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DYNAMIC MECHANICAL PROPERTIES OF EPOXY COMPOSITES MODIFIED WITH POLYSULPHIDE RUBBER

Kostyantyn Sukhyy[1], Elena Belyanovskaya[1], Alla Nosova[1, ✉], Irina Sukha[1], Mikhailo Sukhyy[1], Yudong Huang[2], Yuriy Kochergin[2], Tetiana Hryhorenko[2]

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Abstract. Study of the dependence of the dynamic mechanical properties of polymers based on mixtures of epoxy resin with polysulphide rubber, depending on the conditions for combining the components (carrying out a preliminary thioetherification reaction or mechanical mixing), the rubber content and the curing modes has been carried out.

Key words: epoxy resin, polysulphide rubber, preliminary thioetherification reaction, dynamic mechanical properties.

1. Introduction

Epoxy polymers came into sharp focus as a special class of materials with unusually wide possibilities of application, due to their specific and, moreover, almost universal properties: the ability to solidify at room temperature; minimal shrinkage during solidifying; excellent adhesion to various materials combined with a high range of physical and mechanical properties; good anti-corrosion characteristics; high electrical insulating properties, *etc.*[1-9]

Despite the aforementioned advantages of EP, their significant disadvantages are low shock, vibration and crack resistance, which sharply limits their use under the action of dynamic loads and thermal cycles.

It was previously shown[10-16] that in order to increase the adhesive and physicomechanical properties of epoxy-thiokol compositions cured without heat treatment, it is necessary to carry out a preliminary thioetherification reaction (PRTE) prior to the introduction of the curing agent due to the interaction of thiokol mercaptan groups and oxirane cycles of epoxy resin at elevated temperature, and then use the product of this reaction to cure at room temperature. Composite materials based on the products of the thioetherification reaction are revealed to significantly surpass analogs based on mechanical mixtures of epoxy resin and thiokol in terms of cohesive and adhesive strength, deformation capacity, fracture work, and specific impact number.

As it is known,[17-20] the study of the dynamic mechanical properties of polymers is the task of great theoretical and applied importance. In particular, the dynamic modulus of elasticity is a very important indicator of the stress-strain properties of polymers. Along with the indicator of mechanical losses, the dynamic modulus of elasticity is the most sensitive indicator of all forms of molecular mobility in polymers, primarily in the glassy state. In addition to purely theoretical interest in understanding the mechanisms of molecular motion in polymers, mechanical losses are of great practical importance, largely determining other mechanical properties of polymers. Therefore, the absolute values of the indicators of mechanical losses, the position of temperatures, and frequencies at which the maxima of losses observed are of particular interest. High mechanical losses in polymeric materials can be both advantage and disadvantage. On the one hand, losses reduce vibrations and prevent the occurrence of resonant vibrations with a sharp increase in amplitude. On the other hand, high losses are generally indicative of reduced dimensional stability, which is highly undesirable in structures operating under load for long periods. Many other mechanical properties, such as service durability, the specific surface energy of destruction and impact strength, frictional factor, abrasion, and wear are closely related to mechanical losses. Determination of mechanical losses can serve as an effective method for determining the molecular weight of polymers, the composition of copolymers, the degree of crosslinking heterogeneity, the effect of heat treatment on the morphology of crystals in crystalline polymers, the composition of mixtures of polymers and block copolymers, and the degree of curing the thermosetting resins. The dynamic mechanical properties of polymers can be significantly changed by the heat treatment of the samples. Thus, induration of

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[1] Ukrainian State University of Chemical Technology, Gagarin Avenue, 8, Dnipro, Ukraine, 49005
[2] Harbin Institute of Technology, 92 Xidazhi street, Nangang District, Harbin, Heilongjiang Province,
People's Republic of China
✉ ikichemistry@gmail.com

amorphous polymers, in contrast to annealing, usually results in an increase in mechanical losses. Dynamic mechanical properties, especially mechanical losses, are very sensitive to all types of temperature transitions, relaxation processes, structural inhomogeneities, and features of the morphological structures of multiphase systems such as partially crystalline polymers, polymer mixtures, and filled polymer compositions.

In continuation of the research, the aim of the present work is to study the dynamic mechanical properties of epoxy-thiokol composites based on the products of the preliminary thioetherification reaction.

