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ВЛИЯНИЕ ВОЛОКНИСТОГО НАПОЛНИТЕЛЯ НА МЕХАНИЗМ И КИНЕТИЧЕСКИЕ ЗАКОНОМЕРНОСТИ КРИСТАЛЛИЗАЦИИ БАЗАЛЬТОПЛАСТИКОВ НА ОСНОВЕ РАНДОМ СОПОЛИМЕРА ПОЛИПРОПИЛЕНА

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В работе приводятся результаты исследования влияния содержания волокнистого базальта на закономерность кристаллизации композитов на основе рандом сополимера полипропилена. Исследования проводили методом ступенчатой дилатометрии в диапазоне температур 20 – 210 °С. Исследовалась зависимость удельного объема композита от температуры и содержания наполнителя. В результате были определены значения температуры фазового перехода первого рода, температуры стеклования и занятого удельного объема. Приближенная оценка температуры стеклования композитов определялась графическим методом путем экстраполяции верхней ветви дилатометрической кривой до пересечения с нижней ветвью. Удалось установить, что по мере повышения содержания базальта вплоть до 30%масс. наблюдается закономерное снижение удельного объема или повышение плотности композитов. Изучена закономерность изменения свободного удельного объема от температуры. Показано, что с увеличением содержания волокнистого базальта наблюдается закономерное снижение свободного удельного объема. Установленная закономерность однозначно свидетельствует о том, что в процессе кристаллизации и роста кристаллических образований наполнитель вытесняется в межсферолитное аморфное пространство, отличающееся высоким содержанием свободного объема. Снижение величины свободного удельного объема свидетельствует о том, что наполнитель заполняет незанятый объем в аморфном пространстве. Для описания кинетики кристаллизации композитов в изотермических условиях использовано уравнение Колмогорова-Аврами. Определены значения обобщенной константы зародышеобразования и роста кристаллов К и постоянной n. Было установлено, что с увеличением содержания волокнистого базальта в составе рандом полипропилена наблюдается изменение механизма кристаллизации и типа роста кристаллических структур от трехмерного сферолитного до одномерного – стержневидного при непрерывном образовании гетерогенных и гомогенных центров зародышеобразования.

Ключевые слова: волокнистый базальт, ступенчатая дилатометрия, кристаллизация, температура стеклования, удельный объем, свободный удельный объем

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INFLUENCE OF FIBROUS FILLER ON THE MECHANISM AND KINETIC REGULARITIES OF CRYSTALLIZATION OF BASALT CONTAINING PLASTICS BASED ON POLYPROPYLENE RANDOM COPOLYMER

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The paper presents the results of a study of the influence of fibrous basalt content on the regularity of composites crystallization based on polypropylene random copolymer. The studies were carried out using the method of stepwise dilatometry in the temperature range 20 – 210 °C. The dependence of the specific volume on temperature and filler content was studied, as a result of which the values of the temperature of the first-order phase transition, the glass transition temperature and the occupied specific volume were determined. An approximate estimate of the glass transition temperature of the composites was determined graphically by extrapolating the upper branch of the dilatometric curve to the intersection with the lower branch. It was possible to establish that as the basalt content increases up to 30 wt. %, a natural decrease in the specific volume or an increase in the density of the composites is observed. The regularity of free specific volume change from temperature was studied. It was shown that with the increase in fibrous basalt content, there is a regular decrease in free specific volume. The established regularity clearly indicates that in the process of crystallization and growth of crystalline formations, the filler is forced out into the interspherolitic amorphous space, which is characterized by a high content of free volume. A decrease in the value of free specific volume indicates that the filler fills the unoccupied volume in the amorphous space. In accordance with the Kolmogorov-Avrami equation, the kinetic regularity of crystallization of composites under isothermal conditions was studied, and the values of the generalized nucleation and growth constant of crystals K and the constant n were determined. It was found that with an increase in the content of fibrous basalt in the composition of random polypropylene, a change in the crystallization mechanism and the type of growth of crystalline structures is observed from three-dimensional spherulitic to one-dimensional - rod-shaped with the continuous formation of heterogeneous and homogeneous nucleation centers.

