

Thermal treatment effect on piezoelectric and pyroelectric properties of BaTiO₃ ceramics

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ABSTRACT

Structural, piezoelectric and pyroelectric characteristics of sol gel processed BaTiO₃ ceramics calcined at different temperatures (500, 600, 700, 800, 900 and 1000 °C) and sintered at 1100 °C for 8 hours were investigated. The effect of heat treatment (calcinations) on the piezoelectric coefficient (d_{33}) and the voltage constant (g_{33}) was shown to be similar to that on the mechanical quality factor (Q_m). The values of d_{33} , g_{33} and Q_m increase with increasing temperature and reach their maximum at 1000°C ($d_{33}=79.16$, $g_{33}=28.01$ and $Q_m=199$). The results revealed that the full width at half maximum (FWHM) of the bands B₁ and E(TO₃+ LO₂) has high value and is essential for obtaining BT ceramics having good properties. Copyright © 2015 VBRI Press.

Keywords: Structural characteristics; full width at half maximum (FWHM); piezoelectric parameters; pyroelectric.

Introduction

The micro technology has experienced a significant growth since the fifties. Hence, manufacturing techniques have evolved and have contributed to a reduction in production cost, and nowadays many applications incorporate small devices. Indeed, the actuators and sensors are a class of these devices, which utilize piezoelectric materials as active elements in medical imaging (scan) and velocimetry (piezoelectric motors, sonar sensor, electromechanical filters, low-power ultrasonic transducers injection mechanisms, sonar and ultrasonic cleaning tanks) [1-4]. Besides, the use of the piezoelectric properties of certain materials [5] is a very promising source of alternative energy harvesting methods [6]. Energy harvesting is the process of extracting, converting and storing energy from the environment that can also be described as a response of smart materials when they are subjected to an external stimulus such as pressure, vibrations, motion and temperature [7-10]. Consequently, piezoelectric materials are particularly interesting compounds for this type of application. In particular, lead oxide based ceramics are among the most used materials.

There are three widely used and studied systems: the PZT (Lead zirconate titanate) known for their large piezoelectric constants at relatively high temperature, the PMN-PT and the PZN-PT [11-27]. However, the toxicity of lead has raised concerns about lead-based piezoelectric materials [28], and results related to piezoelectric and pyroelectric properties of lead free BaTiO₃ (BT) based materials (lead free materials) are rare. Therefore, they meet the environmental requirements and are considered

standard materials for studies of the properties of pyroelectricity and piezoelectricity of, in particular, barium titanate [29-31], and hence development of piezoelectric and pyroelectric lead free devices is essential. However, literature survey reveals that the relationship between the Raman vibrational modes and pyroelectricity for BT ceramics have not been reported so far.

It should be noticed that BaTiO₃-based ceramics are usually fabricated by the conventional solid-state method. Recently, considerable efforts have also been devoted to the synthesis of materials by various wet chemical methods as it was found that piezoelectric ceramics made from powders synthesized by alternative methods exhibit improved sinterability, poling property and piezoelectric performance, because of their higher homogeneity of chemical composition with well dispersed fine particles and/or unique morphologies. The aim of the present paper is to study the relationship between Raman vibrational modes and piezoelectric and pyroelectric properties of BT ceramics, and to point out the role played by heat treatment on these properties. At this end, we examined the structure and microstructure of a series of sol gel processed BT ceramics, calcined at different temperatures, using X-ray diffraction (XRD) and Infrared IR techniques, the scanning electron microscopy (SEM) and Raman spectroscopy. In particular, the pyroelectric and piezoelectric constants, the full width at half maximum (FWHM) for two bands of Raman spectra and the mechanical quality factor were calculated and their behavior as a function of heat treatment was studied.

