INVESTIGATION OF THE EFFECT OF STRUCTURING METHODS ON THE CHANGE IN RESIDUAL STRESSES IN POLYMER COMPOSITE MATERIAL

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The process of forming the cohesive strength of PCM is associated with a decrease in its volume. If there is no mechanical impact on the material, then this process is called shrinkage. Shrinkage occurs during cooling, when the solvent evaporates and during structuring. The free shrinkage of the PCM is hindered by its adhesive bond with the surface of the product, which is filled with a polymer compound, as a result of which more or less shrinkage stresses develop in the PCM over time. In addition to the latter in the PCM there are thermal internal stresses. Their occurrence is due to the combination of different materials in PCM, which differ significantly in the coefficients of thermal expansion. An effective way to reduce internal stresses is to add to the composition of PCM various fillers and plasticizers, what improves the relaxation properties of PCM. The aim of this work was to study the influence of different methods of structuring (polymerization), i.e. by convection and in the field of high frequency currents of epoxy, acrylic and epoxycrylic PCM. The study of the emerging internal stresses, both shrinkage and temperature, was performed by the method of digital strain gauge, which allows not only to record the final level of residual stresses, but to monitor it in the process of structuring. The studies have shown that a more effective method of structuring is the process of structuring in the field of high-frequency current, which reduces residual stresses, increases the modulus of elasticity and forcing temperature of the studied compounds, what increases the strength and performance properties of PCM, and significantly reduces structuring time, providing uniform heating over the whole volume of PCM. It is recommended to use the obtained data in various industries related to the process of gluing and sealing both homogeneous and heterogeneous materials, as well as PCM products

KEYWORDS: internal stress, shrinkage, curing, high frequency current field, polymer composite material

Residual stresses arising in a polymer composite material (PCM) at curing (structuring), and the relaxation processes, occurring at the same time, affect the strength of PCM and often determine time variation in their operational and technological properties. The reasons for the residual stresses occurrence are: presence of temperature gradients, difference in the coefficients of thermal linear expansion of PCM components, manifestation of shrinkage, occurrence of microcracks, method and mode of curing, and some other factors [1-3].

The magnitude of the residual stresses and the rate of their development depend on the nature of the binder, and fillers, plasticizers and other components contained in it.

Besides, the temperature-time method of structuring or polymerization of PCM is extremely important for the residual stresses occurrence [4-5].

The occurrence of the residual stresses is caused by the incompleteness of relaxation processes in the PCM. Therefore, the problem of the occurrence and reduction of residual stresses is of great practical importance.

The purpose of this work is to study the level of the residual stresses in epoxy, acrylic and epoxy-acrylic compositions with different structuring methods applied: by convection heating and structuring in the field of high-frequency currents.

TEST METHODS

To prepare PCM the following materials were used: Epoxy oligomer of ED-20 grade, GOST 10587, dibutyl ester of phthalic acid (DBP) GOST 8728 was used as a plasticizer. Oligoester acrylate MGP-9 (Specification 6-01-450) was used as a modifying agent. Distilled polyethylene polyamine (PEPA) (Specification 6-02-594) was used as a curing agent. The content of the curing agent in the PCM made 10-15% of the oligomer weight. The low-level amine curing agent dicyanoethylidiethylene-triamine UP-0633 (Specification 6-05-1803) was used as a diluter. AST-T plastic (Specification 64-2-226) was used as a binder for the acrylic composite materials. The curing time of the AST-T plastic at the temperature of 20 ± 0.5 °C ranges from 60 to 90 minutes.

Molybdenum disulfide (Specification 48-19-133), aluminum oxide (Interregional Specification 6-092046), and tale (Specification 2-036707) were used as dispersed fillers.

The content of the compositions investigated are shown in Table 1.

In the course of investigation, a comparative assessment of two methods of structuring (polymerization) – by convection, i.e. heating the compositions under study in the oven, and by curing these compositions in the field of high-frequency currents (HFC) was carried out. For this purpose, a high-frequency device HFD 2-2.5/81 was used, whose basic technical characteristics are given in Table 2.
Table 1.

