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Dielectric and microwave properties of ceramics of the Bi-Ti-O system

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Abstract. Dielectric and microwave properties of ceramics based on binary systems $Bi_2Ti_2O_7$-TiO_2 and $Bi_2Ti_4O_{11}$-TiO_2 were studied. In order to obtain dielectric materials with high stability of temperature coefficient of resonance frequency, high values of dielectric permittivity and quality factor modification of ceramics by chemical elements with different valence and ionic radii was performed. It is established, that on the basis of $Bi_2Ti_4O_{11}$-TiO_2 system, it is possible to receive microwave materials for manufacturing of resonators. It is shown that the introduction of different valence ions in the $Bi_2Ti_2O_7$-TiO_2 system allows to change its basic dielectric parameters in given limits and directed to choose the optimal composition of ceramics for the development of materials that can be used, for example, when creating storage capacitors.

1 Introduction

The Bi_2O_3-TiO_2system has attracted the attention of many material scientists due to the variety of physical properties (dielectric, ferroelectric, magnetic, piezoelectric) of the compounds formed in this system [1]. Ceramic materials based on bismuth titanates have a complex of properties interesting for practical applications [2-3]; therefore, they are actively studied to obtain new materials based on them. For example, $Bi_2Ti_2O_7$ pyrochlore [2-12] is a dielectric with high dielectric permittivity, small dielectric losses, and low temperature coefficient of capacitance. These properties may allow to reduce, for example, the size of capacitors and, consequently, to reduce their cost. Substituted bismuth titanates based on the well-known ferroelectric $Bi_2Ti_4O_{11}$ are promising as ferroelectric materials [2, 4].

However, bismuth titanates with pyrochlore structure are characterized by thermal instability of properties. Possible ways to improve the thermal stability are the doping of these compounds with atoms of various elements and the use of high pressure cold pressing before the sintering process. Studies of the effect of substitution defects on the physical and microwave properties of bismuth titanates are sparse. The properties of ceramics based on binary systems $Bi_2Ti_2O_7$-TiO_2 and $Bi_2Ti_4O_{11}$-TiO_2 are insufficiently studied, and the data on production of doped binary systems are few and scattered.

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This work is devoted to the study of dielectric and microwave properties of ceramics based on binary systems $Bi_2Ti_2O_7$-TiO_2 and $Bi_2Ti_4O_{11}$-TiO_2.

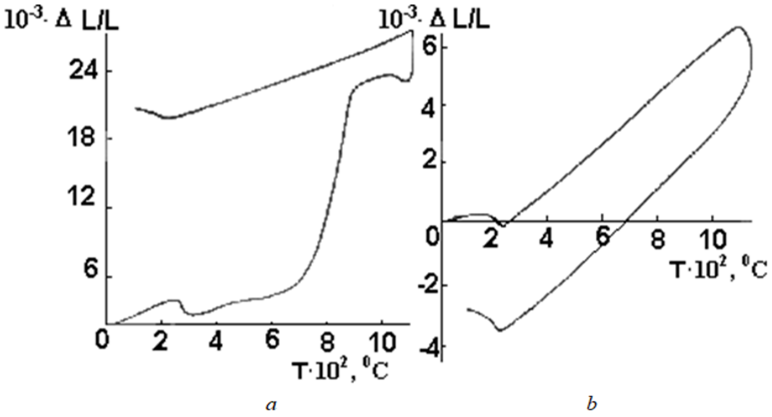
2 Experimental part

The synthesis of $Bi_2Ti_4O_{11}$ and $Bi_2Ti_2O_7$ compounds was studied by diffractometry, dilatometry and thermogravimetry. Research samples were synthesized from high-purity powders of Bi_2O_3 and TiO_2 taken in a stoichiometric ratio (Bi_2O_3+4TiO_2) and (Bi_2O_3 +2TiO_2) and pressed under pressure is 108 Pa.

