



Article Nano- and Micro-Modification of Building Reinforcing Bars of Various Types

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Abstract: Fiber-reinforced plastic (FRP) rebar has drawbacks that can limit its scope, such as poor heat resistance, decrease its strength over time, and under the influence of substances with an alkaline medium, as well as the drawback of a low modulus of elasticity and deformation. Thus, the aim of the article is the nano- and micro-modification of building reinforcing bars using FRP rebars made of basalt fibers, which were impregnated with a thermosetting polymer binder with micro- or nanoparticles. The research discusses the major results of the developed composite reinforcement with the addition of micro- and nanosized particles. The microstructure of FRP has been studied using scanning electron microscopy. It was revealed that dispersion-strengthened polymer composites with the inclusion of microsilica (SiO₂) and nanosized aluminum oxide (Al₂O₃) particles have a much higher modulus of elasticity and strength when compared with the original polymer materials. In the course of the experiment, we also studied the retained plastic properties that are characterized by the absence of fragility. However, it was found that the high strength of materials was attained with a particle size of 10–500 nm, evenly distributed in the matrix, with an average distance between particles of 100-500 nm. It was also exhibited that composite reinforcement had improved the adhesion characteristics in comparison with both steel reinforcement (1.5-2 times, depending on the diameter), and with traditional unmodified FRP rebar (about 1.5 times). Thus, the use of micro-/nanosized powders increased the limit of the possible temperature range for the use and application of polymeric materials by almost two times, up to 286–320 °C, which will undoubtedly expand the range of the technological applications of products made of these materials.

Keywords: composite rebar; polymeric materials; aluminum oxide; microsilica; nanoparticles; thermomechanical properties of materials

1. Introduction

Reinforced concrete is the main building material that is used for the construction of a wide range of structures up to the most important ones [1,2]. In the vast majority of cases, steel reinforcement is used [3,4]. However, with all its advantages, steel reinforcement has a number of disadvantages, which include high weight [5,6], corrosion [7,8], electrical



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). conductivity [9,10], high thermal expansion [11,12], difficulty in bending during installation [13], and a small size range [14]. Fiber-reinforced plastic (FRP) rebar can be a good solution along this path [15].

The material for the manufacture of FRP is carbon, basalt, aramid, or glass. Fibers of raw materials are interconnected into a single rod, on which an additional rib is wound. The standard diameter of the composite rebar ranges from 4 to 24 mm.

It is known that composite reinforcement has a number of indisputable advantages, which include its low weight [16], high strength characteristics [17,18], and low susceptibility to corrosion [19]. Compared to steel, FRP is eight times lighter, which leads to a decrease in the total weight of the structure and the load on the foundation, as well as savings in transportation. FRP rebars have tensile strength several times higher than that of steel one, i.e., 1000 MPa versus 390 MPa, respectively. Smaller FRP can be used instead of steel, resulting in significant savings. The service life of FRP rebars is over 80 years, due to resistance to alkalis, acids, and corrosion. FRP does not interfere with electromagnetic waves and radio signals. The thermal conductivity coefficient of steel reinforcement is $46 \text{ W}/(\text{m}^{\circ}\text{C})$, but the coefficient of FRP is $0.35 \text{ W}/(\text{m}^{\circ}\text{C})$, that is, 100 times lower, which allows for avoiding cold bridges. FRP and concrete react in the same way to temperature cycling, which prevents stress and cracking inside the concrete structure.

However, at the same time, composite reinforcement has its drawbacks, which can limit the scope of its application, including weak heat resistance [20], a decrease in strength over time and under the influence of substances with an alkaline medium [21], as well as a low modulus of elasticity and deformation [22,23]. The modulus of elasticity of FRP is four times less than that of steel. The temperature limit at which it does not lose its physical and mechanical properties is also low, at only 200 °C.

A promising solution to these problems can be the use of micro- and nanoparticles in dispersion-strengthened polymer composite materials, in the matrix or in an impregnating composition, in which the authors included reinforcing elements in the form of specially introduced micro- and nanosized particles. As nanofillers for dispersion-strengthened FRP rebars, one can consider the use of nanotubes, fullerenes, and nanoplates. However, as the literature analysis shows, the use of various types of nanomaterials does not significantly affect the thermomechanical properties of FRP rebars.

