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On the Strength of the CF/Al-Wire Depending on the Fabrication Process Parameters: Melt Temperature, Time, Ultrasonic Power, and Thickness of Carbon Fiber Coating

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Abstract: The process of the production of a CF/Al-wire by pulling carbon fibers through an aluminum melt has at least 15 parameters. The main parameters include the power of ultrasonic treatment, the time of contact of the fiber with the matrix melt, and the melt temperature. In addition, the presence of a barrier coating on the fiber surface and its thickness significantly affects the properties of the resulting material. The importance of these parameters is due to their direct effect on the chemical interaction between the aluminum matrix and the carbon fiber. This interaction leads to the formation of aluminum carbide, a hygroscopic, brittle phase that ultimately significantly reduces the strength of such composites. In this regard, limiting a chemical reaction at the matrix/fiber interface in the production of CF/Al composites is one of the main technological problems. The main goal of this work is to pragmatically elucidate the effect of the above parameters on the strength of CF/Al composites. It is shown that the strength of a CF/Al-wire can reach 2000 MPa.

Keywords: carbon fiber/aluminum matrix composite; CF/Al-wire; carbon fiber coating

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1. Introduction

A composite with an aluminum matrix and carbon fiber is of interest from the point of view of replacing aluminum alloys. This is especially true for applications where, for some reason, aluminum structural members cannot be replaced with lightweight and durable CFRP. Such applications include structural components of the airframe of airplanes and other aircraft. In addition to the aviation and space industry, this material can be used in the electric power industry as a self-support cable core of an electric transmission line. Similar composite material is used today around the world under the trade name ACCR of the 3M company.

The most common method for producing composites with carbon fiber and an aluminum matrix is injection molding. However, in the opinion of the author of this work, the most technologically advanced method is drawing a carbon fiber filament through an aluminum melt subjected to ultrasonic treatment. This technological scheme permits controlling a larger number of process parameters, and the proposed method allows controlling an important parameter, such as the time of contact of the fiber with liquid aluminum over a wider range. This turns out to be crucial from the point of view of the mechanical properties of the composite.

The mentioned technological scheme allows obtaining a CF/Al-wire of an unlimited length. Such a composite wire can be used as an independent product (e.g., a self-support cable core) or as a raw material for additive technology or CF/Al prepregs for thixoforming technology.

The work of a scientific team from Japan [1–3] considered the effect of a large number of parameters on the course of the process of obtaining a composite wire and on its mechanical properties. However, the mentioned work does not consider the effect of important parameters such as melt temperature, ultrasonic treatment power, and carbon fiber barrier coating.

The importance of these parameters is due to their direct effect on the chemical interaction between the aluminum matrix and the carbon fiber. This interaction leads to the formation of aluminum carbide, a hygroscopic, brittle phase that ultimately significantly reduces the strength of such composites [4]. In this regard, limiting a chemical reaction at the matrix/fiber interface in the production of CF/Al composites is one of the main technological problems.

The main goal of this work is to pragmatically elucidate the effect of the above parameters on the strength of CF/Al composites. There is little doubt that this influence is carried out by controlling the chemical interaction between the matrix and the fiber. This results in the state of the interface, which is of great interest from the point of view of fundamental science. However, it is not the object of research in this work and will certainly be investigated later.

Let us consider the mentioned parameters in more detail. The carbon fiber barrier coating is one of the key factors affecting the strength of a CF/Al composite. For example, in [5], a fiber with a CVD coating made of pyrolytic carbon was used to reinforce an aluminum matrix. This allowed the authors to increase the tensile strength of the CF/Al composite from 710 to 1360 MPa; this is the highest strength of such a composite known in open sources. The authors of this work note that a pyrolytic carbon coating not only performs a barrier function, preventing the formation of aluminum carbide, but also plays the role of weak boundaries that ensure non-brittle fracture. Another example of increasing the strength of a CF/Al composite is shown in [6], where C/SiC/Si CVD laminated fiber was used. The tensile strength achieved by the authors was 1250 MPa.

The disadvantages of these coatings are the complexity and low manufacturability of the CVD process. The sol-gel method can be used as a more technologically advanced alternative that almost does not require specialized equipment and special conditions. The works [7,8] show the possibility of applying such coatings; however, the regulation of the thickness of such coatings within one cycle remained an unsolved problem until recently. The solution became possible due to the use of a method combining a sol-gel process and electrochemical deposition [9]. This method is used in the present work.

