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# Study Of Viscoelastic Properties of Epoxyurethane Compositions for Vibration Protection of Metal Products

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**Abstract.** The dynamic mechanical and vibration-absorbing properties of epoxyurethane coatings for vibration protection of metal products have been studied. It has been established that the chemical structure of modifying epoxy oligomers and their content in a mixture with oligester cyclo-cab oligomer affects the structural parameters of the epoxyurethane polymer matrix, which makes it possible to regulate their viscoelastic and damping properties. As a result of dynamic mechanical studies, it was determined that with an increase in the content of epoxy oligomers (ED-20, T-111 and UP-655) in a mixture of oligester cyclo-cab oligomer L-803 from 10:90 to 30:70 wt.% observed of increase the dynamic modulus of elasticity and glass transition temperature. This is due to the fact that the epoxyurethane polymer is enriched with more rigid components: when using ED-20 and T-111, which contain aromatic fragments in their structure and chloraliphatic oligomer UP-655, which formation the matrix of strong intermolecular interactions due to strongly polar chlorine atoms. It has been established that epoxyurethane systems are characterized by one area of «devitrification», as evidenced by the monomodal peak of the maximum  $tg\delta$ , and therefore it is possible to indicate the creation of a relatively homogeneous system.

## INTRODUCTION

Currently, the problem of reducing vibration is relevant in all industries (construction, space technology, transport, etc.). There is a need to protect building metal structures and products from high levels of vibration and noise with various vibration-absorbing materials with the development of modern industrial technologies [1, 2].

The creation of such effective polymer-based vibration-absorbing materials has been solved by modifying polymer epoxy materials by introducing urethane-linked oligomers into their structure [3, 4]. This allows us to purposefully improve the elastic-deformation characteristics of epoxy polymers [5, 6]. Thus, the regulation of viscoelastic properties can be achieved not only by synthesizing new polymer materials, but also by modifying the structure and properties of already known polymer materials.

To obtain an effective vibration-absorbing material based on the polymer composition, it is necessary to create such compositions that would have in the required temperature and frequency range maximum values of the mechanical loss tangent ( $tg\delta$ ) or the mechanical loss modulus  $G''$ , which are a measure of the dissipated energy, respectively [7, 8].

The maximum values of  $tg\delta$  are observed in the region of the main relaxation transition, i.e., in the region of the transition from the glassy transition to the highly elastic state, where the frequency of coordinated motion of the segments of the polymer chains (10-50 carbon atoms) has the same value as the frequency of mechanical action. The

transition temperature from the glassy state to the highly elastic, called the glass transition temperature ( $T_g$ ). Usually, in a dynamic experiment, an increase in frequency by a factor of 10 has been accompanied by an increase in  $T_g$  by 3-7 °C [9].

The scientific and technical problem of creating effective vibration-absorbing materials based on polymers was solved by using commercially available reactive oligomers. This approach is also economically feasible and therefore relevant.

## ANALYSIS OF THE PREVIOUS RESEARCH DATA

It is known [10] that the viscoelastic characteristics of polymers are determined by their chemical nature, the structure of the polymer chain, and intermolecular interactions between them. Therefore, polymers that combine the flexibility of the polymer chain and high values of intermolecular interaction have a high damping ability.

Earlier [11, 12], it was shown that from the point of view of the technology of production and use of vibration-absorbing coatings, it is most expedient to use curing systems based on mixtures of oligester cyclo-cab oligomer and epoxydian oligomers. Crosslinked epoxy-hydroxyurethane compositions are obtained when curing these mixtures of oligomers with amines at room and elevated temperatures. These compositions combine high values of the adhesive strength [13, 14] and mechanical loss tangent.

The aim of this work was to study the effect of the chemical structure of modifying epoxy oligomers in a mixture with oligester cyclo-cab oligomer on the viscoelastic and damping properties of crosslinked epoxyurethane polymers.

## MATERIALS AND METHODS

Taking into account the above, we have chosen as the objects of study oligester cyclo-cab oligomer Laprolat-803 (L-803) and modifying epoxy oligomers: ED-20, T-111 and UP-655. Diethylenetriamine (DETA) was chosen as the general hardener.