Experiments to determine the formation temperature of the compound $Bi_2Ti_4O_{11}$ were carried out according to the following scheme. The samples pressed in the form of disks with a diameter of 0.01 m and a thickness of (0.004−0.005) m were heated in a furnace to a certain temperature, kept at this temperature, then quenched in air. After that X-ray phase analysis was carried out. Diffractograms were taken in CuK monochromatic radiation at 0.05° increments in the range of angles (9−60)°.

At the same time the mixture was subjected to differential-thermal analysis and thermogravimetric studies using a thermal analyzer TGA-92 of the French company "SETARAM". Thermogram of prepared mixture of stoichiometric composition Bi_2O_3+4TiO_2 under heating in the temperature range (20−1100) °C is shown in Fig. 1. The heating rate is 10 °C/min. The type of dependence of heat flux on temperature indicates that the formation process of Bi2Ti4O11 compound is multistage. In the temperature range of 20−700 °C there is moisture escape (~100 °C) and burnout of the bond (~350 °C). Two processes are observed in the range (700-750) °C: the phase transition of Bi_2O_3 [2] and the formation reaction of the compound $Bi_4Ti_3O_{12}$. The endothermic effect (Figure. 1) observed at (735±2) °C is followed by the exothermic effect, which depends on the heating rate. At a heating rate of 15 °C/min this exothermic effect is determined at (748±2) °C.

In parallel with the DTA studies, dilatometric measurements were performed (Fig. 1). For dilatometric measurements, the initial charge was pressed into columns in the form of cylinders with a diameter of 0.008 m and a height of 0.016 m. The experiments were carried out with a dilatometer D1-24 of the French company "ADAMEL LHOMARGY". The rate of temperature change during heating was 2.5 °C/min. In order to optimize the conditions of obtaining ceramic samples based on the compound $Bi_2Ti_4O_{11}$ dilatometric studies were carried out in two cycles: heating in air at a rate of 2.5 °C/min to 1100 °C, holding for 2 h and cooling to room temperature, then reheating (Fig. 1).



a *b*

Fig. 1. Results of dilatometric studies: *a*− first cycle; *b*− second cycle

According to the dilatometric measurements (Fig. 1, *a*) in the temperature range (200−300) °C, shrinkage due to binder burnout is observed. In the temperature range (870−1050) °C linear expansion occurs. The anomaly at 1070 °C is associated with the reaction of the formation of the compound $Bi_2Ti_2O_7$. X-ray phase analysis after dilatometric and thermogravimetric measurements showed that upon heating to 1100 °C at low rate and two-hour holding time the samples contain (6−7) % of $Bi_2Ti_2O_7$ phase. When the measurements were repeated, the dilatogram (Fig. 1, *b*) recorded the phase transition for the compound $Bi_2Ti_4O_{11}$ at (210±2) °C and a slight deviation from linearity in the temperature range (940−1100) °C. The compound $Bi_2Ti_2O_7$ has high defectivity, unstable, so the compound $Bi_2Ti_2O_7$ in the temperature range (940−1100) °C decomposes. After repeated cycle of dilatometric measurements, according to the results of X-ray analysis, the content of $Bi_2Ti_4O_{11}$ phase increases and the amount of $Bi_2Ti_2O_7$ phase decreases. This suggests that the formation process of the $Bi_2Ti_4O_{11}$ compound is largely dependent on the heating rate. To obtain from the initial mixture of oxides $Bi_2Ti_4O_{11}$ compound containing no pyrochlore phase $Bi_2Ti_2O_7$, you can conduct the heating process at speeds not lower than 10 °C/min, in this optimal conditions for obtaining single-phase ceramic samples based on $Bi_2Ti_4O_{11}$ compound are synthesis temperature 1150 °C (2 h) and sintering temperature 1220 °C (2 h).

X-ray microanalysis of the ceramics under study was carried out using an energy dispersive SiLi- semiconductor detector by "Röntec" (Germany). Microstructural studies were carried out on a Raster electronic microscope (REM) of LEO 1455 VP (Germany).

Frequency measurements of dielectric parameters were performed at room temperature using an Agilent E4991A impedance analyzer. Temperature studies of dielectric characteristics were performed at 1 kHz using the Bridge Method.

