Chem. Chem. Technol., 2022,
Vol. 16, No. 2, pp. 337–344

Chemical Technology

HIGH-STRENGTH GLASS-CERAMIC MATERIAL WITH LOW TEMPERATURE FORMATION

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https://doi.org/10.23939/chcht16.02.337

Abstract. Prospects for development of glass-ceramic materials on the lithium aluminosilicates base in order to increase the reliability of armor protection elements have been analyzed. Compositions of lithium aluminosilicate glasses with low content of lithium oxide have been developed, spodumene glass-ceramic materials were obtained on their base in conditions of low-temperature thermal treatment. Formation of structure of glass-ceramic materials based on model glasses after thermal treatment has been investigated and the influence of phase composition on mechanical properties has been established. It was determined that the developed glass-ceramic materials are feasible for the application against the action of high-energy munitions with significant penetrating ability, especially when used in combination with ceramic elements.

Keywords: lithium aluminosilicate glasses, low temperature formation, spodumene glass-ceramics, high-strength material, armor protection element.

1. Introduction

High-strength glass-ceramic materials have become widely used in multiple branches of science and engineering because of unique combination of physico-chemical and performance characteristics. Despite significant achievements in the creation and application of the wide range of glass-ceramic materials that are currently known, the prospects for improving existing and developing fundamentally new materials and coatings for this purpose and technologies for their production are relevant. This is especially related to medicine, radiation safety, the military industry, etc.[1] One of the effective solutions of this problem is development of high-strength glass-ceramic materials with increased values of mechanical strength and reduced weight for their use as means of personal and collective armor protection.

Currently known ceramic materials (corundum, boron carbide and silicon carbide) used in personal protective equipment have high values of physico-chemical properties: Young's modulus E = 350 – – 407 GPa, Poisson ratio μ = 0.17 – 0.22, critical stress intensity factor K_{1C} = 3.2 – 4.0 MPa·m$^{1/2}$, bending strength σ_{bend} = 220 – 440 MPa, specific fracture work 180 – 260 J/m^2.[2] However, the high cost of B_4C and the significant weight of armor elements based on Al_2O_3 (ρ = 3.9 g/cm^3) and SiC (ρ = 3.0 g/cm^3) limits the possibility of their use as elements of composite armor. Also, when studying the influence of technological factors on the effectiveness of ceramic-based armor elements, the expediency of introducing an additional damping layer into the armor element structure was established. One of the effective solutions to this problem is the development of new types of high-strength glass-ceramic materials with increased values of mechanical strength, thermal resistance and, at the same time, reduced weight.

Recent advances in development of new glass-ceramic materials based on magnesium aluminosilicate glasses are concerned, in particular, with the creation of high-strength glass-ceramics for protection against high-energy weapons. Cordierite glass-ceramic materials[3] with high mechanical properties, Knoop hardness and low density (Table 1) as constituent of the composition are effective protection against high-energy weapons

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with high penetrating power and allow increasing the mobility of equipment due to weight reduction. However, the relatively high temperatures of melting and heat treatment of glass for obtaining these glass-ceramic materials limits their use as protective armor elements. Anorthite glass-ceramics[4] obtained according to the low-temperature regime (melting temperature at 1793 K for 120 min; heat treatment: first stage – T = 1223 K, duration 30 min, second stage – T = 1293 K, duration 4 hours) are characterized with insufficiently high values of mechanical properties for their use as effective armor protection under conditions of significant dynamic loads.

