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ИССЛЕДОВАНИЕ СВОЙСТВ БАЗАЛЬТОПЛАСТИКА В ПРОЦЕССЕ КЛИМАТИЧЕСКОГО СТАРЕНИЯ

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Приведены результаты климатических испытаний однонаправленных базальтопластиков после выдержки в климатической камере GRONLAND при температуре 60 ºС и влажности 100% в течение 1, 2 и 3 мес. Показано, что в первый месяц механические свойства не меняются, после второго и третьего месяца экспозиции модуль упругости образцов возрастает на 6-10 %, предельная деформация уменьшается на 5-7%, прочность практически не изменяется. По результатам термомеханических исследований методом дифференциальной сканируещей калориметрии установлено постепенное повышение температры стеклования образцов от 124.4 °С (1 мес.) до 125.8 °С (2 мес.) и 126.4 °С (3 мес.). Это свидетельствет о дополнительной полимеризации связующего в температурно-влажностных условиях климатической камеры. По результатам цифровой обработки микрофотографий поверхности образцов установлена качественная корреляция между изменением свойств и состоянием поверхности.

Ключевые слова: *однонаправленные базальтопластики, климатическое старение, механические свойства, продольный изгиб, температура стеклования, диференциальная сканируюшая калориметрия, микрофотографии по-верхности, цифровая обработка.*

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BASALT PLASTIC PROPERTIES UNDER CLIMATIC AGING CONDITIONS

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The paper presents results of climatic tests of unidirectional basalt plastics after curing in GRONLAND climatic chamber at 60 ºC and 100% humidity for 1, 2, and 3 months. The mechanical properties do not change in the first month, but after the second and third months of exposure elasticity modulo of samples increases by 6-10%, ultimate strain decreases by 5-7%, strength sees almost no change. The results of thermomechanical research using differential scanning calorimetry show the gradual increase of temperature of glass transition of samples from 124.4 °C (1 month) up to 125.8 °C (2 months) and 126.4 °C (3 months). It means that the binder is additionally polymerized in the temperature and humidity conditions of the climatic chamber. After the digital processing of photomicrographs of samples' surfaces, we have established a qualitative correlation between the change in properties and the surface condition.

Key words: *unidirectional basalt plastics, climatic aging, mechanical properties, longitudinal bending, glass transition temperature, differential scanning calorimetry, surface photomicrography, digital processing.*

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INTRODUCTION

The climatic tests are necessary because of the increasing use of polymer composite materials in chemical industry, aviation, car manufacturing, shipbuilding, various branches of mechanical engineering, and modern construction [1]. The polymer composite materials (PCM) age under operating conditions, which is the result of various physical, chemical, and structural transformations on the surface and inside the structural elements. It is not possible yet to predict reliably the change of mechanical parameters of PCM when using more than 30-50 because there is insufficient knowledge about their ageing considering the synergistic effect of daily and seasonal cycles of temperature, humidity, solar radiation, precipitation, wind, and mechanical loads [1].

There is a significant impact on polymer composites used in construction that comes from atmospheric factors (temperature, humidity, solar radiation, etc.), which, as aging facilitators, contribute to the development of physical and chemical processes in the materials and during the operation may significantly reduce their properties [1, 2]. Most PCM used in construction are in direct contact with air or water. A great number of modern studies in Russia [3-8] and internationally [9-18] are devoted to problems of durability of PCM under the effect of increased temperature, humidity, and mechanical loads.

There are studies of the durability of carbon and glass plastics in seawater [10, 11], UV resistance of wood-polymer [12] and hybrid composites [13]. The durability of fiberglass plastics under the combined effects of moisture and mechanical loads was investigated in [14], of temperature and load — [15]. Glass-epoxy, carbon-epoxy and hybrid composites under hydrothermal aging conditions were studied in [9, 16]. The long-term strength of polymer composite reinforcement was also studied in a concrete environment [17], and the durability of basalt plastics was studied at an increased temperature [18].

