DOI: 10.32703/2617-9059-2023-42-5

UDC 666.972.16

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Use of modified basalt fiber in transport construction

Basalt fiber is a promising material that can be used to create a new class of building materials. It has a number of advantages, including high strength, low weight and resistance to chemicals. One of the disadvantages of basalt fiber is its low alkali resistance. In an alkaline environment, the fiber is destroyed, which limits its use in building materials exposed to alkalis. The paper investigates the effect of heat treatment on the alkali resistance of basalt fiber. It was found that heat treatment at a temperature of 500 ^{0}C increases the alkali resistance of the fiber by 80%. This is due to the compaction of the fiber structure and the formation of iron-oxygen tetrahedra on its surface, which are resistant to alkalis. The authors of the article substantiate the feasibility of using heat-treated basalt fiber in transport construction. This material can be used to manufacture new types of building materials that are resistant to alkalis.

Keywords: *basalt fiber, transport construction, heat treatment, fiber structure, alkali resistance of fibers, X-ray diffraction patterns, spectra.*

Introduction. Among the characteristics of fiber-reinforced concrete, its flexural and compressive strengths are usually distinguished. The value of this indicator in fiber concrete exceeds the value of conventional concrete by 10...15%. Only fiber concrete or fiber and rod or wire reinforcement are used in structures. Domestic and foreign experience shows that fiber concrete is a versatile material and is increasingly used in the field of transport construction.

Analysis of recent research and problem statement. The length of the road network with cement concrete pavements in countries such as Germany is 31%, the United States – 35%, and Belgium – 41%. The average service life of cement concrete pavements is more than 20 years. Even in those countries where asphalt pavements have economic advantages over cement concrete pavements, in order to improve and develop a competing technology, government subsidies support the construction of cement concrete pavements at the level of 10...20 % of the total volume of road construction with capital types of pavements [1,2]. In the 70s, the Soviet Union began massive construction of cement concrete pavements using machines with sliding formwork on caterpillar tracks. However, the practice of operating such pavements in the Soviet Union revealed a number of significant drawbacks. Failures in temperature joints, peeling of the concrete surface layer, chipping of slab edges and corners, and the formation of potholes appeared. Premature damage to pavements was usually caused by violations of construction technology, a shortage of high-quality cements, the use of low grades of concrete, insufficient air entrainment in concrete, and excessive exposure to saline solutions at the early stage of concrete hardening [3,4].

Unlike the Soviet Union, in many countries of the world, the construction of cement concrete pavements continued in parallel with the construction of asphalt pavements. Stable transport and operational characteristics and high durability have proven their superiority over pavements built with organic binders.

Cement concrete pavements have a high distribution capacity, low abrasion wear, and a high coefficient of adhesion that is not highly dependent on the moisture content of the pavement. The strength and deformation characteristics of cement concrete practically do not change with changes in temperature, humidity and loading rate. Vehicles driving on roads with such pavements consume about 5...10% less fuel than asphalt concrete roads. In addition, there are large raw material resources for the production of cement concrete pavements [5,6].

Modern technologies for the construction of cement concrete pavements involve full automation of the main processes for placing and compacting concrete mixtures, treating the concrete surface, arranging thermal joints and caring for concrete.

In recent years, there has been a trend toward the creation of road concrete with increased strength and durability. This is achieved by modifying the structure of concrete with chemical additives: plasticizing, air-entraining and gas-forming [7,6]. A new generation of high-quality concrete (High performance concrete) has been developed that has increased frost resistance, early strength (at least 35 MPa after 24 hours) and other high construction and technical properties. Such concretes are also used in our country, including at transport construction sites.

In industrialized countries (USA, Canada, Australia, Great Britain, Germany, Sweden, Norway, Spain and many others), the construction of road pavements made of rigid cement concrete mixtures compacted by rolling is expanding [8,9].

Rolled concrete means rigid cement concrete transported by heavy-duty dump trucks or concrete mixers to the construction site and compacted with rollers of various types. Such concrete can be used in the construction of dams and dam retaining walls. In Europe and North America, concrete is used in transport construction for pavement layers for roads intended for heavy vehicles (container ships, heavy military vehicles, forestry machinery); container terminals, parking lots access, rural, forest roads with high traffic loads; secondary roads and streets, local roads and highways, road accesses to airports, airfield runways; as a reinforcement layer for the reconstruction of old road surfaces [10].