A combination of manufacturability, low density values ρ = 2.35–3.45 g/cm^3, high mechanical strength and thermal resistance to ensure resistance to the action of energy-destructive constituents and the ability to absorb and dissipate impact stresses (shock wave velocity v = 6.0–6.4 km/s) makes it possible to distinguish glass-ceramics based on the Li_2O–Al_2O_3–SiO_2 system as the most promising group of materials for armor protection elements (Table 1). However, the known spodumene glass-ceramic materials are characterized by a sufficiently high content of lithium oxide and significant temperatures of melting and heat treatment of the initial glasses and their duration,[5-7] and glass-ceramic materials based on lithium disilicate are distinguished by low heat resistance and prolonged heat treatment (40–120 hours).[8] High-strength glass-ceramic material for multifunctional application, containing 30–65 wt. % of lithium disilicate and 20–60 wt. % of β-spodumene,[9] due to the peculiarities of its structure is characterized by low values of density and fracture toughness. Although the values of hardness indicators according to Knoop = 680 and TCLE α = (65.0–80.0) · 10^{-7} deg^{-1} limits the use of these materials as fire-resistant elements of armor protection. Therefore, today, the relevant objective of increasing the reliability of special equipment protection, which is operated under conditions of high temperatures and mechanical loads, is the development of compositions of lithium aluminosilicate glasses with a low content of lithium oxide and obtaining of spodumene glass-ceramic materials of the indicated purpose on their basis under conditions of low-temperature heat treatment. Exactly this, as well as the study of the influence of their phase composition on the mechanical properties, is the purpose of this work.

Table 1. Known domestic and foreign glass-ceramic materials for elements of armor protection

System	Crystalline phase	Properties	Country, inventor
MgO–Al_2O_3–SiO_2	α-cordierite	ρ = 2.7–3.1 g/cm^3; HK = 608–1100 E = 105–150 GPa σ_{bend} = 175–229 MPa	USA, R.W. Jones[3]
CaO–Al_2O_3–SiO_2	anorthite	ρ = 2.7 g/cm^3; HV = 9.3–10.0 GPa; σ_{bend} = 150–300 MPa, E = 100 GPa; α = 52·10^{-7} °C^{-1}	USA, A. Raichel, A. Nachumi, S. Raichel[4]
Li_2O–Al_2O_3–SiO_2; ZrO_2, TiO_2	keatite	n < ±0.3	USA, F. Siebers[6]
Li_2O–Al_2O_3–SiO_2; ZrO_2, TiO_2,As_2O_3	β-quartz, solid solutions	ρ = 2.55 g/cm^3; HK = 600; E = 90 GPa; K_{1C} = 0.91 MPa·m$^{1/2}$	USA, L.R. Pinckney[7]
Li_2O–Al_2O_3–SiO_2; ZnO, P_2O_5	β-spodumene	ρ = 2.45 g/cm^3; HK = 580; E = 104 GPa; σ_{bend} = 220 MPa	USA, R.W. Jones[3]
Li_2O–Al_2O_3–SiO_2; P_2O_5; Fe_2O_3; CeO_2	β-spodumene	ρ = 2.4 g/cm^3; HK = 535; E = 88 GPa; σ_{bend} = 250 MPa	

2. Experimental

2.1. Research methods

The presence of the crystalline phase was established by X-ray diffraction and petrographic analysis methods using DRON-3 apparatus and a MIN-8 optical microscope. The microstructure of glasses was investigated using "EMV 100 AK" electron microscope, microhardness H and Vickers hardness HV were established with PMT-3 and TP-2 devices. The Young's modulus was determined by the deflection arrow of thread of glass samples. The viscosity of the glass was determined by the method of stretching the fiber using viscometer of JSC "Institute of Glass". The presence of sybotaxic groups in the structure of glasses was

determined by IR spectroscopy using a Specord-M80 instrument. Ballistic tests were carried out in accordance with the requirements of STANAG 4569.

2.2. Development of compositions of lithium aluminosilicate glasses for obtaining spodumene glass-ceramic materials

High strength of lithium aluminosilicate glass-ceramic materials can be achieved by providing finely dispersed volume crystallization of the initial glass with the formation of β-spodumene crystalline phases via directional crystallization using the phase separation mechanism.

