THERMAL STABILITY OF ORGANIC-INORGANIC COMPOSITES BASED ON DIMETHACRYLATE-TETRAETHOXYSILANE SYSTEM

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Abstract. The influence of composition of hybrid organic-inorganic composites based on oligoesteracrylate (MGF-9)−tetraethoxysilane (TEOS) system on their thermal properties and molecular structure was investigated. Thermograms and curves of thermal destruction of samples were obtained, from which temperature ranges, weight loss of samples and thermal effects of each stage were defined. It is shown that introducing an inorganic filler into polymer matrix promotes increasing of thermal stability of the material. Values of characteristic parameters of studied organic-mineral composites were calculated as the result of thermomechanical analysis of obtained curves. The composite MGF-9:TEOS = 90:10 (vol %) was found to have the maximal thermal resistance and improved thermomechanical properties.

Keywords: thermogravimetry, differential thermal analysis, thermomechanical analysis, organic-inorganic composite, sol-gel system.

1. Introduction

Development of new composite materials based on organic-inorganic systems is a perspective trend in modern science of materials. Successful combination of organic and inorganic components of the system, their ratio and conditions of synthesis makes it possible to aggregate the properties of individual components in composites obtained. This helps improving their performance and allows getting new unique properties. Hybrid organic-inorganic composites (HOIC) are promising as protective coatings, polymeric electrolytes, membranes for use in medicine, optics, microelectronics, *etc*. [1].

One of the common methods of getting the HOIC is sol-gel synthesis and their formation in the process of compatible polymerization from liquid mixtures of organic and inorganic components. When using sol-gel technology an inorganic component is binding (covalently or by means of physical interactions) with the organic component with the formation of composite materials that are characterized by an uniform distribution of inorganic component in the matrix and the absence of significant phase segregation [2]. The use of silica fillers leads to improving physico-mechanical properties and durability of coatings, heat and water resistance, temperature and solar radiation. To achieve a desired set of properties it is often enough to introduce a small amount of functional silica additives into a composition based on traditional polymer [3].

However, the overall patterns of interrelations between the ratio of initial components and properties of composites are not defined. Among the factors affecting the HOIC properties the physical factors play an important role. The emergence of interphase interaction, geometric limitation of polymerization space, growth of polymer free volume while adding an inorganic component − all these facts affect physical, chemical and mechanical properties of the composites [4].

Recently we have studied the kinetics of photoinitiated polymerization of oligoesteracrylate−tetraethoxysilane system to deep conversions. It was shown that values of the maximum polymerization rate, conversion and time to achieve this conversion depend on MGF-9:TEOS ratio in the composition. It was found that the polymerization rate decreases with the increase of inorganic components content both at the equal time of the previous gelation, as well as with its increase [5]. Thus for the compositions with TEOC low content (2.5 and 5 vol %) the parameters of polymerization practically do not differ from pure MGF-9, and the increase of the inorganic component leads to a sharp decrease in the maximum rate of polymerization (2-3 times regardless of previous gelling time). Such behavior may indicate

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different mechanisms of interaction between the components in the polymer as a function of the quantitative composition of the composite. It is known that the singularities of the structural molecular organization of the polymer affect their thermomechanical properties [6]. This work is aimed to study the effect of TEOS content on the thermal resistance and thermo-mechanical properties of organic-inorganic compositions MGF-9:TEOS and their molecular structure.

2. Experimental

For the studies we used oligoesteracrylate monomer (MGF-9) of the formula $CH_2=C(CH_3)–C(O)–(CH_2CH_2O)_3–O–C(O)–C_6H_4–C(O)–O–(OCH_2CH_2)_3–C(O)–C(CH_3)=CH_2$ (P.A. purity, Aldrich); photoinitiator 2,2-dimethoxy-1,2-diphenylethane-1-on (IRGACURE 651) of formula $C_6H_5–C(OCH_3)_2–C(O)–C_6H_5$ (P.A. purity, Fluka); tetraethoxysilane $Si(OC_2H_5)_4$ ("ECOS-1", Russia); ethanol C_2H_5OH (P.A. purity); orthophosphoric acid H_3PO_4 (P.A. purity). The monomer was purified by mixing with pre-activated Al_2O_3 powder and subsequent centrifugation.