Content of the compositions under study

| Component          | Amount, weight % |
|--------------------|-----------------|
|                    | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| ED-20              | 100| 100| 100| 100| 100| 100| 100| 100| 100| 100| 100| 100 |
| PEPA               | 1  | 0  | 1  | 0  | 1  | 0  | 1  | 0  | 1  | 0  | 1  | 0  |
| Dibutylphthalat    | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| UP-0633            | 30 | 20 |    |    |    |    |    |    |    |    |    |    |
| MG-9               |    |    | 20 | 20 | 20 | 20 | 20 | 20 | 20 | 20 | 20 | 20 |
| AST-T              |    |    |    |    |    |    |    |    |    |    |    |    |
| CE-6               |    |    |    |    |    |    |    |    |    |    |    |    |
| MoS₂               |    |    | 20 | 20 |    |    |    |    |    |    |    |    |
| Talc               |    |    | 25 | 25 |    |    |    |    |    |    |    |    |
| Al₂O₃              |    |    |    |    |    |    |    |    | 30 | 30 |    |    |

Table 2.

Technical characteristics of the HFD 2-2.5/81 unit

| Parameter name                  | Norm                      |
|---------------------------------|---------------------------|
| Oscillatory power, kW           | 2.5±0.25                  |
| Operating frequency, MHz        | 81.36±0.8136              |
| Rated supply voltage, V         | 380/220                   |
| Weight of the heated material, kg | 0.25±0.6                 |
| Anode current, A                | 0.7±0.3                   |
| Grid current, A                 | 0.2±0.3                   |
| Generating lamp filament voltage, V | 6.0±0.3              |
| Anode voltage, kV               | 5.0±0.25                  |

Standard samples were made of the compositions under study by free pouring into open metal molds. Combining all the components, included into the composition, was carried out in polyethylene containers, with pre-dosing them by weight or volumetric method. In case the samples were made on the bases of an epoxy oligomer, the polymer mass, which before its pouring was thoroughly stirred and evacuated for 10-15 minutes to remove air bubbles, then was poured into metal molds, pre-treated with an anti-adhesive composition (10% solution of HSR rubber in toluene). When pouring samples made of acrylic and mixed compositions, the combined components, polymer and monomer, were previously brought to swelling for 10-20 minutes to the state of the required viscosity, after which they were poured into the molds treated with the above-mentioned separable composition.

The PCM samples, based on epoxydiane oligomer, were structured by convection in an oven at 100 °C for 2 hours; the samples, based on acrylic oligomer, were cured at room temperature (20 ± 2) °C for 60-90 minutes.

When the PCM samples, based on an epoxy oligomer and acrylic polymer were cured in the field of HFC, they were cured in the optimal mode calculated according to a standard computer program.

**RESEARCH METHODS**

The physical and mechanical properties of epoxy and acrylic PCM specimens were determined by standard and laboratory methods, which are described in [6].

To measure stresses, the wire strain gauge method was used [7-9]. This method was used both to determine the residual stresses at various points of the compound and to measure the contact pressure.

To measure the contact pressure, a thin-walled metal cylinder was used, on whose inner surface wire strain gauges were bonded to measure axial and circumferential deformations, what allowed to control the uniformity of shrinkage deformations in the compounds. The cylinder with the bonded strain gauges was put into the mold. Then the model of the tested compounds was molded so that it formed a uniform layer of a given thickness around the cylinder and did not get inside the cylinder. In the course of curing the compound in the curing chamber, some residual stresses in the samples occur, which deform the cylinder. The cylinder deformation and, hence, the forces that caused it were determined by the magnitude of the change in the resistance of the strain gauges.

In the tested mold the metal cylinder relative deformation, caused by contact pressure, was determined by the formula for calculating the deformation of a thin-walled cylinder loaded with a uniformly distributed external load:

\[
\varepsilon = \frac{P \cdot r}{E \cdot \Delta} \left(1 - \frac{\mu}{r}\right),
\]

where: \(\varepsilon\) - relative circumferential deformation of the cylinder;
\[ P = 6.47 \Delta R \]

where \( \Delta R = (D_2 - D_1) \)

\( D_2 \) is the pressure of the unloaded cylinder;

\( D_1 \) - pressure of the loaded cylinder filled with compound;

6.47 - the coefficient for the cylinder.

