FILME SUBȚIRI PE BAZĂ DE TANTALAT DE BARIU ŞI MAGNEZIU OBȚINUTE PRIN PROCESARE SOL-GEL
BARIUM MAGNESIUM TANTALATE THIN FILMS OBTAINED BY SOL-GEL PROCESSING

CRISTINA BUSUIOC[1, 2*], SORIN ION JINGA[1], ECATERINA ANDRONESCU[1]
[1] *Universitatea "Politehnica" Bucureşti, Str. Gh. Polizu, nr. 1-7, 011061, Sect. 1, Bucureşti, România*
[2] *Institutul Național pentru Fizica Materialelor, Str. Atomiştilor, nr. 105bis, 077125, Măgurele, Jud. Ilfov, România*

Ba(Mg$_{1/3}$Ta$_{2/3}$)O$_3$ (BMT) compound is one of the most studied complex perovskite oxide with adequate properties for microwave applications, such as filters or antennas. BMT thin films were obtained by a modified sol-gel method, using the spin coating technique. Pt-coated Si or α-Al$_2$O$_3$ plates were used as substrates. X-ray diffraction, energy dispersive X-ray spectroscopy, scanning electron microscopy and atomic force microscopy were employed for compositional, structural and morphological characterization of BMT samples. BMT thin films present single-phase compositions and cubic structure. The dielectric properties of BMT thin films were investigated at low frequency.

Compusul Ba(Mg$_{1/3}$Ta$_{2/3}$)O$_3$ (BMT) este unul dintre cei mai studiați oxizi perovskitici complecşi, cu proprietăți adecvate pentru aplicații de microunde, de exemplu, filtre sau antene. Filmele subțiri BMT au fost obținute printr-o metodă sol-gel modificată, utilizând tehnica "spin coating". Ca substraturi au fost folosite plăcuțe de Si acoperit cu Pt sau de α-Al$_2$O$_3$. Pentru caracterizarea compozițională, structurală şi morfologică a probelor BMT, s-a apelat la difracție de raze X, spectroscopie de raze X cu dispersia energiei, microscopie electronică de baleiaj şi microscopie de forță atomică. Filmele subțiri BMT prezintă fază unică şi structură cubică. Proprietățile dielectrice ale filmelor subțiri BMT au fost investigate la frecvență joasă.

Keywords: *Sol-gel; Thin films; Dielectrics; Microwaves*

1. Introduction

With the recent revolution in mobile phone and satellite communication systems, using microwaves as carrier, the research and development in the field of components miniaturization has been one of the biggest challenges [1, 2]. The required properties in the case of high frequency dielectric materials are: high dielectric constant (ε_r), low dielectric loss (high quality factor - *Q*), thermal stability (small temperature coefficient of the resonant frequency - τ_f, and of the permittivity - τ_ε) and good mechanical properties [1, 3].

$Ba(Mg_{1/3}Ta_{2/3})O_3$ (BMT) compound is an important member of the $A(B'_{1/3}B''_{2/3})O_3$ (*A* = Ba, Sr, Ca; *B′* = Mg, Zn, Ni, Co, Sr, Ca, Mn, Cd; *B″* = Nb, Ta) complex perovskites family and has a ε_r of 25, a Q value of 36000 at 10 GHz and a temperature coefficient of resonant frequency of 4.4 ppm/°C [1]. Moreover, BMT presents a order - disorder phenomenon: when Mg and Ta cations are arranged in a random way over the *B* site, the crystal structure shows a disordered simple cubic symmetry, while the ordered compound adopts a 1:2 (Mg–Ta–Ta sequence) ordered hexagonal symmetry [4]. Many studies on the structure and electrical properties of BMT bulk ceramic revealed that the excellent microwave properties are given by the ordered structure [5]. The evolution of the ordered and disordered phases was described in terms of the nucleation and growth of small ordered domains with increasing annealing time and temperature [4, 6].

BMT bulk ceramic obtained by the conventional solid-state reaction method has been extensively studied owing to its potential applications in the electronics field. However, for achieving very good microwave dielectric properties, it requires high sintering temperatures (~ 1600 °C) and long annealing times (~ 100 h). Many investigations have been carried out regarding the use of sintering aids, dopants or glasses to reduce the sintering parameters without affecting the microwave dielectric properties [1, 5, 7, 8]. In order to overcome the disadvantages of the solid-state reaction method, soft chemistry techniques have been also used, but, in most cases, the researchers had to compromise on the microwave dielectric properties [9, 10].

