Methods for developing carbon composites with functional properties

Vladimir Nelyub*

Bauman Moscow State Technical University
1053005, Moscow, 2-Baumanckya 5, Russia

* mail@emtc.ru

Abstract. The paper discusses the results of experimental analyses of carbon tape properties for FibArmTape-230/300 and LUP grades, and carbon composites based on the same. Metal coatings were applied to carbon tapes by magnetron sputtering. To apply metal coatings, two magnetron sputters were used: a laboratory MIR-2 and an industrial MMR-188M. Metal coatings were applied using stainless steel and copper targets. The values of breaking force under tension, relative elongation, and filament diameter before and after applying the metal coating, as well as the carbon composite interlayer shear strength, have been experimentally determined. The parameters of electrical conductivity, thermal conductivity, and shielding properties have been assessed. It has been found that applying a copper coating on carbon tapes allows to increase their thermal conductivity coefficient almost twice and decreases their heat capacity. The electrical resistance of metal-coated carbon tapes and carbon composites was lower than that of the same uncoated materials. The electrical conductivity of the LUP carbon tape, both with a metal coating of different thickness and without a coating, was higher than that of FibArm Tape-230/300 carbon tape. Metal coating on carbon tapes allows to improve significantly the carbon composites shielding properties.

1. Introduction

Carbon composites are widely used as construction materials in various industries, including aircraft industry, automotive industry, aerospace production in the manufacture of load-bearing elements which require a combination of high strength, rigidity, durability, and minimal weight [1, 2]. These materials have a set of unique mechanical and thermal properties, which provides their effective use as construction materials [3-5]. The main advantages of carbon composite structures are their low weight, high strength, rigidity, and wear resistance, as well as unique thermo-physical and tribological properties [6, 7]. Carbon composite applications are constantly expanding due to improved molding methods, which have allowed to significantly reduce their cost [8-10].

Heavy operating conditions of carbon composites products and constantly increasing demands to enhance their reliability make it necessary to improve the performance characteristics of existing materials [1, 3, 6]. Various methods are used to modify the properties of carbon fabrics: oxidation, dressing, galvanic processing, etc. A method of controlling the carbon-based composite properties is applying a metal coating [11, 12].

The main purpose of metal coating is to change the physicochemical properties of the carbon filler surface, which allows to control their reactivity, biocompatibility, thermophysical properties, etc. This paper uses the magnetron sputtering method, the essence of which is that the sprayed metal is initially...
converted to gaseous phase and then transferred to the fabric surface, where it turns into a solid material, forming a thin metal coating.

Applying various types of metal coatings on the carbon tape and fabric surfaces allows to obtain carbon composites with a new set of functional properties, that will significantly improve the product competitiveness. Thus, the authors of [13] found that carbon tapes coated with stainless steel have been effectively used at negative temperatures as reinforcing materials in the manufacture of repair bandages.

The purpose of this paper is to study such characteristics of carbon composites as their thermal conductivity, electrical conductivity and radio shielding properties.

2. Methodology
FibArmTape-230/300 and domestic (LUP) carbon tapes (Table 1) were used as objects of research. Laboratory and industrial magnetron sputters were used to apply metal coatings on carbon tapes (Figs. 1, 2, Table 2).

| Table 1. Carbon Tape Properties |
|---------------------------------|
| Properties | Carbon tape grade | LUP | FibArm Tape-230/300 |
| Regulatory documents | State Standard GOST 28006-88 | Technical Specifications TU 1916-018-61664530-2013 |
| Surface density (g/sq.m) | 170 | 230 |
| Thickness (mm) | 0.12 | 0.21 |
| Elastic modulus (GPa) | 260 | 230 |
| Poisson’s ratio | 0.3 |

When used for applying metal coatings, carbon tape sample (1), see Fig. 1, was placed on rotating table (2) in chamber (4). Using vacuum pumps (3), air was pumped out to pressure $5\times10^{-5}$ tor. Plasma gas (argon) was supplied from cylinder (5). Glowing discharge was ignited by applying voltage to the cathode of magnetron (7) from DC power supply unit (8). In the process of metal coating deposition
on the carbon tape, the discharge current, voltage, pressure, plasma gas flow rate, deposition time, distance from the target to the substrate were controlled.