It should be noted that dispersion-strengthened composite materials can be obtained on the basis of most materials used in construction. The most widespread are alloys based on aluminum—sintered aluminum powder (SAP), which consists of aluminum and dispersed flakes of aluminum oxide (Al_2O_3), was used in the experiment. However, in the future, applications for alloys such as VDU-1 (nickel, strengthened with hafnium dioxide) and VD-3 (matrix Ni + 20% Cr, strengthened with thorium oxide) deserve consideration. These alloys have a fairly high degree of heat resistance, which is of interest to the authors [24–26], who set one of the main goals as improving this indicator of the starting material.

In addition, the possibility of using some wastes of metallurgical production, such as corundum powder and silica fume, which has a low cost and high thermomechanical properties, deserves further attention [27]. Because of the optimally selected distribution of inclusions, a significant improvement in the properties of the starting material, and accordingly, the products obtained from it, can be achieved [6].

In addition, the possibility of using corundum powder consisting of crystalline aluminum oxide obtained from aluminum melting in a crucible furnace is of scientific interest. At the same time, corundum is distinguished by its purity, increased hardness, and regularity of forms, and as practice shows [28], it is not toxic, does not contain substances harmful to health, does not cause silicosis, and is not hygroscopic. The use of silica fume, a waste product from the production of silicon-containing alloys, can be just as effective; its low cost is especially important, according to the authors, for the profitability of the production and marketing process in the future [29]. Silica fume is a highly reactive pozzolan that has a hardening effect on the material. During the melting of the charge and the reduction of quartz (at a temperature of about 1900 °C), gaseous silicon is formed. Upon cooling and further contact with air, silicon oxidizes and then condenses, representing ultrafine particles with a high proportion of amorphous silica. Studies by the authors of [30–32] have shown that silica fume differs from other mineral additives in a relatively small particle size (from 0.1 to 0.5 microns) and a high-specific surface area. According to Reference [33], silica fume binds the matrix of the material more intensively than other mineral additives such as zeolite tuff, blast furnace, and boiler slag.

Thus, the purpose of the article is nano- and micro-modification of building reinforcing bars.

2. Materials and Methods

2.1. Materials

The objects of the research are FRP rebars made of basalt fibers, which are impregnated with a thermosetting polymer binder with micro- or nanoparticles (Figure 1). When planning the composition of raw materials for the experiment, the authors proceeded from the fact that the following components are included in the nanocomposite reinforcement: resin, an accelerator, a hardener, a thread, basalt fiber, and micro- or nanodispersed powder.



Figure 1. Cross-sectional diagrams of fiber-reinforced plastic (FRP) rebars with inclusions of various components: (**a**) microsilica (SiO₂), (**b**) nanoparticles of aluminum oxide (Al₂O₃), (**c**) micro-corundum, 1—basalt fiber, 2—binder (epoxy resin), 3—microsilica, 4—nanoparticles of Al₂O₃, 5—microcorundum.

Research was carried out on composite basalt-plastic FRP rebars with a diameter d equal to 8 and 10 mm, with inclusions in the matrix of nanoparticles of aluminum oxide (Al_2O_3) with particle sizes in the range of 5–60 nm, as well as with spraying along the outer diameter of the rod, and baking a mixture of microcorundum and microsilica (Figure 2).



Figure 2. Materials under investigation: **1**—reinforcement bar diameter (d) = 10 made of basaltplastic mixture; **2**—same d = 8 mm; **3**—traditional reinforcement bar d = 8 mm made of fiberglass, with two-start coiling (transverse ribs).

At the same time, the pH value of the aqueous suspension of microsilica averaged 7.74, and its bulk density was $0.16-0.21 \text{ t/m}^3$ in the unconsolidated state, and $0.39-0.71 \text{ t/m}^3$ in the compacted state. These values of the bulk density make it difficult to transport and use this material during research and production. The expediency of using microsilica during the experiment is explained by its unique ability to have a beneficial effect on increasing strength, frost resistance, chemical resistance, sulfate resistance, and wear resistance. Because of these abilities, the resulted composite should resist technogenic influences for a long time.