As the main method for studying viscoelastic properties, we chose the method of dynamic mechanical spectroscopy, which was implemented on a torsion pendulum-dynamic relaxometer [15]. From the experimental data, the dynamic elastic modulus ( $G'$ ,  $E'$ ), the mechanical loss tangent  $tg\delta$  and the loss modulus  $G''$  were calculated. These indicators are related  $tg\delta = G''/G'$ . The maximum value of  $tg\delta$  corresponds to the mechanical glass transition temperature ( $T_g$ ).

According to some data [16], from the point of view of minimizing the influence of external mechanical influences on changes in the polymer structure during the experiment, the range in which it is most expedient to study the viscoelastic properties of polymers lies in the ultralow frequency range of  $10^{-3}$  -  $10^1$  Hz. In this regard, the frequency of damped torsion oscillations of the pendulum in our experiments was 1.0 Hz in the temperature range from -100 °C to +100 °C. The measurement error for the dynamic shear modulus did not exceed 5%, and for the mechanical loss tangent - 10 %. In addition to the chemical structure of the oligomer chain and physical nodes of the matrix, due to intermolecular interaction, many operational properties of crosslinked epoxyurethane polymers are largely determined by the parameters of the spatial network - the average molecular weight of the segments of macromolecules between the network nodes ( $M_c$ ), as well as the effective crosslinking density ( $n_c$ ) [17].

The  $M_c$  and  $n_c$  values were calculated using the equation [18]:

$$M_c = \frac{3\rho RT_{he}\nu}{E_\infty}, \quad (1)$$

$$n_c = \frac{E_{he}\nu}{3RT\nu}, \quad (2)$$

where  $\rho$  - the density of the polymer;  $R$  - the universal gas constant;  $T$  - the absolute temperature;  $\nu$  - structural coefficient, which depends on the nature and topology of the mesh.

The modulus of elasticity  $E_{he}$  of the studied polymers was determined under uniaxial compression at a temperature exceeding the glass transition temperature by 50 degrees, and  $M_c$  and  $n_c$  were calculated from the above ratio. Polymer samples were cured for 7 days at 25 °C (cold curing - regime I) and during heat treatment - 24 h (25 °C) and 4 hours at 100 °C (regime II).

## EXPERIMENTAL INVESTIGATIONS

The results of studying the effect of the ratio of Laprolat-803 and various epoxy oligomers on the structural parameters of the cured reticulated epoxyurethane compositions are presented in Table 1.

**TABLE 1.** The values  $M_c$  and effective crosslinking density ( $n_c$ ) for epoxyurethane compositions

The ratio of the epoxy oligomer mixed with Laprolat-803, wt. %	Density, kg/m <sup>3</sup>	$E_{he}$ , MPa	$M_c$ , g/mole	$n_c$ , mole/g
Laprolat-803 (100)	1149/1174	2.5/3.1	1780/1455	0.67/0.82
T-111 (100)	-/1150	-/12.2	-/456	-/2.50
ED-20 (100)	-/1180	-/10.5	-/470	-/2.45
ED-20: Laprolat-803				
10:90	1224/1250	3.37/3.49	1321/1329	0.9/0.94
20:80	1210/1212	3.5/3.71	1272/1000	0.94/1.21
30:70	1208/1223	4.5/4.9	989/926	1.21/1.32
T-111: Laprolat-803				
10:90	1189/1201	3.0/3.8	1481/1178	0.81/1.0
20:80	1252/1252	3.1/4.73	1445/982	0.83/1.27
30:70	1165/1165	3.47/5.64	1283/766	0.93/1.52

Note: up to a slash - curing according to regime I; after the line - according to regime II.

From Table 1 it is seen that the  $M_c$  of the polymer based on the bifunctional epoxydian oligomer ED-20 is greater value than the polymer based on the organosilicon epoxy oligomer T-111.