![Diagram of UMN-1800N magnetron sputtering system](image)

**Figure 2.** Diagram of UMN-1800N magnetron sputtering system: 1 – vacuum chamber; 2 – diffusion pump; 3 – backing vacuum pump; 4 – tape rewinder; 5 – treated material; 6 – magnetron; 7 – magnetron power supply unit; 8 – argon feed system; 9 – gas feed system; 10 – power supply unit designed to control surface activation processes before applying a metal coating.

When using an industrial device (Fig. 2), deposition was carried out on a roll of carbon tape on both sides.

**Tables 2.** Characteristics of devices for applying metal coatings on carbon tape

| Parameters                        | Device models | MMR-1800M | MIR-2 |
|-----------------------------------|---------------|-----------|-------|
| Production type                   |               | Mass production | To-order production |
| Vacuum chamber capacity (cu. m)   | 15            | 0.7       |
| Discharge current of one magnetron (A) | 1 to 25      | 0.5 to 5  |
| Maximum fabric size (mm)          | Roll up to 1500 mm in diameter | 210×297 |
| Number of magnetrons              | 4             | 2         |
| Output capacity (sq.m per hour)   | 10            | 0.3       |

The following was used as targets: M1 grade copper and 12X18H10T grade stainless steel. All metal coatings were applied to carbon tapes from two sides with a thickness of 100 nm. Before applying metal coatings, carbon tape was treated with air plasma for 3 min on UPM-500 plasma activation device. The purpose of plasma activation was to remove adsorbed moisture and other gaseous products from carbon tapes, which allowed to increase the strength of the metal coating adhesion to the tape.

The mechanical properties of fibers (before and after applying metal coatings) were assessed on a Zwick/RoellZ010 tensile testing machine. To determine the average strength, relative elongation, and filament diameter, we tested 20 samples for each type of coating.

Carbon fiber molding was performed by the vacuum infusion method [10]. In the manufacture of carbon composites, we used the composition of epoxy-dian resin ED-20 as a binder and isomethyltetrahydrophthalic anhydride as a hardener. The choice of this binder was due to its good process properties and low cost [7].
The electrical resistance of the carbon tape was measured using a special instrument: a measuring cell. It consisted of a base member on which two rods made of aluminum alloy were fastened, with a diameter of 15 mm and a length of 70 mm. During the measurements, the electrical resistance of a square-shaped carbon tape sample was measured when voltage was applied to its two opposite sides. Aluminum alloy rods were installed in parallel and interconnected by means of a plate made of fluoroplastic (which was a dielectric). The distance between the rods was 60 mm. The width of the carbon tape samples, on which the measurements were made, was also 60 mm. Each of the rods was a contact of the measuring cell and was connected to the instrument (V7-22A), allowing to measure the electric resistance. The values obtained in such measurements characterize the electrical resistivity of a carbon tape with particular geometrical shape; therefore, this parameter is usually called “resistivity per square” and denoted as Ohm/square. In this paper, when describing the results of experimental studies, Ohm was used as unit of measurement for the sake of simplicity. With this test method, the electrical resistance measured value does not depend on the geometric dimensions of the carbon tape sample under investigation and is determined only by its length-to-width ratio.

The thermophysical properties of carbon composites were assessed by the method of differential scanning calorimetry on a DSC 204 F1 Phoenix instrument and laser flash method using an LFA 457 device.

The shielding properties of carbon composites were determined by measuring the electromagnetic waves transmission coefficient using a R2-57 instrument, a FCVU 1534100 selective voltmeter in the frequency range of 20 MHz to 1 GHz with a coaxial extender. The dynamic measurement range was at least 90 dB for the power relative to 1 µV.

