

Determination of Adhesion Interaction between Carbon Fiber and Epoxy Binder

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Received April 2, 2014

Abstract—Experimental results of the concentration of paramagnetic centers have been given for carbon fibers of Elur-0.1P, UOL-300, LUP-0.1, HTS-45, AS-4, T-300, and T-700 brands and carbon plastics designed on their basis with ENPB binder. The strength of carbon plastics at interlaminar shear has been used as a measure of adhesion interaction. It is shown that the highest concentration of paramagnetic centers and the highest strength of adhesion interaction are intrinsic to carbon plastics based on T-700 brand fiber. It is noted that the oxidation of carbon fibers leads to the increase in the concentration of paramagnetic centers on their surface.

Keywords: carbon fiber, carbon plastics, epoxide binder, paramagnetic center, strength at interlaminar shear, adhesion interaction

DOI: 10.1134/S1995421215010116

INTRODUCTION

It is known that, for polymer composition materials (PCMs), the characteristics of adhesion interaction, the values of which are determined just as is any strength of adhesive bonding, in megapascals, depend on the properties of the filler–matrix pair. Assuming a high abundance of carbon plastics as structural materials, the attention of researchers has recently been focused on studying ways to increase adhesion interaction between high-modulus carbon fiber and epoxy binder.

The conventional approach to increasing adhesion involves a choice (or design) of the binder providing the highest values of adhesion strength [1–3]. This approach is viable; however, the chemical, physical, and even mechanical features of the structures of surface layers of carbon fibers also significantly affect the adhesion interaction.

The aim of this work was to study the features of adhesion interaction between ENPB epoxy binder and carbon fibers of domestic (Elur-0.1P, UOL-300, and LUP-0.1) and foreign production (HTS-45, AS-4, T-300, and T-700).

EXPERIMENTAL

Experiments on the intensity of adhesion interaction between carbon fiber and binder were carried out on a Varian radiospectrometer [4]. For these purposes, the area under the absorption curve (Fig. 1), which is directly proportional to the concentration of paramagnetic centers (PMCs), was measured [5]. Each

paramagnetic center represents a free radical, which has the ability to strong intermolecular interaction (Fig. 2). The paramagnetic center is on the amorphous part of the carbon fiber (Fig. 3); for this reason, oxida-

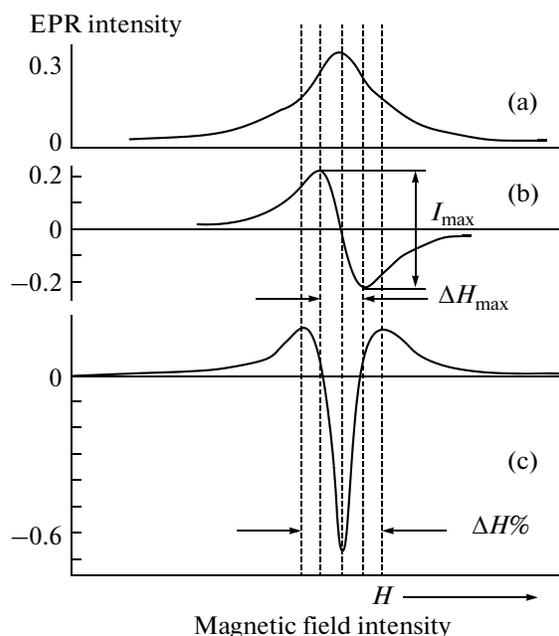


Fig. 1. Intensity of electron paramagnetic resonance (EPR) vs. magnetic field intensity: (a) absorption curve, (b) first derivative of absorption curve, and (c) second derivative of absorption curve.