Based on the results of previous studies on the glass formation region of glasses in the R_2O – LiF – CaF_2– RO – RO_2 – R_2O_3 – P_2O_5 – SiO_2 system, their crystallization ability and mechanical properties,[10,11] the authors of the work chose the SP-9 composition, which is characterized by a sufficiently high indicators of hardness. To increase indicators of impact stress and fracture viscosity, the SP-10 composition was synthesized, which is characterized by differences in chemical composition in the direction of the formation of an interlocked sitallized structure.[12] To form the volume-crystallized fine structure by the liquation mechanism, TiO_2, ZrO_2, and SnO_2 were introduced into the SP-10 composition along with P_2O_5 (3 wt. %) and ZnO (3 wt. %) (Table 2). The presence of CeO_2 (0.5 wt. %) and B_2O_3 (1.5 wt. %) in the compositions of glass-ceramic materials will allow nucleation to proceed and the formation of crystalline phases in the lower temperature range.

Technological parameters of melting and heat treatment are given in the Table 2. Glass-ceramic materials were obtained using pressing by ceramic technology. The initial glasses were milled until passing completely through sieves: No. 125 with a particle size of 63–125 μm ≈ 70 vol. %, No. 063 – 25 – 63μm – 15 vol. %, No. 025 no more than 25 μm – 15 vol. %. The size of the fractions and their ratios were selected taking into account the data on ensuring a high packing density of materials. The solution of xanthan gum in an amount of 10 wt. % was used as a binder. A three-stage pressing of the material mass in a hydraulic press with the pressure at the first stage – 7.36 MPa, at the second stage – 11.78 MPa, at the third stage – 14.71 MPa was carried out. The pressed materials with the marking were dried at the temperatures of 393 – 423 K to a residual moisture content of not more than 0.5 %, followed by a two-stage short-term heat treatment.

Table 2. Differences in the chemical composition of model glasses, crystalline phases that form after melting and heat treatment, and technological parameters

Marking	Chemical composition of model glasses, wt. %						Technological parameters		Crystalline phase characteristics	
	Phase-forming components			Nucleation catalysts			Melting temperature, K	Temperature of heat treatment stages, K; (τ – 2–4hours)	Type	Content, vol. %
	Li_2O	Al_2O_3	SiO_2	ZnO	ΣTiO_2, ZrO_2	SnO_2				
SP-9	8.0	20.0	60.0	3.0	–	–	1673	Stage I – 803 Stage II – 1123	β -$LiAlSi_2O_6$	80
SP-10	7.0	18.0	60.0	–	3.0	1.0	1673	Stage I – 803 Stage II – 1123	β -$LiAlSi_2O_6$	80

3. Results and Discussion

3.1. Investigation of the structure formation and phase composition of glass-ceramic materials based on model glasses during their heat treatment

Petrographic analysis of SP-9 and SP-10X-ray amorphous model glasses showed that they were formed on the basis of a colorless anisotropic eutectic melt. This fact indicates that the composition of model glasses contains fluctuation inhomogeneities, which acquire the greatest development near the eutectic points, where the nucleation of two phases at once is possible. This is what suggests that during the subsequent heat treatment of these glasses, a strengthened finely dispersed structure can form. After a two-stage heat treatment, glass-ceramic materials are characterized by volume finely dispersed crystallization of a high-strength crystalline phase of β-spodumene in an amount of 80 vol. %.

It is known[5] that the fluctuation structure has a kinetic nature and is therefore capable of developing most strongly in systems with low energy barriers on the path of structural and chemical transformations, with a short relaxation time, in particular, in systems with low viscosity. However, the high viscosity of glass determines an important contribution of the kinetics of the process: in the allotted time the glass cannot separate into two layers, the precipitated glassy phase forms fine droplets, which leads to the formation of a developed droplet two-frame structure in a short time.