The work [10] demonstrates the effect of temperature and time on the chemical interaction between the carbon fiber and liquid aluminum. It is shown that the chemical reaction rate increases with an increasing temperature. Based on the data presented in the aforementioned work, it can be assumed that the chemical interaction in the temperature range up to 900 °C has an incubation period of about 10 s. The chemical interaction leads to the degradation of the fiber surface that obviously negatively affects the strength of a CF/Al composite.

In contrast to the other parameters mentioned, nothing is known about the effect of ultrasonic treatment power on the strength of a CF/Al composite in open sources. The effect of this parameter has been studied in this work for the first time.

2. Materials and Methods

As a reinforcing agent, we used a commercially available continuous thread of carbon fiber grade UMT40-3K-EP (manufactured by UMATEX Group, Moscow, Russia) thermally purified from the sizing in a vacuum at 400 °C for 15 min (according to ISO 11566:1996). According to the manufacturer, the tensile strength and tensile modulus of the carbon fiber are 4.0 GPa and 260 GPa, respectively. Commercially available aluminum alloys of Al-6Mg grades and commercially pure aluminum (99.3%) were used as a matrix material.

2.1. Coating Deposition

The SiO_2-coating was applied to the carbon fiber by the electrochemical sol-gel method, which is described in detail in [9]. Table 1 shows the parameters of the reaction medium and the mode of coating. Four types of coatings were used; they differed from each other in thickness that was 0.1, 0.3, 0.7, and 1 μm. Coatings of different thicknesses were obtained by varying the application time.

Table 1. Parameters of the reaction medium for coating.

C_{IPA}, % vol.	MR	C_{salt}, g/L	pH	J, mA/cm^2	τ, min
67	62	20	2.23	5.3	0.1–1.7

2.2. Production of CF/Al-Wire

A schematic of CF/Al-wire production is shown in Figure 1.

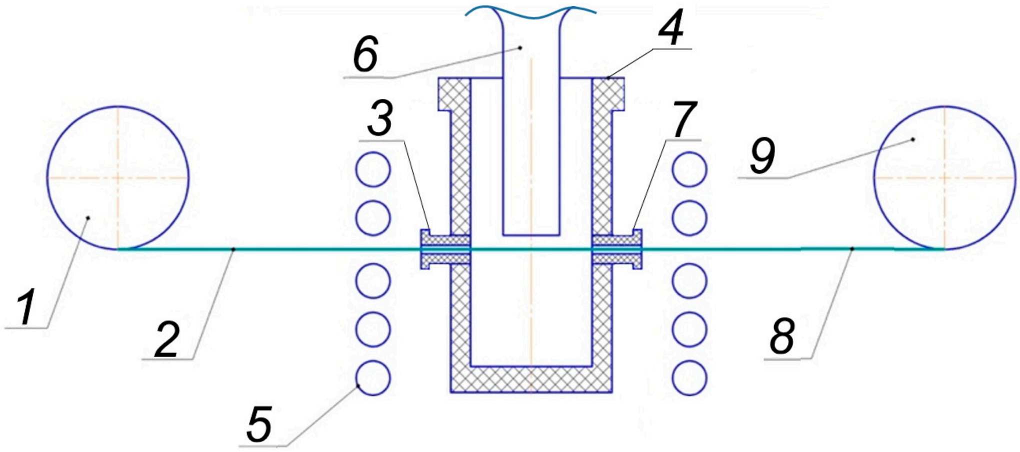


Figure 1. Scheme of CF/Al-wire production.

From release spool (1), carbon fiber thread (2) enters the matrix melt inside the crucible through the inlet die (3) in the wall of graphite crucible (4). The crucible is heated by induction heating (5). To ensure impregnation in the matrix melt, cavitation is created using niobium waveguide (6). Having passed through the outlet die (7) in the crucible wall, the fiber impregnated with the melt enters the cold region. After the melt has crystallized, the resulting composite wire (8) is wound onto receiving spool (9).

The time of the carbon fiber residence in the melt was controlled by adjusting the rotation speed of the stepper motor to the shaft of which the receiving coil was attached. Time control accuracy was not less than 0.1 s. The amplitude of the melt temperature fluctuations did not exceed 3 °C. The regulating thermocouple was located directly in the melt. Ultrasonic treatment of the melt was carried out by using the LUZD-1.5/1 system (manufactured by Kriamid LLC, Russia). The power of ultrasonic treatment at the end of the waveguide was estimated from the magnitude of the bias current on the emitter coil. The distance between the end of the waveguide and the carbon fiber filament was 5 mm.

