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«ХАЛЫҚ» ЖҚ

# Х А Б А Р Л А Р Ы

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## ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ  
АКАДЕМИИ НАУК РЕСПУБЛИКИ  
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## N E W S

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*NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.*

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## **SYNTHESIS OF CERAMIC GRANITE BASED ON DOMESTIC FELDSPAR RAW MATERIALS**

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**Abstract.** Porcelain granite tile is one of the priority building materials in the Republic of Kazakhstan according to the latest programs for industrial and innovative development of country.

The porcelain granite tiles production is high-tech process based on kaolins, white-burning clays, feldspar and quartz sands, specially selected according to their mineral and chemical compositions.

The plant for the production of ceramic granite tiles LLP ZERDE-KERAMIKA and its branch in of Aktobe city are so far the only ones in Kazakhstan. They cover requirements of country in ceramic granite tiles by more than 30%. Currently, a project has been launched in Shymkent, according to which foreign investors are building a porcelain granite tiles plant with capacity of 12 million m<sup>2</sup> to meet the needs of Kazakhstan and Central Asian countries. On the way to solving the problems of increasing production capacity is the problem of raw materials import substitution, among which is given special attention to feldspar mineral raw materials.

The objects of study are feldspar and other types of mining raw materials involved in this technology, developed new raw mixes and synthesized samples of ceramic granite tiles

The mineral-petrographic and technological aspects of selected feldspar raw materials were studied, and experiments series to find the optimal compositions and conditions for ceramic granite tiles synthesis have been studied.

Physicochemical processes and phase transformations have been studied during heat treatment both in individual components and in the developed ceramic granite masses.

A very promising porcelain tile has been produced based on domestic import-substituting feldspathic raw materials.

The research results will serve as the basis for the introduction into production. This development is the beginning of innovative involvement in the ceramic granite production of new genetic types of feldspar raw materials sources. It will expand the range and scale of acute deficiency of investigated component in this rapidly developing business every year.

**Key words:** porcelain granite tiles, minerals, feldspar, synthesis, import substitution

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## **ОТАНДЫҚ ДАЛАЛЫҚ ШПАТ ШИКІЗАТЫ НЕГІЗІНДЕ КЕРАМОГРАНИТТІ СИНТЕЗДЕУ**

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**Аннотация.** Керамогранит Қазақстан Республикасында елдің индустриялық-инновациялық дамуының соңғы бағдарламаларына сәйкес басым құрылыс материалдарының қатарына жатады.

Керамогранит өндірісі – минералды және химиялық құрамы бойынша арнайы зерттеліп таңдалған каолин, әктүсті күйетін саздар, дала шпаты және кварц құмдары негізінде орындалатын жоғары технологиялық процесс.

Керамогранит өндіретін “ЗЕРДЕ-Керамика” ЖШС зауыт және оның Ақтөбе қаласындағы филиалы әзірге Қазақстан Республикасында жалғыз, олар ел қажеттілігінің 30% астамын қанағаттандырады. Қазіргі уақытта шетел инвесторлары Шымкент қаласында Қазақстан мен Орталық Азия елдерінің қажеттіліктерін қамтамасыз ету үшін қуаты 12 млн. м<sup>2</sup> керамогранит шығаратын жобаны іске қосуда. Өндірістік қуаттарды ұлғайту саласындағы жоспарларды іске асыру жолында шикізат материалдарын импорт алмастыру проблемасы тұр, олардың арасында дала шпаты минерал шикізаты ерекше назарда.

Зерттеу объектілері – дала шпаты және осы технологияға қажетті тау-кен шикізаттарының басқа түрлері, жергілікті шикізаттар негізінде зерттеп табылған жаңа құрамдар және синтезделген керамогранит үлгілері.

