Визначено основні властивості керамічної цегли різного призначення. Досліджено мікроструктуру та фазовий склад рядової, лицьової та клінкерної цегли. Встановлено взаємозв'язок мікроструктури і фазового складу досліджених матеріалів з водопоглинанням, механічною міцністю при стисканні та морозостійкістю. Результати досліджень дали можливість визначити особливості формування керамічного черепка та пояснити фізико-хімічні процеси при спіканні.

Встановлено, що рядова цегла містить переважно термічно змінену глинисту речовину з невисокою кількістю склофази. У зв'язку з зазначеним, через неповне рідкофазне спікання, рядова цегла має високі значення водопоглинання (10–14 %) при низькій міцності (7,5–12,5 МПа).

Лицьова цегла має більш розвинену склофазу, яка міцно з'єднує кристалічну фазу. Остання представлена такими мінералами, як β-кварц, мікроклін, альбіт, муліт та ін. Головним завданням виробництва лицьової цегли є забезпечити оптимальну дисперсність вихідних сировинних матеріалів і досягти рівномірного розподілу мінералів по всьому об'єму виробу.

Клінкерна цегла має більш складний механізм спікання, оскільки при використанні вихідної грубодисперсної маси необхідно отримати щільну однорідну структуру виробів. Головними особливостями керамічної маси є введення до її складу опіснюючих добавок, які забезпечать стійкість виробів до деформації під час випалу, і плавнів, які мають забезпечити інтенсивне рідкофазне спікання. При випалі таких виробів потрібно правильно вибрати температурно-часовий режим, який буде відповідати інтервалу спікання основного глинистого матеріалу. Це необхідно для того, щоб, з одного боку, отримати міцну щільну структуру виробу з водопоглинанням 4–5 %, а з іншого – уникнути таких видів браку, як деформація, розтріскування, «перевипал», спучування і т. д.

Результати досліджень можуть бути застосовані у виробничих умовах на підприємствах галузі з метою контролю якості продукції і усунення можливих причин браку, пов'язаного з порушенням технологічного режиму виробництва

Ключові слова: керамічна цегла, мікроструктура керамічного матеріалу, спікання глини, випал будівельної кераміки

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ANALYSIS OF THE INTERACTION BETWEEN PROPERTIES AND MICROSTRUCTURE OF CONSTRUCTION CERAMICS

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1. Introduction

At present, ceramic materials are especially significant among many building materials. Owing to their properties, construction ceramics make it possible to create a healthy climate at home, safe from the point of view of fire resistance and have long service life [1, 2]. However, production of construction ceramics is rather resource-and energy-intensive because it is a multistage technology that implies a high temperature annealing [3].

Therefore, one of the most important tasks in this industry is to ensure that the products are of the highest quality. That would be facilitated by controlling the quality of products based on studying the microstructure and the phase composition of sintered material. Such an analysis will make it possible to operatively intervene in the technological process and to ensure a full-fledged progress of physical-chemical processes to form a ceramic material with the predefined properties.

The relevance of work in this field necessitates conducting a study into the microstructure in order to explain the physical-chemical processes of ceramic material sintering and to allow influencing them through the rational selection of technological factors.

2. Literature review and problem statement

Ordinary, facing and clinker ceramic bricks are different both in terms of operating properties and physical appearance and in the microstructure and phase composition of the sherd. The latter directly define almost all the properties of the products. Thus, the ordinary ceramic brick has rather large water absorption indicators and low strength at compression and frost resistance, while the clinker brick, by contrast, is characterized by high durability and frost resistance and a low value for water absorption [4].

The specified properties are due to the peculiarities of the microstructure and phase composition of ceramic material that gradually forms at each stage of production: when preparing the bulk, when molding a semi-finished product, while drying and annealing a ceramic sherd. Therefore, studying and being able to regulate the processes of forming the microstructure of ceramic bricks would enable obtaining high-quality, competitive products.

Thus, authors of [5] conducted a study into the microstructure when determining an optimal composition of the ceramic brick based on fusible clays and industrial waste at different temperatures of annealing. It was established that during annealing the ceramic bricks based on polymineral fusible clay, ash-slag material and saline waste from the recycling of aluminum-containing slags and broken products, mullite forms at 1,000–1,100 °C. The above study reports investigation into a change in the phase composition of a material at an increase in the temperature of annealing, but does not explain the relationship between microstructure and properties of the products.

Authors of [6] studied the microstructure of ceramic brick in order to select the optimum mode of annealing. Thus, it is recommended to apply fast annealing at the temperatures that are 50–100 °C higher than those at slow annealing. That leads to forming the optimum porosity and density of the ceramic brick. However, the work reports a study into a particular type of brick with the emphasis on investigating technological factors of production, rather than the microstructure. Consequently, a given work provides no answers regarding the relationship between the microstructure of ceramic brick and water absorption, mechanical strength and frost resistance.

The phase composition and microstructure of construction ceramics that contain water-soluble salts of magnesium sulfate were addressed by authors of [7], using an electron microscope. They reveal the mechanisms of influence of soluble salts on the destruction of a ceramic material. However, this work implied using the electron microscope, which is difficult to maintain, and not exploited in practical work, for example under industrial conditions.

In paper [8], an electron-microscope method is employed to investigate the phase composition of ceramic bricks made from beidelite clay, ash-slags and phosphorus slag at different annealing temperatures. It is shown that at a temperature of 1,000 °C there forms a significant amount of the glass phase while increasing the temperature to 1,050 °C contributes to the further increase in the content of the glass phase in brick. However, yet again, the study was carried out using an electron microscope, which is why it is almost impossible to apply results in practical work.

In paper [9], shredded brick wastes (grog) was utilized for the production of bricks. Grog was introduced to the mixtures of fusible clays for obtaining traditional red ceramic bricks. Authors estimated the effect of adding grog on the molding capability of the bulk during extrusion, as well as on the properties and microstructure of bricks, annealed at 700 °C. The results show the effectiveness of adding grog in quantities not exceeding 5 % by weight. Increasing the amount of grog reduces mechanical strength of both dry and annealed ceramic products. This is related to the increased porosity at the stage of annealing as a result of grog behavior. The drawback of this work is the «narrow» range of the considered ceramic materials.