2. Experimental

The objects of study were Epikote-828 epoxy resin (mass fraction of epoxy groups (EG) 22.6 %, viscosity 12 Pa·s at 298 K), 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 433 K for 2 h with a thorough stirring. For comparison of properties, mechanical mixtures of epoxy resin and rubber were also studied. Curing of the compositions was carried out with diethylenetriamine DETA (amine number 1600–1650 mg (KOH/g, density 948–952 kg/m^3) in two modes: I – 289 K/24 h + 323 K/8 h (curing at moderate temperature) and II – 289 K/24 h + 393 K/3 h (curing with heat treatment at elevated temperature).

Dynamic mechanical characteristics (dynamic modulus of elasticity E', loss modulus E'' and tangent of the angle of mechanical losses *tg* δ) were measured on DMA 983 unit of the DuPont 9900 thermoanalytical complex on 25x4x2 mm samples with heating at a rate of 10 K/min. The rubber elasticity modulus (E_{re}) was determined at a temperature equal to T_g + 50 K, where T_g is the glass transition temperature of the polymer.

The mechanical properties under uniaxial tension (tensile strength σ_t and deformation at rupture ε_s) were determined on an automatic tensile testing machine C610H from Labthink instruments Co. Ltd.

Internal stresses were determined using the setup described elsewhere[20] by measuring the deflection of an elastic substrate lying on two supports, on which a polymer composition was applied. The measurement of the deflections of the substrates during the curing of the composition and the subsequent decrease in temperature was carried out with a cathetometer KM-6 equipped with an additional lighting device. The value of internal stresses was calculated by formula (1):

$$\sigma_{int} = \frac{4 f_{max} E_n \delta^3}{3 l^2 (\delta + \Delta\delta)\Delta\delta}, \quad (1)$$

where f_{max} is a maximal value of substrate deflection; l refers to a length of a substrate; δ and Δδ are a thickness of substrate and composition respectively; E_n is an elastic modulus of a substrate.

Water absorption (W) was determined by the change of samples mass after boiling during 3 hours by formula (2):

$$W_{(t)} = \frac{m(t) - m_0}{m_0} \times 100\%, \quad (2)$$

where m_0 is the initial mass of a sample; m(t) is the mass of a sample after exposure in water during time *t*.

Density of nodes of the chemical network (n_c) was calculated by formula (3):

$$n_c = E_{re} / 3RT, \quad (3)$$

where R is an absolute gas constant; T is a Kelvin temperature; E_{re} is a rubber elasticity modulus.

The degree of curing of the epoxy polymer was determined by extracting the unreacted soluble part of the epoxy resin in acetone.

3. Results and Discussion

The temperature dependences of the tangent of the angle of mechanical losses (*tg* δ) and the dynamic modulus of elasticity *vs.* the test temperature for specimens cured without a heat supply are given in Fig. 1. As the thiol concentration increases, the position of the maximum *tg* δ_m maximum is observed to shift towards lower temperatures, its value is decreased (this is more clearly evidenced by the data presented in Fig. 2a). This effect can be explained by the fact that the intensity of this transition associated with devitrification of the epoxy matrix is proportional to the content of the epoxy component in the mixture. The lower the intensity, the higher the concentration of thiol in the composition. The decrease in the temperature at which the maximum *tg* δ is observed can be explained by the plasticizing effect of thiokol, which has a glass-transition temperature below 273 K.

When the plots *tg* δ *vs.* T are analyzed, the following experimental fact attracts attention. For the base epoxy polymer (Fig. 1a, curve 1), cured according to mode I, after passing the main maximum *tg* δ at first decreases rather quickly, then at temperatures above 373 K, the decrease in *tg* δ stops, and in the range of 373–433 K a second wide diffuse maximum is observed, after which *tg* δ decreases rather rapidly again. This behavior can be explained by the fact that after the devitrification of the epoxy polymer, molecular mobility is accelerated, which contributes to the post-curing process. The interaction of epoxy and amine groups, which have not reacted during curing without heating consumes part of the supplied thermal energy, resulting in the formation of a maximum with a top at a temperature of ~408 K. Another confirmation of the post-curing reaction is an increase in the dynamic elasticity modulus in the temperature range of 373–408 K (Fig. 1b).

