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RESEARCH ARTICLE

Oscillation rheometry of curing process of epoxy binders modified with polyetherimide

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Abstract

Objectives. The aim of this study is to ascertain the influence of polyetherimide on the curing process of epoxy binders.

Methods. The storage modulus and loss modulus of epoxyamine systems were measured as a function of curing time on the Anton Paar MCR 302 rheometer. The experiments were carried out at an oscillation frequency of 1 Hz, with an amplitude aligned with the linear viscoelasticity region, and across a range of temperatures (160, 170, and 180°C). The crossover point was determined when the components of the complex modulus of elasticity are equal according to the obtained dependencies.

Results. The influence of polyetherimide on the curing process of epoxyamine binders was investigated at a thermoplastic content of 5 to 20 pts. wt at three temperatures. In a system modified with 20 pts. wt of polyetherimide, phase separation was observed during the curing process. In systems modified with 10 and 20 pts. wt of polyetherimide, the limiting value of the modulus of elasticity was observed to be higher at 170°C than at 180°C.

Conclusions. The modification of epoxyamine binders with thermoplastic in an amount of 5–20 pts. wt has been observed to extend the time required to reach the crossover point. Furthermore, the curing process markedly slows down in the system comprising 10 pts. wt of thermoplastic content, in which it takes the longest time to reach the crossover point at all three experimental temperatures.

Keywords

oscillation rheometry, crossover point, epoxy oligomer, polyetherimide, phase separation

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НАУЧНАЯ СТАТЬЯ

Осцилляционная реометрия процесса отверждения эпоксидных связующих, модифицированных полиэфиримидом

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Аннотация

Цели. Определение влияния полиэфиримида на процесс отверждения эпоксидных связующих.

Методы. Методом осцилляционной реометрии фиксировали зависимость модуля накопления и модуля потерь эпоксиаминных систем от времени отверждения на приборе MCR 302 фирмы Anton Paar с частотой колебаний 1 Гц и амплитудой, соответствующей области линейной вязкоупругости, при трех температурах 160, 170 и 180°C. По полученным зависимостям определяли точку кроссовера при равенстве составляющих комплексного модуля упругости.

Результаты. Установлено влияние полиэфиримида на процесс отверждения эпоксиаминных связующих при содержании термопласта от 5 до 20 мас. ч. на 100 мас. ч. эпоксидного олигомера при трех температурах. Для системы, модифицированной 20 мас. ч. полиэфиримида, зафиксировано фазовое разделение в процессе сшивания. В системах, содержащих 10 и 20 мас. ч. полиэфиримида, предельное значение модуля упругости оказывается выше при 170°C, чем при 180°C.

Выводы. Введение полиэфиримида в эпоксиаминные связующие в количестве 5–20 мас. ч. увеличивает время достижения точки кроссовера. При этом наиболее сильно замедляется процесс отверждения для системы, содержащей 10 мас. ч. термопласта, время достижения точки кроссовера которой оказывается наибольшим при всех трех температурах эксперимента.

Ключевые слова

осцилляционная реометрия, точка кроссовера, эпоксидный олигомер, полиэфиримид, разделение фаз

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INTRODUCTION

Epoxy resins are used as binders in reinforced plastics in many industries. The advantages of this class of compounds include good adhesion, high dielectric properties, low curing shrinkage, and excellent chemical resistance [1]. The use of hot curing hardeners enables the attainment of relatively high heat resistance in epoxy polymers. However, deficiencies exhibited in such materials in terms of crack resistance and impact strength limit the range of their potential application.

The resistance of epoxy polymers to brittle fracture can be improved by modifying them with thermoplastics [2–4]. In comparison to the utilization of rubbers and active diluents, this approach offers the distinct advantage of maintaining the glass transition temperature of the cured epoxy oligomer at its original level [5, 6].