Study [10] focused on manufacturing ceramic bricks from clay materials from the region Beruas (Malaysia). The annealing temperatures were changed from 800 to 1,250 °C with aging at a maximum temperature for one hour. Research into the influence of annealing temperature on phase changes, microstructure, strength at compression, water absorption, and porosity of bricks, established the optimum temperature of annealing, which was 1,200 °C. When temperature varied from 1,000 to 1,250 °C, the porosity of products changed in the range from 39.33 % to 5.87 %. Thus, the authors proved that the properties of bricks can be controlled by changing the temperature of annealing; they provided recommendations on the product's optimal technological parameters, however, not enough attention was paid to the microstructure of products.

Paper [11] studied the effect of ratio of the components on the structure and basic physical-mechanical properties of ceramic bricks modified by the technogenic mineral systems. The authors developed optimal compositions for ceramic charges that contain, %: 67 % – overburden, 28 – waste heaps, and 5 – red mud. The charges are used to obtain ceramic bricks at a temperature of 850 °C, grade M 150, with an average density of 1,650–1,730 kg/m^3, which makes it possible to reduce energy cost of annealing. It was established that the temperature of the beginning and end of sintering of the modified mixture reduces by 150–200 °C compared with that of the non-modified. Thus, the work reveals the relationship between the physical-mechanical properties of ceramic bricks, technological factors of production and the microstructure of an annealed material. However, the results reported relate to solving a particular problem, which is the use of industrial waste, while broader studies into construction materials with a different composition are lacking.

Paper [12] reports results of studying the formation of mineralogical composition of ceramic bricks made at VAT «Revdinskiy Brick Factory» (Russia) using an electron microscope. Such a study made it possible to determine the mineralogical composition. However, scientific research is limited to the products of a single manufacturer, which complicates the application of results to other building materials. In addition, the results were obtained by means of an electron microscope, which makes it impossible to employ a given method to control quality under industrial conditions.

Thus, an analysis of the scientific literature revealed [5–12] that the microstructure and phase composition of construction ceramics are examined in separate studies. We could not find any systematic data on a comprehensive comparative analysis of the microstructure of ceramic bricks for different purposes. In addition, studying the microstructure most often employs an electron-microscopic method of analysis, which has practical, rather than scientific value. At the same time, microstructure analysis using an optical microscope is a more affordable, inexpensive and revealing method. Such a method can be implemented at any enterprise in order to control product quality and reduce defects.

3. The aim and objectives of the study

The aim of this work is to study the microstructure and phase composition of ordinary, facing, and clinker bricks, and to establish their relation to such performance properties as water absorption, mechanical strength at compression, and frost resistance.

To accomplish the aim, the following tasks have been set:

– to determine the water absorption, mechanical strength at compression and frost resistance of ordinary, facie, clinker ceramic bricks;

– to explore the microstructure and phase composition of the specified types of ceramics using an easily-maintained optical microscope;

– to compare quality and properties of the annealed samples of the examined ceramics to the microstructure and phase composition of sherd;

– to give an explanation to the possible types of defects in construction ceramic products, and to substantiate ways to improve the quality of products.

4. Materials and methods of research

For our research, we selected the following samples of ceramic bricks (Table 1), which differed in their purpose. Thus, a standard brick is used in construction when making internal parts of bearing and enclosing structures. Its application is not recommended for external masonry as the brick quickly breaks down in contact with the environment. Facing products are intended to line the buildings, erect fences and any external structures. The clinker brick is used when constricting facades of buildings, for making pillars for fencing, in the construction of basement spaces and columns, walls, columns, fireplaces indoor, for landscape works to lay paths, playgrounds and sidewalks, etc. That is possible because it combines high physical-mechanical properties and decorative qualities.

Table 1

Examined ceramic materials

Sample No.	Brick type	Manufacturer
1	Ordinary	TOV Kirovogradskij Zavod Stroitelnyh Materialov № 1, Ukraine
2	Ordinary	TOV Kerambrok, Ukraine
3	Facing	TOV SP Zymohir'yivs'kyy Tsehel'nyy Zavod
4	Facing, yellow	TOV SBK, Ukraine
5	Facing, yellow-pink	TOV SBK, Ukraine
6	Facing, red-brown	Bilotserkivsky tseghelny zavod TOV «Bilotserkivski budivelny materialy», Ukraine
7	Clinker, red	TOV Kerameya, Ukraine
8	Clinker, brown	Wienerberger, Poland
9	Clinker, yellow	TOV Evroton, Ukraine

Basic properties of the examined samples were measured according to standard procedures [13]. The water absorption of samples was determined by saturating them with water under vacuum and determining the volume of the absorbed water. Mechanical strength limit was determined based on the indicator of maximum compression strain that a sample resists prior to destruction. Frost resistance was determined based on the number of cycles of freezing and thawing the samples at temperatures from −15 to +15 °C.

The microstructure of examined samples was examined by using the optical stereoscopic microscope MBS-10 (VO Rubin, Russia), at a magnification from 4.8 to 64 times, using reflected light [13].

To determine the phase composition of samples, we applied an X-ray phase analysis [14] using the diffractometer DRON-3 (NVP Burevestnik, Russia).

5. Results of studying the properties of ceramic bricks

Results of determining the basic properties of the examined samples of ceramic bricks are shown in Fig. 1.

