

ВЛИЯНИЕ КОНЦЕНТРАЦИИ АЛЮМИНИЕВОЙ ПУДРЫ НА МЕХАНИЗМ И КИНЕТИЧЕСКИЕ ЗАКОНОМЕРНОСТИ КРИСТАЛЛИЗАЦИИ КОМПОЗИТОВ НА ОСНОВЕ ПОЛИЭТИЛЕНА НИЗКОЙ ПЛОТНОСТИ

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Приводятся результаты исследования влияния концентрации алюминиевой пудры на механизм и закономерность изменения зависимости удельного объема от температуры в интервале 25-180 °С в композитах на основе полиэтилена низкой плотности. Путем экстраполяции верхней и нижней ветви дилатометрической кривой найдены приблизительные значения температуры стеклования композитов. Экстраполяция нижней ветви дилатометрической кривой рассматриваемых полимерных композитов к абсолютной температуре позволили определить величину «занятого» удельного объема и «свободного» удельного объема. Методом ступенчатой дилатометрии найдена температура фазового перехода первого рода композитов, которая изменяется в зависимости от концентрации алюминиевой пудры в составе полиэтилена низкой плотности. Концентрацию алюминиевой пудры с размером частиц 1-2 мкм варьировали в пределах 0,5 – 30 %масс. Установлено, что введение 0,5%масс. алюминиевой пудры практически не влияет на закономерность изменения дилатометрической кривой исходной полимерной матрицы. При концентрации наполнителя 1,0%масс. и выше происходит существенное изменение в закономерности изменения этих кривых, выражающееся в снижении величины удельного объема композитов. Резкое снижение удельного объема или возрастание плотности композитов в вязкотекучем и твердом состояниях однозначно свидетельствовали об усиливающей роли алюминиевой пудры. В композитах на основе полиэтилена низкой плотности определена зависимость свободного удельного объема от температуры и от концентрации алюминиевой пудры. Показано, что увеличение концентрации алюминиевой пудры в составе полиэтилена низкой плотности сопровождается закономерным снижением свободного объема полимерной матрицы. Полученные данные подтверждают наше представление о том, что частицы алюминиевой пудры участвуют не только в формировании гетерогенных центров кристаллизации, но и вытесняются в аморфную область по мере роста сферолитных кристаллических образований. В координатах Авраами исследованы кинетические закономерности процесса изотермической кристаллизации в области фазового перехода первого рода. Установлено, что с увеличением степени наполнения полимерной матрицы механизм кристаллизации из сферического (трехмерного) типа роста кристаллических образований переходит в пластинчатый (двухмерный) при непрерывном образовании центров кристаллизации. Даны рекомендации по практическому использованию результатов исследования процесса кристаллизации композитов применительно в технологии их переработки методом литья под давлением.

Ключевые слова: кристаллизация, дилатометрия, удельный объем, фазовый переход, алюминиевая пудра, полимерная матрица

Для цитирования:

Аллахвердиева Х.В., Кахраманов Н.Т., Абдуллин М.И., Мустафаева Ф.А. Влияние концентрации алюминиевой пудры на механизм и кинетические закономерности кристаллизации композитов на основе полиэтилена низкой плотности. *Изв. вузов. Химия и хим. технология.* 2020. Т. 63. Вып. 2. С. 77–83

For citation:

Allakhverdieva Kh.V., Kakhramanov N.T., Abdullin M.I., Mustafayeva F.A. Influence of aluminum powder concentration on mechanism and kinetic regularities of crystallization of composites based on low density polyethylene. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* [Russ. J. Chem. & Chem. Tech.]. 2020. V. 63. N 2. P. 77–83

INFLUENCE OF ALUMINUM POWDER CONCENTRATION ON MECHANISM AND KINETIC REGULARITIES OF CRYSTALLIZATION OF COMPOSITES BASED ON LOW DENSITY POLYETHYLENE

