

CARBON-FILLED POLYMER COMPOSITES DERIVED FROM A HIGH-TEMPERATURE THERMOPLASTIC BINDER

A. A. Beev, D. A. Beeva, I. V. Musov,
E. V. Rzhetskaya, and S. Yu. Khashirova

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Carbon fillers, in particular, various types of graphite, carbon fibers and nanofibers, globular nanocarbon, and carbon fiber tapes improve the usefulness of polymers and have attracted attention for the creation of polymer composites, namely, carbon-fiber-reinforced plastics (or carbon composite) with new outstanding properties such as mechanical strength, heat and electrical conductance, chemical resistance to aggressive media, resistance to radiation damage, and other useful characteristics [1-3].

In light of their specific properties, carbon composites have found various uses from adsorbents and electrical heating elements to parts for airplane bodies and space rockets. There have been many reports on the creation of carbon-fiber-reinforced plastics derived from epoxy resins [4-11]. Epoxy carbon composites often have insufficient heat stability and physicomechanical indices. Thus, work is currently underway on the development of new and improved polymer matrices for the preparation of carbon composites with good thermophysical and physicomechanical properties.

Superconstructional plastics hold special interest for the creation of a new generation of carbon composites. However, the known coupling agents (dressings) of carbon fibers (CF) used for hardening binders are unsuitable for high-temperature thermoplastics. This accounts for the need to develop heat-resistant coupling agents of CF. Information on the surface treatment of carbon and glass fibers over high-temperature thermoplastic matrices is very limited. There is no specific information on the coupling agent compositions and the conditions for their deposition. The compositions and techniques for dressing CF for high-temperature thermoplastics are the subject of know-how development.

The major problems in improving the compatibility of fiber with polymer and improvement of the physicomechanical properties of derived composites lie in the development of techniques for activation of the surface of carbon materials and their coating with coupling agents similar in structure to high-temperature polymer binders.

In the present work, we studied the functionalization and dressing of the surface of carbon fibers and tapes with the aim of obtaining carbon composites on a thermoplastic high-temperature binder. The carbon materials used were Umatex UMT 49-12k-EP fiber and Ordinance RK-300TH tape. The polymer binder was PS-N polysulfone. The dressing of the carbon material was carried out using polyhydroxyether and polysulfone synthesized in the Advanced Polymer Laboratory at Berbekov Kabardino-Balkaria State University [12-15].

The electronic microphotographs were obtained on a Tescan Vega 3 electron microscope manufactured in the Czech Republic.

The thermogravimetric testing was carried out on a Perkin-Elmer TGA 4000 instrument at a heating rate of 5°C/min in the air.

The IR spectrum was taken on a Perkin-Elmer Spectrum Two spectrophotometer.

The thermogravimetric analysis of the starting carbon materials in Fig. 1 shows that their surface is coated with approximately 1.5% coupling agent, whose decomposition begins at 230°C. The thermal decomposition of the CF after removal of the coupling agent begins after 590°C.

The flushing and subsequent IR study of the material found on the carbon fiber surface showed that it is a non-hardened epoxy substance (Fig. 2).

Figure 3 gives electron microscopy images of the initial UMT 49-12k-EP carbon fiber (*a*) and this sample after removal of the epoxy coupling agent (*b*).

In examining the electron microscopy images, we note that microheterogeneities formed by the epoxy coating are characteristic for the surface of the initial CF. After flushing out the coupling agent, there are virtually no heterogeneities on the fiber surface. The carbon materials obtained upon removal of the epoxy compound are suitable for further activation and coating with heat-resistant coupling agents.

N. M. Berbekov Kabardino-Balkaria State University, Nalchik, Russian Federation. E-mail: difenol@mail.ru (A. A. Beev). Translated from *Khimicheskie Volokna*, Vol. 50, No. 6, pp. 66-68, November-December, 2018.

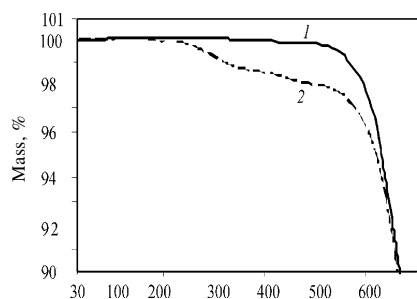


Fig. 1.

Fig. 1. TGA results: 1) CF without dressing and 2) initial dressed fiber.

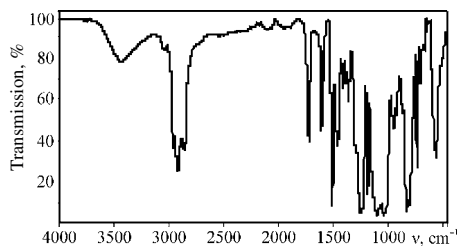


Fig. 2.

Fig. 2. IR spectrum of material removed from the CF surface,

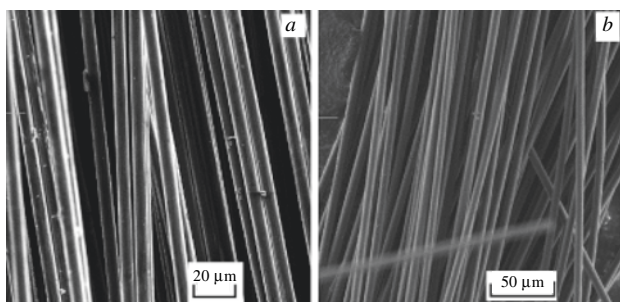


Fig. 3.