The main difference between the metastable phase separation process is that it takes place under conditions of increased viscosity of both the initial melts and the forming phases, that is, the process proceeds relatively slowly. This affects the formation of a significant number of crystal nuclei at the temperature of the onset of softening T_f and the formation of a sitallized structure at the final crystallization temperature. Under such conditions, it is possible to create two-phase materials with small dimensions of phase new formations, which is the key to high mechanical strength of the final product.[6]

According to the results of studying the viscosity (Fig. 1), it was established that a feature of the SP-9 and SP-10 glasses is an anomalous increase in viscosity in the glass transition range T_g–T_f = 723–823 K, which is associated with the formation of fluctuations. The high structural viscosity of the SP-9 and SP-10 glasses is determined by the degree of connectivity of the silicon-oxygen glass frame f_{Si} = 0.28. For SP-10 glass, an exponential increase in viscosity is observed in the temperature range of 723–823 K and moderate increase in viscosity – 823–923 K, with the maximum at 873 K ($\eta = 10^{9.26}$ Pa·s), which is associated with the formation of fluctuations in the range of lower temperatures due to phase separation in comparison with SP-9 with the maximum at 923 K ($\eta = 10^{9.17}$ Pa·s). The intense formation of fluctuations at the temperature of 823 K indicates a high crystallization viscosity of the SP-10 glass-ceramic material, which will have a positive effect on the formation of a finely crystalline structure in the range of low temperatures for a shorter period.

To study the mechanism of nucleation and structure formation in the developed materials, the following characteristic temperatures were chosen: temperature $t1$ after melting; temperature $t2$ after heat treatment near the temperature of the softening onset T_S; temperature $t3$ is higher than T_E; and the "final" crystallization temperature $t4$. To identify the structure of the glass material at the initial stages of nucleation, areas free from crystallization were selected.

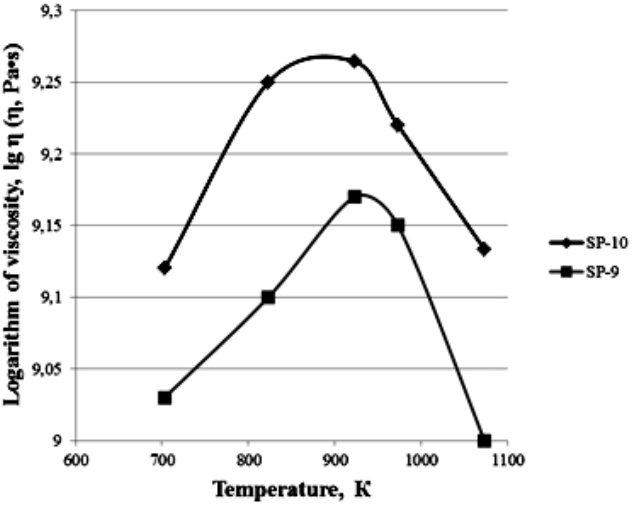

Fig. 1. Dependence of the glass-ceremic materials viscosity on temperature

According to the results of electron microscopy, the samples of SP-9 and SP-10 glass materials after melting represent a multiphase system, which is formed from parent glass, drop-shaped inhomogeneities with the size of 0.07–0.09 μm and clusters of spherical formations with the size of 0.01–0.10 μm. These inhomogeneities have arisen within such formations 0.5–1.0 μm in size (Fig. 2a, I). Secondary separation of spherical formations in the experimental glass materials, as a stage of heterogeneous nucleation of crystal nuclei, is associated with heterophase fluctuations (Fig. 2b, I).

After heat treatment of the experimental glass materials near T_S, a liquation inhomogeneous structure is observed with the enlargement of individual inhomogeneities to 0.1–0.2 μm, which were formed as a result of a gradual change in the composition of neoformations from the periphery of the drops to the center with distinct phase boundaries (Fig. 2c,d I). Their distinctness on the surface of spherical inhomogeneities is confirmed by shadows outside the contours of drops (Fig. 2c,d II). The SP-10 glass material is characterized by the presence of a significant amount of spherical nanoinhomogeneities, which can act as nucleators of lithium metasilicate (Fig. 2d).

