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Processes of structure formation and neoformation of basalt fiber in an alkaline environment

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Abstract. Several approaches can be used to determine the effect of an alkaline environment on glass materials (basalt fibers). One of the most common approaches for the determination of the alkali resistance of a substance is to measure the relative mass loss after the interaction of the object of research with the alkaline environment. According to the practice guidelines, the approach involves determining the alkali resistance of a glass by measuring the mass loss per unit area of the glass under test after boiling it in an alkaline solution. But the disadvantage of this approach is it can't be applied to basalt or another multicomponent fiberglass as it doesn't introduce different speeds of fiber dissolution.

1. Introduction

There are many ways to determine the effect of an alkaline environment on glass materials (e.g. basalt fibers). One of the most common ways to determine the alkali resistance of the substance is to measure the relative mass loss after the interaction of the study object with the alkaline environment. This approach has a significant advantage over the measurement of the relative mass loss due to the increase of the sample surface; therefore, the shape of the sample has no particular importance.

2. Analysis of recent research and publications

In recent years, research on the effect of an alkaline environment on the structure of the basalt fiber, as well as problems of obtaining and use of silicate melts of various functional purposes became the subject of the works by Jigiris, Makhova, and Aslanova.

3. The purpose of this work

The purpose and objective of the present research are to study the morphology of the basalt fiber surface, its elemental and phase composition before and after its interaction with an alkaline environment. These objectives allow identifying the details of the fiber etching process in an aggressive environment and the impact of this process on the coating process.

4. Materials and methods of research

Basalt fibers and alkaline solutions were used as raw materials. The first group includes a basalt fiber that completely dissolves in etchant solution; second group covers the basalt fiber that partly dissolves with formation of basalt fiber fragments without the change of its structural properties; third group includes the basalt fiber that keeps the initial form with formation of surface micropore structure [1].



Thus, divergence in interaction of basalt fibers with etchant solution became a pre-condition for the necessity of research (table 1).

Table 1. Chemical composition of basalts of deposits

Name of deposit	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MgO	CaO	TiO ₂	R ₂ O
basalt	50.6	16.0	14.8	8.1	5.1	9.8	0.9	3.2

5. Research results

The morphology of the basalt fiber surface involves significant changes after 8 days of holding the fiber in an alkaline solution. Shows basalt fiber data of the scanning electron microscope (SEM), made at different intervals of time in Figure 1.

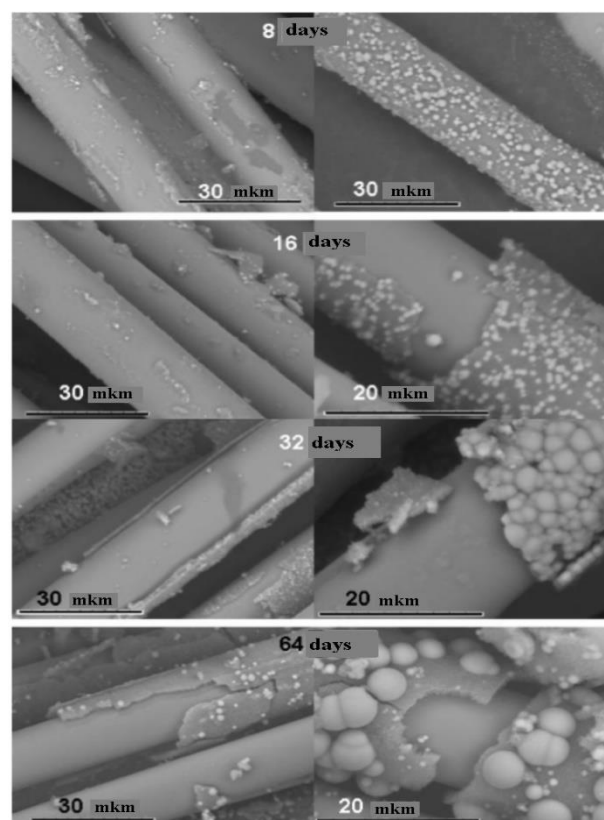


Figure 1. Uncoated basalt fiber after etching in the sodium hydroxide solution.