The objects of the study were composites samples of MGF-9:TEOS systems at ratios of the initial components (vol %): 80:20 (Sample 1); 90:10 (Sample 2), 95:5 (Sample 3); and 97.5:2.5 (Sample 4). In addition, pure MGF-9 + IRGACURE 651 (2 mol %) was investigated for the comparison (Sample 5). These composites were prepared by the method of photoinitiated polymerization. The content of photoinitiator in all cases was 2 mol % per monomer. The solution of photoinitiator in monomer was mixed with the previously prepared sol-system with the ratio of $TEOS:H_2O:C_2H_5OH:H_3PO_4 = 2.2:0.36:4.08:0.0072$ (ml) by using a magnetic stirrer during 20 min at room temperature. The initial compositions obtained on the basis of MGF-9:TEOS system were placed in a glass test tubes with the help of dispenser. The tubes were closed by cover glass that excludes air access. The compositions were exposed to photoinitiated polymerization under the influence of UV-irradiation of a mercury-quartz lamp DRT-400 with intensity of 48 W/m^2 for full completion of the polymerization. The obtained samples were kept in the oven at 313 K for a week to complete the passage of sol-gel processes in the polymer matrix. Samples for thermomechanical analysis were prepared in the form of a cylinder with the diameter of 9.0 mm and the height of 10.0 mm. To perform thermal studies, the obtained samples were grinded to powdered state by using a vibrating mill.

Thermal studies of powders of MGF-9:TEOS system have been conducted on the basis of integrated thermogravimetric analysis and differential thermal

analysis using Q-1500D derivatograph (F. Paulik−J. Paulik−L. Erdey). Analytical signals were registered by computer. The samples were analyzed under dynamic mode at the heating rate of 5°/min in air atmosphere. The sample weight was 200 mg. The reference substance was alumina. The studies were conducted in the temperature range of 293–873 K. The sensitivity on a scale of thermogravimetric analysis (TG) was 200 mg, on a scale of differential thermal analysis (DTA) − 250 mV, on a scale of differential thermogravimetric analysis (DTG) − 500 mV. This method consists in measuring weight loss of the sample during its continuous heating and provides detailed information about the physical and chemical processes, temperature and intensity of their occurrence.

Thermomechanical analysis of the synthesized samples was performed using the device made by "Heckert" company (Medingen, Germany) under the mode of uniaxial compression with the load of $5.3\cdot10^5 N/m^2$ and the heating rate of 1.5°/min. Sample deformation was determined using a null indicator with the accuracy of 0.01 mm. The temperature of the sample during the experiment was measured using a standard TCK thermocouple. Thermomechanical method of analysis reflects the behavior of composites under the influence of temperature and mechanical stress.

3. Results and Discussion

As a result of thermolysis of investigated composites thermograms were obtained, which are a set of curves of TG, DTG and DTA. TG and DTG curves reflect the change of weight loss of sample and its rate of change. Curve DTA reflects the change of temperature difference of the sample and inert substance and characterizes the type and amount of thermal effect. Figs. 1-3 show a comparison of TG, DTG and DTA curves of investigated samples.

As seen from the data obtained, curves of thermal destruction of composites despite being similar in shape are characterized by certain differences depending on the content of inorganic component in the MGF-9:TEOS system.

