The effect of processing in a SHF electromagnetic field on the moisture absorption of cured polymer composite materials

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Abstract. Comparative studies of moisture absorption by cured polymer composite materials (PCM) under conditions of being in a climate chamber for 7 days, previously subjected to short-term exposure to a SHF electromagnetic field with a frequency of 2.45 GHz, have been performed. It has been found that processing with an energy flux density \((17-18) \times 10^4 \text{ mW/cm}^2\) for 2 minutes contributes to a significant decrease in moisture absorption, which is expressed in a decrease in weight change after the presence of carbon fiber and fiberglass prototypes in the chamber compared with control samples by 16.7 and 25%, respectively. The reason for this result, determined on the basis of microstructure analysis, presumably lies in the reduction of the pore size and the increase in the relief of agglomerates, which increases the resistance to liquid penetration. Taking into account the previously established positive effect on improving the strength of PCM processed in a SHF electromagnetic field, the results obtained confirm the feasibility of using this method as a finishing technological operation in the production of PCM products.

The use of polymer composite materials (PCM) in the production of various products is constantly expanding, despite high cost indicators in comparison with metals and alloys. So, the cost of 1 kg of plastic based on PAN-fiber is according to some data 35.5-40 dollars., based on pitch fiber reached up to $ 300., while for steel, this figure is only $ 8.9. Despite this, the advantages of PCM reinforced with carbon fibers allow us to predict a steady growth in their use in the short and long term. In particular, according to Markets&Markets, the market for high-quality carbon fiber is forecast to grow from $ 3.9 billion 2019, up to $ 8.0 billion in 2026. The largest volume of consumption of carbon plastic in the world is accounted for the petrochemical industry, energy and aerospace equipment production. The use of PCM in automotive industry and shipbuilding is constantly expanding [1-4].

The performance requirements for PCM are of a paramount importance. The following requirements are applied to PCM and, in particular, to carbon fiber and fiberglass used in the production of aviation equipment: heat resistance, water and weather resistance, tensile strength along the fibers, compressive strength along the fibers, transversal (across the fibers) strength, interlayer shear, impact strength, and manufacturability [5, 6].

However, composite materials are characterized by a pronounced anisotropy of physico-mechanical characteristics, determined by the type and orientation of the reinforcing components [3, 6-8], which causes the need for additional reinforcement of the structure in some dangerous areas, leading to an increase in weight. This fact is highly undesirable for aerospace products, especially for high-maneuverable and high-speed objects and requires constant improvement of technologies for the
production of reinforcing components and matrix materials for PCM, as well as their reinforcement schemes. Carrying out these activities is a very complex and costly task. Analysis of the use of various chemical and physical technological methods to improve the functional and, in particular, strength properties of PCM shows that most of them have almost run their course. Improvement of existing technologies in relation to the processes of manufacturing components and forming PCM requires a serious re-equipment of production [9, 10]. Based on the above, the development of alternative technologies for strengthening modification of finally cured PCM products is an actual scientific and practical task.

Analysis of domestic and foreign scientific publications [11-15] shows that for local control effects on the structure and strength properties of a three-dimensional or two-dimensional object made of non-metallic materials, the most effective method is the use of a SHF electromagnetic field, the effect of which allows either to dramatically intensify the flow of thermal processes, or to form the required set of properties on the surface and in the volume of the material. At the same time, known applications of this electrical technology affect intermediate operations of the technological cycle – synthesis and modification of components, matrix curing. In this case, the achieved effects are influenced by technological heredity (subsequent molding, dimensional processing and assembly operations), which reduces the predictability of results.

We carried out the research on the possibility of improving the performance characteristics of blanks and parts made of composite carbon fiber and fiberglass by using a SHF electromagnetic field with a frequency of 2450 MHz to impact on the finally cured PCM for 1-2 minutes. It was found that such processing provides, in particular, an increase in the cutoff limit stresses by an average of 40%, bending by 7-13%, and interlayer shear by 14-16% [16-19]. The achieved results were obtained on samples located in the room immediately after the impact of SHF electromagnetic field or a limited time (several days) later.