The same method was used to determine the residual stresses in the compounds in the process of structuring (polymerization) in the HFC generator.

**EXPERIMENTAL RESULTS AND THEIR DISCUSSION**

The residual stresses, arising in a polymer material at curing, and the relaxation processes, which occur in this case, affect the PCM strength and so often determine the time variation of their operational properties. The reasons for the occurrence of residual stresses are: presence of temperature gradients, the difference in the coefficients of thermal expansion of PCM components, manifestation of shrinkage, occurrence of microcracks, method and mode of curing, and others [10-13].

The magnitude of the residual stresses and the rate of their development depend on the nature of the binder, on the fillers, plasticizers and other components introduced into it. Besides, the temperature-time regime of structuring or polymerization of PCMs is extremely important for the occurrence of the residual stresses [14-17]. The occurrence of residual stresses is caused by the incompleteness of relaxation processes in the PCM. Therefore, the problem of the occurrence and reduction of the residual stresses is of great practical importance [18-23].

The residual stresses in PCMs, cured in the field of high frequency current and by the method of convection heating, were investigated. The nature of structural transformations and the degree of the relaxation processes completion are significantly influenced by the method of curing. Thus, the use of HFC for the formation of PCM products allows to carry out uniform curing over the thickness of the material and to obtain PCM with a homogeneous ordered structure.

Figures 1, 2 present the kinetics of the residual stresses formation in the process of convection curing with a smooth rise in temperature for 2 hours, in the field of high frequency current – for 180 seconds.

![Figure 1](image-url). Kinetics of the residual stresses formation in the process of curing by the convection method

When the cured compound is heated, two competing processes occur: a decrease in the system viscosity, which causes a decrease in the residual stresses, and the cross-linking process. The residual stresses will decrease to the point of gel, after which the cross-linking process, which causes a uniform increase in the residual stresses, begins to prevail.
As the figures show, the gel point of epoxy compounds, cured by the convection method, occurs in the temperature range from 60 °C to 90 °C, subject to the compound composition. When the compounds are cured with a highly active curing agent PEPA, gelation occurs at a lower temperature, than when they are cured with a low-active curing agent UP-0633. In the presence of the incompatible DBP plasticizer, the gelation process starts at the temperature 10 °C higher, than with the unplasticized compound. The residual stresses, after completion of the curing process for compounds cured in the HFC field, is 11-10 MPa lower than for those, cured by the convection method. With participation of the MGP-9 modifier, the temperature range of gelation is 42-31% narrower than that for the unplasticized compound, and is shifted towards lower temperatures by 18 °C. The gelation process in the compounds cured with UP-0633 starts, as it has been expected, at the highest temperature.

The considered results show that almost all the compounds, based on oligomer of ED-20 grade, were cured under the optimum temperature conditions, which were close to T_g. The exception makes the compound with MGP-9, whose curing temperature and T_g are somewhat different, what is undesirable. In the course of curing in HFC, the gelation point for all the compounds occurs at lower temperatures, although the process itself proceeds in approximately the same temperature range as for the compounds cured by the convection method, but is shifted towards temperatures lower by 20-25 °C. This fact can be attributed to a more uniform distribution of the temperature field at PCM curing in the high frequency current. The residual stresses after the curing process completion, as can be seen from Table 3, for all the compounds without exception, cured in HFC field, are significantly lower than for those, cured by the convection method.

