1. Introduction

The search for the optimal filling of epoxides aimed at enhancing their properties is underway [1, 2]. The nanobasalt fibers are a sought-after material for the technologically simple amplification of epoxy, polyester, and other mesh polymers. The fibers that are connected at the level of nanotechnologies should provide strong bonding. This is achieved through the mechanical interweaving and adhesion between the composite and a polymer. Such technologies are promis-
ing from a practical point of view. Being fibers, in contrast to other promising nano dispersions (aerosils, nanotubes, graphene, nano alumina, nano metals), they should have a significantly higher filling threshold (40–50 % by weight and above). Such a percentage of their filling makes it possible to expand the prospects for varying their formulations.

In addition, similarly to other inorganic oxide materials (glass, granite, aluminosilicates), basalt exhibits an excellent affinity for epoxides and other polar resins [3, 4]. It appears a relevant task to investigate a series of experimental searches for the formulations of epoxy polymer composites with elevated resistant properties [5, 6].

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2. Literature review and problem statement

Interest in the epoxy-basalt composites has not waned for decades. They are studied both separately and in a mixture with other fillers [7, 8], examining their strength and durability. Here are examples of studies into such composites.

Paper [8] reports the results of studying the effect of basalt fiber on epoxy composite. It was shown that after ultrasonic processing, when compared, the relaxation processes in composites with basalt, glass, and carbon fiber are of the same order [8]. Moreover, basalt fiber yields (for polyepoxide with soot in the amount of 30 % by weight) higher adhesivity (approximately, 70 MPa), cohesion, elasticity module, and relative deformation, compared to other fibers [8]. However, there are many unresolved issues related to testing the composite material at temperatures above 200–250 °C. The reason for this may be objective difficulties associated with the fact that the cited paper examined the contact temperature of the experimental sample in the zone of mechanical interaction between surfaces. At the same time, the established values of friction factors limit the temperature mode of the pair sample–surface, which makes the relevant studies impractical. The option to overcome the corresponding difficulties may be the development of composites with increased mechanical properties, allowing the application of the material over a wide range of temperatures. This is the approach used in works [9, 10]. These works show that even small concentrations of basalt fiber (for example, 0.1 % by weight) can significantly improve the properties of epoxy, which is traditionally explained by good interphase interaction. However, the use of graphene nano pellets in the composite's formulation significantly narrows the scope of its application due to the increased cost component. Given this, further relevant studies do not seem appropriate.

Paper [11] reports the research results that show the high resistance of epoxy basalt against alkaline environments, it is indicated that they are cheaper than carbon and aramid plastics. It is demonstrated that the properties of basalt- and fiberglass are sometimes similar, for example, the curves of Differential Scanning Calorimetry and the glass transition temperature [11]. The authors found that basalt plastics have a higher elasticity module and better strength at bending than similar fiberglass. However, there are unresolved issues related to testing the samples of different geometric shapes. The reason for this may be the purpose of the cited paper – to determine the interlayer shift of composite annular samples. Such a statement does not reveal the effect of alkaline environments on samples executed in a different shape. The option to overcome the difficulties may be the manufacture of flat-shaped samples as part of a more complex geometric object. This is the approach used in works [12–24]. Study [9] shows the possibilities to strengthen the epoxy polymer with nano-basalt fiber, in the amount between 15 and 80 % by weight. It is shown that the most effective strengthening is observed in the interval of 50–80 % by weight, whereby the composite changes the microstructure significantly. Composites with “dispersed basalt” (that is, containing fiber) were examined in [12]. It is shown that the growth of the size of basalt particles (their size was within 140 μm) leads to an increase in the degree of hardening, from 84 to 94 %, and at 90 °C – from 98.7 to 99.7 %. The optical-microscopic photographs of the compositions are given, which show the even distribution of basalt particles. However, the temperature of degradation almost does not change at filling (from 200 to 210 °C), although the oxygen index grows from 19–26 without filling to 37. The authors of [13] studied the water- and lye resistance revealing the threshold of resistance of basalt plastic structures in northern reservoirs (16 years). After 1.5 months of aging in acidic, alcoholic, aquatic, and salt environments, the decrease in tensile strength was 5–10 % (worst of all in the alcohol environment). Papers [22, 23] examined the possibility and effectiveness of the use of dispersed basalt as a filler for epoxy resin and showed that the introduction of dispersed basalt had an impact on the structural formation processes of the epoxy composite. This influence is manifested in the increase in the viability of the composition – the duration of gel formation and curing processes increases from 30 to 32–75 minutes and from 70 to 72–95 minutes; the authors noted an increase in the physical and mechanical characteristics when introducing dispersed basalt in the amount of 30–50 % by weight. However, such studies do not give an idea of the properties of the same composites but containing micro- or nano-fibers. This suggests that it is appropriate to conduct a study on the chemical durability of composite formulations containing micronano fibers as a filler.