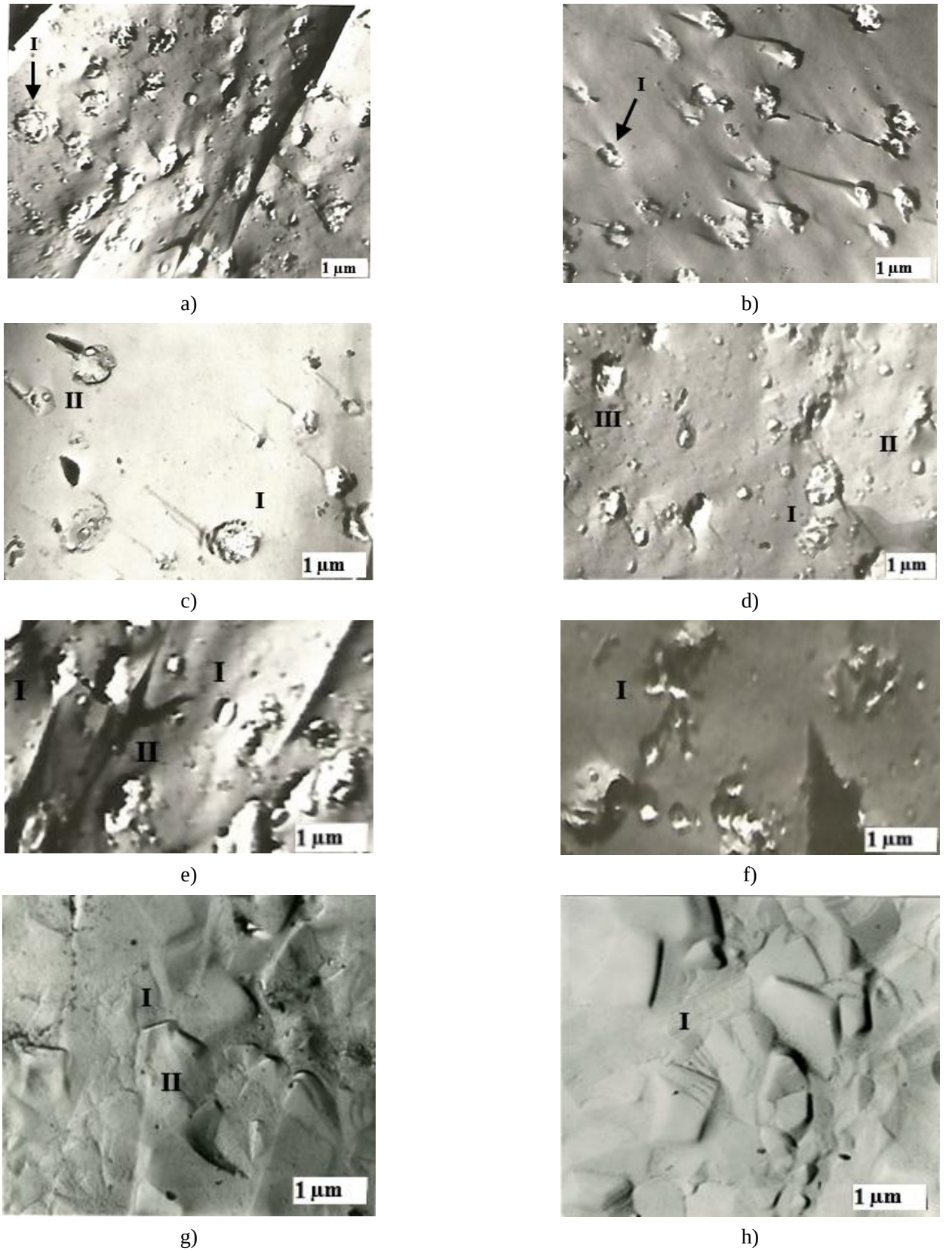

Fig. 2. The structure of glass materials after melting at the temperature *t*1 SP-9 (a) and SP-10 (b); after heat treatment at temperatures: *t*2 823 K SP-9 (c), SP-10 (d), *t*3 923 K SP-9 (e), SP-10 (f), *t*4 1123 K SP -9 (g), SP -10 (h)