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The process of thermal destruction of all investigated samples is a multistage, which is indicated by the presence of at least four DTG-curves. Table 1 shows the temperature ranges of each stage and change in mass of all samples. The most intensive weight loss of samples is observable in the temperature range of 523–683 K.
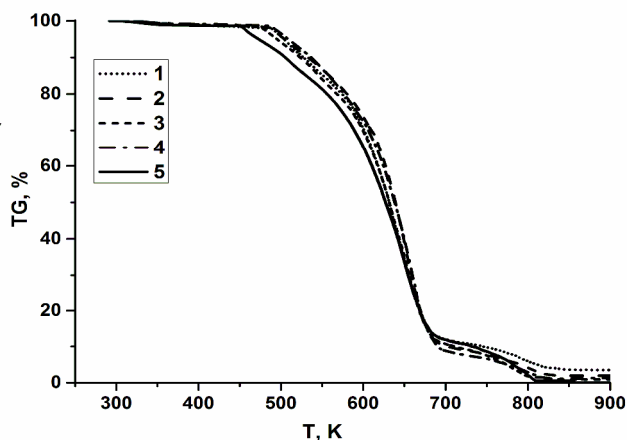
Fig. 1. TG-curves of thermal destruction of MGF-9:TEOS composites with different content of TEOS, vol %: 20 (1); 10 (2); 5 (3); 2.5 (4) and 0 (5)
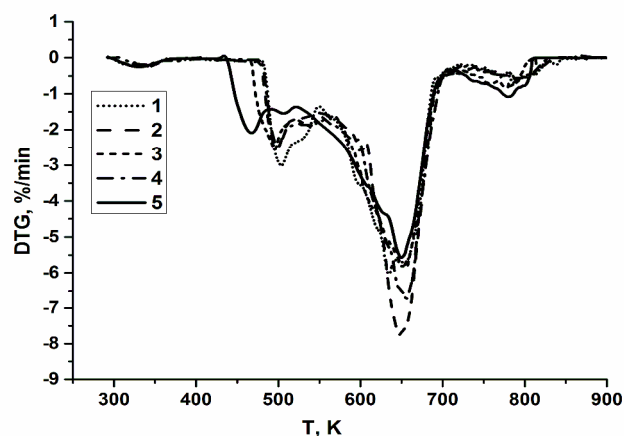

Fig. 2. DTG-curves of thermal destruction of MGF-9:TEOS composites with different content of TEOS, vol %: 20 (1); 10 (2); 5 (3); 2.5 (4) and 0 (5)
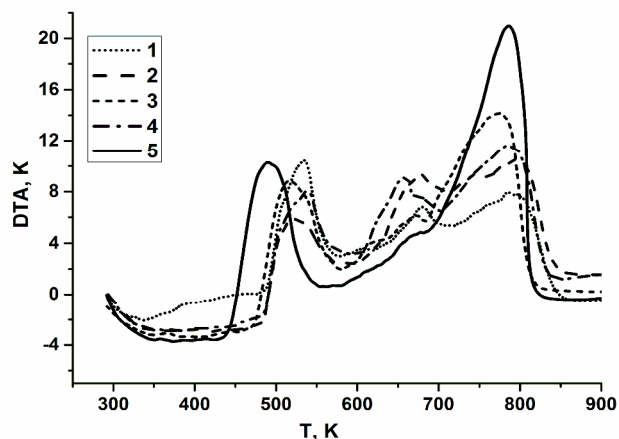

Fig. 3. DTA-curves of thermal destruction of composites MGF-9:TEOS with different content of TEOS, vol %: 20 (1); 10 (2); 5 (3); 2.5 (4) and 0 (5)

Table 1

The results of thermal analysis of samples of MGF-9:TEOS system

	Stage	MGF-9:TEOS system				
		Sample 1 80:20	Sample 2 90:10	Sample 3 95:5	Sample 4 97.5:2.5	Sample 5 Pure MGF-9
Temperature range, K	I	293–485	293–481	293–471	293–485	293–439
	II	485–576	481–586	471–573	485–597	439–527
	III	576–721	586–716	573–693	597–704	527–688
	IV	721–863	716–849	693–833	704–846	688–933
Weight loss, %	I	2.04	1.62	1.57	1.38	1.33
	II	18.5	20.03	19.63	25.19	13.11
	III	68.24	69.31	67.49	64.38	72.73
	IV	7.54	7.32	10.2	7.35	12.83

The occurence of endothermic effect is observable in Samples 2−5 on the DTA-curves at the first stage of the thermolysis (temperature range of 293−485 K). This effect is accompanied by negligible weight loss and corresponds to the release of volatiles from the composition, and beginning of samples softening.