In the majority of cases, the initially compatible system of epoxy oligomer and thermoplastic undergoes phase decomposition during the curing process. This is due to the increase in molecular weight of the cured polymer, which in turn contributes to the enhancement of the physical and mechanical properties of the material [7]. Different types of structures are formed in the cured polymers [8]. In [9], examples of extended structures with direct and inverse “matrix–dispersion”, in which the dispersion medium is reactoplastic and thermoplastic, respectively, are demonstrated.

The incorporation of thermoplastics into epoxy oligomers markedly alters their intrinsic characteristics, including viscosity. Consequently, it is of considerable scientific interest to examine the curing process of systems modified in this way. Rotation or oscillation rheometry can be used to measure viscosity and determine gelation time [10]. During the latter, the dependencies of two components of the complex modulus (|G*|): the storage modulus G′ and the loss modulus G″ on time are recorded. The moment of intersection of the values of these parameters, referred to as the crossover point, is associated in the literature with gel formation in the system [11]. In epoxy binders modified with thermoplastics, the release of the phase enriched in the more viscous component occurs prior to gelation. Consequently, through the utilization of rotation or oscillation rheometry, it is occasionally feasible to ascertain the precise moment of phase separation. This is evidenced by the emergence of distinctive discontinuities in the relationships between shear viscosity or loss modulus and curing time [12].

Previously, the curing process of epoxy oligomer modified with cardo polysulfone copolymer PSFF-70K was investigated by rotational rheometry in constant shear mode [13]. For the system containing 10 pts. wt of thermoplastic content, there is a slight decrease in shear viscosity at the 30th minute at an experimental temperature of 180°C, indicating phase separation during the structuring process. Interestingly, for the binder modified with 5 pts. wt PSFF-70K the longest gelation time was recorded compared to the unmodified binder, which can probably be attributed to the fact that at this content of modifier there is no phase separation of the mixture.

The modification of epoxy oligomer with polyetherimide (PEI) was also studied in [14]. On the infrared spectra of the cured epoxyamine polymer containing 2 wt % of PEI, the authors observed a shift towards lower wave numbers of the peak characteristic of the hydroxyl group, which is also contained in the initial epoxy A type oligomer and is formed after the reaction of the epoxide cycle with the amino group. This can probably be explained by the occurrence of hydrogen bonds between the hydroxyl groups of the epoxy polymer and the imide groups of PEI.

The objective of this work is to study the curing process of PEI-modified epoxyamine binders.

MATERIALS AND METHODS

The binders were prepared on the basis of epoxy oligomer ED-20 (GOST 10587-84[1], *Ya.M. Sverdlov Plant*, Russia). Ultem 1010 grade PEI (*Sabic*, USA) was used as a thermoplastic modifier, which was added to ED-20 at 160°C in an amount of 5, 10, 15, and 20 pts. wt per 100 pts. wt of epoxy oligomer and stirred using a top-drive stirrer until homogeneous solutions were obtained at 160–180°C. 4,4’-diaminodiphenylsulfone was used as a hardener for the binders; this is not indicated in the captions. The hardener was mixed for 30 min at 120°C in an amount of 30 pts. wt per 100 pts. wt of ED-20, which is close to the stoichiometric ratio.

Oscillation rheometry was performed in a plane-to-plane measuring system with a working gap of 1 mm on an MCR 302 rheometer manufactured by *Anton Paar* (Austria) at an oscillation frequency ω = 1 Hz and an amplitude corresponding to the linear viscoelasticity region. The experiments were conducted at three distinct temperatures: 160, 170, and 180°C. Prior to the experiment, the binders were preheated to 130°C in the thermal cabinet, then transferred to the lower fixed plane of the instrument. Next, the gap was set up and the binders were heated to the measurement temperature. During the experiments, the dependencies of the accumulation modulus G′ and loss modulus G″ on the curing time were recorded.

RESULTS AND DISCUSSION

Earlier studies have shown that the epoxy-PEI system is compatible prior to the curing process and characterized by an upper critical solution temperature of 40°C. The initial viscosity of PEI-modified epoxies was also found to be between 2 and 6 kPa·s at room temperature (20°C) [15].

Figure 1 shows the dependencies of the storage modulus and loss modulus on the curing time of epoxyamine binders in semi-logarithmic coordinates at three temperatures.