Іріктеп алынған дала шпатының минералдық-петрографиялық және технологиялық аспектілері зерттелді, сондай-ақ керамогранит синтезінің оңтайлы құрамдары мен шарттарын іздеу бойынша эксперименттер жүзеге асырылды. Жеке компоненттерде, сондай-ақ олардың негізінде зерттеп табылған керамогранит массаларында жоғары температурада өңдеу кезінде орын алатын физика-химиялық процесстер және фазалық өзгерістер зерттелді.

Бірінші рет отандық дала шпаты негізінде болашағы аса зор керамогранит алынды.

Зерттеу нәтижелері өндіріске шикізаттың жаңа түрін енгізуге негіз болады. Бұл жұмыс нәтижесі дала шпаты шикізаты көздерінің жаңа генетикалық түрлерін керамогранит өндірісіне инновациялық тартудың бастамасы. Ол жыл сайын қарқынды дамып келе жатқан осы өндіріске өте тапшы компоненттің номенклатурасы мен ауқымын кеңейтеді.

**Түйін сөздер:** керамогранит, минералды шикізат, дала шпаты, синтез, импорт алмастыру

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## **СИНТЕЗ КЕРАМОГРАНИТА НА ОСНОВЕ ОТЕЧЕСТВЕННОГО ПОЛЕВОШПАТОВОГО СЫРЬЯ**

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**Аннотация.** Керамогранит в Республике Казахстан согласно последним программам индустриально-инновационного развития страны в числе приоритетных строительных материалов. Производство керамогранита – высокотехнологичный процесс на основе каолинов, беложгущихся глин, полевых шпатов и кварцевых песков, специально подобранных по минеральному и химическому составам.

Завод по производству керамогранита ТОО «ЗЕРДЕ-Керамика» и его филиал в г. Актобе – пока единственные на территории Республики Казахстан, и они удовлетворяют более 30% потребности страны. В настоящее время в г.Шымкент запущен проект, по которому иностранные инвесторы строят керамогранитовый завод мощностью 12 млн. м<sup>2</sup> для обеспечения потребностей Казахстана и стран Центральной Азии. На пути решения задач по наращиванию производственных мощностей стоит проблема импортозамещения сырьевых материалов, особое внимание среди которых падает на полевошпатовое минеральное сырье.

Объекты исследования – полевошпатовое и другие вовлеченные в данную технологию виды горно-рудного сырья, разработанные новые сырьевые составы и синтезированные образцы керамогранита. Изучены минералогическо-петрографические и технологические аспекты выбранного полевошпатового сырья, а также проведены серии экспериментов по поиску оптимальных составов и условий синтеза керамогранита. Исследованы физико-химические процессы и фазовые превращения при термообработке как в отдельных компонентах, так и в разработанных керамогранитовых массах.

Впервые на основе отечественного импортозамещающего полевошпатового сырья получен весьма перспективный керамогранит.

Результаты исследований послужат основанием для внедрения в производство нового вида сырья. Данная разработка является началом инновационного вовлечения в керамогранитовое производство новых генетических типов источников полевошпатового сырья. Она расширит номенклатуру и масштабы остродефицитного исследуемого компонента в этом бурно развивающемся с каждым годом деле.

**Ключевые слова:** керамогранит, минеральное сырье, полевой шпат, синтез, импортозамещение.

**Introduction.** The synthesis of ceramic granite tiles is aimed at creating a material that has the quality of natural granite. They are obtained on the basis of kaolin, white-burning clays, feldspars and quartz sands, taking into account their mineral and chemical compositions.

Of all the listed components, feldspars play the most complex and significant role in this technology. In the production of porcelain granite tile, feldspathic raw materials usually mean only K-Na feldspars (a subclass of orthoclase) in this huge group of aluminosilicates, because they are relatively widespread and contain the necessary minimum of flux-forming alkalis.

