

INVESTIGATION OF THE STRENGTH AND ELASTIC PROPERTIES OF GLASS-CERAMIC OF A LITHIUM ALUMOSILICATE COMPOSITION

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This paper presents the results of investigations of the strength and elastic properties of the glass-ceramic obtained by novel technology with the use of the method of casting blanks from high-density aqueous suspensions of lithium aluminosilicate glass into porous moulds followed by their sintering and crystallization under combined conditions of thermal treatment. Comparison of the properties of the proposed glass-ceramic with the properties of the glass-ceramics obtained by conventional technology enabled us to gain a better insight into its potentialities and provide the necessary information for calculating the structural strength.

The mechanical strength of materials is one of the major characteristics determining in most cases their suitability for a particular practical application. It is desirable that materials retain their mechanical strength in the conditions of environmental impacts, at heightened temperatures, and under the influence of operational and other factors. This is particularly important for materials meant for use in the structures of flying vehicles, since a structural failure can make an expensive article inoperative with unpredictable consequences.

At the same time, publications devoted to the investigation of the strength properties of glass-ceramics are fragmentary in character, which makes it difficult to explore the possibility of using them in particular structures in more detail.

This paper presents the results of investigations of the strength and elastic properties of the glass-ceramic of a lithium aluminosilicate composition obtained by the method of slip casting of blanks into porous molds followed by their sintering and crystallization under combined conditions of thermal treatment. In a number of cases, comparison of the level of properties of different materials is given, which makes it possible to gain a deeper insight into the glass-ceramics and provide the designer with the necessary source material for making calculations in developing particular objects proceeding from the conditions of impacts of various factors.

Flexural strength measurements at room and higher temperatures were made on standard devices by standard procedures according to the scheme of three-point loading on glass-ceramic specimens measuring $7 \times 7 \times 70$ mm whose surface was subjected to abrasive treatment to attain a roughness of $R_a = 0.6\text{--}0.7$ μm .

In [1], it is noted that the glass-ceramics of the lithium aluminosilicate system covering the class of materials with a low thermal linear expansion coefficient (TLEC) are inferior in their mechanical strength to the pyroceramics containing cordierite as the main crystalline phase. The results of the tests of the glass-ceramic specimens obtained by ceramic technology were not an exclusion (Table 1).

The lower flexural strength values of the pyroceramics and glass-ceramics of a β -spodumene composition compared to the pyroceramics of a cordierite composition can be attributed to the lower thermal linear expansion coefficient of these materials. As a result, in the glassy phase, at the boundaries of the crystals formed, on cooling, tensile stresses leading, in the final analysis, to a decrease in the strength can arise. This phenomenon can also explain the lower mechanical strength in materials in which β -eucryptite is present as the main crystalline phase because it has even lower values of the thermal linear expansion coefficient. However, the glass-ceramic strength is high enough and comparable to the strength of the pyroceramics of a similar composition obtained by the traditional glass technology (Table 1).

Attention is drawn to the fact that most of the tested glass-ceramic specimens ($\sim 75\%$) have a flexural strength higher than the mean value; in 9% of the specimens this value is in the 90–95 MPa range, and only 3% of the specimens have the lowest values (85 MPa) (Fig. 1).

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TABLE 1. The Most Common Literature Values of the Flexural Strength at Room Temperature of Certain Kinds of Pyroceramics, Glasses, and Ceramics

Material	Flexural strength σ_{flex}
Pyroceram 9606 (pyroceramic of the cordierite system, USA)	120–260
Pyroceram 9608 (pyroceramic of the β -spodumene system, USA)	110–140
AS-370 (pyroceramic of the cordierite system, Ukraine)	160–180
AS-418 (pyroceramic of the β -spodumene system, Ukraine)	100–120
OTM-357 (glass-ceramic of a β -spodumene composition, Russia)	100–120
Quartz glass	70–80
Borosilicate glass	50–70
Quartz ceramics (Russia)	40–60
High-alumina ceramics (Russia)	210–350

