

CERAMICI $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ DOPATE CU Nb^{5+} , Zr^{4+} SAU Eu^{3+} $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ CERAMICS DOPED WITH Nb^{5+} , Zr^{4+} OR Eu^{3+}

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The ceramics of Nb^{5+} , Zr^{4+} or Eu^{3+} doped $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ complex perovskite oxide were obtained by the conventional solid-state reaction method, being sintered in air, at temperatures between 1400 and 1600 °C, for 2 h. X-ray diffraction, scanning electron microscopy and dielectric measurements were used for the compositional, structural, morphological and dielectric characterization. All $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ ceramics present single-phase compositions after removing the surface layer in which zinc volatilization occurred. Furthermore, the 1:2 ordering of zinc and tantalum cations on the octahedral positions of the perovskite structure was observed for all sintering temperatures. The microstructure of $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ ceramics is strongly influenced by the dopant type, in terms of grains size and shape. The dielectric constant varies between 16.9 and 31.5. $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ resonator doped with 1.00 % Nb_2O_5 and sintered at 1600 °C, 2 h, exhibits the highest value for the quality factor, $Q \times f \sim 157$ THz at 10 GHz.

Ceramicile oxidului perovskitic complex $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ dopat cu Nb^{5+} , Zr^{4+} sau Eu^{3+} au fost obținute prin metoda convențională a reacțiilor în fază solidă, fiind sinterizate în aer, la temperaturi între 1400 și 1600 °C, 2 h. Pentru caracterizarea compozițională, structurală, morfologică și dielectrică au fost utilizate difracția de raze X, microscopia electronică de baleaj și măsurătorile dielectrice. Toate ceramicile $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ prezintă fază unică după îndepărtarea stratului superficial în care a avut loc volatilizarea zincului. În plus, a fost observată ordonarea 1:2 a cationilor de zinc și tantal pe pozițiile octaedrice ale structurii perovskitice pentru toate temperaturile de sinterizare. Microstructura ceramicilor $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ este puternic influențată de tipul dopantului, în ceea ce privește dimensiunea și forma granulelor. Constanta dielectrică variază între 16,9 și 31,5. Rezonatorul $\text{BaZn}_{1/3}\text{Ta}_{2/3}\text{O}_3$ dopat cu 1,00 % Nb_2O_5 și sinterizat la 1600 °C, 2 h, prezintă cea mai mare valoare pentru factorul de calitate, $Q \times f \sim 157$ THz la 10 GHz.

Keywords: dielectric, perovskite, microwave resonator

1. Introduction

Barium tantalate based complex perovskites are very attractive solutions for microwave applications due to their very low dielectric loss. These materials are used as dielectric resonators for fabricating wireless communication devices, which demand the miniaturization of the size and weight, as well as temperature stability. The microwave devices miniaturization is very important for filters, antennas and oscillators used in the communications field at frequencies higher than 10 GHz. [1-3]

$\text{Ba}(\text{Zn}_{1/3}\text{Ta}_{2/3})\text{O}_3$ (BZT) exhibits a moderate dielectric constant ($\epsilon_r \sim 28$), a high quality factor ($Q \times f \sim 168$ THz) and a low temperature coefficient of the resonant frequency ($\tau_f \sim 0.5$ ppm/°C) [1]. It presents either a disordered-type structure (Zn and Ta cations are arranged in a random way over the B site) with a cubic symmetry ($Pm\bar{3}m$ space group) or a 1:2 ordered-type structure (Zn and Ta cations are arranged in Zn-Ta-Ta sequences) with a hexagonal symmetry ($P3m1$ space group) [1, 4].

Galasso [4] described the evolution of the ordered and disordered phases in terms of the nucleation and growth of small ordered domains with increasing annealing time and temperature. The ordering behaviour of B cations and the ceramic microstructure have a pronounced effect on the dielectric loss at microwave frequencies [1, 5]. Moreover, sintering at temperatures above 1500 °C or an extended thermal treatment leads to ZnO volatilization from the surface of BZT samples, which determines the formation of zinc-deficient phases, such as $\text{Ba}_8\text{ZnTa}_6\text{O}_{24}$, $\text{Ba}_3\text{Ta}_2\text{O}_8$ or BaTa_2O_6 , phases that degrade the quality factor [1, 5, 6]. Bieringer *et al.* [7] proposed a mechanism in which a fully ordered BZT phase grows at the expense of a slightly Zn-deficient and partially ordered phase. As a consequence, preparation of such complex dielectric materials requires special thermal treatments in order to control the densification, microstructure, cation ordering and secondary phases presence and to enhance the quality factor. The highest three values reported for

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the $Q \times f$ product of BZT resonators were: ~ 236 THz (doping with V₂O₃) [8], ~ 210 THz (solid solution with (Sr_{0.25}Ba_{0.75})(Ga_{1/2}Ta_{1/2})O₃) [9] and ~ 200 THz (adding BaWO₄) [10].