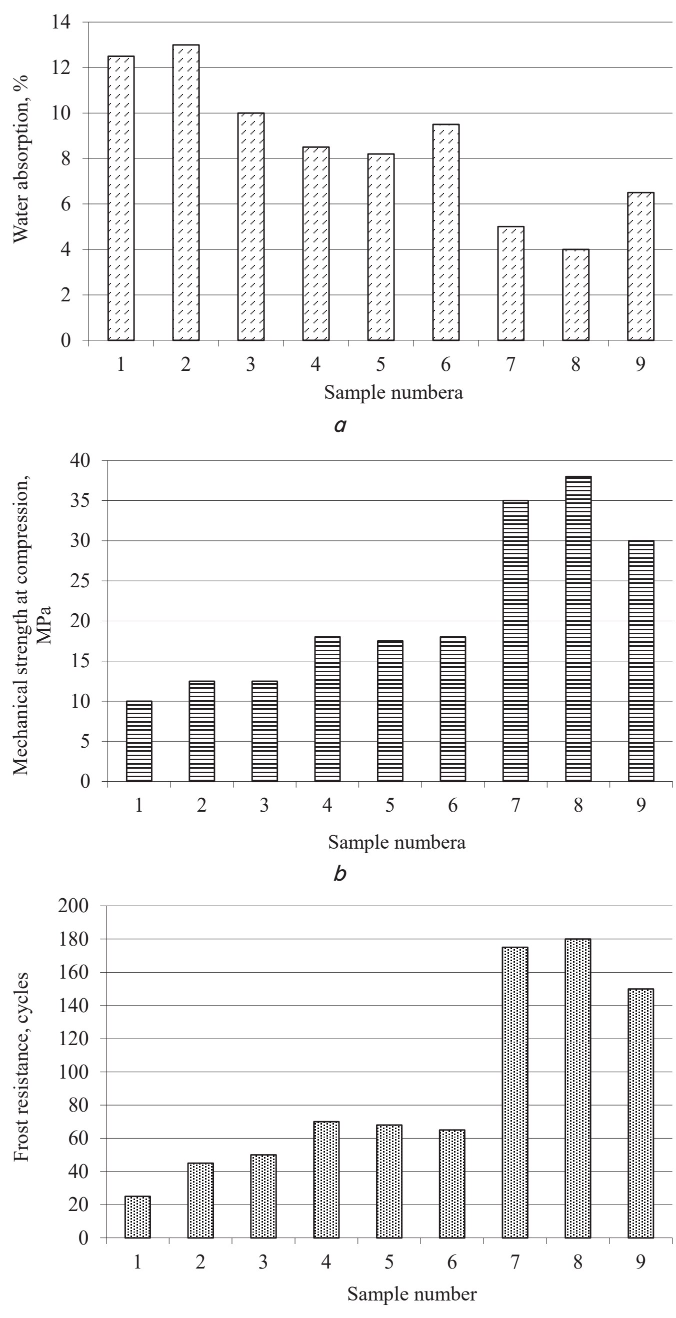


Fig. 1. Basic properties of the examined samples of ceramic bricks: *a* — water absorption, *b* — mechanical strength at compression, *c* — frost resistance

The highest value of water absorption (12.5–13 %) and the lowest strength at compression (10–12.5 MPa) and frost resistance (35–45 cycles) are characteristic of the ordinary ceramic bricks. These properties are intercorrelated: at a high porosity, water from the environment penetrates the pores of the product, thereby changing the volume during freezing and thawing, causing the larger microstresses, and eventually destroys the product.

The lowest value of water absorption (4–6.5 %) and the highest indicators for mechanical strength at compression (30–38 MPa) and frost resistance (175–150 cycles) were established for the clinker bricks.

A comparative analysis of the basic properties of the examined ceramic bricks is given in Table 1.

Table 1

Basic properties of ceramic bricks for various purposes

Property name	Ceramic brick		
	ordinary	facing	clinker
Water absorption, %	12.5–13	8.2–9.6	4–6.5
Mechanical strength at compression, MPa	10–12.5	12.5–18	30–38
Frost resistance, cycles	35–45	47–70	150–180

Images in Fig. 2 show that the microstructure of the examined construction ceramics differs in: density of the sintered sherd, the content and size of rocky impurities, the size and distribution of pores, the degree of vitrification of the main thermally altered clay mass. These components of the microstructure intrinsically determine the basic properties of a ceramic material [16, 17].

The microstructure of samples of ordinary brick No. 1 and No. 2 differs by the presence of a large number of pores while the main thermally altered clay mass contains a small amount of the glass-like phase. The microstructure of facing brick samples No. 3–6 contains considerably fewer pores, the well-developed glass- and crystalline phase, which, in comparison with the ordinary bricks, are represented by more diverse minerals (β-quartz, microcline, albite, mullite, etc.). The main features of the clinker ceramic mass are the introduction to its composition of emaciated additives that would provide the resistance of products to deformation during annealing, and marshes, which must ensure the intense liquid-phase sintering at annealing. It is due to the presence of flooded areas that the microstructure of clinker ceramics is represented by an elevated content of the glass phase, which firmly binds the crystalline phase thereby ensuring a high density of products.

6. Discussion of results of studying the relationship between microstructure and properties of ceramic bricks

Sample No. 1 of the ordinary ceramic brick is quite loose, contains a small amount of the glass-like phase, which typically must bind well the thermally modified clay and stone-like particles [18]. There is a heterogeneity of structure in the outer layer and inside the product, which could arise at the incomplete agitation of the bulk, insufficient temperature or duration of annealing.

Since for ordinary bricks, according to requirements of standards, a low strength at compression would suffice (7.5, 10, or 12.5 MPa), it is not always that a manufacturer pays attention to the uniformity of the structure. However, such heterogeneities may lead to the emergence of shrinkage processes and deformation of different magnitude, which, in turn, predetermines the occurrence of micro-cracks (Fig. 3) and even the destruction of products.

Total porosity of the sherd is high, visually estimated to be 20–22 %, which, combined with the insufficient adhesion of particles at sintering, explains the low strength of ordinary products.

Rocky inclusions the size greater than 0.1 mm are almost absent (Fig. 4, *a*). Evenly distributed in the main thermally modified clay mass are the fine-dispersed quartz grains. The starting clay mass is represented by a mixture of two types of clays – fusible, with red coloration, and refractory, of light color, which are rather homogeneously mixed and complement each other's properties. Thus, the plastic refractory clay is introduced to the mass composition in order to improve the molding capacity of emaciated fusible clay material [9]. Iron-containing minerals occur in the form of individual grains and do not exert a significant influence on the properties of products.

The outer surface of ordinary products, including sample No. 1, has a low density (Fig. 4, *b*), there are microcracks with a length from 0.5 to 3 mm, as well