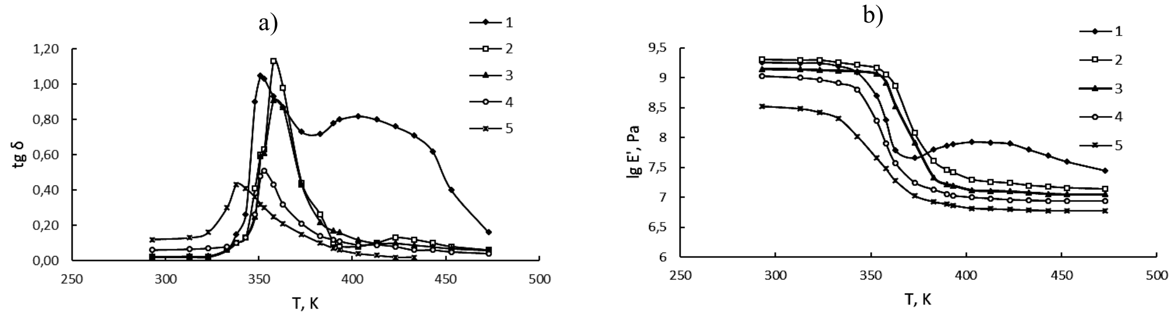


Fig. 1. Dependence *tg* δ (a) and *lg E'* (b) *vs.* temperature (*T*) for the samples based on PRTE, cured by the mode I. Content of thiokol: 0 (1), 10 (2), 20 (3), 60 (4) and 100 (5) pts.wt. per 100 pts.wt. EO Epikote-828

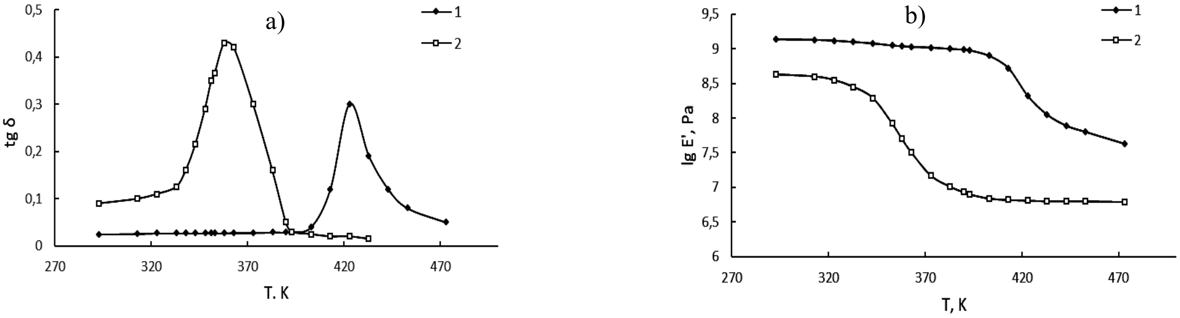


Fig. 2. Dependence *tg* δ (a) and *lg E'* (b) vs. temperature (T) for the samples based on PRTE, cured by the mode II. Content of thiokol: 0 (1) and 100 (2) pts.wt. per 100 pts.wt. of epoxy resin.

Finally, if the sample was previously heat-treated at 393 K, *i.e.* the post-curing process had already been completed before the start of scanning the sample, then this secondary maximum does not appear on the plot *tg* δ *vs. T* for it (Fig. 3b). In this case, the intensity of the primary maximum decreases greatly (from 1.05 to 0.30, and its position is shifted significantly towards higher temperatures (from 351 to 423 K).

In the case of samples containing rubber, the maximum associated with the post-curing rapidly degenerates as the modifier content increases. This may indicate that the presence of thiokol in the composition promotes a greater conversion of reactive groups. As a consequence, as can be seen from Fig. 3b, with the addition of thiokol up to 60 pts. wt., the temperature of the maximum value of the tangent of the angle of mechanical losses (*tg* δ_m) increases by 278–280 K relative to the base polymer.