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3. The aim and objectives of the study

The aim of this study is to define the optimal ratio for the polymer-composite filled with basalt micronano fibers with increased resistant properties.

To accomplish the aim, the following tasks have been set:
- to determine the structure and morphology of basalt fiber and the polymer composite;
- to determine the fire resistance of the hardened composite; to derive a dependence between the amount of a filler and the level of fire resistance;
- to determine the mechanical characteristics of the material's developed formulations;
- to determine the wear resistance, as well as the adhesion of the composites to steel;
- to determine the chemical durability of the material's developed formulations.

4. Materials and research methods to study the effect of the micronobasalt fiber content as the main filler of the polymer composite

4.1. Examined materials

The samples were prepared on the base of ED20 resin and PEPA hardener (5:1), with 10 % dilution by ethyl alco-
hol (the commercially-available sample “Epoxy Glue”). The hardening of the formulations was carried out by mixing these components with micronanobasalt fiber (made in Ukraine, Zhytomyr region) without vacuuming.

4.2. Procedure for determining the samples’ properties

The structure of the filler particles was examined using a scanning electron microscope (SEM) JSM-35 JEOL (Japan).

Compression tests (GOST 4651-68) involved the samples in the form of a cylinder with a diameter of 6.5 mm and a height of 10–12 mm (at the LouisShopper press machine), manufactured at 25 °C and thermally-treated. The tests for adhesive detachment (GOST 14760-69) involved the glued metal “cones” with a diameter of 2.2 cm using the tested binder, at the installation “UMM-10 Armavir”. All rounding, including averaging, implies a larger value; the lowest 1–2 values are not taken into account (if they are greatly understated). The tests for adhesive shift (GOST 14760-59) were carried out at the installation ZIP DI-1. The bend tests (GOST) involved the samples measuring 12×60×2 mm on a base of 40 mm. The abrasion of the composites was measured based on a change in the mass of the sample after rubbing the sample with the sandpaper P180, over a 100-fold cycle at the diameter of the cycle path 5 cm.

The wear resistance was calculated from the following empirical formula

\[ T = \frac{\rho}{X \rho_0} \]  

(1)

where \( \rho \) is the ratio of the densities of the filled to unfilled polymers, \( X \) is the abrasion value.

In fluid resistance tests, the tablets (the size of 1×1×0.2 mm) were placed in the acetone/ethyl acetate mixture and 35 % hydrogen peroxide. A change in the mass was recorded gravimetrically as a percentage of the weight gain.

The changes in the mass, mass change rate, the thermal effect magnitudes in the heating of the samples were studied using the thermogravimetric analysis method employing a derivatograph of the Paulik-Paulik-Erdey system, made by MOM, brand-1500D, GOST 29127-91. The thermograms were acquired under the following mode: a batch of 100.3 mg; sensitivity, 100 mg; TG-500, DTG-500, DTA-250; heating rate, 10°/min. The tests with open fire were carried out by a gas burner.