Experimental

Material synthesis

In our preparation we have used barium acetate trihydrate ($\text{Ba}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$), titanium alkoxide $\text{Ti}[\text{OCH}(\text{CH}_3)_2]_4$, and lactic acid ($\text{CH}_3\text{CH}(\text{OH})\text{COOH}$) as peptizing agent and distilled water as a solvent. The first step consisted in preparing a colloidal solution of titanium oxide TiO_2 , with molar ratios of alkoxide/lactic acid equal to 2, and of alkoxide/water equal to 3 to have a stable titanium sol. The preparation of titanium sol of 500 ml to 1 mol/L required 143.5 g of titanium alkoxide, 22.5 g of lactic acid and 300 g of water corresponding respectively to 0.5, 0.25 and 16.66 mol. The aqueous solution of lactic acid was stirred at 60 °C for few minutes. Then, the titanium alkoxide was added carefully and quickly to the mixture to avoid the hydrolysis of the alkoxide (very sensitive to moisture) as much as possible. An excess of titanium of 1% was added to compensate losses in Ti due to hydrolysis. The resulting mixture, under continuous agitation, gradually transited from a milky solution to a fully transparent sol. The final sol was adjusted with distilled water to 1 mol/L of Ti. Barium acetate and Ti were mixed in stoichiometric proportions, depending on the chemical formulation BaTiO_3 under agitation for 5 min. The destabilization of this solution was achieved by evaporation of the solvent in an oven at the temperature of 60 °C for 72 h (Fig. 1). The xerogel obtained was grinded in an agate mortar. Raw powder, after grinding, was calcined in air at different temperatures (500, 600, 700, 800, 900 and 1000 °C) for 4 h, in a programmable oven.

These compositions were then pressed into pellets of 12 mm diameter and a thickness of 1 mm under a uniaxially pressure equal to 10 tons/cm². The pelletized samples were finally sintered at 1100 °C for 8 h in a programmable furnace. Silver paste was added to both faces of the disks. These compositions were then pressed into pellets of 12 mm diameter and a thickness of 1 mm under a uniaxially pressure equal to 10 tons/cm². The pelletized samples were finally sintered at 1100 °C for 8 h in a programmable furnace. Silver paste was added to both faces of the disks, which were then fired at 60 °C as electrodes.

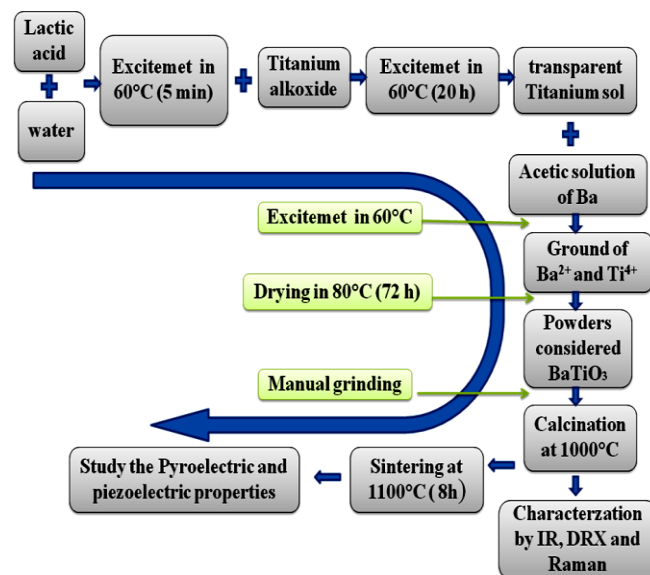


Fig. 1. Flowchart of the BS_xT synthesis by the sol gel process.

Measurements and characterizations

The obtained powders were characterized by X-ray diffraction (DRX), Raman spectroscopy, and Fourier transform infrared (IR). The determination of the piezoelectric parameters (d_{33} and g_{33}) is based on the measurements of the resonance and antiresonance frequencies, using an Agilent 4294A precision impedance analyzer (Hewlett-Packard, Palo Alto, CA). Dielectric characterization was performed as a function of the frequency from 1 kHz up to 2 MHz. These dielectric measurements were carried out in the temperature range of 30–225 °C and under sub weak level of excitement (1v). BT ceramics samples were polarized using a dc electric field of 0.1kV mm⁻¹ (30–225 °C). To remove any residual space charge the two electrodes were short-circuited for 20 minutes.

