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INFLUENCE OF CONCENTRATION OF THIOKOL, AMOUNT OF THE HARDENER AND FILLER ON PROPERTIES OF EPOXIDE-POLYSULPHIDE COMPOSITES

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Abstract

A system research of the possibility of regulating the deformation-strength, adhesion and dynamic mechanical properties of epoxy-polysulphide composites based on the products of the preliminary thioetherification reaction (PRTE) by varying the concentration of polysulphide rubber, as well as changing the amount of the hardener and using a mineral filler has been carried out. An increase in the content of rubber in the product of the preliminary thioetherification reaction was found to promote an increase in the characteristics of cohesive and adhesive strength, deformation at break and the work of destruction of the material. An increase in the complex of properties is assumed to result from the formation of higher molecular weight products with an increase in the rubber content in the composition subjected to PRTE. The formation of such products is confirmed by the data of dynamic mechanical spectrometry. It has been shown that by changing the amount of the introduced hardener, i.e., by changing the ratio of amine and epoxy groups, it is possible to effectively regulate the deformation and strength characteristics of epoxy-polysulphide mixtures. With an increase in the content of the hardener, the tensile strength, deformation at break and the work of fracture of the material increase very significantly. It was found that the introduction of a filler into an epoxy system containing a large amount of rubber promotes an increase in cohesive strength with a relatively small decrease in adhesion strength and deformation at break.

Keywords: epoxy resin; polysulphide rubber; preliminary thioetherification reaction; hardener; deformation-strength; adhesion and dynamic mechanical properties; filler

ВПЛИВ КОНЦЕНТРАЦІЇ ТІОКОЛУ, КІЛЬКОСТІ ТВЕРДНИКА І НАПОВНЮВАЧА НА ВЛАСТИВОСТІ ЕПОКСИДНО-ПОЛІСУЛЬФІДНИХ КОМПОЗИТІВ

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Анотація

Проведено системне дослідження можливості регулювання деформаційно-міцнісних, адгезійних і динамічних механічних властивостей епоксидно-полісульфідних композитів на основі продуктів попередньої реакції тіоетерифікаціі (ПРТЕ) варіюванням концентрації полісульфідного каучуку, а також зміною кількості твердника і за допомогою мінерального наповнювача. Встановлено, що збільшення вмісту каучуку в продукті попередньої реакції тіоетерифікаціі сприяє збільшенню показників когезійної і адгезійної міцності, деформації при розриві і роботі руйнування матеріалу. Припущено, що підвищення комплексу властивостей обумовлено утворенням більш високомолекулярних продуктів у міру збільшення вмісту каучуку в композиті на основі ПРТЕ. Утворення таких продуктів підтверджено даними динамічної механічної спектрометрії. Показано, що зміною кількості введеного твердника, тобто зміною співвідношення амінних і епоксидних груп, можна ефективно регулювати деформаційно-міцнісні характеристики епоксидно-полісульфідних композитів. За збільшення вмісту твердника вельми істотно зростають міцність при розтягуванні, деформація при розриві і робота руйнування матеріалу. Встановлено, що введення наповнювача в епоксидну систему, яка містить велику кількість каучуку, сприяє збільшенню когезійної міцності і деформації при розриві.

Ключові слова: епоксидна смола; полісульфідний каучук; попередня реакція тіоетеріфікаціі; отверджувач; деформаційно-міцнісні, адгезійні та динамічні механічні властивості; наповнювач.

