

# STUDY OF THE INFLUENCE OF NANO-CARBON MATERIALS ON THE STRUCTURE AND SOME PHYSICAL PROPERTIES OF POLYMERS

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## Abstract

Studies have been conducted of the influence of nanocarbon materials on the structure and some physical properties of amorphous and crystalline polymers. It has been shown that an increase in filler content in polymers is accompanied by changes in structure, mechanical and thermal properties. The formation of crystal solvates in the ternary polymer-fullerene-solvent system in crystalline polymers and the formation of superstructures-spherulites in amorphous polymers were discovered. The observed changes are explained in terms of the competing influence of the interaction of the polymer with fullerene and solvent, changes in the state of the amorphous regions of the polymer upon introduction of the filler, and the technology for producing nanocomposites.

**Keywords:** nanocarbon materials, structure, property, polymer, strength, deformation.

## Introduction

It is well known that depending on the nature of the fillers, the shape and size of the particles, the nature of their distribution in the matrix, the interaction of filler molecules with the polymer, technological factors, etc., various options for changing the structure and performance properties of polymer composites can be observed [1-10]. However, in the technological aspect, there are few works in the literature devoted to the study of the structure and some physical properties of polymers when modified with various nanocarbon substances.

The following polymers were used as research objects: low-density polyethylene (LDPE), high-density polyethylene (HDPE), isotactic polypropylene (IPP), polyamide-6 (PA-6), polymethyl methacrylate (PMMA), polyvinyl alcohol (PVA), polystyrene (PS), polytetrafluorethylene (PTFE). The fillers were fullerene carbon (FC), C<sub>60</sub> fullerene, multi-walled nanotubes (MWNTs), and nanodiamonds (ND). Non-oriented nanocomposite polymers were obtained by forming them from solutions in aromatic solvents (benzene and its derivatives) and from melts [3-7]. The concentration of fillers was varied within the range C = 0.1-10 mass. Mechanical tests of the samples were carried out on a standard tensile testing machine RM-1 with a strain rate of 0.01 s<sup>-1</sup>, thermal tests were carried out on a DSK 204 F Netzsch thermal analyzer with a heating rate of 100/min, radiographic studies were carried out on DRON-2 and KRM-1 installations, copper Nickel filtered radiation. Optical studies were



carried out using a polarizing microscope MIN-8. All studies were carried out at a temperature of 20°C.

### Methods

Using polarization optical microscopy, it was established that in crystalline polymers LDPE, HDPE and IPP, the main morphological form of the supramolecular structure is spherulitic, the presence of which is also confirmed by studies using an atomic force microscope (AFM) [7-10]. The sizes of spherulites are 10-15 microns for LDPE, 100 microns for HDPE, and 130 microns for IPP. From small-angle X-ray diffraction patterns (XRA) of polymers it follows that these structural formations are characterized by tangential periodicity with a large period of 25-30 nm. High-angle X-ray diffraction patterns (HAXRD) showed the presence of crystalline aggregates of 5-6 nm in size in the structure. When introducing fillers within the concentration range  $C = 0.1-10\%$  mass. in the BR pictures there are no changes in the positions and radial half-widths of the reflections. The observed slight decrease ( $\sim 10-15\%$ ) in the intensity of the reflections is apparently due to the effect of absorption of radiation by the substance. Consequently, nanocarbon materials ( $C_{60}$  fullerene, etc.) do not interact with polymer crystallites and are not included in their crystal lattices.

### Results

In the MR images, with increasing filler concentration in the range  $C = 0-1\%$ , there is first an increase in the discrete scattering intensity  $I_m$ , and then at  $C > 1\%$  constancy of  $I_m$  is observed. Analysis of BR and MR patterns based on a two-phase model of the supramolecular structure of polymers shows that  $\sim 10\%$  of filler molecules penetrate into interlamellar amorphous regions, and most of them ( $\sim 90\%$ ) are located in interspherulitic amorphous regions, which is also confirmed by AFM studies [7]. Dispersion of the filler to the molecular level and their uniform distribution in the volume of the matrix determine the absence of any aggregates of additives.