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The results of the study of the influence of the aluminum powder concentration on the mechanism and the regularity of changes in the dependence of specific volume on temperature in the range of 25-180 °C in composites based on low density polyethylene are presented. By extrapolating the upper and lower branches of the dilatometric curve, approximate values of the glass transition temperature of the composites are found. Extrapolation of the lower branch of the dilatometric curve of the considered polymer composites to the absolute temperature allowed us to determine the value of the "occupied" specific volume and the "free" specific volume. Using the step dilatometry method, the temperature of the first-order phase transition of composites was found, which varies depending on the aluminum powder concentration in the composition of low density polyethylene. The concentration of aluminum powder with a particle size 1-2 μm varied between 0.5 – 30 wt. %. It has been established that the loading of 0.5 wt. % of aluminum powder practically does not affect the regularity of change in the dilatometric curve of the initial polymer matrix. At a filler concentration of 1.0 wt. % and higher, a significant change occurs in the regularities of change in these curves, expressed in a decrease in the value of the specific volume of the composites. A sharp decrease in the specific volume or an increase in the density of the composites in viscous-flow and solid states clearly indicated the reinforcing role of aluminum powder. In composites based on low density polyethylene, the dependence of the free specific volume on temperature and on the concentration of aluminum powder is determined. It is shown that an increase in the concentration of aluminum powder in the composition of low density polyethylene is accompanied by a regular decrease in the free volume of the polymer matrix. The obtained data confirms our idea that particles of aluminum powder are involved not only in the formation of heterogeneous crystallization centers, but are also forced into the amorphous region as the spherulite crystalline formations grow. In the coordinates of Avraami, the kinetic regularities of the process of isothermal crystallization in the region of the first-order phase transition are investigated. It has been established that with an increase in the degree of filling of the polymer matrix, the mechanism of crystallization from spherical (three-dimensional) type of growth of crystalline formations passes into a plate-like (two-dimensional) with the continuous formation of crystallization centers. Recommendations are given on the practical use of the results of a study of the crystallization process of composites as applied to the technology of their processing by injection molding.

Key words: crystallization, dilatometry, specific volume, phase transition, aluminum powder, polymer matrix

INTRODUCTION

As technique and technology improved in various areas of industry, more and more new requirements began to be placed on the quality of polymeric

materials designed to produce construction products on their basis. In particular, this applies to aviation, cosmic, military, shipbuilding, engineering, etc. industries. In order to improve the complex properties of polymers, various methods of modifying their structure

and properties are undertaken. The most preferred method of modifying the polymer matrix is loading into its composition various finely dispersed mineral and metal fillers, which in one way or another allow obtaining composite materials with predetermined operational properties [1-7].

In recent years, interest in the development and research of metal-polymer systems has been expanded, making it possible to obtain materials with properties that are promising from a practical point of view [8-10]. But at the same time, despite the above, to this day there are still open questions regarding the technological characteristics of their processing by methods of injection molding and extrusion. At the same time, the quality of plastic products directly depends on the rate of their cooling (crystallization) in the mold of an injection molding machine or a forming die extruder.

In this regard, comprehensive information on the crystallization process of the composites polymer matrix can be obtained using the method of step dilatometry. This method allows to investigate the effect of various ingredients on the kinetic regularity and mechanism of crystallization in isothermal conditions. For a long time, the dilatometry method was considered as the theoretical basis of the processing technology. However, the results of experimental studies suggest the practical significance of research conducted in this direction [11,12].

In this regard, the purpose of the study was to study the effect of filler concentration on the kinetic regularities and the crystallization mechanism of polymer composites.

EXPERIMENTAL PART

As the object of the study the low density polyethylene (LDPE) was used with the following properties: ultimate tensile stress – 13.1 MPa, flexural modulus – 196 MPa, density – 927 kg/m³, elongation at break – 720%, heat resistance– 85 °C, melting point – 106 °C, melt flow index MFI = 1.3 g/10min, degree of crystallinity 60%.