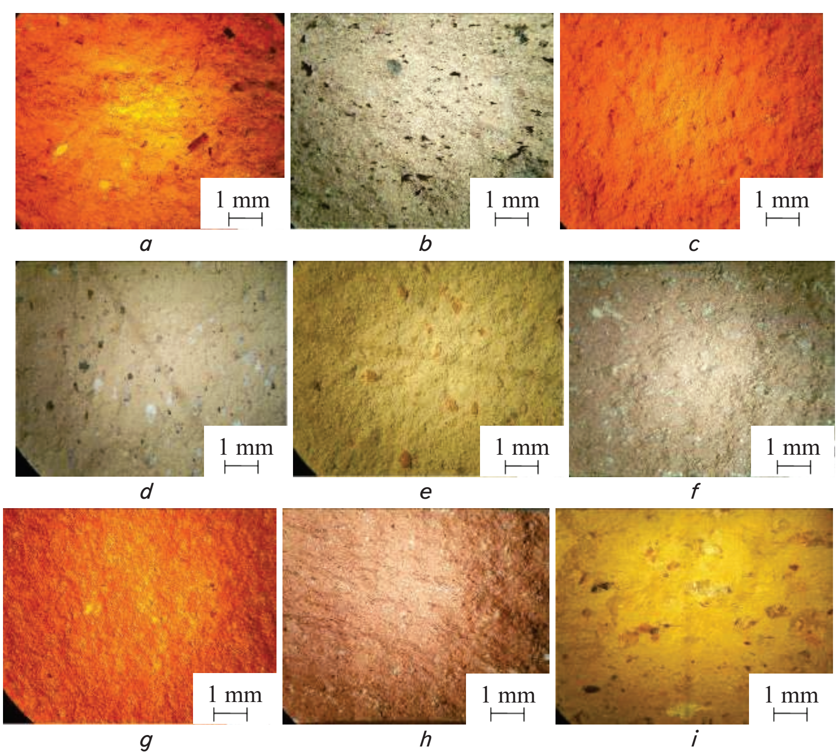


Fig. 2. Microstructure of the examined ceramic samples, reflected light: *a* – № 1; *b* – No. 2; *c* – No. 3; *d* – No. 4; *e* – No. 5; *f* – No. 6; *g* – No. 7; *h* – No. 8; *i* – No. 9

as pores the size of 0.5–0.8 mm. This explains the low frost resistance of ordinary construction products, which requires additional treatment – applying a protective cover the type of engobe or laying the plaster when constructing buildings.

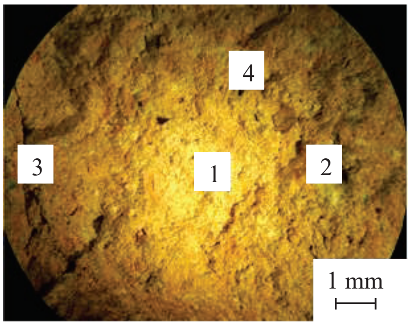


Fig. 3. Microstructure of sample No. 1, reflected light: 1 – main thermally altered clay mass; 2 – pore; 3 – microcrack; 4 – inclusion of an iron-containing mineral

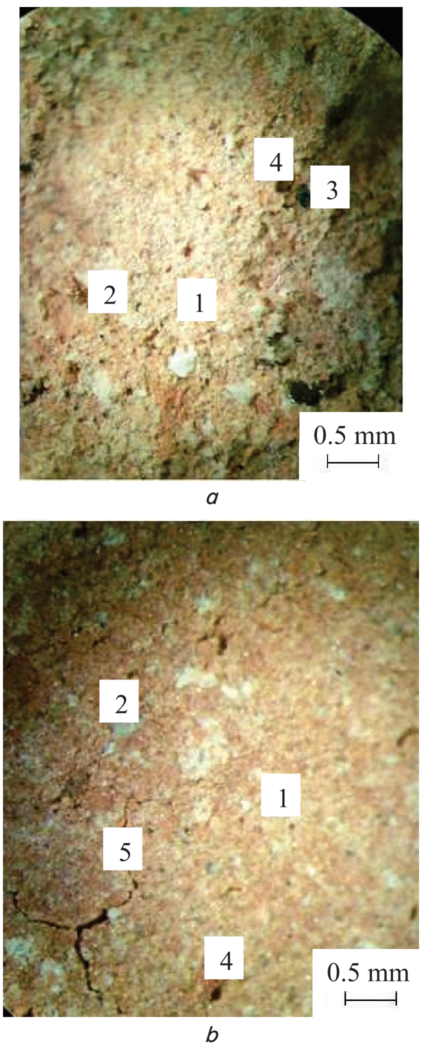


Fig. 4. Microstructure of sample No. 1, reflected light: *a* – inside the product; *b* – at the surface; 1 – main thermally altered clay mass; 2 – grain of quartz; 3 – inclusion of iron-containing mineral; 4 – pore; 5 – microcrack

Thus, the characteristics of sample No. 1 are the insufficient content of the glass-like phase, which predetermines the formation of loose structure and low durability of the product (10 MPa). The sample is also characterized by high porosity, which occurred most likely at the low vacuuming of the plastic mass, as well as the difference in the structure between the inner and outer layer of sherd.

Sample of ceramic brick No. 2 has even greater porosity (Fig. 5), and the size and shape of the pores are quite diverse. Small pores the size of up to 0.1–0.3 mm mainly have a rounded shape, pores of a larger size are elongated; in this case, most of them are perpendicular to the force of mass compression during molding. At some sections of the breaking surface the size of pores reaches a length of up to 1.5–2.5 mm.

However, if we compare the degree of sintering of ceramic mass of a given sample to the sample of ordinary brick No. 1, sample No. 2 has a more baked state, which manifests itself in the larger amount of the glass-like phase, which binds stone-like particles. This explains the higher indicators of the sample's strength (12.5 MPa) compared to those for sample No. 1 at the high values of water absorption (13 %). The presence of the glass phase is observed well at a large magnification of the sample's surface (Fig. 6), when the entire breaking surface has a distinctive glass shine.

Similar to sample No. 1, the clay mass is represented by the thermally modified clay materials of two types – fusible clay with red coloration, and refractory clay of light color.

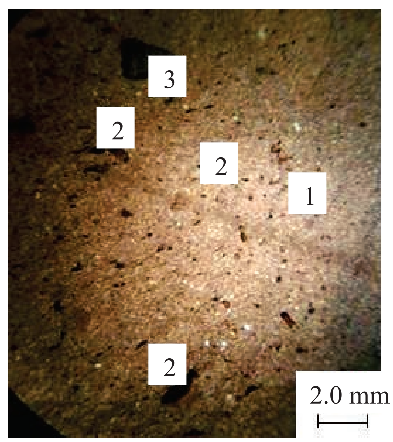


Fig. 5. Microstructure of sample No. 2, reflected light: 1 – main thermally altered clay mass, 2 – pore; 3 – impurities

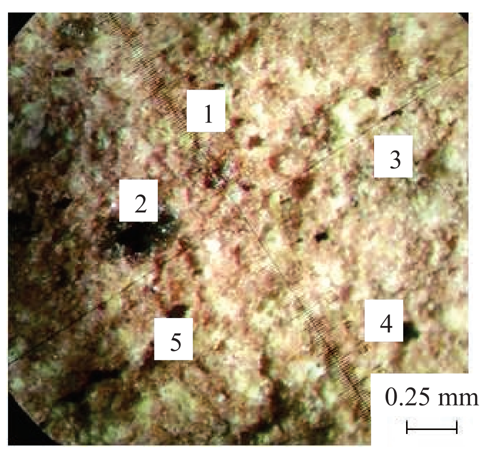
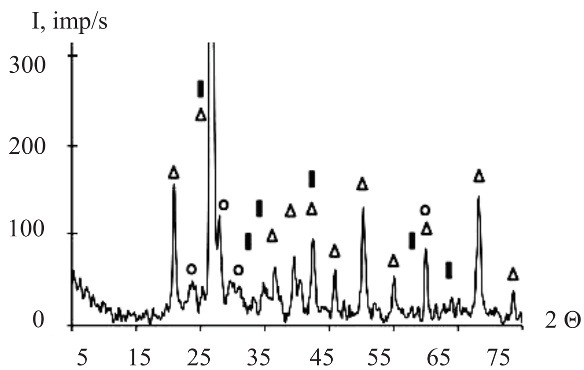