However, deviation of DTA-curve in the region of exothermic effects is observed on the thermogram of Sample 1 having maximum amount of TEOS, unlike others samples, in the temperature range of 383−485 K. This can be explained by the completion of polymerization processes of composite inorganic component.

At the second stage of thermolysis (the temperature range of 471−597 K) on the TG curves of Samples 1−4, we observe small weight losses with occurrence of clear cut exothermic effects on DTA curves. All this corresponds to simultaneous processes of thermal and thermooxidative destruction in composites. Sample 5 (pure MGF-9) is characterized by lower thermal resistance compared to composite samples; thermooxidative and destructive processes in this sample are shifted into the region of lower temperatures (439−527 K).

Sample 3 is characterized by lower thermal resistance in comparison with other composite samples (MGF-9:TEOS=95:5 (vol %)). This sample loses weight much intensively than the others and beginning of thermal destruction and maximum value of DTG curve extremum are shifted to the region of lower temperatures (Fig. 2). Thermooxidative processes in Sample 3 cause more intense exothermic effects on DTA curves compared to other composites (Fig. 3).

Samples 1, 2 and 4 (content of TEOS are 20, 10 and 2.5 vol %, respectively) are characterized by higher thermal resistance. Thermooxidative and destructive processes in these samples occur with lower weight losses and the extremums appeared on DTG curves are not so deep (Figs. 2 and 3), which may indicate their higher thermal stability.

The combustion process of thermal destruction products of composite samples at the third stage of thermolysis takes place in the temperature range of 573−721 K. This process is characterized by the most intensive weight loss of samples (Fig. 1), occurrence of the deepest minimum on DTG curves (Fig. 2) and the most intensive exothermic effect on DTA curves (Fig. 3). For Sample 5 (Pure MGF-9) combustion of residual matter of thermal destruction is displaced to the region of lower temperatures (527−688 K).

The fourth stage of thermolysis exhibits the combustion of carbonated residual matter of samples in the temperature range of 688−933 K. This process corresponds to the emergence of the most intensive extremums on DTA-curves. The combustion process of pyrolitic residual matter in Sample 5 ends with the full weight loss and is accompanied with the most intensive thermal effect on DTA curve (Fig. 3).

Summarizing the results of thermal study one can say that the presence of inorganic component in composite samples leads to the increase of their thermal resistance. However, dependence of thermal resistance on the TEOS amount in samples is quite complicated, which is probably associated with the structure of the composites.

For determination of structural and molecular parameters of the studied composites and for the study of their mechanical behavior under action of both temperature and mechanical stress, thermomechanical analysis of the composites MGF-9:TEOS with different composition of the inorganic component was conducted.

The results of thermomechanical analysis of MGF-9:TEOS composites are presented as strain dependence ε on temperature ($\varepsilon = \Delta h/h_0$, where Δh − sample deformation, h_0 − sample initial size; Fig. 4).

The obtained thermomechanical curves (Fig. 3) generally look like spatial cross-linked polymers, but the nature of TMA curves of composites markedly differs from pure MGF-9 polymer. Three temperature areas typical of spatial cross-linked polymer can be distinguished at TMA data curves: initial area of glassy state, high elastic area and thermomechanical fluidity area (final one).

TMA method allows to define characteristic temperatures T_{glass}, T_{hel} and T_{tmf} of composites and assess physical, mechanical, structural, and molecular characteristics of composites in the form of high elastic equilibrium modulus E_∞ (N/m^2), kinetic molecular weight segment (macrochain segment between nodes of structural net) of composites M_c (g/mol) and amount of segments (effective cross-linking nodes) in volume unit n_c (mol/m^3).

Within statistical elasticity theory of molecular grid of spatial crosslinked polymer the dependence of crosslinking density (M_c) on the high elastic modulus E_∞ can be described as [7]:

$$M_c = \frac{3grRTu}{E_\infty u_0} \qquad (1)$$

where M_c − molecular weight of kinetic segment; E_∞ − the high elastic modulus; γ − structural coefficient depending on nature and topology of the grid; ρ − sample density; v_0 − total number of cross-links; v − number of physically effective reactions that form spatial structure; R − gas constant; T − transition temperature in the high elastic state, К.