In order to modify the properties of the LDPE, aluminum powder (AP) with a size of 1.0-2.0 μm was loading into its composition. The amount of AP in the composition of the LDPE varied within 0.5, 1.0, 5.0, 10, 20, 30 wt. %. The components were mixed on hot rollers at a temperature of 150-160 °C, by loading the AP into the LDPE melt for 7-8 min.

Dilatometric studies were performed on an IIRT-1 device converted to a dilatometer with a load of 5.3 kg and in the temperature range from 180 °C to room temperature.

RESULTS AND DISCUSSION

The advantage of the dilatometry method is that it allows one to estimate the influence of the filler concentration on the nature of the change in the crystallization process, the first-order phase transition temperature, and the crystallization rate. The construction of a dilatometric curve of the dependence of the specific volume on temperature makes it possible to estimate the volume shrinkage processes not only in the viscous-flow, but also in the solid state [11].

Fig. 1 shows the dilatometric curves of the dependence of the specific volume on temperature, depending on the AP concentration. It was important to identify the effect of minimum and high concentrations of AP on the nature of changes in these curves. Analyzing the dilatometric curves in this figure it can be established that the loading of 0.5 wt. % AP almost does not affect the regularity of changes in the dilatometric curve of the initial LDPE. A further increase in concentration from 1.0 to 30 wt. % is accompanied by a slight decrease in the value of the specific volume of samples. It is characteristic that, regardless of the AP content, the nucleation process on the dilatometric curve or the first-order phase transition occurs in the temperature range of 80-90 °C. This temperature characterizes the transition of a polymer from a viscous-flow to a solid state. It is this process that takes place in the cooling mold of the injection unit.

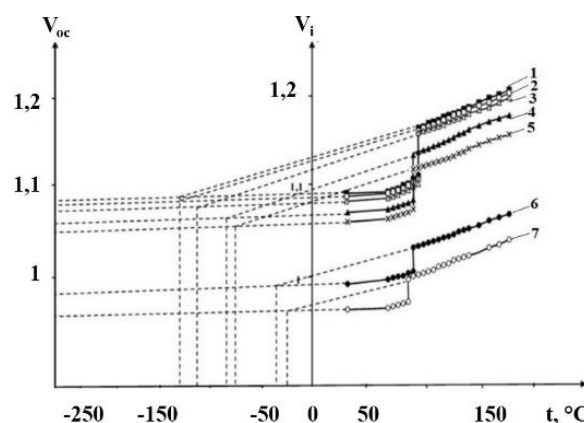


Fig. 1. The effect of AP concentration on the regularity of specific volume change on temperature for composites based on LDPE + AP, wt. %: 1 - LDPE, 2 - 0.5; 3 - 1.0; 4 - 5.0; 5 - 10; 6 - 20; 7 - 30
Рис. 1. Влияние концентрации АП на закономерность изменения удельного объема от температуры для композитов на основе ПЭНП+АП, в %масс.: 1 - исх.ПЭНП, 2 - 0,5; 3 - 1,0; 4 - 5,0; 5 - 10; 6 - 20; 7 - 30

It is clear that if the density of the composites is the reciprocal of the specific volume, then a decrease in the specific volume should be considered as an increase in the density of the composites. And, the

greater the concentration of the AP, the higher the density of the composites. The data obtained clearly indicate that the loading of AP is accompanied by compaction of the polymer volume without loosening it. In this case, it would be appropriate to state that the AP, as a filler, is an amplifier of LDPE-based composites [13].