With an increasing holding period (16 days or more), it can be observed that the corrosion layer consists of two layers [2, 3]. The first one is on the surface and is tightly adjacent to the fiber and consists of particles of hexagonal lamellar shape directed perpendicular to the surface of the basalt fiber Figure 2.

The second – an external layer – consists of spherical particles. With the increasing etching period, there is an increase in the size and number of spherical particles, as well as an increase in the thickness of the corrosion shell. After 64 days of holding the basalt fiber in an alkaline solution, the number of fibers with peeling corrosion significantly increases. The shell thickness makes 650-750 nm after 16 days of etching and increases up to 1.5-2 μm after 64 days of etching. Changes in the elemental composition of the fiber surface, depending on the holding time in alkaline solution, were studied by elemental microanalysis [4]. The selection criteria were the integrity of the “corrosion shell – fiber” pair

and the morphological features of the corrosion shell for each sample. The results of the EDS analysis of fiber surface studies after 8 days of etching are shown in Figure 3.

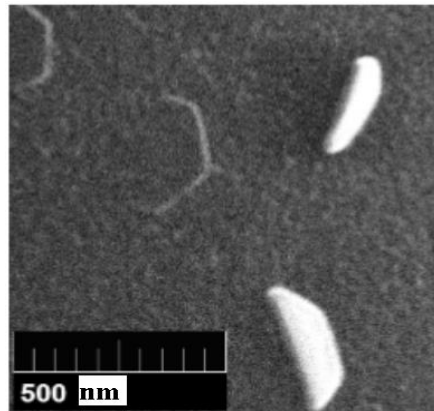


Figure 2. Hexagonal lamellar particles on the basalt fiber after etching in a sodium hydroxide solution

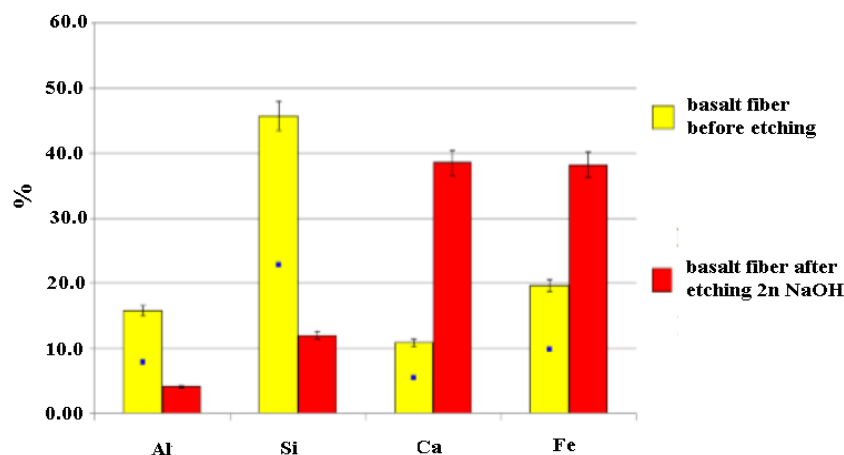


Figure 3. Results of the EDS analysis of the uncoated basalt fiber before and after etching (8 days)

According to the results of studies, it can be concluded that the concentration, the main components of basalt fibers, silicon, and aluminum - are decreasing, and the concentration of iron and calcium - are increasing. The concentrations of MgO and TiO₂ are too low to be detected by the EDS analysis [5].

To determine the phase composition of basalt fiber before and after etching, the methods of IR spectroscopy and X-ray phase analysis were used.

The IR spectra of the initial fibers and fibers after etching in an alkaline medium are shown in the Figure 4.

The bands attribution is shown in Table 2. There is a group of very intense bands in the range 1000-1200 cm⁻¹.

According to the data [6,7] the bands can be assigned to the valence fluctuations of the Al-O-Si composition. In the region of 1400-1700 cm⁻¹, there is a group of bands of medium intensity. These bands can be attributed to the valence fluctuations of the MeOH bonds (under Me are meant the metals that make up the fiber). In the infrared spectrum of the etched basalt fiber, there is a change in the intensity of the bands, namely, the intensity in the region of 1000-1200 cm⁻¹ decreases. It can be assumed that the decrease in the intensity of the bands in the region of 1000-1200 cm⁻¹ is due to the dissolution of the aluminosilicate composition in the surface area of the fiber, whereas the increase of the intensity of the bands in the region of 1400-1700 cm⁻¹ is associated with the formation of metal

hydroxides on the fiber surface. A very intense band of ~ 1450 cm⁻¹ is present in the IR spectrum of the corrosion shell, which is exfoliated. It can be attributed to the valence vibrations of the carbonate group. The X-ray phase analysis confirms the presence of sodium carbonates and iron bicarbonates in the corrosion layer Figure 5.