The evidence of the intense formation of the Li_2SiO_3 nucleator for glass materials are the absorption bands in the ranges 550–750 cm^{-1} and 880–1100 cm^{-1} (Fig. 3a), which are characteristic of the spectra of metasilicates. The number of frequencies in this range corresponds to the symmetric vibration of Si–O–Si and, according to A. N. Lazarev, it makes it possible to estimate the number of tetrahedra in the repetition period. For the SP-10 glass material, the presence of absorption spectra of Li_2SiO_3 in the 550, 600, 720, and 780 cm^{-1} bands is more intense. This may indicate the possibility of the formation of a significant amount of a finely dispersed crystalline phase.

The continuous growth of the phase at *t*2 is a characteristic feature of the occurrence of metastable liquation as a phase transition. The presence of inhomogeneities in the glass materials in the form of depressions and alignment of drops indicates the completion of the phase separation process followed by the process of structure ordering, which is more intense for SP-10. Crystals of the most symmetric metastable phases, wedge-shaped, appear at the drop boundary (Fig. 2d, III). These crystals are most easily wetted by glass and, as a consequence, are more closely bound to glass.

During treatment of the SP-9 and SP-10 glass materials at temperature *t*3, along with the coarsening of the formation of the fluctuation structure (Fig. 2e,f I), the growth of wedge-shaped crystals is observed, which have arisen at the boundary of spherical inhomogeneities (Fig. 2e, f II). This may indicate the presence of *β*-eucryptite with a hexagonal lattice. The presence of *β*-eucryptite is observed in the IR absorption spectra of glass materials in the bands of 480, 660, 750, and 1003 cm^{-1} (Figs. 2,3, b). Crystallization of *β*-eucryptite will negatively affect the mechanical properties of glass materials. Therefore, prolonged exposure of glass materials at the temperatures of formation of a given crystalline phase should be avoided.

The structure of the SP-9 and SP-10 glass materials at the temperature of *t*4 = 1123 K is characterized by a dense network of crystals of a columnar planar prismatic habit with shading with clear cleavage, connected by their ends to each other (Fig. 2g,h I), characteristic of *β*-spodumene. This is confirmed by the presence of clear absorption bands of *β*-spodumene at 740 cm^{-1} in the SP-9 and SP-10 materials. The presence of this band indicates that the silicon – oxygen tetrahedra are connected to each other at all four vertices (Figs. 2,3, c). For glass materials, a significantly narrowed band of 1150–1000 cm^{-1} is observed for β-spodumene (a typical wide band for chain silicates is 1200–800 cm^{-1}). In this case, absorption is most intensely observed in the 1026 cm^{-1} band.