3 Results and discussion

Ceramics based on the $Bi_2Ti_4O_{11}$ compound has a dielectric permittivity of 53−56 and a dielectric loss tangent of 0.015 [2, 4].

Theoretical calculations of dielectric parameters for samples of the binary $Bi_2Ti_4O_{11}$-TiO_2 system based on the logarithmic law of mixing were performed based on the equations:

$$\ln\varepsilon = V_1\ln\varepsilon_1 + V_2\ln\varepsilon_2; \quad (1)$$

$$\tau_f = V_1\tau_{1f} + V_2\tau_{2f}, \quad (2)$$

whereV_1 and V_2 are volume percentages of $Bi_2Ti_4O_{11}$ and TiO_2, respectively;

ε_1 and $\varepsilon_2$are values of relative dielectric permittivity's of $Bi_2Ti_4O_{11}$ and TiO_2, respectively;

τ_{1f} andτ_{2f} are the temperature coefficients of the resonant frequencies of $Bi_2Ti_4O_{11}$ and TiO_2, respectively.

Calculations have shown that the resonant frequency coefficient τ_f close to zero at high values of dielectric permittivity should have compositions close to the phase ratio 43.5%$Bi_2Ti_4O_{11}$-56.5%TiO_2. Based on theoretical calculations to obtain in the system $Bi_2Ti_4O_{11}$-TiO_2 ceramic materials with higher values of the relative permittivity ε (80−100) and close to zero coefficient of the resonant frequency τ_f were investigated compositions 43%$Bi_2Ti_4O_{11}$-57%TiO_2, 45%$Bi_2Ti_4O_{11}$-55%TiO_2 and 40%$Bi_2Ti_4O_{11}$-60%TiO_2, which corresponds to the following ratio of the initial oxides:

HF7 – (0.081Bi_2O_3 + 0.919 TiO_2); HF8– (0.075Bi_2O_3 + 0.925 TiO_2)

HF9 – (0.087Bi_2O_3 + 0.913 TiO_2)

According to Table 1 dielectric parameters of ceramic materials based on the binary system $Bi_2Ti_4O_{11}$-TiO_2 are frequency dependent. With increasing frequency from 1 kHz to 1 MHz dielectric permittivity decreases from 95 to 45, the tangent of the angle of dielectric losses decreases from 0.25 to 0.0037. Thus, the highest permittivity at the lowest dielectric losses at frequency 1 MHz have samples of solid solution composition 43%$Bi_2Ti_4O_{11}$-57%TiO_2, which corresponds to composition HF7, which is close to the theoretically calculated composition.

Samples of the composition 43%Bi2Ti4O11-57%TiO_2before the sintering stage were subjected to high pressure under cold pressing (HPCP) conditions, which allowed to reduce the sintering temperature from 1220 to 1080 °C (Table 1).

Table 1. Dielectric parameters of the ceramic system $Bi_2Ti_4O_{11}$-TiO_2

Compound		wt. %					
TiO_2		0.57	HF7	0.55	HF9	0.60	HF8
$Bi_2Ti_4O_{11}$		0.43		0.45		0.40	
ε	1 kHz	89		82		95	
	10 kHz	65		52		65	
	20 kHz	63		48		62	
	1 MHz	61		45		59	
tgδ	10 kHz	0.13		0.25		0.17	
	20 kHz	0.085		0.018		0.11	
	1 MHz	0.0039		0.007		0.007	

According to Table 2, a decrease in the dielectric loss tangent is observed for ceramics. This is due to the fact that the use of HPCP leads to a reduction of bismuth oxide losses in the sintering process and, accordingly, to an increase in the density of the material. In addition, as shown by EDX analysis (Fig. 2), the homogeneity of the distribution of chemical elements in the grains of both phases increases.

