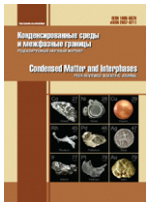

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Lead-free smart materials based on alkali metal niobates: phase formation, crystal structure, macroscopic responses

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Abstract

Objectives: In the global practice of researching various materials for piezoelectric devices, one of the first places is occupied by functional (smart) materials based on lead-containing compositions. However, the transition to environmentally friendly products, necessitated by the formation of new Russian and European legislative frameworks, has forced the search for other materials science solutions by eliminating lead from the elemental base of materials. An alternative to known compositions are solid solutions based on alkali metal niobates from morphotropic heterophase regions of the corresponding binary and ternary systems, characterized by extreme properties near the interphase boundaries. However, they have not found wide application in practice due to difficulties in phase formation during synthesis and the formation of a dense ceramic framework during sintering.

Experimental: In this work, using mechanical activation and hot pressing procedures, which were not previously used in such environments, it was possible to obtain lead-free, non-toxic ferroelectric piezoelectric ceramics with improved macroscopic responses due to the transformation of the phase coexistence regions.

Conclusions: As a result of the research, multi-frequency materials have been developed and created, including those with an elevated Curie temperature, piezoelectric sensitivity, thermal stability, and pyroelectric effect for various piezoelectric applications.

Keywords: Lead-free piezoceramics, Alkali metal niobates, Solid solutions, Interphase boundaries, Mechanical activation, Hot pressing

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1. Introduction

Recently, in the practice of using various materials in real sectors of the global economy, functional materials, primarily smart materials (SMs), "capable of actively counteracting external factors and adapting to them after assessing the nature of the external impact and their own condition," have come to the forefront. [1]. Among them, those whose operation is based on the piezoelectric effect, the conversion of mechanical force into an electrical signal and vice versa, are in high demand. Their main disadvantage is the presence of lead in the compositions, an element belonging to the first hazard class [2, 3]. The transition to environmentally friendly products that has now begun is due to the formation of a new Russian [4] and European [5, 6] legislative frameworks, which has forced us to look for other materials science solutions by excluding lead from the elemental base of SMs. An analysis of bibliographic information and patent literature [7–17] showed that the undisputed alternative to global brands of materials are SMs based on alkali metal niobates (AMN) from morphotropic heterophase regions of the corresponding binary and ternary systems, characterized by extreme properties near the interphase boundaries: rhombic-rhombohedral in the (Na,Li)NbO_3 system and rhombic-rhombic of different multiplicity, characteristic of different configurations of rotations of oxygen octahedra, in systems based on (Na, K)NbO_3 [18]. However, they have not yet found wide application in practice. This is largely due to the impossibility of obtaining them by traditional methods, which, in turn, is associated with the complexity of their crystalline structure; the presence of extensive polymorphism, which destabilizes the structure of materials; the ease of violation of the stoichiometry of a given composition due to the increased volatility of alkaline cations; polarization electrolysis; strong dependence of properties on the thermodynamic history (preparation conditions) [19].

This study, driven by current trends in the greenification of electronic components, aims to demonstrate the potential for improving the properties of SMs with reduced mass capacity and dimensions for use in devices where weight is critical; an extended frequency range, enabling the use of SMs in digital information processing systems; a high operating temperature limit for the creation of contact thermal sensors; increased thermal stability and, consequently, improved reliability and resistance to environmental factors.

2. Experimental

The objects of the study were sodium, potassium, and lithium niobates and solid solutions (SS) based on them, including those modified with various elements. The objects were obtained by one/two-stage solid-phase synthesis followed by sintering using conventional ceramic technology (CCT) or hot pressing (HP) with the simultaneous application of temperature and pressure to the powder press blank. Process procedures for obtaining objects are shown in the table: temperatures T_1, T_2 and isothermal holding time τ_1, τ_2 first and second synthesis respectively, sintering temperature, T_{si}, and the holding time during sintering, τ_{si}. In all cases, mechanical activation (MA) procedures were used for 10 min at the stages of preparing the initial precursors or preparing the batches. Mechanical activation (mechanical impact on objects by crushing them) was carried out in an AGO-2 high-energy grinding planetary ball mill manufactured by Novits (Novosibirsk). Grinding was carried out in an ethyl alcohol environment; the drum rotation frequency was 1050 rpm. Hydrocarbonates, carbonates and oxides of the corresponding elements of at least analytical grade (pure for analysis) were used as raw materials. Hot press sintering was carried out in a UGP-1 (disks with dimensions Ø10×1 mm^2) under pressure, R, equal to 200 kg/cm^2, developed and designed at the Research Institute of Physics [20]. The selection of the HP modes was made based on shrinkage curves and adjusted in accordance with the data from microstructural analysis. Polarization of the samples was carried out in a polyethylenesiloxane fluid PES-5 at 413 K for (15–20) min in a field of strength of (5–6) kV/mm.