In this context, growing thin films is a solution for both miniaturization and cost related problems. Thin films of dielectric materials offer the advantage of much lower crystallization temperatures, larger

* Autor corespondent/Corresponding author,
Tel.: +4 0727 039 699, e-mail: cristinab@infim.ro

capacitance than bulk samples and can be integrated in microelectronics devices. Few studies regarding the properties of BMT thin film are available in the literature [11-16].

Zhang et all. [11] prepared high-quality BMT thin films on Si (100) or fused silica substrates by pulsed laser deposition, with the substrate heated at 800 °C. The films are fully disordered, but the good optical performance indicates the possibility of using them in the optical field, besides the microwave field. Scarisoareanu et all. [12] reported on the structural, dielectric and optical properties of BMT thin films prepared by radiofrequency assisted pulsed laser deposition. All films exhibit a polycrystalline cubic structure, with a slight preferential orientation for the films obtained at lower substrate temperature, and a dielectric constant of ~ 23. Lin et all. [13] investigated the growth behaviour of BMT thin films on bare or Pt-coated Si substrates and their buffering effect on the subsequently deposited $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) films. The preferred orientation of BMT layers varies pronouncedly with the deposition parameters and the subsequently deposited PZT films inherit the texture characteristics of BMT buffer layers; the buffer layer not only suppresses the film-to-substrate interdiffusion, but also enhances PZT nucleation kinetics. Chen et all. [14] prepared a BMT thin films by a 2-step pulsed laser deposition process on MgO substrate. The films post-annealed at 600 °C present a grain average size of 200 - 300 nm. Zhou et all. [15] obtained BMT thin films on Si (111), Si (100) or Pt-coated Si substrates by a sol-gel process involving the reaction of barium isopropoxide, magnesium acetate and tantalum ethoxide in 2-methoxyethanol and subsequently hydrolysis, spin-coating and heat treatment. The films tend to crystallize with grains sized below 100 nm and dominant disordered cubic perovskite structure if annealed below 1000 °C. Joshi et all. [16] reported on the characterization of BMT thin films synthesized the metalorganic solution deposition technique, using barium acetate, magnesium methoxide and tantalum ethoxide as precursors. It was possible to attain a 0.3 μm thick polycrystalline BMT thin film with 1:2 ordered perovskite structure when annealing at 700 °C. All films present smooth surface, with no cracks, dense microstructure and very fine grain size; the dielectric constant has values in the 23.5 - 25 range, while the temperature coefficient of the capacitance is - 145 ppm/°C in the 25 - 125 °C temperature range.

In this paper, we report on the synthesis and characterization of BMT thin films deposited by a modified sol-gel method associated with the spin coating technique. Sol-gel processing presents the advantages of homogeneity at the molecular level due to the mixing of liquid precursors, precise composition control, low processing temperature, simple and economical fabrication technology and uniform deposition over large area substrates. The resulting BMT films were compositional, structural morphological and electrical characterized.

2. Experimental procedure

2.1. Method description

BMT thin films were obtained by a modified sol-gel method associated with the spin coating technique. Spin coating (Fig. 1) is a chemical procedure used to apply uniform thin films to flat substrates. In short, an excess amount of a solution is placed on the substrate, which is then rotated at high speed in order to spread the fluid by the centrifugal force. Rotation is continued while the fluid spins off the edges of the substrate, until the desired thickness of the film is achieved. The applied solvent is usually volatile and simultaneously evaporates.

Thus, the higher the rotation speed or time, the thinner the film. The thickness of the film also depends on the solvent and on the concentration of the solution.

Spin coating is widely used in the fabrication of thin films with thickness below 10 nm, but also to deposit layers of about 1 μm thick.

2.2. Synthesis

Barium carbonate (Fluka, >99%), tetrahydrated magnesium acetate (Merck, >99%) and tantalum butoxide (Aldrich, 98%) were used as sources of metallic cations. To magnesium acetate and citric acid (CA) dissolved in ethylene glycol, tantalum butoxide was added in inert atmosphere, corresponding to a molar ratio (Mg + Ta) : (CA) of 1:2. Then, barium citrate obtained by dissolving barium carbonate in 4M aqueous solution of CA was added and Ba-Mg-Ta clear citrate solution was formed. The precursor BMT