The analysis of the technical and operational characteristics of all types of fibers currently used in road construction suggests that it is most advisable to use basalt fiber as a reinforcing element in cement concrete [11,12,13].

Given the shortage of funds, there is a need to develop a program for the construction of highways using fiber concrete in the structural layers of the pavement that is acceptable from a financial and technical point of view.

Modern conditions of road construction dictate the widespread use of industrial waste - slag, burnt shale from coal mine dumps, ash, as well as virtually inexhaustible resources - chalk deposits and igneous rocks (basalts), which are currently of little use in transport construction. Given the conditions and international experience, it is advisable to accelerate developments in the field of fiber concrete technology and calculation, to use fiber concrete structures more widely in the design of transport construction facilities, and to use materials that are optimal in terms of cost and volume of deposits in Ukraine as fiber.

Basalt fibers are one of the most effective modern materials, characterized by high physical and mechanical properties and can be used in a wide range of temperatures.

The production of fibrous concrete in modern construction based on basalt fibers is promising because they work in aggressive environments (resistance to acids and alkalis), have good operational, physical and mechanical characteristics. All these properties of basalt fibers make it important to create high-performance building materials and products for various areas of the construction industry. [14]

It has been established [15] that the fiber in solution during cement hydration is characterized by insufficient alkali resistance.

The purpose and objectives of the study. As noted earlier [15], prolonged storage of fiber in solution contributes to its dissolution. In this regard, it is relevant to develop methods for modifying the surface layer of basalt fiber that will provide high corrosion resistance.

The efficiency of using basalt fiber in cement concrete can be increased by heat treatment of the fiber.

Materials and methods of the study. To study the effect of heat treatment on fiber properties, the fiber was subjected to heating in the temperature range from 300 to 700 ^{0}C with a step of 100 ^{0}C. The isothermal exposure was 30 minutes. Cooling took place at room temperature in an air environment (Table 1).

Table 1: **Fiber modified by heat treatment gradient**

Manufacturer	300^{0}C	400^{0}C	500^{0}C	600^{0}C	700^{0}C
Armbud					
	Normal color	Normal color	Lightened color	Changes in color – more red	Changes in color – more red

The temperature treatment time was chosen empirically and amounted to 30 minutes.

In the course of the research, a significant change in fiber color was noted depending on the processing temperature (Table 1). The probable explanation for this fact is the oxidation of iron in the fiber structure, which resulted in the fiber acquiring a red hue.

