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THE EFFECT OF THE MODIFICATION OF AN EPOXY RESIN BY LIQUID OLIGOMERS ON THE PHYSICAL-MECHANICAL PROPERTIES OF COMPOSITES

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The effect of polyurethane and silicone rubbers as modifiers providing an increase in the toughness of ED-20 (cold curing) and Etal-Inject-T (hot curing) epoxy resins is investigated. The ED-20 resin has an initial strength of 97 MPa and the impact strength of 20.1 kJ/m^2, while Etal-Inject-T resin has an initial strength of 106 MPa and the impact strength of 42.34 kJ/m^2. It is established that polyurethane and silicone rubbers equally affect the strength properties of the epoxy resins. The polyurethane rubber was found to be a more effective modifier. The addition of polyurethane rubber to ED-20 resin leads to an increase in impact viscosity, however a slight decrease in the compressive strength is observed. An optimal content of polyurethane rubber in ED-20 resin is about 5%; this ensures a twofold increase in the impact viscosity, the compressive strength being decreased insignificantly (only by 14%). With an increase in the content of modifiers in the ED-20 resin (more than 5%) results in a slight increase in the impact viscosity, however the reduction of the compressive strength becomes significant. The addition of 6.5% of polyurethane rubber to Etal-Inject-T resin causes an increase in both the impact viscosity (by 67% up to 71 kJ/m^2) and the compressive strength (by 7.5% up to 114 MPa). When the content of the modifiers in Etal-Inject-T is more than 6.5%, the impact viscosity remains the same, but the compressive strength begins to diminish. An increase in the impact viscosity of the material can be attributed to the formation of a rubber's dispersion phase, which seems to loosen the rigid structure of a cured epoxy resin. The microstructures of the epoxy resin samples modified by polyurethane and silicon rubber were also investigated.

Keywords*:* carbon fiber reinforced plastic, epoxy resin, modifier, liquid oligomer, impact viscosity, compression, strength.

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Introduction

The carbon fiber reinforced plastic is a composite material consisting of a binder matrix and reinforced carbon fibers. It possesses such good properties as lightness, high values of strength and modulus of elasticity, low coefficient of thermal expansion, enhanced vibration and fatigue strength. Nevertheless, carbon fiber reinforced plastic is still not widely used in space rockets production. This is due to the high cost of this material as well as its weakness against shocks and chipping. The carbon fiber reinforced plastics of the first generation turned out to be very fragile. In addition, up to date they are used where the material is exposed only to the static loads. Table 1 presents the comparative characteristics of different materials, including some metals and carbon fiber reinforced plastics. It is clearly seen from Table 1 that the main problem of

Table 1

Comparative characteristics of different materials

Material	Strength-weight ratio, $\times10^{-3}$, m^2/s^2	Impact viscosity, kJ/m^2
Aluminium alloy AMg-6	1.2	400
Duralumin D16	1.6	250
High-resistance steel	1.8	400–1300
Titanium alloy	2.8	300–500
Carbon fiber reinforced plastic	4.0	20

carbon fiber reinforced plastic is a low impact viscosity (i.e. impact strength), which reduces its competitive capacity as compared with metal alloys.

Nowadays, the search for the ways to increase significantly the impact viscosity of carbon fiber reinforced plastics and maintain the achieved level of static capacity is a topical problem. Some impressive results were reported: the impact viscosity was increased by 15–110% [1,2]. This creates prerequisites for the use of impact-modified types of carbon fiber reinforced plastics in the manufacture of aircraft industry, space rocketry, etc.

One of the methods to increase the impact viscosity of carbon fiber reinforced plastics is to increase the impact viscosity of their matrix, the epoxy resin (ER).

The impact strength of the epoxy resins can be enhanced by the introduction of modifiers into primary carbon plastics. Various types of thermoplastics, plasticizers, liquid oligomers, carbon nanotubes, etc. are often used as modifying additives. The application of modifiers allows changing controllably the structure of epoxy composites, which largely determines the strength characteristics [3–13].