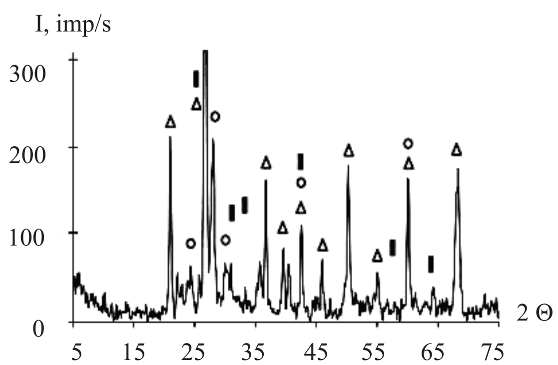
Fig. 6. Microstructure of sample No. 2, reflected light: 1 – main thermally altered clay mass; 2 – melted grain of sintering additive; 3 – grain of quartz; 4 – pore; 5 – iron-containing mineral

Fine-dispersed quartz grains are also present and evenly distributed throughout the mass, but there are also stone-like grains the size of up to 0.5 mm. In the mass, we also trace the contours of the melted particles of a substance, which, most likely, was introduced as a sintering additive (Fig. 6). Visually, the amount of these grains can be estimated to be approximately 5–7 %, they almost have no clear-cut contours because they are excessively melted to the main thermally modified clay glass-mass, which generally leads to the formation of a solid structure of the sherd. There are also melted grains of iron-containing impurities. Thus, for the sample of ordinary brick No. 2, we can conclude that, despite the much greater porosity, strength of the sintered sherd is higher than that in sample No. 1, due to the formation of the more vitrified thermally altered clay mass. It turns out that the above has a greater impact on the formation of a solid structure, rather than the presence of pores in the material.

X-ray phase analysis of samples No. 1 and No. 2 revealed (Fig. 7) the presence of the crystalline phase, mainly in the form of β-quartz (intensive diffracted maxima d=4.25; 3.34; 2.45; 2.28; 1.98; 1.81 Å, and others). Also present are the inclusions of microcline (d=3.75; 3.21; 2.88; 1.98; 1.81 Å) and primary mullite (d=3.34; 2.68; 2.50; 1.58; 1.45 Å). The amount of the latter is extremely low – within 5–7 % as the diffraction maxima, characteristic of mullite, have low intensity.



a



b

Fig. 7. Radiographs of samples of ordinary brick: *a* — sample No. 1; *b* — sample No. 2; Δ — β-quartz; o — microcline; I — mullite

Radiographs of both samples are similar – both qualitatively and quantitatively, the only difference being that in sample No. 2 there are other crystalline phase-impurities (radiograph has a greater density of diffraction maxima). That can be associated with a more diverse mineralogical composition of the starting raw materials. The amount of such impurities ranges within 5 %, so they do not exert any significant influence on the properties of ordinary bricks, which is why there is no need to identify them.

The microstructure of sample of facing brick No. 3 has a more varied phase composition (Fig. 8). This can be caused by either the original properties of clay raw materials or by the intended creation of a multi-component compositional mix by the manufacturer [16]. Thus, we observed in the samples many rocky impurities of quartz, iron-containing minerals, residues of the fine-dispersed carbonate decomposition products, grains of artificial emaciater and a sintering additive (Fig. 9).

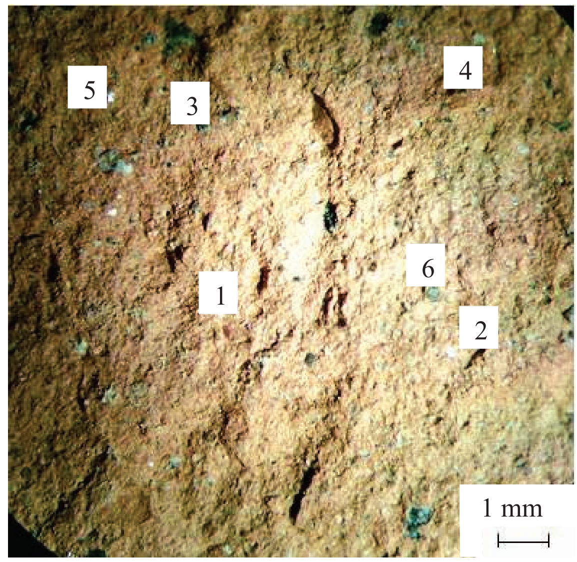


Fig. 8. Microstructure of sample No. 3, reflected light: 1 — main thermally altered clay mass; 2 — pore; 3 — grain of iron-containing mineral; 4 — finely dispersed carbonate residue; 5 — scale of hydromica; 6 — grain of quartz

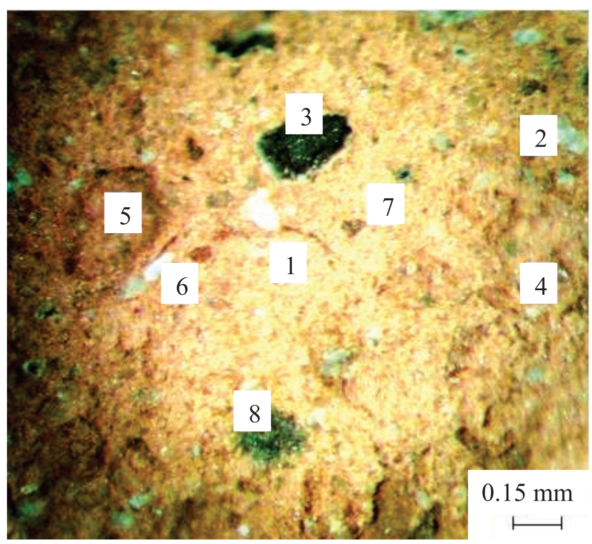


Fig. 9. Microstructure of sample No. 3, reflected light: 1 — main thermally altered clay glass-mass; 2 — grain (aggregate) of quartz; 3 — residue from the decomposition of carbonate; 4 — scale of mica; 5 — pore; 6 — iron-containing mineral; 7 — grain of artificial emaciater; 8 — grain of sintering additive

The total porosity of sample No. 3 is significantly less compared with samples No. 1 and 2. There are small pore the size of 0.2–0.3 mm of round shape, evenly distributed throughout the entire volume. The sample is well sintered, the basic clay mass is sufficiently vitrified, the strength of particles adhesion is high.