Results and discussion

IR characterization

The IR spectra of the BT depending on the temperature of calcinations are plotted in Fig. 2. The large absorption band at 1396 cm⁻¹ can be interpreted as the vibration of C=O groups because of small traces of inevitable carbonate, which will be confirmed by X-ray diffraction spectra (Fig. 3). A band in low frequencies domain (512 cm⁻¹) was observed, from the vibration of the metal -oxygen- metal group (MOM) from the amorphous powder [32]. It disappears at 1000 °C, indicating the formation of the octahedron (Ti)O₆ [33] characteristic of the good crystallization of the powder [34]. The bands located at 3626 and 3732 cm⁻¹ associated with the OH group (water stretching vibration) disappear at 1000 °C. All these indicate that most organic groups involved in the precursor are decomposed and disappear with heat treatment.

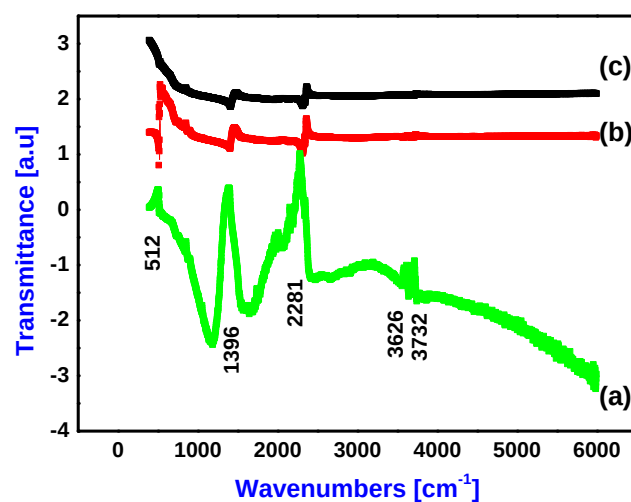


Fig. 2. IR spectrum of pure BT calcined to 600 (a), 900 (b) and 1000 °C (c).

X-ray diffraction studies

Fig. 3 shows the XRD spectra corresponding to the BaTiO_3 powder calcined at different temperatures from 500 to 1000 °C, for 4 hours. These spectra indicate the evolution of the reaction to permit the formation of the BT powder

depending on the temperature. The XRD pattern of the BT powder calcined at 500 °C is substantially amorphous; excluding high diffraction peaks (diffuse) which was identified as peaks of reflection characteristic of the TiO_2 precursor. No crystalline phase is apparent. The peak located around 600 °C assigned to the TiO_2 phase disappears completely and we note also the emergence of reflection peaks characteristic of the BT -phase. The powder is then totally crystallized in the perovskite phase at 600 °C with the presence of a secondary phase (BaCO_3) at $2\theta=24^\circ$, which decreases with increasing temperature. On the other hand the crystallization peaks of the perovskite phase become narrow and intense with increasing temperature, indicating that the phase is more crystalline at 1000 °C.

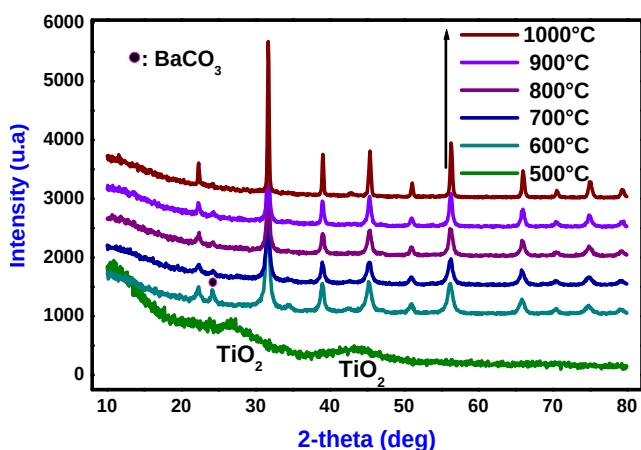


Fig. 3. XRD patterns of the poled BaTiO_3 ceramics calcined at 500 1000 °C.