Quantitative checking of this correlation is rather complicated since the proportion of functional groups participating in the formation of physically active links is unknown. Even the greater uncertainty is associated with the assessment of structural factor γ. If $E_¥ \leq 6{\cdot}10^7$ N/m^2 we can assume that $u = u_o$, $g = 1$ [8]. In this case, the calculation of Eq. (1) is substantially simplified.

The high elastic modulus E_∞ is calculated by Eq. (2):

$$E_\infty = \frac{P}{F \cdot e} \qquad (2)$$

where P − load on the sample, N; F − cross sectional area of the sample to which force m^2 is applied; ε − relative deformation of the sample in high elastic area.
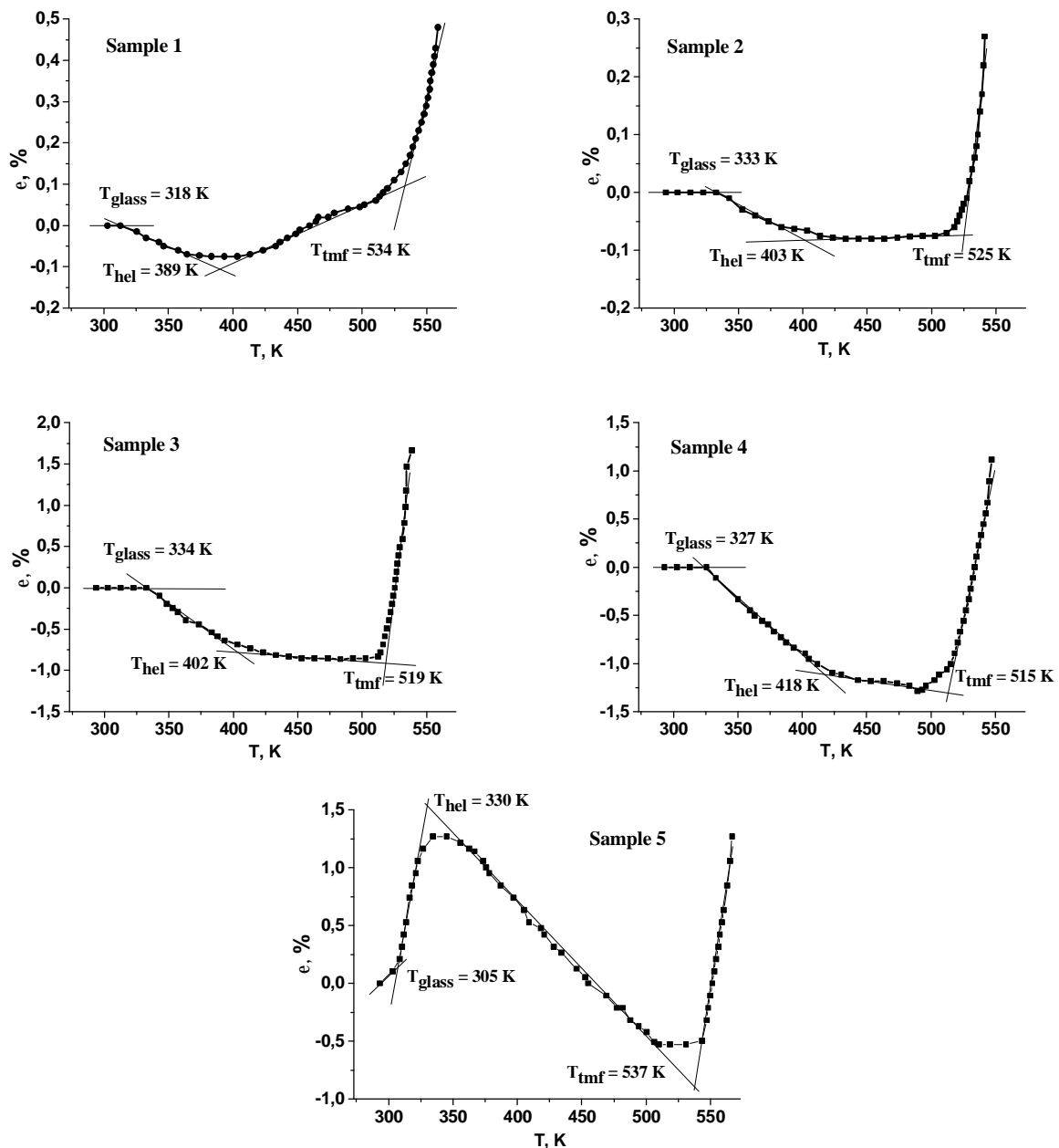
Fig. 4. Thermomechanical curves of TEOS:MGF-9 composites
with components ratio (vol %): 80:20 (1); 90:10 (2); 95:5 (3); 97.5: 2.5 (4) and 100: 0 (5)