At the same time, dispersion-strengthened FRP rebars include a polymer matrix in which the specified nanoparticles of aluminum oxide (Al_2O_3) are distributed. Polymethyl methacrylate (otherwise called organic glass or acrylic resin) was used as a polymer matrix. To assess the significance of the results obtained, a comparison was made with concrete of the developed FRP rebars, both with traditional unmodified FRP rebars and steel reinforcement bars.

2.2. Methods

Experimental studies to determine the structure of the material were carried out using a Zeiss Sigma scanning electron microscope (Figure 3a, Table 1). At the same time, the tensile strength, modulus of elasticity (deformation), and the adhesion of the composite reinforcement to concrete were determined using an Instron 8802 dynamic testing machine and an Epsilon extensometer (Figure 3b,c, Table 1).



Figure 3. Equipment used in the experimental research: (a) Zeiss Sigma scanning electron microscope, (b) Instron 8802 dynamic testing machine, and (c) Epsilon longitudinal strain sensor (extensometer).

In the course of the experiment to determine the ultimate strength and elastic modulus in samples of basalt-plastic reinforcement (Figure 4), corundum aluminum oxide (Al_2O_3) and microsilica (SiO_2) were used.



Figure 4. Testing of the specimens of basalt-plastic reinforcement bars d = 10 mm: (**a**) test specimens, and (**b**) testing machine with a specimen.

Characteristics	Value			
Zeiss Sigma scanning electron microscope				
Full zoom range without distortion	×12-×1,000,000			
Spatial resolution	20 kV at 1.3 nm, 1.5 kV at 15 nm, and 1 kV at			
	2.8 nm			
Graphics Resolution	3072×2304 pixels			
Electron source	field emission (thermionic type)			
Working chamber dimensions:	diameter 365 mm, height 270 mm			
Instron 8802 dynamic testing machine				
Type of drive	hydraulic			
Piston stroke	150 mm			
Maximum frequency of cyclic tests of samples	up to 100 Hz (limited by vibration amplitude)			
in tension, compression, and bending				
Maximum developed force	100 kN			
Load measurement error	$\pm 0.5\%$ of measured value			
Epsilon longitudinal strain sensor (extensometer)				
Туре	Suspended, axial			
Experienced deformations	Tension, compression, cyclic testing			
Base measurement length	3–600 mm			
Measurement range	5%-100%			

Table 1. Main technical characteristics of the equipment.

Testing of the adhesion of reinforcement to mortar was carried out using an Instron 8802 dynamic testing machine, in a movable frame into which a mortar cube of $100 \times 100 \times 100$ mm was fixed (Figure 5). Because of the low strength of basalt-plastic reinforcement in the transverse direction, ISO 10406-1 recommends fixing the ends of the reinforcement in special anchors for axial tensile tests, thus preventing its destruction due to compression by the grips of a mechanical machine. To assess the adhesion of the reinforcing rod and concrete, the method of pulling reinforcement out of a concrete cube, the so-called Pull-Out test, was used. It is noted in various publications that this method gives a slightly overestimated result due to the presence of a noticeable hydrostatic component in the stressed state of concrete due to the influence of the base plate. However, ISO 10406-1 provides the exact Pull-Out test, and, in addition, most of the studies were carried out in this setting, so the authors of the paper used the Pull-Out test method.



Figure 5. Equipment and specimens for testing an adhesion of the basalt-plastic reinforcement bars d = 10 mm with a mortar cube $100 \times 100 \times 100 \text{ mm}$: (a) test specimens, and (b) test specimen in a movable frame of an Instron 8802 dynamic testing machine.

3. Results and Discussion

Production of the FRP Rebars

The inner core is made up of parallel fiberglass fibers, interconnected by a polymer resin. It should be noted that it is the inner fiberglass reinforcement bar that forms its

strength characteristics. The outer layer of the reinforcing bar made of fiberglass is made in the form of a bi-directional winding from the fibers of a composite material with additional dusting of a fine-grained abrasive powder.