2.3. Strength Tests

The strength of the wire was determined at three-point bending. The cross-sectional shape of the wire was taken to be ellipsoidal. The strength values were calculated by the formula (for ellipsoidal cylinders):

$$\sigma = [8FL]/[\pi ab^2], \quad (1)$$

where F is the load preceding failure—a is the major axis of the ellipse in the cross-section of the specimen, b is the minor axis of the ellipse in the cross-section of the specimen, and L is the distance between the supports. The dimensions of a and b did not exceed 1 mm, and the distance between the supports was 20 mm. The loading rate was 5 mm/min.

The effective fiber strength σ_{eff} was calculated as the ratio of the fiber strength in the composite (calculated from the rule of mixtures) to the initial fiber strength (4000 MPa according to the manufacturer's data). The bending strength of the matrix was taken to be 450 MPa (the highest strength of Al-6Mg alloy).

The tests were carried out under normal conditions. The force was measured using a H3-C3-100KG-3B-D41 strain gauge (manufactured by ZEMIC CO. LTD., Hanzhong, China), and the measurement error did not exceed 3 g. The transverse dimensions of the wire were measured using a Mitutoyo 293-240-30 0-25 mm/0.001 digital micrometer (Japan) with an accuracy of 1 µm.

The samples for each experimental point were carried out from a piece of a composite wire with a length of at least 1 m. The specified segment was tested for bending strength in different areas equidistant from each other. The distance between the adjacent load application points was at least 50 mm. Each experimental point resulted from at least 5 tests.

2.4. Scanning Electron Microscopy (SEM)

The fracture surfaces of the composite wire were investigated using a SUPRA 50VP (ZEISS, Oberkochen, Germany) high-resolution scanning electron microscope. The images were obtained in the mode of secondary electrons with an accelerating voltage of 10 kV at magnifications up to 50000×.

3. Results

3.1. Coating Effect

Preliminary experiments have shown that coated fibers are not melt impregnated at temperatures below 850 °C. In this case, impregnation occurs only at the highest ultrasonic power (2 W/mm^2). In this regard, the effect of the coating thickness was studied under these conditions. Al-6Mg alloy was used as a matrix.

Figure 2 shows the dependencies of the effect of the coating thickness and contact time on the strength of a CF/Al-wire.

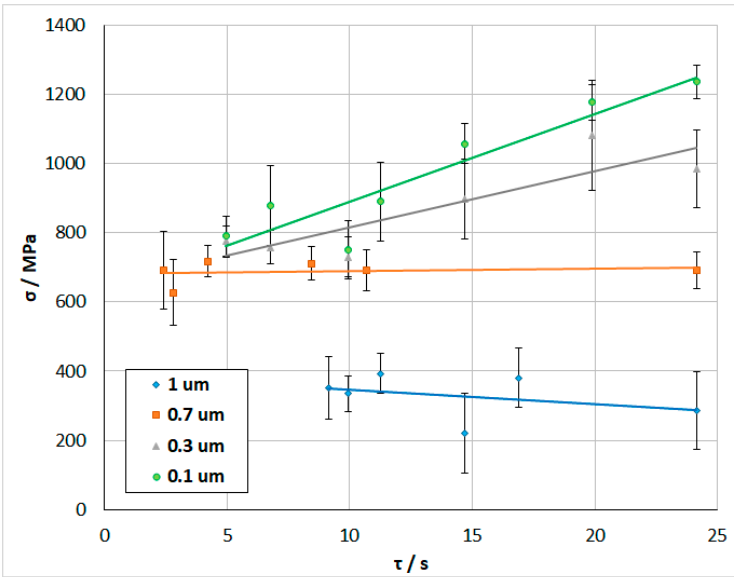


Figure 2. Effect of the coating thickness and contact time (τ) on the strength (σ) of a CF/Al-wire.

The CF/Al-wire reinforced carbon fibers with coatings with thicknesses of 1 and 0.7 µm have strengths not exceeding 500 and 800 MPa, respectively. In this case, the time of contact of the fiber with the melt almost does not affect the strength. With an increase in the contact time from 5 to almost 25 s, the strength of a wire reinforced with fibers with coatings with the thicknesses of 0.3 and 0.1 µm increases monotonically, reaching values of about 1000 and 1250 MPa, respectively.

The time of contact of the fiber with the melt is determined by the speed of drawing the fiber. In addition, with an increase in the drawing speed, an increase in the volume fraction of fiber in the composite wire occurs. Thus, the volume fraction of the fiber increases with a decreasing contact time.