However, we recall that the organosilicon epoxy oligomer T-111 is an analogue of an epoxydian oligomer chemically modified with an organosilicon component and therefore has a slightly higher average molecular weight (620 g/mol) compared to the ED-20 oligomer ( $M = 450$  g/mol). Therefore, the expected  $M_c$  value for the T-111 based polymer should be higher than for the ED-20 based polymer. This contradiction, apparently, can be explained by the influence on the value of  $M_c$  additional physical bonds due to the presence of elastic organosiloxane groups (...Si-O-Si ...) in the oligomer T-111, which contributes to a more close contact of chains between crosslinking units.

It is also seen that the effective cross-linking density  $n_c$  for the crosslinked polymer based on the oligester of cyclo-cab oligomer Laprolat-803 is more than three times lower than that for the polymer based on ED-20 and T-111. At the same time the effective cross-linking density  $n_c$  for the crosslinked polymer based on the oligester of cyclo-cab oligomer L-803 is more than three times lower than that for the polymer based on ED-20 and T-111. This is primarily due to the presence flexible aliphatic ether (oxyalkylene) fragments between cyclocarbonate functional groups in the structure of oligomer L-803.

There is a regular monotonic increase of modulus of high elasticity  $E_{he}$  and the crosslinking density, and, accordingly, a decrease in  $M_c$  as increasing the content of ED-20 and T-111 in the mixture. At the same time, the crosslinking density for compositions ED-20:Laprolat-803 during curing without heat supply is noticeably higher than for compositions T-111:Laprolat-803.

An inverse relationship is observed for the heat-treated samples and, as can be seen, the absolute values of the effective crosslinking density of polymers based on T-111:Laprolat-803 mixtures are higher compared to mixed compositions ED-20:Laprolat-803.

The results also show that the relative increase in the crosslinking density for the compositions T-111:Laprolat-803 after heat treatment is significantly higher than that for the ED-20:Laprolat-803 mixtures. Therefore, that the processes of structuring of mixed compositions under conditions without heat supply are more retarded in the presence of the epoxy-organosilicon oligomer T-111 in comparison with the epoxydian oligomer ED-20.

The results of the studies of the dynamic mechanical properties of crosslinked epoxyurethane compositions are shown in Figure 1-3, which shows the temperature dependences of the mechanical loss tangent ( $tg\delta$ ) and the dynamic shear modulus ( $G'$ ) depending on the ratio of epoxy oligomer: oligester cyclo-cab oligomer (Fig. 1, 2), chemical the nature of the epoxy oligomer (Fig. 3).

It can be seen from Fig. 1-3 that the temperature dependences of  $G'$  and  $tg\delta$  for all compositions have approximately the same appearance. Three areas can be distinguished on the curves of the above dependences.

For the first region at low temperatures, where a comparatively slow and smooth decrease in the shear modulus is observed, as well as a smooth increase in the mechanical loss tangent. Epoxyurethane polymers are in a glassy state in this area. For some compositions, low intensity  $tg\delta$  peaks ( $\beta$ -transition) are observed, which are apparently

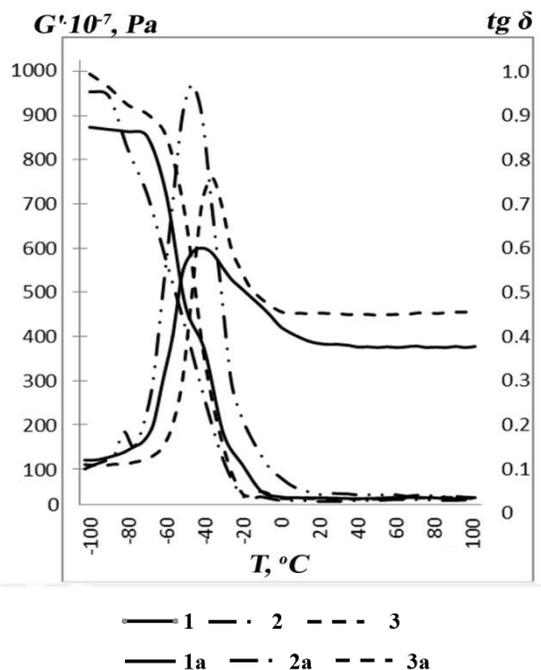
associated with the mobility of small-scale aliphatic oxyether chain fragments and unrealized terminal functional groups in this temperature range.