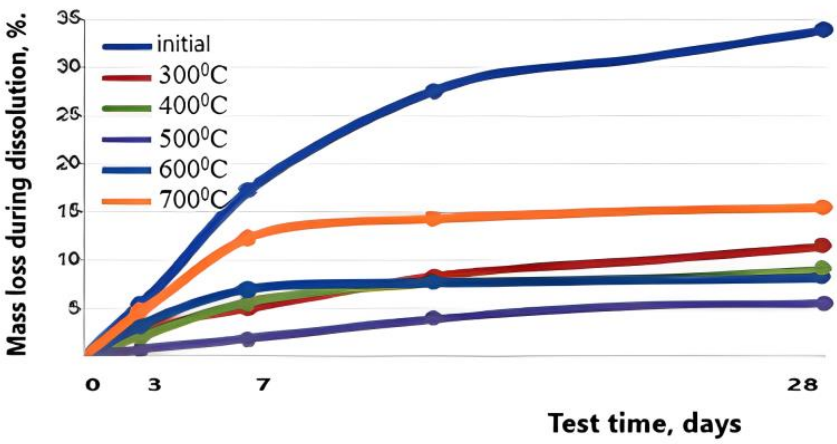

Fig. 1. **Fiber weight loss during dissolution as a function of test time**

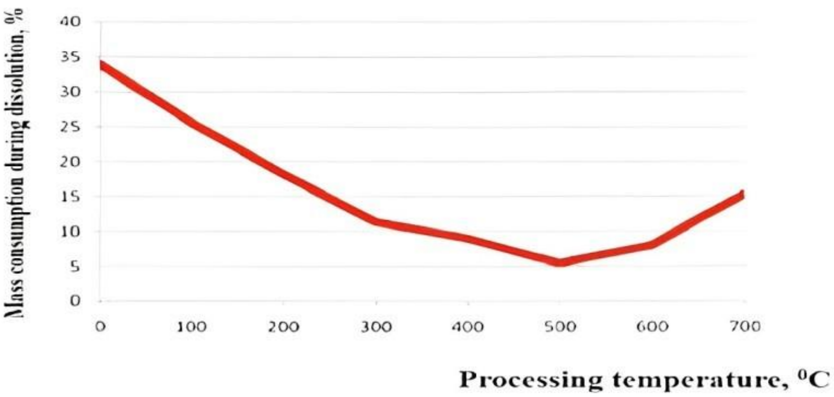

Fig. 2. **Mass loss during dissolution depending on temperature treatment after aging in an alkaline environment for 28 days**

As a result, it was found that an increase in temperature from 300 to 500 0C increases the alkali resistance of the fiber. The mass loss during aging in an alkaline solution after 28 days is about 5 %, while the original fiber loses more than 30 % of the fiber mass during this time. Further increase in the temperature of isothermal aging is not effective due to the decrease in alkali resistance of fibers and economic inexpediency.

When analyzing the microstructure of both the initial and heat-treated reinforcing component, there are no special changes (Fig. 3). This applies to both the size (diameter) (Fig. 3 a, d) and the surface of the fibers (Fig. 3 b, d). The diameter of the individual fibers varies in a wide range – from 2 to 30 microns. The surface of the fiber is quite smooth, but in some parts it is covered with globular formations, presumably carbonation products, up to 1 μm in size.

However, if the surface of the original fiber is highly coordinated (Fig. 4 (a, b), a-c), then after heat treatment it is covered with globular formations (Fig. 4 (b), d-f). The samples of the original anisotropic material have "eaten out" areas, longitudinal "furrows" in the direction of fiber elongation, which may indicate their internal structure (Fig. 4 (a, b), b, c). In this case, the destruction occurs along the weakest part - the interface, presumably, of individual fiber strands from aluminosilicate chains. If we consider the area of confirmed corrosion, it has a clear boundary (Fig. 4 (a), b) and a leaching depth of up to 400 nm (Fig. 4 (b), c). However, the actual amount of damage is greater due to corrosion of the entire surface layer of disordered aluminosilicate glass. There is a globular substance on the fibers themselves, which can be attributed to both X-ray amorphous calcium carbonate and alkali reaction products with the aluminosilicate material of the fiber itself.