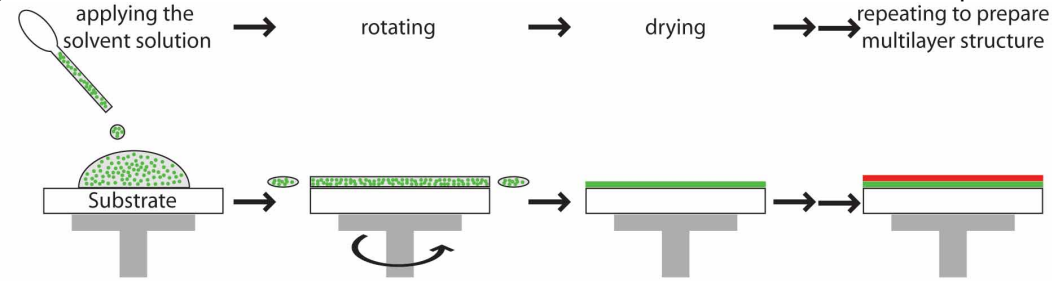


Fig. 1- The stages of the spin coating technique / *Etapele tehnicii de depunere prin centrifugare*.

films were deposited by spin coating on two types of substrates: platinum coated silicon (100) (Pt / Si) and α-alumina (α-Al_2O_3). The suspended impurities were removed from the solution by filtering through a 0.2 mm syringe filter. After spinning with a rotation speed of 3000 rpm / 30 s, the precursor films were kept on a hot plate at ~ 200 °C / 2 min, then at ~ 400 °C / 4 min, in air. This step was repeated after each coating in order to ensure a partial removal of the volatile matter. The post-deposition annealing of the films, required in order to develop crystallinity, was carried out at 750 °C / 2 h or 800 °C / 1 h, with a heating rate of 10 °C/min, in air. Four BMT thin films were obtained, as follows:

- BMT3 / Pt / Si thin film composed of 3 layers and annealed at 750 °C / 2 h;
- BMT4 / Pt / Si thin film composed of 4 layers and annealed at 800 °C / 1 h;
- BMT / α-Al_2O_3 thin film composed of 4 layers and annealed at 800 °C / 1 h;
- BMT12 / Pt / Si thin film composed of 12 layers (the precursor solution was diluted 10 times with a 0.3M aqueous solution of CA) and annealed at 750 °C / 2 h.

2.3. Characterization

BMT thin films were investigated by X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), atomic force microscopy (AFM) and electrical measurements. A Bruker D8 ADVANCE diffractometer with Ni filtered Cu-K_α radiation, 2θ ranging between 20 and 60 °, was used to identify the crystalline phases and the structure of the resulting films. The unit cell parameter values and crystallite average sizes were calculated using the PANalytical X'Pert HighScore Plus software. The morphology of the films was visualized with a FEI Quanta Inspect F scanning electron microscope and a MFP-3D-SA atomic force microscope. The temperature dependence of the capacitance at 100 kHz frequency was recorded with a HIOKI LCR-meter, in the - 100 °C to + 100 °C temperature range.

3. Results and discussion

3.1. X-ray diffraction and energy dispersive X-ray spectroscopy

The XRD patterns (Figs. 2 and 3) indicate a polycrystalline single-phase with disordered cubic structure (JCPDS 01-070-9200) for all BMT films. There is not a preferential growth on different crystallographic directions, as it can be found in the case BMT thin films obtained by PLD [12, 13]. The supplementary diffraction peaks are attributed to the substrate, in the first case to Pt (Fig. 2b) and in the second one to α-Al_2O_3 (Fig. 3). BMT3 / Pt / Si thin film (Fig. 2a) does not present the Pt diffraction peak because the XRD analysis was performed at