There are significant differences in the *tg* δ_m values for unheated and heat-treated samples in the region of relatively low thiokol concentrations (Fig. 3a). The high *tg* δ_m values for samples cured according to mode I can be explained by the superposition of effects caused by the glass transition and conformational rearrangements of molecules associated with the reaction of post-curing of the epoxy matrix at elevated temperatures, where this maximum is revealed. After heat treatment at 393 K, the post-curing process has already been finished, and this leads to a significant decrease in the intensity of this relaxation transition. The observed effects can be explained by the fact that as a result of the PRTE, longer sections of the chain are formed between the junctions of the network. These long sections cause large losses of mechanical energy during the transition to a highly elastic state, which is reflected in an increase of *tg* δ_m values. The value of *tg* δ_m itself associated with the devitrification of the epoxy polymer for the samples cured without heat supply decreases monotonically (Fig. 3a) with an increase in the rubber concentration, especially rapidly in the region of small additives. This phenomenon can be explained by a decrease in the proportion of the epoxy component in the mixture with an increase in the thiokol content. At the same time, for the heat-treated samples, value of the *tg* δ_m does not decrease, but, on the contrary, slightly increases with an increase in the thiokol concentration. Apparently, this effect results from the difficulties in the displacement of the sections of the epoxy polymer chain responsible for revealing the maximum *tg* δ_m because of the grafting of a certain fraction of rubber molecules to them due to the chemical interaction of oxirane and mercaptan groups during heat treatment at 393 K.