According to the data presented in Fig. 1, for the initial LDPE and its composites with an AP content of 0.5–1.0 wt. %, the dilatometric curves have a first-order phase transition at a temperature of 90 °C. A further increase in the concentration of AP from 5.0 to 20 wt. % is accompanied by a decrease in the temperature of the phase transition to 85 °C. When the degree of filling 30 wt. % first-order phase transition is reduced to 82 °C. To interpret the observed regularities, let us turn to the well-known theoretical aspects of the formation of the supramolecular structure of filled composites. Most of the foreign solid particles loading into the composition of polymers are distinguished by the fact that the melt exhibits the characteristic properties of nucleating agents [14–17]. This is expressed in the fact that these dispersed particles are capable to form heterogeneous crystallization centers. As a result, in melts in addition to homogeneous, there are heterogeneous crystallization centers that form crystalline formations during the melt cooling process. Therefore, in the process of crystallization in the region of the first-order phase transition, spontaneous formation of small-spherulite crystalline structures occurs at a time, which in the process of growth push a part of the filler into the interspherulite or amorphous region. The latter circumstance allows us to state that a part of AP particles takes part in the formation of heterogeneous crystallization centers, while another part is displaced into the amorphous region. According to the experimental data obtained, it seems to be correct to say that the filler particles can be spent only on the formation of heterogeneous nucleation centers with their minimum concentration in the composition of the LDPE, within 0.5–1.0 wt. %.

From Fig. 1, extrapolation of the upper and lower branches leads to their intersection. Perpendicular, lowered on the abscissa axis from the point of their intersection, allows to fix the temperature, which with the maximum approximation can be considered as the glass transition temperature of polymeric materials. According to the data obtained, the glass transition temperature of the considered composites is respectively: LDPE and LDPE + 0.5 wt. % AP = -125 °C, LDPE + 1.0 wt. % AP = -117 °C, LDPE + 5.0 wt. %

AP = -88 °C, LDPE + 10 wt. % AP = -81 °C, LDPE + 20 wt. % AP = -39 °C, LDPE + 30 wt. % AP = -25 °C. The data obtained show that with an increase in the concentration of AP in the composition of the composite over 5.0 wt. % embrittlement of the composite occurs, which, as a rule, leads to a sharp decrease in the value of their glass transition temperature. And indeed, when assessing the deformation-strength characteristics of composites, it was found that with an AP concentration of 10 wt. % and the higher the elongation at break deteriorates sharply to 20–30%.

Another important point to note is the definition of the occupied specific volume (V_{oc}), which it is possible to estimate by extrapolating the lower branch of the dilatometric curve to absolute zero (-273 °C). At an absolute zero temperature, the free specific volume (V_f) in the polymer becomes zero. Since the specific volume (V_i) consists of the sum of the occupied and free specific volumes, then $V_f = V_i - V_o$ [18–20].

Fig. 2 shows the temperature dependence of V_f for composites with different AP contents. Analyzing the dilatometric curves in this figure, it can be established that with increasing AP concentration V_f in composites it decreases markedly. The results obtained clearly indicate that the AP particles are embedded in the free volume, which is contained mainly in the amorphous region. If we take a more concrete approach to the interpretation of this process, then we can see that when 0.5 wt. % AP is added to LDPE, the value of free specific volume practically does not undergo any changes, thus confirming our arguments about the role of the concentration factor of composite organization. There is reason to believe that when the concentration of AP in the range of 0.5 wt. % all filler particles are spent on the formation of heterogeneous nucleation centers with their subsequent transition to heterogeneous crystallization centers. At AP concentrations above 0.5 wt. %, the decrease in free specific volume should be interpreted as the participation of AP particles simultaneously in the process of nucleation and migration to the amorphous regions in the process of growth of crystalline formations. And, the greater the concentration of the filler, the greater part of it in the process of growth of crystalline formations forcibly migrates to the amorphous regions. Thus, when discussing the role of the concentration factor of the filler, it will probably be appropriate to talk about the threshold concentration that determines the nucleation abilities of the filler particles.