Feldspars are genetically related to magmatic activity and, as part of various rocks, make up a colossal mass of the earth's crust - about 50% of its weight. In terms of chemical composition, feldspars are aluminosilicates of Na, K, Ca -  $\text{NaAlSi}_3\text{O}_8$ ,  $\text{KAlSi}_3\text{O}_8$ ,  $\text{CaAl}_2\text{Si}_2\text{O}_8$  and have the ability to form isomorphous series (Figure 1) (Betehtin, 2008:736; Boki, 2003:584)

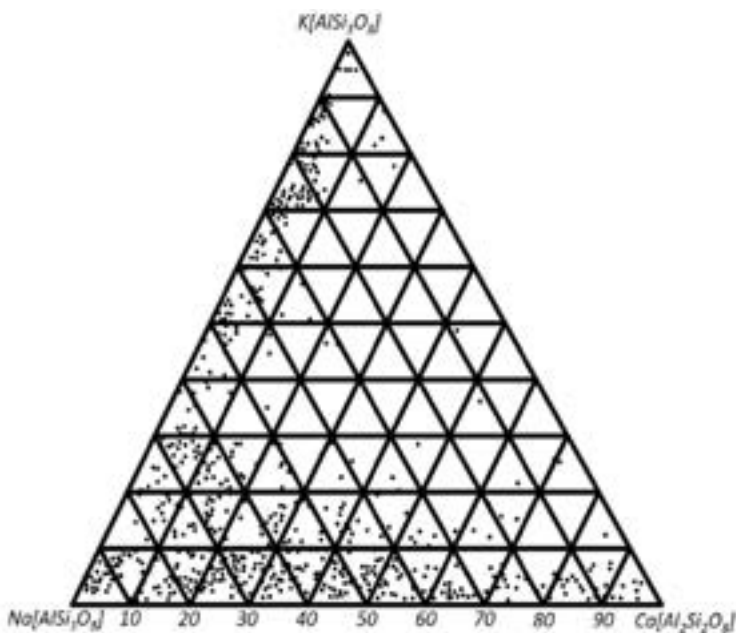


Figure 1 - Diagram of fluctuations in the chemical composition of feldspars

Feldspars are crystal-chemically aluminosilicates, where all 4 free half-oxygen ions at the vertices of Si and Al tetrahedra are bonded to adjacent tetrahedra in such a way that three-dimensional framework structures are formed. From half to a quarter of the tetrahedra, as a rule, are occupied by aluminum ions and the framework as a whole has a negative charge - one minus for each Al tetrahedron. K and Na ions are placed in large voids in the frame, neutralizing the charge, which give these silicates unique flux-forming qualities.

The production of porcelain granite tile in the Republic at ZERDE-Ceramics LLP and its recently launched branch satisfies only about one third of the Republic's needs. An urgent problem in further increasing the volume of porcelain granite tile production is a justified transition to the use of import-substituting feldspathic raw materials.

As the analysis shows, for the production technology of porcelain granite tile in many countries there has been and remains a problem of shortage of high-quality feldspathic raw materials. There has been a noticeable increase in targeted research to clarify the geological and mineralogical content and prospects of local deposits of feldspathic mineral raw materials as possible own sources (Adylov, 2007:3; Adyrbayev, 2017:5; Albertazzi, 2010:6; Baucia, 2010:11; Betehtin, 2008:736; Gacki, 2011:9; Guzman, 2003:496; Kulinich, 2000:372; Lewicka E, 2010:4; Moshnyakov, 2020:4; Tereshchenko, 2000:3; Zubehin, 2014:4).

The mineralogical, petrographic and experimental studies outlined below, aimed at developing the compositions of porcelain granite tile masses exclusively based on domestic feldspathic raw materials, were carried out taking into account the above goal.

During the work, for the first time, feldspars from mineral deposits previously explored as raw materials for household porcelain and glass were tested in the mineral raw material composition of porcelain granite tiles. It is known that potassium feldspars are of greatest interest for the production of ceramics, including porcelain granite tiles. The use of feldspars is based on their ability to transform at relatively low temperatures into a melt, which, when solidified with kaolin and quartz, forms a dense white, slightly translucent mass - the mullite mineral basis of silicate ceramics.