| Compound composition | σ_{\text{init}}, MPa | σ_{\text{min}}, MPa | σ_{\text{max}}, MPa | Δ σ₁=Σ_{\text{init}}-σ_{\text{min}}, MPa | Δ σ₁=Σ_{\text{max}}-σ_{\text{min}}, MPa | Gelation temperature, °C | Gelation completion temperature, °C |
|----------------------|----------------------|----------------------|----------------------|--------------------------------|--------------------------------|-----------------------------|-------------------------------|
| ED-20+PEPA           | 2.0/1.0              | -3.8/-4.0            | 5.8/6.0              | 9.6/10.0                      | 14.6/10.0                    | 45/45                       | 70/53                        |
| ED-20+PEPA+Mo2S      | 1.1/1.0              | -3.2/-2.6            | 6.1/6.2              | 9.3/8.8                       | 14.5/10.3                    | 45/43                       | 72/48                        |
| ED-20+PEPA+talc      | 1.1/1.3              | -3.5/-3.3            | 7.2/4.8              | 10.7/8.1                      | 16.6/4.8                     | 45/50                       | 63/50                        |
| ED-20+PEPA+Al2O3     | 0.8/1.1              | -4.8/-1.5            | 4.8/3.4              | 9.6/4.9                       | 16.6/4.8                     | 45/43                       | 63/53                        |
| ED-20+PEPA+DBP       | 2.5/0.9              | -4.8/-3.9            | 12.1/6.1             | 14.6/10.0                     | 16.6/4.8                     | 50/43                       | 65/55                        |
| ED-20+PEPA+DBP+Mo2S  | 2.8/0.5              | -4.5/-5.0            | 9.9/5.5              | 14.5/10.3                     | 16.6/4.8                     | 55/46                       | 70/54                        |
| ED-20+PEPA+DBP+talc  | 1.3/1.0              | -5.1/-1.3            | 11.5/3.5             | 16.6/4.8                      | 16.6/4.8                     | 55/42                       | 65/52                        |
| ED-20+PEPA+DBP+Al2O3 | 1.4/0.9              | -3.4/-3.3            | 8.1/2.5              | 11.5/5.8                      | 11.5/5.8                     | 60/43                       | 70/43                        |
| ED-20+PEPA+MGP-9     | 0.5/0.9              | -6.3/-8.0            | 9.2/5.9              | 15.2/13.9                     | 15.2/13.9                    | 50/42                       | 78/55                        |
For acrylic compositions (Figure 1, 2), cured both by the convection method and in the field of HFC, a slightly different nature of the residual stresses formation in the process of curing is observed. The residual stresses transition to the negative region for AST-T and CE-6 is 0.5 MPa and 0.6 MPa at convection heating, and 1.0 MPa and 1.9 MPa in the HFC field, respectively. For epoxy polymers, the residual stresses range from 5.5 MPa to 9.2 MPa. This indicates, that the segmental mobility of the acrylic polymers is limited as compared to the epoxy PCMs. This fact is confirmed by the dynamic mechanical characteristics, from which it can be seen, that the value of the modulus G’ in the glassy state is 1.11-1.61% higher, than that of epoxy compounds, cured by PEPA. The glass transition temperature difference is 25 °C.

The residual stresses of epoxyacrylic composition CE-6, cured by the convection method, are higher than those of epoxy compounds, and when cured in high-frequency current, the residual stresses decrease and make 2.2 MPa. Thus, the residual stresses magnitude depends on the rate of relaxation processes, which, in turn, is determined by the degree of curing and the polymer structure, the presence and nature of the plasticizer, and the conditions for the formation of a three-dimensional polymer lattice.

Subsequently, the following fillers were introduced into the investigated binders: aluminum oxide, molybdenum disulfide, and talc. In the filled PCMs, the value of the residual stresses depends on the size and concentration of the filler particles, on the differences in the physical properties of the polymer and filler, and on their interaction. The role of relaxation phenomena in the presence of the fillers, which affect the strength of PCM and often determine the time variations in their performance properties, should be especially emphasized. This is caused by the fact, that the residual stresses are formed in PCM due to the shrinkage of the polymer during its structuring, and due to the external impacts.

As Table 3 shows, the fillers have an ambiguous effect on the residual stresses in the epoxy compounds with PEPA, cured by the convection method. In PCM with aluminum oxide they decrease by 15%, while in PCM with talc, on the contrary, they grow to 23%. The fillers did not significantly affect the nature of the kinetics of the curing process. When the compounds were cured in HFC, the fillers had a more significant effect on the relaxation processes during crosslinking, as well as in the cured state. This especially becomes apparent, when aluminum oxide is used in the compounds, in which the residual stresses decreased by 45%. The width of the α-relaxation transition became 18% narrower, although the temperature of the gelation point remained unchanged. When the compound was cured in HFC, in contrast to the convection method, Talc also had a positive effect on the value of the residual stresses, although less noticeable than that of aluminum oxide. In general, the value of the residual stresses in the solid-state compounds, cured in the field of high-frequency current, is 50-60% lower, than that at the convection method of curing. The area of gelation shifted towards the temperatures lower by 20-25 °C.