The common method for preparing BZT ceramic is the conventional solid-state reaction technique. However, its sinterability is poor [1, 6, 11, 12] and many investigations have been carried out in order to reduce the sintering parameters without affecting the microwave dielectric properties. Several authors reported improvements in sinterability by using sintering aids or dopants or by adding glasses [1, 8, 13-16]. Soft chemistry techniques have been also used, but, in most cases, the microwave dielectric properties values were diminished [17-19].

In this work, we report on the synthesis and characterization of Nb₂O₅, ZrO₂ or Eu₂O₃ doped BZT ceramics. The dopants were added in order to improve the densification, granular growth and $Q \times f$ product of BZT resonators. The sinterability, phase composition, crystalline structure and microwave dielectric properties were investigated and correlations were established.

2. Experimental

BaZn_{1/3}Ta_{2/3}O₃ (BZT) ceramics were prepared by the conventional solid-state reaction method, using barium carbonate (BaCO₃; Fluka; > 99 %), zinc oxide (ZnO; Fluka; ≥ 99 %) and tantalum pentoxide (Ta₂O₅; Alfa Aesar; 99.993 %) as starting materials. The precursor powders were weighted according to BZT stoichiometry, homogenized by ball milling in ethanol, for 24 h, using zirconia jar and balls, then dried at 80 °C. The dry precursor mixture was uniaxial pressed at 150 MPa into discs and calcined at 1310 °C, for 2 h, in air, then the discs were ball milled and dried in the same conditions as mentioned earlier. The resulting powder and the selected dopants were weighted according to the desired composition (0.25, 0.50, 0.75 or 1.00 mol % dopant), homogenized and dried in the same conditions as mentioned earlier. Niobium pentoxide (Nb₂O₅; Alfa Aesar; 99.9985 %), zirconium dioxide (ZrO₂; Aldrich; 99.99 %) and europium trioxide (Eu₂O₃; Aldrich; 99.9 %) were used as dopants. The resulting powders were granulated using a polyvinyl alcohol solution and uniaxial pressed at 150 MPa into pellets. The pellets were sintered at 1400, 1500 or 1600 °C, for 2 h, in air.

BZT ceramics were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). A Shimadzu XRD-6000 diffractometer was used to identify the phase composition and crystal structure. The microstructure of BZT ceramics was analyzed with a Hitachi S-2600N scanning electron microscope. The XRD and SEM investigations were performed after removing a layer of about 1 mm thickness from the surface of the sintered samples through disc cutting. This operation was executed in order to eliminate the layer in which ZnO volatilization occurred during the sintering process, phenomenon that leads to secondary phases formation. Hakki-Coleman method was applied for the microwave dielectric properties (dielectric constant and dielectric loss) measurement at room temperature. A computer aided measurement system containing an HP 8757C scalar network analyzer and an HP 8350 B sweep oscillator was used. The dielectric properties were determined on the initial BZT ceramics because of the minimum height required for the resonators measurement.

3. Results and Discussions

Regarding the dopants of BZT ceramic, there were chosen the following cations: Nb⁵⁺, with an ionic radius value identical with the one of Ta⁵⁺, Zr⁴⁺, with an ionic radius value higher with less than 15 % than the one of Ta⁵⁺, and Eu³⁺, with an ionic radius value much different from the one of Ta⁵⁺, all cations being considered in hexacoordination with the oxygen (Table 1). As a consequence, only Nb⁵⁺ and Zr⁴⁺ can substitute Ta⁵⁺ in the perovskite structure.