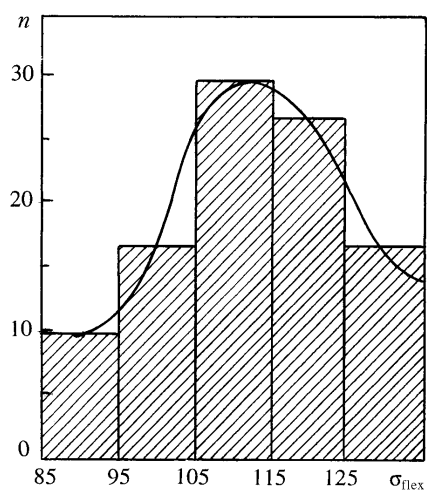


Fig. 1. Histogram of the flexural strength distribution of glass-ceramic specimens of a lithium alumosilicate composition (shaded rectangles show the number of specimens in % of the tested ones with the flexural strength values in the given ranges). $T = 20^{\circ}\text{C}$, $\sigma = 111.7 \text{ MPa}$, $K_v = 12\%$. n , %; σ_{flex} , MPa.

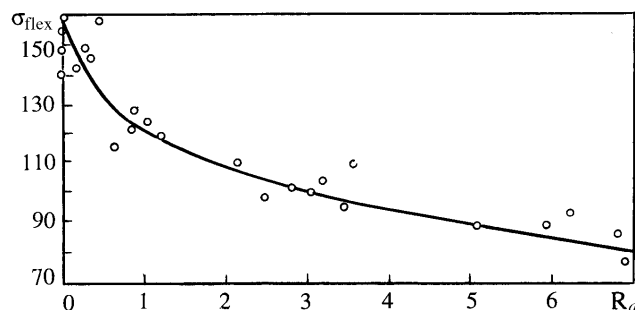


Fig. 2. Flexural strength of glass-ceramic of a lithium alumosilicate composition versus surface roughness of test samples. σ_{flex} , MPa; R_a , μm .

The results obtained point to the statistical character of the strength values of glass-ceramics, which is typical of all ceramic materials and glasses. For example, the variation coefficient of the strength values for quartz ceramics comes to 23–25%; for densely sintered high-alumina ceramics — 10–17%; for ceramics from reaction-bonded silicon nitride — 15–20%; and for glass ceramics of a lithium alumosilicate composition — 8–15% [2–5].

It should be remembered, however, that the above values of the variation coefficient can only be regarded as the general trend of the character of the flexural strength distribution in the specimens, since the authors often give neither the size of the specimens, nor the testing conditions, nor the history of the specimen preparation for testing, although all these factors produce a significant effect on the strength of materials.

For instance, the investigations on the influence of the quality of the treated surface of pyroceramic AS-418 specimens on the strength properties revealed a considerable decrease in the strength with increasing roughness of the test specimens with a simultaneous increase in the variation coefficient [3].

Although comprehensive studies on the influence of the degree of surface roughness of specimens of glass-ceramic of a similar composition on its strength properties confirmed the fact of a decrease in the latter (Fig. 2), we attributed the observed differences in the changes in the flexural strength magnitudes and variation coefficients of

TABLE 2. Influence of the Surface Roughness of Specimens on the Flexural Strength of Pyroceramic AS-418 and Glass-Ceramic OTM-357

Material	Conditions for the preparation of specimens and their size	Surface roughness of specimens, R_a	Mean flexural strength, σ_{flex}	Variation coefficient, K_v
Pyroceramic AS-418	Treated with carborundum wheel (120 × 25 × 10 mm)	0.04	124	7.8
		0.82	109	1.4
		1.50	54	18.5
Glass-ceramic OTM-357	Treated with free abrasive of different dispersiveness (7 × 7 × 70 mm)	0.02	154	10.7
		0.60	123	11.3
		3.00	103	13.0
		5.80	91	15.2

TABLE 3. Influence of the Velocity of Application of Load on the Flexural Strength of Pyroceramic AS-418 and Glass-Ceramic OTM-357

Material	Specimen size	Velocity of application of load, $V_{a,load}$	Mean flexural strength, σ_{flex}	Variation coefficient, K_v
Pyroceramic AS-418	120×25×10 mm	0.002	80	12.5
		5.0	111	8.1
		50.0	119	8.4
Glass-ceramic OTM-357	7×7×70 mm	0.04	114	15.2
		0.4	112	15.3
		4.0	116	15.8
		40.0	118	14.2
		100.0	113	11.8

pyroceramic AS-418 and glass-ceramic (Table 2) to the differences in the sizes of the tested specimens and the character of their preparation for testing.