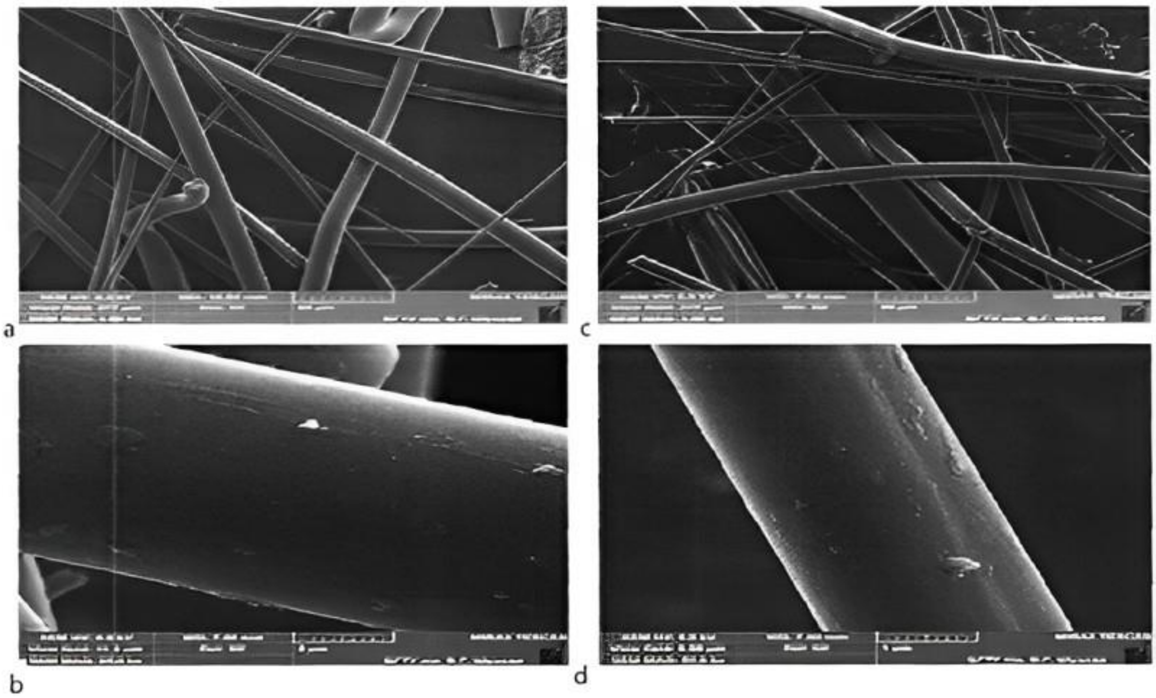

Fig. 3. **Microstructure of basalt fiber:**
a, b – initial; c, d – after heat treatment at 500 ^{0}C

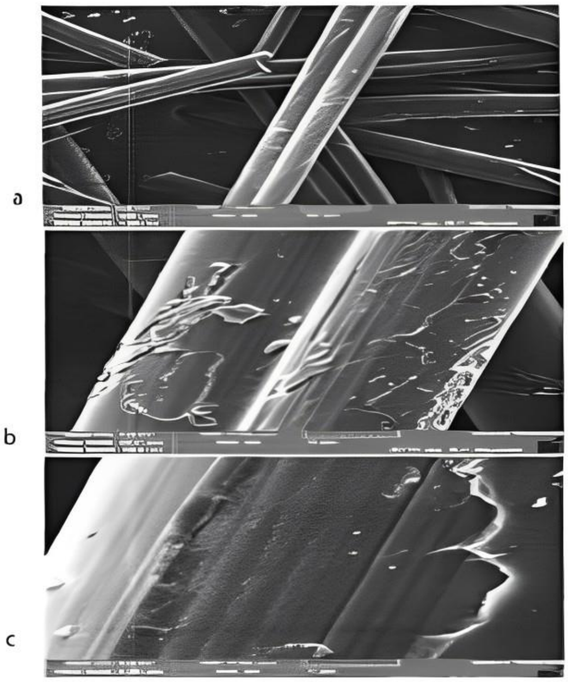

Fig. 4 (a). **Effect of alkaline environment of the model system on the microstructure of basalt fiber: a…c – initial; d…e – after heat treatment at 500 °C**

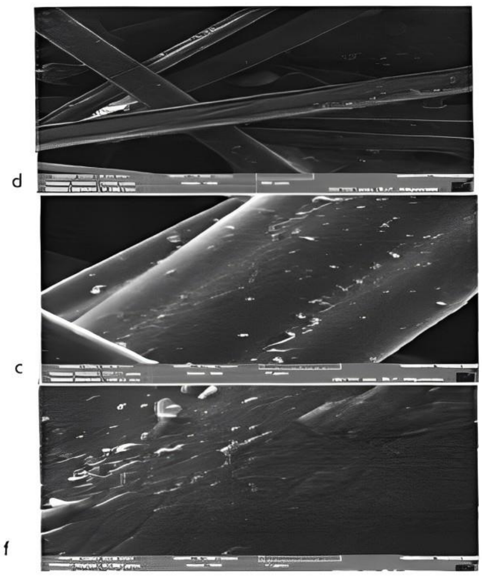

Fig. 4 (b). **Effect of alkaline environment of the model system on the microstructure of basalt fiber: a…c – initial; d…e – after heat treatment at 500 °C**

No obvious traces of leaching were found on the fibers after heat treatment (Fig. 4 (b), d-f). The absence of longitudinal «furrows» is noteworthy, which is a consequence of the high resistance of the modified basalt microfiber to alkaline environment. In general, it can be said that the surface morphology of the heat-treated fiber does not undergo serious changes under the influence of the aggressive environment of cement milk (Fig. 3).