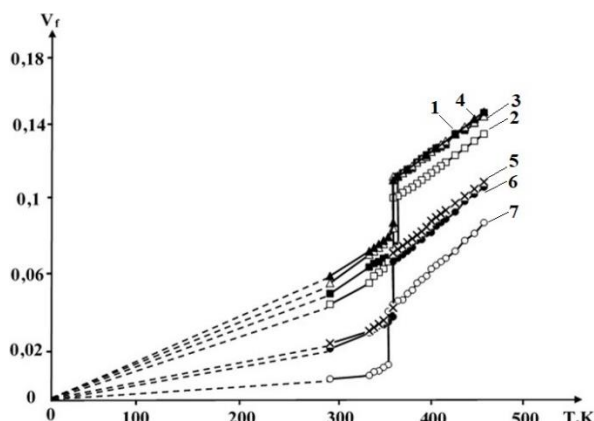


Fig. 2. The effect of AP concentration on the dependence of the free specific volume on the temperature (K) for composites based on LDPE, wt. %: 1 - LDPE, 2 - 0.5; 3 - 1.0; 4 - 5.0; 5 - 10; 6 - 20; 7 - 30

Рис. 2. Влияние концентрации АП на зависимость свободного удельного объема от температуры (K) для композитов на основе ПЭНП, в % масс.: 1 - ПЭНП, 2 - 0,5; 3 - 1,0; 4 - 5,0; 5 - 10; 6 - 20; 7 - 30

The results of kinetic measurements of the crystallization process are interpreted using a model developed by Avraami for phase transformations in metals [10]. Previously, we have shown the applicability of the Avrami theory to the study of the crystallization process in the region of the first-order phase transition in polymer composites [11]. According to this theory, the crystallization process proceeds in accordance with the expression:

$$\varphi = e^{-K\tau^n}$$

where φ – is the part of the polymer that has not yet undergone transformation into the crystalline phase; K – generalized nucleation and crystal growth constant; n – constant ranges from 1-4. Its value depends on the nature of the nucleation process and growth. [11, 18-20].

If V_0 – is the initial specific volume of the polymer at T_{cr} , V_τ – is the specific volume by the τ time, V_∞ – is the final specific volume of the polymer at a given crystallization temperature in the phase transition region, then the ratio $(V_0 - V_\tau)/(V_0 - V_\infty)$ there is a part of the polymer that has undergone transformation into a crystalline state by the τ time. In this case, φ in equation (1) is:

$$\varphi = 1 - (V_0 - V_\tau)/(V_0 - V_\infty) \quad (2)$$

Double logarithm of the Avraami equation gives:

$$\lg(-\ln\varphi) = \lg K + n \lg \tau \quad (3)$$

In accordance with the above equation, this dependence is a straight line in the coordinates $\lg(-\ln\varphi)$ from $\lg \tau$. As a result of experimental measurements, the applicability of the theory of Avraami to the study of the crystallization process of LDPE and its composites was established.

In fig. 3 the coordinates of Avraami presents the results of studying the effect of AP concentration on the kinetic regularity of crystallization of composites based on LDPE + AP. Analyzing the curves in this figure, you can see that for the initial LDPE and the composite with 1.0 wt. % AP, they coincide. With the loading of 0.5 wt. % AP, the crystallization rate slightly increases, which can be well interpreted by the slope of the curves and the results of the evaluation of the crystallization rate constants. Thus, for example, according to equation (3), at a zero crystallization time ($\lg \tau$), $\lg(-\ln\varphi)$ becomes equal to $\lg K$. For the initial LDPE and the sample with 1.0 wt.% AP, the generalized crystallization rate constant (K) respectively is $1.2 \cdot 10^{-5}$ and $0.6 \cdot 10^{-5}$. At 0.5 wt. % content of the AP $K = 4.1 \cdot 10^{-5}$, i.e. has the highest value of the crystallization rate constant, even higher than that of the initial LDPE. Increasing the concentration of AP in the range of 5, 10, 20, 30 wt. % is accompanied by a decrease in K , respectively, in the following sequence – $0.39 \cdot 10^{-5}$, $0.31 \cdot 10^{-5}$, $0.19 \cdot 10^{-5}$, $0.08 \cdot 10^{-5}$. From the obtained results of the analysis it can be established that with an increase in the concentration of AP from 0.5 to 30 wt. % a regular decrease in K is observed, approximately, by 50 times, and in relation to the initial LDPE – by 15 times.