Thus, the results presented (Fig. 2, Table 2) point to a strong dependence of the mechanical strength of materials on the surface state. This question deserves serious attention, since the manufacture of a particular article calls for mechanical treatment of the surface. And the pressure on the surface of specimens or articles from pyroceramics, glass, or ceramics of microcracks caused by mechanical treatment explains both the scale factor of strength and its statistical character, since the number of defects depends on the surface size and the probability of the appearance in a given particular specimen of the most dangerous defect in the form of a crack obeys statistical laws. This is clearly evident in testing specimens close in phase composition but differing in the character of their preparation for testing and in size (Table 2). A factor of a less pronounced trend towards a decrease in the flexural strength of glass-ceramic compared to pyroceramic AS-418 is the presence in the former of an insignificant number of closed pores serving as barriers to the propagation of a crack, whereas in pyroceramic AS-418 surface defects can lead to a catastrophic destruction of the material.

An important characteristic of a material meant for articles operating in the conditions of dynamic loading is the deformation rate or, which is the same, the rate of application of load.

The results of testing glass-ceramic OTM-357 on 7 × 7 × 70 cm specimens with a surface roughness of $R_a = 0.6 \mu\text{m}$ compared to the data of analogous testing of pyroceramic AS-418 on 120 × 25 × 10 specimens, according to the data of [6], are presented in Table 3.

In [6], it was concluded that a decrease in the rate of application of load with a corresponding increase in the time of action of the environment (i.e., water vapors contained in the air) on the crack tip at its stable development leads to a decrease in the pyroceramic strength.

In our experiments, a change in the rate of application of load by more than four orders of magnitude (from 0.04 to 100 mm/min) did not lead to any marked changes in the flexural strength and the spread of strength values is

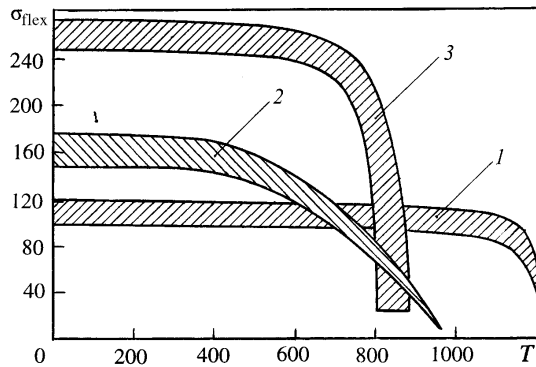


Fig. 3. Flexural strength of glass-ceramic (1) and hardened pyroceramics AS-418 (2) and Pyroceramic 9606 (3) versus the testing temperature. σ_{flex} , MPa; T , °C.

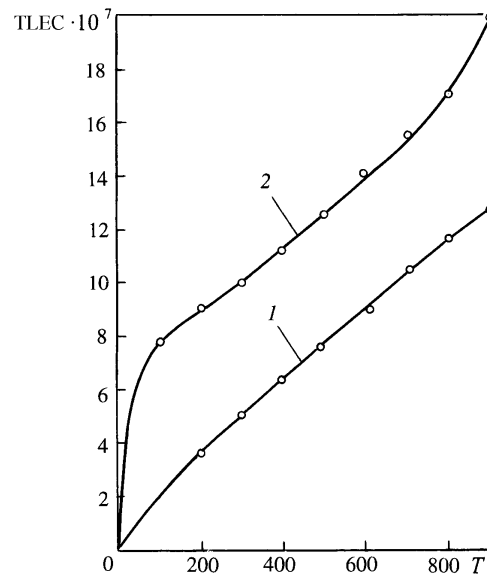


Fig. 4. Temperature dependence of the mean values of the thermal linear expansion coefficient of glass-ceramic (1) and pyroceramic AS-418 (2). TLEC, deg^{-1} ; T , °C.

approximately equal. Therefore, we believe that the statements of the authors of [6] that the water vapors contained in the air influence the decrease in the flexural strength in these experiments are somewhat incorrect because the tests of the specimens are rather short-term. The decrease in the strength of pyroceramic AS-418 in the experiment at a rate of application of load of 0.002 mm/min is likely to be due to the fatigue of the material when under the influence of a sustained load the cracks can grow to sizes capable of causing a fracture of the material under a given load. In all probability, increasing the loading duration, one can also attain a specimen destruction at lower loads.