X-ray diffraction studies were carried out by powder diffraction using a DRON-3 diffractometer (filtered Co$K\alpha$-radiation, Bragg-Brentano focusing scheme). The study involved crushed ceramic objects, which made it possible to exclude the influence of surface effects, stresses, and textures that arise during the ceramic

Table. Elemental compositions, production conditions, and electrophysical characteristics of the objects under study are the basis of the materials being developed

No.	Elemental composition, Wt.%	Production conditions (T_1, T_2, T_{si}) K $\tau_1 = \tau_2 = 5$ hours $\tau_{si} = 1.5$ hours	Electrophysical characteristics (with MA and HP; without MA and HP*)			
			$\varepsilon^T_{33}/\varepsilon_0$	K_p	$\lvert d_{31}\rvert$, pC/N	Q_M
low-frequency materials						
1	Na_2O = 8.75–9.72; K_2O = 5.32–5.37; Nb_2O_5 = 75.05–75.76; CdO = 9.15–10.88	HP T_1 = 1220 K; T_2 = 1240 K; T_{si} =1240 K;	1360–2020 (1280–1930)*	0.32–0.33 (0.30–0.31)*	67–70 (62–64)*	1000–1090 (960–1040)*
mid-frequency materials						
2	Na_2O = 8.49–8.67; K_2O = 11.00–11.25; Li_2O = 0.49–0.65; NiO = 0.82–0.83; Nb_2O_5 = 60.68–61.98; Ta_2O_5 = 11.20–11.44; Sb_2O_5 = 5.35–7.15	HP T_1=1223 K; T_{si}=1393 K;	1095–1097 (1040–1045)*	0.42–0.43 (0.415–0.42)*	80–84 (72–76)*	45–49 (55–60)*
high-frequency materials						
3	Na_2O = 16.20–16.68; Li_2O = 1.12–1.15; Nb_2O_5 =8 1.25–81.98; SrO = 0.19–1.43	HP T_1 = 1143 K; T_2 = 1193 K; T_{si} = 1433 K;	110–125 (114–135)*	0.136–0.225 (0.125–0.193)*	7.1–11.3 (7.0–11.0)*	904–1338 (855–1060)*
4	Na_2O = 8.56–8.75; K_2O = 12.75–13.04; Nb_2O_5 = 77.28–77.35; CuO = 1.16–1.17	HP T_1=1223 K; T_{si} =1373 K;	240–355 (290–370)*	0.20–0.32 (0.18–0.29)*	20.0–30.0 (19.0–27.0)*	215–500 (205–400)*
high-temperature materials						
5	Li_2O = 21.15–21.65; Nb_2O_5 = 76.11–77.89; NiO = 0.22–1.32; TiO_2 = 1.24–1.41	HP T_1= 1050 K; T_{si} =1240 K;	48–51 $T_к$ > 1273 K (50–52)*	0.015–0.020 (0.010–0.012)*	0.37–0.51 (0.30–0.35)*	60–70 (50–57)*
highly sensitive materials						
6	Na_2O = 8.69–8.91; K_2O = 13.20–13.53; Nb_2O_5 = 74.51–76.37; CdO = 3.60–3.61	HP T_1=1043 K; T_2= 1093 K; T_{si} =1233 K;	510–610 (540–650)*	0.27–0.34 (0.25–0.32)*	28–32 $\lvert g_{31}\rvert$ = 5.9–6.2 mV·m/N (26–29)* ($\lvert g_{31}\rvert$=5.0–5.4 mV·m/N)*	115–123 (115–125)*
thermostable materials						
7	Na_2O = 8.54–8.67; K_2O = 11.06–11.22; Li_2O = 0.32–0.33; Sb_2O_5 = 3.44–3.49; Ta_2O_5 = 11.28–11.44; Nb_2O_5 = 61.05–61.95; NiO = 1.94–2.87; B_2O_3 = 0.97–1.44	HP T_1= 1223 K; T_{si} =1273 K;	1194–1200 ($\Delta\varepsilon^T_{33}/\varepsilon_0$ = 2–3 %) (1140–1150)* ($\Delta\varepsilon^T_{33}/\varepsilon_0$ = 4–5 %)*	0.30–0.32 (ΔK_p=5–6 %) (0.28–0.30)* (ΔK_p = 6–7 %)*	59–62 ($\Delta\lvert d_{31}\rvert$=5–6 %) (53–58)* ($\Delta\lvert d_{31}\rvert$ =6–7 %)*	80–82 (ΔQ_M = 3 %) (72–76)* (ΔQ_M = 4 %)*
pyroelectric materials						
8	Na_2O = 18.38–18.73; Nb_2O_5 = 78.79–80.32; TiO_2 = 0.49–1.46; CoO = 0.46–1.37	HP T_1=1070 K; T_2= 1120 K; T_{si} = 1380 K	180–220 $\gamma = (1.44–1.5)\cdot10^{-4}$ C/m² ·K (205–240)* ($\gamma = (1.3–1.4)\cdot10^{-4}$ C/m² ·K)*	0.16–0.168 (0.145–0.150)*	11–12 (10–11)*	340–390 (280–320)*
9	Na_2O = 16.21–18.73; Nb_2O_5 = 69.52–80.32; TiO_2 = 0.49–7.37; NiO = 0.46–6.90	HP T_1 = 1070 K; T_2 = 1120 K; T_{si} = 1380 K	170-250 $\gamma = (1.6–1.7)\cdot10^{-4}$ C/m² ·K (195–230)* ($\gamma = (1.5–1.6)\cdot10^{-4}$ C/m² ·K)*	0.13-0.16 (0.12–0.14)*	9-10 (8–9)*	320-380 (250–310*