Microstructure and surface morphology

Fig. 4 shows microstructures of BT ceramics sintered at 1100 °C for 8 h. The grain size was determined by averaging over the total number of grain sizes. the grains formed have a relatively homogeneous size fairly regular, and has circular or quasi-spherical.

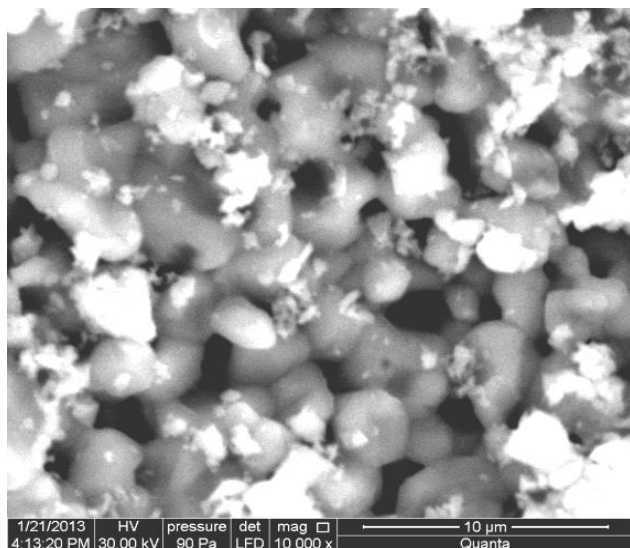


Fig. 4. The scanning electron micrographs recorded on the surfaces of BT ceramics.

Raman characterization

The Raman spectra of the calcined BT powder at various temperatures are shown in Fig. 5. Between 200 and 300 cm^{-1} , three modes are observed- $A(\text{TO}_1)$, $A(\text{LO}_1)$ and $A(\text{TO}_2)$ - at 205, 237 and 287 cm^{-1} respectively; however, the increase of the temperature of calcinations causes the progressive decrease in the intensity until the occurrence of a single broad band at 1000 °C. One can also observe that there is a narrow band at around 305 cm^{-1} associated with the modes B_1 and $E(\text{TO}_3+\text{LO}_2)$, and a wide and asymmetrical band around 520 cm^{-1} associated with the modes $A_1(\text{TO}_3)$ and $E(\text{TO}_4)$ and another wide band (not very intense) at 720 cm^{-1} associated with the modes $A_1(\text{LO}_3)$ and $E(\text{LO}_4)$. One also notes the increase in intensity of the modes $E(\text{TO}_3+\text{LO}_2)/B_1$ and $E(\text{LO}_4)/A_1(\text{LO}_3)$. Those are the consequence of the shift of frequencies of the Raman modes. Furthermore, we note the presence of a new band at 804 cm^{-1} , which was never mentioned in the literature; this band does not appear in the 1000 °C spectrum and may closely be related to the BaCO_3 secondary phase observed on the X-ray diffraction patterns. Moreover, FWHM of B/E ($\text{TO}+\text{LO}$) mode increases with increasing temperature (Fig. 6(a)). An abrupt increasing of FWHM was observed above 900 °C. In this state, the vibration modes are affected by the fluctuations caused by the phase change, especially as the intensity of the peak at 305 cm^{-1} assigned to the modes $B_1+E(\text{TO}_3+\text{LO}_2)$, disappears when the phase transition from the ferroelectric to the paraelectric occurs.