Figure 6 shows that, under equal conditions, the elasticity (extensibility) of the composite reinforcement (curve 2) is 5–7 times lower than the traditional metal reinforcement (curve 1).



Figure 6. Dependence of tensile values on load: 1—metal rebars; 2—composite rebars, 3—composite rebars with the inclusion of micro-SiO₂ and nano-Al₂O₃.

Accordingly, the scope of FRP rebars is limited to applications in structures that primarily work in compression rather than bending. However, dispersion-strengthened polymer composites with the inclusion of micro-SiO₂ and nano-Al₂O₃ particles have a much higher modulus of elasticity and strength (capable of taking large loads), when compared with the original polymer materials. In the course of the experiment, it was confirmed that at the same time, they retain plastic properties and are characterized by the absence of fragility. High strength of the materials is provided with a particle size of 10–500 nm, evenly distributed in the matrix, with an average distance between particles of 100–500 nm.

The starting point for planning the experiment was the judgment that the effect of composites on the initial materials should be calculated, taking into account the critical ("ineffective") length, which is an important characteristic of the interaction of the composites. Modeling the processes in the composite, the authors proceeded from the assumption that the growth of stresses in the basalt-plastic fiber continues until the stress reaches the average level that is observed in the continuous fiber. The length l_c , at which this phenomenon is observed, is critical, or "ineffective", and is calculated by the formula:

$$\frac{l_c}{d_f} = \frac{\sigma_f}{2\tau_m} \tag{1}$$

where d_f and σ_f are the fiber diameter and strength, respectively, and τ_m is the yield stress of the matrix.

Microsilica, located in the matrix along the outer diameter of the rod, increases the density, strength, impermeability, and durability of the polymer composite (Figure 7a).

The structure of the basalt FRP rebar, where d = 10 mm, is shown in Figure 7b,c. From the analysis of Figure 7b,c, it was possible to determine the composition of the composite matrix, the elements of which are positioned as follows: 1—unreacted part of the basalt fiber with the liquid elements of the composite (brown); 2—reacted and hardened basalt fiber material (black); 3—Al₂O₃ nanoparticles in dimensions from 5 to 40 nm (white). A high-density monolithic nanostructure of a dispersion-strengthened matrix was noted, which provides improved performance characteristics compared to metal reinforcement as well as the unmodified FRP rebar.





An important place in the research process was given to the assessment of the stress state, where the average bond stress τ between the studied rebars and concrete was calculated using the following formula:

$$\tau = \frac{F}{C \times l} \tag{2}$$

where *F* is the tensile load, *C* is the equivalent circumference of the reinforcing bar, and *l* is a length of anchorage of rebar in concrete (d = 10 mm, Table 2).

It was found that composite rebar has improved adhesion characteristics in comparison with both steel bar (1.5–2 times, depending on the diameter) and traditional unmodified FRP rebar (about 1.5 times). The tensile strength of the developed modified rebar was almost three times higher than that of steel bar and almost 20% higher than that of traditional FRP rebar. However, the relative strain at breaking for steel is almost an order of magnitude higher than for FRP. Even so, modified FRPs showed an increase in this characteristic from 2.5% to 2.7%, which is a rise of about 8% compared to traditional FRPs. The same tendency was observed for the tensile modulus, the maximum values for steel (average 200 GPa) were four times lower for traditional FRP rebar and three times lower for modified FRP rebar.

Moreover additional pollination of the reinforcement surface with a mixture of microcorundum and microsilica led to an improvement in the adhesion of rebar to concrete, and accordingly, to an increase in the strength of the structure as a whole, which was confirmed by the conducted studies (Figure 8).

Rebar Sample	Tensile Strength, MPa	Elongation at Break, %	Tensile Modulus, MPa	Bond Stress, MPa
FRP rebar d = 8 mm	1160	2.5	55,800	9
FRP rebar d = 10 mm	1130	2.6	54,600	14
FRP-M rebar d = 8 mm	1349	2.7	63,800	14
FRP-M rebar d = 10 mm	1325	2.8	62,600	21
Steel bar d = 8 mm	395	24	203,000	8
Steel bar d = 10 mm	385	25	197,000	11

Table 2. Mechanical properties of the FRP rebars.