Table 2

**Thermomechanical properties and structural parameters
of molecular composites synthesized on the basis of MGF-9:TEOS**

MGF-9:TEOS system		T_{glass}, K	T_{hel}, K	T_{tmf}, K	$E_{\infty} \cdot 10^{-8}$, N/m²	M_c, g/mol	$n_c \cdot 10^{-3}$, mol/m³
Sample	vol %						
1	80:20	318	389	534	4.90	24.34	50.53
2	90:10	333	403	525	6.43	19.06	64.01
3	95:5	334	402	519	0.68	178.33	6.79
4	97.5:2.5	327	418	515	0.48	271.37	4.61
5	pure MGF-9	305	330	537	0.35	289.12	4.25

The M_c value is a measure of crosslinking density, which, consequently, is the determining factor of the full range of polymer physical and mechanical properties (strength, hardness, relaxation properties, *etc*.). This value gives the opportunity to estimate the amount of segments (effective cross-linking nodes) in volume unit n_c (mol/m³):

$$n_c = \frac{r}{M_c} \qquad (3)$$

An average density ρ defined as mass per unit volume of sample dried to constant weight in a natural state. Because the sample has correct geometric form (cylinder), its volume is determined by measuring the length, width and diameter using calipers (accuracy – 0.05 mm) and weight − weighing on an analytical balance (accuracy − 0.0002 g).

The obtained values of characteristic parameters of studied organic-mineral composites are shown in Table 2.

The initial segment of thermomechanical curve characterized by a dramatic increase of deformation in a narrow temperature range, resulting in mobility increasing of kinetic segments of polymer matrix (α-relaxation process) corresponds to a structural transition of polymer from the glassy state into the high elastic one (Fig. 4, *curve* 5). However, introducing the inorganic component into system leads to descending branch of TMA curve and shifts it into higher temperature areas. Negative values ε for studied samples indicate the presence of internal stresses, which value is higher than the external load applied. It is associated primarily with the method of sample preparation in the block for TMA analysis. So when heated to the polymer softening temperature, these internal stresses relax, and sizes of samples grow. If the temperature grows then high elastic deformation of polymer occurs independently of external factors or internal forces. In this case the temperature of polymer vitrification T_{glass} can be defined by extrapolation of linear section at the temperature axis [6].

It is known that T_{glass} of filled system is defined by the concentration of cross-link units, deposition of interfacial layers and physical structuring of polymeric system driven by filler. The results obtained show that introducing the inorganic component into system leads to significant increase in vitrification temperature compared to pure polymer (from 305 to 330 K for the system containing 2.5 vol % of TEOS). This can be explained by physical structuring of polymeric system with small concentration of filler (2.5 vol % of TEOS). According to the microheterogeneous concept of radical polymerization polymer MGF-9 is characterized by granular structure in which polymer grains are connected with each other by hydrogen bonds or separate monomer molecules into a single network structure [9-11]. This structure is charac-

terized by certain defect of structure. When introducing a small amount of inorganic component, the process of its inclusion into defective and weakly linked areas of polymeric matrix takes place [12, 13].