An important role in the process of mechanical destruction of materials is played by the temperature. However, in the literature only fragmentary information on the results of investigations of the influence of this factor is known. Therefore, the explanations of the behavior of glass-ceramics and ceramics at elevated temperatures have a rough character, although most authors believe that the strength of pyroceramics of different brands decreases rapidly from the initial values at temperatures as low as 300–500°C [1, 7, 8].

Practically all authors concerned with these investigations arrive at the conclusion that the decrease in the strength of glass ceramics of various compositions is caused by the weakening of the atomic bonding forces and the formation of microdefects because of the difference in the thermal linear expansion coefficients between the glassy and crystalline phases forming the materials.

However, the investigations of the temperature dependences of the flexural strength of glass-ceramic OTM-357 revealed a fundamental difference between the results. We revealed no decrease in the strength of the specimens tested in the 20–1175°C temperature range. With increasing test temperatures the OTM-357 glass-ceramic specimens under the action of increasing load exhibit plasticity, although even in testing at 1200°C the residual flexural strength of OTM-357 glass-ceramic is high enough (over 40 MPa) (Fig. 3).

So far there has been no definitive explanation of the fundamental difference in the flexural strength between pyroceramics and glass-ceramics, and in this direction a set of more comprehensive experimental data on various materials, obviously lacking today, is needed.

At the same time, it may be suggested that the stability of the values of the strength properties of OTM-357 glass-ceramics in the region of temperatures up to 1175°C is due to the smaller fraction of the glassy phase in the material, the presence of the ceramic-like structure, and the lower values of the thermal linear expansion coefficient compared to the pyroceramics obtained by the traditional glass technology (Fig. 4).

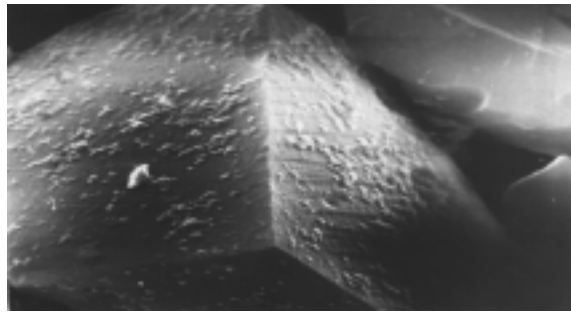


Fig. 5. The structure of glass particles in specimens treated at 700°C.

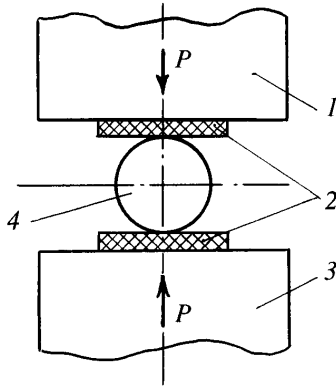


Fig. 6. Scheme of specimen loading in determining the tensile strength: 1) top plate; 2) calico spacer; 3) bottom plate; 4) sample.

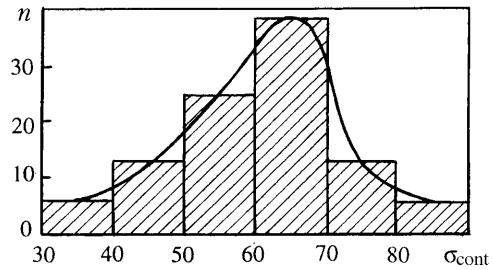


Fig. 7. Histogram of the radial compression contact strength distribution of glass-ceramic specimens (total number of tested specimens — 30. Shaded rectangles show the number of specimens in % of the tested ones with the contact strength values in the given ranges). n , %; σ_{cont} , MPa.

