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Nonequilibrium carbon black suspensions used in synthesis of polymer composite material

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Nonequilibrium carbon black suspensions used in synthesis of polymer composite material

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Abstract. Nowadays polymer matrix-based composite material with various carbon fillers are widely used to protect radioequipment from different interference, to improve characteristics of radar absorbing coatings. Current synthesis processes are sophisticated and rather costly. The challenge is to develop new methods of producing composite materials by efficient knowledge intensive technologies to reduce the cost of products. The paper studies possibility of producing composite material on the basis of elastic polyurethane foam with carbon fillers using polyurethane impregnation in nonequilibrium black carbon suspension. Suspension composition: running water and carbon-bearing powders of nanometer range: fullerene black carbon, Taunite (multi-layer carbon nano tube material), technical carbon T900, wood soot. Nonequilibrium suspension was produced by treatment in hydrodynamic generator of rotor type (cavitation mode). Angular rotation speed of the rotor ω =10000 rpm.

1. Introduction

To protect equipment from external interference is a challenge of today’s material science and design. To ensure electromagnetic compatibility of the screening device and the source of radiation, to reduce the weight of the structure requires development of new volume composite radio materials. Concentration conditions under which the conducting graphite coatings can form on cardboard and polymer dielectric substrates by settling graphite particles from colloid graphite dispersions were studied in [1].

Effect of fullerene modifier on radar-absorbent characteristics of fibrous composite material and structure of panel from the same material were studied in [2]. Measurements of absorption coefficient of panels with modifier and without it are given. Feasibility of applying the proposed compositions in aircraft structures is evaluated.

Synthesis of high-performance and economic composite carbon materials containing carbon fibers, graphite, black carbon for electromagnetic screening in electronics, communication, aviation, space and civil infrastructure is presented in [3, 4]. In [5] recent works in the field of development of polymer materials and nanocomposites protecting from electromagnetic interference are reviewed.

In [6] radar-absorbing materials for broad-band electromagnetic wave absorbers are considered, advantages and disadvantages of various types of absorbers with dielectric and magnetic loss are analyzed. Prospects for development of high-performance broad-band radar-absorbing materials are outlined.

2. Materials & Methods

2.1. Object of research

The objects under study are porous composite materials made by impregnation of elastic polyurethane foam in low-concentration carbon black suspensions produced by treatment in rotor-type hydrodynamic generator in cavitation mode. The working liquid of the suspension was running water. Low concentrations of the solid phase in suspensions were used to eliminate active coagulation of carbon particles.

Under study were systems in which the filler was uniformly distributed over the entire volume of the polymer matrix and systems with undoubtedly non-uniform distribution of the filler. The elastic polyurethane foam fillers are fullerene soot (FS) with fullerene content C_{60} 11% mass and particle size 20-100 nm; multiwall nanotubes with outside diameter 15–40 nm (“Taunite” material), wood soot (WS) with globules sizing ≈60 nm. By XRF method wood soot samples were found to contain small quantities of C_{60} and C_{70} fullerenes [7]. For elastic polyurethane foam filler we also used fine natural graphite powder from Kureika deposit with particles in the form of thin plates sizing up to 100 nm and technical carbon T900 with globules sizing up to 100 nm. Impregnation time, pressure, suspension concentration, drying time were identical for all samples.

2.2. Synthesis process parameters

Low concentration carbon black suspensions have been produced in hydrodynamic generator at angular rotation speed of the rotor ω=10000 rpm, exposure time – up to 3 min. The exposure process components were turbulent microstirring and bubble cavitation. These are cavitation-activated suspensions that under intensive turbulence acquired properties of nonequilibrium systems. Polyurethane foam was filled with carbon by two impregnation methods. The first method was to submerge polyurethane foam under pressure for 5 minutes followed by removal and drying. This procedure was repeated 3 times on the same sample. In the case of the second method the sample was impregnated one time under pressure for 24 hours followed by drying.

Efficiency of carbon absorption by polyurethane foam from the suspension and microwave radiation absorption by the samples were examined by optical microscopy (microscope HIROX KH-7700), gravimetric method (electronic balance KERN-770-60), resonance microwave method (“Obzor 304” instrument, working frequency range 0.3 MHz ÷ 3.2 GHz). The samples under study were uniformly stained black leaving no marks on the white paper (figure 1). Electronic properties of initial and cavitation-activated suspensions were studied with SE/X-2544 EMR spectrometer at frequency 9 GHz, 3 cm at 293 K, 110 K.