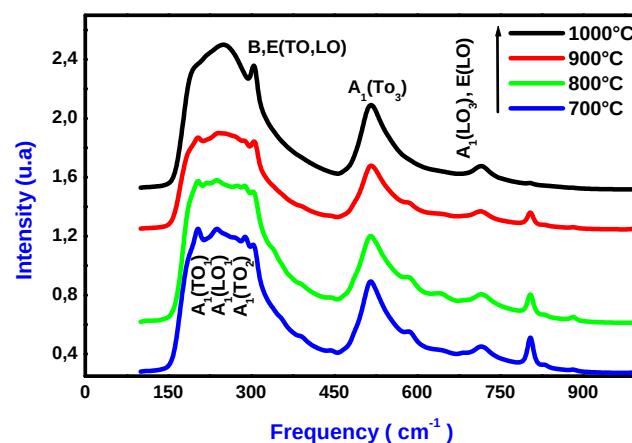


Fig. 5. Room temperature depolarized Raman spectra of the BT calcined at 700, 800, 900 and 1000 °C.

Pyroelectric and piezoelectric properties

The temperature dependence of the pyroelectric coefficient (p) was calculated from the temperature change rate (T) and the pyroelectric current (I_p) according to the relation:

$$p(T) = \frac{I_p}{S \cdot T} \quad (1)$$

where, S is the surface of an electrode. The result of the calculation is graphically shown in Fig. 6(b). In this figure, it is seen that the pyroelectric current p shows a broad peak at a lower temperature, T_C , which is a characteristic of the paraelectric-to-ferroelectric phase transition, and then

decreases to zero at about 480 K. The pyroelectric coefficient p takes the value $150 \mu\text{C cm}^{-1} \text{K}^{-2}$ at T_C and remains almost constant with the increase of the temperature, then it increases rapidly to $540 \mu\text{C cm}^{-1} \text{K}^{-2}$ to 1000°C . This is closely related to the sudden increase of the FWHM of the bands B_1 and E ($\text{TO}_3 + \text{LO}_2$) which we have mentioned above (Fig 5).

The determination of the piezoelectric parameters is based on the measurements of the resonance and anti-resonance frequencies of the sample. The latter were calculated using the following formulas [35, 36]:

$$K_p^2 = \frac{\eta^2 - (1 - \alpha^{E^2})}{2(1 + \alpha^E)} * \frac{f_a^2 - f_r^2}{f_a^2} = \frac{2.51}{2} * \frac{f_a^2 - f_r^2}{f_a^2} \quad (2)$$

(Planar coupling coefficient)

$$d_{33} = K_p \sqrt{\frac{(1 - \alpha^E) \varepsilon_r \varepsilon_0}{2E}} (C/N) \text{ at } 25^\circ\text{C} \quad (3)$$

(Piezoelectric constant)

$$E = \left[\frac{\pi \Phi f_r}{\eta} \right]^2 * (1 - \alpha^{E^2}) * d(N/m^2) \quad (4)$$

(Young's modulus)

$$g_{33} = \frac{d_{33}}{\varepsilon_0 \varepsilon_r} \quad (5)$$

(Voltage constant)

where:

Φ : Sample diameter (m)

α^E : Fish coefficient (0.31 for ceramics)

η : The root of the Bessel equation ($\eta=2.05$)

D : Density (kg/m^3)

ε_0 : Permittivity of the empty = 8.85×10^{-12} (F/m)

ε_r : Relative dielectric permittivity

f_r : Resonance frequency

f_a : Antiresonance frequency

The evolution of the piezoelectric parameters depending on the heat treatment for the five compositions sintered at 1100°C is shown in Fig. 6(a). It is observed that these coefficients increase with increasing temperature. However, from these results, piezoelectric parameters show a linear increase with temperature and they remain almost constant above 900°C , while the pyroelectricity increases rapidly from this temperature, although this increase is very weak between 600 and 900°C . Therefore, it can be concluded that the FWHM of the high bands B_1 and E ($\text{TO}_3 + \text{LO}_2$) is essential for obtaining BT ceramics having good pyroelectric properties, however the enhancement of the piezoelectric is not the only factor that affects the pyroelectric properties.