The presence of such a large quantity of large rocky grains contributes to the formation of products' frame, which at drying and annealing leads to lower shrinkage processes. However, sintering of such materials requires strict compliance with the technological regime.

Sample No. 4 of facing brick is also a multicomponent material by its phase composition (Fig. 10). However, according to the degree of thermal change in the mass, to the sintering of particles that are different in nature to the main mass of clay, such a polyphasicity is, over all, achieved not through the natural features of clay raw materials, but the targeted formation of such a structure [4].

Thus, the structure contains clearly differentiated grains of quartz with a rather large size (to 0.25–0.35 mm) and grains of feldspar minerals the size of 0.3–0.45 mm. These raw materials are introduced to the composition of the mass in order to emaciate it and shape the frame of products at the stage of molding (to reduce shrinkage and deformation).

At the stage of annealing, feldspar minerals typically take a role of marsh [18]. However, the samples show that the grains of minerals are only slightly melted as a result of insufficiently high temperature during ceramics annealing. One can predict the comprehensive effect of grains of feldspar minerals in this case: in addition to a minor sintering action, they create a frame for the products similar to quartz. However, the excessive amount of quartz in the sherd structure can lead to the emergence of microstrains as a result of the modification transformations [19]. Thus, the amount of quartz should be limited.

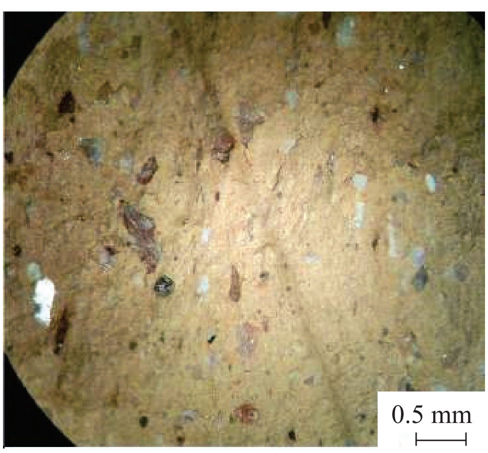


Fig. 10. Microstructure of sample No. 4, reflected light: 1 – main thermally altered clay glass-mass; 2 – grain of quartz; 3 – grain of feldspar mineral; 4 – scale of mica; 5 – iron-containing mineral, pore; 6 – grain of artificial emaciater; 7 – pore

The degree of sintering (vitrification) of the main clay mass is quite high, rocky particles are well bound by the glass-phase. One should note an almost absence of large (exceeding 0.1 mm) pores in the sherd structure, which is achieved by high-quality vacuuming of the mass and the absence of burning impurities in the raw materials.

Sample No. 5 of facing brick is identical to sample No. 4 in its phase composition. It is polyphase (Fig. 11), well vitrified, and contains almost no pores larger than 0.1 mm. It should be noted that the formation of such microporosity, characteristic of samples No. 4 and 5, is especially important for facing bricks as water does not penetrate pores the size less than 10 μm; frost resistance of such products is higher (68–70 cycles) than that of ordinary bricks.

The structure of a given product differs from sample No. 4 by that the mass was added with pieces of broken products whose particles size reach 0.5–0.8 mm. Fig. 11 shows that such segments sinter poorly with the main mass and thus there may be voids, and even micro cracks due to a shrinkage difference between the already sintered emaciater (pieces of broken products) and the composition of the clay-containing mass that is sintered.

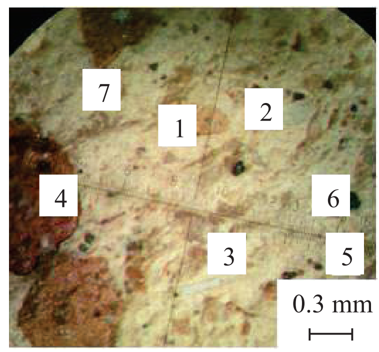


Fig. 11. Microstructure of sample No. 5, reflected light: 1 – main thermally altered clay glass-mass; 2 – grain of quartz; 3 – grain of feldspar mineral; 4 – pieces of broken products, 5 – iron-containing mineral, 6 – grain of artificial emaciater; 7 – pore

In addition, the mass also contains an artificial additive of black color in the amount up to 10 %, whose particles size do not exceed 0.1 mm. Such an additive was probably introduced as an intermediate phase between the fairly large particles of quartz, pieces of broken products, spars, and fine-dispersed clay materials, in order to create dense packing [20].

Sample No. 6 has a dense homogeneous structure (Fig. 12) whose base is the thermally modified glass-mass with particles mainly of grains of quartz.

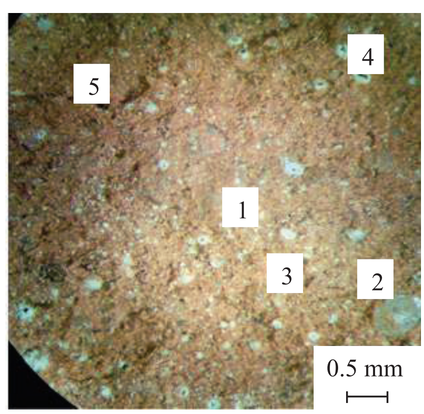


Fig. 12. Microstructure of sample No. 6, reflected light: 1 – main thermally altered clay glass-mass; 2 – grain of quartz; 3 – iron-containing mineral; 4 – finely dispersed residue from the decomposition of carbonates; 5 – pore

Vitrification of the mass is expressed poorly. Quartz grains are evenly distributed throughout the entire volume, basic size is 0.1–0.2 mm, separate grains reach 0.45 mm. In addition, the main mass contains fine-dispersed residues (0.05–0.15 mm) from products of decomposition of carbonate compounds. Such compounds typically do not substantially alter the properties of the ceramic sherd in construction ceramics, but contribute to the activation of sintering [21]. Radiographs of samples of facing bricks are shown in Fig. 13.