Fig. 3. Structure of UMT 49-12k-EP carbon fiber: a) initial and b) after removal of the coupling agent.

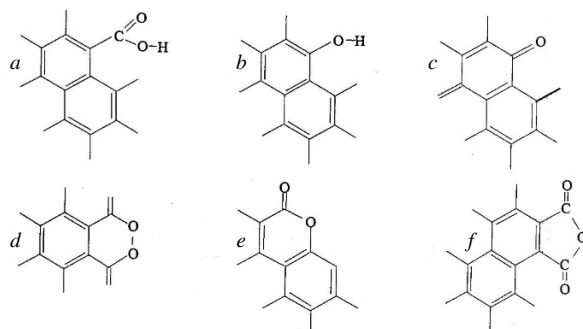


Fig. 4.

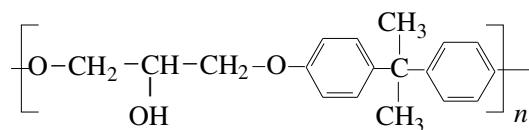
Fig. 4. Possible oxygen-containing groups on the surface of activated CF: a) carboxyl, b) hydroxyl, c) quinoid, d) cyclic peroxide, e) lactone, and f) acid anhydride groups.

Activation of the surface of carbon fibers and tapes was carried out with various concentrations of sulfuric acid. Chemically-active carboxyl, hydroxyl, quinoid, cyclic peroxide, lactone, and acid anhydride functional groups, which facilitate the coupling of the filler with polymer particles, are formed in the structure of carbon fibers and tapes (Fig. 4).

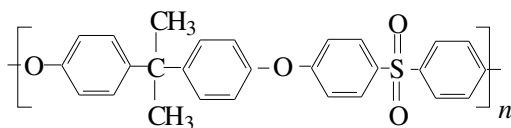
Analysis of the electron microscopy images of the carbon fibers activated by sulfuric acid indicates that their structure depends on the duration of the treatment. Thus, weakly-pronounced grooves are seen on the surface of CF maintained in sulfuric acid for 30 min (Fig. 5a). Upon longer treatment, the nature of the structure changes and craters as well as protuberances are seen (Fig. 5b), indicating partial destruction of the fiber structure.

The dressing of the carbon nanofillers activated for 30 min was carried out in 2-6% sulfuric acid solutions of polyhydroxyether and polysulfone with the following structures:

Polyhydroxyether derived from bisphenol A:



Polysulfone derived from bisphenol A:



Comparison of the microphotographs of the samples of dressed carbon fibers in Fig. 6 clearly shows polymer coatings on their surface formed during the dressing both by polyhydroxyether and polysulfone.

Table 1. Compressive Strength of Carbon Composites Derived from Polysulfone

Carbon composite	Coupling agent concentration, %	Strength, MPa
PPS-CF	–	490
PPS-activated CF	–	536
PPS-dressed CF	3	666-686
Same	5	715-735
" "	7	686-735

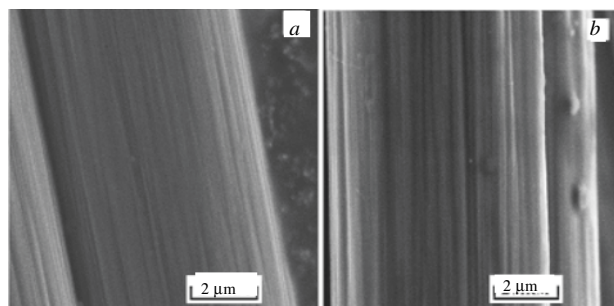


Fig. 5.

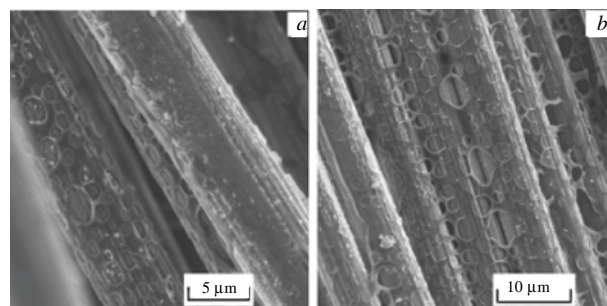


Fig. 6.

Fig. 5. Structure of the CF surface after chemical activation with 70% sulfuric acid at 60°C for 30 (a) and 60 min (b).

Fig. 6. Electron microscopy images of CF dressed with polyhydroxyether (a) and polysulfone (b).

However, we can see that upon dressing, the surface of the carbon fiber is not coated by a smooth homogeneous layer of polymer. This can happen because single treatment of the fiber by a coupling agent apparently does not provide for a sufficient coating of its surface by polymer and requires multiple treatments.

In order to determine the efficiency of the resultant dressed carbon materials, samples of carbon composite derived from polyphenylene sulfone as the thermoplastic binder were prepared by pressure casting.

Table 1 gives the results of testing the compressive strength of the carbon composite with a CF dressed with polyhydroxyether derived from bisphenol A of different concentrations. These findings suggest that the compressive strength of the polysulfone carbon composite is enhanced by 40-50% due to surface activation and the introduction of a thermoplastic polyhydroxyether dressing, which increases the wettability of the filler and strengthens the interaction between the filler and polysulfone matrix.

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