At the initial stage of the curing process, the loss modulus G″ appears to be higher than the storage modulus G′, which is due to the inability of liquids to store energy under mechanical loading. However, during the curing process, the components of the complex modulus of elasticity increase. At a certain moment at which G′ turns out to be higher than G″, the system under study acquires the characteristics of a solid state. This point in time is called the crossover point (G′ = G″) and is associated with gel formation. The crossover point corresponds to the moment of appearance of a continuous grid of chemical bonds that are formed during the curing process. The time to reach the crossover point is about 18–33 min longer in epoxy binders with thermoplastic modifiers as compared to unmodified ED-20. This is probably due to the slowing down of the curing process, i.e., slowing down of the formation of the chemical bonding network in the epoxyamine binder in the presence of PEI, since the thermoplastic dilutes the reactive system and increases its viscosity as correlated with the literature

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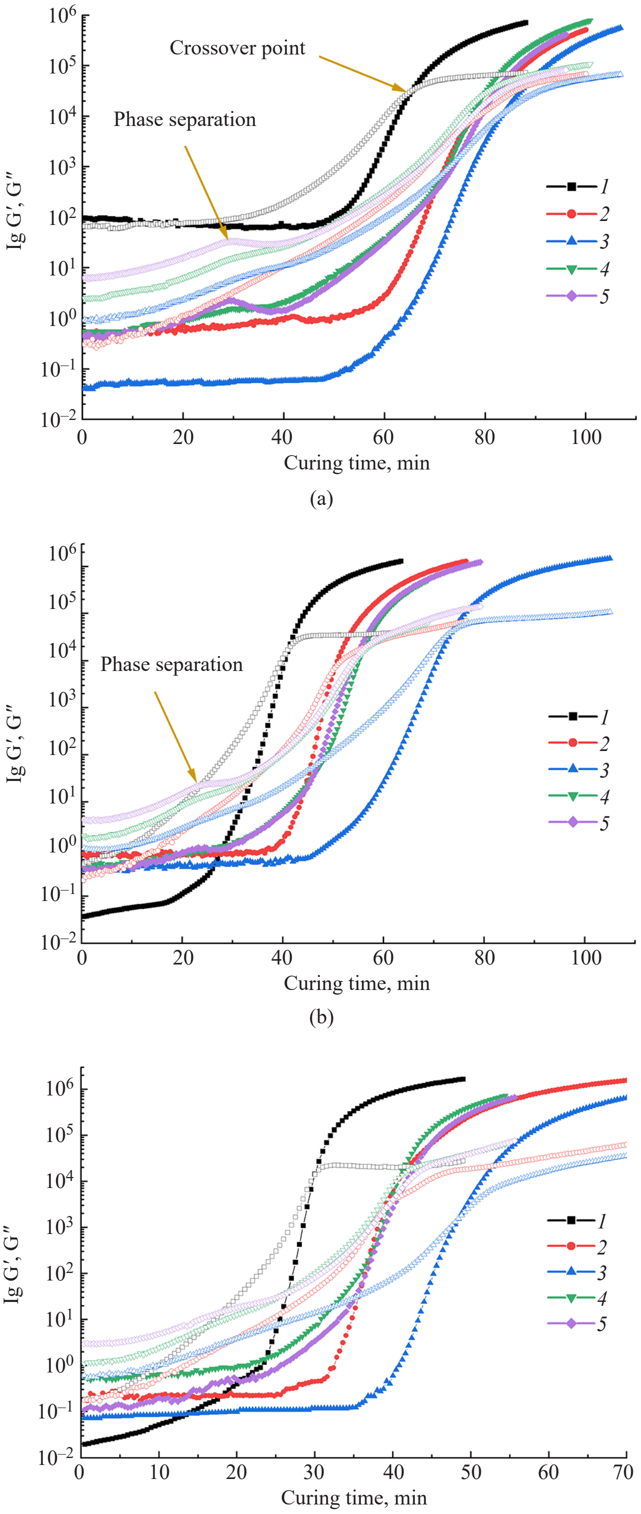
[1] GOST 10587-84. State Standard of the USSR. Uncured epoxy resins. Specifications. Moscow: USSR State Committee for Product Quality Management and Standards; 1985.