The XRD patterns of some BZT ceramics doped with Nb₂O₅ or Eu₂O₃, sintered at different temperatures, are shown in figures 1a and 2. Generally, the X-ray patterns present intense and sharp diffraction peaks attributed to the perovskite structure with cubic symmetry (JCPDS 01-070-9200), as well as the weak diffraction peak from 17.7°, peculiar to the hexagonal superstructure (JCPDS 01-070-0197), which means that nanodomains with 1:2 ordered structure have nucleated and crystallized in the cubic matrix [4]. All samples exhibit a high crystallinity degree and contain 1:2 ordered nanodomains even for the lowest sintering temperature (1400 °C). Ba₈ZnTa₆O₂₄ zinc-deficient secondary phase was

Table 1

Ionic radius values of targeted cations in hexacoordination with the oxygen [20]
Valorile razei ionice a cationilor vizați în hexacoordinare cu oxigenul [20]

Element	Charge Sarcina	Anion	Coordination	Ionic Radius (Å) Raza ionică
Ta	+5	O ²⁻	VI	0.64
Nb	+5			0.64
Zr	+4			0.72
Eu	+3			0.95

identified only in the case of BZT ceramic doped with 1.00 % Nb_2O_5 and sintered at 1600 °C; the mentioned phase is a result of partial volatilization of ZnO in a deeper layer from the sample surface. It is interesting to observe the fact that in the case of BZT ceramics doped with Nb_2O_5 , a shift of 100% diffraction peak to higher angles can be identified (figure 1b). The shift increases with dopant concentration increasing from 0.25 to 1.00 %, which indicates that a part of niobium cations substitutes tantalum cations in the perovskite structure.

Using the XRD data, the order parameter (S) values were calculated (Equation 1). $[I_{100}/I_{(110,102)obs}]$ is the ration of the observed intensity of the 100 superstructure reflection to that of the 110 and 102 reflections from the sub-cell, while $[I_{100}/I_{(110,102)cal}]$ is the corresponding intensity ratio calculated for a fully ordered structure [1].

$$S = \left(\frac{I_{100}}{I_{(110,102)obs}} \right)^{\frac{1}{2}} \frac{I_{(110,102)cal}}{I_{100}} \quad (\text{Eq. 1})$$

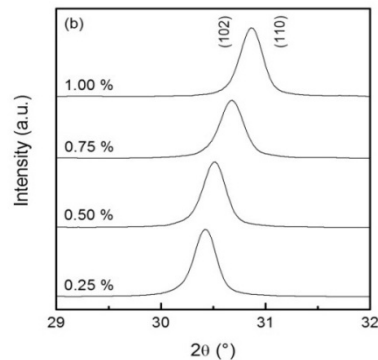
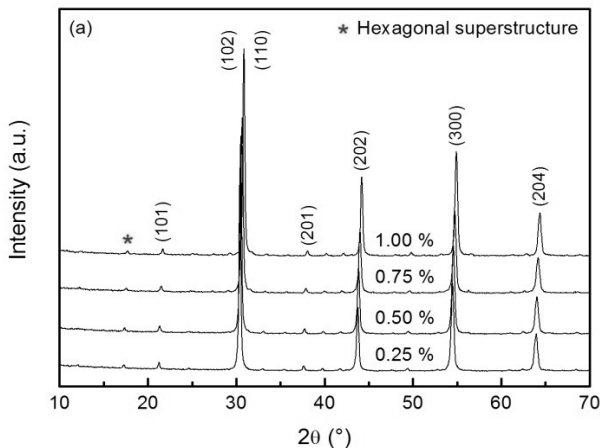


Fig. 1 - XRD patterns of: (a) BZT ceramics doped with Nb_2O_5 and sintered at 1500°C and (b) the same ceramics for a narrow angle range
Diffractogramele de raze X ale: (a) ceramicilor BZT dopate cu Nb_2O_5 și sinterizate la 1500 °C și (b) aceleși ceramici pentru un interval îngust de unghi.

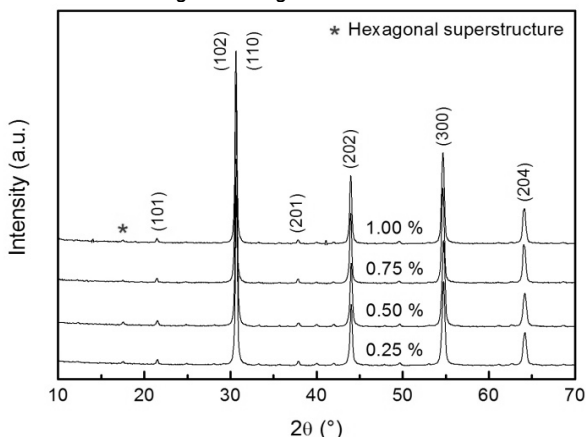


Fig. 2 - XRD patterns of BZT ceramics doped with Eu_2O_3 and sintered at 1600°C / Diffractogramele de raze X ale ceramicilor BZT dopate cu Eu_2O_3 și sinterizate la 1600 °C.