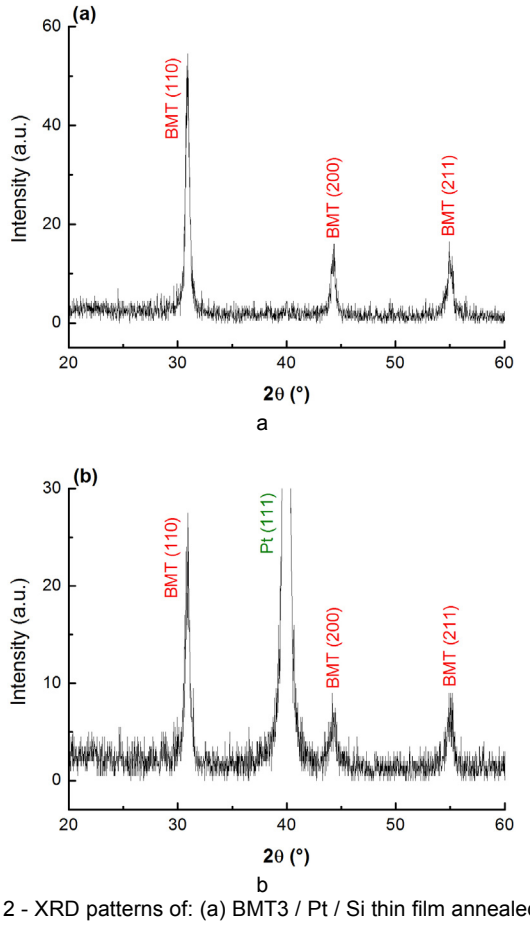


Fig. 2 - XRD patterns of: (a) BMT3 / Pt / Si thin film annealed at 750 °C / 2 h and (b) BMT4 / Pt / Si thin film annealed at 800 °C / 1 h / *Difractogramele de raze X ale: (a) filmului subțire BMT3 / Pt / Si tratat termic la 750 °C / 2 h şi (b) filmului subțire BMT4 / Pt / Si tratat termic la 800 °C / 1 h.*

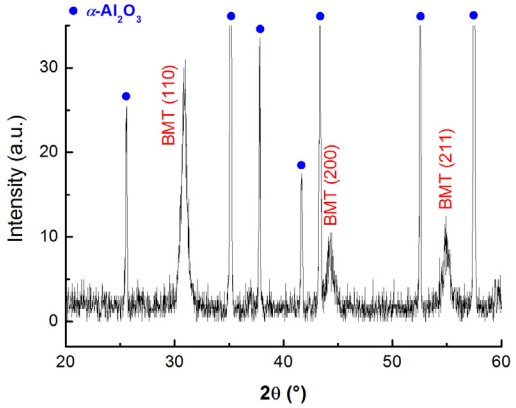


Fig. 3 - XRD pattern of XRD patterns of BMT / α-Al_2O_3 thin film annealed at 800 °C / 1 h / *Difractograma de raze X a filmului subțire BMT / α-Al_2O_3 tratat termic la 800 °C/1h.*

grazing incidence (2°). Several authors confirmed the disordered cubic structure of BMT thin films annealed at temperatures below 1000 °C [11, 12, 15].

The calculated values of the lattice parameter are: *a* = 4.0885 Å for BMT3 / Pt / Si thin film, *a* = 4.0929 Å for BMT4 / Pt / Si thin film and *a* = 4.0920 Å for BMT / α-Al_2O_3 thin film, while the crystallite average sizes reach 17 nm for BMT3 / Pt / Si thin film, 20 nm for BMT4 / Pt / Si thin film

Tabelul 1

Theoretical and EDS weight composition of BMT12 / Pt / Si thin film annealed at 750 °C / 2 h.
Compoziția gravimetrică teoretică şi EDS pentru filmul subțire BMT12 / Pt / Si tratat termic la 750 °C / 2 h.

Composition / *Compoziție*	Ba (wt%)	Mg (wt%)	Ta (wt%)	O (wt%)
Theoretical / *Teoretică*	43.73	2.58	38.41	15.28
EDS EDS	44.53	1.84	39.41	14.22

and 19 nm for BMT / α-Al_2O_3 thin film.

In the case of BMT12 / Pt / Si thin film, there was not possible to record the XRD pattern, probably because of the small thickness of this film. As a consequence, the EDS investigation was used and, as it can be seen in Tabel I, there is a good agreement between the theoretical and measured weight composition of BMT12 / Pt / Si thin film, so that it can be stated that BMT compound was formed.

3.2. Scanning electron microscopy

Figures 4-7 show the morphology of all BMT thin films. Unfortunately, the surface of BMT3 / Pt / Si thin film (Fig. 4) is covered by a lattice of branched cracks with width of ~ 0.5 μm. The high concentration of the precursor solution and the long annealing treatment can be two of the reasons for this defective surface.

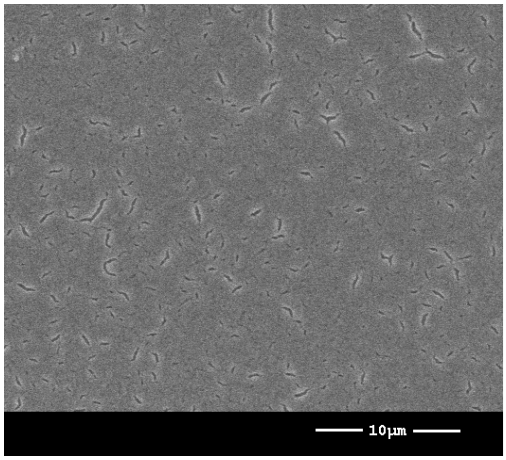
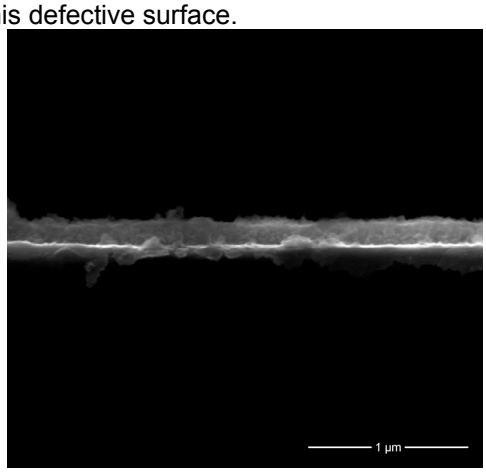