The current world practice is to establish relationships between surface degradation and changes in composite material properties [19-24]. This allows a non-destructive way to perform an express analysis of its condition and evaluate the critical change of properties as a result of aging. The authors propose an original method for analyzing climatic aging of PCM based on digital processing of surface photomicrographs [25], which follows the advanced trends in the field of polymer construction materials science. The method has been tested and the relationship between the sample surface condition and the change in physical-mechanical and thermomechanical properties as a result of climatic aging has been established [8].

The purpose of this work is to study the degradation of basalt plastic properties during climatic aging under conditions of elevated humidity and temperature.

EXPERIMENT

Samples for research were produced in the form of unidirectional basalt plastics using basalt roving BCF 17-2520-KV13 (Kamenniy Vek, Dubna) and hot-cured epoxyhydride binder EDI: ED-22 epoxy resin (100 wt. %), iso-MTGFA hardener (85 wt.%), accelerator UP-606/2 (1 wt. %) [26].

Unidirectional samples of basalt plastics were made by winding using the authors' patented method [27]. The technology of making such samples is described in more detail in [28]. The method of producing unidirectional plates is based on tight winding (turn to turn) of a cylindrical shell of binder-impregnated roving on a metal mandrel (Fig. 1, a), followed by cutting (Fig. 1, b), unfolding to sheet (Fig. 1, c), under-pressing in the mold, and polymerizing the product as follows: 0.5 h at 120°C, then 4 h at 150°C. Thus, three unidirectional sheet samples of basalt plastic (named Z1, Z2, Z3 for convenience) were produced. All 3 sheets were produced with the same binder, same batch of roving, same winding, pressing, and curing.

After the sheets were cured, thin plates were cut out of them as samples (12 mm wide and with the same sheet thickness without treating the outer surface) and tested as is by the longitudinal bending method [29, 30] (Fig. 1, d). The test results are shown in Table 1.

The samples from each batch were subjected to unloaded climatic testing at 60°C and 100% humidity in a GRONLAND climatic chamber: samples from batch Z1 – for 1 month, samples from batch Z2 – for 2 months, samples from batch Z3 – for 3 months.

The choice of temperature and humidity for testing was based on publicly available data and previous studies of the authors. The territory of Russia is notable for its large geographical length that covers a large number of climatic zones – from extremely cold (Yakutsk, Far North, and the Arctic) to warm humid sea (Sochi, Gelendzhik). It is established that cold and moderate climate renders the least destructive influence on a composite, and warm humid climate impacts it the most. Therefore, the choice of temperature 60 °C and humidity of 100% in the climatic chamber is due to the worst-case scenario of PCM operation.

After climatic aging, the samples were subjected to mechanical longitudinal bending tests, DSC analysis to determine the thermomechanical properties and examine the surface state of the samples by digital processing of photomicrographs.

The data of mechanical tests for longitudinal bending of samples after climatic exposure are given in Table 2.

The data in Tables 1, 2 show that in the first month of exposure there was almost no change in the mechanical properties of basalt plastic samples. In the second and third months the elastic modulus E increased noticeably (by 6-10%), the critical strain ε, respectively, decreased by 5-7% with the strength σ practically unchanged. Additional polymerization of material at temperature 60°C and humidity 100% could result in the increase of elastic modulus (increase of stiffness of samples) [8].

To study the thermomechanical properties, tests were conducted according to ISO 11357-2:1999 [31] by differential scanning calorimetry (DSC). We determined exo-effects on NETZSCH DSC 204 F1 with the speed of heating samples 10 ° C / min to 200-250 ° C in an inert nitrogen environment flowing through the measuring cell at 30 ml / (min· ° C). Typical DSC diagrams of the field samples of climatic tests are shown in Fig. 2.