ВЛИЯНИЕ КОНЦЕНТРАЦИИ ТИОКОЛА, КОЛИЧЕСТВА ОТВЕРДИТЕЛЯ И НАПОЛНИТЕЛЯ НА СВОЙСТВА ЭПОКСИДНО-ПОЛИСУЛЬФИДНЫХ КОМПОЗИТОВ

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Аннотация

Проведено системное исследование возможности регулирования деформационно-прочностных, адгезионных и динамических механических свойств эпоксидно-полисульфидных композитов на основе продуктов предварительной реакции тиоэтерификации (ПРТЭ) варьированием концентрации полисульфидного каучука, а также изменением количества отвердителя и с помощью минерального наполнителя. Установлено, что увеличение содержания каучука в продукте предварительной реакции тиоэтерификации способствует увеличению показателей когезионной и адгезионной прочности, деформации при разрыве и работы разрушения материала. Предположительно, повышение (улучшение?) комплекса свойств обусловлено образованием более высокомолекулярных продуктов по мере увеличения содержания каучука в композиции, подвергаемой ПРТЭ. Образование таких продуктов подтверждено данными динамической механической спектрометрии. Показано, что изменением количества вводимого отвердителя, т. е. изменением соотношения аминных и эпоксидных групп, можно эффективно регулировать деформационно-прочностные характеристики эпоксидно-полисульфидных смесей. При увеличении содержания отвердителя весьма существенно возрастают прочность при растяжении, деформация при разрыве и работа разрушения материала. Установлено, что введение наполнителя в эпоксидную систему, содержащую большое количество каучука, способствует увеличению когезионной прочности при относительно небольшом снижении адгезионной прочности и деформации при разрыве.

Ключевые слова: эпоксидная смола; полисульфидный каучук; предварительная реакция тиоэтерификации; отвердитель; деформационно-прочностные, адгезионные и динамические механические свойства; наполнитель

Introduction

It was previously shown [1; 2] that in order to increase the adhesive and physicomechanical properties of compositions based on epoxy resin and polysulphide rubber (thiokol), hardened without heat treatment, it is necessary to carry out a preliminary thioetherification reaction (PRTE) before introducing the hardener due to the interaction of mercaptan groups of rubber and oxirane cycles of epoxy resin at an elevated temperature, and then use the product of this reaction to harden at room temperature. It is shown that composite materials based on the products of the thioetherification reaction based surpass analogues significantly mechanical mixtures of epoxy resin and thiokol in terms of cohesive and adhesive strength, deformation capacity, fracture work, and specific impact strength.

In continuation of the research, the purpose of this work is to study the possibility of regulating the deformation-strength, adhesive and dynamic mechanical properties of epoxy-polysulphide composites by changing the concentration of thiokol in the initial product of the preliminary thioetherification reaction, as well as the amount of the hardener and the use of the filler.

Models and methods

The objects of study were Epikote-828 epoxy resin (mass fraction of epoxy groups (EG) 22.6 %, viscosity 12 Pa·s at 25 °C), polysulphide rubber (liquid thiokol grade I with a viscosity of 28 Pa,

content of sulphhydryl groups 3.1%). The combination of epoxy resin and thiokol was carried out at $160\,^{\circ}\text{C}$ for 2 h with thorough stirring. Hardening of the compositions was carried out with diethylenetriamine DETA in two modes: I – $25\,^{\circ}\text{C}/24\,\text{h} + 50\,^{\circ}\text{C}/8\,\text{h}$ (hardening at moderate temperature) and II – $25\,^{\circ}\text{C}/24\,\text{h} + 120\,^{\circ}\text{C}/3\,\text{h}$ (hardening with heat treatment at elevated temperature).

The mechanical properties under uniaxial tension (tensile strength σ_t and deformation at rupture ϵ_s) were determined on a Polyany-type device with a rigid dynamometer and automatic recording of measured values [3]. The deformation rate was $3.83\cdot 10^{-5}$ m/s. The elastic modulus (E) was calculated from the slope of the initial portion of the σ - ϵ curve. The area under the stress-strain curve served as a measure of the fracture work (Af).

The adhesion strength in shear (τ_{sh}) and tear off (σ_{to}) was determined according to State Standards 14759 and 14760, respectively. The surfaces for gluing steel samples (Steel 3) were processed on a grinding plate using electrocorundum to a uniform roughness, then degreased with acetone.

Dynamic mechanical characteristics (dynamic modulus of elasticity E', loss modulus E'' and tangent of the angle of mechanical losses tg δ) were measured on a DMA 983 unit of the thermoanalytical complex DuPont 9900 on 25x4x2 mm samples with heating at a rate of 10°C/min. The rubber elasticity modulus (E_{re.})

was determined at a temperature equal to $T_{\rm g}$ + 50°C, where $T_{\rm g}$ is the glass transition temperature of the polymer.