In such a way the decrease of matrix defects, the increase of ordering and density of defective areas causes the increase of structure rigidity of tallowed polymer and speed stagnation of α-relaxing processes in polymeric matrix. Simultaneously, length of interfacial segment of polymeric matrix is decreasing from 289 to 271 g/mol and the value of rubber elasticity modulus is growing, indicating the appearance of "short-range" ordering.

The increase of TEOS amount to 5 vol % marginally changes the thermomechanical properties of the composite. Further increase in rubber elasticity modulus and reduction of interganglionic composite segment length, increase of the glass transition temperature (T_{glass}) and thermomechanical fluidity temperature (T_{tmf}) at simultaneous decrease of temperature of high elasticity of composite (T_{hel}) are observed.

Further growth of inorganic component concentration up to 10 vol % stabilizes vitrification temperature at the level of 333 K but at the same time sharp increase of high elastic modulus (from $6.38 \cdot 10^7$ to $6.43 \cdot 10^8$ N/m²) and decrease in molecular mass of kinetic segments by an order of magnitude ($M_c = 19.06$ g/mol) is observed. These changes are probably caused by formation of tridimensional net of inorganic component leading to greater ordering of polymeric matrix. Additional linked nodes are forming between polymer molecules explaining the increase in elasticity level.

Simultaneously, the increase of the crosslinking degree of the polymer with increasing concentrations of inorganic component leads to limited mobility of kinetic segments of the polymer matrix and, correspondingly, to the decrease in internal pressure of the system. This is confirmed by the decrease in the value of sample deformation. (Fig. 4, *curve* 2).

High concentration of inorganic component (TEOS content is 20 vol %) leads to demixing of formed phases, as indicated by the decrease in the value of high elastic module and increase of molecular mass of kinetic segment. It causes the decrease in vitrification temperature and temperature of high elasticity of composite (up to $T_{glass} = 318$ K and $T_{hel} = 389$ K).

TMA-curve horizontal area is an area of elastic deformation. Composite behavior strongly depends on the presence of inorganic component in the system. We can observe decrease of relative deformation up to changing the sign for pure monomer sample of this area at the temperature growth. It is associated with temperature influence on the grain structure of the polymer matrix. Temperature growth leads to weakening of ties among separate densely cross-linked polymer grains, enhances

their mobility and gradually reduces internal stresses of polymer chain in some grains, which causes growth in resistance of polymeric matrix to external load [9, 10, 14].

Introduction of inorganic component changes the nature of high elastic area of composite. Viscoelasticity plateau is distinctive for spatial-structured polymers, *i.e.* the appearance of this area is caused by formation of spatial net that blocks mobility of polymer macromolecules. Plateau length indicates the growth of frame stiffness that retains a transition of polymeric matrix into plastic state. This is correlated by shifting value T_{tmf} in the direction of higher temperatures at quantitative growth of inorganic phase from 2.5 to 10 vol %. Jump-like growth of deformation on TMA-curve for composite with the highest content of inorganic component (Fig. 4, *curve* 1), is associated primarily with micro disintegration of phases formed. The destruction of ties in each phase takes place at different temperatures. Deformation in the range of 373–473 K corresponds to softening of the polymer component of the composite, at temperatures above 473 K the partial destruction of inorganic three-dimensional net takes place.

Sharp deformation growth can be observed at the temperatures higher than 503–523 K in narrow temperature range that corresponds to the area of nonreversible deformation. Such type of deformation is related not only to disruption of interchain correlations but also to macromolecules disruption. This is an area of thermomechanical fluidity of polymeric matrix, T_{tmf} value is a measure of deformational stability of polymeric composite under mechanical and temperature loads.

According to the results, increasing the number of inorganic phase leads to T_{tmf} increase, which indicates growing role of inorganic component. A high T_{tmf} or pure polymer (Sample 5) can be explained by the occurrence of polymerization of residual monomer that happens due to the formation of additional crosslinking units of the polymer matrix under the temperature [14].

