## **Electrophysical and strength characteristics of** polychlortricfluoroethylene filled with carbon nanotubes dispersed in graphene suspensions

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Introduction: It is known that one of the decisive factors that determines the effectiveness of using carbon nanotubes (CNTs) for strengthening polymer matrices is their uniform distribution. At the same time, it is shown, for example [1], that the current threshold, determined by electrical conductivity data, shifts to the region of smaller values with a more uniform distribution of CNTs and correlates with the structural and strength characteristics of filled polymers. In this study, the preliminary deagglomeration of CNTs was performed using ultrasonic treatment in a dispersion of graphene nanoparticles (GNPs).

Synthesis of graphene nanoparticles. GNP was obtained by secondary "intercalation" of electrodes from expanded graphite (EG) foil (according to TU U 26.8-30969031-002-2002) in an alkaline electrolyte (KOH) of low concentration with a current transmission of 6.0 to 60.0 mA/cm<sup>2</sup>. The separation of nanoparticles was provided by a membrane made of polypropylene fabric (Fig. 1)



Fig. 1. shows a block diagram of one of the possible installation for obtaining nanoparticles of graphene, where: 1 -electrolytic bath, 2 - cathode, 3 - cassette device with a strip of fiberglass-reinforced EG foil, 4 - anode, 5 - polypropylene membrane, 6 - filter, 7 - tank with electrolyte solution of potassium hydroxide, 8 - float valve, 9 - part of the electrolytic bath for suspension of graphene nanoparticles, 10 - photodiode, 11 - LED, 12 - control unit, 13 - control valve drive, 14 - control valve, 15 - tank for suspension of graphene nanoparticles, 16 - tap for removing coarse suspension of EG, 17 - container for coarse suspension of expanded graphite.



Fig. 2. SEM image of graphene structures on the surface of gold (the substrate is inclined at an angle of 60° to the electronic probe): a, b — graphene-like particle; b — enlarged images of sections of graphene-like particle (b), respectively (selected square-volume); c — the curve of change in\ brightness of the image along the line AB

Table 1. Particle sizes of graphene material obtained under different conditions of electrochemical oxidation of EG, determined by the LCS method (spectrometer "ZetaSizer 3" Malvern Instrument (UK) with a correlator 7032 and helium-neon laser LG-111 with a power of 25 mW for wavelength  $\lambda = 633$  nm)

N*	Average size,	Fraction I size, nm			Fraction II size, nm			Polydispersity
sample	nm	minimum	maximum	probable	minimum	maximum	probable	Polydispersity



## Multi-walled carbon nanotubes (TU U 24.1-03291669-009: 2009. Authors: Yu.I. Sementsov, O.V. Melezhik). Fig. 3.



L	_				1			1	
	1	2162	8	130	40	130	31000	7954	1
	2	623	12	300	54	300	25000	4133	1
	3	1529	11	200	35	200	68000	16500	1
	4	3251	11	280	35	280	70000	16500	1
	5	1264	12	300	55	300	43000	10048	1
	6	1349	12	200	40	200	3200	642	0.6
	7	7049	33	800	42	800	8000	1664	1



Fig. 4. Particle size distribution in suspension (sample 6, table 1): a -"monomodal" mode; b, c - "polymodal" mode: b - mode of particle distribution by volume (mass at constant density); c - particle distribution

Preparation of materials. Deagglomeration of CNTs was carried out by ultrasonic treatment in a dispersion of graphene nanoplates. The GNP content was 0.1 m. f. (mass fraction) to 1 m. f. of CNTs, the mass ratio of GNPs to CNTs did not change. Three aqueous systems with a CNTs content of 0.5 (S0.5), 0.25 (S0.25), and 0.125 (S0.125) wt.% were dispersed. The CNT@GNP suspension was thoroughly mixed with polychlortricfluoroethylene (PCTFE) powder pre-moistened with ethyl alcohol. The resulting dispersion was dried in a rotary evaporator at a temperature of 75 °C. After removing the water, the resulting mixture was poured into a mold and samples were formed at a temperature of 232 °C and a pressure of 5 MPa. In the work, composites with the content of hybrid filler BNT@GNP from 0.0005 to 0.1 vol. f. were studied (volume fraction (VF)) for each obtained system.



 $\phi_c$  – content value corresponding to the percolation threshold; t - a critical index

dispersion of CNTs for PCTFE-CNT@GNP systems

u				Experime		
f CNTs	system #	suspension during CNT dispersion, % wt.	$\phi_c$ , volume fraction	t	$\sigma_{i}$ , ( $\Omega$ ·cm)-1	(Ω·cm
	S0.5	0.5	0,0047	2,01	9,9*10+1	1,44.10
	S0.25	0.25	0,0032	1,78	8,6*10+1	9,89.10
	S0.125	0.125	0,00097	1,75	$7,1*10^{+1}$	1,66.10

Conclusion. It is shown that the joint ultrasonic treatment of aqueous mixtures of carbon nanotubes and graphene nanoplates makes it possible to obtain their stable dispersions at a ratio of 0.1 ppm. GNP up to 1 m.h. CNTs and water content of 0.5-0.125% by mass. It was established that the reduction of the percolation threshold in polymer-filled PCTFE – CNTs@GNP systems can be achieved by reducing the concentration of CNTs in water during ultrasonic dispersion in the presence of GNPs. At the same time, higher levels of electrical conductivity at low frequencies and dielectric losses at microwaves are achieved with a lower content of CNTs. It is shown that the percolation threshold in the PCTFE-CNTs@GNP system decreases from 0.0048 to 0.00097 volume fraction when the content of CNTs in water decreases from 0.5 to 0.125%. At the same time, the CNTs content at which the maximum flexural strength values are observed shifts according to the shift in the percolation threshold. The maximum values of the strength limit will be achieved with a more uniform distribution and stretching of CNTs in the volume of the polymer matrix.

<sup>1</sup>Ivanenko K.O., Ushakova L.M., Avramenko T.G. et.al. Influence of Nanofillers Concentration on Physical and Mechanical Characteristics of Their Polymer Composites // Springer Proceedings in Physics.-2021.-246.-P. 685-698..