At the same time, developers of composite materials and designers are facing the task of ensuring the preservation of the characteristics of materials during long-term use of products in various climatic conditions. This is due to the fact that the perception of external loads by structural elements is strongly influenced by the aging processes of materials under the influence of the time factor and the environment (changing temperature, humidity, solar radiation, chemical effects). In particular, the experience of the aviation equipment usage shows that the most significant influence on the performance of its elements is not mechanical wear and power factors, but the aging process and the impact of moisture accumulated in the porous structure due to capillary effects. In relation to PCM, external factors cause the development of physical and chemical processes in materials, especially in the zone of “matrix-fiber” contact interaction with metal fixing elements, which causes changes in performance and, in particular, strength indicators [20-22].

The purpose of the research, the results of which are given below, is to study the effect of the SHF electromagnetic field on the change in the moisture content of carbon fiber and fiberglass samples after they are kept in a climate chamber.

We used samples in the form of flat-parallel plates with dimensions of 75x35x5 mm. The material of the samples is carbon fiber and fiberglass produced by Eurocomplete Ltd, Kaluga.

The samples were divided into control and experimental groups.

SHF processing was performed at the "Zhuk-2-02" installation (LLC NPP "Agroecotech", Obninsk) with a beam-type camera with an unlimited volume (radiation into the open space) at a frequency of 2.45 GHz and a distance from the sample surface to the aperture plane of the radiating horn equal to 200 mm for 2 minutes. Studies [16-18] have confirmed that under these conditions the best results are obtained on increasing the strength of PCM, on bending, and interlayer shear stresses. Three samples were processed simultaneously (figure 1).
After processing, the control samples and prototypes were weighed on electronic balance RM200 with an accuracy of 0.0001 g, then placed in climate test chamber KRK-400 (head. No. 039/84, certified until 20.03, 2020) for 7 days. In the chamber, the following modes were set: temperature +60°C, humidity 80%, which are used for climatic tests of aviation PCM in laboratory conditions. After being in the chamber, the samples were re-weighed. Moisture absorption was estimated by weight change under accepted test conditions. The effect of SHF processing was determined in relation to the average weight change in the batch of prototypes to the control ones.

To identify the mechanism of changing moisture absorption, the microstructure characteristics were determined using the Mira II LMU Tescan scanning electron microscope in the laboratory of special-purpose materials of the Chernyshevsky State University (Saratov). Quantitative assessment of changes in the microstructure was carried out on computer analyzer of microstructures AGPM-6M using the Metallograph software package.

The results of the research are presented in tables 1 and 2. The marked increase in the weight of samples after being in a climate chamber at 80% humidity is associated with the penetration of water through the pores and capillaries between the matrix agglomerates into the PCM structure. The decrease in the weight of the prototypes after being in the chamber is presumably associated with a decrease in the size of open pores, which leads to an increase in the surface tension in the meniscus of the water drop and makes it difficult for it to penetrate the structure.

### Table 1. Change in the weight of control samples and prototypes of carbon fiber after being in the climate chamber (g).

| №  | Condition of the specimen | Before  | After  | Changes | Average value |
|----|---------------------------|---------|--------|---------|---------------|
| 1  | Control                   | 17,0444 | 17,0646| 0,0202  |               |
| 2  | Control                   | 17,5537 | 17,5770| 0,0233  | 0,0221        |
| 3  | Control                   | 17,3458 | 17,3686| 0,0228  |               |
| 4  | Control                   | 16,5482 | 16,5644| 0,0162  |               |
| 5  | Prototype                 | 17,2172 | 17,2372| 0,0020  | 0,0184        |
| 6  | Prototype                 | 17,0887 | 17,1077| 0,0190  |               |

\[ \Delta = \text{Prototypes} \div \text{Control} \]

\[ = 0.8326 \quad (\text{-16.7\%}) \]
Therefore, water only penetrates into large pores, the number of which has decreased in comparison with control samples. A smaller effect on reducing the moisture content of carbon fiber prototypes may be associated with better wettability of carbon fibers and greater porosity compared with fiberglass (or a large open pore size). This assumption is confirmed by images of the microstructure of the matrix of the studied materials obtained at different magnifications (figure 2 and 3).