Currently, significant success has been achieved in the modification of ER, both by thermoplastics with reactive groups and by physically mixed systems. These systems include epoxy resins modified with polyetherimides (PEI), polysulfones (PSF), polyethersulfones (PESF), polyphenylene oxide (PPO) and polycarbonate (PC) [3,4]. The effect of thermoplastic modifiers PSF and PESF on the strength characteristics of hardened epoxy materials has been investigated [5]. The modifier (5, 10, and 20 wt.%) and modifier mixtures (2.5 to 7.5; 5 to 5; and 7.5 to 2.5 wt.%) in the form of granules were added to the epoxy oligomer at the temperature of 100–105°C. The impact strength was considerably increased (from 24.9 to 75.7 kJ/m^2 at the PSF content of 20%). The reinforcing effect was explained by the structure of the material, in particular the two-phase structure resulting from the curing.

Many authors have detected an increase in the impact viscosity of ER when it was modified by plasticizers: diglycidyl ether diethylene glycol (DEG-1), tricresyl phosphate (TCP) and trichloroethyl phosphate (TCEP) [5–7]. Mostovoy et al. investigated the ER of ED-20 type modified with tricresyl phosphate (TCP) and APP-2 ammonium polyphosphate filling material [6]. The plasticized filled formulations showed an increase in the impact viscosity by 1.6–1.8 times. This effect was explained by the high adhesion of epoxy resin and plastic filler particles.

The effect of carbon nanotubes (CNTs) on the strength properties of ER was studied [5–7,9] and an increase in the impact viscosity of ER from 16 kJ/m^2 to 97 kJ/m^2 under the action of introduced 0.01 wt.% of CNT was observed. The modification of ER by some liquid oligomers, butadiene rubber, silicone rubber, polyurethane rubber and butadiene-styrene rubber, resulted to an increase in the impact viscosity by 1.8 times, 1.5 times, 15-50%, and 1.6 times, respectively [7–10].

Two brands of ER are widely used for the production of composite materials: cold curing ED-20 and hot curing Etal Inject-T. It is important to increase the impact strength of these ERs. To this end, generally available silicone and polyurethane rubbers can be used.

Thus, the aim of this work was to study the effect of polyurethane and silicone rubbers modifiers on the impact viscosity and strength of the following epoxy resins: cold curing ED-20 and hot curing Etal Inject-T.

Experimental

Two types of epoxy resins and liquid oligomers were used in this study. Their characteristics are described below.

Resins

ED-20 resin and Etal Inject-T compound were used as an epoxy base of the systems under investigation.

ED-20 epoxy-diane resin (National State Standard 10587-84) is a reactive oligomeric product based on the diglycidyl ether of diphenylolpropane. It can be treated at indoor temperature and gives high strength at curing. An amine hardener PEPA is used for its curing, which is a composition comprising a certain amount of ethylene polyamines, from diethylenetriamine to hexaethyleneheptamine and their piperazine-containing analogues. The ratio is 15 wt.% of PEPA (with respect to the weight of resin). The curing mode was observed at a temperature of 20–25°C.

Etal Inject-T: these hot cured epoxy compounds are modified epoxy resin (component A) and modified hardener of amine type (component B). The ratio of the resin to curing part is 100:49.9 (in wt.%). The curing mode lasted 4 hours at 150°C and then 1 hour at 180°C.

Liquid oligomers

Polyurethane rubber is a two-component compound based on polyurethane with a unique combination of the following properties: high strength, wear-resistance, impact resistance, low glass transition temperature and preservation of highly elastic properties in a wide range of the temperatures. The general characteristics are given in Table 2.

Table 2

Characteristics of the cured polyurethane compound

Type	Silagerm 5045
Shore A hardness	40
Ratio (by weight)	2A/1B
Color	Beige
Hardening time, hour	24
Density, g/cm^3	1.07
Viscosity, cP s	3000–3500
Elongation at break, %	400–600
Breaking tenacity, MPa	3.5–5.0

SKTN-A silicone rubber is a low molecular weight OH-based silicone rubber. The materials based on SKTN-A rubber have good elasticity and springiness, high hydrophobic properties, chemical inertness, dielectric properties, and resistance to the action of ozone, oxidizing substances UVR and vibrations. The general characteristics are shown in Table 3.