Generally, S values range between 0.70 and 0.99. It is not possible to estimate the efficiency of the selected dopants from this point of view because the values are spread out over a wide range for each dopant. However, all S values calculated in this paper are much higher than those of undoped BZT ceramics, for which a maximum value of 0.60 was attain (own results). Briefly, in the case of Nb_2O_5 doped BZT ceramics sintered at 1400°C, S presents values slightly higher than 0.70, while in the case of the same ceramics sintered at 1500 or 1600°C, it falls in the 0.80 - 0.99 range, increasing with sintering temperature increasing. On the other hand, for ZrO_2 and Eu_2O_3 doper BZT ceramics, S varies in the 0.70 - 0.9 range.

Figures 3 - 5 present the microstructure of some doped BZT ceramics sintered at different temperatures. BZT samples sintered at 1400 °C show a porous microstructure and a relatively small number of inter-granular bridges, aspects generated both by the uniaxial pressing process and by an insufficient sintering temperature. BZT samples sintered at 1500°C share a very good densification; in the fracture section, the individual

grains can be differentiated only inside the inter-granular pores dispersed in the material mass (Figure 4a), their size reaching maximum 2 μm . Generally, the grain size distribution is monomodal for all sintering temperatures and the increase of the sintering temperature from 1400 to 1600°C favours the granular growth, Eu_2O_3 doped BZT ceramics being the best example, with grain average size values of 0.8 μm for 1400 °C, 1.5 μm for 1500°C and up to 7.0 μm for 1600°C (Figure 5a). The SEM images display, for most of the samples, quasi-spherical grains (Figures 3a and 5a), but also polyhedral grains with well defined edges and corners, joined by common sides (Figure 4b).

An interesting morphology and curious at the same time is typical of BZT ceramic doped with 1.00 % Nb_2O_5 and sintered at 1600°C, for which

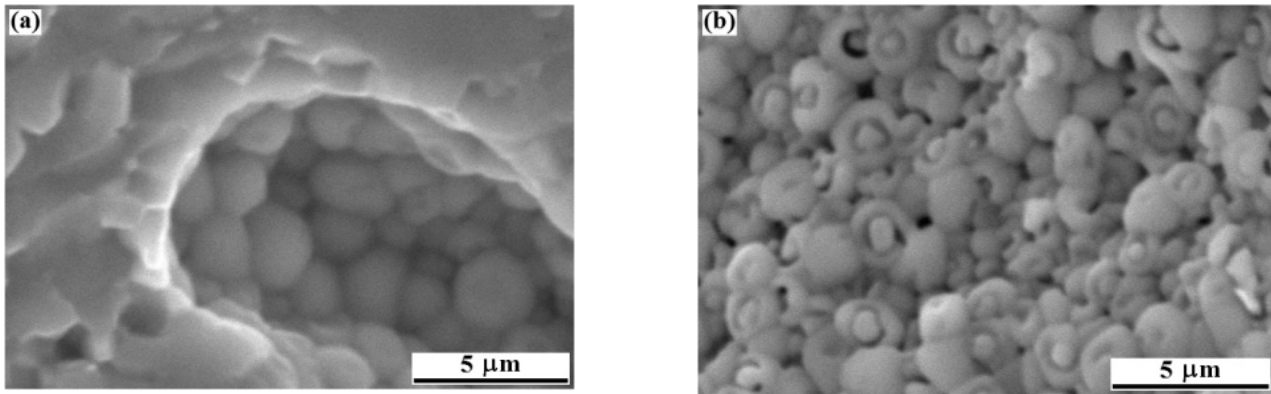


Fig. 3- SEM images of BZT ceramics sintered at 1600 °C and doped with: (a) 0.50 % Nb_2O_5 and (b) 1.00 % Nb_2O_5 . / Imagini SEM ale ceramicilor BZT sinterizate la 1600 °C și dopate cu: (a) 0,50 % Nb_2O_5 și (b) 1,00 % Nb_2O_5 .