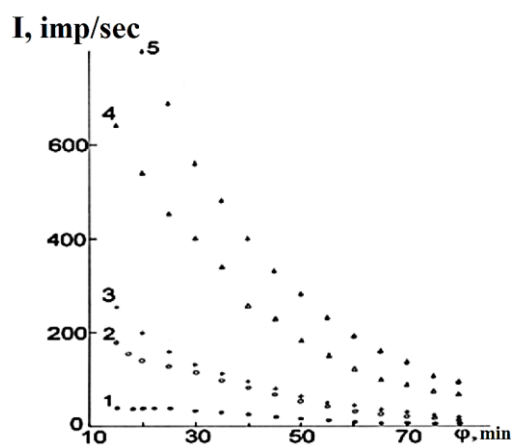


Fig.1. Small-angle X-ray diffraction patterns of LDPE+ND obtained from the melt. A different situation is observed in the case of the introduction of nanodiamonds into polymers. Apparently, it is due to the fact that nanodiamonds are a hard dispersed phase in a polymer dispersion medium. At a concentration of nanodiamonds  $C = 5-10\%$ , scattering from nanodiamonds is observed in the BR patterns of amorphous and crystalline polymer



composites. The sizes of crystalline grains of nanodiamonds are 4-5 nm. The introduction of nanodiamonds is accompanied by a gradual increase in the intensity of diffuse scattering in the MR patterns of polymer composites, which indicates the development of microheterogeneity in the structure of the material, the emergence of microinhomogeneities such as pores, cracks, etc. (Fig. 1).

The interaction of  $C_{60}$  fullerene with a polymer and a solvent leads to the formation of  $C_{60} \cdot 2BrBe$  crystal solvates in the ternary polymer-fullerene-solvent system in LDPE, HDPE and IPP composites [8]. In the BR pictures of polymers at a concentration of fullerene  $C_{60}$  in samples  $C = 10\%$ , the most intense reflections of crystal solvates (CS) appear in the angle range  $2\theta = 8-20^\circ$ . The size of the CS is  $\sim 30$  nm and its share is 8-10%. From these data it follows that almost the entire solvent, that is, bromobenzene (BrBe), is spent on the formation of CS. In amorphous polymers PMMA and PS, crystal solvates are not formed, but superstructures-spherulites of giant sizes of 1-5 cm or more appear (Fig. 2).

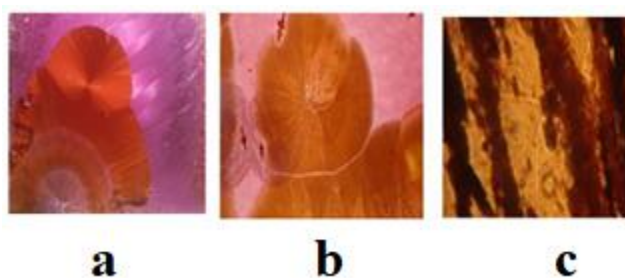


Fig.2. Microphotographs of PMMA + 10%  $C_{60}$  (a), PS + 5%  $C_{60}$  (b), photo rays (b) at a magnification of x1600 (c).

### Discussion

In addition, against the background of these structures, aggregations of  $C_{60}$  fullerene of a fractal nature are observed. The rays of spherulites are a conglomerate of accreted growths, hairs and different-sized aggregates-crystallites of BrB, fullerene, etc. The observed superstructures turned out to be thermodynamically unstable. In the thermogram of the first heating cycle of LDPE samples, along with the main melting peak at  $107^\circ$ , a number of peaks of different intensities and positions on the temperature scale appear, which correspond to different structural organizations [7]. With increasing concentration of fullerene  $C_{60}$ , the multiplicity gradually disappears, the main melting peak is observed at  $107^\circ$ , and in the region of  $70-90^\circ$  a diffuse peak appears, which apparently corresponds to the melting of CS. In the second cycle, the thermograms show only a singlet peak of melting of the polymer composite at  $107^\circ$ . In amorphous polymers, with increasing fullerene content, some small fluctuations in the transition temperature (softening or glass transition) of the composites relative to the transition temperature of the original pure sample are observed. Figure 3 shows the deformation curves of fullerene-containing samples made from a composite of LDPE and PS.



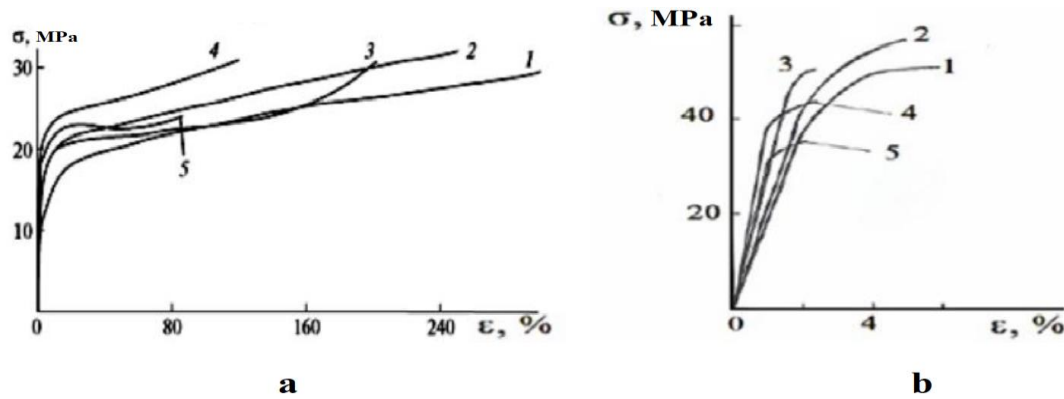


Fig.3. Stress-strain curves of LDPE+C<sub>60</sub> films (a) cast from solutions in DCB, PMMA+C<sub>60</sub> (b) from solutions in BrBe. Concentration of fullerene C<sub>60</sub> in films: 1 – 0; 2 – 1; 3 – 3; 4 – 5; 5 – 10%.