To assess the contribution of fiber to the composite strength using simple mathematical calculations, the dependencies of the effective fiber strength on the thickness of the coating and fiber content in the composite were determined (Figure 3). These dependencies have a general tendency: with an increase in the volume fraction of the fiber, its effective strength decreases. However, note that with a coating thickness of 0.7 and 1 μm, a decrease in the effective fiber strength with an increase in the volume fraction occurs more slowly.

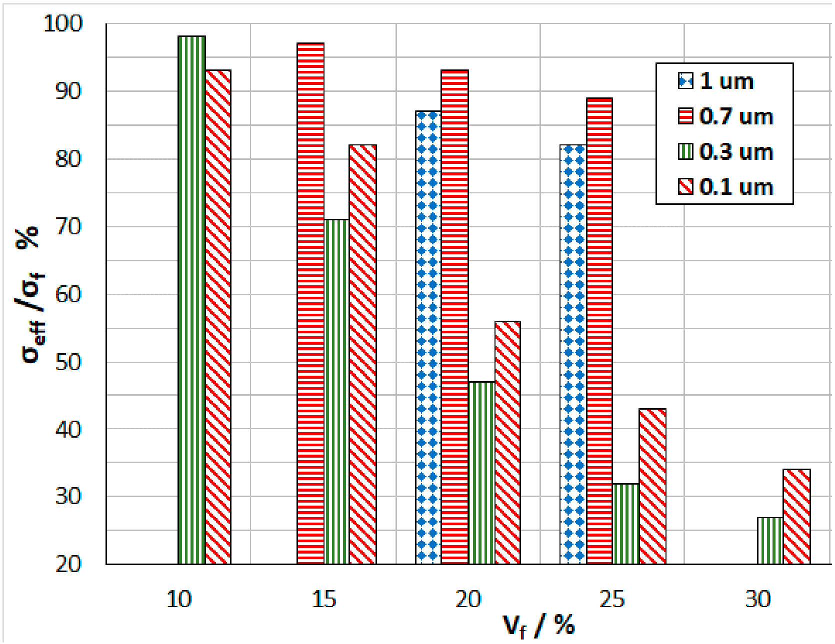
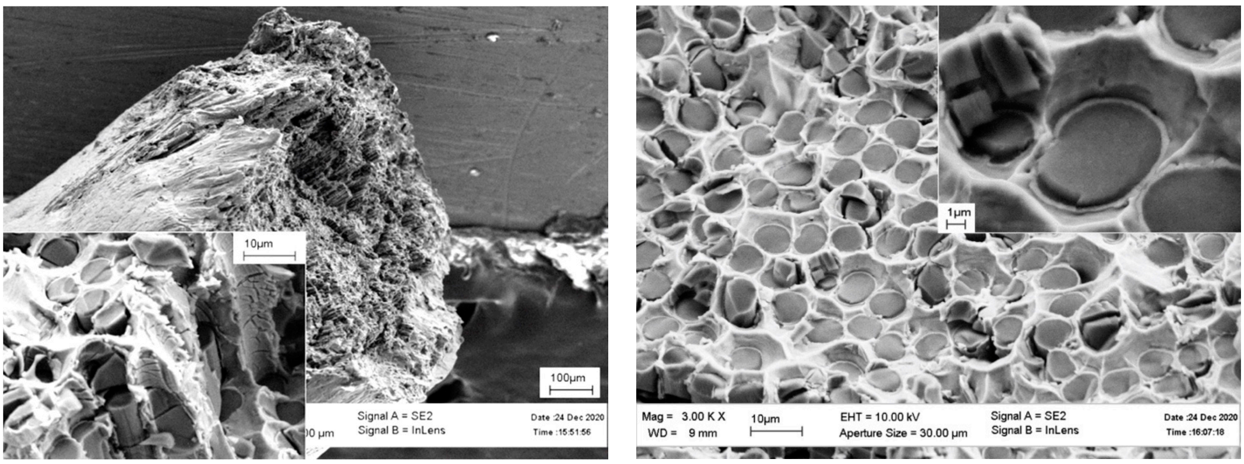