Table 2. Dielectric parameters of ceramics of composition
43%Bi2Ti4O11-57%TiO_2as a function of the DXP value at 1 MHz

Pressure, GPa	0	1	2	3	4.5	6.5	7.5
ε	61	80	84	85	85	85	78
tgδ	0.0039	0.0011	0.001	0.001	0.0021	0.0023	0.0024

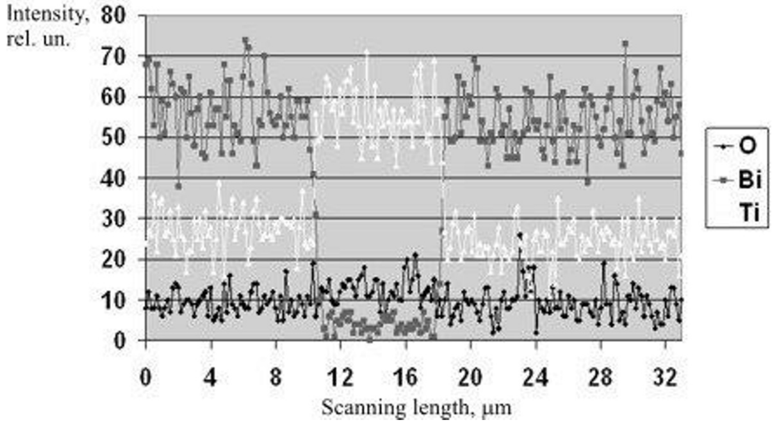


Fig. 2. Results of EDX analysis of 43% $Bi_2Ti_4O_{11}$-57%TiO_2 ceramics produced using high pressure (2 GPa) cold pressing

In order to obtain ceramics with high stability of temperature coefficient of resonant frequency and high values of dielectric permittivity the ceramics were modified with microadditives specified in Table 3. As follows from Table 3 data, presence of microadditives in ceramics composition more than 3 wt % leads to decomposition of $Bi_2Ti_4O_{11}$ compound according to the scheme resulting in a two-phase solid solution based on the system $Bi_2Ti_2O_7$-TiO_2.

$$Bi_2Ti_4O_{11} \rightarrow Bi_2Ti_2O_7 + 2TiO_2$$

Ceramic materials of the $Bi_2Ti_4O_{11}$-TiO_2 system change the basic dielectric parameters within specified limits, which allows directional selection of the optimal ceramic composition for each specific task. By modifying $Bi_2Ti_4O_{11}$-TiO_2ceramics, it is possible to obtain microwave materials with more than 100 and tg of the order of 0.002.

From Table 3 we can see that ceramics based on the system TiO_2+$Bi_2Ti_2O_7$ can be very promising for the microwave region. The synthesis of solid solutions of the $Bi_2Ti_4O_{11}$-TiO_2binary system was carried out in air by solid-phase reactions at temperatures of 850−1000 °C for 2−4 h. After synthesis secondary mixing was carried out, then milling, after which powders were pressed into tablets 0.008 or 0.012 m in diameter at 108 Pa and sintered at temperatures (1200−1220) °C for 2−8 h in tightly closed crucibles using bismuth-containing filler.

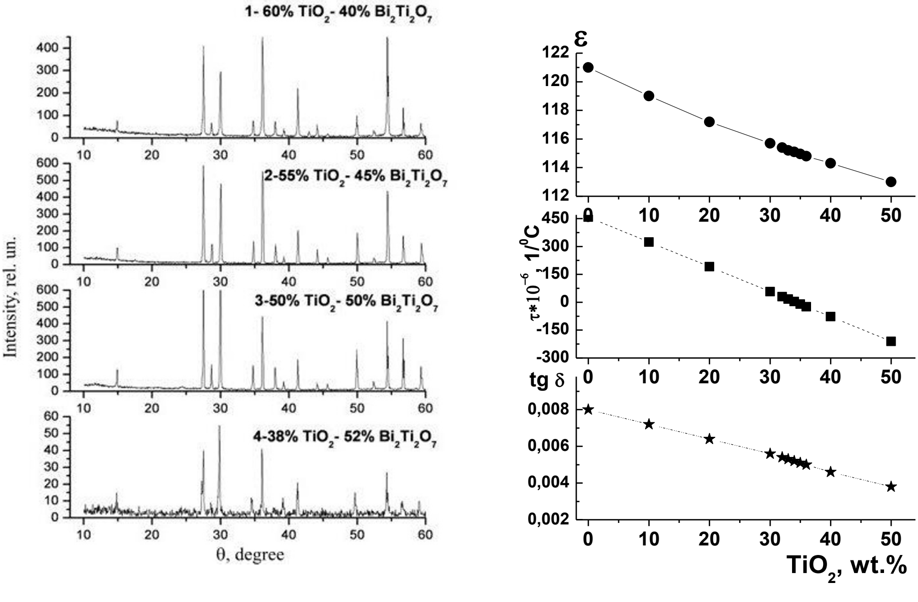
Table 3. Dielectric parameters and phase composition of doped samples binary system 43%$Bi_2Ti_4O_{11}$-57%TiO_2