Almost similar changes in mechanical properties are observed in composites containing other nanocarbon materials (PS, MWCNT, NA). From the analysis of the results of mechanical tests of composites, one can notice some common features of changes in the mechanical properties of polymers when they are modified with nanocarbon materials. At low filler contents in the range  $C = 0.1-1\%$ , the strength of polymers increases to 30-50% while deformability, melting temperature and softening (glass transition) remain unchanged. At high filler concentrations of more than  $C > 1\%$ , a sharp decrease in deformability is observed while maintaining or slowing down the decrease in strength, constancy of the melting point and softening of the composites. This change in the mechanical properties of composites is apparently associated with a change in the physical state of the amorphous regions, that is, their transition from a highly elastic state to a glassy state.

The results of mechanical tests of LDPE and PVA composites with multiwalled nanotubes (MWCNTs) are interesting. Electromicroscopic studies have shown that MWCNTs are tortuous and intertwined tubes of different lengths, that is, they have a felt-like structure [9]. When interacting with a polymer, they can be one of the reasons for a change in the state of amorphous regions, that is, their transition from a highly elastic state to a glassy state and vice versa. Figure 4a shows the deformation curves  $\sigma(\epsilon)$  of the LDPE+DND system obtained from the melt, and Figure 4b shows the same curves for LDPE+DND obtained from solutions in BrB.

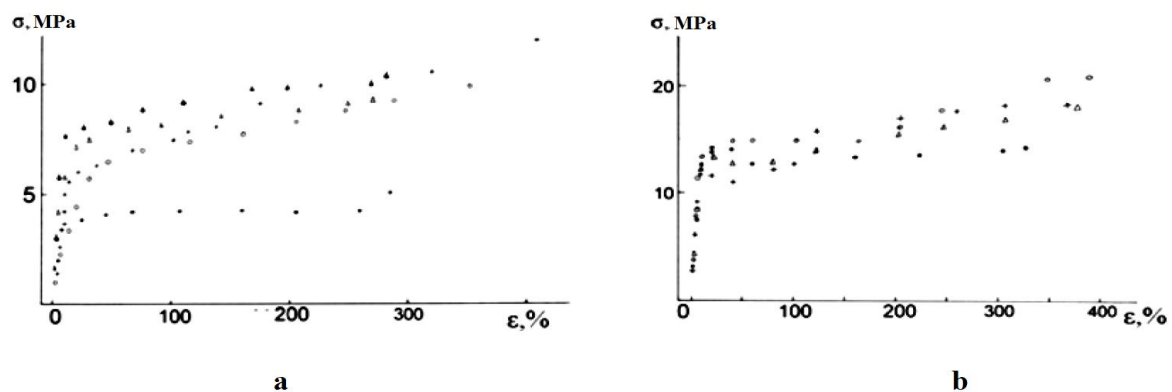


Fig.4. Stress-strain curves of LDPE+NA (a) obtained from the melt: + - initial,  $C=0$ ;  $\circ$  – 1;  $\Delta$  – 3;  $-$  5;  $\bullet$  – 10% wt. ON THE; LDPE+NA (b), obtained from a solution in BrB:  $\bullet$  – initial,  $C=0$ ;  $\circ$  – 0.1; + - 0.5;  $\Delta$  – 1% mass.





## Conclusion

It can be seen that the  $\sigma(\varepsilon)$  curves for both samples are of the same type. With an increase in the DND concentration in the range  $C = 0.1-3$  mass %, mechanical properties improve and/or remain unchanged - strength and deformability, and at concentrations  $C > 3\%$  there is a slight decrease in the strength, deformability and yield strength of the samples. An essential point is that at the maximum concentrations of ND used, the samples do not lose their strength and elasticity life, as was observed in the case of fullerene-containing polymers [9]. From a comparison of the results of studies of the mechanical properties of composites, it follows that the use of multilayer nanotubes and nanodiamonds as fillers is much more effective in comparison with fullerenes, since they allow maintaining the strength and elasticity of composites. Thus, the brightness of the manifestation of changes in the properties of polymer nanocomposites depends on their chemical structure, the presence of side substituents of different nature, molecular and supramolecular morphology of polymers and fillers, their activity, polymer production technology, etc.

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