**Materials and basic methods.** The objects of study are feldspar and other types of mining raw materials involved in this technology, developed raw masses and synthesized ceramic granite tiles.

The studies were carried out using physical and chemical methods, including mineralogical and petrographic, chemical, X-ray phase, differential thermal analyses, electron microscopic and traditional laboratory methods for studying mineral raw materials, ceramic granite masses and obtained material samples.

The chemical compositions of the most optimal raw materials, selected according to the results of comprehensive studies, are shown in the table 1.

Table 1. Chemical compositions of raw materials included for the development of ceramic granite masses

Mineral raw materials	Content of oxides, % by mass								loss on ignition
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	
Alekseevsky kaolins	71,02	20,07	-	-	0,48	0,55	1,24	0,46	6,11
Berezovsky clays	51,36	29,64	1,44	1,10	1,46	1,39	1,38	0,90	11,22
Akzhar quartz sands	89,36	4,47	0,46	0,69	0,03	1,43	1,95	0,40	0,10
Aksoran feldspars	70,0	19,3	0,8	0,57	0,49	0,12	0,21	8,55	-

The deposit of Aksoran feldspars in terms of the mineral phases composing it is unique in its own way and is localized in Permian granites, represented by more than ten stock-shaped almost monomineral feldspar bodies up to 100 m long and up to 30 m wide, exposed to the surface. Taking into account the content of alkali oxides, the mineral in its petrographic composition belongs to the albitite feldspathic rock (Figure 2). The reserves of raw materials, according to the forecasts of exploration geologists, will be enough to operate factories for tens of hundreds of years (Kulinich, 2000:372).



Figure 2 - Albitite of the Aksoran deposit and porcelain tiles based on it

The process of preparing ceramic masses included the following main technological steps: grinding of lumpy materials in a jaw crusher, wet grinding of raw materials until the crushed fractions pass through a 60  $\mu\text{m}$  sieve, magnetic cleaning and preparation of a plastic mass.

The developed composition of the ceramic granite mass, obtained by analyzing the curves of flexibility on phase diagrams, by studying the physical-chemical and structural transformations into multicomponent systems during burning, and based on the results of technological experiments, is shown in Table 2.

The quality of the obtained samples of ceramic granite tiles were evaluated by the following characteristic properties: water absorption, frost resistance, heat resistance, wear resistance and bending strength.

Table 2. Raw material composition of the developed ceramic granite mass

Components	Contents of components in mass, %
Kaolins	28,0
Clays	26,0
Quartz sands	8,0
Feld spars	38,0
Total	100

To establish the optimal burning temperatures and holding duration, a burning cycle of the investigated mass of ceramic granite tiles were carried out at temperatures of 1000, 1050, 1100, 1150, 1200, 1250 and 1300  $^{\circ}\text{C}$ .

Below, Figure 3 shows curves of the dependence of the main properties of the studied porcelain granite tiles samples depending on the firing temperature.

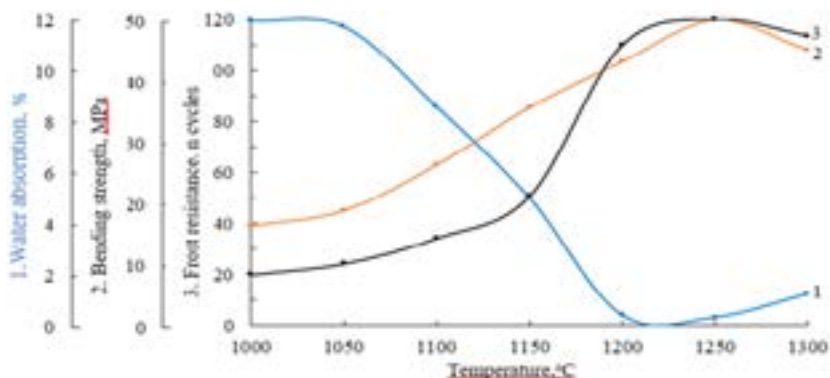


Figure 3 - Curves of changes in water absorption (1), bending strength (2) and frost resistance (3) of porcelain granite tiles samples fired in the range of 1000-1300 °C

In all cases, 5 porcelain granite tiles samples were used to calculate the most representative average physical and mechanical indicators.