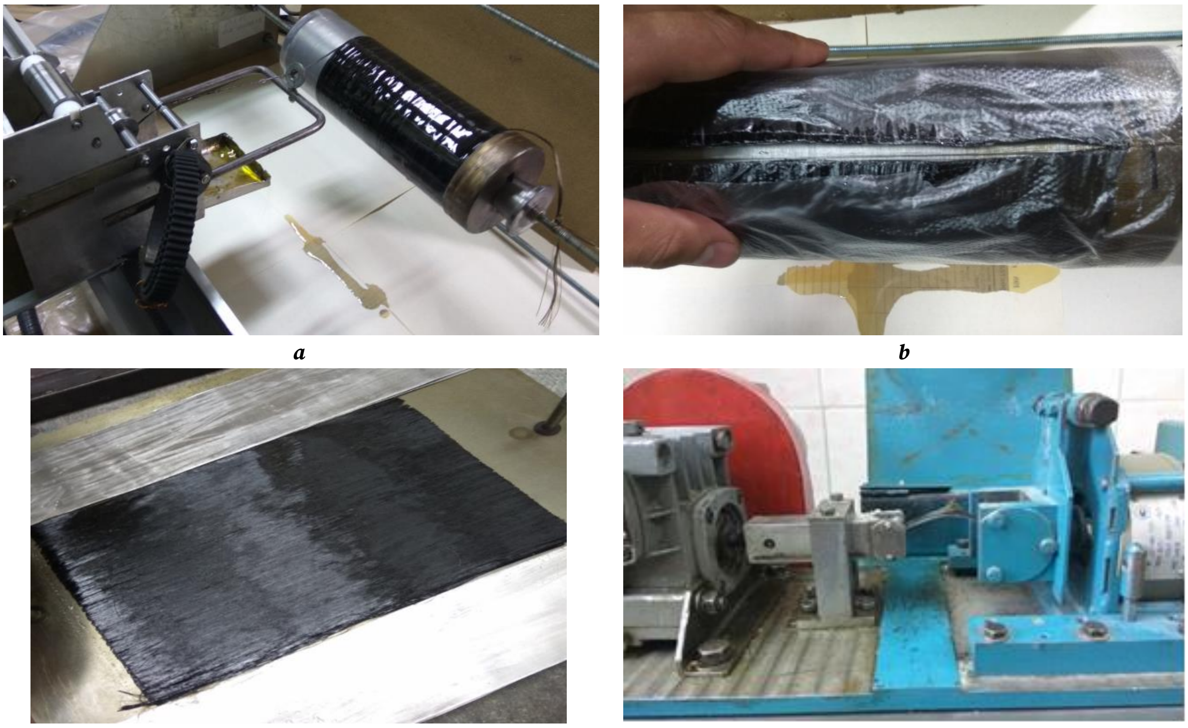


Fig. 1. Demonstration of the method of manufacturing sheet samples of winding products [28]: a-wet winding of the roving on the mandrel (turn to turn); b – cutting of the wound billet along the axis; c - reaming and pressing of the sheet; d - testing of samples for longitudinal bending

