

PROPERTIES OF FULLERENE- AND NANODIAMOND-CONTAINING COMPOSITES BASED ON AROMATIC POLYAMIDE NOMEX

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Introduction

Aromatic polyamides such as NOMEX belong to "superplastics" and are promising for engineering of heat-resistant and high-strength materials. The progress of modern polymeric materials, including polyamides, is based on the development and application of polymeric composites and nanocomposites which are widely used in tribology, biomedicine, hydrogen power engineering, space technologies (including Stealth technology), etc.

In the present work novel composite materials, NOMEX/nanostructured carbon, were created and characterized by EPR and photoluminescence (PL) spectroscopy. The structure of the composites, their magnetic properties, a role and characteristics of defects in the materials are discussed.

Results and discussion

As the initial polymer matrix we used NOMEX –linear heterocyclic co-polymer containing in the main macromolecule chain amide group HNCO-linked from both sides by phenyl fragments. The polymer matrix was obtained by means of condensing dichloranhydride of terephthalic acid with the mixture of p- and m- phenylenediamines taken in equimolar ratio. For preparation of composites, the mixture was homogenized in the vortex layer apparatus in which the components were mixed by non-equiaxial ferromagnetic (FM) particles placed in a rotary electromagnetic field [1]. After ending of the process FM particles were extracted using magnetic separation. Then the composition was pelleted and placed into the press mold where it was maintained in a plastic condition at $T \approx 598$ K during 5 minutes without pressure and 10 minutes under pressure of $P=40$ MPa. At the final stage the material was purified.

In the initial NOMEX polymer $T=300$ K two types of EPR signals were revealed, a narrow line ($g \approx 2.004$, $\Delta H_{pp} \approx 10$ G) and a broad one ($g \approx 2.23$, $\Delta H_{pp} \approx 400$ G) (lines 1 and 2 in Fig. 1, respectively).

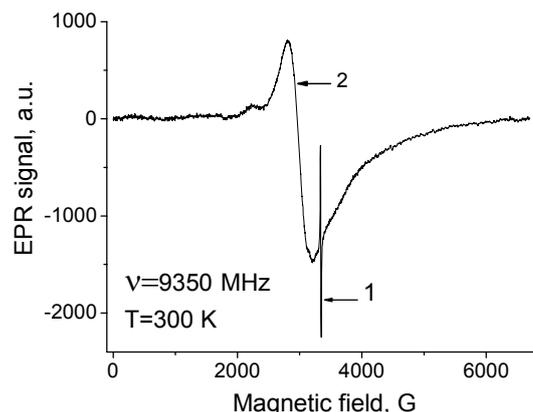


Fig. 1. EPR spectrum of polyamide NOMEX.

In NOMEX based nanocomposites the magnetic resonance response is essentially modified. New EPR signals caused by presence of filling compound are registered (Fig. 2 and Fig. 3). The values of g -factors of the EPR lines observed in composites coincide entirely with that for the initial filling compound, $g=2.0024-2.0027$.

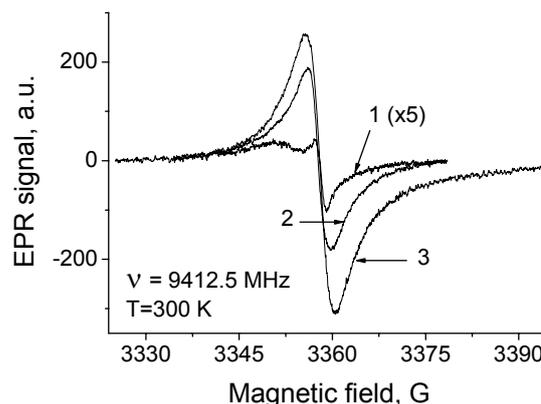


Fig. 2. EPR spectra of composites NOMEX+3% fillers, notably fullerene C_{60} (1), fullerene black (2) and fullerene soot (3). The supplementary broad line in the spectrum (1) belongs to NOMEX.