Addition	Amountofadditive, wt. %	ε	tgδ	Results X-ray phase analysis
$MnCO_3$	0.2	99	0.0034	$Bi_2Ti_4O_{11}$+TiO_2
	0.4	99	0.0037	$Bi_2Ti_4O_{11}$+TiO_2
	0.8	105	0.0030	$Bi_2Ti_4O_{11}$+TiO_2
	1	100	0.0053	$Bi_2Ti_4O_{11}$+TiO_2
	3	115	0.0050	$2TiO_2$+$Bi_2Ti_2O_7$
Al_2O_3	0.2	94	0.0028	$Bi_2Ti_4O_{11}$+TiO_2
	1	86	0.0024	$Bi_2Ti_4O_{11}$+TiO_2
	4	88	0.0032	$2TiO_2$+$Bi_2Ti_2O_7$
Nd_2O_3	0.2	87	0.0016	$Bi_2Ti_4O_{11}$+TiO_2
	0.4	102	0.0020	$Bi_2Ti_4O_{11}$+TiO_2
	1	90	0.0020	$2TiO_2$+$Bi_2Ti_2O_7$
	3	80	0.0050	
SnO_2	0.1	80	0.0050	$Bi_2Ti_4O_{11}$+TiO_2
	1	80	0.0070	
	2	89	0.0050	
	3	85	0.0010	$2TiO_2$+$Bi_2Ti_2O_7$
	4	73	0.0050	$2TiO_2$+$Bi_2Ti_2O_7$
	6	98	0.0009	$2TiO_2$+$Bi_2Ti_2O_7$

The composition of the ceramics depending on their titanium dioxide content was controlled by X-ray diffraction analysis (Fig. 3, *a*). The intensity peak on the X-ray radiographs at angle value $\theta = 27.38°$ corresponds to 100 % of the TiO_2 titanium dioxide line, and the peak at $\theta = 29.99°$ corresponds to 100 % of the $Bi_2Ti_2O_7$ compound line.

Analysis of physical properties of $Bi_2Ti_2O_7$-TiO_2 ceramics depending on their titanium oxide content (Fig. 3, *b*) showed that high quality factor (respectively lower dielectric loss tangent) with the resonance frequency factor τ_f close to zero has composition with phase ratio $0.66Bi_2Ti_2O_7$-$0.34TiO_2$. The area of titanium oxide concentrations from 30 % to 40 % was investigated in more detail, since in this area the sign of the resonance frequency coefficient of changes. From the dependencies shown in Fig. 4, it follows that the resonance frequency coefficient is close to zero ($\sim +4 \cdot 10^{-6}$ $°C^{-1}$) have compositions $(0.655-0.66)Bi_2Ti_2O_7$-$(0.345-0.34)TiO_2$, with their dielectric permittivity at 1 MHz frequency being ~115 and the tangent of the dielectric loss angle ~0.005.