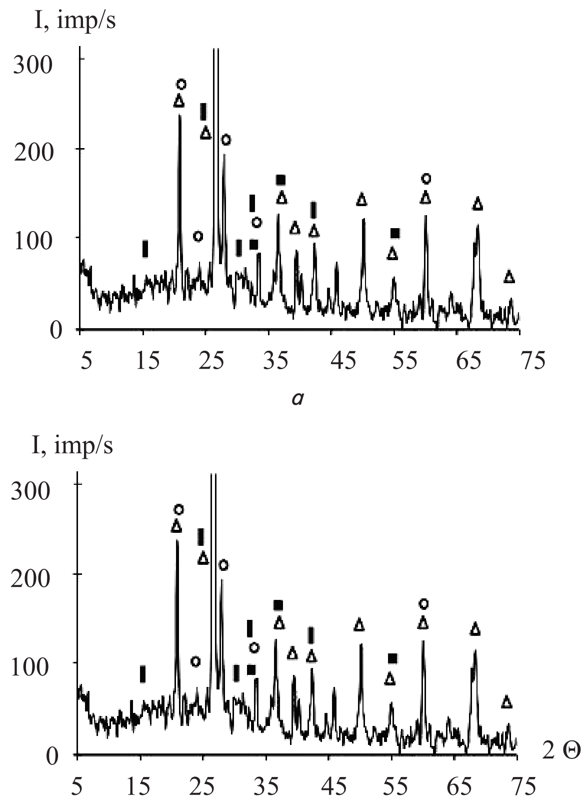


Fig. 13. Radiographs of samples of facing brick: *a* — sample No. 4; *b* — sample No. 6; Δ — β-quartz; o — microcline; I — mullite; ◊ — biotite; ■ — pyrite

Compared with the samples of ordinary bricks (Fig. 8), these radiographs demonstrate a more diverse phase composition. That can be explained by the fact that requirements to ceramic masses in the production of facing bricks are stricter. Therefore, to compile the charge, compositions from several clays and correcting admixtures are applied.

The structure of ceramic sherd No. 7 (Fig. 14) is similar to that of No. 6, however, it differs by the larger vitrification of the main thermally altered clay mass and the greater content pf components. This is due to that sherd No. 7 relates to the clinker ceramics, which is why during formation of the structure in the course of annealing it is required to achieve high strength and low water absorption of a material (4–6 %) [18]. The source of the glass phase in a given sherd is fusible clay, as well as the feldspar minerals and grains of artificial marsh. Such a set of marshes is necessary to ensure the gradual formation of the glass-like phase over a wide temperature range. In order to avoid deformation of the products during sintering, it is required to introduce an artificial emaciater [4], which is well identified in the structure.

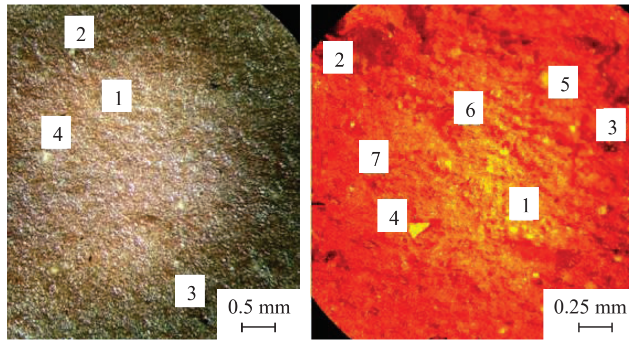


Fig. 14. Microstructure of sample No. 7, reflected light: *a* —magnification ×32 times; *b* — magnification ×64 times; 1 — main thermally altered clay glass-mass; 2 — grain of quartz; 3 — iron-containing mineral; 4 — grain of feldspar mineral; 5 — grain of artificial emaciater; 6 — grain of artificial marsh; 7 — pore

The structure of the clinker product No. 8 (Fig. 15) is interesting, which is very different from the outside and from the inside. Outside, it is the homogeneous glass-like thermally modified clay mass, with the vitrification very strongly expressed and is observed even by the naked eye. At the surface, there is a relief that plays a positive role, for example at when glazing or engraving such a surface. Open pores are completely missing, which explains the high frost resistance of clinker products (170–180 cycles of freezing and thawing).

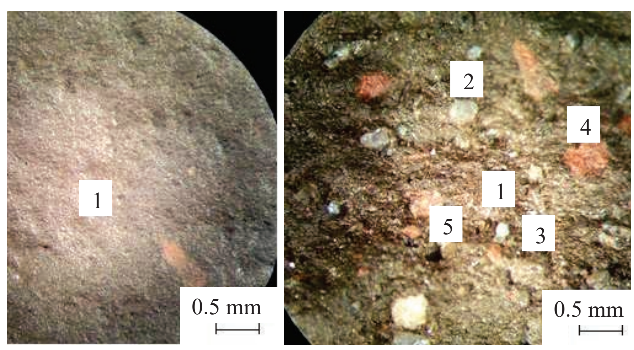


Fig. 15. Microstructure of sample No. 8, reflected light: *a* — external surface; *b* — inside the product; 1 — main thermally altered clay glass-mass; 2 — grain of quartz; 3 — grain of marsh; 4 — grain of artificial emaciater; 5 — pore

Strong vitrification of the main thermally altered clay mass will definitely lead to the deformation of products, which is why they introduce to the mass weight the emaciated additives. It is precisely in order to prevent deformation, the shard was introduced with a large amount of emaciating components, with a fairly large size. The size of quartz grains is 0.3–0.4 mm, and the size of grains of the artificial emaciater (pieces of broken products) is from 0.3 to 0.6 mm. Feld-spar minerals are also of large size (0.35–0.4 mm) and produce both emaciating and sintering effect. The grains are melted along the surface, which is why they are firmly bound to the main clay glass-mass. In addition, feldspar, along with the grains of quartz and artificial emaciater, due to its large size, creates a «frame» of the products. The pores are almost absent in the internal structure of the sherd.

The microstructure of clinker sherd No. 9 (Fig. 16) differs from the previous two by strong heterogeneity. The main clay glass-mass is well sintered, vitrified, but to a lesser extent than in samples No. 7 and 8, because it probably contains fusible clay with a less content of iron oxide (the lighter color of sherd). The structure contains large (up to 0.7–0.9 mm) grains of the natural emaciater – quartz, even in the form of aggregates (most likely, quartz was introduced to the mass not in the form of sand but rather in the form of quartz waste).