5. Results of studying the indicators of properties of the examined formulations

5.1. Determining the structure and morphology of basalt fiber and the polymer-composite

The properties of composites are significantly dependent on the morphology and supramolecular structure. Introducing BF should lead to significant changes in the structure as the fiber-like filler particles would serve a reinforcing element. Around them are the regions of the polymer with compaction and the modified conformation of macromolecules. As the concentration grows, the number of new types of centers would increase, which are uncharacteristic of an unfilled polymer. These could include both strengthening structures (reinforcing, increasing the heat and chemical resistance) and defective or concentrating stresses. Some preliminary information can be provided by microscopy.

The acquired SEM-photographs show that BF is composed of fibers up to 60 μm long and 3–5 μm in diameter (Fig. 1, a, b), as well as their aggregates. The epoxy with BF in the amount of 50 % by weight is a heterogeneous mass containing the pores in addition to the fibers (Fig. 1, c).

5.2. Studying the fire resistance of composites

One can see that the addition of BF naturally increases the resistance against open fire, up to the formation of self-extinguishing composites (Table 1). This is a direct consequence of the non-combustibility of the filler and can be important for the creation of fire-safe materials.

The data acquired in determining the heat resistance of the samples are summarized in Table 2 and Fig. 2. One can see that the onset of irreversible noticeable destruction (characterized by a 5 % loss of the mass) in the examined epoxy polymers begins quite late – at 250 °C (according to other authors, for such systems – as early as 200 °C [22–24]. On
the one hand, this shows the advantage of the manufacturing technology used; on the other hand, it makes it difficult to simply increase heat resistance by filling (since it is more difficult to exceed the limit of 250 °C than that of 200 °C). However, the example of T°, the temperature of a 10 % loss of the mass, shows that this is possible – for the case of filling in the amount of 50 % by weight (Table 2, Fig. 2). In turn, at an average filling (in the amount of 15 % by weight), it is possible to increase the temperature of the maximum mass loss, which is important for the fire safety of articles made from such composites.

### Table 1

| Characteristic temperatures | 0 by weight (N-polymer) | 15 by weight | 50 by weight |
|-----------------------------|-------------------------|--------------|--------------|
| N-polymer (0 %)             |                         |              |              |
| 1-1.5 (easily ignites spontaneously) | 250 | 250 | 250 |
| 2-3 (ignites spontaneously) | 275 | 275 | 285 |
| 4-5 (difficult to ignite spontaneously) | 275/300 | 285/315 | 275/280 |
| 5-6 (self-extinguishing)   | | | |

### Table 2

| No. | Characteristic temperatures | 0 by weight (N-polymer) | 15 by weight | 50 by weight |
|-----|-----------------------------|-------------------------|--------------|--------------|
| 1   | 1° C, a 5 % loss of the mass | 450±1.9 (100 %)         | 13.7±0.3    | 13.7±0.4    |
| 2   | 2° C, a 10 % loss of the mass | 435±1.8 (97 %)         | 14.15±0.3   | 14.3±0.4    |
| 3   | DTG peak 1 (maximal mass losses)/DTA exo-peak corresponding to it | 440±1.9 (98 %)         | 14.3±0.3    | 14.3±0.4    |
| 4   | 3° C, exo-peak 2 of the combustion end | 495±2.0 (110 %)       | 14.05±0.3   | 14.1±0.4    |

### Table 3

| No. 1 | Compression C, all load values per column 7.5×10⁻³ mm | Cmean (% to C for N) | Module ×10⁴ Ecomp, kgs/cm² | Ymean Ecomp (%) for E for N |
|-------|------------------------------------------------------|----------------------|----------------------------|-----------------------------|
| H (0 %) | 450±1.9 (100 %)         | 12.25±0.2           | 12.3±0.3 (100 %)           |
| 15 %   | 435±1.8 (97 %)          | 14.15±0.3           | 14.3±0.4 (116 %)           |
| 50 %   | 440±1.9 (98 %)          | 13.7±0.3            | 13.7±0.4 (111 %)           |
| 80 %   | 495±2.0 (110 %)         | 14.05±0.3           | 14.1±0.4 (115 %)           |