One of the main quality indicators of porcelain granite tiles is water absorption. This property is associated with the early destructuring of feldspathic components and the reaction of their interaction with the formation of new crystalline phases, melting of low-melting eutectics with the formation of a glassy phase and polymorphic transformations of the  $\text{Na}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2$  and  $\text{K}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2$  systems, already at temperatures of 600 – 700 °C.

The nature of the curves shows that the water absorption of the mass decreases evenly, starting from 1050 °C and approaches the minimum value at a temperature of 1200 °C. Its zero value is achieved at a temperature of 1200 – 1250 °C, upon completion of the above thermal processes.

It should be emphasized that, unlike traditional ceramics, when molding porcelain tiles, higher pressure is used, reaching 500 kgf/cm<sup>2</sup>. This circumstance has a very positive effect on the water absorption rates of porcelain granite tiles samples.

Water absorption at an increase in firing temperature of 1200 °C for all studied samples is 0.2%, which meets the requirements of the standard.

Frost resistance reaches its highest level at a firing temperature of 1200-1250 °C, naturally correlating in the opposite direction with water absorption indicators. The nature of this indicator depends on the intensity of physical and chemical processes in the porcelain granite tiles mass during firing, when the shard begins to sinter, the mass becomes compacted and a monolith is formed from individual grains. Sintering occurs due to the formation of the liquid phase, reactions in the solid phase and the fusion of newly formed crystalline forms, as well as due to the recrystallization of primary compounds.

As is known, in ceramics, including porcelain granite tiles, the mechanical properties are influenced by the phase composition and microstructure, and there is also a direct connection between frost resistance and porosity or water absorption

of the material (Martín-Márquez, 2010:7; Nori, 2010:6; Sanchez, 2006:8; Tereshchenko, 2000:3; Wyszomirski, 2012:20; Zubehin, 2014:4).

The bending strength of prototypes obtained on the basis of the test mass increases rapidly starting from a firing temperature of 1000 °C, due to the contact interactions of particles in the solid phase. From this temperature, solid-phase sintering processes begin in feldspars with a change in physical and optical properties.

The presence of small mineral impurities in the form of secondary hydroxyl-containing silicates leads to the formation of low-melting eutectics in limited quantities, providing sufficient bonding of mineral particles to impart initial strength to the shard of products. According to chronology, this takes place long before the melting of feldspars - albite  $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$  at a temperature of 1118 °C and orthoclase  $\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$  at 1150 °C

The most important property for porcelain floor tiles in particular is abrasion resistance.

The dynamics of changes in the wear resistance of prototypes of the developed mass showed that wear resistance approaches the minimum value at the firing temperature of the samples in the range of 1200 -1250 °C. It is under these conditions that in the studied masses, according to mineralogical and petrographic studies, mass mullitization and the formation of a uniformly granular structure of the material are observed.

It is not surprising that the heat resistance of tiles based on the developed porcelain granite tiles masses approaches the maximum value also at the firing temperature of the samples in the range of 1200 - 1250 °C.

At the final stage of firing, at temperatures above 1200 °C, the diffusion process of dissolution of the kaolinite residue and quartz begins in the feldspathic melt, which is so necessary for the construction of a crystal lattice of new mullite formations - the ultimate goal of porcelain granite tiles technology.

In these processes, as confirmed by experiments, the viscosity of the initial melt, which begins to appear in the body of the heat-treated mass upon reaching a certain temperature, is of decisive importance. An excessively viscous melt does not ensure complete cementation of particles, while a low-viscosity melt wets crystalline particle well, but later contributes to the appearance of unwanted deformation in the form of curvature of products.