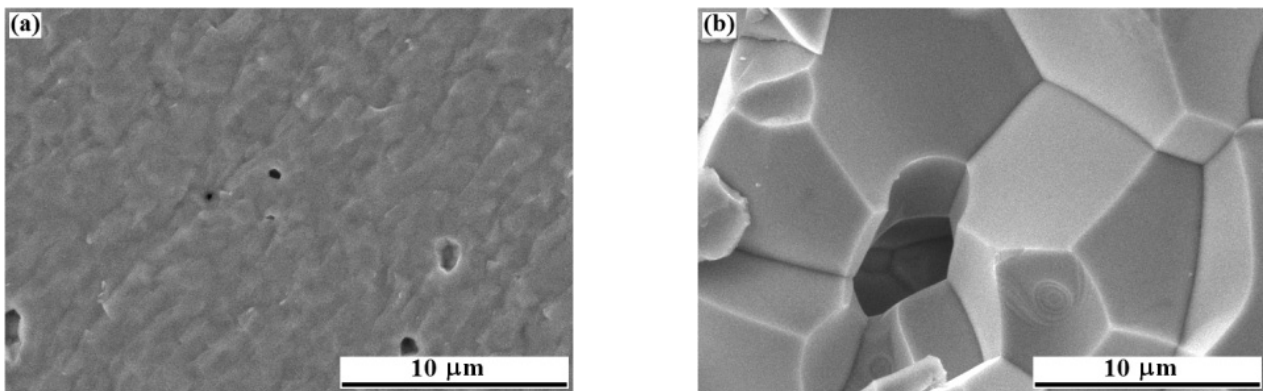


Fig. 4 - SEM images of BZT ceramics doped with 0.75 % ZrO_2 and sintered at: (a) 1500 °C and (b) 1600 °C / Imagini SEM ale ceramicilor BZT dopate cu 0,75 % ZrO_2 și sinterizate la: (a) 1500 °C și (b) 1600 °C.

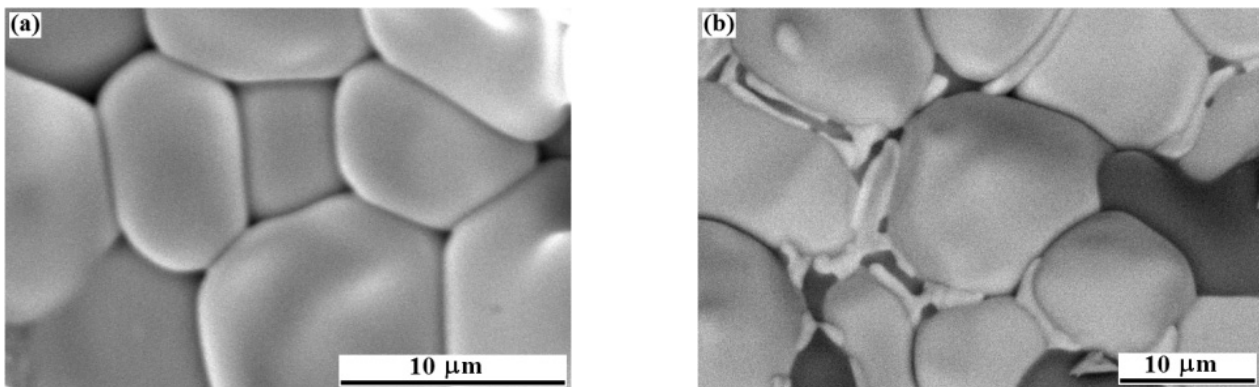


Fig. 5- SEM images of BZT ceramics sintered at 1600 °C and doped with: (a) 0.25 % Eu_2O_3 and (b) 1.00 % Eu_2O_3 . / Imagini SEM ale ceramicilor BZT sinterizate la 1600 °C și dopate cu: (a) 0,25 % Eu_2O_3 și (b) 1,00 % Eu_2O_3 .

tortellini-like grains can be observed (Figure 3b).

BZT ceramics doped with ZrO_2 and sintered at 1600°C also present a particular morphology, composed of big polyhedral grains, the granular growth steps being very well observed in some areas of the samples (Figure 4b), which means that this dopant greatly favours the diffusion processes.

In the case of BZT ceramic doped with 1.00 % Eu_2O_3 and sintered at 1600 °C, the SEM image created using the back-scattered electrons reveals the presence of two secondary phases (probably $Ba_8ZnTa_6O_{24}$ and $BaTa_2O_6$) in a small area of the sample, as well as their spatial distribution in the material, both as distinct grains and as crystallized

melt at BZT grain boundaries, resulting in a very suggestive compositional map (Figure 5b). The existence of these secondary phases can be explained by the partial volatilization of ZnO in a deeper layer from the sample surface.