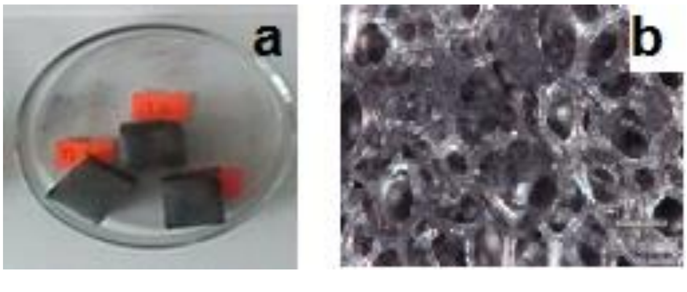

Figure 1. (a) Polyurethane foam sample with technical carbon T900 and (b) structure of polyurethane foam with filler T900, optical microscope HIROX KH-7700, magnification x100.

3. Experimental results and discussion

The study showed that polyurethane foam most actively absorbs carbon from the fullerene-containing carbon black suspension (table 1). When impregnated for 24 hours the sorption of carbon by polyurethane foam is 11% of the carbon black available, T900 (7%) goes second. Other samples exhibited less absorption. When the fullerene soot suspension was produced by simple mechanical stirring, the sample left marks on the paper, the soot flaked from the surface. This can be due to non-uniform distribution of the filler in the polyurethane foam, because mechanical stirring does not make a suspension with good sedimentation properties.

Absorption of carbon depends on the shape of primary particles of the carbon black powder. E.g. absorption of fullerene soot, T900 and wood soot with spherical shape of the primary carbon black particles is better than that of the graphite powder (plates) and Taunite powder (nanotubes). Impregnation of polyurethane foam depends on the composition of carbon black globules (figure 2a). E.g. composition of T900 is carbon with minimum amount of impurities, and the composition of the wood soot globule is carbon, organic carbon and considerable amount of various inorganic impurities (figure 2b, c).

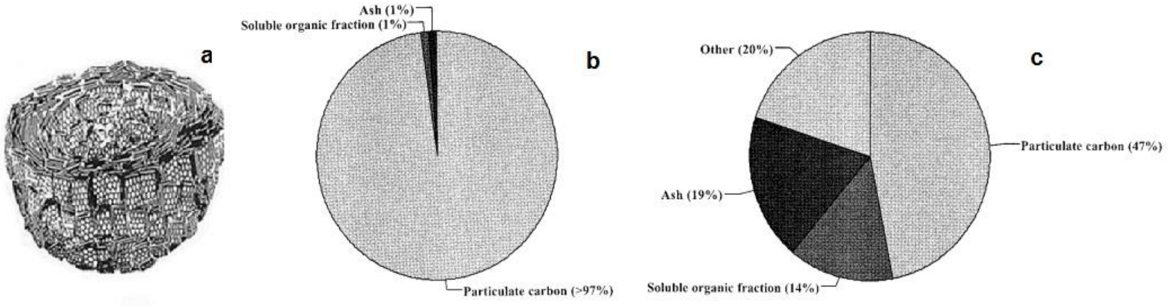

Figure 2. (a) Model of a carbon black particle showing the arrangement of the turbostratic domains [8] and comparison of chemical composition among (b) carbon black and (c) domestic chimney soot from a wood-burning fireplace (soot was collected from flue surfaces of domestic fireplaces burning hardwood) [9].

Single impregnation for 24 hours after which almost all samples exhibit better carbon sorption than in short-time three-times is more efficient.

Capacity of produced samples to absorb electromagnetic radiation was measured with "Obzor 304" instrument, working frequency range 0.3 MHz ÷ 3.2 GHz. Results are shown in the table 1.

Table 1. Absorption properties of polyurethane foam with different fillers.

Filler	Sample impregnation time in black carbon suspension (hours)	Initial weight of polyurethane foam sample, m_0 (mg)	Weight of polyurethane foam sample after impregnation, m (mg)	Weight fraction of absorbed carbon, Δ (%)	Absorption frequency (GHz)	Signal attenuation (dB)
T900	0.25	201	215	6.96	1.82	6.72
T900	24	202	217	7.42	1.82	6.25
FS	0.25	169	188	11.24	1.82	-
FS	24	166	185	11.44	1.82	-
Graphite	0.25	204	209	2.45	1.82	2.59
Graphite	24	164	169	3.01	1.82	2.64
Taunite	0.25	168	175	4.16	1.82	-
Taunite	24	213	220	3.29	1.82	-
Wood soot	0.25	192	202	5.21	1.82	1.7
Wood soot	24	210	221	5.24	1.82	-

* Weight fraction of absorbed carbon $\Delta=(m-m_0)*100\%/m_0$.