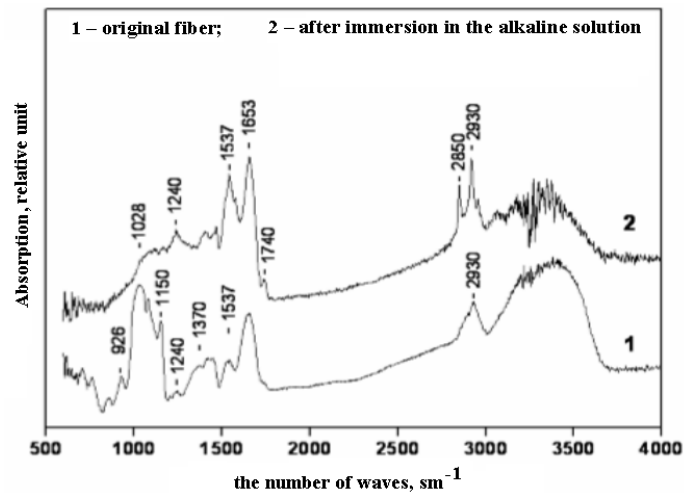


Figure 4. IR spectra of the basalt fiber: 1 – original fiber; 2 – after immersion in the alkaline solution

Table 2. Results of the basalt fiber IR spectra studies

Name of deposit	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MgO	CaO	TiO ₂	R ₂ O
basalt	50.6	16.0	14.8	8.1	5.1	9.8	0.9	3.2

Corrosion in a saturated Ca(OH)₂ solution

After etching of the original basalt fiber in a saturated Ca(OH)₂ solution during 16 days, two types of morphological features can be observed on the fiber surface, namely, not tightly placed crystals with the length of 2-3 μm Figure 6. According to the SEM analysis, both neoformations and their clusters consist of tightly placed elongated plate crystals [8].

The EDS analysis of different basalt fiber surface areas with no clusters shows no significant changes in the elemental composition of the surface compared to the one of the not etched fiber Figure 7.

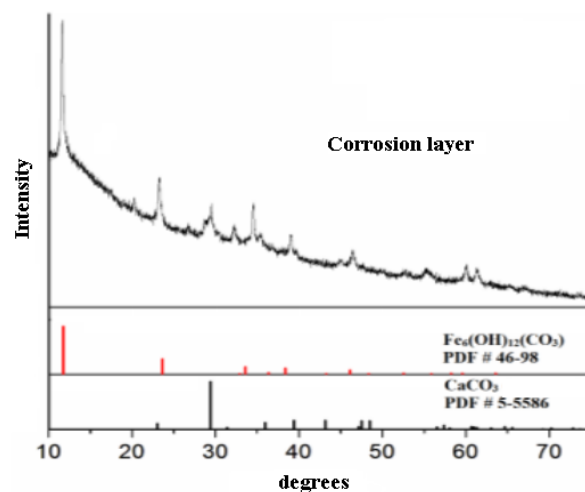


Figure 5. X-ray pattern of the corrosion layer

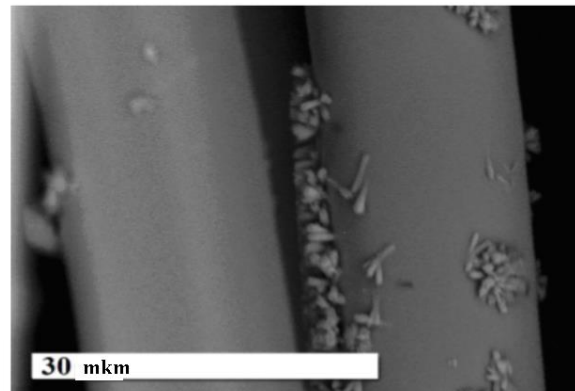


Figure 6. An SEM image of the basalt fiber after 16-days etching in a saturated Ca(OH)_2 solution