Fig. 4 - SEM image (surface) of BMT3 / Pt / Si thin film annealed at 750 °C / 2 h / *Imagine SEM (suprafață) a filmului subțire BMT3 / Pt / Si tratat termic la 750 °C / 2 h.*



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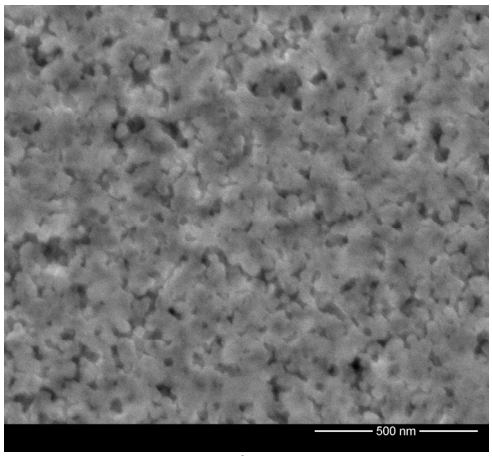

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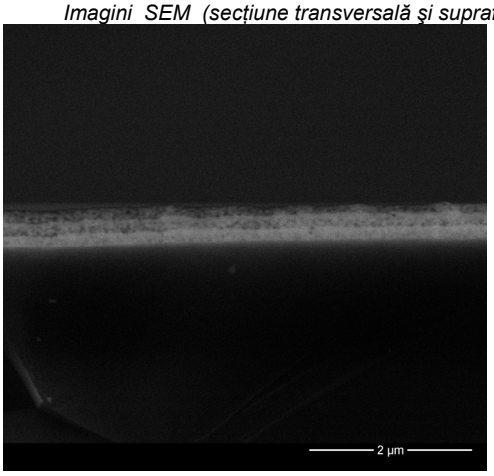
Fig. 5 - SEM images (cross section and surface) of BMT4 / Pt / Si thin film annealed at 800 °C / 1 h.
Imagini SEM (secțiune transversală şi suprafață) ale filmului subțire BMT4 / Pt / Si tratat termic la 800 °C / 1 h.



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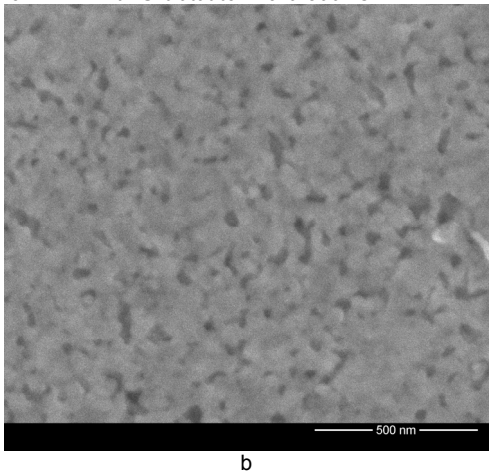

b

Fig. 6 - SEM images (cross section and surface) of BMT / α-Al_2O_3 thin film annealed at 800 °C / 1 h.
Imagini SEM (secțiune transversală şi suprafață) ale filmului subțire BMT / α-Al_2O_3 tratat termic la 800 °C / 1 h.