The molecular weight of the chain section between the nodes of the chemical network (M_c) was calculated by the formula:

$$M_c = 3\rho RT / E_{r.e.}$$

где R refers to the absolute gas constant; T is an absolute temperature; ρ is the density of the polymer.

Density of nodes of the chemical network (n_c) was calculated using the formula:

$$n_c = \frac{\rho}{M_c}$$

The density of the samples was measured by the gradient column method according to State Standard 15139.

Results of research and their analysis

Epoxy materials in technology are usually used' in the form of multicomponent curable compositions containing, in addition to resin and hardener, solvents and plasticizers, active diluents and flexibilizers, fine and granular fillers, dyes and pigments, and other functional additives that impart specific properties [4–9]. In practice, epoxy resins are always modified to improve processing properties and reduce their cost. However, the main goal of epoxy polymer modification is to enhance them [10, 24]. Strengthening implies, first of all, an increase in the mechanical strength, hardness, elastic modulus, wear resistance of the material, i.e., the degree of its resistance to external force effects. In addition, the term "reinforcement of polymers" also includes an increase in resistance to external destructive factors, in particular, chemically and physically active media.

Generally, the methods for modifying crosslinked polymers obtained on the basis of reactive monomers and oligomers can be divided into three groups: chemical, physicochemical, and physical.

The possibilities of chemical modification are extremely large, since dozens of types of epoxy resins and individual hardeners (as well as their mixtures) make it possible to obtain cross-linked polymers with a huge variety of chemical structures. Therefore, chemical modification based on varying the structure of molecules of epoxy resins, and, even more often, on the choice of one or another hardener, is the main way to change the technological and operational properties of binders, matrices, film-forming [10-18]. In technological practice, however, it is necessary (often for economic reasons) to create materials with specified technological and operational properties, using a limited set of industrial epoxy resins (98 % Dian) and several hardeners. Therefore, they often resort to the methods of physicochemical and, less often, physical modification [10; 26]. In the first case, this is plasticization, finely-dispersed filling, combination with liquid rubbers and other polymers [19-23]. The essence of physical modification is the effect of mechanical, electrical and electromagnetic fields on the initial reactive systems in order to change the supramolecular structure of cross-linked polymers [10; 23-26].

In order to further improve the complex of properties of epoxy-thiokol composite materials based on PRTE, we studied the possibilities of regulating their properties by modifying them due to changing the concentration of thiokol in the initial product of the preliminary thioetherification reaction, the amount of the introduced hardener, and other factors.

Let us consider the effect of thiokol concentrations in PRTE products on the properties of samples containing the same amount of polysulphide rubber. For this purpose, the products of PRTE containing 60 and 200 parts by mass of thiokol, diluted with epoxy resin to obtain a mixture containing 20 parts by mass of modifier, and compared with the base composition based on a mixture of 100 parts by mass including the same epoxy resin Epikot-828 and 20 parts by mass of thiokol subjected to PRTE. As can be seen from Fig. 1, as the content of thiokol in the PRTE increased, the property indices increase quite significantly in comparison with the base sample.

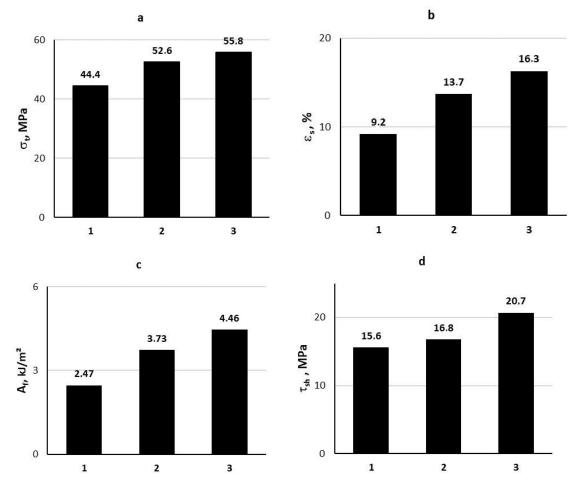


Fig. 1. Dependences σ_t (a), ϵ_s (b), A_f (c) and τ_{sh} (d) vs. content of thiokol in the base product of PRTE. Rubber content in PRTE-products: 20 (1), 60 (2) μ 200 (3) parts by mass per 100 parts by mass Epikote-828. The samples were hardened by the mode I