3. Results and Discussion

Table 3 shows the average values of the filament mechanical properties before and after applying a stainless-steel coating on carbon tapes.

| Parameters                  | Metal coating type     | Parameter value | Dispersion |
|-----------------------------|------------------------|-----------------|------------|
| Breaking stress (MPa)       | FibArm Tape-230/300    |                 |            |
| Relative elongation (%)     | Without coating        | 3,110           | 870        |
| Diameter (nm)               |                        | 2.05            | 0.62       |
| Breaking stress (MPa)       | Stainless steel        | 8,660           | 260        |
| Relative elongation (%)     |                        | 4,640           | 620        |
| Diameter (µm)               |                        | 1.88            | 0.52       |
|                            |                        | 8,680           | 440        |

| Parameters                  | Metal coating type     | Parameter value | Dispersion |
|-----------------------------|------------------------|-----------------|------------|
| Breaking stress (MPa)       | LUP carbon tape        | 1,750           | 280        |
| Relative elongation (%)     | Without coating        | 0.8             | 0.2        |
| Diameter (nm)               |                        | 5,900           | 200        |
| Breaking stress (MPa)       | Stainless steel        | 1,850           | 300        |
| Relative elongation (%)     |                        | 0.75            | 0.1        |
| Diameter (nm)               |                        | 6,200           | 200        |

Table 4 shows the same filament parameters after applying a copper coating when using laboratory and industrial magnetron sputters.
Table 4. Average values of the filament mechanical properties of FibArmTape-230/300 copper-coated carbon tape when using a laboratory (MIR-2) and an industrial (MMR-1800M) magnetron sputters

| Parameters                  | Copper coating device | Parameter value | Dispersion |
|-----------------------------|-----------------------|-----------------|------------|
| Breaking stress (MPa)       | MMR-1800M             | 4,490           | 140        |
| Relative elongation (%)     | MIR-2                 | 1.9             | 0.5        |
| Diameter (nm)               |                       | 8,710           | 120        |
| Breaking stress (MPa)       | MIR-2                 | 4,430           | 680        |
| Relative elongation (%)     | MIR-2                 | 1.95            | 0.55       |
| Diameter (nm)               |                       | 8,630           | 490        |

Analysis of the results shows that the filament used to make FibArmTape-230/300 carbon tape is several times stronger than carbon LUP.

The measurement results of the specific surface resistance of stainless-steel and copper coated carbon tapes are given in Table 5. The higher the electrical resistance characteristics, the worse the electrical properties, i.e. the lower the electrical resistance, the higher the conductivity.

Table 5. Electrical resistivity of stainless-steel and copper coated carbon tapes

| Metal coating thickness (nm) | Surface resistance, Ohm, for carbon tape grades |
|-----------------------------|-----------------------------------------------|
|                             | LUP                | FibArm Tape-230|300           |
| Stainless-steel coating     |                   |                 |
| Without coating             | 1.2                | 2.9             |
| 30 to 50                    | 1.0                | 2.7             |
| 100                         | 0.9                | 2.5             |
| 200                         | 0.78               | 1.9             |
| Copper coating              |                   |                 |
| Without coating             | 1.2                | 2.9             |
| 30 to 50                    | 0.8                | 2.1             |
| 100                         | 0.75               | 1.9             |
| 200                         | 0.6                | 1.3             |

The accuracy of the instruments used to measure the electrical resistivity is relatively low and amounts to ± 0.1, which does not allow to precisely assess the difference between the metal coating thickness of a few tens of nm. However, it is possible to establish the general nature of the patterns for the two types of metal coatings under investigation: the greater the thickness of the metal coating, the lower the electrical resistance characteristics.

Therefore, the study showed that the electrical conductivity of LUP carbon tape, both without metal coating and with coating of different thickness, is higher than that of FibArm Tape-230/300 carbon tape. This is probably due to the fact that, on the surface of FibArm Tape-230/300 carbon tape, there was a dressing, which was probably not completely removed before applying the metal coating. Thus, the use of LUP carbon tape will allow to obtain carbon composites with higher values of electrical conductivity.