Indeed, glass-ceramic is formed from fine-grained particles of the source glass whose active crystallization begins with the surface, thus decreasing the fraction of the glassy phase at the grain boundaries (Fig. 5). This fact is corroborated by the results of x-ray phase and microstructural analyses.

Proceeding from this, one would expect a decrease in the flexural strength only in the region of the material deformation temperature, which was observed in experiments (Fig. 3).

However, there are also other strength characteristics, for example, tensile strength, compressive strength, and impact strength, whose values are needed in calculating the working capacity of structures.

It is extremely difficult to determine the tensile strength of brittle materials, since at the places where the specimen is gripped for its loading, even in very carefully conducted experiments, concentrations of stresses arise and specimens fail not in the working zone. Therefore, measurement data for this parameter cannot be considered to be reliable.

Because of the methodological difficulties of determining the tensile strength by the direct method, a number of works present data obtained by the relatively simple method of radial compression [9–11]. In [11], the results on the ultimate tensile strength of some glasses and pyroceramic represented by solid solutions of the β -spodumene series are presented. Specimens of various sizes were subjected to axial and radial compression tests. The results of the tests were processed with the use of the failure theory [12] according to which the ultimate tensile strength is related to the measured values of the axial and radial compression strength by the following relation:

$$\sigma_{\text{tens}} = \frac{2\sigma_{\text{comp}}\sigma_{\text{cont}}}{\sqrt{\pi^2\sigma_{\text{comp}}^2 - 48\sigma_{\text{cont}}^2}}, \quad (1)$$

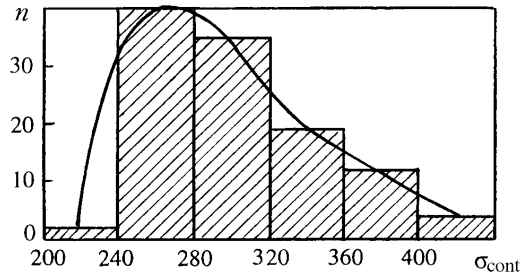


Fig. 8. Histogram of the compressive strength distribution of glass-ceramic specimens (total number of tested specimens — 30. Shaded rectangles show the number of specimens in % of the tested ones with the compressive strength values in the given ranges). n , %; σ_{comp} , MPa.

TABLE 4. Comparative Data on Mechanical Properties of Some Materials

Material	Contact strength, σ_{cont}	Compressive strength, σ_{comp}	Flexural strength, σ_{flex}	Tensile strength, σ_{tens}
Pyroceramic A-1	22	570	50	45
Quartz ceramic	27	150	40	19
Glass ceramic	62	280	100	45

$$\sigma_{\text{cont}} = \frac{P}{dh}. \quad (2)$$

In [1], a considerable influence of the scale factor on the strength properties of test specimens is noted. An increase in their diameter from 10 to 30 mm caused a 48% decrease in the contact strength. At the same time, also a high spread in values of both contact strength and axial compression strength is noted. The value of variation coefficients in these expressions reached 36–40%.

Investigations of glass-ceramic OTM-357 to determine its tensile strength were carried out on specimens 30 mm in diameter and 10 mm in height according to the scheme depicted in Fig. 6. The contact strength and tensile strength values were calculated by equations (1) and (2).

The contact strength of glass-ceramic specimens, as the other strength properties, has a statistical character of the distribution and a fairly high variation coefficient — up to 47% (Fig. 7).

The mean values of the strength properties of glass-ceramic compared to the data of [11] are presented in Table 1. The tensile strength values obtained for glass-ceramic OTM-357 of 45 ± 15 MPa correspond to the conventional ratio $\delta_{\text{flex}}/\delta_{\text{tens}} \cong 1.8\text{--}2.8$ observed in concretes, oxide ceramic, and other materials [13].

Despite its insufficient accuracy, the radial compression method is relatively simple and permits quantitative assessment of the tensile strength, which is very useful in calculating and designing articles.