So the parameters σ_t increase by 18.2 and 25.4; ϵ_s – by 48.1 and 76.2, A_f - by 48.7 and 80.6, τ_{sh} - by 7.6 and 32.7 %, respectively, for samples based on PRTE containing 60 and 200 parts by mass of thiokol. The observed effect can be explained as follows. In the base mixture containing 20 parts by mass of thiokol, there is a large excess of epoxy. Assuming that 2 molecules of epoxy resin are consumed per thiokol molecule

and taking into account the fact that the molecular weight of thiokol is 2000, and of epoxy resin 380, it is easy to calculate that only 7.6 mass parts of resin is enough for the complete exhaustion of SH-groups (for a mixture of 100 parts by mass of epoxy resin and 20 parts by mass of thiokol).

PRTE results in a product that is a block oligomer with the structure

$$H_{2}C - CH - R' - CH - CH_{2} - S - R - S - CH_{2} - CH - R' - CH - CH_{2}$$
where
$$R = - \left(C_{2}H_{4} - O - CH_{2} - O - C_{2}H_{4} - S - S \right)_{n} C_{2}H_{4} - O - CH_{2} - O - C_{2}H_{4}$$

$$R' = -CH_{2} - O - CH_{2} - CH_{2}$$

As the content of thiokol increases as a result of PRTE, the probability of the formation of high molecular products of the general formula

OH OH
$$H_2C$$
— CH — CH_2 — S — R — S — CH_2 — CH — R' — CH — CH_2

which have a greater modifying effect on a complex of properties.

The formation of structures of this type can be confirmed by the data of dynamic mechanical spectrometry. It can be seen (Table 1) that when the PRTE products with a high thiokol content are introduced into the epoxy resin (for samples hardened according to mode II), the parameters

 $E_{\rm r.e.}$, n_c , $T_{\rm g}{}^{\rm i}$, $T_{\rm g}{}^{\rm f}$, and temperatures tg δ_m and E''_m increase, which may be due to the smaller amount of epoxy groups that react with thiokol by thioetherification. And, therefore, the larger number of unbound epoxy groups will participate in the formation of a denser chemical network when interacting with the amine groups of the hardener.

 $Table\ 1$ Influence of thiokol content in PRTE-products on dynamic mechanical properties of epoxy-thiokol composites \$^1\$)

Parameters —	Thiokol content in the PRTE-product, parts by mass ²⁾		
	20	60	200
Dynamic modulus of elasticity, E' at 25°C, GPa	1.58/1.43 ³⁾	1.45/1.21	1.39/1.20
Rubber elastity modulus, $E_{r.e.}$ at $T = T_g + 50$ °C, MPa	18.6/28.5	15.5/34,9	13.7/38.5
Molecular weight of chain segment between cross-linking, M _c , kg/kmol	621.9/396.0	717.3/348.2	810.5/317.1
Density of nodes of the chemical network, n_c , $kmol/m^3$	1.77/2.78	1.54/3.16	1.36/3.47
The temperature of the initial section of the transition from the glassy state to the highly elastic, $T_{\rm g}{}^{\rm i}$, ${}^{\rm o}{}^{\rm C}$	71.1/92.8	75.1/103.3	69.1/104.9
The temperature of the final section of the transition from the glassy state to the highly elastic, $T_g{}^f,{}^\circ C$	89.2/129.8	90.5/135.5	86.8/138.4
Maximal value of tangent of the angle of mechanical losses tg δ_{m}	0.9802/0.3614	0.8971/0.3423	0.9152/0.3403
Temperature tg δ_m , °C	88.3/128.3	91.5/131.2	84.3/135.2
Maximal value of loss modulus, E" _m , MPa	177.4/96.2	209.7/80.1	181.1/85.1
Temperature T _{E"m} , °C	82.8/115.0	85.7/125.8	77.4/128.5