The microwave measurements were performed in the 5 - 12 GHz frequency range. Figures 6 - 8 presents both the variation of the dielectric constant and the variation of the $Q \times f$ product (the product between the quality factor and the resonant frequency) of BZT resonators versus sintering temperature, for all dopants concentrations. The dependence of the dielectric constant on the sintering temperature is similar for

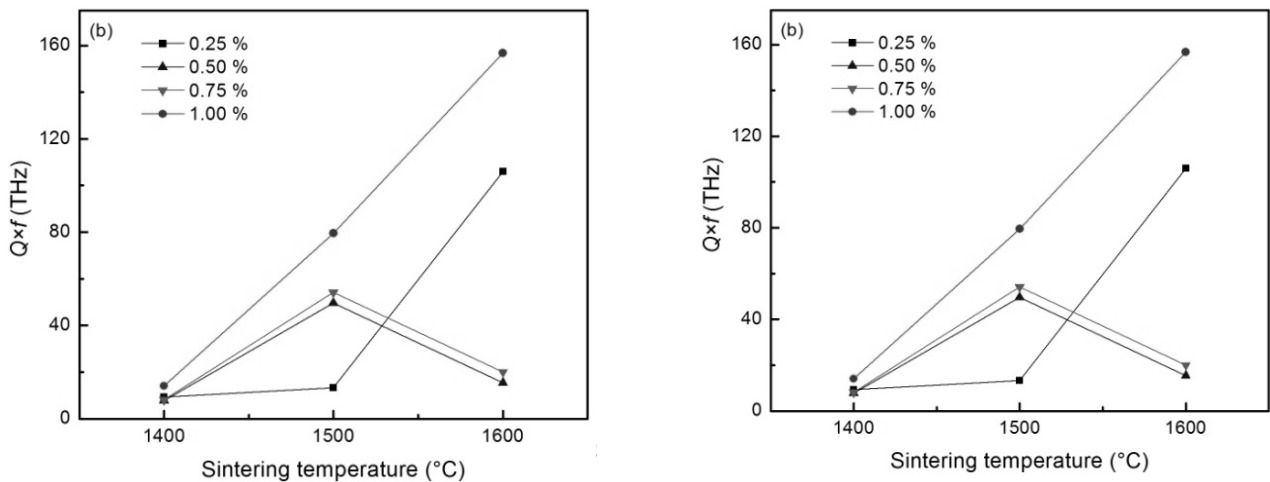


Fig. 6 - (a) Dielectric constant and (b) $Q \times f$ product of BZT ceramics doped with Nb_2O_5 versus sintering temperature, for all dopant concentrations / (a) Constanta dielectrică și (b) produsul $Q \times f$ ale ceramicilor BZT dopate cu Nb_2O_5 versus temperatura de sinterizare, pentru toate concentrațiile dopantului.