The values of thermal conductivity for carbon composites made using carbon tapes with copper coating, 100 nm thick, are given in Table 6. Table 7 shows the carbon composite interlayer shear strength.
Table 6. Carbon composite thermal conductivity values

| Material                                         | Thermal conductivity (W/m·K) |
|-------------------------------------------------|-----------------------------|
| Without metal coating                           | Copper coated               |
| Carbon composite, FibArm Tape-230|300 carbon tape             | 1.077                       | 2.14             |
| Carbon composite, LUP carbon tape               | 1.126                       | 2.16             |

Table 7. Carbon composite interlayer shear strength

| Metal coating type                           | Interlayer shear strength (MPa) |
|---------------------------------------------|---------------------------------|
|                                            | LUP                             | FibArm Tape-230|300 |
| Without metal coating                       | 46                              | 49              |
| Stainless steel, 12X18H10T grade            | 64                              | 54              |
| Copper                                      | 52                              | 69              |

The study showed that applying metal coatings on carbon tapes increases the carbon composite interlayer shear strength.

When using LUP carbon tape, applying a stainless steel or copper coating provided an increase in strength of 39% and 13%, respectively. For FibArm Tape-230|300 carbon tape, the greatest increase in strength (by 41%) was obtained using copper coating.

Carbon composite parts made of metal-coated carbon tapes are suggested to be used as shielding materials. To determine the frequency range in which the use of these materials is most effective, we found in our research the values of shielding coefficients in a wide range of wavelengths. Studies were carried out using a R2-57 instrument, which allows to measure the reflection coefficient. Electromagnetic waves of various frequencies were applied to a carbon composite sample; then, we measured its power after passing through the carbon composite. Table 8 presents the results for carbon composites based on carbon tape with and without metal coating. For the convenience of analyzing the results, shielding coefficients are given for different frequencies and wavelengths. Each value shown in Table 8 has been obtained by averaging the measurement results at five points with different sample positions. All studies were performed on 3mm thick samples made using vacuum infusion method and an epoxy binder and FibArm Tape-230|300 carbon tape. The position angle of all samples was 0°.

Table 8. Shielding coefficient measurement results

| Frequency (MHz) | Shielding coefficient for carbon composites based on carbon tapes (dB) |
|-----------------|-------------------------------------------------------------------------|
|                 | Without metal coating | Metal coated               |
| 30              | -4                     | -3                         |
| 100             | -4                     | -3                         |
| 200             | -4                     | -4                         |
| 500             | -5                     | -4                         |
| 1,000           | -8                     | -5                         |
| 2,000           | -10                    | -6                         |
| 3,000           | -20                    | -6                         |

The studies showed that, when increasing the incident electromagnetic wave frequency, there is an increase in shielding coefficient, the maximum values of which reach -20 dB at a frequency of 3,000.
The carbon tape metal coating provides the greatest effect at high frequencies of 2,000 MHz and higher, whereas a plain carbon composite slightly changes its properties with increasing frequency. The equipment used did not allow for testing at high frequencies; however, we can assume that the effect of applying a metal coating will increase. At frequencies below 30 MHz, the shielding effect is very low and there is no significant difference in the properties of carbon composites made of carbon tapes with and without metal coating.

4. Conclusions
The MIR-2 and MMR-1800M magnetron sputters used for our study allow to apply a thin layer of metal coating on carbon tape.

Studying the properties of carbon tapes with metal coatings and properties of carbon composites based on the same showed that they are substantially dependent on the properties of the coating materials. The relationship has been established between the elementary filament strength and the metal coating type. When applying stainless steel and copper coatings, the strength of filaments increased by more than 40%. The filament relative elongation for all types of coatings was lower than for the original one (i.e. without coating). If an industrial MMR-1800M device is used to apply carbon tape to the surface, there is a significant decrease in the dispersion values for all parameters, whereas average values vary slightly.

The use of metal-coated carbon tapes allows to obtain carbon composites with higher interlayer shear strength values.

The conductivity of carbon composites made of LUP carbon tape after applying a stainless-steel coating increased by 48%, and when applying a copper coating, by 72%. The electrical properties of carbon composites made using FibArm Tape-230|300 carbon tape have changed in a similar way.

The tests have shown the effectiveness of using copper-coated carbon tapes in the manufacture of parts that require special properties related to shielding electromagnetic waves.

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