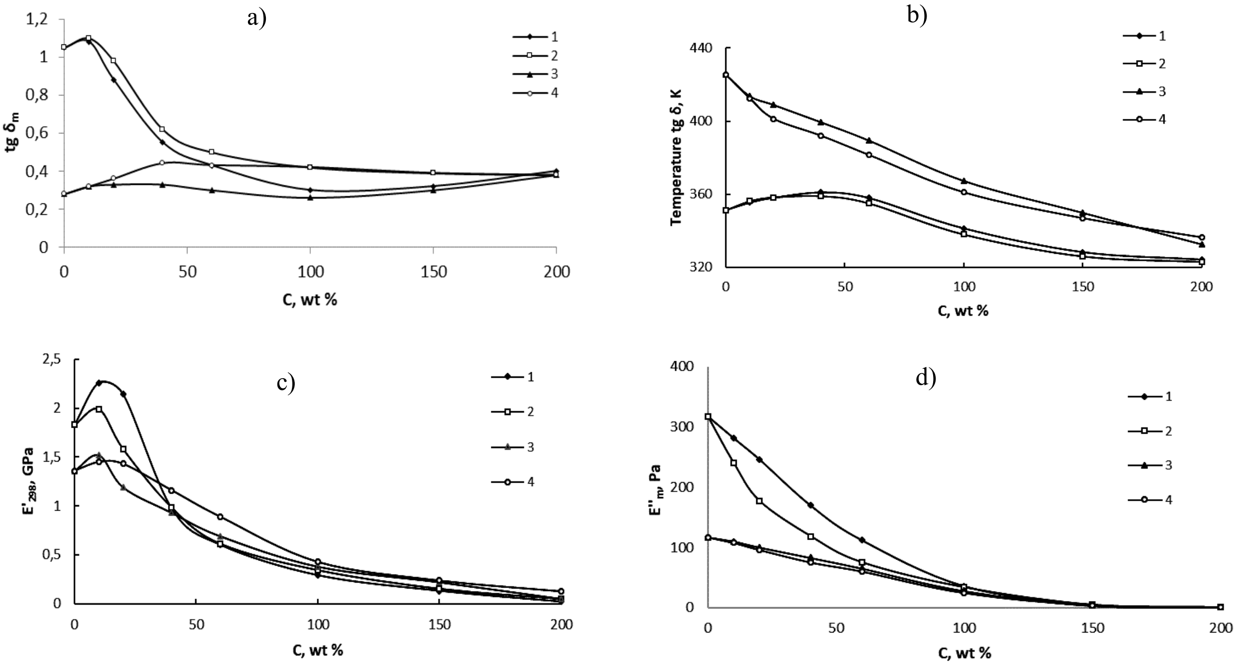


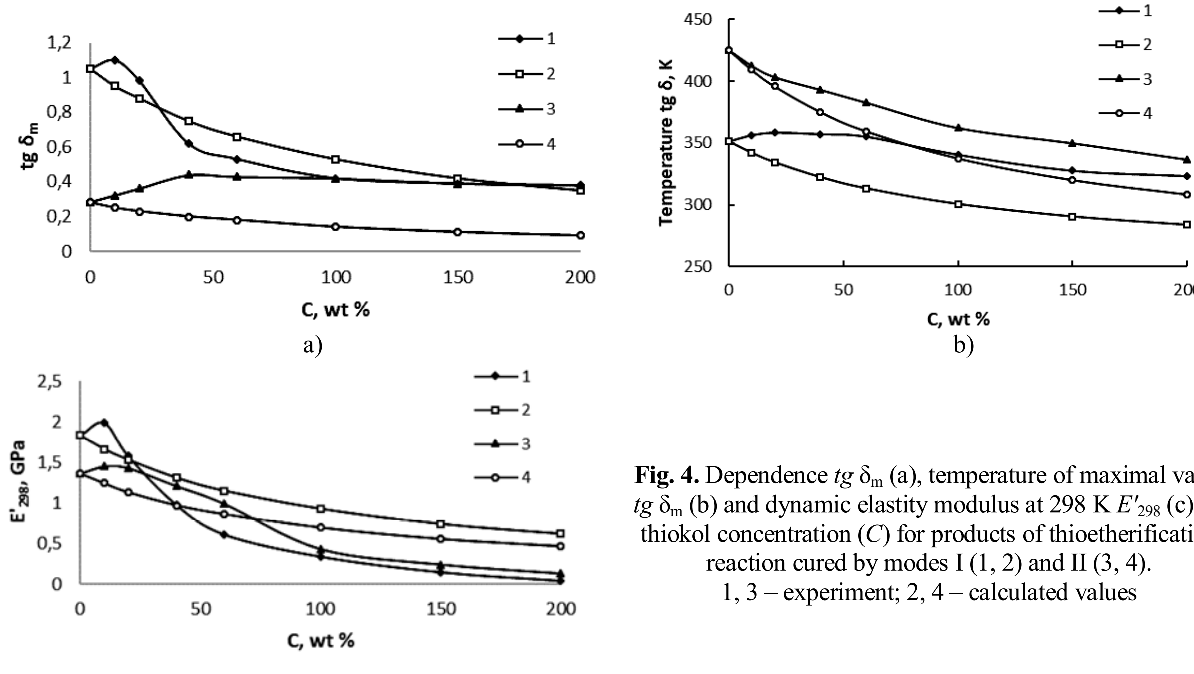
Fig. 3. Dependence *tg* δ_m (a), temperature of maximal value *tg* δ_m (b), dynamic elastity modulus at 298 K E'_{298} (c) and maximal modulus of losses E''_m (d) *vs.* thiokol concentration (C) for mechanical mixtures (1, 3) and products of reactions of thioetherification (2, 4). The samples are cured by the mode I (1, 2) and II (3, 4)

The nature of the change in the parameter *tg* δ_m with an increase in the concentration of thiokol for the products of PRTE and mechanical mixtures (MM) is seen to be different (Fig. 3a). At low modifier contents for the samples cured by mode I, an increase in *tg* δ_m is observed for the product of PRTE and MM with the formation of a maximum at a thiokol concentration of 10 pts. wt. Then, for the PRTE product, there is a monotonic decrease in *tg* δ_m with a plateau at $C > 100$ pts. wt. For MM, a decrease in *tg* δ_m also occurs up to $C = 100$ pts. wt., but then an increase in *tg* δ_m to values inherent in the PRTE product is observed. For the samples cured according to mode II, at low C, *tg* δ_m also increases. Only for MM, an increase in *tg* δ_m is observed to be much smaller in value and after the formation of a small maximum at $C = 35$ pts. wt. its decrease is stated with the formation of minima at $C = 100$ pts. wt., after which *tg* δ_m increases again. For the PRTE product, *tg* δ_m grows to $C = 40$ pts. wt., after which it practically reaches the plateau zone. According to Fig. 4a, the analytical dependence *tg* δ_m *vs.* C describes quite well the course of the experimental curve obtained for samples based on PRTE products cured according to mode I. At the same time, for samples cured according to mode II, the type of the dependences *tg* δ_m *vs.* C is opposite. For the experimental dependence, a monotonic increase in *tg* δ_m is observed, while the analytical dependence exhibits a monotonic decrease in the parameter.