### 5. Studying the mechanical strength of composites

In general, the destruction of epoxy samples by compression is usually accompanied by the passage of the load-deformation curve through two thresholds – the limit of elastic resistance (the limit of fluidity) and the limit of final destruction. The latter is visually characterized by the destruction of a sample into small parts, the collapse along straight cracks, barreling. An unfilled N-sample usually collapses this way. Often, a compressed N-sample can “squeeze in a barrel” – “to barrel.” When filled with basalt fiber, the sample should become more elastic, and this is observed by the growth in the E elasticity module (Table 3). At the same time, the limit of elasticity (fluidity) is not observed explicitly (or is seen as a small threshold on the curve) because further loading does not lead to the plastic deformation of the sample; the final destruction eventually occurs. Unlike the more plastic N-sample (or low-filled compositions), the final destruction proceeds along the bottom-up diagonal by Chernov-Luders that shift the planes, or along the longitudinal cracks.

The introduction of composites is a positive quality of the material – the loaded component retains the predefined volume until the load reaches the limit of strength, after which a crack is formed. However, a composite material, depending on a filler, can be plastic as well.

### 5.4. Studying the abrasion resistance and the adhesion of composites

It was found that filling in the amount of 15 % by weight increases the adhesion at detachment by almost 1.5 times, and in the amount of 50 % by weight – by 67 %. This can be explained by the high affinity of the basalt phase to a steel surface (Table 4).

The density of composites at small and medium filling content varies insignificantly. However, at 50, it increases markedly and especially markedly at 80 % by weight (Table 4).
5.5. Studying the chemical durability of the developed composite formulations

The resistance of the composite material against swelling in aggressive environments was tested in the acetone-ethyl acetate mixture. The aging in the mixture lasted 5 days. The result is shown in Fig. 3.

Note that acetone and its solutions are very aggressive environments for polyepoxides, as is known [25] from studies [26, 27]. Since acetone is found in many industrial and household solvents, the issue of increasing the durability of composites in it is very important. It is equally important to know the limits of resistance in oxidizing (for example, peroxides) and acidic environments. The affordable and used 35 % hydrogen peroxide (Fig. 4) and nitric acid (Fig. 5) are good for modeling. Continuous fiberglass (CFG) and continuous basalt fiber (CBF) were further considered as the material for our study.

The need to study the durability of a formulation in seawater is obvious due to the use of epoxides in shipbuilding and construction as compounds and coatings (Table 5).

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(estimated based on the temperature of a 10 % loss of the mass) does not change at 15 % filling but grows somewhat for BF in the amount of 50 % by weight (Table 2, line 2). However, the temperature, maximum weight loss, and exo-effect increase when filling with BF in the amount of 15 % by weight. This indicates the formation of more heat-resistant polymer-composite structures over this filling interval. In addition, the derivatograms show that with the increase in filling by up to 50 % by weight, the character of the manifestation of the exo-effects changes significantly, which reflects changes in the structure of the composite.

The introduction of microbasalt can increase strength at compression to 10 % (with a measurement error less than 5 %), and only at a very high filling in the amount of 80 % by weight (Table 3, No. 1). True, after a week of aging in water, the basalt-composites were much more stable than a conventional polymer. The strengthening effect of microbasalt is expressed in an increase in the compression load of a composite aged in water and its elastic modulus up to 6–12 %. A slight increase in the modulus of elasticity is also observed for the N-polymer aged in water (Table 3, series No. 1a).

The growth in the elasticity module \( E_{\text{comp}} \) when the epoxy basals are compressed (as opposed to load/strength \( C \)) becomes noticeable even with a slight filling (15 % by weight). It was expected that at higher fillings in water there would be a further increase in the module but a given experiment did not reveal it (Table 3, No. 1). It is characteristic that, for compression, simply introducing basalt roving into the resin does not produce such a noticeable effect (in contrast to glass roving, Table 3, No. 2).