In general, all the compounds, cured with PEPA in the presence of DBP, both with fillers and without them, when using the convection method of curing, have higher residual stresses, than the same compounds without a plasticizer. When considering the effect of the filler, it can be seen, that in PCMs the aluminum oxide has a significant positive effect on the nature of the formation and the magnitude of residual stresses; the residual stresses have decreased by 30%. Other fillers reduced the residual stresses, but less significantly, namely, molybdenum disulfide by 25%, and talc by 15%.

For compounds with MGP-9, as well as for those with DBP, the aluminum oxide had the maximum effect on the value of residual stresses. The residual stresses decreased by 11%, and Δσ for the compounds, cured by the convection method, made 9.8 MPa, and for those, cured in HFC, it was 8.4 MPa, what testified to a high degree of the compound homogenization. Talc had the effect on the residual stresses formation process itself. This process proceeds in a narrower temperature range, although the residual stresses slightly increased as compared to the process with the unfilled composition, but the character of the curve slope indicates, that in the process of further structuring their value decreased, what was confirmed by further investigations.

In the course of structuring in the HFC field, the residual stresses in compounds 1 and 3 are close in value, and the width of the gelation region is narrower for the compounds with fillers, what confirms positive contribution of the fillers.
in the process of structuring. After the gelation process completion, the filled compounds, cured with UP-0633 by the convection method, have residual stresses at approximately the same level as the compounds, cured with PEPA containing DBP and MGP-9. For all the compounds, structured in the field of high frequency current, the value of residual stresses is 40-50% lower. When the convection structuring method is used, the inhibitory effect of the fillers on the curing process and on the formation of a three-dimensional structure is observed. In HFC field this influence is ignored. It should be noted, that aluminum oxide behaves similarly both at the convection heating and when exposed to HFC field. Molybdenum disulfide and talc do not change their effect. All mentioned above is illustrated by diagrams (Figure 3-6).

Figure 3. The effect of the filler on the kinetics of residual stresses formation in the process of structuring

Figure 4. The effect of the filler on the kinetics of residual stresses formation in the process of structuring

Figure 5. The effect of the filler on the kinetics of residual stresses formation in the process of structuring
Figure 6. The effect of the filler on the kinetics of residual stresses formation in the process of structuring

For CE-6 compound, cured by the convection method and in HFC field, the best of the tested fillers is aluminum oxide, which reduces the residual stresses by 43% and 51%, respectively (Fig.6)

Thus, it is shown that HFC has a positive effect on the curing process and on the formation of residual stresses, both in unfilled and filled compounds. As a result, the performance properties of PCM, structured in the field of HFC, are characterized by a higher level and higher stability as compared to the properties of PCM, cured by the convection method [24-25].

CONCLUSIONS

1. A complex of researches on the effect of methods for structuring (polymerization) of epoxy, acrylic and epoxyacrylic PCMs on the value of residual stresses, occurring in PCM during structuring by two different methods, i.e. convection heating and structuring in the field of high-frequency current, was carried out.

2. It is shown, that structuring in the field of high-frequency current can significantly reduce the level of residual stresses, what has a positive effect on the strength properties of the material and the degree of curing.

3. It has been established, that the HFC electric field has the same effect on the properties of the compounds under study, irrespective of the polymer chemical nature, what is, apparently, due to the distribution of the electron density of epoxy and acrylic polymers.

4. The nature of the relaxation processes in PCM, which are structured in the field of HFC, has been studied. An increase in the dynamic modulus of elasticity and glass transition temperature for a number of PCMs, as compared to the PCMs structured by the convection method, has been determined. The increase in the dynamic modulus of elasticity and glass transition temperature is associated with an increase in the frequency of the spatial lattice and, as a consequence, an increase of the intermolecular interaction. In this case, the level of the residual stresses in PCM, structured in the HFC field, is lower as compared to the convection method, what allows to recommend this method for formation of sealing and adhesive joints, due to the achievement of a stable complex of strength, plasticity, and performance properties.

