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ФИЗИКО-МЕХАНИЧЕСКИЕ И ТЕРМОДЕФОРМАЦИОННЫЕ СВОЙСТВА ПЛАСТИФИЦИРОВАННЫХ ВЫСОКОНАПОЛНЕННЫХ КОМПОЗИТОВ НА ОСНОВЕ ПОЛИПРОПИЛЕНА И КВАРЦА

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Аннотация. Рассмотрено влияние содержания кварца (диоксида кремния) на основные физико-механические свойства композитов на основе полипропилена. Показано, что введение компатибилизатора - сополимера полипропилена с малеиновым ангидридом – в состав композита способствует улучшению свойств и совместимости смешиваемых компонентов смеси. Исследовали такие свойства, как разрушающее напряжение, предел текучести при растяжении, относительное удлинение, теплостойкость и показатель текучести расплава. Использование синтезированного полиэтиленового воска в качестве пластификатора позволило в значительной степени

улучшить деформационные способности высоконаполненных композитов. Концентрацию кварца варьировали в пределах 5,0–25 % масс., а пластификатора 1,0–3,0 % масс. Методом термомеханических исследований показаны закономерности изменения термомеханических кривых в температурном диапазоне 20–250 °С, в зависимости от концентрации кварца, пластификатора и вулканизующего агента. Комплекс проведенных исследований открыл новые возможности упрочнения композитов и повышения их теплостойкости.

Ключевые слова: кварц, полипропилен, композит, термомеханические кривые, разрушающее напряжение, предел текучести при растяжении, вулканизация

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Original article

PHYSICAL-MECHANICAL AND THERMAL DEFORMATION PROPERTIES OF PLASTICIZED HIGHLY FILLED COMPOSITES BASED ON POLYPROPYLENE AND QUARTZ

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Abstract. The influence of quartz content (silicon dioxide) on the main physical-mechanical properties of composites based on polypropylene is considered. It is shown that the loading of a compatibilizer – a copolymer of polypropylene with maleic anhydride – into the composition of the composite improves the properties and compatibility of the mixed components of the mixture. Properties such as ultimate tensile stress, tensile yield strength, elongation at break, Vicat softening temperature and melt flow index were studied. The use of synthesized polyethylene wax as a plasticizer made it possible to significantly improve the deformability of highly filled composites. The quartz concentration was varied within 5.0–25 % wt., and the plasticizer concentration was 1.0–3.0 % wt. The method of thermomechanical studies shows the patterns of change in thermomechanical curves in the temperature range of 20–250 °C, depending on the concentration of quartz, plasticizer and vulcanizing agent. The complex of studies carried out has opened up new possibilities for strengthening composites and increasing their Vicat softening temperature.

Keywords: quartz, polypropylene, composite, thermomechanical curves, ultimate tensile stress, tensile yield strength, vulcanization

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Introduction

Polyolefins are increasingly being used in various industries every year. Interest in polyolefins is due to the fact that they are characterized by a unique combination of structure and properties, which makes it possible to obtain high-quality structural products on their basis [1–3 extrusion, injection molding, vacuum-pneumatic molding, pressing, etc. [4–6]. One of the representatives of

the class of polyolefins is polypropylene (PP), which is also widely used in the production of extrusion and injection molded products. The intensive development of technology and the improvement of production technology put forward new and more stringent requirements for the quality of PP. To this end, various methods are being taken to modify its structure by using various types of fillers [7, 8]. However, the use of various mineral fillers contributes to a sharp deterioration in strength characteristics, due to a significant increase in brittleness. In this regard, one of the important scientific-technical problems is the adoption of the necessary measures aimed at maintaining the basic physical-mechanical characteristics of filled PP composites at the proper level.

Based on the foregoing, the purpose of this work is to improve the properties of highly filled PP composites during its plasticization.

]. One of the main advantages of polyolefins is the possibility of their processing by almost all methods:

Experimental part

Isotactic polypropylene (PP) grade HP500M (SOCAR-POLYMER) was used as a polymer matrix) — ultimate tensile stress 33.0 MPa, elongation at break 30 %, Vicat softening temperature 160 °C, melting temperature 169 °C, density 903 kg/m^3, crystallinity 65 %, melt flow index 3.6 g/10 min.

Plasticizer (PEV) – low molecular weight copolyethylene wax with an average molecular weight of 2500 [9–19].

Quartz – one of the most common minerals in the earth's crust, is a polymorphic modification of silicon dioxide. Chemical formula is SiO_2.