There is a sharp decrease of shear modulus and an increase of mechanical loss tangent with a further increase in temperature. Such significant changes in the temperature dependence of these parameters are usually associated with the main temperature transition (the region of the  $\alpha$ -relaxation process) of the polymer from the glassy state to the highly elastic state.

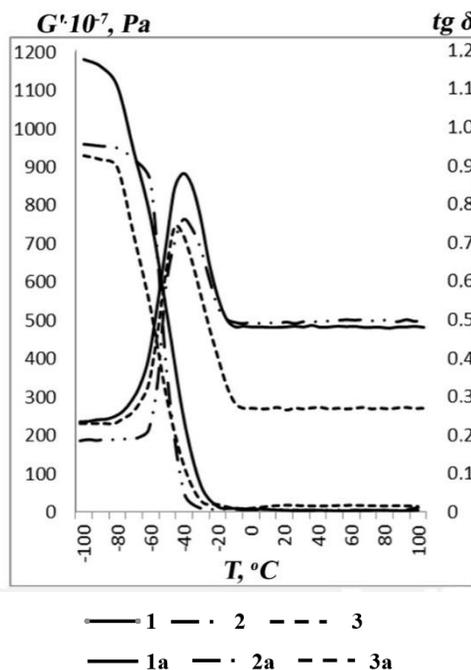
The maximum value of  $tg\delta$  in this temperature range corresponds to the mechanical glass transition temperature  $T_g$ .

The third region is the region of the highly elastic state (high elasticity plateau), which characterized by an almost constant and low shear modulus. The  $tg\delta$  values in the highly elastic state are also characterized by relative constancy.

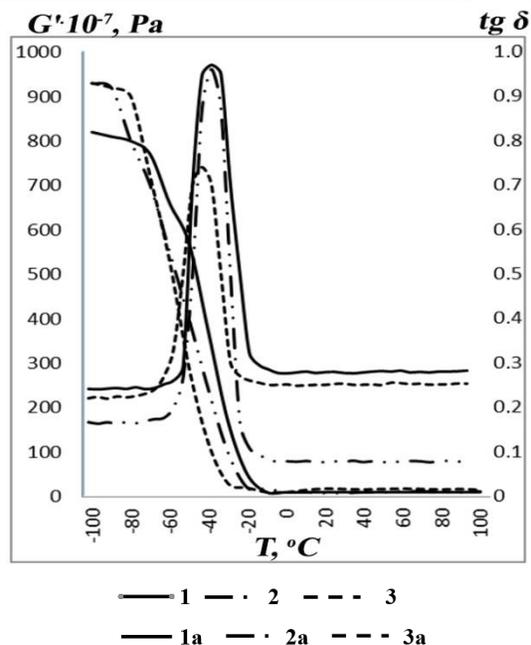
Let us consider in more detail the dynamic mechanical properties of the studied epoxyurethane systems.



**FIGURE 1.** Temperature dependences of the dynamic shear modulus  $G'$  (1-3) and mechanical loss tangent  $tg\delta$  (1a-3a) of epoxyurethane compositions on the ratio of oligomers ED-20:Laprolat-803=10:90 (1, 1a), 20:80 (2, 2a), 30:70 (3, 3a) wt.%.



**FIGURE 2.** Temperature dependences of the dynamic shear modulus  $G'$  (1-3) and mechanical loss tangent  $tg\delta$  (1a-3a) of epoxyurethane compositions on the ratio of oligomers T-111:Laprolat-803=10:90 (1, 1a), 20:80 (2, 2a), 30:70 (3, 3a) wt.%.



**FIGURE 3.** Temperature dependences of the dynamic shear modulus  $G'$  and mechanical loss tangent  $tg\delta$  of epoxyurethane compositions based on the mixture epoxy oligomer: Laprolat-803 for ED-20 (1, 1a), T-111 (2, 2a), UP-655 (3, 3a) wt.%.