**Table 2.** Change in the weight of control samples and prototypes of fiberglass after being in the climate chamber (g).

| №.  | Condition of specimen | Before  | After  | Changes | Average value |
|-----|-----------------------|---------|--------|---------|---------------|
| 1   | Control               | 22,9350 | 22,9468| 0,0118  |               |
| 2   | Control               | 22,7600 | 22,7600| 0,0156  | 0,01643       |
| 3   | Control               | 23,2275 | 23,2494| 0,0219  |               |
| 4   | Control               | 22,2277 | 22,2427| 0,015   |               |
| 5   | Prototype             | 21,8810 | 21,8901| 0,0091  | 0,01227       |
| 6   | Prototype             | 21,6505 | 21,6632| 0,0127  |               |

Δ = Prototypes / Control 0,747 (-25,3%)
Figure 2. Microstructure of the matrix of control samples (a, c, e) and prototypes (b, d, f) of carbon fiber after being in a climate chamber. Magnification: x5000 (a, b), h50000 (c, d), x200000 (e, f)
As shown in the analysis of the porous agglomerated structure, the matrix of carbon fiber and fiberglass at different magnification levels looks denser. At maximum magnification of the matrix, there is presence of microfragments of 0.1-0.2 microns on the surface of carbon fiber agglomerates and 0.05-0.1 microns on the surface of fiberglass agglomerates, which becomes more developed. The observed changes in the surface microrelief lead to the curvature of the pore channels and the increased roughness. The differences in the parameters of the porous agglomerated structure of carbon fiber are less pronounced.

The porosity of the prototypes detected using the Metallograph software package with an increase of x5000 decreases in comparison with the control samples in the structure of carbon fiber, respectively, from 53% to 30% (by 43.4%), and fiberglass – from 50% to 33% (by 34%). At the same time, the average pore size in the carbon fiber structure does not change and is about 10-12 microns. For fiberglass, the pore size is reduced from 10-15 microns to 7-8 microns. At x50000 increase pores have been revealed in the structure of control samples and prototypes of carbon fiber of 2-5 and 0.5-1.5 microns, respectively. For fiberglass-3, 5 and 0.5-2 microns. At x 200000 increase, the pore sizes of both carbon fiber and fiberglass do not change and are 0.2-1 microns. It can be noted that the presence of micropores detected at x50000 and x200000 increase will practically not affect the moisture absorption of samples due to high values of resistance to liquid penetration, which will be prevented by the surface tension.

In fact, the saturation of the structure of the studied PCM samples will be influenced by the macro- and mesoporous structure, characterized by both porosity (relative pore volume) and pore size. Additionally, the hydro-resistance of the prototypes will increase due to the marked increase in the relief of agglomerates.

Summarizing, we can conclude that the reason for the decrease in moisture absorption of PCM samples processed in a SHF electromagnetic field is in increase of the resistance to liquid penetration (pore size reduction and increase in their surface roughness ) and in the volume reduction available for liquid placement (porosity decrease). At the same time, the greater effect noted for fiberglass is explained by the fact that when porosity decrease differs slightly from carbon fiber, the pore size decreases by 66%, in average which is not typical for changes in the structure of the latter. Thus, a significant decrease in penetration of liquid into a structure treated in a SHF electromagnetic field of PKM has been found. Bearing in mind the previously established positive effect on improving the
strength of PKM treated in a SHF electromagnetic field, this confirms the feasibility of this method as a finishing technological operation in the production of structural carbon fiber and fiberglass.

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