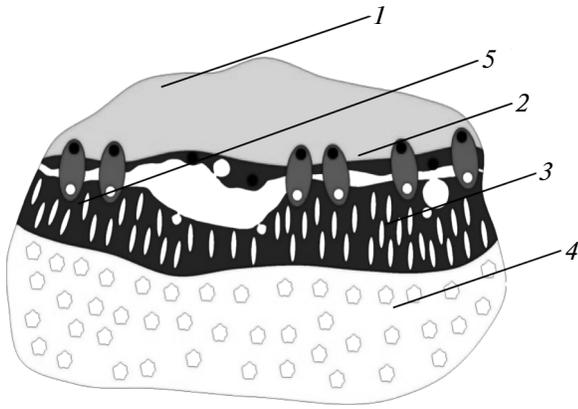


Fig. 2. Structural scheme of carbon plastics: (1) carbon fiber, (2) paramagnetic center, (3) interfacial zone, (4) polymer matrix, and (5) pores and other defects of interfacial zone.

tion of fibers leads to an increase in the concentration of paramagnetic centers, because oxidation increases the fraction of the amorphous phase.

The role of paramagnetic centers is played by carboxylic ($-\text{COOH}$), carbonyl ($-\text{C=O}$), and other groups containing oxygen that are located on the surface of carbon fibers [4]. The higher their number, the more active this fiber to the binder.

The effectiveness of the adhesion interaction was estimated by the difference between the concentration of centers in fiber and carbon plastics. This is related to the fact that the concentration of paramagnetic centers in carbon plastics should be lower than that in the fiber, because the fraction of paramagnetic centers interacted with the binder.

Two groups of materials were used as samples: carbon fibers and carbon plastics based on them. Two characteristics were evaluated for them, namely, half-width of absorption spectrum ΔH (Fig. 1c) and concentration of paramagnetic centers N , which was determined by the area under the absorption curve (Fig. 1a). The concentration of the paramagnetic cen-

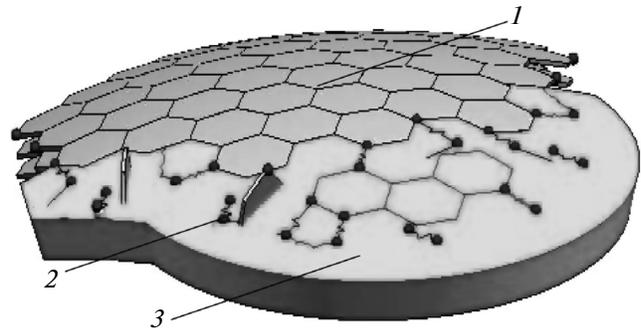


Fig. 3. Scheme of the section of carbon fiber surface: (1) graphite-like part of carbon fiber surface, (2) paramagnetic center, and (3) amorphous part of carbon fiber surface.

ters was determined from comparison of the spectral intensities of the studied sample and reference, namely, diphenylpicrylhydrazyl. For this purpose, the dependence given in Fig. 1a was experimentally obtained, from which the first (Fig. 1b) and second (Fig. 1c) derivatives were consecutively taken.

RESULTS AND DISCUSSION

In Table 1, experimental values are given for carbon fibers and carbon plastics based on them. As follows from the obtained data, the &-700 carbon fiber is the most active. Concentration of paramagnetic centers in fiber N_f was 7.1×10^{-17} spin/cm³; in carbon plastics, it decreased and corresponded to $N_{km} = 1.2 \times 10^{-17}$ spin/cm³. By analogy, the half-width of the signal of the spectrum for carbon fiber ΔH_f and carbon plastics ΔH_{km} changed.

The higher the concentration of paramagnetic centers in fiber and the lower the number thereof remaining in carbon plastics, the higher the adhesion interaction of fiber with binder.

Adhesion strength was determined indirectly by comparing the strength at interlaminar shear for carbon plastics prepared by pressing. For convenient

Table 1. Results of studies according to electron paramagnetic resonance

| Brand of carbon fiber | Fibers | | Carbon plastics | |
|-----------------------|------------------------|--|---------------------------|---|
| | ΔH_f , oersted | $N_f \times 10^{-17}$, spin/cm ³ | ΔH_{km} , oersted | $N_{km} \times 10^{-17}$, spin/cm ³ |
| Elur-0.1P | 25 | 13.5 | 20 | 0.59 |
| HTS-45 | 56 | 3.3 | 30 | 2.6 |
| UOL-300 | 21 | 3.4 | 40 | 3.4 |
| LUP-0.1 | 52 | 4.8 | 17 | 1.1 |
| AS-4 | 20 | 3.3 | 28 | 1.6 |
| T-300 | 31 | 3.5 | 53 | 3.7 |
| T-700 | 32 | 7.1 | 21 | 1.2 |