A maleic anhydride-functionalized PP (PPMA) compatibilizer Exxelor PO1020 (Vanderbilt Chemicals, LLC) designed to improve compatibility with mineral fillers. The degree of MA grafting in PP is 5.6 % wt.

Dicumyl peroxide (DP) is a cross-linking agent, light yellow powder, with Tmelt = 40 °C, designed to obtain cross-linked structures in polymer compositions.

Ultimate tensile stress, tensile yield strength and elongation at break of PP composites were determined in accordance with State Standard 11262-80, bending strength in accordance with State Standard 9550-81. The relative experimental error did not exceed 5 %.

The size of quartz was determined on a laser diffraction analyzer model Mastersizer-3000 (Malvern) and was 1–2 μm. The method is based on the measurement of the angular dependence of the scattered light intensity during the passage of a laser beam through a dispersed sample. The range of particle size determination is 0.01–3000 μm.

Softening temperature was determined by the Vicat method.

The melt flow index (MFI) was determined on a rheometer brand MELT FLOW TESTER, CEAST MF50 (INSTRON, Italy) at a temperature of 190 °C and a load of 5 kg.

Thermomechanical properties were determined on a Kanavets instrument. The deformation was measured at successively changing temperatures (T). The tests were carried out on polymer composite tablets with a diameter of 20 mm and a thickness of 6 mm.

In order to modify the properties of PP, quartz with a size of 1.0–2.0 μm was loaded into its composition. On hot rolls at a temperature of 180 °C, 1.0–3.0 % wt. PPMA was preliminarily loaded into the PP melt. Then 1.0–3.0 % wt. PEV was added. The amount of quartz in HDPE was varied within 5.0, 10, 15, 20, and 25 % wt. The components were mixed on rollers at a temperature of 180 °C by loading quartz into the PP melt for 7–8 min. Pressing plates for testing physical-mechanical properties was carried out under pressure at a temperature of 190–200 °C and a pressure of 50 t.

Results and discuss

It is quite obvious that before proceeding with the development of a composite material based on plasticized PP, it was necessary, first of all, to consider the possibility of improving the compatibility and miscibility of the components of the mixture. For this purpose, the use of the PPMA compatibilizer made it possible, first of all, to solve the problem of the compatibility of nonpolar PP with polar quartz [20–22]. To achieve this goal, it was necessary to have data on the optimal content of PPMA and PEV in composites based on PP. In this regard, it was necessary to consider a phased study of composites before and after the loading of PPMA and PEV into the composition of the composite.

Table 1 presents the results of a study of the effect of quartz concentration on the physical-mechanical characteristics of PP-based composites. In this experiment, PPMA was additionally used, the concentration of which in the composition of the composite was varied within 1–3 % wt. Comparing the data given in Table 1 (samples 1–6), it can be established that with an increase in the quartz concentration from 5.0 to 25 % wt., a regular decrease in strength indicators is observed. Only at 5.0 % wt. quartz content, the ultimate tensile stress and tensile yield strength acquire relatively high values. As can be seen from Table 1, the elongation at break drops sharply with increasing quartz content. At the same time, attention should also be paid to the fact that at a quartz content of more than 10 % wt., elongation at break and MFI of the samples are equal to zero. The brittle state of composites corresponds to the equality of the tensile yield strength and ultimate tensile stress [23]. This state corresponds to the fact that no «neck» is formed in the samples at the moment of stretching. From a comparative analysis of the MFI data, it can be seen that the compatibilized composites (samples 7–21) have relatively high values. It follows from the obtained data that the compatibilizer contributes to the improvement of the strength characteristics and the fluidity of the melt. By changing the content of PPMA, we can practically control the main properties of composites.

Table 1. Influence of a compatibilizer (PPMA) on the physical-mechanical properties of filled composite materials based on pp and quartz