**TABLE 2.** Viscoelastic and damping properties of epoxyurethane compositions at different contents of epoxy oligomers (ED-20, T-111, UP-655) in a mixture with oligester cyclo-cab oligomer Laprolat-803

The ratio of the epoxy oligomer mixed with Laprolat-803, wt.%		$G'$ (glassy state), GPa	Glass transition temperature ( $T_g$ ), °C	$tg\delta_{max}$	$G'$ at $tg\delta_{max}$ , GPa	$G''$ at $tg\delta_{max}$ , GPa	Devitrification interval, degrees
ED-20	10	8.5	-40	0.6	3.0	1.8	54
	20	9.2	-45	0.97	3.4	3.3	60
	30	9.7	-34	0.76	2.4	1.8	40
T-111	10	9.6	-42	0.76	0.75	0.56	36
	20	9.7	-45	0.74	2.0	1.48	50
	30	11.8	-38	0.88	2.3	2.0	38
UP-655	20	8.3	-38	0.98	3.2	3.14	46

As can be seen from Fig.1 and Table 2 with an increase the content of the epoxidian oligomer ED-20 from 10:90 to 30:70 wt.% in mixture with oligester of cyclo-cab oligomer the glass transition temperature  $T_g$  of epoxyurethane compositions first decreases by 5°C passing through minimum at content of 20 wt.% ED-20, and then shifted to the region of higher temperatures by 11 °C with simultaneous expansion of the peak  $tg\delta$  and decrease in maximum values. At the same time, for the composition of ED-20:Laprolat-803= 20:80 wt.% there is a narrowing of the peak of mechanical losses and an increase in the maximum value of  $tg\delta$  to 0.98. A similar simultaneous change in the indicated characteristics of the peak of mechanical losses indicates an increase in the cooperatively of the process of thawing the molecular mobility of the main segments of the chain polymer network, which is typical for polymers with a more

uniform structure. On the other hand, with this ratio in a highly elastic state, the highest level of  $tg\delta$  is characteristic of the composition ED-20:Laprolat-803=20:80 wt.% and is about 0.45.

Data analysis (Fig. 1, 2 and in Table 2) also shows that in the low-temperature region (-100 to -80 °C), the dynamic modulus of elasticity increases monotonically with an increase in the content of the epoxydian oligomer in the mixture of oligester cyclo-cab oligomer (Fig. 1). Obviously this is primarily due to the fact that the epoxyurethane polymer composition is enriched with a more rigid component, due to the presence of aromatic fragments in the chemical structure of ED-20 and the appearance of additional polar hydroxyl groups as a result of the reaction of the epoxy group with an amine, which contribute to the formation of a network of stronger intermolecular interactions. A similar increase of the shear modulus in the glassy state is observed in the case of an increase in the proportion of the organosilicon epoxy oligomer T-111 to 30% in the T-111:L-803 system. However, the level of shear modulus values for polymer compositions T-111:L-803 is slightly higher compared to compositions ED-20:L-803. Apparently, this is due to the presence in the structure of T-111, in addition to hard aromatic rings, also flexible siloxane fragments, which provide a greater intermolecular interaction of chains between cross-linking sites in the glassy state. It should be noted that the presence of siloxane units in the main chain of oligomer T-111 is also reflected in the glass transition temperature of binary compositions (Table 2). Thus, the glass transition temperature (peak of the maximum  $tg\delta$ ) for the composition T111:L-803=10:90 and 30:70 is shifted to the region of lower temperatures by two to four degrees in comparison with similar compositions for the ED-20 oligomer.

The results obtained also show that the change of dynamic properties with an increase in the content of ED-20 and T-111 are ambiguous.