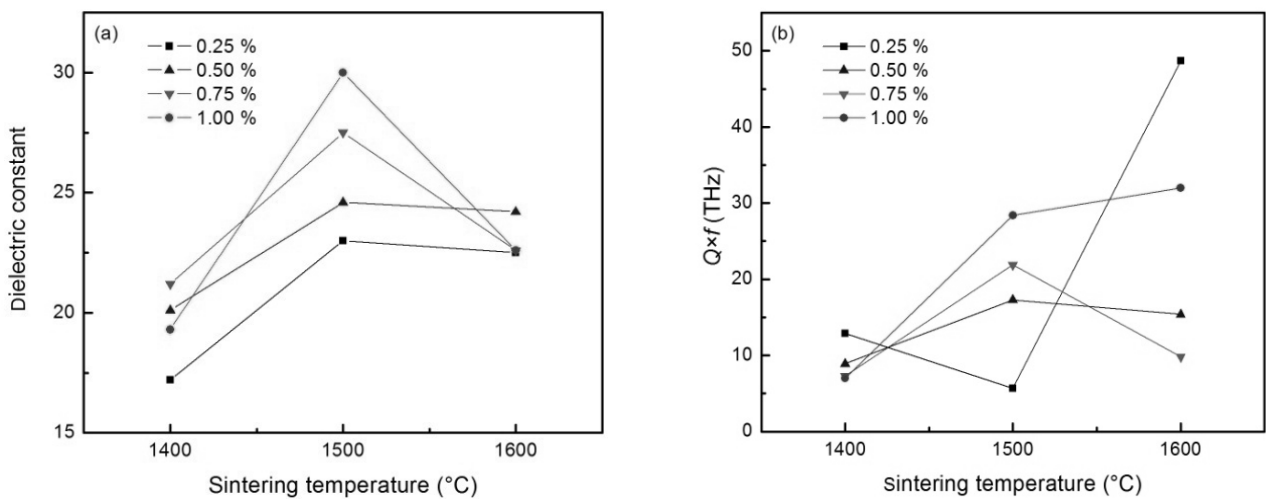


Fig. 7 - (a) Dielectric constant and (b) $Q \times f$ product of BZT ceramics doped with ZrO_2 versus sintering temperature, for all dopant concentrations / (a) Constanta dielectrică și (b) produsul $Q \times f$ ale ceramicilor BZT dopate cu ZrO_2 versus temperatura de sinterizare, pentru toate concentrațiile dopantului.

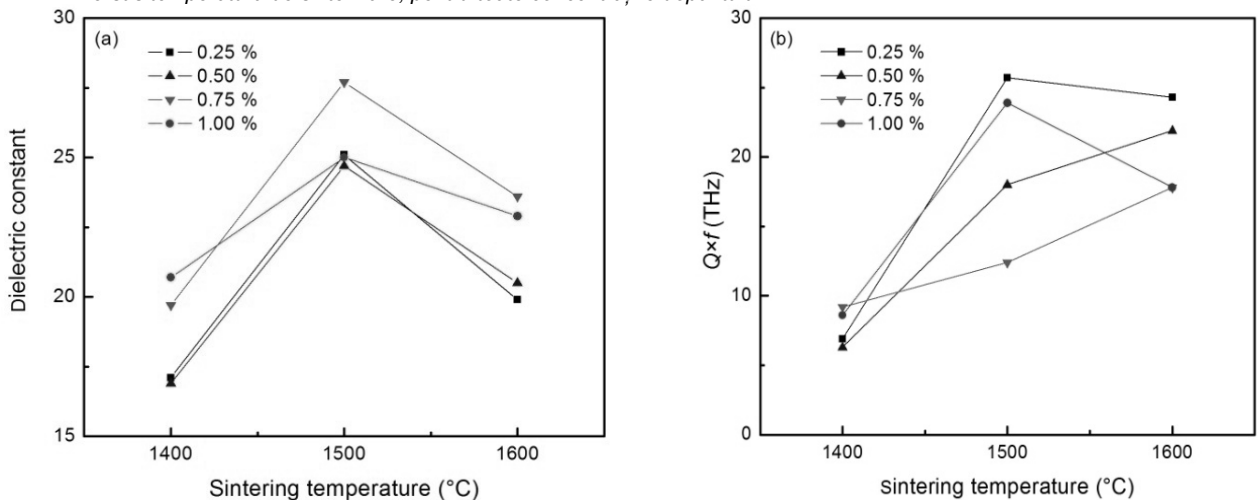


Fig. 8 - (a) Dielectric constant and (b) $Q \times f$ product of BZT ceramics doped with Eu_2O_3 versus sintering temperature, for all dopant concentrations / (a) Constanta dielectrică și (b) produsul $Q \times f$ ale ceramicilor BZT dopate cu Eu_2O_3 versus temperatura de sinterizare, pentru toate concentrațiile dopantului.

all samples, namely the increase of the sintering temperature from 1400 to 1500 °C leads to the increase of the dielectric constant, while the increase of the sintering temperature from 1500 to

1600 °C determines the decrease of it. The dielectric constant values ranges between 16.9 and 31.5, the lowest values being characteristic to the poorly densified samples. Regarding the influence of the

dopants concentration, no variation criterion can be formulated.

The situation is similar in the case of the $Q \times f$ product; no correlation can be made with other parameters, the variations being random. Even the scientific literature reports that it is possible to obtain different values for the quality factor of two ceramic samples obtained from the same starting materials and in the same processing conditions, this being due to small differences in the intrinsic crystal structure, microstructure, density and impurity concentration, cationic order and stoichiometry [1]. The highest values for the $Q \times f$ product, considering each dopant, are: ~ 157 THz for BZT ceramic doped with 1.00 % Nb_2O_5 and sintered at $1600^\circ C$, ~ 49 THz for BZT ceramic doped with 0.25 % ZrO_2 and sintered at $1600^\circ C$ and ~ 26 THz for BZT ceramic doped with 0.25 % Eu_2O_3 and sintered at $1500^\circ C$. The highest $Q \times f$ product value achieved in the case of using Nb_2O_5 as dopant is close to that reported by other papers (~ 161 THz [8] and ~ 159 THz [13]), the only difference being the value of the doping concentration (0.5 %). Globally, the results obtained in this paper for the $Q \times f$ product are better than those reported by Ioachim *et al.*, who used the same dopants [14].