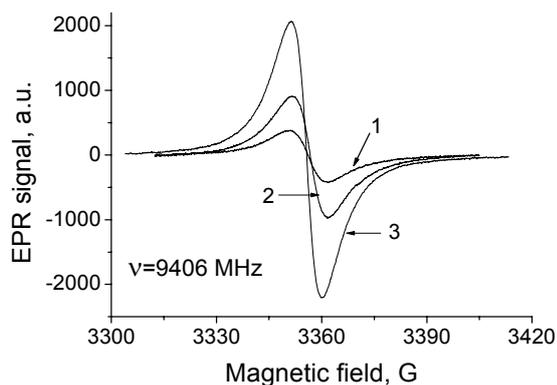


Fig.3. EPR spectra of NOMEX/ultradisperse diamond (UDD) composite. Diamond content is (1) 0.2%, (2) 0.5%, and (3) 1%. T=300 K.

The EPR signals are caused mainly by carbon dangling bond defects. Concentration of paramagnetic centers (PC) N_s and EPR linewidth ΔH_{pp} depend on specific filler type.

Minimal $N_s \approx 10^{16} \text{ cm}^{-3}$ occurs for composite NOMEX/fullerene C_{60} (Fig.2, curve 1). The values of ΔH_{pp} are 1.9 G, 3.7 G and 4.5 G for composites based on fullerene C_{60} , fullerene black and fullerene soot, respectively (Fig.2). In addition, the effect of EPR line broadening occurs in the fullerene-based composites as compared with initial filler components. This makes it possible to estimate matrix-filler interaction in the composite.

In composites based on ultradisperse diamond (UDD) maximal $N_s \sim 10^{20} \text{ cm}^{-3}$ caused by carbon dangling bonds [3] is observed (Fig. 3). It is due to small sizes of crystallites ($d \sim 4 \text{ nm}$ [3]) and large "diamond" surface in the sample. The linewidth $\Delta H_{pp} \approx 10 \text{ G}$ that observed (Fig. 3) is formed by dipole and exchange interactions in paramagnetic system i.e. depends on N_s . Integral intensity of EPR signals in the composites is proportional to mass of entered filler (Fig. 3) and therefore allows estimating quantitatively its content in a composite.

The broad absorption lines connected with nanoparticles of iron oxides are also present in all the composite samples. Strong photoluminescence was observed in the composites as well as in NOMEX (Fig.4). Maximal PL intensity was registered in NOMEX/UDD composites.

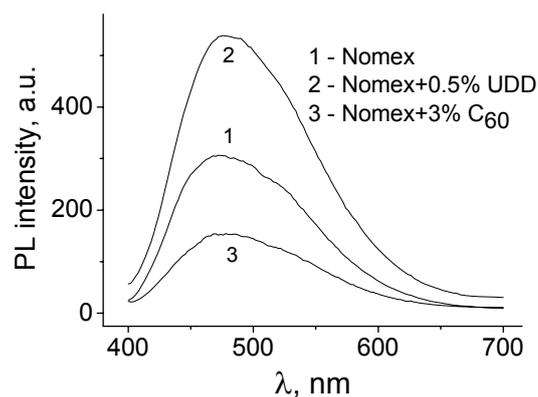


Fig. 4. PL spectra for the initial polymer NOMEX and its composites. Excitation wavelength 375 nm, T=300 K.

Conclusions

1. Novel composite materials based on aromatic polyamide NOMEX and nanostructured carbon are created.

2. Magnetic resonance response in the systems studied is caused as by defects in polymeric chains of NOMEX molecules and superparamagnetic nanoparticles of iron oxides in the initial polymer well as by PC connected with presence of the filler.

3. Analysis of ESR and PL properties allows to determine a fraction content of the filler in the composite and to characterise matrix-filler bonds in the composite.

References

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