4. Discussion

Carbon particles produced from cavitation-activated suspension exhibit enhanced adhesion capacity. With the generator operating in cavitation mode high-energy effect of cumulative liquid microjets and shock waves forming in cavitation bubble collapse is observed. The temperature and pressure can, at this, reach values higher than 2000 K and 100 MPa, respectively, these initiates changes in physical-chemical properties of the carbon black particles and intensive dispersion. As a result of intensive turbulence, the cavitation-activated suspensions acquired properties of nonequilibrium systems.

At 293 K the EMR spectrum of T900 sample (figure 3a) is simulated by three Lorentz-shape lines corresponding to impurities with magnetic ions contained in the sample. They are oxides of iron, chromium and cobalt (<1%) surrounded by ions of oxygen, sulfur, silicon, potassium, etc. Hydrodynamic impact on T900 powder under different rotation conditions in high-speed laboratory blender changes the form of the spectrum (figure 3b). At 3000 rpm the spectrum is simulated by 2 Lorentz lines, at 5000 rpm – by three, the basic line decomposes into two (appearance of anisotropy in the electron structure). Under cavitation conditions the form of EMR spectrum of T900 (figure 3c) changed because of changed magnetic properties of impurity magnetic centers attributed to fine superparamagnetic particles. They are recorded to enlarge; more uniform particles of ions of iron oxides and other elements are observed to form.

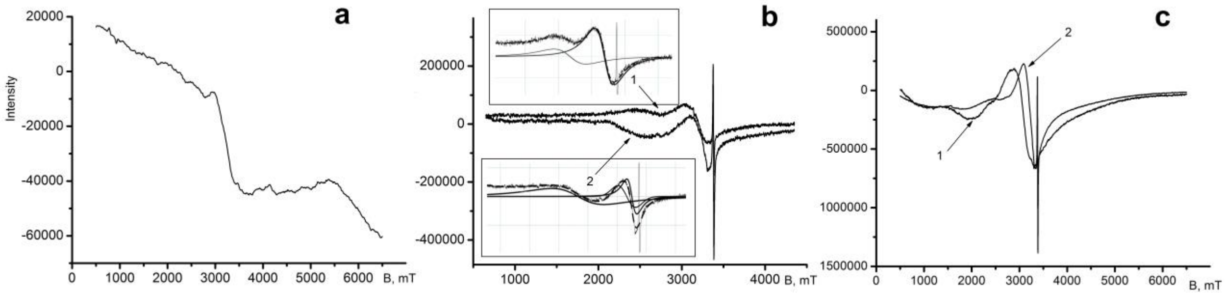

Figure 3. EMR spectra of technical carbon T900: (a) initial T900 carbon at 293 K; (b) T900 after treatment in hydrodynamic generator (line 1 – ω=3000 rpm, line 2 – ω=5000 rpm, 293 K); (c) T900 after treatment under cavitation condition sat ω=10000 rpm (line 1 – 293 K, line 2 – 110 K).

According to the data of microprobe analysis cavitation changes chemical composition of the surface of T900 particles. Due to surface segregation of impurities localized at the collapsing cavitation bubbles the surface of T900 was found to have chemosorbed oxygen atoms; this increases adhesion of T900 in the polyurethane foam.

Active dispersion makes cavitation exert higher influence on the properties of graphite. Its structure is formed by parallel graphene planes. Graphene is known to have high adhesion capacity. Similar to single graphene planes thin graphite plates also exhibit enhanced adhesion capacity. However, in this experiment thinning of plates was not sufficient for the adhesion to be higher than that of T900.

The absorption capacity of polyurethane foam samples with T900 grade technical carbon fillers in the microwave band is not worse than of industrial samples. Therefore, absorbing composite materials made by impregnation of polyurethane foam in cavitation-activated suspensions can be produced with use of cheap technical carbons.

5. Conclusion

Cavitation process used to synthesize absorbing materials in the microwave range on the basis of elastic polyurethane foam with different carbon fillers is shown to reduce the cost of manufactured products.

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