manufacturing process. The calculation of structural parameters was carried out according to the methodology presented in the work [20]. The errors in measuring the structural parameters had the following values: linear $\Delta a = \Delta b = \Delta c = \pm(0.002–0.004)$ Å; angular $\Delta\alpha(\beta) = 0.05^{o}$; volume $\Delta V = \pm 0.05 Å^3$.

X-ray density was calculated using the formula: $\rho_{xray} = MZ / (N_A V)$, where M is the molecular weight, Z is the number of formula units per unit cell, N_A – Avogadro's number, V – volume of the unit cell. The experimental density, ρ_{exp}, of the samples was determined by hydrostatic weighing in octane. Relative density, ρ_{rel}, was calculated using the formula $(\rho_{exp}/\rho_{xray})\cdot 100$ %.

To certify the electrophysical properties of the investigated SSs, measurements of their dielectric, piezoelectric and electroelastic parameters were carried out at room temperature in accordance with [21]. In this case, the relative permittivity of polarized samples was determined, $\varepsilon^{T}_{33}/\varepsilon_0$, piezomodule, $|d_{31}|$, the coefficient of electromechanical coupling of the planar oscillation mode, K_p, mechanical quality factor, Q_M, including specific parameters of the developed materials (Curie temperature, T_C, piezo sensitivity, $|g_{31}|$, pyroelectric coefficient, γ, thermal stability – $\Delta\varepsilon^{T}_{33}/\varepsilon_0$, ΔK_p, $\Delta|d_{31}|$, ΔQ_M).

3. Results and discussion

The Table shows the optimized elemental compositions of the studied objects: the basis of the developed materials, the technological conditions for their production, and the main electrophysical characteristics.

As can be seen from the table, the use of MA and HP operations during the two main stages (synthesis and sintering) of ceramic production led to a significant improvement in the macro properties of the objects. The reason for the observed phenomenon may be the transformation of their internal structure (crystalline, granular) as a result of the influence of external factors. Thus, the grinding of batches during MA led to the acceleration of diffusion phenomena during the synthesis process, and sintering of synthesized powders under pressure led to the activation of mass transfer during recrystallization. This ensured a decrease in the temperatures of synthesis and sintering and, as a consequence, a reduction in the extent of morphotropic heterophase regions in the corresponding SS systems and a "shift" of the selected objects into single-phase regions.