So on one side, at the content of 20% of the indicated oligomers in the mixture, an increase in the peaks of the maximum  $tg\delta$  values and their narrowing are observed, which indicates the ordering of segmental mobility and an increase in structural homogeneity. On the other side, the shift of the  $tg\delta$  peaks and, accordingly, the glass transition temperature by 3-5 °C to lower temperatures, an expansion of the defitrification interval, a steeper decrease in the shear modulus in the glassy state and the appearance of additional  $tg\delta$  peaks of low intensity in the range (-80) to (-70) °C speak about some loosening of the structure. With a further increase of concentration of ED-20 and T-111 to 30 wt.% and with an increase of temperature, an even sharper decrease in the elastic modulus in the glassy state (at -100 °C) is observed, which indicates a further loosening of the structure of the corresponding polymers in the glassy state. Obviously, this is explained by the fact that the density of the cross-links increases (Table 1), and the number of bulky aromatic structures that reduce the efficiency of intermolecular interaction in the glassy state, as a result of which the molecular mobility of a local nature, due to the movement of fragments of hydroxyurethane chains, will be "thawed" at lower temperatures with an increase in the content of ED-20 and T-111. Besides, increasing the crosslinking density and the content of aromatic fragments in the epoxyurethane polymer with the introduction of 30% ED-20 and T-111 contributes to a shift in temperature to the region of high temperatures by 11 degrees and 7 degrees, respectively. Wherein, as can be seen from Table 2, the transition interval from the glassy state in the highly elastic state is noticeably narrowed and makes 40 degrees (for 30% ED-20) and 38 degrees (for 30% T-111).

As you can see from the above data, the chemical structure of the epoxy oligomer significantly affects the viscoelastic and damping properties of epoxyurethane compositions. Therefore, we also studied compositions containing an aliphatic chlorine-containing epoxy oligomer UP-655 in a mixture with L-803 (Fig. 3 and Table 2). As seen, all investigated epoxyurethane systems are characterized by one region of «defitrification», as evidenced by the monomodal peak of the maximum  $tg\delta$ , and therefore we can speak of the formation of a relatively homogeneous system. The introduction of 20% aliphatic epoxyoligomer UP-655 in comparison, for example, with aromatic ED-20, leads to a decrease in the shear modulus in the glassy state (at -100 °C) by 10% and its slight change with an increase in temperature up to -70 °C, to a narrowing the temperature range of «defitrification» of the epoxyurethane polymer by 24 °C and an increase in the glass transition temperature by 7 °C. This behavior of the polymer in the presence of the UP-655 epoxy oligomer, is associated with its aliphatic nature, namely the presence in the main chain of flexible oxyethylene units and side strongly polar chlorine atoms between crosslinking sites This provides a higher degree of intermolecular interaction between the chains in the glassy state and as a result of which the local molecular mobility of aliphatic fragments of the chains characteristic of a mixture of L-803 and UP-655 is «defrosting» at a higher temperature and at the same time a high damping capacity is achieved (the maximum  $tg\delta$  is 0.98, the loss modulus is 3.14 GPa).

As can be seen from Fig. 2, the highest values of  $tg\delta=0.48-0.5$  in highly elastic state are observed for epoxyurethane materials based on a mixture of ED-20:L-803 at a ratio of 20:80 wt.%, so this composition can be used as the basis for creating vibration-absorbing mastics.

## CONCLUSION

The dynamic mechanical and vibration-absorbing properties of epoxyurethane coatings for vibration protection of metal products have been studied. It has been established that the chemical structure of modifying epoxy oligomers and their content in a mixture with oligester cyclo-cab oligomer affects the structural parameters of the epoxyurethane polymer matrix, which makes it possible to regulate their viscoelastic and damping properties. As a result of dynamic mechanical studies, it was determined that with an increase in the content of epoxy oligomers (ED-20, T-111 and UP-655) in a mixture of oligester cyclo-cab oligomer L-803 from 10:90 to 30:70 wt.% observed of increase the dynamic modulus of elasticity and glass transition temperature. This is due to the fact that the epoxyurethane polymer is enriched with more rigid components: when using ED-20 and T-111, which contain aromatic fragments in their structure and chloraliphatic oligomer UP-655, which formation the matrix of strong intermolecular interactions due to strongly polar chlorine atoms. It has been established that epoxyurethane systems are characterized by one area of «devitrification», as evidenced by the monomodal peak of the maximum  $tg\delta$ , and therefore it is possible to indicate the creation of a relatively homogeneous system.

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