The mechanical quality factor Q_m measures the mechanical losses in the ceramic [37, 38], and may be calculated using the following equation.

$$Q_m = \frac{1}{4\pi RC(f_a - f_r)} \quad (\text{The mechanical quality factor}) \quad (6)$$

where, C : Capacity (F), R : Resonance resistance (Ohm)

The evolution of the mechanical quality factor (Q_m) as a function of the heat treatment is the same as that observed

for d_{33} and g_{33} . That is to say, there is an increase of the Q_m factor with increasing temperature of calcinations, which takes the maximum value at the temperature of 1000°C (Fig. 6a). Concerning the values found for this factor, the BT offers more attractive characteristics.

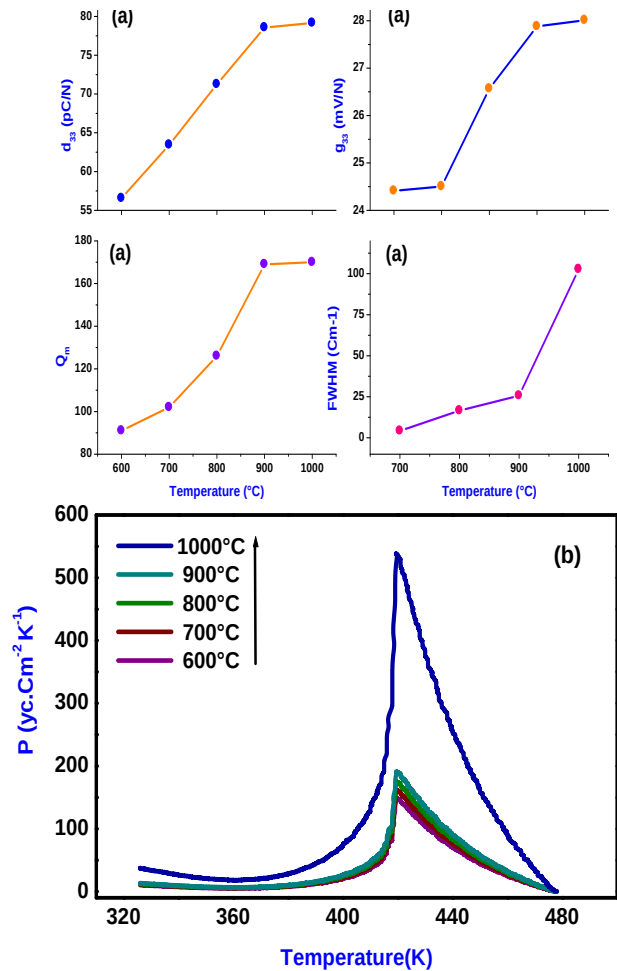


Fig. 6. (a) Piezoelectric constant d_{33} , voltage constant g_{33} , the mechanical quality factor (Q_m), variation of the Raman FWHM for the band $B_1/E(\text{TO}_3 + \text{LO}_2)$; (b) Pyroelectric coefficient of the BaTiO_3 ceramics as functions of the calcination.

Conclusion

Structure, microstructure, pyroelectric and piezoelectric properties of a series of sol gel processed BT ceramics were studied as functions of heat treatment. In particular, a pure perovskite phase, without the presence of any secondary phase, was obtained at a temperature of 1000°C . Moreover, this study shows that the increase of the temperature of calcinations leads not only to a good crystallization of the BaTiO_3 samples, but also to a large increase in the piezoelectric coefficients d_{33} and g_{33} . A similar behavior under heat treatment was also observed for the calculated full width at half maximum and mechanical quality factor. On the other hand, the results obtained are important particularly in terms of the relationship between the piezoelectric parameters, pyroelectricity and the Raman vibrational modes. Indeed, it was observed that the gradual emergence as a function of temperature of the band inherent to the mode $B_1/E(\text{TO}_3 + \text{LO}_2)$, which was followed

via the estimation of the corresponding FWHM, is a sign of an enhancement of the pyroelectric properties of the BT ceramics.

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