Such aggregates are dangerous in terms of modification transformations during heat treatment [19], since they may cause the cracking of products at cooling after annealing and even during operation. It also contains many grains of iron-containing minerals, including pyrite and biotite.

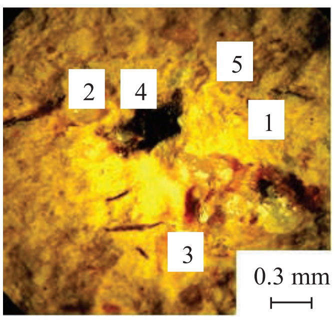


Fig. 16. Microstructure of sample No. 9, reflected light: 1 – main thermally altered clay glass-mass; 2 – grain (aggregate) of quartz; 3 – grain (aggregate) of pegmatite; 4 – iron-containing mineral; 5 – pore

Results of X-ray phase analysis of samples of clinker bricks are shown in Fig. 17.

Radiographs of samples No. 7 and No. 9 demonstrate a large amount of the crystalline phase with a multicomponent composition. The main crystalline phases are β-quartz, feldspar minerals – albite and microcline, there are also impurities of biotite and pyrite; mullite is identified.

Radiograph of sample No. 8 differs by the presence of the elevated amount of the glass-like phase, which manifests itself in lower intensity of the diffraction maxima. The amount of mullite phase in samples No. 7 and 8 is much smaller than that in samples of facing bricks (Fig. 13), which may possibly relate to the annealing of products at higher temperatures when there occurs the recrystallization of mullite [22].

Thus, the main benefit of our study is the establishment of relationship between the microstructure and properties of ceramic bricks. During research, we used a simple and affordable method of analysis by using an optical microscope with reflected light. Analysis requires practically no prior preparation of samples, and the microscope is easy to maintain. In contrast to results found in literary sources [5–12], which tackled a particular type of product, our research reports a comparative analysis of building ceramics for different purposes. A drawback might be a limited number of samples of ceramic bricks. In the future, it is possible to extend the range of examined products.

The research results could be applied under industrial conditions at enterprises in order to control product quality and eliminate possible causes of defects associated with violation of the production technological regime.

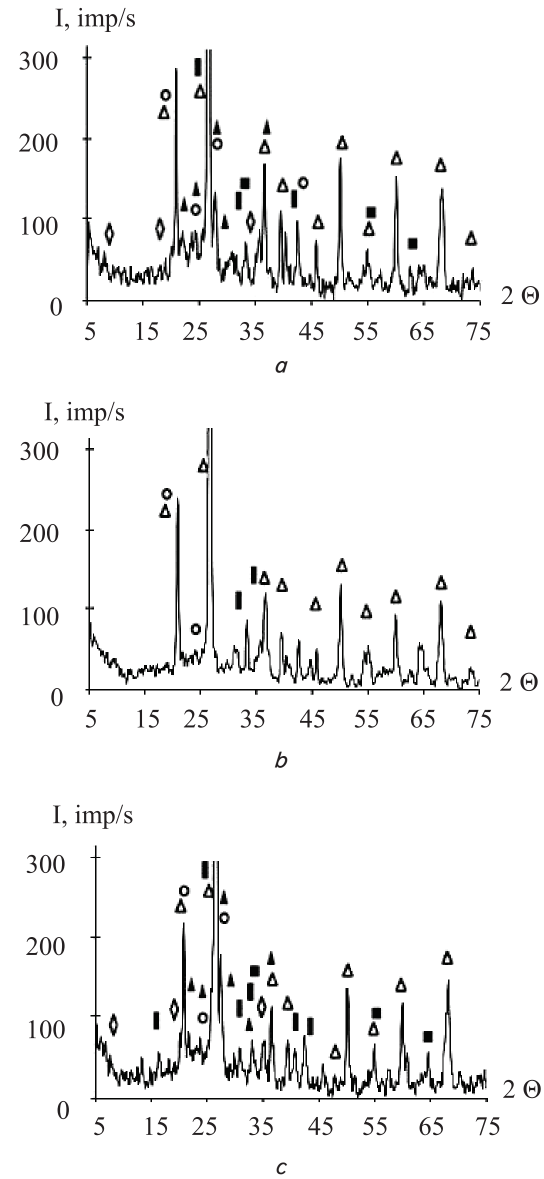


Fig. 17. Radiographs of samples of clinker bricks: *a* – sample No. 7; *b* – sample No. 8; *c* – sample No. 9; Δ – β-quartz; o – microcline; I – mullite; ▲ – albite; ◊ – biotite; ■ – pyrite

7. Conclusions

1. We have defined the basic properties of ordinary, facing, and clinker ceramic bricks. It was established that the greatest values of water absorption (10–14 %), low strength at compression (7.5–12.5 MPa), and frost resistance (25–42 cycles of freezing and thawing) are demonstrated by ordinary bricks. Clinker ceramics have the lowest water absorption (4–5 %) at high indicators of strength (larger than 30 MPa) and frost resistance (155–180 cycles). The specified differences in properties are due to various factors: the characteristics of raw materials, charge composition, grinding grade, molding, and, mainly, to the annealing of products.

2. We have investigated the microstructure and phase composition of different types of construction ceramics, which are formed under specific industrial conditions under the influence of the comprehensive action from all factors

in the technological process. It is shown that studying the microstructure of ceramics using an optical microscope in reflected light makes it possible to operatively control quality of products. It is also possible to specify negative technological factors that lead to cracking, deformation, and other kinds of defects in construction products.

3. We have established the relationship between the microstructure and phase composition of ceramic bricks and its basic properties. Thus, the ordinary brick mainly contains the thermally modified clay substance, which in most cases is not sufficiently vitrified. Therefore, due to the incomplete rare-phase sintering, ordinary brick has high values of water absorption at low strength. The facing and clinker bricks have a better developed glass-phase, which firmly binds the crystalline phase. Crystalline phase is represented by the greater diversity in comparison with ordinary bricks – by β-quartz, feldspar minerals (albite and microcline), there are also impurities of biotite and pyrite; mullite is also identified. The degree of vitrification of ceramic sherd directly defines the water absorption of products: the largest content of the glass phase was found in the composition of clinker products that have the lowest values of water absorption. Special attention needs to be given to the amount and dispersion of grains of quartz, which when heated and cooling are capable of modification transformations with a change in the volume, thereby causing the destruction of products.

Thus, our study has shown the relationship between the microstructure of a ceramic sherd in construction ceramics and its properties; therefore, controlling it could enable the timely and operative regulation of products quality, in order to bring down the quantity of defects under industrial conditions.

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