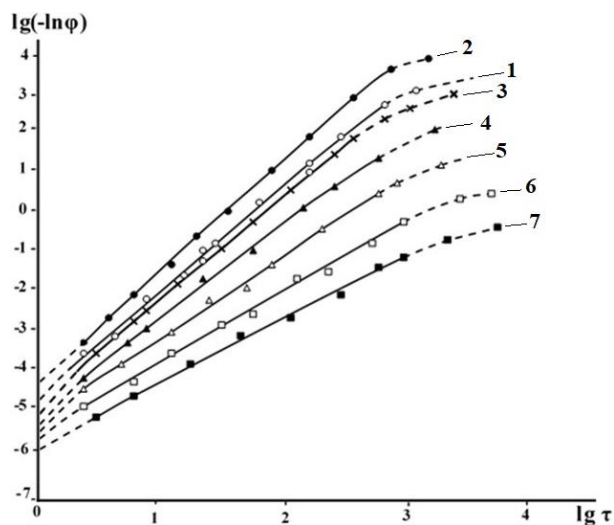


Fig. 3. The effect of AP concentration on the kinetic regularity and the mechanism of crystallization of composites based on LDPE + AP in the area of the first-order phase transition, in wt. %: 1 - initial LDPE; 2 - 0.5; 3 - 1.0; 4 - 5.0; 5 - 10; 6 - 20; 7 - 30

Рис. 3. Влияние концентрации АП на кинетическую закономерность и механизм кристаллизации композитов на основе ПЭНП+АП в области фазового перехода первого рода, в % масс.: 1 - исходный ПЭНП; 2 - 0,5; 3 - 1,0; 4 - 5,0; 5 - 10; 6 - 20; 7 - 30

To evaluate the growth mechanism and the formation of crystals, we found the value of n , which, as it turned out, depends on the degree of filling. So, for

example, for the initial LDPE and composites with the content of AP in the range of 0.5-10 wt. %, the value of n is approximately 3, which corresponds to the spherical (three-dimensional) type of growth of crystalline structures with the continuous formation of crystallization centers. With a degree of filling of 20-30 wt. % of AP, the value of n becomes equal to about 2, which characterizes the plate-like (two-dimensional) type of crystal growth with the continuous formation of crystallization centers. In this case, the term "continuous formation of crystallization centers" indicates that the rate of formation of these centers remains constant throughout the study of the nucleation process in the region of the first-order phase transition.

It should be noted that the method of dilatometric measurements allows to obtain quite useful information about the nature of the formation of crystal structures in the process of cooling the melt of a polymeric material. At the same time, the process of compact packaging of ordered structural formations is accompanied by a regular decrease in the specific volume [18-20]. This is due to the fact that the processing of polymeric materials at the final stage is characterized by cooling the melt and holding time under pressure in the press mold, as a result of which it is possible to form structural products of various configurations. And depending on how properly the mode of cooling

the molten composite in the press mold of the injection molding machine is observed, the molded plastic product becomes so high-quality. In particular, as a result of studying the kinetic regularities of composites crystallization, it was found that the rate of their crystallization from the melt slows down with the loading of a large amount of filler. The latter circumstance suggests that for relatively highly filled composites it is necessary to increase the time of their cooling in the mold.

CONCLUSIONS

Thus, based on the above, it can be stated that the method of dilatometry of polymer samples during stepwise cooling allows us to investigate not only the regularity of changes in specific volume from temperature, but also to fix the first-order phase transition, determine the temperature dependence of the occupied and free specific volumes, calculate approximate temperature values of glass transition of composites and establish the mechanism and type of growth of crystalline formations depending on the concentration of AP. Along with this, it becomes obvious that the method of dilatometric measurements provides the necessary information to select the optimal cooling conditions for the polymer composite during the process of injection molding.

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Поступила в редакцию 16.04.2019
Принята к опубликованию 19.12.2019

Received 16.04.2019
Accepted 19.12.2019