The ceramic material of composition $0.66Bi_2Ti_2O_7$-$0.34TiO_2$ was alloyed with manganese, tin, europium and gadolinium ions in the amount from 0.1 wt. % to 7 wt. % at the stage of synthesis. For ceramic samples based on solid solutions of the $Bi_2Ti_2O_7$–TiO_2 system, both fast polarization mechanisms, which include elastic ion polarization, and slower ones, such as jump (thermal) polarization, in which electrons and ions localized near structural defects participate, can contribute to polarization. Consequently, oxides that could influence the above mechanisms were chosen as alloying microadditives.



a *b*

Fig. 3. Research results of ceramic samples of the $Bi_2Ti_2O_7$-TiO_2system depending on their TiO_2 content: *a*– radiographic; *b*– microwave parameters

Bismuth is volatile, so during sintering in the crystal lattice of pyrochlore $Bi_2Ti_2O_7$ bismuth defect formation is possible. As the alloying oxides were chosen oxides whose cations had radii less than the bismuth ionic radius (1.095 Å), but greater than the titanium ionic radius (0.6152 Å), the introduced ions had different valence: Sn^{+2} (0.9948 Å), Mn^{+2} (0.7966 Å), Gd^{+3} (0.9710 Å), Eu^{+3} (0.9876 Å). The alloying ions occupied the positions of bismuth ions and were substitution defects. The position of the introduced ions in the positions of trivalent bismuth ion Bi^{+3} due to the difference of ionic radii resulted in the change of basic interatomic distances in the pyrochlore structure, which in turn caused the change in the magnitude of shift of active cation Ti^{+4} relative to its centrosymmetric position. As a result, the introduction of ions as substitution defects leads to a change in dielectric permittivity and dielectric loss tangent of the obtained ceramic samples.

The results of dielectric permittivity tests carried out at a frequency of 1 MHz for compositions of the modified system with the highest dielectric parameter values are shown in Fig. 5. At that, the samples having the highest temperature stability of dielectric permittivity and dissipation factor in the working temperature range from 0 to 250 °C were chosen. Dielectric parameters of ceramic samples of composition $0.66Bi_2Ti_2O_7$-$0,34TiO_2$ vary within a wide range (εfrom 88 to 140, tgδ from 0,0009 to 0,0001) depending on the type and amount of the alloying additives. Their values largely depend on the type and valence of the introduced ions (Gd^{+3}, Eu^{+3}, Mn^{+2}, Sn^{+2}) (Fig. 4)

Compounds containing divalent tin ions Sn^{+2} (0.9948 Å) and trivalent ions Eu^{+3} (0.9876 Å), whose radii are close in value, have dielectric permittivity values of 88 and 120, respectively, at room temperature. In this case, for the above compositions, the contributions from elastic polarization can be commensurate, while different contributions are given by the hopping polarization, which is determined by the density of the electron cloud concentrated near the defect.

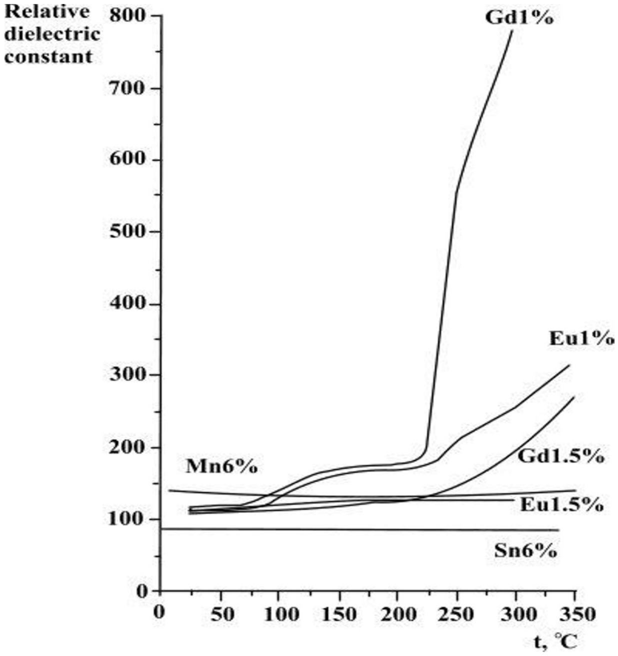


Fig. 4. Temperature dependences of dielectric permittivity of ceramics of composition 0.66%$Bi_2Ti_2O_7$-0.34%TiO_2 modified with manganese (Mn), tin (Sn) ions, europium (Eu) and gadolinium (Gd)

Introduction of europium ions (1.5 wt %), tin (6 wt %) and manganese (6 wt %) into the $Bi_2Ti_2O_7$-TiO_2 system achieves good temperature stability of dielectric permittivity and dielectric loss tangent. The temperature coefficient of dielectric constant (TCε) varies from $-4\cdot10^{-6}$ $°C^{-1}$ to $+10\cdot10^{-6}$ $°C^{-1}$.