Keywords: fibrous basalt, stepwise dilatometry, crystallization, glass transition temperature, specific volume, free specific volume

Despite the large number of works in the field of obtain and research of composite materials based on polyolefins, very little attention has been devoted to the use of fibrous fillers [1, 2]. This circumstance is due to the fact that fibrous fillers are difficult to disperse and mix during processing [3, 4]. For a long time, this was associated with the lack of sufficiently efficient screw designs to improve mixing in the extruder [5-7]. However, this version has not received proper development and therefore most scientists have become increasingly inclined to use effective compatibilizers [8, 9]. The main function of the compatibilizer was not only to improve the compatibility of the mixed components of the blend, but also to ensure stability in maintaining the compatibilizer in the composition of the composite during its long-term operation under harsh extreme operating conditions. Therefore, when selecting a compatibilizer, we proceeded from the principle of eliminating the possibility of its sweating during the processing and operation of composite materials.

It should also be noted that the lack of systematic research on the structure and properties of composite materials with a fibrous filler dictates the need, first of all, for a more detailed study of its behavior during the cooling process - crystallization and the mechanism of growth of crystalline structural units.

In this regard, the purpose of this work was, using the example of a polypropylene random copolymer (RPP) and fibrous basalt (FBS), to study the influence of a multifunctional compatibilizer on the kinetic patterns of crystallization and the growth mechanism of crystalline formations in composite materials.

EXPERIMENTAL PART

Materials

Polypropylene random copolymer (RPP) brand RP2400 ("SOCAR POLYMER, Azerbaijan") is a thermoplastic statistical copolymer of ethylene/propylene with the following characteristics: tensile strength 28.5 MPA, elongation at break 600%, melt flow rate 1.78 g/10 min, density 904 kg/m^3, melting temperature 146 °C, heat resistance 131 °C, crystallinity degree – 57%.

Fibrous basalt (FBS) («Company "Russian Basalt", Chelyabinsk region») is an effusive igneous rock of green-black color, which has a fibrous structure and belongs to the main composition of the normal alkalinity series of the basalt group. From a chemical and mineralogical point of view, basalt has a complex structure. It contains intertwined crystalline formations and fine-grained suspensions of magnetite, silicates and metal oxides. The structure of the mineral consists of amorphous volcanic glass, feldspar crystals, sulfide ores, carbonates, and quartz. Agvit and feldspar form the basis of the mineral.

The content of silica (SiO_2) ranges from 42 to 52-53%, the sum of alkalis Na_2+K_2 up to 5%, in alkaline basalts up to 7%.

Compatibilizer – (PP-g-MA) – Exxelor PO1020 (ExxonMobil Chemical) PP modified with maleic anhydride, designed to improve the compatibility of mineral fillers with all brands of PP, including RPP. It is introduced into the composition of the composite based on RPP in an amount of 1.0-3.0 wt. %. The melt flow rate (MFR) is 34 g/10 min.

Stepwise dilatometry method

Dilatometric studies were carried out on an IIRT-1 device, converted to a dilatometer with a load of 5.3 kg and in a temperature range from 210 °C to room temperature.

Preparation of composites

In order to modify the properties of RPP, a compatibilizer (PP-g-MA) in an amount of 1.0-3.0 wt. % was first introduced into its composition on hot rollers at a temperature of 160-175 °C. Then basalt was added to the molten polymer mixture in parts. The roller friction was equal to 1.29. The objective of the study was to conduct a preliminary assessment of the dispersion of filler particles in the composite composition under conditions of intense mixing in the melt mode.

RESULTS AND ITS DISCUSSION

The use of the stepwise dilatometry method to study the kinetic regularities of crystallization of RPP composites with FBS opens up a promising opportunity to use the results obtained for an approximate assessment of the technological features of the cooling mode of a product in a mold in the injection molding unit. In the process of studying the dependence of the specific volume of composites on temperature, it seems possible to determine the first-order phase transition, the glass transition temperature, and the occupied and free specific volume of composites [10]. RPP composites with different FBS contents were used as the object of study. The concentration of FBS varied within the range of 5.0-30.0 wt. %. The objective of the study was to study the effect of fibrous filler on the regularity of changes in the temperature dependence of the specific volume and free specific volume.