Table 3

Characteristics of SKTN-A rubber

Physical configuration	Transparent liquid
Type of SKTN-A rubber	A
Flame temperature, ^{0}C	200
Content of OH, ppm	1530
Viscosity at 23^{0}C, mPa s	2000
Density at 25^{0}C g/cm^3	0.98

The impact strength of epoxy resin was determined by the Charpy test in accordance with the National State Standard 4647-2015. This standard applies to plastics and establishes a method for determination of Charpy impact strength using notched and non-notched samples. The established method is used to evaluate the characteristics of test samples under the action of shock stresses and determine the brittleness or viscosity of the samples within the limits established by the test conditions. The impact-viscosity tests of the ER were carried out by using the MK-30A Charpy impact machine. For the impact resistance test of epoxy rubber, a non-notched type of sample was selected (Fig. 1).

To get samples of required dimensions (with the length (l) of 80 mm, the width (b) of 10 mm and the thickness (h) of 4 mm), a curing mold made of aluminum was used. The LOCTITE 770-NC antiadherent was pre-applied to the walls of the mold before pouring to prevent the sample from sticking; then it was dried for 30 minutes. The cured samples were adjusted to exact dimensions on a SAPHIR 320 E grinding machine.

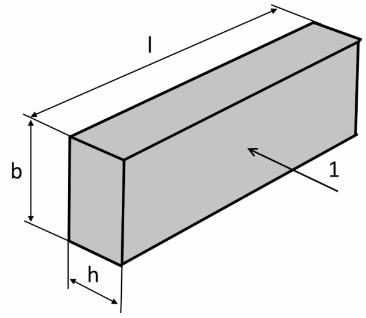

Fig. 1. Sample for impact-viscosity test

After carrying out the Charpy impact tests, the impact viscosity of the material was calculated based on the obtained data of the energy spent on the destruction of the samples.

The impact viscosity of the samples without notching (A_n) in kJ/m^2 was calculated by the following formula:

$$A_n=A\cdot10^3/(b\cdot h),$$

where A is the impact energy spent on the destruction of the sample without notching (J), b is the width of the sample at its middle (mm), h is the thickness of the sample at its middle (mm).

Results and discussion

The primary objective of this work is to increase the impact viscosity of epoxy resin with preservation of compressive strength. The test results of the samples of ED-20 and Etal-Inject-T resins with the addition of liquid oligomers are shown in Figs. 2 and 3. As can be seen, the liquid oligomers are

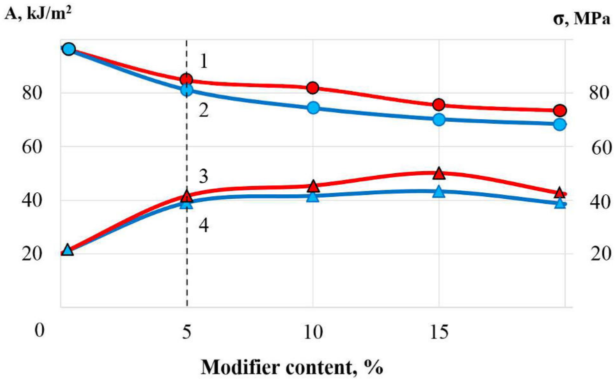

Fig. 2. Dependence of strength and impact viscosity on the content of modifier in ED-20 ER: 1 and 2 – compressive strength of ER with polyurethane and silicone SKTN-A rubber, respectively; 3 and 4 – impact viscosity of ER with polyurethane and silicone SKTN-A rubber, respectively