Table 2. Characteristics of carbon fibers and carbon plastics

| Brand of carbon fiber | Strength of carbon fiber at elongation, MPa | $N_{\text{km}} \times 10^{-17}$, spin/cm ³ | Strength of carbon plastics, MPa, at | |
|-----------------------|---|--|--------------------------------------|------------|
| | | | interlayer shear | elongation |
| T-700 | 4500 | 1.2 | 64 | 2050 |
| HTS-45 | 4000 | 2.6 | 34 | 1780 |
| T-300 | 3500 | 3.7 | 30 | 1400 |
| LUP-0.1 | 2500 | 1.1 | 56 | 1110 |
| Elur-0.1P | 2400 | 0.59 | 77 | 1020 |
| UOL-300 | 2200 | 3.4 | 40 | 1250 |

Table 3. Characteristics of carbon fiber AS-4 and carbon plastics based on it prior to and after oxidation of fiber

| Carbon fiber | Strength of carbon fiber at elongation, MPa | $N_{\text{km}} \times 10^{-17}$, spin/cm ³ | Strength of carbon plastics, MPa, at | |
|--------------------|---|--|--------------------------------------|------------|
| | | | interlaminar shear | elongation |
| Prior to oxidation | 4250 | 1.6 | 46 | 1950 |
| After oxidation | 5100 | 2.1 | 60.6 | 2130 |

comparison of the obtained result, all used brands of carbon fibers are given in Table 2 in descending order of strength. As follows from the obtained data (see Table 2), the highest strength at interlaminar shear was obtained on carbon plastics, which was prepared using T-700 fiber. At the same time, all results of the strength of carbon plastics at interlaminar shear given in Table 2 depend not only on the value of adhesion interaction, but also on many technological factors, and therefore may not serve as direct evidence that the concentration of paramagnetic centers has a positive effect on the value of the adhesion strength.

With the goal of determining the effect of the concentration of paramagnetic centers on the value of the adhesion strength for AS-4 carbon fiber, the oxidation of its surface at 650°C was performed for 10 min. The obtained results (Table 3) show that oxidation leads to an increase in the concentration of paramagnetic centers by more than 30%. Simultaneously with the increase in the concentration of paramagnetic centers, there was also an increase in the strength of carbon plastics at interlaminar shear, as well as an increase in the strength of carbon fibers.

CONCLUSIONS

It was experimentally proved that the concentration of paramagnetic centers depends on the brand of carbon fiber and can be varied by oxidation thereof.

The intensity of adhesion interaction between fiber and binder can be determined from the difference between the concentrations of paramagnetic centers

in fiber and carbon plastics. The higher the concentration of paramagnetic centers in fiber and the less their number in carbon plastics, the higher the adhesion interaction of fiber with binder.

The highest number of paramagnetic centers was detected on the surface of T-700 fiber, which also has the highest strength at interlaminar shear of all the studied carbon plastics.

REFERENCES

1. S. L. Bazhenov, A. A. Berlin, A. A. Kul'kov, and V. G. Oshmyan, *Polymer Composite Materials* (Izd. Dom Intellect, Dolgoprudnyi, 2010) [in Russian].
2. N. I. Baurova, "The dynamics of the processes of destruction of polymer composites," *Entsikl. Inzh.-Khim.*, No. 2, 19–25 (2013).
3. I. A. Aleksandrov, A. N. Muranov, and G. V. Malysheva, "Study of the influence of the deformation properties of binders on CFRP destruction processes," *Vse Mater. Entsikl. Sprav.*, No. 7, 40–45 (2012).
4. L. A. Blyumenfel'd, V. V. Voevodskii, and A. G. Semenov, *Application of Electron Paramagnetic Resonance in Chemistry* (Akad. Nauk SSSR, Novosibirsk, 1962) [in Russian].
5. V. I. Volga, A. S. Kotosonov, V. S. Tverskoi, and N. N. Ryabikina, in *Structure and Properties of Carbon Materials: Collected Papers* (Metallurgiya, Moscow, 1984), pp. 56–62 [in Russian].

Translated by A. Muravev