The drop in strength at bending (by about 2 times) after filling is a trend typical of almost any epoxy resin filler. Basalt fiber was no exception (Table 3, No. 3). The natural exception is only the samples with basalt roving, which increase strength at bending (Table 3). However, at high fillings (but not at 15 % by weight), there is an almost twofold growth of the module at bending, higher than that for the composite with roving (Table 3, No. 3), which is very important from a practical point of view.

The swell graph (Fig. 3) in acetone-ethyl acetate shows that the unfilled polymer swells very actively in the first days and, after 2 days, reveals the signs of physical destruction (decay into parts). As a result, the swelling is replaced by reverse processes of the mass loss, which can be observed in Fig. 3 where the curves of the unfilled samples take similar shapes (the difference in swelling is due to differences in the sample masses). Filling with basalt in the amount of 15 % by weight retains the likeness of a swelling curve to the curve for \( N1 \) and \( N2 \) (unfilled), severely distorting its shape towards smoothing the swelling and loss of mass processes. The sample with 15 % by weight also deconstructs, albeit after 5 days.

The filling with micro basalt reduces the rate and degree of swelling in 35 % \( H_2O_2 \) (Fig. 4) – the more active the higher the percentage of filling. Visually, they demonstrate the signs of oxidation by peroxide (white) but no significant destruction (as in acetone) is detected. As the curves demonstrate, in this case, the filling in the amount of 15 % by weight (insignificant) does not lead to a drastic change in the nature of swelling, which indicates the preservation of the structure of the polymer as a whole. The same cannot be said for composites with a high degree of filling, in the amount of 50 and 80 % by weight, whose swelling patterns are different than those for the unfilled and low-filled samples. This can be explained by the compaction of the structure of the composite (with a decrease in the number of pores and permeable areas) and its increasing resistance against aggressive effects due to the increase in the proportion of inorganic phases.

However, the resistance to swelling in nitric acid after filling with BF, in the amount of 50–80 % by weight, decreases (Fig. 5), which is probably due to the nature of basalt. It can be assumed that the porosity would increase after the filling with a medium amount (15 % by weight), which facilitates the penetration of small acid molecules into the structure of the composite. At higher fillings, the swelling decreases but remains above the level of the N-polymer, Fig. 5), which may be due to compaction of the composite structure.

The resulting data also show (Table 5) that in seawater the composites with BF do not actually swell (as well as an unfilled polymer) although the composites of epoxy polymer with glass- and basalt fiber tend to swell.

Further study of the resulting formulations implies the use of computer-based universal testing equipment applying the combinations of composite formulations. This approach was implemented with the use of laminates based on the basalt fiber reinforced with tungsten [28], as well as with the use of fly ash [29]. Such methods are promising.

### 7. Conclusions

1. The introduction of basalt fiber into epoxy resin is possible over a very wide concentration interval – up to 80 % by weight, with the formation of viscose black masses, cured normally. The high filling significantly increases the density of the composites (by 2.2 times for 80 % by weight).

2. The cured composites have increased fire resistance, growing with the growth of filling. At the same time, however, the method of thermogravimetry did not reveal the effect of a basalt filler on the nature of thermo-oxidative destruction.

3. The introduction of BF provides an opportunity to increase strength at compression (within 10 %) and the elasticity module at compression (by 10–20 %). Moreover, basalt composites are insensitive to aging in water, in contrast to unfilled, which reduces the strength after aging. The highly filled composites (BF in the amount of 50–80 % by weight) are characterized by a very noticeable increase in the elastic modulus at bending (by almost twofold) although the bending strength itself is also proportionally reduced.

4. It has been found that filling in the amount of 15 % by weight increases the adhesion at detachment by 31 %, and in the amount of 50 % by weight – by 67 %, while the resistance against abrasion – by 12 % and 24 %, respectively.

5. The resistance in acetone and ethyl acetate at high fillings increases significantly. At 50–80 wt %, the sample does not degrade in these solvents - in contrast to unfilled and 15 wt % filled composites. The filling with microbasalt reduces the rate and degree of swelling in 35 % \( H_2O_2 \) – the more active the higher the percentage of filling.
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