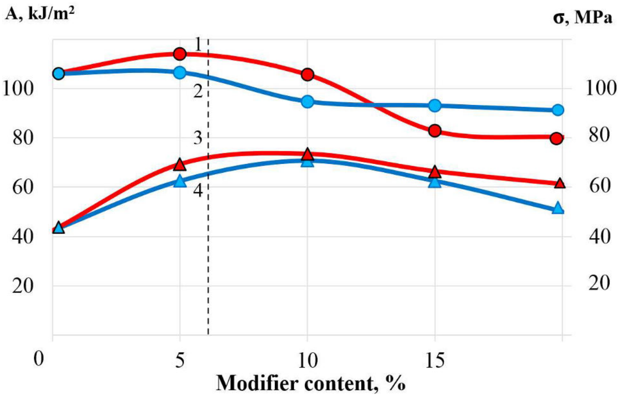

Fig. 3. Dependence of strength and impact viscosity of the content of modifier in Etal-Inject-T ER: 1 and 2 – compressive strength of ER with polyurethane and silicone SKTN-A rubber, respectively; 3 and 4 – impact viscosity of ER with polyurethane and silicone SKTN-A rubber, respectively

effective modifiers that increase the impact viscosity of a resin. However, a decrease in the compressive strength is observed with an increase in the content of modifiers (Fig. 2).

It follows from Fig. 2 that the addition of polyurethane rubber to the ED-20 resin leads to an increase in the impact viscosity, but also a slight decrease in the compressive strength is observed. An optimum content of polyurethane rubber in ED-20 resin can be adopted as 5%, at which the impact viscosity is increased by 2 times with a slight loss of the compressive strength (by 14%). There is a slight increase in the impact viscosity with an increase in the content of modifiers in the ED-20 resin (>5%); however, a decrease in the compressive strength becomes significant.

At the same time, as can be seen from curves 1 and 3 in Fig. 3, the addition of 6.5% of polyurethane rubber to Etal Inject-T contributes to the simultaneous increase in the impact viscosity (from 42.34 kJ/m^2 to 71 kJ/m^2) and compressive strength (from 106 MPa to 114 MPa). Curves 2 and 4 in Fig. 3 show similar behavior for the resin that was cured by 6.5% of SKTN-A. The addition of 10% of polyurethane rubber causes an increase in the impact viscosity with the preservation of compressive strength (Fig. 3, curve 2). The modification of SKTN-A silicone rubber results in an increase in the impact viscosity by 67% (Fig. 3, curve 2) and a decrease in the compressive strength by 28% (Fig. 3, curve 4).

A comparative analysis of the strength properties revealed that the introduction of modifiers into the Etal-Inject-T resin provides a greater hardening effect than that in case of ED-20. This is connected with the fact that the molecules are embedded in the grid structure of the resin with the introduction of liquid oligomers; thereby the elastic phase changes the destructive nature of the glassy matrix reducing the fragility. The particles of liquid oligomer regulate the deformation in the matrix by providing concentrations at local stresses, which leads to an increase in the strength and impact absorbing properties of epoxy resin.

The obtained characteristics well correlate with the features of the microstructure of the Etal-Inject-T ES samples surface when it is modified by liquid oligomers (Fig. 4). Figure 4,b shows the structural inhomogeneity with small inclusions of modifiers as compared with initial ER matrix (Fig. 4,a). This is probably due to the incomplete dissolution of the components in the resin and the presence of air-bubble pores formed during the curing process of the ER with the SKTN-A oligomer. This affects the compressive strength properties. The features of the chemical interaction of components could be the reason for this. Figure 4,c shows the equilibrium distribution of the polyurethane rubber phase. The inclusions of air are absent (as in the case of SKTN-A). The presence of conglomerates was found in the samples of SKTN-A silicone rubber. A similar effect appears at the addition of more than 5% of SKTN-A, which negatively affects the strength (compression) of the ER. In order to eliminate these defects, it is expedient to perform the heat treatment of each component separately.