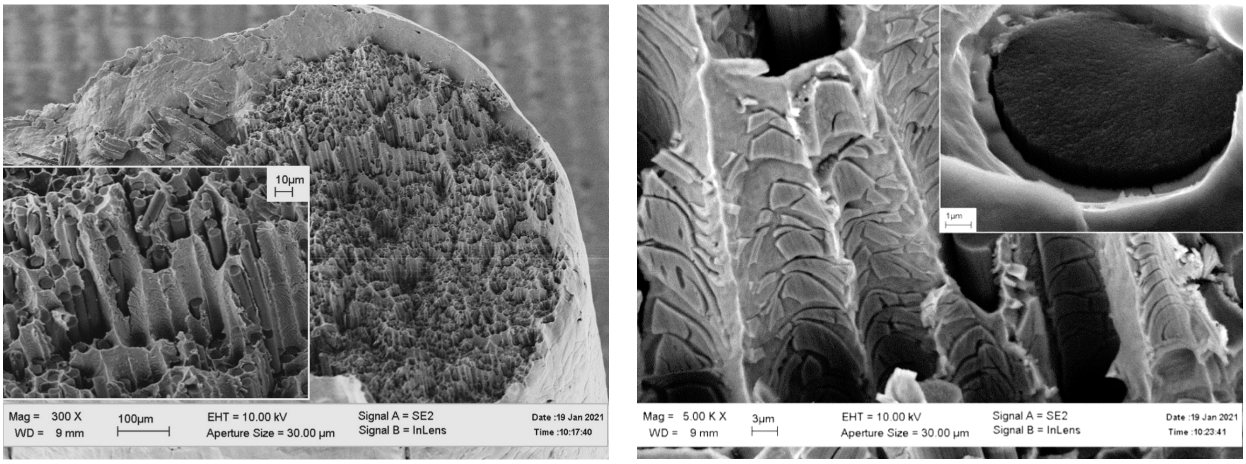
Figure 3. The tendency of changes in the effective strength (σ_{eff}) of the fiber depending on its volume fraction (V_f).

Figure 4 depicts SEM images of the fracture surfaces of a coated fiber-reinforced composite wire. The representative deformation curve is shown in Figure 5. The fracture surface has a developed relief. The deformation curve is nonlinear, which indicates a non-fragile nature of failure.



(**a**) (**b**)

Figure 4. *Cont.*



(**c**) (**d**)

Figure 4. SEM images of the fracture surfaces of a CF/Al-wire reinforced with fibers with coatings with the thicknesses of (**a**,**b**) 0.3 and (**c**,**d**) 0.7 μm.

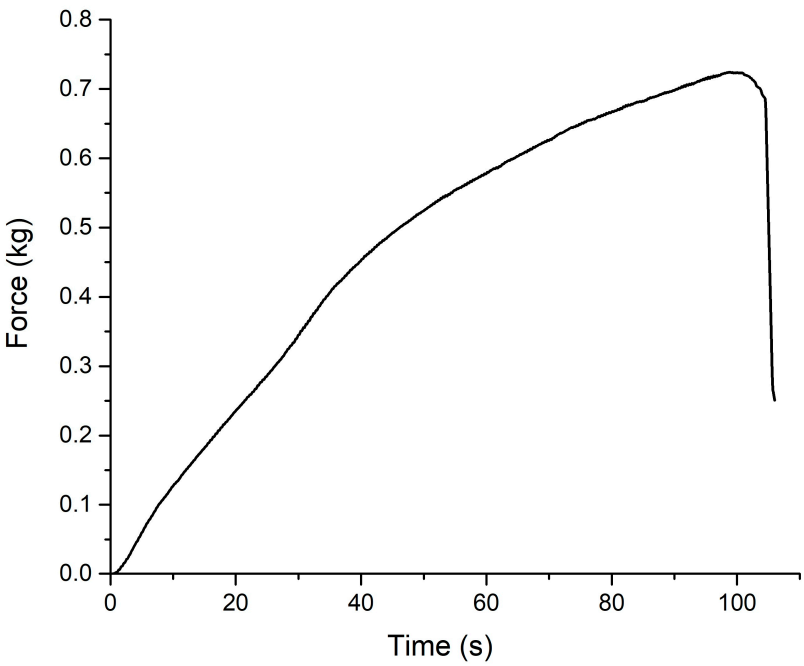


Figure 5. The representative deformation curve.

3.2. *The Effect of Melt Temperature*

Since the melt temperature and ultrasonic processing power when using coated fibers have significant limitations, the effect of these parameters was studied using uncoated fibers.

Figure 6 shows the dependencies of the effect of the melt temperature and contact time on the strength of a carbon-aluminum wire. Al-Mg6 alloy was used as a matrix, and the power of ultrasonic treatment was the highest (2 W/mm^2).

