARATION, CHARACTERIZATION, AND PROPERTIES OF BARIUM TITANATE

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ABSTRACT

Barium titanate ceramic materials were prepared from high purity barium carbonate ($BaCO_3$) and titanium dioxide (TiO_2) powders. These powders were mixed first, then calcinated at different temperatures (1000, 1200, 1350 °C) for periods (4, 8, 12, 16 hr). After that milled to fine powder and pressed in a form of disk with dimensions (20 mm x 5 mm) then sintered at three different temperatures (1200, 1350, 1420 °C) for 12 hr.

Calcinated powder phases were investigated using X-ray diffraction technique. The results indicates that the best sintering temperature was 1420 for 12 hr. Ba/Ti ratio of 1/1, 003 gave the best dielectric properties (relative dielectric constant (K) and apparent density).

INTRODUCTION

Polycrystalline barium titanate is extensively used in the preparation of high permittivity capacitors. Barium titanate ($BaTiO_3$) dielectric constant is influenced by many factors like the $BaCO_3$ - TiO_2 ratios, density, and impurities^[1]. TiO_2 is used as a second phase in excess form to control the grain growth^[2]. It is known that at the curie temperature i.e., at the cubic – tetragonal transition, the intrinsic permittivity for $BaTiO_3$ is very high. In fine grained barium titanate the high permittivity arises from the internal stresses which tend to force each grain back toward the cubic state^[3,6].

Two techniques are generally adopted to carry out the modification of $BaTiO_3$ ceramic powder to achieve high dielectric constant. The first is by self modification of $BaTiO_3$, by controlling the particle size with the best sintering temperature and by using different pressing techniques. The second modification is by addition of a certain additives in different concentrations. In this research the temperature and period of calcination have been studied. The sintering temperature change for different Ba/Ti ratios are investigated and their effect on density, dielectric constant and the dissipation factors are determined^[7].

Materials and Method of Preparation

Barium titanate ($BaTiO_3$) was prepared by using different ratios of $BaCO_3$ and TiO_2 of (1/1.2

, 1/1, 1/1.003). For each ratio the raw materials, $BaCO_3$ and TiO_2 mixed in agate ball mill for 6hr and then were calcinated at 1000, 1200, and 1350 °C for periods of 4hr, 8hr, 12hr and 16hr. Pellets of $BaTiO_3$ of 2cm diameter and 5mm thickness were prepared by pressing the precalcinated powder after grinding, by using a press of 10 tons load, with PVA binder. These pellets were then sintered in air at temperatures (1200, 1350 and 1420)°C 12 hr.

The calcinated samples before pressing were examined by x-ray diffractron technique to determine the phase of the produced $BaTiO_3$. The apparent density is determined by using the straight forward method by calculating the volume of the pellet from the dimensions, and its weight is measured by a sensitive balance, the ratio of mass to volume is the density.

The dielectric constant K and the dissipation factor ($\tan \delta$) are measured by using LCR meter for the aluminum electroded pellets,

$$K = c.d/\epsilon_0.A$$

Where c = capacitance of the prepared pellets (Farad), d = Thickness of pellets, ϵ_0 = Permittivity of vacuum = $8.85 \times 10^{-12} C^2/N \cdot m^2$, A = cross sectional area of the pellets, and $\tan \delta = 1/w.c.R$, $\tan \delta$ = Dissipation factor, R = Resistance, and w = Frequency.

RESULTS AND DISCUSSION

X-Ray Diffraction

The analysis of x-ray diffraction patterns for the calcinated $BaTiO_3$ indicate that the tetragonal $BaTiO_3$ is only the crystalline phase at Ba/Ti ratio (1/1) and (1/1.003) as in fig(1). With excess TiO_2 ($Ba/Ti = 1/1.2$) the phase is hexagonal especially at firing period (16 hr). Fig (1) shows a sample of XRD data with information given for each peak.

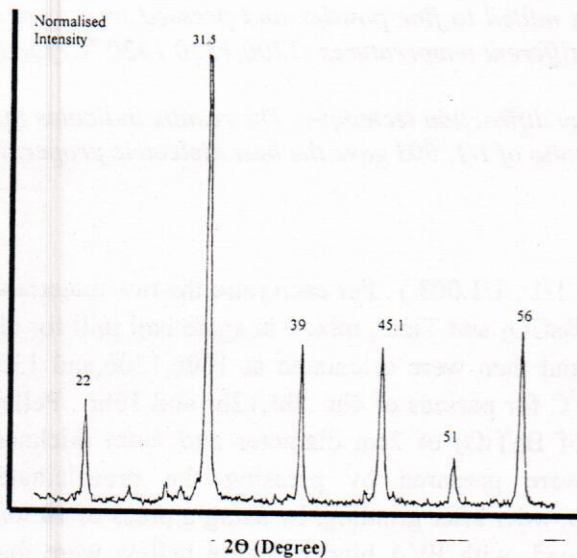


Fig. (1) XRD spectra of calcined barium titanate for Ba/Ti=1/1.003

Apparent Density

Results are showing that the firing temperature has a remarkable influence on the density of the prepared barium titanate as shown in Fig (2, 3, and 4) and table(1). This influence is governed by the mixing ratio and controlling the firing temperature. In the case of $Ba/Ti=1/1.2$ the influence is much clear.

At low temperature $1000^\circ C$, the density starts to decrease in the period of 8 to 12 hrs then nearly diminished in the period of 12-16 hr Fig (2,3,4).