(a)

(b)

(c)

Fig. 1. Dependence of storage modulus G′ (solid symbols) and loss modulus G″ (hollow symbols) on curing time:
(*1*) ED-20,
(*2*) ED-20+PEI (5 pts. wt),
(*3*) ED-20+PEI (10 pts. wt),
(*4*) ED-20+PEI (15 pts. wt),
(*5*) ED-20+PEI (20 pts. wt),
at 160 (a),
170 (b), and
180°C (c)

data [15]. The table below summarizes the crossover point reach times calculated from Fig. 1.

Table. Times of reaching the crossover points of the studied binders

Sample	Time of the crossover point, min		
	160°C	170°C	180°C
ED-20	66	41	30
ED-20+PEI (5 pts. wt)	77	50	39
ED-20+PEI (10 pts. wt)	84	74	48
ED-20+PEI (15 pts. wt)	80	56	41
ED-20+PEI (20 pts. wt)	81	55	41

From Figs. 1a and 1b, it can be seen that in the system containing 20 pts. wt of PEI (curve *5*) at 160°C at the 29th min (Fig. 1a) and at 170°C at the 23rd min of the experiment (Fig. 1b), there is a decrease in the loss modulus value, which is related to the dynamic viscosity by the following relationship:

$$\eta' = \frac{G''}{\omega},$$

where η' is dynamic viscosity, Pa·s; G'' is loss modulus, Pa; ω is frequency, rad/s.

This means that the dependence of the loss modulus has qualitatively the same character as the dependence of the dynamic viscosity under the condition of constant frequency during the experiment. Consequently, it can be assumed that the decrease in the loss modulus value is due to the release of the viscous component, PEI, from the reaction system into a separate phase. At this point, the curing process ceases to be homogeneous and begins to be heterogeneous. Then, due to the ongoing curing process, the loss modulus of the system increases again.

In the technology of reinforced plastics, the concept of binder pot-life parameter is taken as the time to reach a certain viscosity level, above which the binder is no longer able to impregnate the reinforcing filler. This parameter is often taken as ≈100 Pa·s [1, 11]. From Fig. 2, it can be concluded that a system containing 10 pts. wt of PEI (curve *3*) has an increased (about 15–27 min) pot-life compared to other binders studied in this work. As the "process window" for processing the material into composite products increases, this represents a clear advantage of the thermoplastic-modified binder in comparison withneat ED-20. Since epoxy binders are Newtonian fluids at elevated temperatures [15, 16], the complex viscosity can be fairly taken as the effective viscosity based on the Cox–Merz rule.