Fig. 1 shows the dependence of the specific volume of RPP composites on temperature with different contents of FBS. Analyzing the dilatometric curves in this figure, it can be established that the content of FBS has a noticeable effect on the regularity of their changes. As can be seen from this figure, as the temperature of the sample decreases, the specific volume decreases and at the temperature of the first-order phase transition there is a sharp jump in the change in the value of this indicator. The phase transition for samples with 5.0-20.0 wt. % FBS content occurs at a temperature of 126 °C, and for the RPP + 30 wt. % FBS composite, the value of this indicator decreases to 120 °C. There is reason to believe that such a decrease in the phase transition temperature can be interpreted by a sufficiently high content of FBS, at which the steric factor significantly increases, restraining the process of crystallization of the highly filled composite. It is characteristic that the higher the FBS content, the lower the specific volume of the samples and, accordingly, the higher their density. Along with this, with an increase in the FBS content, the magnitude of the abrupt change in the specific volume in the region of the first-order phase transition decreases significantly. So, for example, if for the original RPP the jump in the specific volume in the region of the phase transition is 0.11 cm^3/g, then for composites with 5.0; 10.0; 15.0; 20.0; 30.0 wt. % FBS content, the value of this indicator will correspondingly change in the following sequence: 0.092; 0.088; 0.063; 0.060; 0.041 cm^3/g. This change in the specific value in the phase transition region is due to the fact that with an increase in the filler content in the composite, the proportion of the polymer matrix in it, which is known to be responsible for crystallization processes, correspondingly decreases.

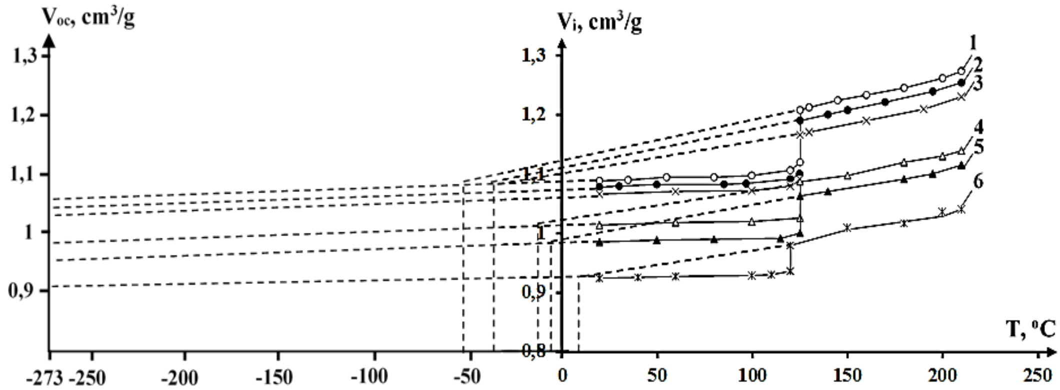

Fig. 1. Influence of FBS content (wt. %) on the temperature dependence of the specific volume of RPP-based composites: 1- initial RPP; 2- 5.0; 3- 10.0; 4- 15.0; 5- 20.0; 6- 30.0.

Рис. 1. Влияние содержания ВБЗ (%масс.) на температурную зависимость удельного объема для композитов на основе РПП: 1- исходный РПП; 2- 5,0; 3- 10,0; 4- 15,0; 5- 20,0; 6- 30,0

Table

Effect of FBS content on the density, occupied volume and glass transition temperature of composites based on RPP