Table 1. Test results of unidirectional basalt plastics in the initial state

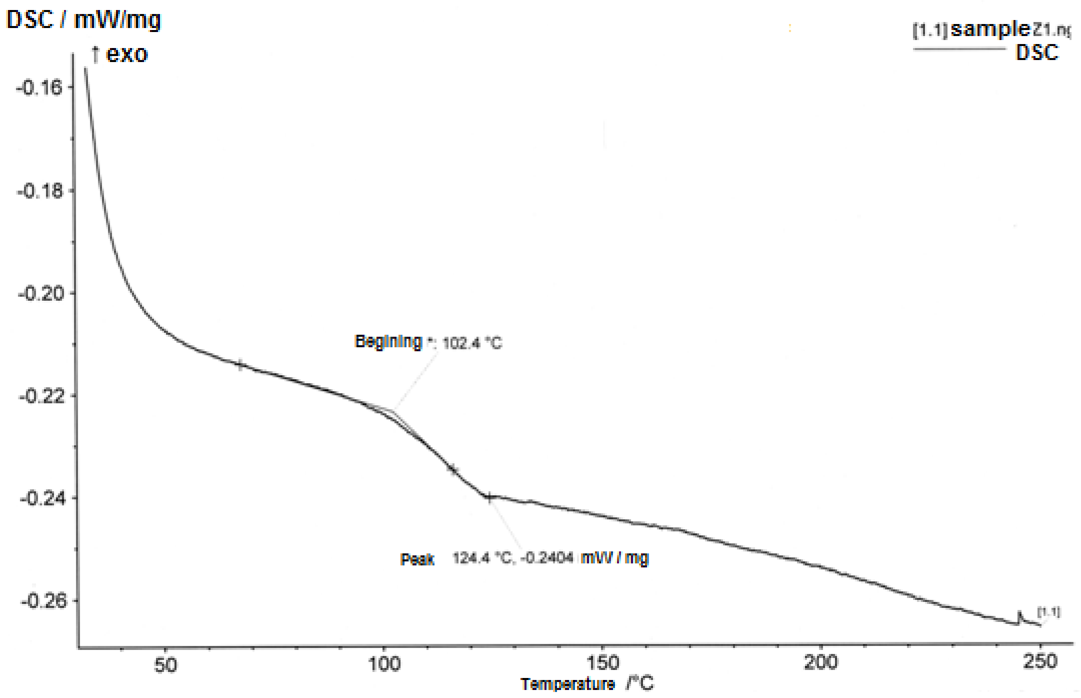
Batch marking	L, mm	b, mm	s, mm	ε, %	E, MPa	σ, MPa
Z1	99.9	11.97	2.09	4.27	42684	1569
	99.8	11.75	2.27	4.20	41900	1629
	100.2	11.53	2.19	4.02	42504	1516
Average value				***4.16***	***42363***	***1571***
Z2	100.0	12.53	1.99	3.97	45739	1613
	100.3	12.75	2.08	3.71	42754	1298
	100.3	11.97	2.06	3.97	45393	1611
Average value				***3.88***	***44628***	***1508***
Z3	100.0	11.89	2.23	4.12	42190	1501
	100.0	11.92	2.20	4.06	41870	1610
	100.0	11.97	2.19	4.02	41398	1488
Average value				***4.06***	***41819***	***1533***
Legend: L – sample length, b – sample width, s – sample thickness, ε – critical strain (at crushing), σ – strength, E – elasticity modulus.						

The peak on the curves corresponds to the glass transition temperature. Note that as the samples stay in the thermo-moisture conditions of the climatic chamber, the glass transition temperature gradually increases - from 124.4 °C (Z1, 1 month) to 125.8 °C (Z2, 2 months) and 126.4 °C (Z3, 3 months). This confirms the effect of binder post-curing, an increase in the elastic modulus observed earlier in [8] and explained by the catalytic effect of moisture on epoxy polymer post-curing in [24, 32, 33]. The essence of the effect: when plasticizing with moisture, the efficiency of intermolecular interaction decreases, the active groups acquire greater mobility, due to which additional transverse bonds are formed. The glass transition temperature, elastic moduli, and strength of epoxy polymers increase after removing moisture [8, 24, 32, 33].