For $Ba/Ti= 1/1.003$ ratio the density has the highest values and for all treating periods. The interesting results is that the density increases with firing period upto 8 hr, then decreases till 12 hrs, then starts to increase again. This can be attributed to the formation of another compounds (Ba_2TiO_4) which react with TiO_2 to produce $BaTiO_3$.

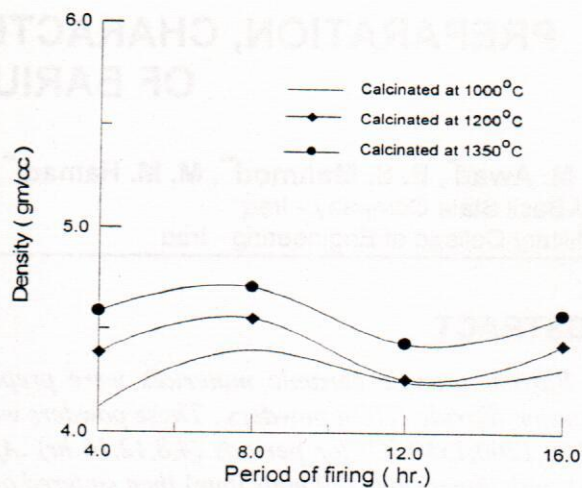


Fig. (2) Sintered density vs. calcinations period for $BaTiO_3$ at Ba/Ti=1/1.2

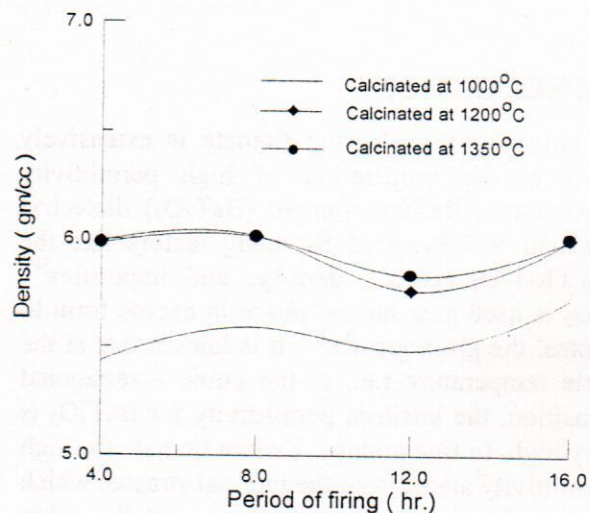


Fig. (3) Sintered density vs. calcinations period for $BaTiO_3$ at Ba/Ti=1/1.003

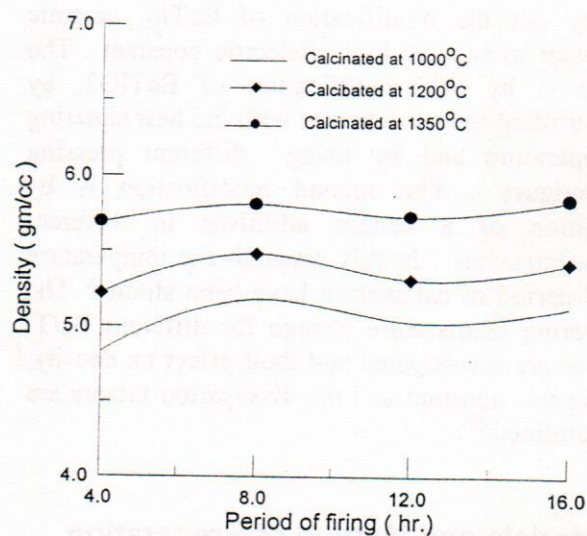


Fig. (4) Sintered density vs. calcinations period for $BaTiO_3$ at Ba/Ti=1/1

Table (1) Influence of sintering temperature on the density and dielectric properties of BaTiO₃ calcinated at 1200°C for 4hr

Property	Ba/Ti=1/1			Ba/Ti=1/1.003			Ba/Ti=1/1.2		
	Sintering Temperature (°C) 12 hr			Sintering Temperature (°C) 12 hr			Sintering Temperature (°C) 12 hr		
	1200	1350	1420	1200	1350	1420	1200	1350	1420
Density gm/cc	4.56	4.75	5.22	5.24	5.43	5.991	4.006	4.210	4.399
Dielectric Constant	1875	1890	2660	3685	4120	6712	1103	1209	1340
Loss Factor Tan δ	0.50	0.35	0.20	0.40	0.25	0.13	0.76	0.62	0.57

Dielectric Properties

Dielectric Properties of BaTiO₃ in ferroelectric state are extremely sensitive to TiO₂ amount. Typical results are shown when Ba/Ti ratio = 1/1.003 is used at calcination temperature 1350 °C for 16 hr and the sintering temperature is 1420°C.

This result suggests that at Ba/Ti ratio of 1/1.003 the grain growth was inhibited. This is in good agreements with literature^[4].

CONCLUSIONS

- 1-The dielectric properties of BaTiO₃ and its density are influenced by the preparation conditions, calcination temperature, period of calcinations, Ba/Ti ratio and sintering temperature.
2. The best calcination temperature found experimentally to be 1350 °C for 16 hr which gave highest density and best dielectric properties.
3. The best Ba/Ti ratio was 1/1.003.
4. The best sintering temperature is 1420 °C for 12 hr.

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