Due to the fact that glass-ceramic, like other materials, always fails at tension, compressive stresses are of no great importance to it. The measured indices of the compressive strength of glass-ceramic OTM-357 on $10 \times 10 \times 15$ mm specimens are at the level of 280–350 MPa and are characterized by a high variance (30–40%), which is due to the shape and size of the specimens. These compressive strength values of glass-ceramic are close to the compressive strength of pyroceramics of a similar composition obtained by conventional glass technology [14].

The behavior of the material under impact loads is also important in assessing the operating capacity of structures. But impact strength measurements for brittle materials are associated with many uncertainties and measurement data depend on experimental conditions.

For instance, in [15] it is reported that the mean value of the impact strength of unhardened pyroceramic AS-418 is in the range from 5.1 to 9.3 kJ/m². But the testing of specimens of glass-ceramic OTM-357 analogous in composition to pyroceramic AS-418 on $120 \times 25 \times 10$ mm specimens on a pendulum testing machine showed lower values of 2.0–2.5 kJ/m², which, however, are close enough to the impact strength values of high-alumina ceramic — 2.2 kJ/m² [1].

TABLE 5. Elastic Properties of Some Materials at 20°C

Material	Elastic modulus, $E \cdot 10^{-4}$	Poisson coefficient, μ
Pyroceram 9606	12.3	0.245
Pyroceram 9608	8.8	0.25
Pyroceramic AS-370	13.2	0.34
Pyroceramic AS-418	8.5	0.29
Glass-ceramic OTM-357	5.3	0.29
Quartz ceramic with a porosity of 10%	4.5	0.25
High-alumina ceramic	28.0	0.32
Quartz glass	7.4	0.25
Borosilicate glass	6.7	0.26

In all probability, the significant differences in the impact strength values of glass-ceramic OTM-357 from the data of [15] are explained by the different methods of determining this parameter and the size and shape of the specimens.

The working capacity of structures is also largely determined by the elastic properties of the materials from which they are made, since these parameters determine the levels of stresses that can arise under the action of operational factors.

For example, sudden heating and cooling, which is inherent in operating the structures of flying vehicles, cause deformations of the material and the structure. In such cases, it is expedient to use materials with a low elastic modulus. In much the same manner, if a structure from a ceramic or another source material is joined to a metal, and this always takes place in joining the fairing to the flying vehicle airframe, the presence of a low elastic modulus of these materials is desirable because under a particular given deformation the stresses in the structure will be lower.

The elastic moduli of pyroceramics are always higher than the elastic moduli of source glasses. It has been reliably established that the elastic modulus for glasses satisfies the additivity criterion depending on the chemical composition. For them, coefficients permitting one to calculate the elastic modulus by the chemical composition have been derived, which makes it possible to control this quantity by formulating adequate source mixtures. The elastic modulus of materials depends on their porosity with whose increase it significantly decreases [16].

In glass-ceramic OTM-357, the elastic modulus is also determined by the elastic constants of the main crystalline phases and by the residual glassy phase. The data on the elastic properties of various materials given in Table 5 demonstrate clearly enough the relationship between the elastic modulus and its chemical composition and porosity. The considerably lower values of the elastic modulus of glass-ceramic OTM-357 compared to pyroceramics are explained exactly by the presence in it of a small fraction of closed pores.

Thus, the complex investigations of the strength and elastic properties of glass-ceramic of a lithium aluminosilicate composition have made it possible to establish the laws of changes in these parameters depending on different factors, and the given comparative characteristics permit using them as basic values for calculations in designing structures.

NOTATION

n , number of specimens that have shown values of strength properties of glass-ceramic in particular ranges, %; K , variation coefficient, %; σ_{flex} , flexural strength, MPa; T , testing temperature, °C; R_a , surface roughness, μm ; $V_{\text{a.load}}$, velocity of application of load in testing, mm/min; σ_{tens} , ultimate tensile strength, MPa; σ_{com} , ultimate axial compression strength, MPa; δ_{cont} , radial compression contact strength, MPa; P , specimen breaking strength, kg; d , specimen diameter, mm; h , sample height, mm; E , elasticity modulus; μ , Poisson coefficient.

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