Notes

It is quite effective to regulate the properties of epoxy-polysulphide mixtures by changing the amount of the introduced hardener (Fig. 2), i.e, by changing the ratio of amine (A) and epoxy (E) groups. When the content of diethylenetriamine increase, the tensile strength, deformation at break, and the work of fracture of the material rise very significantly. The magnitude of the effect achieved in this case is somewhat higher than when using PRTE with an increased content of thiokol (Fig. 1).

At the same time, in contrast to the first method, with an excess of the hardener, the adhesive strength does not increase, but rather decreases. Thus, an excess of the hardener affects positively the deformation and strength properties of the epoxy-thiokol composition, but causes a decrease in the adhesive strength. This may indicate that an excess of the hardener in the volume of the polymer sample shows an enhancing effect on the properties of the material (and the possible release of the hardener on the sample surface does not have a noticeable negative effect). In an adhesive joint, such a release of a part of the free hardener at the adhesive-substrate interface leads to a decrease in the adhesive strength.

The plasticizing effect of an excess of the hardener is clearly seen from the data in Table 2.

¹⁾ Total concentration of thiokol in composition is 20 parts by mass per 100 parts by mass of Epikote-828;

²⁾ per 100 parts by mass Epikote-828;

³⁾ before the line — the samples were hardened by the mode I; after the line – samples were hardened by the mode II.

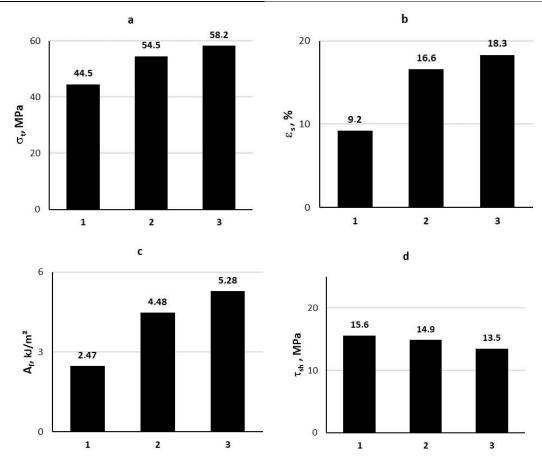


Fig. 2. Dependences σ_t (a), ϵ_s (b), A_f (c) μ τ_{sh} (d) vs. ratio of amine (A) and epoxide groups (E). Thiokol content in PRTE-products is 20 parts by mass per 100 parts by mass Epikote-828. A/E equals: 1 (1), 1.25 (2) and 1.5 (3). Samples were hardened by the mode I

As the ratio A/E increases, the dynamic modulus of elasticity, measured at 25° C (i.e., in the region of the glassy state of the polymer), and the rubber elasticity modulus $E_{\rm r.e.}$, as well as the

parameters $n_\text{c},\,T_g{}^i,\,T_g{}^f$ and temperatures tg δ_m and $E''{}_m$ decrease.

Influence of the amount of hardener on dynamic and mechanic properties of enoxy-thickol composites 1)