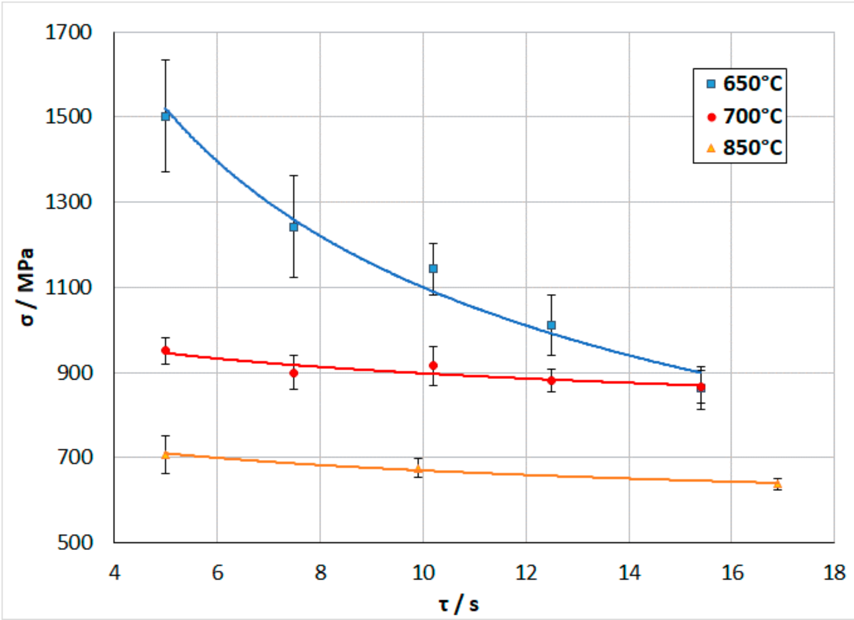


Figure 6. Dependencies of the strength (σ) of the CF/Al-wire reinforced with uncoated fiber on the melt temperature and the time of contact (τ) of the fiber with the melt.

The strength of the wire obtained at 850 °C decreases monotonically from 700 to 650 MPa with a change in the contact time from 5 to 17 s. A decrease in the temperature to 700 °C leads to an overall increase in the strength by about 200 MPa. With a decrease in the temperature to 650 °C, the slope of the curve becomes steeper, while the shortest contact time (5 s) corresponds to the highest average strength of 1500 MPa. A further reduction in the time of contact of the fiber with the melt was not achieved since at shorter times the fiber does not have time to be impregnated with the matrix melt.

3.3. Ultrasonic Treatment Power

Inverse dependence of the composite strength on the shear strength of the matrix is known from [11,12]. In particular, this dependence was demonstrated in [2,3] where it is shown that with an increase in the magnesium content in the matrix (that is, an increase in the shear strength of the matrix), the strength of the composite decreases. Pure aluminum has the lowest shear strength among aluminum alloys. In this regard, in addition to Al-Mg6 alloy, commercially available commercially pure aluminum was used as a matrix.

Figure 7 shows the dependencies of the strength of the carbon-aluminum wire on the power of ultrasonic treatment. The time of contact of the fiber with the melt was 10 s. The temperatures of Al-Mg6 and pure aluminum melts were 650 and 670 °C, respectively.