It has been established that the more the melt is saturated with silica and alumina dissolving in it, the higher its viscosity and the higher the required characteristics of the samples. Firing products at the found optimal temperature ensured the necessary qualities of the material based on the developed masses.

This is due to the fact that, when the feldspathic melt reaches the melting temperature, the latter acts as a solvent for quartz and kaolinite residue, a binder of quartz and kaolinite residue that has not reacted with the melt, as well as an active mineralizer that promotes the occurrence of intramolecular transformations of kaolinite, diffusion processes and the growth of the linear dimensions of new formations.

All the main physical and mechanical properties (Table 3) of the developed experimental porcelain granite tiles samples were determined and analyzed; they meet the requirements for porcelain granite tiles materials.

Table 3 - Physical and mechanical characteristics of the developed porcelain granite tiles samples

Name of indicator	Normalized indicator	Values for developed samples	
		1200 °C	1250 °C
Bending strength, MPa, not less	40	42	49
Water absorption, %, no more	0,5	0,2	0,03
Frost resistance, n cycle, not less	100	106	120
Wear resistance (on quartz sand), g/cm <sup>2</sup> , no more	0,18	0,17	0,09
Thermal resistance, n cycle, not less	125	132	153
Surface hardness on the Mohs scale, not less	6	6-6,5	6-6,5

In this case, the feldspathic melt is saturated with diffusing aluminum ions in the melt.

The completeness of these processes depends on the solubility of the crystalline phase in the liquid phase, the amount of the liquid phase and its properties - the ability to wet solid particles, spread over their surface and penetrate into the capillary gaps between solid particles.

When the temperature reaches 1300 °C, it is clearly visible that the physical and mechanical properties deteriorate, this shows that the optimal burning temperature for this composition is 1200 - 1250 °C.

**Results.** X-ray phase analysis data (Figure 4) of the synthesized porcelain granite tiles indicate mullite ( $d/n = 5.414; 3.408; 2.880; 2.697; 2.545; 2.206;$ ) and quartz ( $d/n = 4,262; 3,345; 2,455; 2,285; 2,129; 1,981; 1,818$ ).

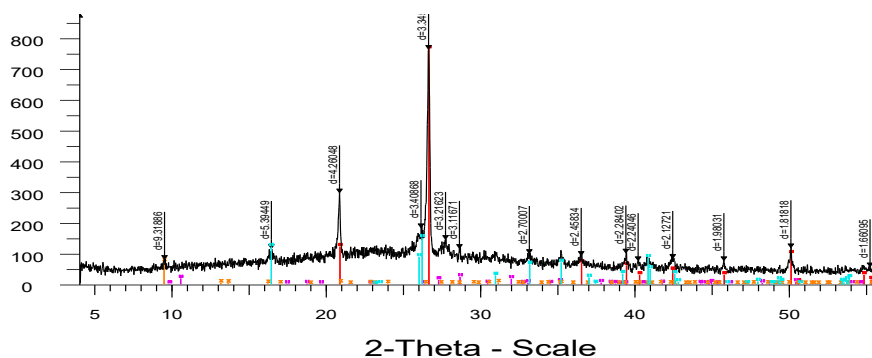


Figure 4 - X-ray image of porcelain granite tiles obtained on the basis of Aksoran feldspars

We studied the microstructures of samples of the developed porcelain granite tiles masses. The samples have (Figure 5) a rather dense structure. The general appearance of the microstructure is represented by clearly distinguishable feldspar

relics consisting of glass and mullite, grains of residual quartz surrounded by rims of high-silica glass and closed pores of various sizes and shapes. In micrographs of the cleavage one can see a structurally glassy phase overgrown with small submicroscopic, evenly scattered mullite crystals. Mullite areas belonging to the original feldspar particles and almost undecomposed masses of clayey substances are clearly visible. In feldspar relics, large mullite needles grow from the surface as the composition changes due to alkali diffusion.