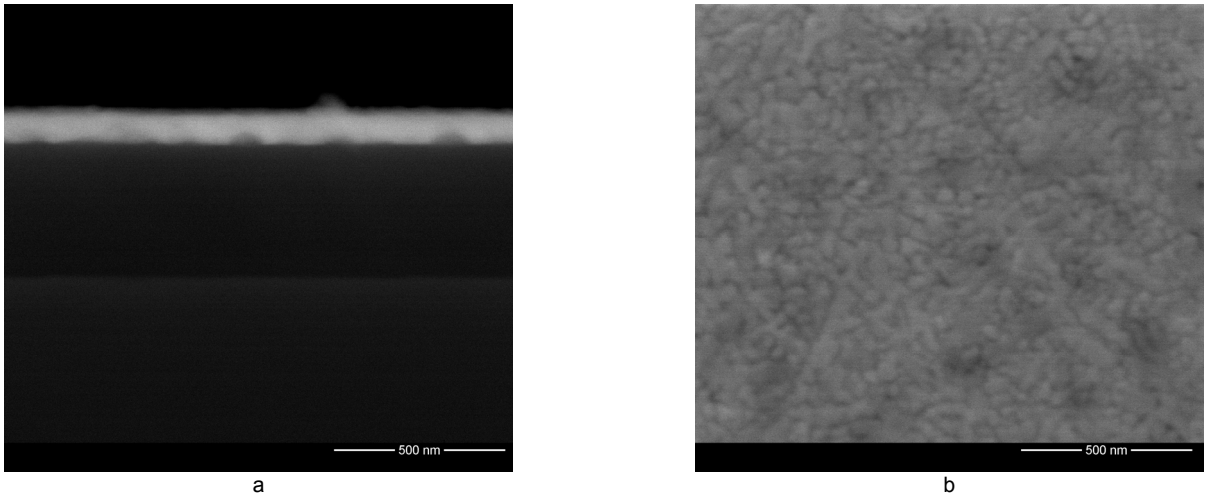

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Fig. 7 - SEM images (cross section and surface) of BMT12 / Pt / Si thin film annealed at 750 °C / 2 h.
Imagini SEM (secțiune transversală şi suprafață) ale filmului subțire BMT12 / Pt / Si tratat termic la 750 °C / 2 h.

BMT thin films annealed at 800 °C / 1 h show thickness values of ~ 160 in the case of Pt / Si substrate (Fig. 5a) and ~ 460 nm for α-Al_2O_3 substrate (Fig. 6a). This difference is very curious because the layers number and the processing parameters (rotation speed and time, annealing temperature) are the same. BMT film deposited on Pt / Si substrate has a homogeneous section, while BMT film deposited on α-Al_2O_3 substrate exhibits three distinct layers, with thickness that decreases from the substrate surface to the film surface, but also with different porosity, which increases from the substrate surface to the film surface. It is obvious that the substrate type has a crucial influence on the thickness and microstructure of BMT thin films. The explanation is very simple: the Si substrate is coated with a Pt (111) layer with a thickness of ~ 30 nm and cubic structure (a = 3.9231 Å), while the α-Al_2O_3 substrate has rhombohedral structure (a = 4.7342 Å and c = 12.9176 Å). BMT thin films deposited by spin coating exhibit cubic structure, therefore, the growth stress is smaller in the case of Pt / Si substrate, this encouraging the deposition of high quality films.

Moreover, both films annealed at 800 °C/1 h present high quality surfaces, smooth, with good compactness and free of cracks (Figs. 5b and 6b). The microstructure is granular, different from the microstructure of those BMT thin films obtained by PLD, which have a columnar microstructure, with columns oriented perpendicular to the substrate surface (own results, not publish yet). The grain size distribution is narrow, with grains presenting spherical shape. BMT4 / Pt / Si thin film shows a grain average size of ~ 25 nm, but in the case of BMT / α-Al_2O_3 thin film it is hard to assess the grain average size from the SEM image. The dimension mentioned above is comparable with the grain average size reported for BMT thin films obtained by other soft chemistry methods [15, 16] and lower that that reported for BMT thin films prepared by PLD [12].

BMT12 Pt/Si thin film deposited from a diluted precursor solution and annealed at 750 °C / 2 h has a constant thickness of ~ 100 nm (Fig. 7a). Unlike BMT3 / Pt / Si thin film annealed at the same temperature, BMT12 Pt/Si thin film has a fine and smooth surface, with near-zero porosity and free of cracks (Fig. 7b). The spherical grains have a narrow size distribution and an average size of ~ 20 nm, smaller than in the case of BMT4 / Pt / Si thin film deposited from a concentrated precursor solution and annealed at 800 °C / 1 h. The sample presented in Figure 7 seems to have the best microstructure from all four films; unfortunately, the electrical measurements on this film could not have been performed because of the small thickness, which generates the risk of short-circuit.

3.4. Atomic force microscopy

The AFM images of BMT4 / Pt / Si and BMT / α-Al_2O_3 thin films (Fig. 8) show smooth surfaces, narrow grain size distributions and grain average sizes of ~ 25 nm and ~ 50 nm, respectively, in good agreement with the SEM investigation. It is obvious that the α-Al_2O_3 substrate determines a grain average size two times higher than the Pt / Si substrate.