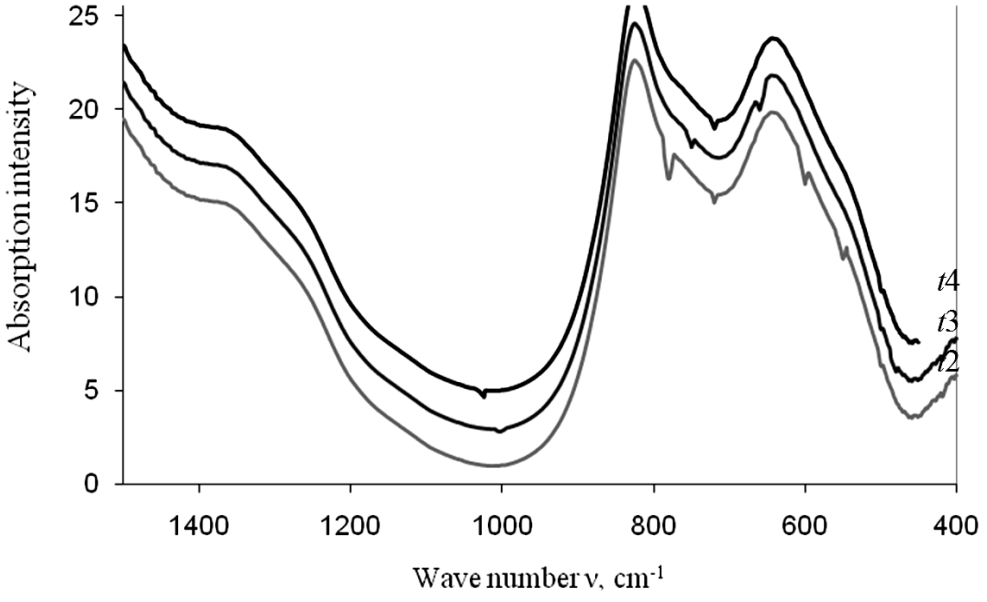

Fig. 3. IR-spectra of experimental glass materials: t2(a), t3(b), t4(c)

The presented course of crystallization is the most typical for the formation of a finely crystalline structure, which provides higher values of the mechanical properties of crystallized glass materials. However, for the SP-10 glass material under the given heat treatment regime, the formation of a volume crystallized structure

is observed already after 2 hours of heat treatment at 1123 K. For SP-9 glass, only for 4 hours when the temperature rises to 1123 K, β-spodumene crystals grow, the cracks heal with glass residues and a sitallized structure is formed.

Taking into account the above results, a two-stage heat treatment mode was selected at temperatures of the first stage – 803 K and the second stage for the SP-10 glass material – 1123 K, for SP-9 glass material – 1123 K for 4 hours (Table 2).

3.2. Development of glass-ceramic materials and investigation of their mechanical properties

Previous low-temperature crystallization introduces significant changes tp the character of crystallization of experimental glass materials. Thus, in the case of crystallization at the temperature of 823 K for the SP-9 and SP-10 glass materials, nucleation and growth of lithium metasilicate crystals occur. As a result, the glass is depleted in lithium oxide so that the β-eucryptite solid solution, which leads to strength loss of the structure, is formed in an insignificant amount.

At the second stage of heat treatment for the SP-9 and SP-10 glass materials, the presence of β-spodumene crystals with the size of ≈ 0.5 – 1.0 μm and ≈ 0.1– 0.5 μm, respectively, in the amount of 80 vol. % together with glass interlayers, which act as a damper, are the key to the high strength of the glass-ceramics (Table 3). The finely dispersed sitallized structure of these glass materials is provided by the ratio of phase-forming oxides, the presence and content of nucleation catalysts.

Therefore, due to the presence of finely dispersed β-spodumene crystals, which are uniformly distributed in the volume of the experimental glass-ceramic materials in the amount of 80 vol. %, they are characterized by such values of mechanical properties (Table 3) which allow them to be operated under conditions of significant mechanical loads.

For the developed glass-ceramic materials based on lithium aluminosilicate glasses, the formation of a dissipative sitallized structure by the mechanism of phase separation under conditions of low-temperature heat treatment makes it possible to ensure their high operational properties and their use under conditions of significant mechanical and thermal loads.