As an analysis of the entire porcelain granite tiles production process shows, the narrowest and most problematic issue in this matter is the selection of feldspathic raw materials. It is the feldspar in the porcelain granite tiles charge that most determines the formation of the petrographic composition and structure of the material, which provides the desired properties of the artificial stone.

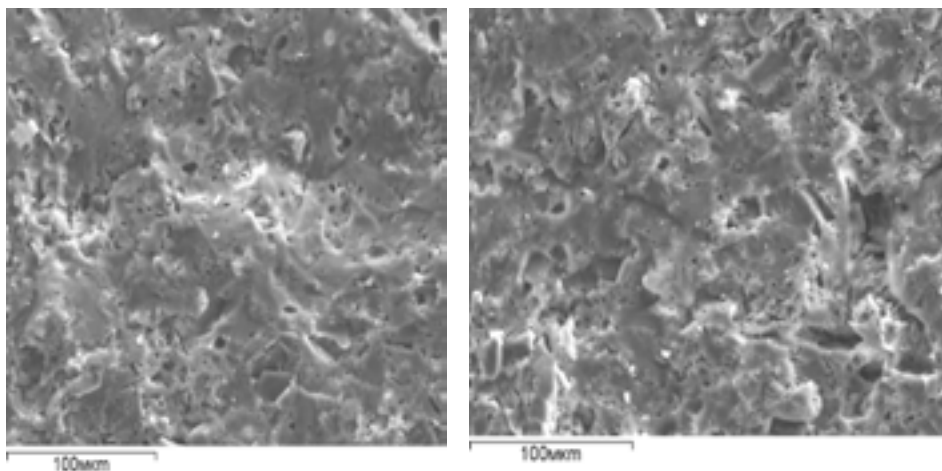


Figure 5 - Microstructure of porcelain granite tiles obtained from Aksoran feldspars

**Discussion.** Feldspars are practically irreplaceable in the production of porcelain granite tiles - a synthetic analogue of the most famous natural material in terms of crystallinity, mineral composition, structure and properties.

It has been established that the Aksoran feldspars, in terms of their material composition, belong to an almost monomineral albitite rock in granites and they are highly effective in melt and phase formation during sintering processes in porcelain granite tiles masses.

According to previously expressed opinions, the best raw materials for porcelain production are potassium-sodium spars with a predominance of orthoclase ( $\text{KAlSi}_3\text{O}_8$ ), because they, along with a low melting point, are characterized by a wide softening range (Guzman, 2003:496). At the same time, in experimental mineralogy there is an established fact that the melting of orthoclase at a temperature of 1250-1320 °C occurs better with the addition of albite.



The optimal parameters of the technology of porcelain tiles with new flux were determined and more than 30 tons of Aksoran feldspars were successfully tested in the conditions of the LLP «ZERDE-KERAMIKA» plant as an import-substituting component.

From the results of the research, the prospects for using these albitites to cover the growing demand for this scarce and functionally important component in the production of porcelain granite tiles are obvious.

**Conclusion.** Based on domestic feldspathic raw material, which is also new to the technology of porcelain tiles, an innovative composition of the charge has been developed with the following ratio of components, %: feldspars - 38, clays - 26, kaolins - 28, quartz sands - 8.

As an analysis of the entire porcelain granite tiles production process shows, the narrowest and most problematic issue in this matter is the selection of feldspathic raw materials. It is the feldspar in the porcelain granite tiles charge that most determines the formation of the petrographic composition and structure of the material, which provides the desired properties of the artificial stone.

All properties of the developed porcelain granite tiles meet the requirements of the current standard. The high indicators of their technical properties are explained by the achievement of optimal mullite and quartz mineral phases and their ratios, as well as the favorable structure of the material.

As a result of the research, porcelain granite tiles, a very promising import-substituting material, was synthesized for the first time on the basis of domestic feldspathic raw materials. Domestic albitite, proposed for introduction into porcelain granite tiles production, occupies the most dense area on the diagram of fluctuations in the chemical compositions of feldspars and, as a new raw material, has high potential for the future.

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