3.5. Electrical measurements

In order to perform the electrical measurements, superior Pt electrodes were deposited on the surface of BMT4 / Pt / Si thin film, which already has an inferior Pt electrode. The metallic contacts deposition was made by RF-sputtering, using a metal mask with dimensions of 0.2 mm^2.

The dielectric constant (ε_r) of BMT4 / Pt / Si thin film is ~ 18, lower than in the case of other BMT thin films [12, 16] and BMT bulk ceramic [1], probably due to a higher porosity.

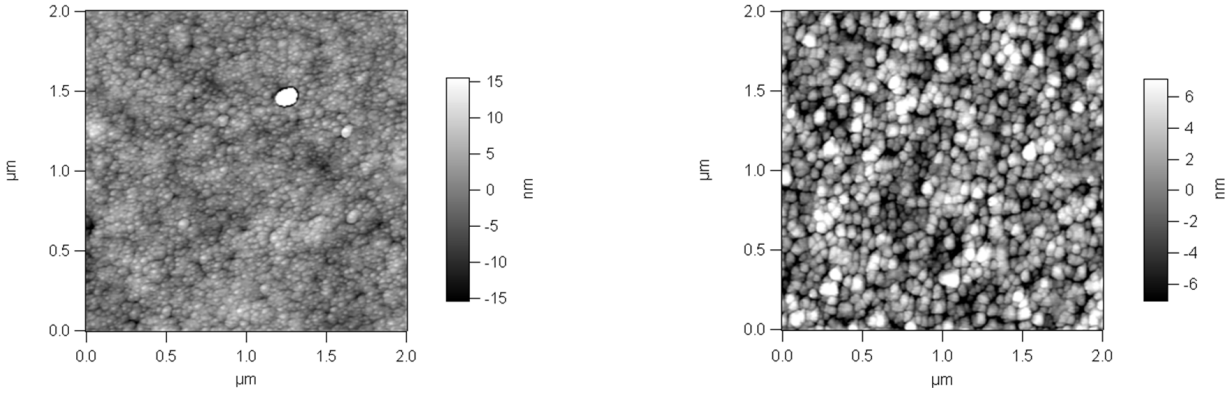

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Fig. 8 - AFM images of BMT4 / Pt / Si thin film annealed at 800 °C (left) and BMT / α-Al_2O_3 thin film annealed at 800 °C / 1 h (right).
Imagini AFM ale filmului subțire BMT4 / Pt / Si tratat termic la 800 °C / 1 h (stânga) şi filmului subțire BMT / α-Al_2O_3 tratat termic la 800 °C / 1 h (dreapta).

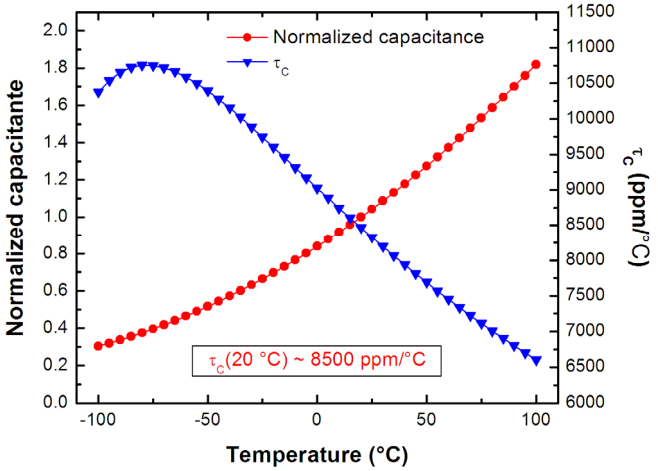

Fig. 9 - The normalized capacitance and the temperature coefficient of the capacitance (τ_C) / of BMT4 / Pt / Si thin film annealed at 800 °C / 1 h versus temperature, at 100 KHz / *Capacitatea normalizată şi coeficientul de temperatură la capacități (τ_C) pentru filmul subțire BMT4 / Pt / Si tratat termic la 800 °C / 1 h versus temperatură, la 100 kHz.*

Figure 9 presents the normalized capacitance and the temperature coefficient of the capacitance (τ_C) of BMT4 / Pt / Si thin film versus temperature (from - 100 °C to + 100 °C), at 100 kHz. The normalized values (this means divided by the capacitance value at room temperature) were used in order to eliminate the influence of the thickness and surface area of the capacitor. The values of τ_C were determined from the curve of normalized capacitance. At 20 °C, τ_C has a value of ~ 8500 ppm/°C, a relatively high value, but that can be improved by obtaining a better microstructure of BMT thin films.