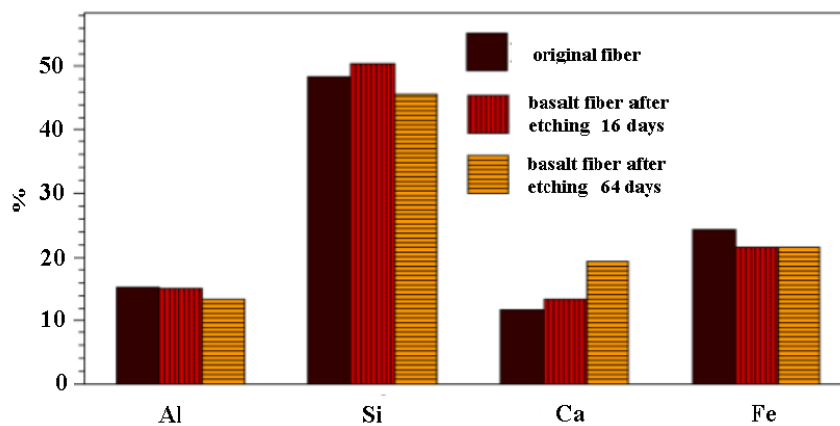


Figure 7. Normalized EDS analysis data obtained from those areas of the basalt fiber surface on which no aggregates of needle-like crystals were observed after immersion of the fiber into the saturated Ca(OH)_2 solution for 16 and 64 days

It should be noted, sufficient changes in the elemental composition of the original basalt fiber could be observed after its immersion into the NaOH solution for 16 days. Thus, the basalt fiber undergoes more changes after its etching in the NaOH solution, than in the solution of Ca hydroxide due to the lower concentration of the latter [9, 10].

The SEM images of the basalt fiber after 64 days of etching in a saturated Ca(OH)_2 solution are shown in Figure 8.

The picture shows the number of neoformations increases with the increasing duration of etching. A larger number of round-shaped particles cover nearly the entire fiber surface (Figure 8 a).

Koga observed a similar morphology for calcite formed during the reaction of Ca(OH)_2 with CO_2 under conditions of 100% humidity. Along with the round-shaped particles, there are plate crystals of hexagonal shape (Figure 8 b) and flower-like clusters present on the surface. The clusters cover a large part of the fiber surface (Figure 8 c). At higher magnification, it can be seen that these clusters are polycrystalline (Figure 8 d). According to the X-ray phase analysis data, the phase composition of the corrosion shell corresponds to calcite (Figure 9).

The method of X-ray phase analysis discovered no Fe-containing phases. However, some of the morphological figures, namely plate hexagonal crystals which are typical for hydroxycarbonate of iron formed at high pH, may indicate the formation of this phase [10]. Perhaps the quantity of the Fe-containing phase on the etched basalt fiber surface is below the visibility limit of the X-ray phase analysis method.