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Полімерним компаундом, в результаті чого в ПКМ з часом розвиваються більш або менш усадкові напруження. Крім процесі структурування. Вільній усадці ПКМ перешкоджає його адгезійний зв'язок з поверхнею виробу, що заливається Полімерний компаунд.

Ключові слова: які пов'язані з процесом склеювання та герметизації, як однорідних, так і різнорідних матеріалів, а також виробів з ПКМ.

Дослідження впливу способів структурування на зміну залишкових напруж

В полімерних композиційних матеріалах

Г.М. Черкашина, В.Л. Авраменко, О.Г. Карандашов

Процес формування когезійної міцності ПКМ пов’язаний з уменьшенням його об’єму. Якщо при цьому відсутнє механічне вплив на матеріал, то такий процес називають усадкою. Усадка має місце при охолодженні, при випаровуванні розчинника і в процесі структурування. Вільній усадці ПКМ перешкоджає його адгезійний зв’язок з поверхнею виробу, що заливається компаундом, в результаті чого в ПКМ з часом розвиваються більш або менш усадкові напруження. Крім останніх, в ПКМ мають місце термічні внутрішні напруж. Їх виникнення обумовлено поєднанням різних матеріалів в ПКМ, які значно відрізняються коефіцієнтами термічного розширення. Ефективним засобом для зниження внутрішніх напруж є додавання до складу ПКМ різних наповнювачів і пластичних, що покращує релаксаційні властивості ПКМ. Метою даної роботи було дослідження впливу різних методів структурування (полімеризації) – конвекційного і в полі струмів високої частоти епоксидних, акрилових та епоксіакрилових ПКМ. Дослідження виникаючих внутрішніх напруж, як усадок, так і температурних проводили методом цифрової тензометрії, яка дозволяє не тільки фіксувати кінцевий рівень

Ісследование влияния спосоbов структурирования на изменение остаточных напряжений в полимерных композиционных материалах

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Процесс формирования когезионной прочности ПКМ связан с уменьшением его объема. Если при этом отсутствует механическое воздействие на материал, такая процесс называют усадкой. Усадка имеет место при охлаждении, при испарении растворителя и в процессе структурирования. Свободной усадки ПКМ препятствует его адгезионная связь с поверхностью изделия, которая заливается полимерным компаундом, в результате чего в ПКМ со временем развивается более или менее усадочные напряжения. Кроме последних, в ПКМ имеется термические внутренние напряжения. Их
возникновение обусловлено сочетанием различных материалов в ПКМ, которые значительно отличаются коэффициентами
термического расширения. Эффективным средством для снижения внутренних напряжений является добавление в состав
ПКМ различных наполнителей и пластификаторов, что улучшает релаксационные свойства ПКМ. Целью данной работы
было исследование влияния различных методов структурирования (полимеризации) - конвекционного и в поле токов
высокой частоты эпоксидных, акриловых и эпоксиакриловых ПКМ. Исследование возникающих внутренних напряжений,
как усадочных, так и температурных проводили методом цифровой тензометрии, которая позволяет не только фиксировать
конечный уровень остаточных напряжений, но отслеживать его в процессе структурирования. Проведенными
исследованиями установлено, что более эффективным методом структурирования является процесс структурирования в
поле ТВЧ, который способствует снижению уровня остаточных напряжений, повышению модуля упругости и температуры
припояжа исследованных компаундов, что способствует повышению комплекса прочностных и эксплуатационных
свойств ПКМ, а также значительно сокращает время структурирования, обеспечивая равномерный нагрев по всему объему
ПКМ. Полученные данные рекомендуется использовать в различных отраслях, связанных с процессом склеивания и
герметизации, как однородных, так и разнородных материалов, а также изделий из ПКМ.

КЛЮЧЕВЫЕ СЛОВА: внутреннее напряжение, усадка, структурирование, поле токов высокой частоты, полимерный
композиционный материал