The low alkali resistance of the original basalt fiber can be determined by several factors. As a result of the rapid cooling of the melt during the formation of fibers, a heterogeneous volumetric and surface loose high-temperature structure is fixed in them. At the same time, the fiber is characterized by surface defects (microcracks, micropores, bond breaks, etc.). All this together intensifies the process of dissolving the silicate framework.

During the heat treatment from 300 to 500 ^{0}C, defects are healed, the chemical composition and surface structure of the fibers change as a result of $Fe^{+2} \rightarrow Fe^{+3}$ oxidation and the formation of iron-oxygen tetrahedra $[FeO_4]Na$ with the involvement of alkaline cations on the fiber surface. The above processes are most active at 500 ^{0}C, a temperature close to the glass transition temperature (the transition from the solid to the plastic state).

At higher temperatures, structural changes associated with pre-crystallization processes (600 ^{0}C) and crystallization (700 ^{0}C) begin in basalt fibers, especially on their surface. Structural changes associated with crystallization are accompanied by the formation of various kinds of defects that make the material more chemically active and lead to accelerated interaction with alkali. At the same time, fiber weight loss increases.

To explain the processes occurring in the fiber structure during its heat treatment, X-ray diffraction patterns (Fig. 5) and spectra (Fig. 6) of the original and heat-treated fibers were obtained.

X-ray diffraction data were used to obtain an idea of the microstructural state of the initial and heat-treated basalt fiber.

The X-ray diffraction patterns of the basalt fiber are typical of structured amorphous materials with a characteristic broadened intensity profile in the region of angles around 30^0 (Fig. 5).

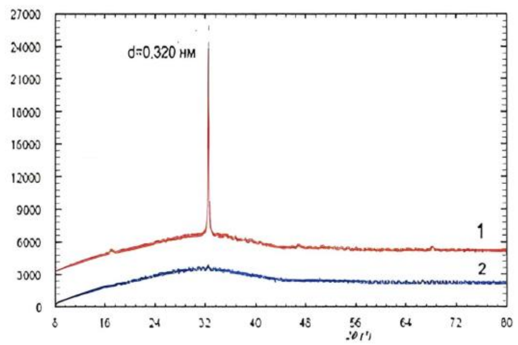

Fig. 5. **X-ray diffraction pattern of basalt fiber before (1) and after (2) heat treatment**

A characteristic feature of the fiber X-ray diffraction patterns is the presence of a single reflection 32.5^0, with this reflection being most intense in the X-ray pattern of the initial fiber. Based on the general ideas about the rock-forming mineral phases of basalts and on the basis of XRD diagnostics performed using the diffraction database, this reflection can be attributed to $Ca(Al_2Si_2O_8)$.

The presence of this mineral in the X-ray diffraction patterns can be interpreted as a consequence of the finely plastic form of the crystallites. These nanoscale minerals on the surface of the fibers are attributed to newly formed phases during the drawing process. During heat treatment, they disappear from the fiber surface due to structural changes.

Comparison of the infrared spectra of basalt fiber before and after thermal modification showed a modification of the absorption band profiles of aluminosilicate groups in the region of 1000...300 cm^{-1} (Fig. 6).

In particular, the heat treatment causes a decrease in the intensities of the absorption bands 1019 and 1052 cm^{-1}, which are characteristic of the valence vibrations of Si-O-Si (Al) bonds in the anionic chain groupings (Q^2) of clinopyroxenes. In view of this, there is an increase in the intensity of the bands of the framework structural fragments (Q^4) in the range of wave numbers 1084...1300 cm^{-1}.