***Таблица*. Влияние содержания ВБЗ на плотность, занятый объем и температуру стеклования композитов на основе РПП**

№	Composition of the RPP+VBZ composite, wt. %	Occupied volume, cm^3/g	Density at (-273 °C), g/cm^3	Density at 20 °C, g/cm^3	Glass transition temperature, °C
1	RPP	1.058	0.945	0.921	-52
2	RPP +5.0	1.045	0.957	0.928	-34
3	RPP +10.0	1.034	0.967	0.939	-34
4	RPP +15.0	0.998	1.002	0.985	-10
5	RPP +20.0	0.967	1.034	1.015	-8
6	RPP +30.0	0.914	1.094	1.082	+10

The dilatometric research method is an effective method for approximate estimation of the glass transition temperature of composite materials [10-12]. As can be seen from Fig. 1, extrapolation of the upper branch of the dilatometric curve to the lower one allows us to determine the glass transition temperature of composites using a graphical method at the point of their intersection. At the same time, extrapolation of the lower branch of the dilatometric curve to absolute zero temperature (-273 °C) allows us to estimate the occupied specific volume, the results of which are summarized in Table. Comparing the data presented in this table, it can be noted that as the FBS content increases, a decrease in the specific volume and, accordingly, an increase in the density of the composite at absolute zero and room temperature are observed. If we analyze the results of studying the glass transition temperature, we can establish that in this case, with an increase in the filler content, there is a natural increase in the glass transition temperature of the composites.

When studying the process of crystallization of composites, problematic issues related to the measurement of free specific volume (V_f), which provides fairly complete information about the nature of the change in the state of the interphase amorphous region of composites depending on the filler content, become of no small importance [12]. It is known that the structure of semicrystalline polyolefins consists of a crystalline and an amorphous phase. The formation of the structure of these polymers during cooling and, accordingly, crystallization proceeds in such a way that the growth of the crystalline phase is accompanied by the displacement of all “foreign particles” and polar groups into the interphase amorphous region [13, 14]. Thus, a more dense crystalline phase is formed, consisting of RPP macrochains and a less dense amorphous region, which contains in its volume defects of crystalline formations, passing chains of spherulites, filler particles, as well as segments of the compatibilizer macrochain containing polar groups of maleic anhydride. In other words, the interphase amorphous region accumulates in its space everything foreign contained in the composite. Therefore, analysis of changes in Vc of composites allows us to obtain a fairly clear idea of the processes occurring in the interphase region [15, 16].

Fig. 2 shows the temperature dependence of V_f for composites with different FBS contents. The value of V_f is determined by the difference between the specific volume at any temperature and the occupied specific volume: $V_f = V_i – V_o$. As can be seen from this figure, a decrease in temperature is accompanied by a natural decrease in the value of this indicator throughout the entire temperature range, down to zero at -273 °C. Attention should also be paid to the fact that in a viscous-flow state, a decrease in temperature leads to a general decrease in V_f of the polymer mass. And only after a first-order phase transition occurs, a segregation process is observed in the structure of the composite with the formation of an amorphous and crystalline component. A further decrease in temperature leads to the fact that after the phase transition, the decrease in V_f occurs predominantly in the interphase amorphous region. If you trace the change in V_f depending on the FBS content, you can pay attention to the fact that with an increase in the amount of introduced filler, the value of this indicator noticeably decreases. This circumstance is important, since it once again confirms the opinion that during the crystallization process of composite, FBS is embedded in a less dense amorphous region.