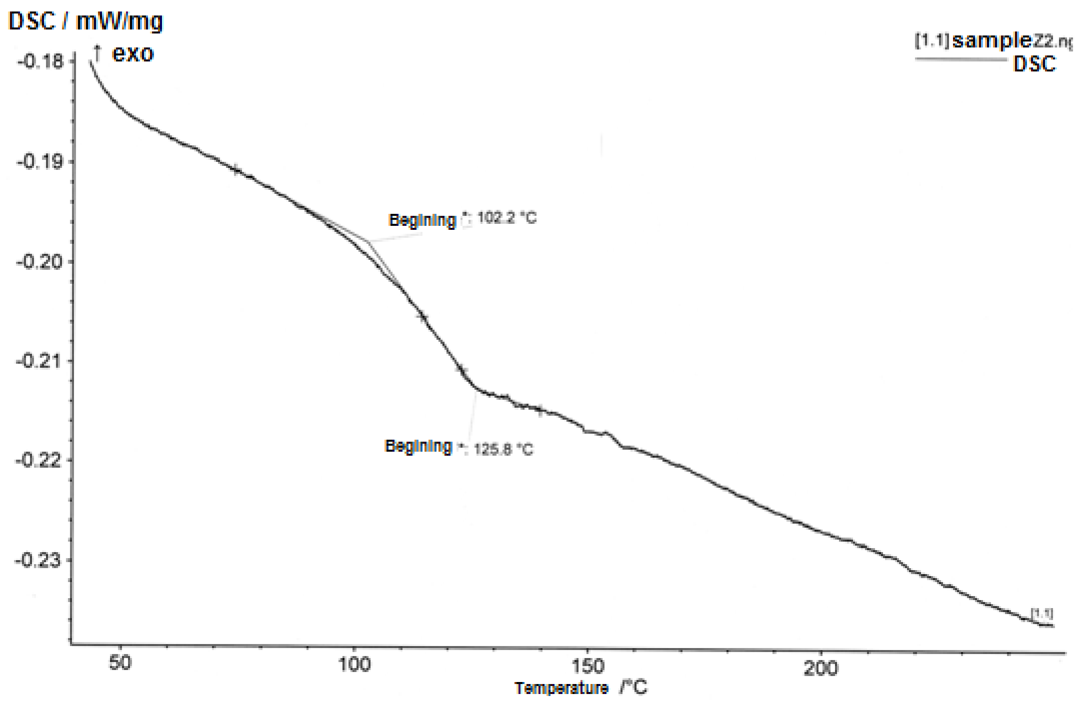
Table 2. Results of longitudinal bending tests of unidirectional basalt plastics after climatic aging in the GRONLAND chamber at a temperature of 60 °C and a humidity of 100%

Batch marking, exposure time	L, mm	b, mm	s, mm	ε, %	E, MPa	σ, MPa
Z1 (1 month)	100.0	11.99	2.07	3.49	40072	1307
	100.0	12.05	2.02	4.20	43703	1562
	100.0	11.95	1.97	4.10	42203	1512
	100.0	12.04	2.23	4.21	40641	1546
	100.0	12.04	2.22	3.97	39650	1433
Average value				**3.99**	**41254**	**1472**
Z2 (2 months)	100.0	12.06	1.88	3.81	46384	1579
	100.0	12.01	1.77	3.67	46150	1487
	100.0	12.07	1.89	3.84	45756	1517
	100.0	12.08	1.90	3.39	48313	1524
Average value				**3.68**	**46651**	**1527**
Z3 (3 months)	100.0	11.99	1.96	3.37	46713	1490
	100.0	12.04	2.12	4.08	43232	1538
	100.0	11.99	2.17	4.08	45181	1543
	100.0	12.11	2.12	3.68	45514	1509
	100.0	11.89	1.93	3.91	46784	1561
Average value				**3.82**	**45485**	**1528**