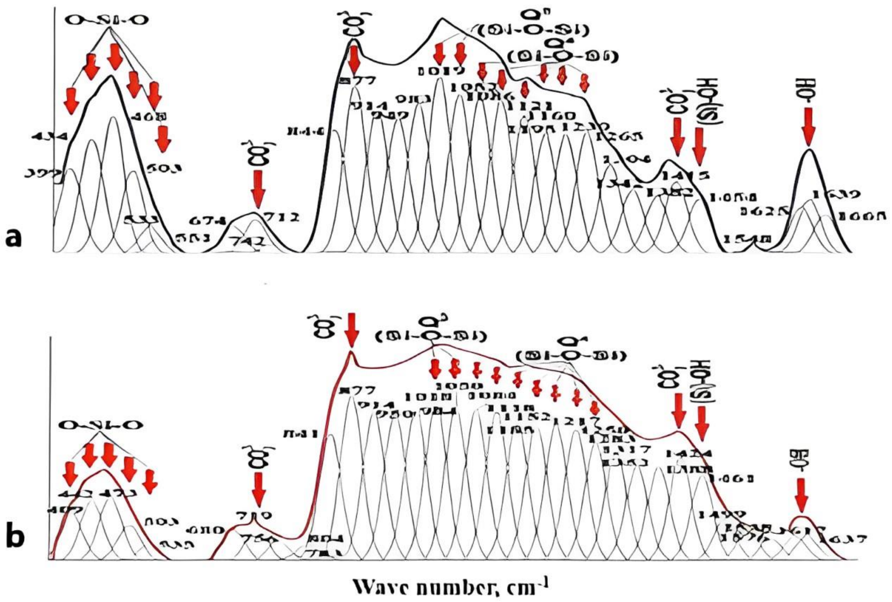

Fig. 6. **Comparison of diagrams of normalized profiles of infrared spectra of the initial (a) and heat-treated (b) fiber**

These circumstances indicate an increase in the level of polymerization, since an increase in the concentration of framework groups (Q^4) occurs due to a decrease in the number of chain fragments (Q^2). This results in a densification of the basalt fiber structure, which ultimately has a positive effect on the fiber's resistance to alkaline attack. Such structural shifts become possible due to the $Fe^{+2} \rightarrow Fe^{+3}$ transition as a result of heat treatment under oxidizing conditions, as noted by many researchers.

In order to select the basalt fiber most suitable for reinforcing the cement matrix, taking into account the wide geography of fiber application in the construction industry and the constantly growing volume of road construction in Ukraine, the fibers of the largest producers of basalt fiber were analyzed.

Based on a variety of initial properties, for a more accurate selection of basalt fiber, we analyzed the raw material composition, the ratio of geometric characteristics (length and diameter) of the fiber, as well as the methods of its production (Table 2).

Table 2: **Properties of basalt fibers from different manufacturers**

Company	Technology	Raw materials	Diameter, μm
Technobasalt - Invest LLC	Centrifugal dispersion	Basalt (SiO_2 – 53%)	13…20
Plant of building materials and basalt products, LLC	Melt blowing by vertical air jet	Underlying basalt (SiO_2 – 49%)	3…5
Basalt fiber & composite materials, LLC	Duplex process	Basalt (SiO_2 – 53%)	1…3
Armbud, LLC	Centrifugal dispersion	Basalt (SiO_2 – 52%)	1…3

One of the important initial parameters of a fiber is its composition. To increase the adhesion to cement stone, it is necessary that the fiber is characterized by a silicate composition, since concrete has an alkaline environment.

The elemental composition of basalt fiber (Table 3) from different manufacturers is characterized by slight variations in the elements.

Table 3: **Composition of basalt fiber**

Components	Technobasalt - Invest LLC	Plant of building materials and basalt products, LLC	Basalt fiber & composite materials, LLC	Armbud, LLC
	Fiber №1	Fiber №2	Fiber №3	Fiber № 4
Na	1,87	3,6	2,3	1,62
Mg	3,07	1,8	1,8	2,52
A1	5,331	6,4	6,9	5,41
Si	19,43	18,4	19,2	18,03
P	0	0,2	0,2	0,23
K	0,33	0,6	0,5	0,59
Ca	3, i	3,2	2,9	3,23
Ti	0,69	0,5	0,5	0,44
Mn	0	0,1	0	0
Fe	3,64	4,2	3,5	7,05
O	62,19	61	62,2	60,75
S	0,46	0	0	0,03

The resistance of basalt fibers in aggressive environments can be judged by the chemical composition of the raw material from which they are extracted. For example, alkali resistance increases with the introduction of structure-sealing oxides such as zirconium, aluminum, iron, tin, etc. Fiber resistance is also significantly affected by the technology and production method.