Figure 8. Diagram of adhesion of rebar to concrete of grade B35: 1—steel rebar; 2—fiberglass rebar; 3—basalt FRP rebar sprayed with a mix of basalt fiber and microsilica.

The operation of basalt-plastic materials based on polyester resins is possible within the range of 60–120 °C, epoxy resin is possible within the range of 80–140 °C, and phenolformaldehyde is possible within the range of 150–250 °C [34]. However, the inclusion of micro- and nanosized powders in the composition of the powders considered above increases this limit of the possible temperature range to 286–320 °C, which will undoubtedly expand the range of technological applications of products made of these materials. As the analysis shows, when materials are heated to 350–400 °C, the physical and mechanical properties are more stable for basalt plastics, which are based on organosilicon and polyamide binders (Figure 9).

Thus, it obtained composite materials reinforced with high-strength and high-modulus continuous fibers. The main load in them is perceived by the reinforcing elements, and the transfer of stresses occurs using a polymer matrix. In this case, the proposed composite materials had anisotropic properties; therefore, the mechanical and physical properties of the materials obtained were determined primarily by the properties of the fibers, and in addition, by their orientation, volumetric content, and the ability of the matrix to transfer the applied load to the fibers.



Figure 9. Dependence of the decrease in material strength on temperature: 1—glass fiber; 2—basalt fiber; 3—fiber with the addition of microparticles.

The materials obtained combine high-strength and dielectric properties. They have a fairly low density and thermal conductivity, and they are also characterized by high atmospheric, water, and chemical resistance. The highest strength and stiffness values include fiberglass plastics, which contain oriented continuous fibers that allow them to be positioned as unidirectional and cross-directional: in the first case, the fibers are located in parallel, and in the second case, at a given angle to each other, constant or variable across the product. At the same time, a change in the orientation of the fibers obviously leads to a change in the mechanical properties of fiberglass materials, as studied by the authors.

4. Conclusions

Fiber-reinforced plastic rebars are promising building reinforcing materials; however, they have their drawbacks that can limit their area of application, such as poor thermal shock resistance, a decrease in strength over time and under the influence of substances with an alkaline medium, as well as a low modulus of elasticity and deformation. The paper discusses the research results of the developed composite reinforcement with the addition of micro-SiO₂ and nano-Al₂O₃ particles. As a result of the comprehensive studies, the following findings were revealed:

- 1. It was established that dispersion-strengthened polymer composites with the inclusion of micro- and nanosized particles have a much higher modulus of elasticity and strength, when compared with the original polymer materials.
- 2. The retained plastic properties that are characterized by the absence of fragility were also studied.
- 3. The high strength of materials is provided with a particle size of 10–500 nm, uniformly distributed in the matrix, with an average distance between particles of 100–500 nm.
- 4. It was found that composite reinforcement has improved adhesion characteristics in comparison with both steel reinforcement (1.5–2 times, depending on the diameter), and with traditional unmodified FRP rebar (about 1.5 times).
- 5. The tensile strength of the developed modified rebar is almost three times higher than that of steel reinforcement and almost 20% higher than that of traditional FRP rebar.
- 6. Modified FRPs showed an increase in strain at breaking from 2.5%–2.7%, which is a rise of about 8% compared to traditional FRPs. The same tendency was also observed for the tensile modulus; the maximum values for steel (on average 200 GPa) were four times lower for traditional FRP rebar and three times lower for modified FRP rebar.
- 7. The use of micro-/nanosized powders can increase the limit of the possible temperature range of application of polymeric materials by almost two times, up to 286–320 °C.
- 8. It was also exhibited that when materials are heated to 350–400 °C, the physical and mechanical properties are more stable for basalt plastics, depending on the organosilicon and polyamide binders.

On the basis of the major findings of this study, we highlighted research points for further study and for extendable contributions by researchers worldwide. It should be noted that as a filler for the rebar under consideration, it is possible to use other high-strength dispersed materials that are industrial waste materials, including SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, and K₂O.

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