It was determined that the issue of component compatibility is of great importance when ER was modified by modifiers. As a rule, the liquid oligomers

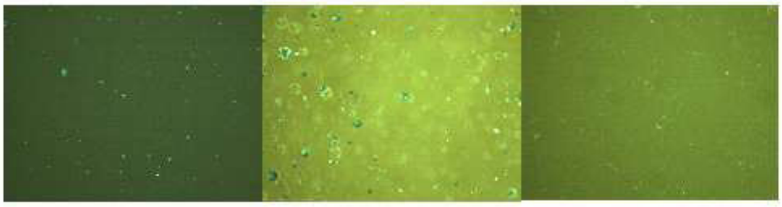
a b c

Fig. 4. The microstructure of the Etal-Inject-T ES samples surface with liquid oligomers (×200): a – epoxy resin; b – 5% of SKTN-A silicone rubber; and c – 5% of polyurethane rubber

are dissolved in epoxy resin. The process begins with the dissolution of unstructured liquid oligomer in a low molecular weight liquid, which is further polymerized. During this reaction, the rubber is released in the form of dispersed phase with the formation of chemical bonds between rubber and epoxy resin. The dispersed phases of rubber thereby loosen the hard and brittle structure of the cured epoxy resin, leading to an increase in its impact resistance.

Conclusions

The cured epoxy resins in initial sate have the following characteristics: Etal-Inject-T exhibits the compressive strength of 106 MPa and the impact viscosity of 42.34 J/m^2, whereas ED-20 resin shows 97 MPa and 20.1 kJ/m^2, respectively. The modifiers, polyurethane and silicone rubbers, similarly affect the strength properties of epoxy resins. The polyurethane rubber is found to be a more effective modifier. The addition of the polyurethane rubber to ED-20 resin leads to an increase in the impact viscosity and a slight decrease in the compressive strength. The optimal content of polyurethane rubber in ED-20 resin can be adopted as 5%, at which the impact viscosity is increased by 2 times with an insignificant loss of the strength (by 14%). The addition of 6.5% of polyurethane rubber to Etal-Inject-T resin yields an increase in both the impact viscosity by 67% (up to 71 kJ/m^2) and the compressive strength by 7.5% (up to 114 MPa). The effect of an increase in the impact viscosity of the material can be attributed to the formation of a rubbers dispersion phase, which seems to loosen the rigid structure of a cured epoxy resin.

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ВПЛИВ МОДИФІКАЦІЇ ЕПОКСИДНОЇ СМОЛИ РІДКИМИ ОЛІГОМЕРАМИ НА ФІЗИКО-МЕХАНІЧНІ ХАРАКТЕРИСТИКИ КОМПОЗИЦІЇ

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Досліджено вплив поліуретанового та силіконового каучуків як модифікаторів підвищення ударної в'язкості епоксидної смоли ЕД-20 холодного твердження та Етал-Інжект-Т гарячого твердження. Смола ЕД-20 має початкову міцність 97 МПа та ударну в'язкість 20,1 кДж/м^2, а смола Етал-Інжект-Т – відповідно 106 МПа и 42,34 кДж/м^2. Встановлено, що поліуретанові та силіконові каучуки, якісно однаково впливають на властивості епоксидних смол. Більш ефективним модифікатором є поліуретановий каучук. Введення поліуретанового каучуку до смоли ЕД-20 приводить до підвищення ударної в'язкості, але і до одночасного зниження міцності при *стисканні. Оптимальною кількістю поліуретанового каучуку у смолі ЕД-20 можна вважати 5%, при якій ударна в’язкість збільшується в 2 рази при незначній втраті міцності при стисканні (14%). Встановлено, що зі збільшенням кількості модифікаторів (>5%) у смолі ЕД-20 спостерігається слабке підвищення ударної в’язкості, однак, зниження міцності при стисканні стає значним. Добавка 6,5% поліуретанового каучуку до смоли Етал-Інжект-Т забезпечує одночасне підвищення ударної в’язкості на 67% (до 71 кДж/м^2) та міцності при стисканні на 7,5% (до 114 МПа). При кількості модифікаторів >6,5% в Етал-Інжект-Т величина ударної в’язкості зберігається, але спостерігається падіння міцності при стисканні. Підвищення ударної в’язкості матеріалу можна зв’язати з утворенням дисперсної фази каучуку, яка розпушує структуру затверділої епоксидної смоли. Досліджені мікроструктури зразків епоксидної смоли з модифікованим поліуретановим та силіконовим каучуками.*

Ключові слова*:* вуглепластик, епоксидна смола, модифікатори, рідкі олігомери, ударна в’язкість, стискання, міцність.

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