As it is known,[17] the glass transition temperature of a polymer mixture can be determined in accordance with the analytical dependence $T_{12} = w_1T_1 + w_2T_2$, where T_{12} is the temperature *tg* δ_{max} for the mixture, T_1 and T_2 are the temperatures *tg* δ_{max} for the components of the mixture, w_1 and w_2 are the mass fractions of the components mixtures. In our case, T_1 is equal to 351 and 426 K for the original epoxy resin cured according to modes I and II, respectively. According to Ref. [21], the temperature T_2 for thiokol is 250 K. Then, for example, for the PRTE product, in which the rubber component and thiokol are taken in 1:1 ratio, the calculated T_{12} values will be 300 K (curing mode I) and 338 K (curing mode II). The experimentally determined values of the glass transition temperature for a composite of this composition are 338 K (Fig. 1a, curve 5) and 358 K (Fig. 2a, curve 2). Consequently, the difference between the experimental and calculated values is 38 (338–300) K and 20.0 (358–338) K, respectively, for the samples cured according to modes I and II. A smaller difference for the composite cured by mode II indicates greater compatibility of its components after heat treatment.

According to Fig. 3b, there is no significant difference in the nature of the concentration dependences of the temperature *tg* δ_m for MM and PRTE products. As given in Fig. 4b (curves 2 and 4), regardless of the curing mode for PRTE products, the decrease in temperature *tg* δ_m with an increase of the rubber content occurs more slowly than predicted by analytical dependences for fully compatible components.

As for the values of the dynamic modulus of elasticity, measured at 298 K, E'_{298} (Fig. 3c), their change depending on the concentration of rubber is in many respects similar to the static modulus of elasticity.[12] Formation of low intensity maxima on the dependences E'_{298} *vs.* C at a thiokol content of ~ 10 pts. wt. can be associated both with the antiplasticization effect (an increase in strength and modulus with a simultaneous decrease in the glass transition temperature T_c) and with the fact that small additives accelerate the relaxation process, thereby reducing unwanted residual stresses in the epoxy matrix. According to Ref. [22], the main and necessary feature of antiplasticization is a simultaneous increase in the elastic modulus and strength of the vitreous polymer with a monotonic decrease in the glass transition temperature. In this case, “antiplasticizers” should be well combined with the polymer, have polar atoms (chlorine, oxygen, nitrogen, sulfur), and glass transition above 323 K. Thiokols correspond with all these characteristics: they perfectly combine with epoxy resin, contain polar oxygen and sulfur atoms, and have T_c > 233 K. It should also be noted that after passing the maximum, the curves E'_{298} *vs.* C fully correspond to the behavior which is typical for the plasticization effect. In this case, the experimentally determined values of E'_{298} (Fig. 4c) in the region of sufficiently high thiokol concentrations (C > 50 pts. wt. per 100 pts. wt. of epoxy polymer) are significantly less than the theoretical values calculated according to the additivity rule. Apparently, this is due to the fact that, with a high content of thiol, the epoxy component of the mixture can no longer form a continuous spatial network, but forms only local zones distributed in the volume of the rubber vulcanizate matrix.



a) b) c)

Fig. 4. Dependence *tg* δ_m (a), temperature of maximal value *tg* δ_m (b) and dynamic elastity modulus at 298 K E'_{298} (c) *vs.* thiokol concentration (C) for products of thioetherification reaction cured by modes I (1, 2) and II (3, 4).
1, 3 – experiment; 2, 4 – calculated values

It should be noted that after heat treatment of the samples (Fig. 3c), the modulus of elasticity, measured at 298 K, E'_{298} decreases, and to the fullest extent this is exhibited for a polymer without thiokol and containing up to 20 pts. wt. of rubber. Such a decrease in E'_{298} may result from the fact that additional chemical crosslinks formed in the epoxy polymer during heat treatment prevent a denser packing of fragments of the molecular chain, thereby reducing the efficiency of intermolecular interaction. A similar effect of the anomalous dependence of the dynamic modulus of elasticity in the vitreous state of the polymer *vs.* n_c was repeatedly observed earlier.[23]

According to Fig. 3d, the nature of the dependences E''_m *vs.* C for the PRTE and MM products is identical.

