Temperature variations at the interface in epoxy polymer nanocomposites with unoxidized multilayer graphene particles

Sigareva N.V., Gorelov B.M., Mistchanchuk O.V., Shulga S.V.

Chuiko Institute of Surface Chemistry of the NAS of Ukraine. General Naumova str., 17, Kyiv-03164.
Ukraine.
E-mail: microft2@ukr.net

ABSTRACT

Polymer composites with graphene distinguish the possibility of producing materials combining significant with high electrical and thermal conductivity. Such a set of physico-mechanical parameters will make composite materials with graphene the functional materials in many spheres of industrial production. The filling of the polymer with graphene particles, varying the kinetic parameters of the composites, also affects their thermal stability. But the mechanism of destruction of polymers filled with graphene remains insufficiently explored and understood. Heat processes at the interface in epoxy resin composites with unoxidized multilayer graphene nanoparticles in the interval of low loadings with multilayered nanoparticles at a content of C ≤5% and temperatures of thermal decomposition of the bulk of the epoxy polymer T ≥ 250 °C have been considered. It has been established that the introduction of multilayer graphene particles with sizes of basal surface up to 5 μm and a lateral surface of up to 50 nm leads to a reduction in the energy of thermal destruction of the polymer structure and simultaneously to an increase in the thermal stability of the epoxy matrix.

EXPERIMENTAL

In the present work Epoxy resin composites with unoxidized graphene were studied upon loading 1.0, 2.0 and 5.0% by weight. Unoxidized graphene was prepared by electrochemical method [1]. Graphene particles were blocks consisting of several dozen loosely bound graphene layers, where the side surface of the particles was chemically active. The specific surface area of graphene is ~ 790 m^2/g. Composites with unoxidized graphene, in order to avoid oxidation in air, were prepared by pouring a suspension with graphene particles into the liquid resin. Oxidation of unoxidized graphene occurs in the liquid matrix during its solidification.

The electronographic study showed the so-prepared low-dimension particles to be really multilayered graphene nanoplatelets of about 5×5 μm in-plane dimensions and 50 nm in thickness (Fig. 1). The X-ray diffraction analysis showed that the multilayered nanoplatelets contain graphene sheets (Fig. 2).
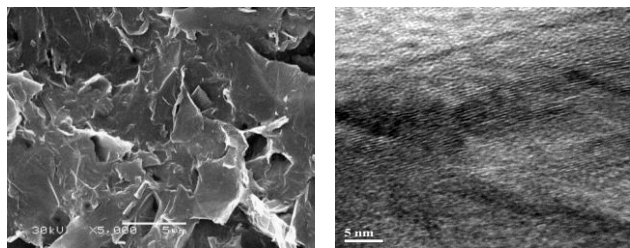

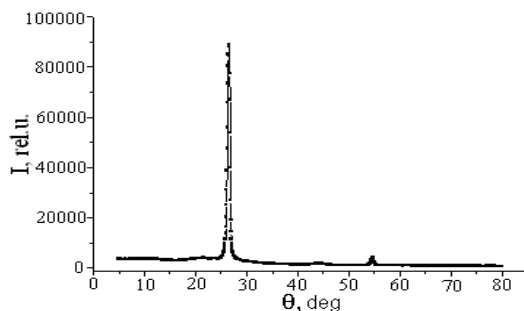**Fig. 1.** SEM images of basic and lateral surfaces of graphene particles

Fig. 2. X-ray diffraction analysis of graphene particles

Studies of the thermal destruction of composites were performed by the method of temperature-programmed desorption with masspectrometric recording of volatile fragments of destruction. Mass spectrometric analysis of atomic products is performed in the range 10-200 *m/z* (*m* is the mass, *z* is charge of the fragment), at a temperature range of 25-800 °C at a pressure of 0.1 Pa and a temperature change rate was 8 °C/min.

RESULTS

Energy selection $\Delta E \sim k \Delta T_e$ (*k* is the Boltzmann constant, ΔT_e is the change in the temperature of the electronic subsystem) occurs at the interface and gives rise to a decrease in the energy of the polymeric phonon subsystem by ΔE and the temperature of the polymer structure at the interface region by ΔT_e. The heat removal from the phonon subsystem of polymer structure to the graphene electron subsystem, which is not involved in the process of thermal decomposition of the matrix, leads to the growth in the composite thermal stability.

Estimates of ΔT_e values from thermal desorption curves of epoxy polymer for thermal decomposition fragments with different *m/z* indicates that ΔT_e reduces with increasing fragment mass. The ΔT_e values for fragments with *m/z* in the range of 17 - 95 are in the interval of 17 - 36 K.

Value of Δ_i is next

$$\Delta_i = T_i (I_c) - T_c(I_c)$$

Estimations give the values $\Delta \approx 34$, 28.5, and 17 ^{o}C for fragments with *m/z* 17, 43, and 94 (Fig.3). Thus, when mass of fastened atomic fragments increases, Δ and the amount of transferred heat to the graphene electronic subsystem reduce.
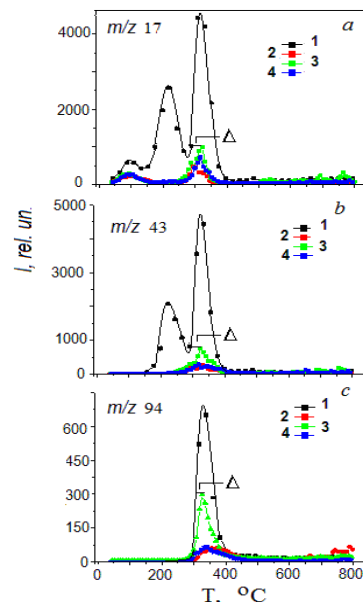

Fig. 3. Temperature dependences of the desorption intensity of volatile fragments with *m/z* 17 (*a*), 43 (*b*), and 94 (*c*) at thermal destruction for neat epoxy (1) and its composites with 1(2), 2 (3) and 5% (4) multilayer graphene particles

SUMMARY

It may be assumed that the growth in the thermal stability of the composite can be related with the removal of a portion of the heat energy coming from an external source through polymer chains to the surface of graphene nanoparticles by the graphene electron subsystem.

REFERENCES

[1] Z. Y. Xia , S. Pezzini , E. Treossi , G. Giambastiani , F. Corticelli , V. Morandi , A. Zanelli , V. Bellani , and V. Palermo // The Exfoliation of Graphene in Liquids by Electrochemical, Chemical, and Sonication-Assisted Techniques: A Nanoscale Study. Adv. Funct. Mater. 2013, DOI: 10.1002/adfm.201203686.