The mathematical equations 1-4 represent the theoretical background regarding the electrical measurements. The equation 3 is the general form, while the equation 4 is specific to isotropic media, the polycrystalline materials being considered such media.

$$C = \varepsilon_o \varepsilon_r \frac{A}{h} = \varepsilon \frac{A}{h} \quad (1)$$

$$\frac{1}{C}\frac{dC}{dT} = \frac{1}{\varepsilon}\frac{\partial \varepsilon}{\partial T} + \frac{1}{A}\frac{\partial A}{\partial T} - \frac{1}{h}\frac{\partial h}{\partial T} \quad (2)$$

$$\tau_C = \tau_\varepsilon + \tau_A - \tau_h \quad (3)$$

$$\tau_C = \tau_\varepsilon + \alpha \quad (4)$$

where: C is the capacitance, ε_0 is the permittivity of vacuum, ε_r is the relative permittivity of the material, A is the surface of the capacitor, h is the height of the capacitor and α is the thermal expansion coefficient (α = 11 ppm/°C for BMT).

In our case it is not necessary to make the expansion correction in order to obtain the value of the temperature coefficient of the permittivity (τ_ε) because the value of τ_C is high, so that we can consider that τ_C is approximately equal to τ_ε.

4. Conclusions

BMT thin films were deposited by a modified sol-gel method associated with the spin coating technique on Pt-coated Si or α-Al_2O_3 substrates. The annealing treatment was performed at 750 °C or 800 °C. All films present a disordered cubic structure. SEM investigation reveals a granular microstructure, a narrow grain size distribution and spherical grains. BMT thin films grown on Pt-coated Si substrate present grain average sizes of 20 - 25 nm, while that deposited on α-Al_2O_3 substrate exhibits a grain average size of ~ 50 nm. Substrate kind has a crucial influence on the thickness and microstructure of BMT thin films.

The dielectric constant (ε_r) of BMT thin film deposited on Pt-coated Si substrate and annealed at 800 °C / 1 h is ~ 18, while the temperature coefficient of the capacitance (τ_C) has a value of ~ 8500 ppm/°C at 20 °C. A future annealing treatment or the deposition of thicker films from a diluted precursor solution could assure a better thermal stability of BMT thin films, so that they will become suitable for microwave communications and integrated capacitor applications.

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NOUTĂȚI / NEWS

Geopolimeri pe bază de caolin grosier de puritate scăzută: Rezistența mecanică funcție de compoziția chimică şi de temperatură / Geopolymers based on a coarse low-purity kaolin mineral: Mechanical strength as a function of the chemical composition and temperature

Un mineral grosier cu 70% caolinit şi 30% cuarț a fost calcinat şi activat chimic cu ajutorul soluțiilor alcaline de Na_2SiO_3 şi NaOH. Evoluția efortului de compresiune a fost cercetată funcție de temperatura de conservare până la întărire la 20 şi 80°C şi de rapoartele molare SiO_2/Al_2O_3 (2,64–4,04) şi Na_2O/Al_2O_3 (0,62–1,54). Pentru conservarea la 20°C, compoziția cea mai bună a fost SiO_2/Al_2O_3 = 2,96 şi Na_2O/Al_2O_3 = 0,62, atingând 85 MPa la 28 zile. Conservarea la 80°C a avut un efect pozitiv asupra dezvoltării rezistenței doar în primele 3 zile. Difracția de raze X a grupărilor geopolimerice a arătat formarea silicoaluminaților amorfi de natură similară. Microstructura constă din cuarț nereacționat şi particule de metacaolinit într-o matrice de polimer silicoaluminat şi din silicagel necondensat din silicatul de sodiu nereacționat.

A coarse mineral with 70% kaolinite and 30% quartz was calcined and chemically activated by alkaline solutions of Na_2SiO_3 and NaOH. The compressive strength evolution was investigated as a function of the curing temperature at 20 and 80°C, and the molar ratios of SiO_2/Al_2O_3 (2.64–4.04) and Na_2O/Al_2O_3 (0.62–1.54). For curing at 20°C, the best composition was SiO_2/Al_2O_3 = 2.96 and NaO/Al_2O_3 = 0.62, reaching 85 MPa at 28 days. Curing at 80°C had a positive effect on the strength development only in the first 3 days. X-ray diffraction of the geopolymeric formulations showed the formation of amorphous silicoaluminates of similar nature. The microstructure consisted of unreacted quartz and metakaolinite particles in a matrix of silicoaluminate polymer and condensed silica gel from the unreacted sodium silicate.

Material prelucrat de / *Material worked by: Alina Melinescu*
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