The obtained results showed that the properties of composites based on inorganic and organic spatial-structured polymers depend on the structure of formed composite. In the oligoesteracrylate MGF-9 the carbonyl groups in the chain lead to formation of weak hydrogen bonds between organic and inorganic components of the system [4]. Thus, the composite material, depending on the ratio of the components can be characterized by different structures at the moment of its formation. On the one hand, there are organic molecules included in the spatial structure of the obtained gel. On the other hand, there are inorganic molecules or their agglomerates integrated inside the organic macrostructures [15].

We can assume that introducing a small amount of TEOS (2.5 vol %) into a polymer matrix leads to formation of a structure in which inorganic component is embedded in the polymer matrix in the form of nanoparticles. Since embedding in the polymer matrix occurs primarily in microareas with lower density, *i.e.* defective and weakly crosslinked areas, it causes reduction of defects in polymeric matrix, ordering of defective areas and their compaction, resulting in improving of physical and chemical properties of the system. One can say that the presence of small amount of inorganic component exhibits reinforcing effect on polymer properties and facilitates the effect of small dopes [12, 13], exerting practically no effect on the process of polymer polymerization.

The increase of inorganic component amount to 5 vol % leads to the changes of composite thermal resistance. Such behavior can be explained by the possible release of inorganic components as a separate phase in the form of clusters [16], leading to the formation of transition disordered organo-silicate structures in composite. The presence of the inorganic component may also prevent monolithicity of polymer, explaining the decrease in temperature of the thermal stress as compared with pure MGF-9.

An increase of TEOS amount to 10 vol % (Sample 2) causes formation of space-crosslinked grid of inorganic component and implements so-called interpenetrating structure, that is characterized by high ordering of system. An increase of the regularity of copolymeric chains structure leads to the strengthened attraction of polymeric balls elements. As a result, it enables density increase and, consequently, determines decrease in intensity of thermooxidative destruction [17]. High value rubber elasticity modulus, cross-link density and high heat resistance in comparison with other composites characterize this composite.

The formed structure for the samples with maximum amount of inorganic component is characterized by separation of formed phases. It leads to decreased thermal stability of the composite and increased burnout of organic component at the interface during thermo-oxidative destruction (Fig. 2).

4. Conclusions

This paper shows that introduction of the inorganic filler TEOS into polymer matrix MGF-9 promotes growth in thermal resistance of the material. The dependence "thermal properties of composite − content of TEOS" is characterized by extremum presence, which is caused by the structure of the formed composite. It was determined that composite MGF-9:TEOS = 90:10 (vol %) has the best properties among HOIC samples. This composite has the highest value of rubber elastic modulus and is charac-

terized by high heat resistance in comparison with composites with concentration of inorganic component from 2.5 to 20 vol %.

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ТЕРМІЧНА СТІЙКІСТЬ ОРГАНО-НЕОРГАНІЧНИХ КОМПОЗИТІВ НА ОСНОВІ СИСТЕМИ ДИМЕТАКРИЛАТ-ТЕТРАЕТОКСИСИЛАН

Анотація. *Досліджено вплив складу гібридних органо-неорганічних композитів на основі системи α, w–диметакри-лоїл(тридиетиленоксидтерефталат) (МГФ-9) – тетра-етоксисилан (ТЕОС) на їх термічні і термомеханічні власти-вості та молекулярну структуру. Одержано термограми та криві термічної деструкції зразків, за якими визначено температурні інтервали, втрату маси зразків та теплові ефекти кожної стадії. Показано, що введення неорганічного наповнювача у матрицю полімеру сприяє збільшенню термо-стабільності матеріалу. Внаслідок термомеханічного аналізу одержаних кривих розраховано значення характеристичних параметрів досліджених органо-мінеральних композитів. З'ясовано, що максимальну термічну стійкість та покращені термомеханічні властивості має композит МГФ-9:ТЕОС = = 90:10 % об.*

Ключові слова: термогравіметрія, диференційний тер-мічний аналіз, термомеханічний аналіз, органо-неорганічний нанокомпозит, золь-гель система.