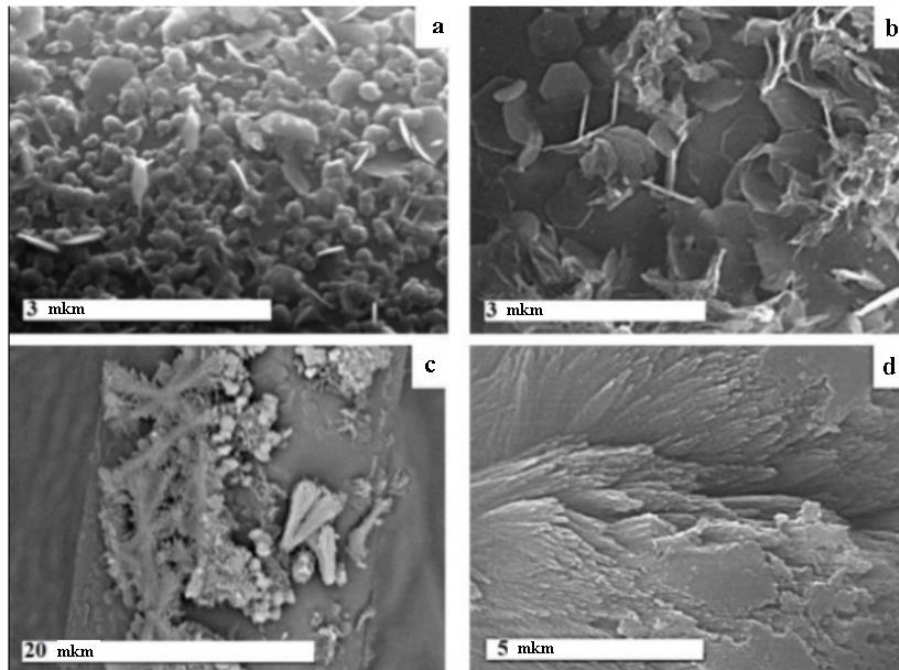


Figure 8. SEM images of the basalt fiber after 64 days of etching in a saturated solution of $\text{Ca}(\text{OH})_2$

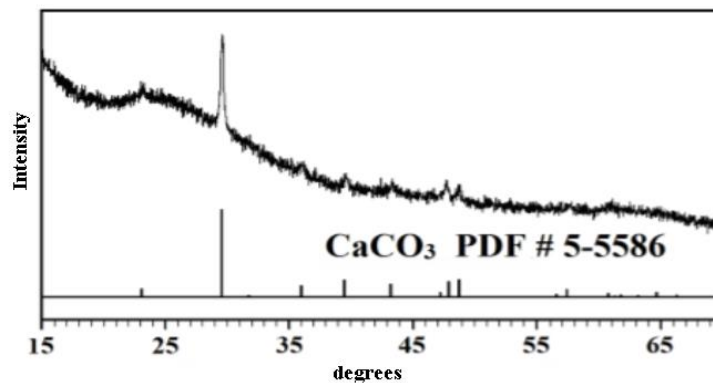


Figure 9. X-ray pattern of the basalt fiber after 64 days of etching in a saturated $\text{Ca}(\text{OH})_2$ solution

The long lasting 64-day etching of basalt fibers in the $\text{Ca}(\text{OH})_2$ solution is followed by the change in elemental composition (Figure 8.), morphology and phase composition (Figure 9). As it can be seen from Figure 8 the content of Al and Si on the basalt fiber surface decreases and on the opposite, the content Ca increases. More than 70% (of the mass) Ca are noticed in the neoformations on the surface after immersion of the fiber in a $\text{Ca}(\text{OH})_2$ solution. These data together with the data of the X-ray phase analysis prove that a neoformation is a calcium-containing phase like for example CaCO_3 calcite.

The origin of calcite on the surface can be explained by the fact that the gaseous CO_2 slowly enters the solution in a saturated $\text{Ca}(\text{OH})_2$ solution and reacts with it, forming CaCO_3 . Then CaCO_3 deposits in the form of crystals on the surface of the basalt fiber, possibly in the places with no defects. The recovery of Ca in the solution takes place due to the dissolution of a new portion of basalt fibers in a $\text{Ca}(\text{OH})_2$ solution. Further increase in the onset of calcite occurs in the process of dissolution and deposition. The SEM analysis showed that the binding of the corrosion shell to the fiber core decreases with increasing etching time. As a result, the corrosion membrane is torn away from the fiber. The basic patterns are revealed during the long-term etching of the fiber in a solution of $\text{Ca}(\text{OH})_2$, similar to those that were established for etching of basalt fiber in a solution of NaOH. The corrosive shell does not prevent the

alkali from penetrating the surface of the basalt fiber, first because of poor bonding to the surface of the fiber and deflection, and second, because of the porosity of the corrosion shell.

6. Conclusions and prospects for further studies

Based on the data of SEM/EDS, X-ray phase analysis and IR-spectroscopy we can conclude that the corrosion shell, formed on the original basalt fiber after its etching in an alkaline solution of NaOH, consists of plate hexagonal crystals of $\text{Fe}_6(\text{OH})_{12}\text{CO}_3$ ferrous bicarbonate directed perpendicular to the surface of the fiber and outer layer, made of globular particles of calcium carbonate.

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