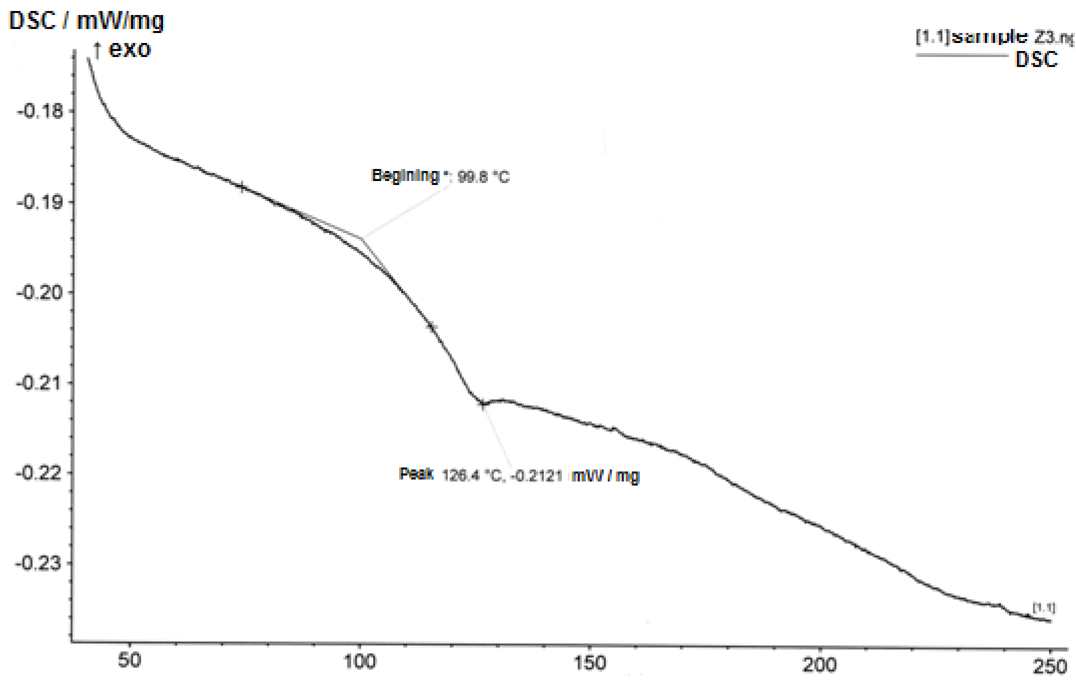
The changes on the surface of samples as a result of climatic effects was studied using the original method of digital processing of surface photomicrographs [25]. Anaconda 3 development environment (https://www.anaconda.com/) was used to make histograms that show the gradation distribution of gray depicted in the photomicrographs of samples taken from three types of basalt plastics in the initial state and after climate aging. Examples of histograms are presented as follows: Top - the original image, next - the histogram of shades of gray (blue columns), and the cumulative distribution function (CDF) - a red curve (https://en.wikipedia.org/wiki/cumulative_distribution_function) (Fig. 3).



a)

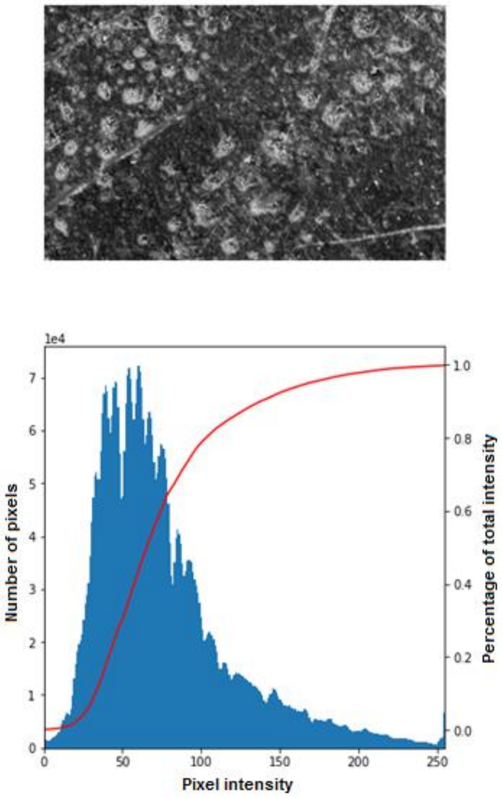


b)