It was of interest to compare the effect of heat treatment in air and exposure to hot water on the dynamic mechanical properties of the studied compositions. It can be seen (Table 1) that after heat treatment of samples cured according to mode I, there is a noticeable increase in the elastic modulus in the highly elastic state, the density of the chemical network nodes, the transition temperature from the vitreous to the highly elastic state, as well as temperatures at which the loss modulus E'' and the tangent of the angle of mechanical losses tg δ reach their maximum values.

Table 1. Effect of heat treatment and water on the properties of epoxy-thiokol composites[1)]

Characteristic quantity	Base sample[2)]	After heat treatment at 393 K during 3 hours	After exposition in water at 373 K during 3 hours
Dynamic elastity modulus, E' at 298 K, GPa	2.11	1.60	1.73
Rubber elasticity modulus, E_{re} at $T = T_g + 50$ K, MPa	21.80	33.67	38.14
Molecular weight of chain segment between cross-linking, M_c, kg/kmol	517.71	362.61	302.38
Density of nodes of the chemical network, n_c, kmol/m^3	2.12	3.03	3.64
The temperature of the initial section of the transition from the glassy state to the highly elastic, T_g^i, K	345.55	375.55	341.85
The temperature of the final section of the transition from the glassy state to the highly elastic, T_g^f, K	358.15	406.85	386.45
Maximal value of tangent of the angle of mechanical losses, *tg* δ_m	0.8634	0.3178	0.2699
Temperature of maximal value *tg* δ_m, K	360.25	402.15	383.15
Maximal value of loss modulus, E''_m, MPa	257.70	78.42	94.03
Temperature E''_m, K	353.1	396.65	373

Notes:
[1)] 100 pts. wt. Epikote-828 + 20 pts. wt. thiokol;
[2)] Curing by the mode I.

As follows from the data given in Table 1, the effect of boiling water leads to a lower decrease in the modulus E' in the vitreous state as compared to heat treatment. This phenomenon is probably due to the formation of additional hydrogen bonds between the polar groups of the polymer and the molecules of sorbed water. At the same time, the sorbed water has a plasticizing effect on the epoxy polymer, which is reflected in a slight decrease at the transition temperature from the vitreous to the highly elastic state and the maximum temperatures of *tg* δ and E''.

Interestingly, in this case, the maximum value of the tangent of the angle of mechanical losses and the loss modulus for the sample exposed to water is somewhat lower than that of the heat-treated composite. The reason for such a change in these parameters as a result of heat treatment and exposure to boiling water is easy to understand if the fact that value of *tg* δ is determined by the formula *tg* δ = E''/E' taken into account. It follows from it that the value of *tg* δ decreases if the loss modulus E'' is reduced or the dynamic elastic modulus increases, and moreover to a greater extent.

As proven by the results of the study, the heat treatment of the samples and their exposure to boiling water results in an increase in the loss modulus at room temperature (41.61, and 76 MPa, respectively, for the samples cured according to modes I and II, and subjected to boiling in water) and the values of *tg* δ (0.02; 0.025 and 0.035, respectively, for specimens cured by modes I and II and boiled in water). Probably, this can be associated with an increase in the level of internal stresses in the samples after heating to elevated temperatures and subsequent rather rapid cooling to ambient temperature. Such hardening of the sample, as was noted earlier, increases the level of mechanical losses. As the sample is heated during the experiment, the relaxation of internal stresses is accelerated. As a result, a very significant decrease in E'' and *tg* δ is observed with an increase in temperature to 373 K for samples cured according to mode II (up to 30 MPa and 0.027, respectively), as well as samples subjected to boiling in water (up to 43 MPa and 0.029). It should be noted that in this case, the samples are in a vitreous state since their temperature T_g^f is higher than 386 K (Table 1).

A higher level of mechanical losses in a sample subjected to boiling in water can be explained by the specific effect of absorbed moisture on the properties of the polymer matrix. A direct experiment to determine the effect of water on the value of σ_{int} indicates a rather tangible rise of this parameter (Table 2). At the same time, cohesive strength and deformation at break grow. So, the presence of water in the curing system is suggested to result in the formation of a denser chemical network. This assumption is confirmed by the results of measuring the degree of curing of the epoxy polymer (Table 2).

