Experimental investigations of mechanical and electrical characteristics of a nanomodified epoxy resin DER-330

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Abstract. Technique preparation of a composite material based on epoxy resin DER-330 and 5 different nanopowders has been developed. The modifiers exert a positive effect on the elastic modulus, flexural strength, and dielectric permittivity of the epoxy-anhydride polymers. Weight content of nanoadditives in the matrix ranged from 0% to 4%. Dependences between mechanical properties of epoxy composites and concentration and nature of the filler were investigated in a three-point bending. Morphology of the fracture surface was studied. The complex analysis of nanofillers allowed us optimum fillers and concentrations when a significant increase of elastic and strength characteristics is achieved. It was showed that addition of silica fume nanoparticles A-380 to the matrix increases fracture stress on 30%. Composites with nanoparticles of aluminum nitride showed the best mechanical values in case of jointly investigated parameters. Growth of the elastic modulus is 12 % and increase of the fracture stress is 7 %. Adding alumina nanoparticles and nanofibers in the range of investigated concentration does not lead to significant changes in the mechanical characteristics of the composite.

1. Introduction
The Epoxy-anhydride polymers are widely used as matrixes for modern composite electrical insulating materials [1]. These substances have great potential for the targeted modification of their properties and, therefore, are of an increasing interest both in scientific research and in engineering applications [2]. Not only the type of resin and the nature of a curing agent can be varied, but also a huge number of organic and inorganic modifiers and fillers can be added to improve the material characteristics. Modifications of the epoxy polymers are directed, first of all, at mechanical properties and dielectric properties. The reason for a significant improvement in the properties is presumably associated with a change in the structure of epoxy matrix near the filler surface [3, 4]. The dynamic development of industrial sectors is impossible without permanent improvement of materials. The use of composite materials (CMs) instead of the traditional ones allows significant reduction of the product weight without deteriorating the strength characteristics, which is particularly important for the aviation and aerospace industry. In addition, CMs may often have unique properties that are not inherent in traditional materials, which makes them indispensable for applications in certain areas of engineering.

This paper deals with the research of physical and mechanical characteristics of nano-modified epoxy composite materials, which are widely used in the construction industry as protective impregnations and coatings that increase the carrying capacity, chemical resistance, and durability of building structures. The goal of this work is to develop a method of epoxy resin binder modification...
by nanoparticles and a technology of obtaining the corresponding composite materials with improved performance.

2. Experimental
A polymeric matrix for the nanocomposite material was made from D.E.R. 330 epoxy resin (Dow Chemical Company, epoxy value 22.5), isomethyltetrahydrophthalic anhydride (IMTHPA) hardener (TU 8.103149 85), and benzylidimethylamine (BDMA) catalyst. Fillers used hydrophilic powders silica A380 (E1) and A200 (E2), differing particle sizes, powders of an aluminum oxide in the form of spherical particles (E3) and nanofibers (E4), and a powder of aluminum nitride (E5).

Dispersing powder in the resin was carried out using an ultrasonic generator Bandelin Sonopuls HD 3200. Stability of the suspension was monitored visually before and after curing. Curing was carried out in a stepwise mode, similar to that used in [5]. The completeness of the curing was monitored by IR spectroscopy.

Strength characteristics were studied in a three point bending mode. Experiments were conducted on a Zwick/Roell Z005 material testing machine according to ASTM D 790 testing standard. Rectangular samples (4 × 10 × 80 mm$^3$) were produced for the experimental using the technique described below. The loading (three point bending with unloading) was set by moving the mobile crosshead with a constant rate of 5%/min, deformation was measured by a crosshead motion sensor and the applied force was measured by an integrated force sensor. The distance between supports is 65 mm. The experiments were performed on up to 8% of deformation. The average test data of at least five samples are given. The error in the determination of the elastic modulus does not exceed 3% and that of tensile is no higher than 10%.

Figure 1 shows the concentration dependence of the Young's modulus of heterogeneous material with a variety of fillings. Judging schedule dependence of Young's modulus of the filler concentration for compositions with powders of silicon oxide (E1, E2) and aluminum oxide (E3) is the same. At a concentration of 0.2% to 1.2% modulus increases, reaching a maximum and then gradually decreases. Maximum increase of 7% is realized by using Al2O3 additive at a concentration of 0.8%. The dependence of the composition of the powder of aluminum nitride (E5) differs from others modulus monotonically increases and reaches a maximum at a concentration of 3.2% (E = 3890 MPa, an increase of 12% compared to baseline).

![Figure 1. The dependence of the Young's modulus at the three-point bending from the weight concentration of fillers](image)

Figure 2 shows graphs of the concentration of stress fracture powders. In a heterogeneous material containing particles of E1, E4, E5, realized higher stress at fracture over the whole range. The greatest
positive influence nanosized powder silica A-380 (E1). Fracture to stress 155 MPa base composition, adding 4% A-380 is increased by 30%. Powders E2 and type E3 have a strengthening effect on the epoxy composition at a concentration of 0.4-1.2%.

![Figure 2. Dependence of fracture stress with three-point bending of the weight concentration of powders](image)

2.1. The capacity and dielectric constant epoxy compositions
Compositions of epoxy resins with hardeners anhydrides are mainly used as a base insulating compounds. Determination of dielectric characteristics carried out for the samples containing alumina nanofibers at a concentration of 1.8 and 3.2%. Sample sizes - 5x5 cm, thickness 0.15 cm. Capacitance measurement carried out on the digital bridge R5058 at a frequency of 10 kHz. The measurement results are shown in Table 1.

| Parameters            | Epoxy resin without filler | Epoxy resin with alumina nanofibers |
|-----------------------|----------------------------|-------------------------------------|
| Concentration         | 45                         | 44                                  |
| Capacitance           |                            | 42.2                                |
| The dielectric constant | 3.05                      | 2.98                                |

It was established that the additive alumina nanofibers improves the capacitance and dielectric constant epoxy compositions. Previously it demonstrated that the filler does not impair the Young's modulus and fracture stress. Collectively, the data developed promising heterogeneous characterize materials based on epoxy resin for encapsulation of electrical equipment.

3. Conclusions
Epoxy nanocomposites based on polymer matrix DER330 + IMTHPA and five different nanofillers were obtained under the same conditions. The influence of the nature and amount of inorganic additives on the mechanical properties of epoxy anhydride nanocomposites was investigated. It has been established that the incorporation of silica nanoparticles at an amount of 0.2–1.2% of the weight of the binding agent leads to an increase in Young’s modulus. Additives of silica A 380 with a particle size of 7 nm have the greatest effect on the fracture stress: a 30% increase over the base composition.
The addition of aluminum oxide nanoparticles and nanofibres does not lead to significant changes in the mechanical characteristics of the composite in the investigated concentration range. While alumina nanofibers additives improve the capacitance and dielectric constant epoxy compositions. Composites with aluminum nitride nanoparticles showed the best mechanical values on the strength of all investigated parameters: the growth in the elastic modulus is 12% upon an increase in the fracture stress by 7%.

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