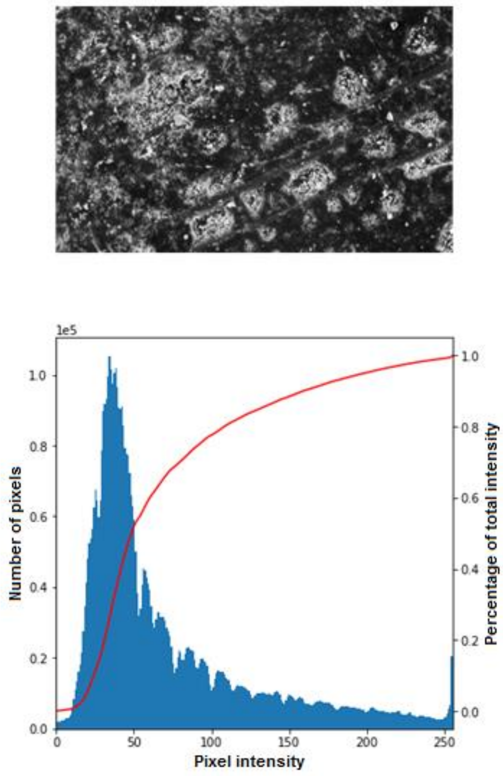


c)

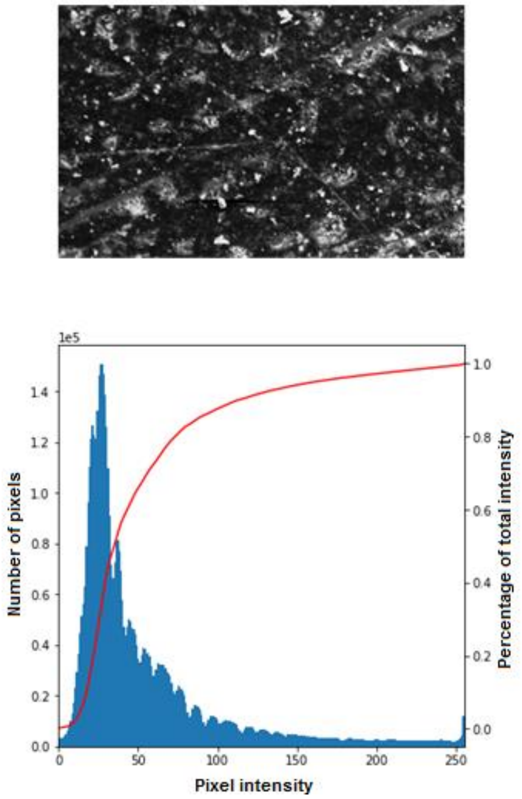
Fig. 2. DSC diagrams of basalt plastic samples after climatic aging in the GRONLAND chamber at a temperature of 60 ° C and humidity of 100%:
a - sample Z1 after exposure for 1 month, b - sample Z2 after exposure for 2 months,
c - sample Z3 after exposure for 3 months



a)



b)



c)

Fig. 3. Photomicrographs of the surface of the samples basalto-plastica (magnification x 500) after the climatic aging in the camera GRONLAND at a temperature of 60 ° C and humidity 100% and their digital processing:
a – Z1 sample after incubation for 1 month, b – Z2 sample after incubation for 2 months, c – sample Z3 after incubation for 3 months

The difference between the states of exposure (initial, after climatic effect) were assessed by comparing the cumulative distribution function (CDF) (Fig. 4).

The difference of CDF functions for samples Z1 and Z2, Z1 and Z3 shows the structural changes in the surface of the samples after their exposure in the climatic chamber for 2 and 3 months. Comparing these data with the results of mechanical and thermomechanical tests, it can be stated that there was a climatic impact, but the destruction of the surface and structure of the composite did not occur (the properties even slightly improved). This is due to the short time of the basalt plastic samples in the climatic chamber.