All this together can determine the possible submicron inhomogeneities associated with different Fe^{+2}/Fe^{+3} contents in the fiber, according to a number of researchers [11…12], determines the possible submicron inhomogeneities associated with the presence of iron outside the silicon-oxygen network, which in turn makes the fibers unstable to oxidation, and can also lead to crystallization at elevated temperatures.

As noted earlier, the geometric characteristics of the fiber are also important in the design of micro-reinforced materials. According to the literature, it is known that to achieve the optimal plastic-aggregate state of a micro-reinforcing component in a mixture, the initial components with an optimal length-to-diameter ratio of more than 10 should be selected [13…15]. The fiber produced by Armbud is characterized by the required ratio of geometric characteristics.

Cement milk was used as an aggressive medium, characterized by a sufficient alkaline environment (pH = 12.9). Fluffed fiber was introduced into the specified solution (Fig. 7). The concentration of basalt fiber in the solution was chosen taking into account the maximum fiber content in cement composites (3...12% by weight of the binder), established on the basis of literature data. The concentration was 7%.

The samples were aged at room temperature for 7, 14, 28, 72 days. After the exposure period, to remove the remaining cement component, the fibers were washed with a weak hydrochloric acid solution (0.1%) through filter paper. To remove impurities, the sample was further washed with 1 liter of distilled water. Then the basalt fiber was dried at room temperature. The solubility of the fiber was determined by the difference in masses before and after the experiment, as a dry residue as a result of exposure to alkaline medium. The mass loss was expressed as a percentage (Table 4). Possible fiber losses during washing (technological losses) were assumed to be comparable for each experiment.

Table 4. **Results of the study of alkaline resistance of basalt fiber**

№	Test time, days	Fiber weight before the experiment, g	pH initial	Weight after aging, g	Weight loss, %	pH after the experiment	Color
1	7	4,9	11,9	4,2	14,3	11,9	normal
2	14	5,2	11,9	3,6	30,7	11,5	normal
3	28	5,0	11,9	3,4	32,0	11,4	poorly lit
4	72	5,1	11,9	2,1	58,8	11,3	illuminated

During the observation on day 7, a slight precipitation of white precipitate was detected on the surface of the fibers, probably due to the crystallization of CaO on the keratinized surface of the basalt.

In the period from 14 to 28 days, as a result of thinning of smaller fiber villi, the total mass of basalt fiber gradually crumbled with slow deposition of small fiber particles (up to 20%). On day 28, the amount of white sediment increased and covered more and more large fibers. Examination of the sample on day 72 revealed a change in the color of the fiber surface from lightly colored for small villi to gray for larger fibers.

It was found that the change in the pH of the cement mortar significantly depends on the time the fiber samples were in it. Thus, on day 7, no increase in pH was recorded, on day 14, a decrease of 2% was noted, and in the period from 28 to 72 days, a decrease of 4.3% was achieved relative to the initial one.

After 28 days of exposure to the solution, the weight loss was about 32%. Long-term storage of fiber (72 days) in solution leads to a mass loss of up to 59%.

The above facts are consistent with the results of microstructural studies of basalt fibers (Fig. 7). There is a significant leaching of the initial aluminosilicate materials after aging for 28 days in the alkaline environment of cement milk. This contributes to the appearance of characteristic traces of corrosion on the surface (Fig. 7 c, d), which negatively affects the reinforcing function of the anisotropic component of the cement matrix.