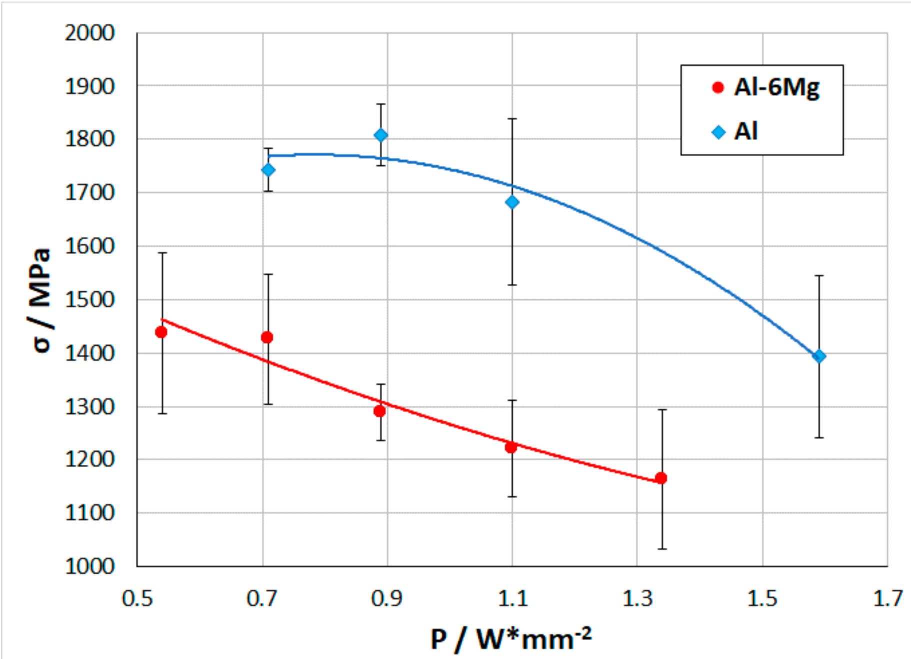


Figure 7. Dependencies of the strength (σ) of the CF/Al-wire on the power (P) of ultrasonic treatment.

With a decrease in the power of ultrasonic treatment to 0.7 and 0.9 W/mm^2 for Al-6Mg alloy and pure aluminum, the strength of the composite wire increases to 1430 and 1800 MPa, respectively. A further decrease in the power does not lead to a significant increase in the strength of the wire with an Al-6Mg matrix, and the strength of the wire with a pure aluminum matrix of decreases slightly to 1750 MPa.

3.4. Optimization of the Production Mode

To search for a possible maximum strength, samples of the carbon-aluminum wire with two matrix materials of Al-6Mg and pure aluminum were obtained at different times of contact of the fiber with the melt. Accordingly, for pure aluminum and Al-6Mg, the melt temperature was 650 and 670 °C. The power of ultrasonic treatment corresponded to the highest strength values and was 0.9 and 0.7 W/mm^2.

Figure 8 depicts the dependencies of the strength of the carbon-aluminum wire samples obtained according to the mode described above on the time of contact of the fiber with the melt.

With a decrease in the time of contact of the fiber with the melt from 10 to 5 s, the strength of the wire with an Al-6Mg matrix increases monotonically from 1430 to 1540 MPa. The strength dependence of the wire with a pure aluminum matrix appears to be extreme, with a maximum between 5 and 10 s. The highest recorded value of an average strength of the wire with a pure aluminum matrix corresponded to the contact time of 7.5 s and was 2025 ± 44 MPa. Note that the strength of some samples not shown in the graph due to an insufficient sample size reached 2150 MPa.

To further compare the results obtained for the wire reinforced with coated and uncoated fibers, note that in the latter case, the volume fraction of the fiber almost did not depend on the parameters under study and was in the range from 60 to 70%.

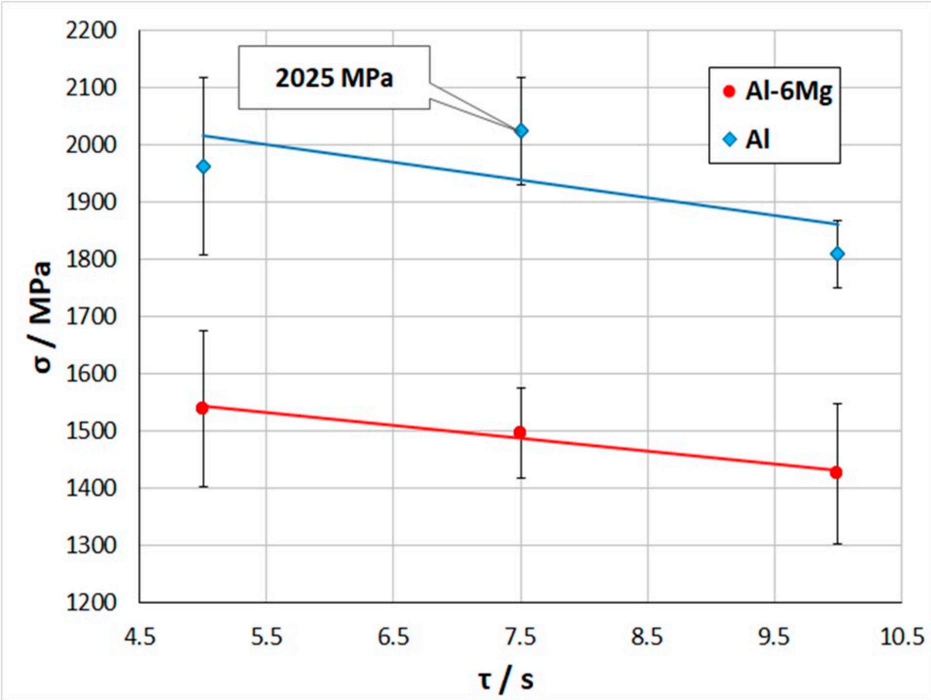


Figure 8. Dependencies of the strength (σ) of the CF/Al-wire on the time of contact (τ) of the fiber with the melt.