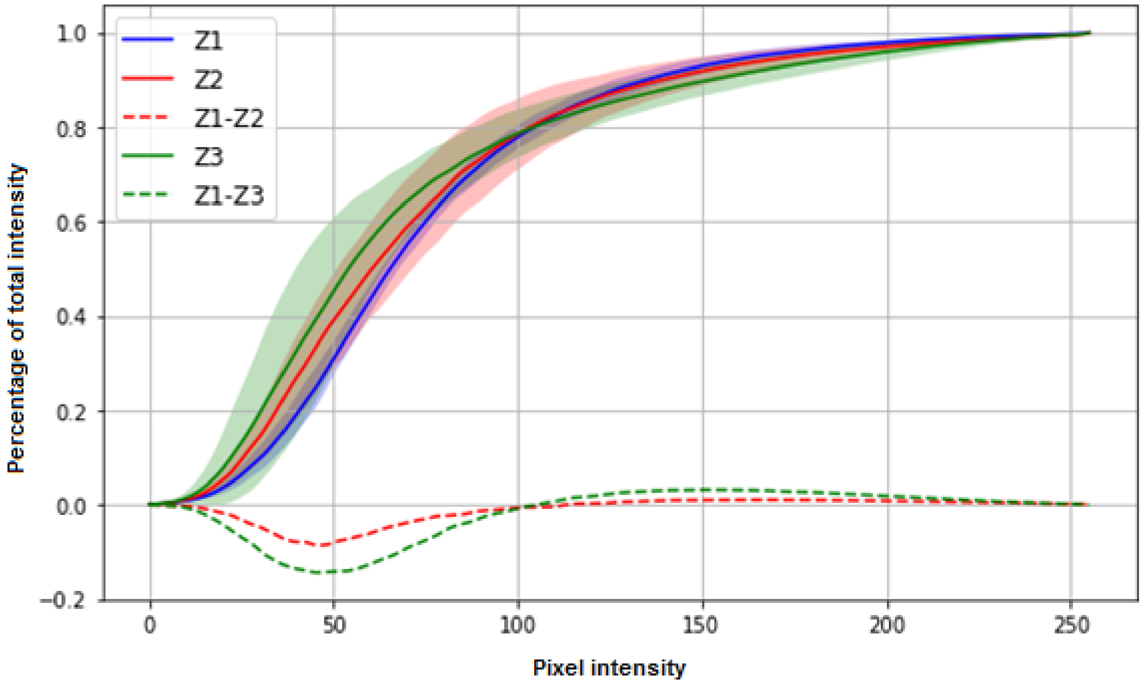


Fig. 4. Comparison of cumulative distribution functions (CDF) of samples from batches Z1-Z3 after climatic aging (solid lines), dotted lines – the difference between the CDF functions

CONCLUSIONS

1. The climatic research of unidirectional basalt plastics has been carried out at exposure in unloaded condition at 60 °C and 100% humidity in climatic chamber GRONLAND for 1, 2, and 3 months.

2. Determining the mechanical properties of the samples by the longitudinal bending method, showed that the properties of the samples stayed almost the same in the first month of climatic aging, but in the second and third months, the elastic modulus increased by 6-10%, the critical deformation during the destruction decreased by 5-7% compared to the initial samples, while the strength also stayed at the same level.

3. The study of the thermomechanical properties of samples by the method of differential scanning calorimetry showed an increase in glass transition temperature from 124.4 °C (1 month) to 125.8 °C (2 months) and 126.4 °C (3 months). This occured due to the catalytic effect of moisture on curing of epoxy polymers.

4. The digital processing of photomicrographs of the samples' surface after climate aging revealed structural changes for samples after 2 and 3 months of exposure. Thus, a qualitative relationship is established between the change in the properties of the samples (on the macro level) and the change in the state of their surface (at the micro level) according to the data obtained from the photomicrographs of the surface.

5. Climatic tests of basalt plastic samples at 60 °C and 100% humidity for 3 months led to an improvement in mechanical and thermomechanical properties. It follows from this that the exposure time was insufficient in order for noticeable destructive changes in composites.

The work was conducted using the equipment of the Biysk Regional Center for Collective Use, Siberian Branch of the Russian Academy of Sciences (IPCET, Siberian Branch of the Russian Academy of Sciences, Biysk).

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