Table 3. Properties of the developed glass-ceramic materials

Marking of materials	Properties					
	H, MPa	*HV*, MPa	K_{1C}, MPa·m$^{1/2}$	*E*, GPa	$\alpha \cdot 10^7$, grad^{-1}	ρ, g/cm^3
SP-9	9084	8667	3.4	100.0	20.78	2.44
SP-10	9095	8680	3.4	95.6	21.34	2.43

The limit of conditioned damage or the ballistic limit V_{50} of protective elements based on theSP-9 and SP-10 glass-ceramic materials is determined by the speed V_{50} = 910 ± 15 m/s of a 5.45 mm bullet (cartridge 7N10), below which reliable protection is provided.[10] The carried out ballistic tests showed that the developed SP-10 withstood shelling according to the requirements of STANAG 4569 and meets the 2nd level of protection.

The effectiveness of the application of glass-ceramic materials in the composition of the composite armor element "ceramics – glass-ceramic material – polymer" is achieved by their simultaneous use as energy-destroying and energy-absorbing layers. This will reduce the weight of the armor element and ensure their high operational properties.

4. Conclusions

Lithium aluminum silicate glasses and high-strength glass-ceramic materials on their basis under conditions of short-term low-temperature heat treatment have been developed.

The features of the formation of a structure in lithium aluminosilicate glass-ceramic materials at the initial stages of nucleation and their influence on the formation of a self-ordering sitallized structure were analyzed. The effect of the thermal prehistory of the

developed glasses on their structure during heat treatment has been established. The mechanism of phase formation in glasses of the $K_2O – Li_2O – RO – RO_2 – Al_2O_3 – B_2O_3 – P_2O_5 – SiO_2$ basic system was determined, which consists in the occurrence of volume fine crystallization of glass due to the intensive formation of nucleation of lithium metasilicate (T = 823 K) in the form of spherulites at the viscosity of $\eta = 10^{9.26}$ Pa·s; the formation of β-eucryptite crystals (T = 923 K) at the viscosity of $\eta = 10^{9.17}$ Pa·s and their recrystallization into β-spodumene crystals of a columnar planar prismatic habit (T = 1123 K), which are firmly connected.

The factors that determine the formation of a finely dispersed volume-crystallized structure under conditions of low-temperature two-stage heat treatment and ensuring a high level of strength of glass-ceramic materials in the mentioned lithium-containing system are: the ratio of phase-forming oxides Al_2O_3: SiO_2 = 1 : (3.3 – 4); type and content of nucleation catalysts P_2O_5, ZnO, TiO_2, ZrO_2, SnO_2, and CeO_2; and a certain temperature-time regime of heat treatment of the initial glasses.

It has been established that providing of high mechanical properties of the developed glass-ceramic materials makes it possible to consider them promising for operation under conditions of exposure to high-energy weapons with significant penetrating ability, especially when used in combination with ceramic elements. The introduction of the developed high-strength glass-ceramic material based on β-spodumene, which is characterized by level 2 of protection according to the STANAG 4569 requirements, will increase the competitiveness of domestic armor materials and provide indicators of their properties at the level of world analogues.

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Received: November 17, 2020 / Revised: December 3, 2020 / Accepted: December 5, 2020

ВИСОКОМІЦНІ СКЛОКЕРАМІЧНІ МАТЕРІАЛИ З НИЗЬКОЮ ТЕМПЕРАТУРОЮ ФОРМУВАННЯ

Анотація. *Проаналізовано перспективи розвитку склокерамічних матеріалів на основі алюмосилікатів літію з метою підвищення надійності елементів бронезахисту. Розроблено склади літійалюмосилікатних стекол з низьким вмістом оксиду літію, на їхній основі отримані сподуменові склокерамічні матеріали в умовах низькотемпературної термообробленя. Досліджено формування структури склокерамічних матеріалів на основі модельних стекол після термічного оброблення і встановлено вплив фазового складу на механічні властивості. Встановлено, що розроблені склокерамічні матеріали доцільні для застосування проти дії високоенергетичних боєприпасів зі значною пробивною здатністю, особливо в поєднанні з керамічними елементами.*

Ключові слова: *літійалюмосилікатні стекла, низькотемпературне формування, сподуменова склокераміка, високоміцний матеріал, бронезахисний елемент.*