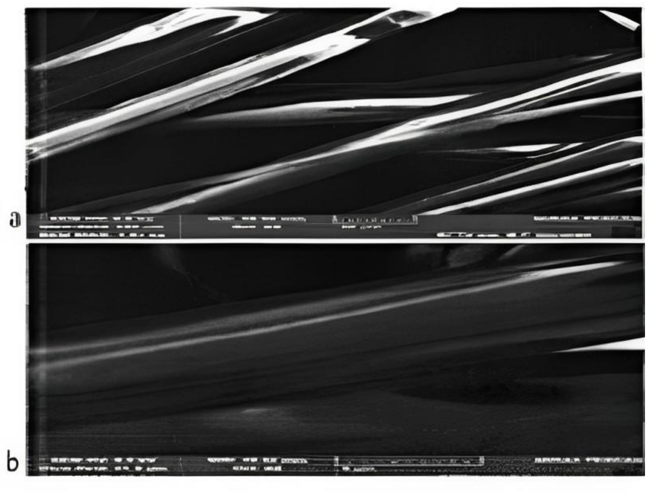

Fig. 7. **Microstructure of basalt fiber:**
a, b - in the initial state; c, d - aged in an alkaline environment for 28 days

When operating basalt fiber in real conditions in concrete, the degree of fiber dissolution will not be so significant, since the dissolution processes will subside as the cement stone hardens and hardens. Nevertheless, the chemical processes occurring in concrete during its service life, in which the fiber is involved, should not be underestimated. In this regard, it is necessary to develop a method of fiber modification to increase its resistance in an aggressive alkaline environment.

Conclusions.

1. The expediency of heat treatment of basalt fiber at a temperature of 500 ^{0}C with subsequent cooling in an air environment at room temperature to increase its alkaline resistance has been substantiated. A model of modification of basalt fiber during heat treatment based on the $Fe^{+2} \rightarrow Fe^{+3}$ transition as a result of heat treatment under oxidizing conditions is proposed. The change in the coordination environment that occurs during this process initiates the formation of bonds between $(Si_2O_6)^{-1}$ and their transformation into a skeletal structural type with the formation of iron-containing plagioclase phases. These processes lead to the compaction of the fiber structure and contribute to the increase of its alkali resistance, which is confirmed by the morphostructural features of the surface of the heat-treated basalt fiber aged in cement mortar.

2. The analysis of the main fibers currently used in the production of building materials as a micro-reinforcing component, according to a number of variable criteria, made it possible to single out basalt fiber as the most rational in terms of its use in the production of fine-grained micro-reinforced cement concrete, including for road construction. A comparative analysis of fibers from 4 manufacturers by a set of indicators: geometry, chemical composition, physical and mechanical characteristics, made it possible to identify the fibers of Armbud, which most closely meet the requirements.

3. It was found that basalt fiber undrgoes significant dissolution during aging in an aggressive and alkaline environment with a pH corresponding to the hydration of cement stone. At the same time, after 28 days of exposure to an alkaline solution, the mass loss is about 30%. Long-term storage of fiber (72 days) in solution leads to a mass loss of up to 59%. It becomes obvious that there is a need to develop a method to increase the alkali resistance of basalt fiber to increase its durability.

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Використання модифікованого базальтового волокна в транспортному будівництві

Базальтове волокно - це перспективний матеріал, який може бути використаний для створення нового класу будівельних матеріалів. Воно має ряд переваг, включаючи високу міцність, низьку вагу і стійкість до хімічних впливів. Одним з недоліків базальтового волокна є його низька лугостійкість. У лужному середовищі волокно руйнується, що обмежує його застосування в будівельних матеріалах, які піддаються впливу лугів. У статті досліджено вплив термічної обробки на лугостійкість базальтового волокна. Було встановлено, що термічна обробка при температурі 500 °С підвищує лугостійкість волокна на 80%. Це відбувається в результаті ущільнення структури волокна і формування на його поверхні залізокисневих тетраедрів, які стійкі до дії лугів. Автори статті обґрунтовують доцільність використання термічно обробленого базальтового волокна в транспортному будівництві. Цей матеріал може бути використаний для виготовлення нових типів будівельних матеріалів, які будуть стійкі до впливу лугів.

Ключові слова: *базальтове волокно, транспортне будівництво, термічна обробка, структура фібри, лугостійкості волокон, рентгенограми, спектри.*