№	Composition of polymer composite, % wt.	Tensile yield strength, MPa	Ultimate tensile stress, MPa	Elongation at break, %	MFI, g/10min	Vicat softening temperature, °C
1	PP	35.2	33.0	30	3.6	160
2	PP+5qu	36.6	35.2	20	1.9	160
3	PP+10qu	32.4	31.8	10	0.4	161
4	PP+15qu	29.7	29.7	–	–	163
5	PP+20qu	25.5	25.5	–	–	165
6	PP+25qu	19.7	19.7	–	–	167
7	PP+5qu+1C	35.8	34.9	30	2.1	160
8	PP+10qu+1C	33.0	32.1	20	1.2	160
9	PP+15qu+1C	28.3	28.3	–	0.3	161
10	PP+20qu+1C	25.9	25.9	–	–	162
11	PP+25qu+1C	20.1	20.1	–	–	163
12	PP+5qu+2C	36.6	35.9	35	2.4	160
13	PP+10qu+2C	34.2	33.7	25	1.5	160
14	PP+15qu+2C	31.5	31.0	15	0.8	162
15	PP+20qu+2C	27.6	27.6	10	0.3	162
16	PP+25qu+2C	21.8	21.8	–	–	163
17	PP+5qu+3C	36.2	35.0	35	2.7	159
18	PP+10qu+3C	33.7	32.5	25	1.8	160
19	PP+15qu+3C	30.4	29.8	15	1.1	161
20	PP+20qu+3C	27.0	27.0	10	05	162
21	PP+25qu+3C	20.2	20.2	–	–	162

Notes: qu – quartz; C – compatibilizer (PPMA).

As can be seen from Table 1, composites with 2.0 % wt. PPMA content have relatively high values in terms of strength. At a higher content of PPMA, we recorded a tendency to a slight decrease in the strength characteristics and heat resistance of the composites. This circumstance is interpreted by the fact that PPMA belongs to the group of polar polymers, is characterized by high melt fluidity and, therefore, at high concentrations, redistributing mainly in the interspherulite space, contributes to a certain decrease in properties [24, 25]. Apparently, it would be correct to assert the existence of a certain threshold concentration of PPMA, at which the best performance in terms of properties is achieved.

It seemed interesting to consider the effect of the content of the plasticizer (PEV) on the pattern of change in compatibilized composites, the results of which are summarized in Table 2. Taking into account that in Table 1 the highest values of composite properties were achieved in samples with 2 wt. % PPMA content, in the studies below it was taken as a basis.

As can be seen from Table 2, the loading of PEV into the composition of HDPE samples filled with quartz is accompanied by a noticeable change in their physical-mechanical characteristics. So, for example, in samples 1–15, an increase in the content of PEV is accompanied by a slight decrease in strength parameters. The exception is composites with 20–25 % wt. quartz content. This is due to the fact that the loading of SEV into the composition of a relatively highly filled composite (samples 4, 5, 9, 10, 14, and 15) is accompanied by some increase in the ultimate tensile stress and tensile yield strength. In all likelihood, the loading of SEV into the composition of the composite in the melt mode leads to the fact that, upon cooling, growing crystalline formations displace quartz particles, polar regions of PPMA macrochains, and low molecular weight SEV macrochains into the interspherolitic region [26, 27].

Table 2. Influence of the content of plasticizer (PEV) on the physical-mechanical properties of composite materials based on PP and quartz

№	Composition of polymer composite, wt. %	Tensile yield strength, MPa	Ultimate tensile stress, MPa	Elongation at break, %	MFI, g/10min	Vicat softening temperature, °C
1	PP+5qu+1PEV	34.7	33.0	30	2.2	160
2	PP+10qu+1PEV	32.9	32.0	20	1.2	160
3	PP+15qu+1PEV	29.0	28.6	10	0.6	161
4	PP+20qu+1PEV	26.1	26.1	–	–	162
5	PP+25qu+1PEV	20.4	20.4	–	–	164
6	PP+5qu+2PEV	32.3	30.8	30	2.7	159
7	PP+10qu+2PEV	30.1	29.2	30	1.8	160
8	PP+15qu+2PEV	28.6	27.5	20	1.0	160
9	PP+20qu+2PEV	27.0	26.4	10	0.3	161
10	PP+25qu+2PEV	21.2	21.2	–	–	161
11	PP+5qu+3PEV	30.2	28.5	40	3.3	159
12	PP+10qu+3PEV	28.3	27.2	30	2.3	159
13	PP+15qu+3PEV	26.7	25.6	20	1.6	160
14	PP+20qu+3PEV	25.9	24.8	10	0.7	160
15	PP+25qu+3PEV	21.5	20.6	10	0.3	160
16	PP+5qu+2C+1PEV	35.1	34.2	30	2.9	160
17	PP+10qu+2C+1PEV	33.5	32.3	20	2.1	160
18	PP+15qu+2C+1PEV	31.0	30.6	15	1.1	160
19	PP+20qu+2C+1PEV	28.0	27.7	10	0.6	161
20	PP+25qu+2C+1PEV	22.3	22.3	–	0.2	161
21	PP+5qu+2C+2PEV	34.4	33.1	40	3.2	158
22	PP+10qu+2C+2PEV	32.0	30.8	40	2.2	159
23	PP+15qu+2C+2PEV	30.1	29.0	30	1.5	160
24	PP+20qu+2C+2PEV	27.4	26.5	25	0.9	160
25	PP+25qu+2C+2PEV	23.0	22.7	10	0.4	160
26	PP+5qu+2C+3PEV	32.5	30.8	45	4.3	157
27	PP+10qu+2C+3PEV	30.1	28.9	40	3.4	158
28	PP+15qu+2C+3PEV	28.3	27.0	35	2.3	158
29	PP+20qu+2C+3PEV	26.3	25.2	25	1.4	159
30	PP+25qu+2C+3PEV	21.4	19.8	20	0.8	159
Notes: qu – quartz; C – compatibilizer (PPMA).						