Mostly, the increase of the sintering temperature should have a positive influence on the dielectric loss, as a consequence of granular growth enhancement, as well as grain boundaries and other structural defects elimination. Some BZT samples presented in this paper exhibit a decrease of the $Q \times f$ product when increasing the sintering temperature from 1500 to $1600^\circ C$, probably due to an intensification of ZnO depletion, which leads to a higher amount of secondary phases.

4. Conclusions

$BaZn_{1/3}Ta_{2/3}O_3$ ceramics doped with Nb_2O_5 , ZrO_2 or Eu_2O_3 were prepared by the conventional solid-state reaction method. The compositional and structural characterization, as well as the morphological characterization were performed after removing a layer of about 1 mm thickness from the surface of the sintered samples through disc cutting.

Almost all samples exhibit single-phase compositions and perovskite structure with hexagonal symmetry after sintering at 1400 , 1500 or $1600^\circ C$, 2 h. The SEM images evidence a porosity decrease, as well as an intense granular growth with sintering temperature increasing; the bigger grains, with quasi-spherical or polyhedral shape, reach $10 \mu m$ when sintering at $1600^\circ C$. The dopant type has a strong influence on the microstructure features, leading to different grain sizes and shapes.

The dopants efficiency decreases in the following series: $Nb_2O_5 > ZrO_2 > Eu_2O_3$. The

highest value achieved for the $Q \times f$ product was ~ 157 THz at 10 GHz, in the case of doping with 1.00 % Nb_2O_5 and sintering at $1600^\circ C$.

Acknowledgements

The authors thank to Dr. A. Ioachim from the National Institute of Materials Physics, Bucharest - Magurele, Romania, for performing the microwave measurements.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

CIMTEC 2014 - 13th International Conference on Modern Materials and Technologies Montecatini Terme, Tuscany, Italy, June 8 to 20, 2014

CIMTEC 2014 will consist of the 13th International Ceramics Congress (June 8-13) and of the 6th Forum on New Materials (June 15-20), each of them including a number of Symposia, Special Sessions, and Conferences. CIMTEC will again gather together a large and qualified audience of materials scientists, physicists, chemists and engineers as well as of experts of a wide range of the most demanding application areas of modern materials, from the molecular and nanoscales to large complex integrated systems.

- Symposium CA** Ceramic Powders: Advances in Synthesis, Processing and Manufacturing
- Symposium CB** Progress in Non Conventional and Novel Manufacturing Routes to Ceramics
- Special Session CB-9** SHS Ceramics
- Symposium CC** Materials Solutions for Highly Demanding Tribological Applications
- Symposium CD** Joining Inorganic Materials at Different Length Scales
- Symposium CE** Innovative Synthesis and Processing of Nanostructured, Nanocomposite and Hybrid Functional Materials for Energy and Sustainability
- Symposium CF** High and Ultra High Temperature Ceramics for Extreme Environments
- Symposium CG** Progress in Nano-laminated Ternary Carbides and Nitrides (MAX Phases) and Derivatives Thereof (MXenes)
- Symposium CH** Porous Ceramics for Environmental Protection, Energy-related Technologies and Advanced Industrial Cycles
- Symposium CI** Ceramic Thin Films and Coatings for Protective, Tribological and Multifunctional Applications
- Symposium CJ** Advances in Electroceramics
- Special Session CJ-6** State-of-the-art Development and Application of Thin Film Piezoelectric MEMS/NEMS
- Symposium CK** Functional Magnetic Oxides
- Symposium CL** Inorganic Materials Systems for Optical and Photonic Applications
- Symposium CM** Inorganic Polymers (Geopolymers) and Geocements: Environmentally Friendly Ceramic Materials for Low-Technology and High-Technology Applications
- Symposium CN** Science and Technology for Silicate Ceramics
- Symposium CO** Refractories: Developments in Raw Material, Production and Installation, Modelling, and Testing/Performance
- CP - 7th International Conference** Advanced Inorganic Fibre Composites for Structural and Thermal Management Applications

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