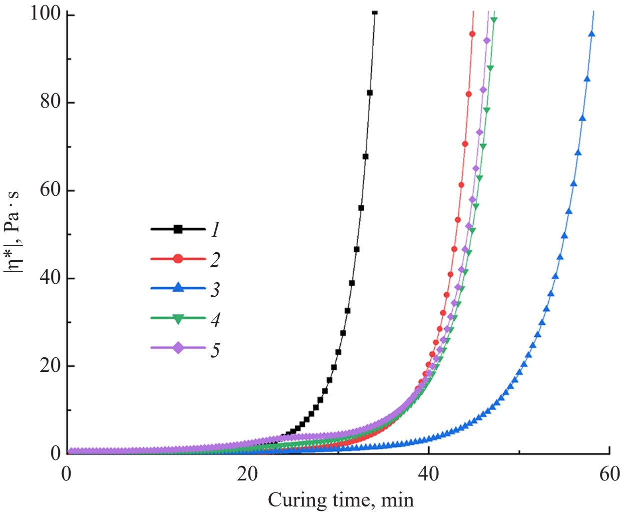


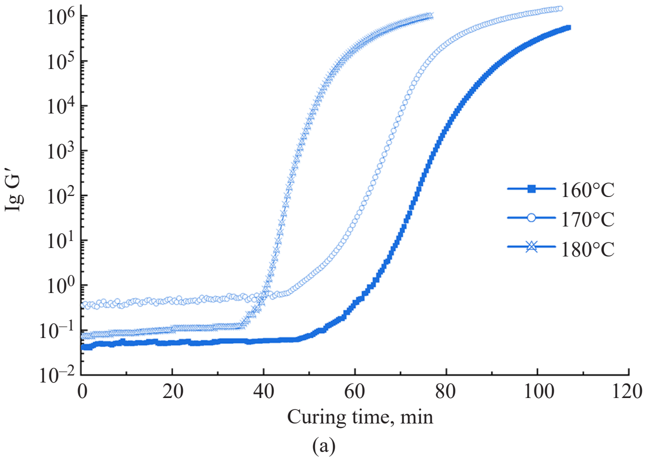
Fig. 2. Dependence of complex viscosity (|η*|) on curing time at 170°C:
(*1*) ED-20,
(*2*) ED-20+PEI (5 pts. wt),
(*3*) ED-20+PEI (10 pts. wt),
(*4*) ED-20+PEI (15 pts. wt),
(*5*) ED-20+PEI (20 pts. wt)

Since higher temperatures lead to more intensive curing processes, it would be expected that the limiting value of the storage modulus would be higher at 180°C. In practice, however, the limiting value of elastic modulus is found to be higher at 170°C, as shown in Fig. 3.

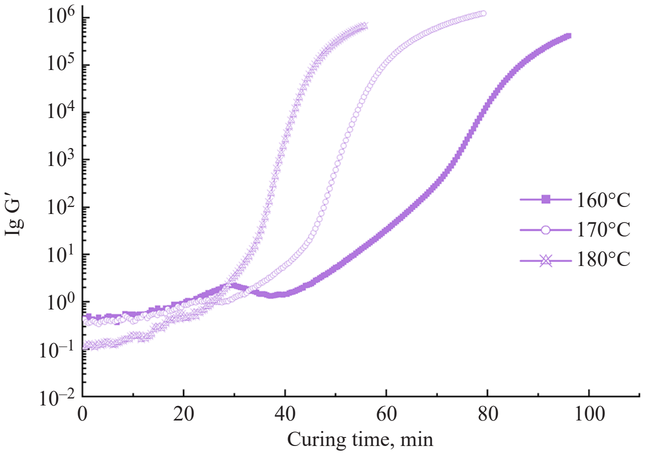
The point to bear in mind here is that the modulus of elasticity is affected by the temperature of the experiment. Since the modulus of elasticity is known to decrease with increasing temperature, so at 180°C, the decrease in the ultimate value of the modulus of elasticity due to the higher temperature prevails over its increase due to curing.

CONCLUSIONS

It has been shown that the addition of a thermoplastic modifier to an epoxy binder increases the gelation time, which is due both to the slowing down of the curing process in the presence of the thermoplastic, which dilutes the reaction system, as well as to the increased viscosity of the modified binder itself compared to the unmodified binder. The highest pot-life was recorded for the system modified with 10 pts. wt of PEI. The higher ultimate value of elastic modulus at a curing temperature of 170°C than that at 180°C is due to the dual effect of temperature.



(a)



(b)

Fig. 3. Dependence of storage modulus (G′) on curing time at three temperatures: (a) ED-20+PEI (10 pts. wt), (b) ED-20+PEI (20 pts. wt)

Authors' contributions

S.V. Polunin—conducting experiments, processing the data obtained, discussing the results obtained, and participating in editing the text of the article.

K.A. Atamas—processing the obtained data, literature review on the topic of the article, writing and formatting the text of the article.

I.Yu. Gorbunova—research idea, planning experiments, analyzing and discussing the results, and editing the text of the article.

P.A. Morozova—conducting experiments and discussing the results obtained.

K.M. Marakhovsky—planning experiments, discussing the results obtained, and participating in editing the text of the article.

The authors declare no conflicts of interest.

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