The microstructure of a ceramic material based on $0.66Bi_2Ti_2O_7$-$0,34TiO_2$ modified with europium and having the highest dielectric parameters is shown in Fig. 5. The grains of dark color are grains of $Bi_2Ti_2O_7$ phase, the grains of light color are TiO_2 phase. The size of pyrochlore grains of $Bi_2Ti_2O_7$ phase having rounded shape varies from $2\cdot10^{-6}$ to $5\cdot10^{-6}$ m. At the same time the TiO_2 phase grains are much smaller ($(1–2)\cdot10^{-6}$ m) and are octahedrons of regular shape. Grains of both phases are densely packed. In the distributions of titanium, bismuth and oxygen within the grains of each phase no fluctuations are observed, and the distribution of elements has a homogeneous character.

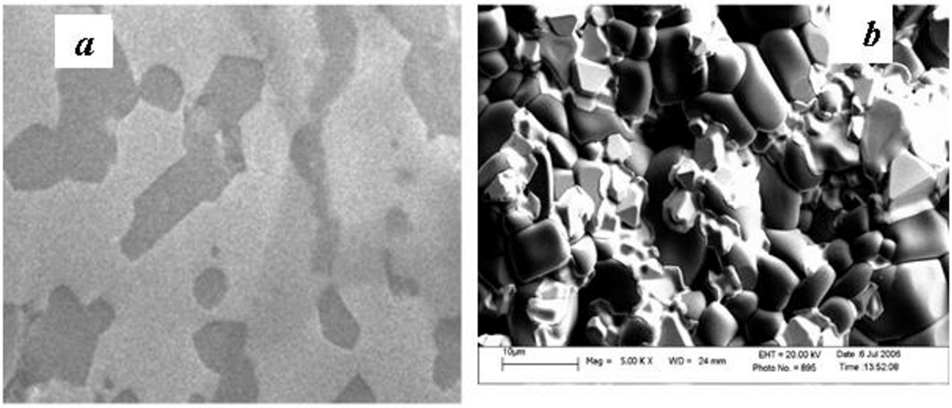


Fig. 5. Microphotograph of ceramics of the composition $0.66Bi_2Ti_2O_7$-$0.34TiO_2$, doped with europium: *a* - surface at magnification of 5 000; *b* - grain structure at magnification of 10 000

4 Conclusions

The results of this research allow us to determine the directions of further practical applications of ceramics of the binary systems $Bi_2Ti_4O_{11}$-TiO_2 and $Bi_2Ti_2O_7$-TiO_2. By modifying $Bi_2Ti_4O_{11}$-TiO_2ceramics we can obtain microwave materials with a dielectric permittivity greater than 100 and tgδ about 0,002, which allows their use for the manufacture of resonators. Ceramic materials based on solid solutions of the $Bi_2Ti_2O_7$-TiO_2system have high values of dielectric parameters, while the introduction of diverse ions allows the basic dielectric characteristics of the ceramics to be varied within given limits, and a choice of optimal ceramic composition for each specific task is made. Low cost of the initial reagents, simplicity of the technology, predictability of the physical properties, high degree of temperature stability of microwave parameters makes the ceramics $Bi_2Ti_2O_7$-TiO_2 promising for use in microwave devices and potentially useful as functional materials. Due to their high dielectric permittivity and low dielectric loss, $Bi_2Ti_2O_7$-TiO_2 dielectrics can be used to create storage capacitors for dynamic random-access memory (DRAM).

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