Fig. 1 shows X-ray diffraction patterns of some of the materials presented for illustration purposes. X-ray diffraction analysis has shown

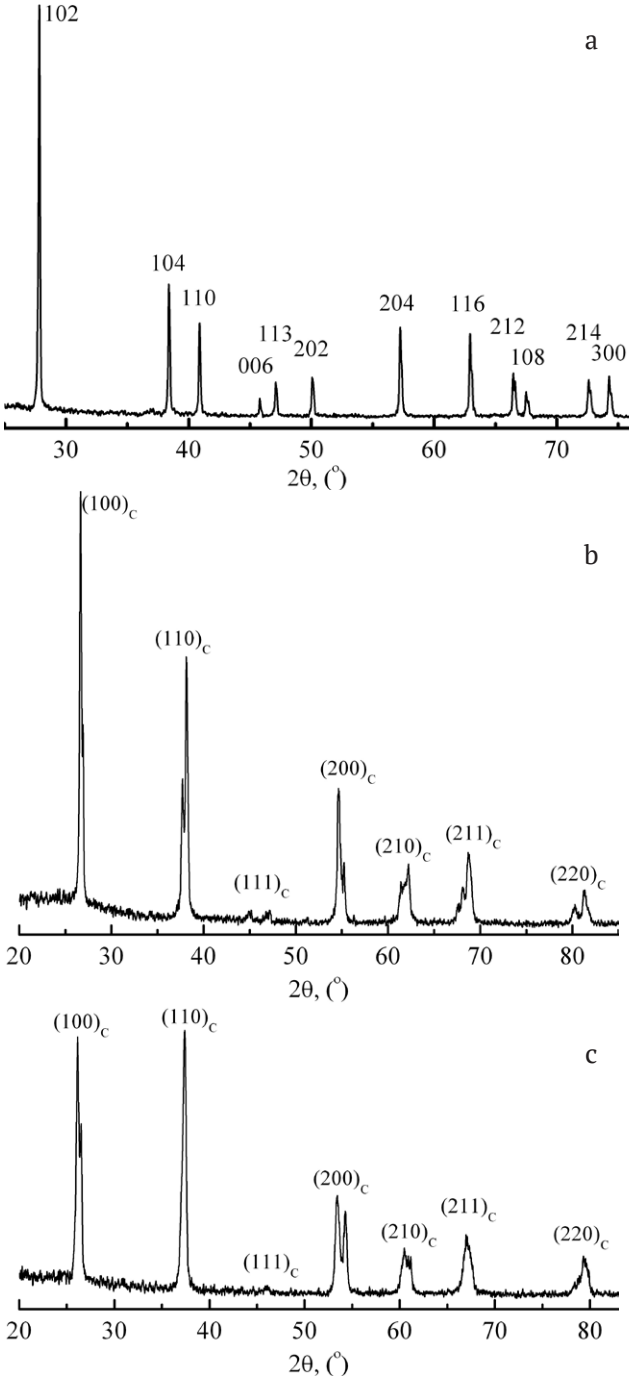

Fig. 1. X-ray diffraction patterns of materials: high-temperature (ilmenite-type structure) – a; high-frequency (perovskite-type structure) – b; high-sensitivity (perovskite-type structure) – c

that the resulting ceramic compositions are pure and have a density higher than 95 % of the theoretical value, which is typical for the technologies used [22]. The X-ray diffraction patterns show that all materials are well structured without impurity phases. This implementation of more favorable conditions for diffusion and recrystallization processes ensures the formation of a more perfect microstructure of ceramics [23] and, as a consequence, improving their dielectric and piezoelectric properties.

The specific gravity of all the developed materials is about 4 g/cm^3, which is half as much as in the known Pb-containing analogues. This allows, as mentioned above, to use these materials in devices in which weight characteristics are decisive.

The obtained low-frequency materials can be used in low-frequency receiving devices – hydrophones, microphones, seismic receivers. Medium-frequency materials can be used in radio-electronic devices operating in reception mode, including in transducers of ultrasonic (US) transmitters.

High-frequency materials can be used for ultrasonic delay lines on bulk and surface waves and medical diagnostic devices. Highly sensitive materials can be useful, for example, in devices for measuring mechanical impacts (pressure). High-temperature materials can be used to create reusable generator-type piezoelectric sensors for monitoring equipment (nuclear reactors, nuclear missile systems) subject to extreme thermal effects. Thermally stable materials will find application in the creation of knock sensors for internal combustion engines. Pyroelectric materials will be useful for creating highly efficient thermoelements in radiation pyrometry devices based on pyroelectric phenomena.

4. Conclusions

Thus, the analysis of the above information showed that it is possible to improve the properties of ferroelectric ceramic materials, free of toxicity and lead, with low specific gravity by using mechanical activation during synthesis followed by hot press sintering for applications in devices where weight characteristics are decisive, as well as in multi-frequency systems as thermocouples and in other electronic devices.

Author contributions

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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