Table 2

	or epoxy-tniokoi c	of epoxy-thiokol composites 1)		
Parameters –	Ratio of amine and epoxy groups			
	1:1	1.25:1	1.5:1	
Dynamic modulus of elasticity, E' at 25°C, GPa	1.58/1.43 ²⁾	1.45/1.26	1.30/1.16	
Rubber elastity modulus, $E_{r.e.}$ при T = T_g + 50°C, MPa	18.6/28.5	16.3/21.6	12.4/13.6	
Molecular weight of chain segment between cross-linking, Mc, kg/kmol	621.9/396.0	679.6/548.7	891.8/857.4	
Density of nodes of the chemical network, n_c , $kmol/m^3$	1.77/2.78	1.62/2.00	1.23/1.28	
The temperature of the initial section of the transition from the glassy state to the highly elastic, $T_g{}^i,{}^\circ C$	71.1/92.8	70.9/92.0	65.5/80.3	
The temperature of the final section of the transition from the glassy state to the highly elastic, $T_g f, {}^\circ C$	89.2/119.8	86.9/114.1	82.2/102.8	
Maximal value of tanget of the angle of mechanical losses tg δ_{m}	0.9802/0.3614	0.8218/0.4831	0.9303/0.5841	
Temperature tg δ_m , °C	88.3/128.3	87.6/111.4	84.3/101.6	
Maximal value of loss modulus, E" _m , MPa	177.4/96.2	154.9/149.7	232.7/179.0	
Temperature T _{E"m} , °C	82.8/115.0	81.6/106.2	77.5/94.9	

Notes:

1) Thiokol concentration 20 parts by mass per 100 parts by mass Epikote-828;

2) before the line — the samples were hardened by the mode I; after the line - samples were hardened by the mode II.

When a filler (quartz sand) is introduced into an epoxy system containing a large amount of thiokol (200 parts by mass per 100 parts by mass of Epikote-828), a slight increase in cohesive strength and a relatively small decrease in adhesion strength and deformation at break are observed (Fig. 3).

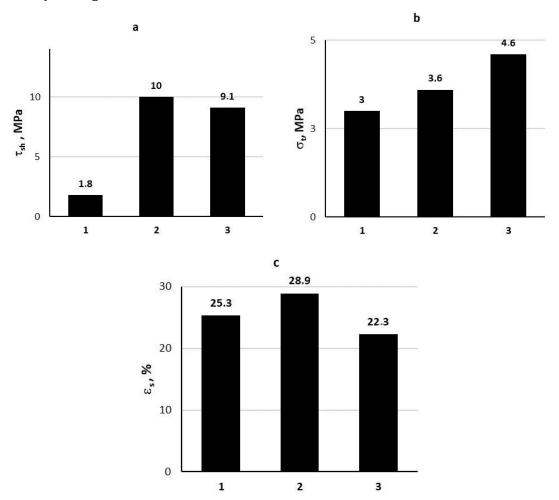


Fig. 3. Dependences τ_{sh} (a), σ_t (b) μ ϵ_s (c) vs. method of method of combining epoxy resin with thiokol and concentration of filler. Ratio: Epikote-828: thiokol = 100: 200 parts by mass. 1 – mechanical mixture; 2 – PRTE-product; 3 – PRTE-product filled with quartz sand. Filler concentration 100 parts by mass per 100 parts by mass of polymer. Samples were hardened by the mode II.

From the results obtained, it can be concluded that the introduction of a cheap filler can significantly reduce the cost of the epoxy-thiokol composition without fear of a noticeable deterioration in the complex of properties.

Conclusion

1. A system research of the possibility of regulating the deformation-strength, adhesive and dynamic mechanical properties of epoxypolysulphide composites by varying the concentration of thiokol in the initial product of the preliminary thioetherification reaction, as well as changing the amount of the hardener and using a mineral filler has been carried out.

- 2. It was found that increasing the thiokol content in the product of the preliminary thioetherification reaction promotes a growth in the indicators of cohesive and adhesive strength, deformation at break and the work of destruction of the material. An increase in the complex of properties is assumed to result from the formation of higher molecular weight products with an increase in the thiokol content in the composition subjected to PRTE. The formation of such products is confirmed by the data of dynamic mechanical spectrometry.
- 3. It has been shown that by changing the amount of the introduced hardener, i.e. by changing the ratio of amine and epoxy groups, it is possible to effectively regulate the properties

of epoxy-polysulphide mixtures. With an increase in the content of the hardener diethylenetriamine, the tensile strength, deformation at break, and the work of fracture of the material increase very significantly.

4. The introduction of a filler into an epoxy system containing a large amount of thiokol was found to promote an increase in cohesive strength with a relatively small decrease in adhesion strength and deformation at break.

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