3.5. Comparison with World Analogs

Among the industrially available analogs of a CF/Al-wire, the closest is a composite wire of the American company 3M [13]. This wire is a long composite piece with an aluminum matrix reinforced with Nextel™ oxide fibers. The tensile strength of such a wire is 1378 MPa, the bending strength is 2085 MPa, and the density is 3.29 g/cm^3. Considering that the density of the CF/Al-wire does not exceed the density of aluminum (2.7 g/cm^3), the specific bending strengths of carbon-aluminum and 3M™ ACCR wires are ~798 $\times$ 10^3 and 633 $\times$ 10^3 m^2/s^2, respectively. Thus, the specific strength of a CF/Al-wire is no less than 25% higher than the closest industrial analog.

4. Discussion

4.1. The Effect of Carbon Fiber Coating

An increase in the volume fraction of the fiber with an increase in the drawing speed is most likely to be due to a decrease in the wire diameter like it occurs when growing a single crystal, the diameter of which decreases with an increase in the drawing speed [14]. A decrease in the wire diameter with a constant number of filaments in the bundle leads to an increase in the volume fraction of the fiber.

It is known [10] that an increase in the time of contact of carbon fiber with an aluminum melt leads to a more complete reaction of the formation of aluminum carbide that in turn worsens the composite strength. However, the strength of the CF/Al-wire reinforced with coated fiber (Figure 2) hardly changes or increases. This fact indirectly indicates good barrier properties of the coating used; however, it does not explain the strength dependence on the contact time.

The obtained dependencies can be explained by the inversely proportional relationship between the contact time and the volume fraction. It is known from [12] that at low volume fractions of fiber in composites with a metal matrix, the effective fiber strength is close to 100%. Considering the above, a longer contact time turns out to correspond to a lower volume fraction of the fiber. This ensures its greater effective strength (Figure 3) and, as a consequence, greater strength of the composite.

The general tendency of the dependence of the effective strength of coated fiber on the volume fraction is such that with an increase in the volume fraction of the fiber, its effective strength decreases; this is in good agreement with the concept of composites with a metal matrix [12]. Note that at a coating thickness of 0.7 and 1 μm, a decrease in the effective fiber strength occurs quite slowly; this gives reason to expect greater strength of the composite with a higher content of fiber with such coatings.

4.2. The Effect of Melt Temperature and Ultrasonic Treatment Power

The results obtained in this work on the effect of temperature, contact time, and ultrasonic treatment power are in good agreement with the concepts described in the introduction. An increase in the above parameters during the production of a CF/Al-wire leads to a decrease in its strength.

Dependencies of the strength of the CF/Al-wire (reinforced with uncoated wire) on the contact time, obtained at different temperatures, are in good agreement with the results in other works [3–5]. An increase in the contact time and temperature leads to a more complete chemical interaction between the matrix melt and the fiber, and that degrades the properties of the fiber and the composite as a whole. An increase in the power of ultrasonic treatment of the melt also leads to a decrease in the strength of the CF/Al-wire. Most likely, this is also associated with the intensification of the chemical interaction between the matrix melt and the fiber since ultrasonic treatment intensifies most chemical reactions. Thus, when reinforcing an aluminum matrix with uncoated fiber, the greatest strength can be obtained with the shortest contact time, a melt temperature close to the liquidus temperature, and the lowest ultrasonic treatment power sufficient for fiber impregnation.

5. Conclusions

1. The strength of the CF/Al-wire increases with a decreasing coating thickness and an increasing contact time; this indicates good barrier properties of the coating. The failure of the wire reinforced with coated fiber is not brittle.
2. An increase in the volume fraction of coated fiber leads to a decrease in its effective strength; this is in good agreement with the concept of composites with a metal matrix. At a coating thickness of 0.7 and 1 μm, a decrease in the effective fiber strength occurs quite slowly; this gives reason to expect greater strength of the composite with a higher content of fiber with such coatings.
3. The investigation of the effect of the melt temperature and ultrasonic treatment power in the production of the CF/Al-wire reinforced with uncoated fiber showed that the greatest strength can be obtained with the shortest contact time, a melt temperature close to the liquidus temperature, and the lowest ultrasonic treatment power sufficient for fiber impregnation.
4. Optimization of the production mode permitted achieving a CF/Al-wire strength of more than 2000 MPa at three-point bending.

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