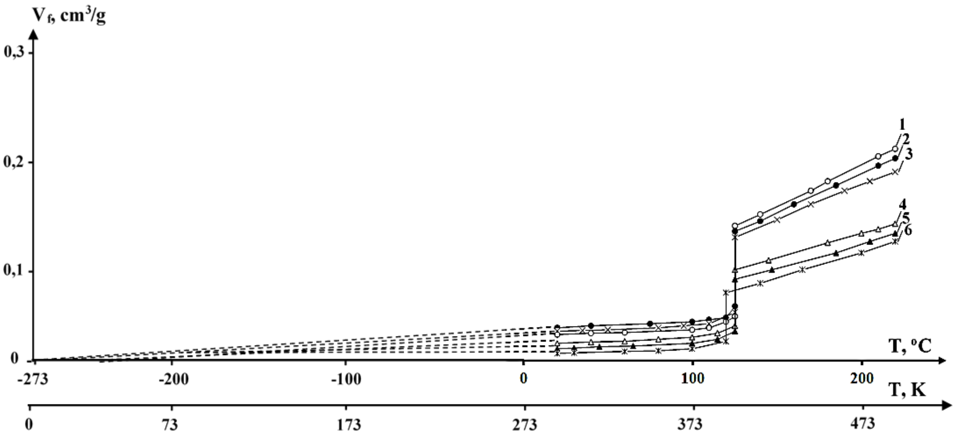

Fig. 2. Influence of FBS content (wt. %) on the temperature dependence of the free specific volume of RPP-based composites: 1- initial RPP; 2- 5.0; 3- 10.0; 4- 15.0; 5- 20.0; 6- 30.0

Рис. 2. Влияние содержания ВБЗ (%масс.) на температурную зависимость свободного удельного объема композитов на основе РПП: 1- исх. РПП; 2- 5,0; 3- 10,0; 4- 15,0; 5- 20,0; 6- 30,0

And finally, the process of dilatometric research ends with the study of crystallization isotherms of composites at the temperature of a first-order phase transition. This method allows one to obtain a fairly clear picture of the formation of crystalline formations under isothermal conditions [9, 17, 18]. Kinetic measurements of the crystallization process were carried out according to the Kolmogorov-Avrami theory, which confirms its applicability for use in polymer composites. According to this theory, the crystallization process proceeds in accordance with the expression:

$$\varphi = e^{-K\tau^n} \tag{1}$$

where φ is the part of the polymer that has not yet undergone transformation into the crystalline phase; K – generalized constant of nucleation and growth of crystals; τ – crystallization time at the temperature of a first-order phase transition, in seconds; n-constant is in the range of 1-4, defined as the tangent of the angle of inclination of the curve to the abscissa axis and depends on the nucleation mechanism and the shape of the growing crystals in the composites under consideration [9, 10, 19].

Taking the double logarithm of the Kolmogorov-Avrami equation yields the following expression:

$$lg(-ln\varphi) = lgK + nlg\tau \qquad (2)$$

this equation (2) represents the dependence in the form of a straight line in $lg(-ln\varphi)$ coordinates on $lg\tau$.

Fig. 3 shows the crystallization isotherms of composites based on RPP with different contents of FBS. Analyzing the curves in this figure, one can notice that with an increase in the FBS content, the angle of inclination of the curve to the abscissa axis decreases, indicating a decrease in the value of the constant (n). In accordance with the numbering of the curves in Fig. 3, the value of n is correspondingly equal to: 1 – 3.75; 2 – 2.85; 3 – 2.61; 4 – 2.15; 5 – 1.83; 6 – 1.48. The lgK value was determined by extrapolating the curves to the ordinate at the value $lg\tau = 0$. In accordance with the curves in Fig. 3, the lgK value changed in the following sequence: 1- (-1.5); 2- (-1.8); 3- (-2.3); 4- (-3.0); 5- (-3.4); 6- (-3.7). Based on the research results obtained, the Kolmogorov-Avrami equations were derived for each composite separately:

$$lg(-ln\varphi) = -1.5 + 3.75lg\tau \qquad (3)$$

$$lg(-ln\varphi) = -1.8 + 2.85lg\tau \qquad (4)$$

$$lg(-ln\varphi) = -2.3 + 2.61lg\tau \qquad (5)$$

$$lg(-ln\varphi) = -3.0 + 2.15lg\tau \qquad (6)$$

$$lg(-ln\varphi) = -3.4 + 1.83lg\tau \qquad (7)$$

$$lg(-ln\varphi) = -3.7 + 1.48lg\tau \qquad (8)$$

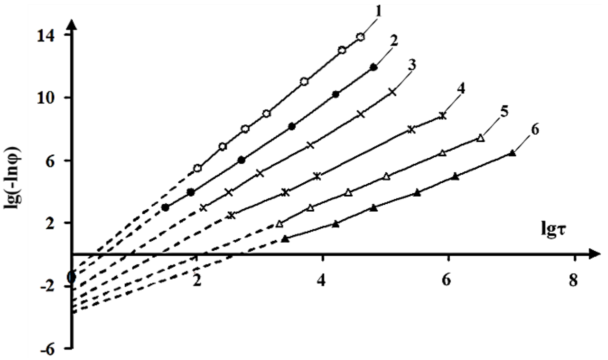

Fig. 3. Influence of FBS content (wt. %) on the regularity of change in crystallisation isotherms of RPP-based composites at the temperature of phase transition of the first kind: 1- initial RPP; 2- 5.0; 3- 10.0; 4- 15.0; 5- 20.0; 6- 30.0

Рис. 3. Влияние содержания ВБЗ (%масс.) на закономерность изменения изотерм кристаллизации композитов на основе РПП при температуре фазового перехода первого рода: 1- исходный РПП; 2- 5,0; 3- 10,0; 4- 15,0; 5- 20,0; 6- 30,0

Given the crystallization time (τ, s), using the given formulas (3-8), one can determine the part of the polymer that has not yet undergone a transition to the crystalline phase in the region of the phase transition. When studying the mechanism of crystallization of RPP composites depending on the content of FBS, it is possible to determine the type of growth of crystalline formations from the value of n. So, for example, if for the initial RPP and composites with 5.0-10.0 wt. % FBS the process of isothermal crystallization in the region of a first-order phase transition is accompanied by a three-dimensional type of crystal growth with the formation of spherulites, then in composites containing 15.0-20.0 wt. % FBS a two-dimensional lamellar type of growth takes place crystals with the continuous formation of nucleation centers. At 30.0 wt. % content, the simplest one-dimensional – rod-shaped type of crystal growth predominates.

Thus, it becomes obvious that the concentration of FBS in the composition of the compatibilized composite based on RPP has a significant effect on the mechanism and type of growth of crystalline formations. The transition from a three-dimensional crystal structure of RPP to a one-dimensional one indicates the ability of the fibrous filler to create significant steric obstacles to the growth and improvement of crystalline formations. As a result, any changes associated with the type of growth of crystalline formations will undoubtedly affect the formation of the corresponding supramolecular structure. This circumstance is very important, since all these structural changes have a significant impact on the basic physical-mechanical and physical-chemical characteristics of composite materials. Therefore, it would be appropriate to state that the process of crystallization and crystal growth itself is ultimately a fundamental factor influencing the processes of segregation and the formation of the interphase region, which is most sensitive to changes in the structure and properties of composite materials [21].

CONCLUSION

Based on the studies conducted, it can be argued that the developed composites based on RPP and FBS generally retain their crystalline structure. Therefore, the resulting composites can be classified as promising structural materials that can be processed using standard methods and, thus, find practical application in various fields of modern engineering and technology. The effect of FBS content in the range of 5.0-30.0 wt. % on the temperature dependence of the specific volume, free specific volume, occupied volume and glass transition temperature of the composites was studied using the stepwise dilatometry method.

The results of a theoretical analysis of the regularity of changes in the dilatometric curve and the first-order phase transition of composite materials in the presence of a fibrous filler are presented. The values of the temperature of the first order phase transition of composites were determined depending on the FBS content. If for composites containing 5.0-20.0 wt. % basalt the phase transition occurs at a temperature of 126 °C, then for a composite with 30 wt. % content the value of this indicator decreases to 120 °C. At the temperature of a first-order phase transition and in accordance with the Kolmogorov-Avrami equation, the crystallization isotherms of the composites under consideration were studied, making it possible to establish the mechanism and type of growth of crystalline formations depending on the FBS content. It is shown that with an increase in the FBS content, a change in the type of growth of crystalline formations is observed from three-dimensional spherulitic to one-dimensional rod-shaped. Based on the research carried out, the Kolmogorov-Avrami equations were derived for basalt-containing RPP plastics, which can be used to determine the part of the polymer that has not undergone crystallization in the region of the phase transition.

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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