Table 2. Effect of water on mechanical properties and cure degree of epoxy resin

Content of water in polymer, pts. wt.	σ_{int} (MPa) at the temperature, K		σ_t, MPa	ε_s, %	Degree of epoxy groups conversion, %
	293	213			
0	2.7	5.9	45.1	5.9	72.8
0.5	3.6	8.3	46.9	6.0	78.2
1.0	4.3	10.5	48.3	6.1	81.4

The complex nature of the effect of water on the degree of transformation of epoxy groups, the level of internal stresses, and deformation-strength properties can be explained on the basis of the previously proposed mechanism[24] of the influence of liquid media on the properties of EP, the essence of which is the imposition of the effects of plasticization and a sharp acceleration of the process of additional curing of the polymer on deep stages of transformation. The liquid sorbed by the polymer weakens physical bonds, which leads to an increase in the intensity of molecular motion. This increases the probability of contact of unreacted reactive epoxy and amine groups, and, consequently, the formation of additional chemical crosslinks.

4. Conclusions

1. A systematic study of the dependence of the dynamic mechanical properties of polymeric materials based on epoxy-polysulphide mixtures, depending on the conditions for combining the components (mechanical mixing and mixing during the preliminary thioetherification reaction), the amount of polysulphide rubber and the curing modes of the composition, has been carried out.

2. The value of the maximum tangent of the angle of mechanical losses associated with devitrification of the epoxy matrix is shown to decrease as the content of polysulphide rubber in it increases. In this case, the position of the maximum is shifted towards lower test temperatures. For composites cured at room temperature, with thiokol additions up to 60 pts. wt. the temperature of the maximum tangent of the angle of mechanical losses was found to increase by 278–280 K relatively to the base epoxy polymer.

3. The nature of the change in the parameter $tg\ \delta_m$ with an increase in the concentration of polysulphide rubber for the products of PRTE and mechanical mixtures (MM) was found to be different. With an increase in the rubber content for PRTE products, regardless of the curing mode, the decrease in temperature $tg\ \delta_m$ occurs more slowly than predicted by analytical dependences for fully compatible components. Heat treatment improves the compatibility of components in epoxy-polysulphide composites.

4. After heat treatment of the samples, the elastic modulus in the vitreous state is shown to decrease, and this is most pronounced for a polymer containing up to 20 pts. wt. rubber. This decrease is explained by the fact that additional chemical crosslinks formed in the epoxy polymer during heat treatment prevent a denser packing of fragments of the molecular chain, thereby reducing the efficiency of intermolecular interaction.

5. A comparative study of the effect of heat treatment in air and exposure to hot water on the dynamic mechanical properties of the studied compositions has been carried out. It is revealed that after heat treatment of the samples, there is a noticeable increase in the elastic modulus in the highly elastic state, the density of the nodes of the chemical network, the transition temperature from the vitreous to the highly elastic state, as well as the temperatures at which the loss modulus E'' and the tangent of the angle of mechanical losses $tg\ \delta$ reach the maximum values. Exposure to boiling water leads to a decrease in the dynamic modulus of elasticity in the vitreous state, as compared to heat treatment. At the same time, sorbed water affects epoxy polymer as a plasticizing agent, this being reflected in reducing the transition temperature from the glasslike state to a highly elastic one and of the temperatures of the maxima of the tangent of the angle of mechanical losses and the loss modulus. The higher level of mechanical losses in the sample subjected to boiling in water is explained by the specificity of the effect of adsorbed moisture on the properties of the epoxy matrix, which contributes to the formation of a denser chemical network.

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ДИНАМІЧНІ МЕХАНІЧНІ ВЛАСТИВОСТІ ЕПОКСИДНИХ КОМПОЗИТІВ, МОДИФІКОВАНИХ ПОЛІСУЛЬФІДНИМ КАУЧУКОМ

***Анотація**. Проведено дослідження залежності динамічних механічних властивостей полімерів на основі сумішей епоксидної смоли з полісульфідним каучуком залежно від умов суміщення компонентів (реакції тіоетерифікації або механічного перемішування), вмісту каучуку та режимів затвердження.*

***Ключові слова:** епоксидна смола, полісульфідний каучук, тіоетерифікація, динамічні механічні властивості.*