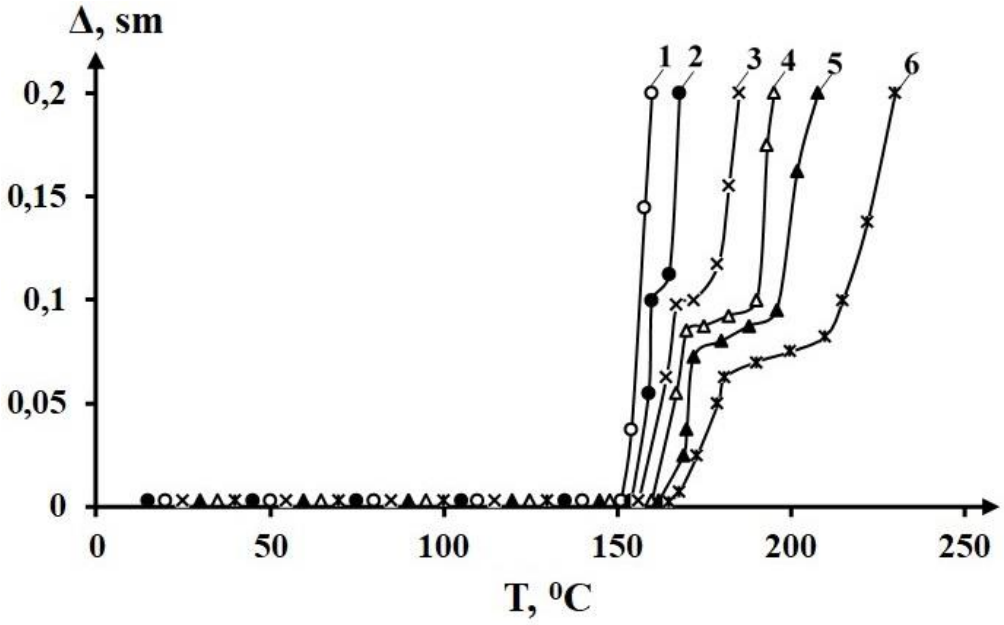
As a result, in the interspherolitic region, quartz particles acquire a certain mobility under the influence of SEV, which improves the conditions for the movement of passing chains relative to each other and quartz particles during uniaxial tension. And as a result, elongation at break and strength characteristics are improved.

When a dispersed filler is loaded into the PP composition, the possibility of stress transfer from the polymer matrix to solid particles decreases to such an extent that opposite intrastructural processes occur in the composite [28, 29]. On the one hand, quartz at certain concentrations increases the strength of the sample, and on the other hand, there is a possibility of brittleness in it. As a result, due to the uneven redistribution of internal stress, an increase in defectiveness and pseudoporosity of the supramolecular structure of highly filled composites, a regular deterioration in the ultimate tensile stress of the polymer matrix occurs [30, 31]. Indeed, according to the data of Tables 1 and 2, it can be established that in those highly filled composites in which there was no elongation (samples 4–6), they acquire in the presence of PEV and PPMA.

A serious technological disadvantage of brittle polymer composites lies in the fact that during operation and especially after exposure to such external factors as UV irradiation, γ-radiation, a sharp temperature drop, prolonged mechanical vibration, chemical reagents, etc. rapidly undergo premature failure and loss of structural characteristics [32]. Therefore, when developing a composite material, it is very important to achieve high strength indicators, provided that the elongation at break is maintained at a satisfactory level. If we analyze the data in Table 2, we can see that the loading of PEV and PPMA is accompanied by an increase in the MFI of composites, which is very important from the point of view of their processing by methods such as injection molding and extrusion.

One of the effective methods for evaluating the behavior of composites in the solid and viscous state is thermomechanical analysis in a wide temperature range. In this regard, Figure 1 shows the thermomechanical characteristics of

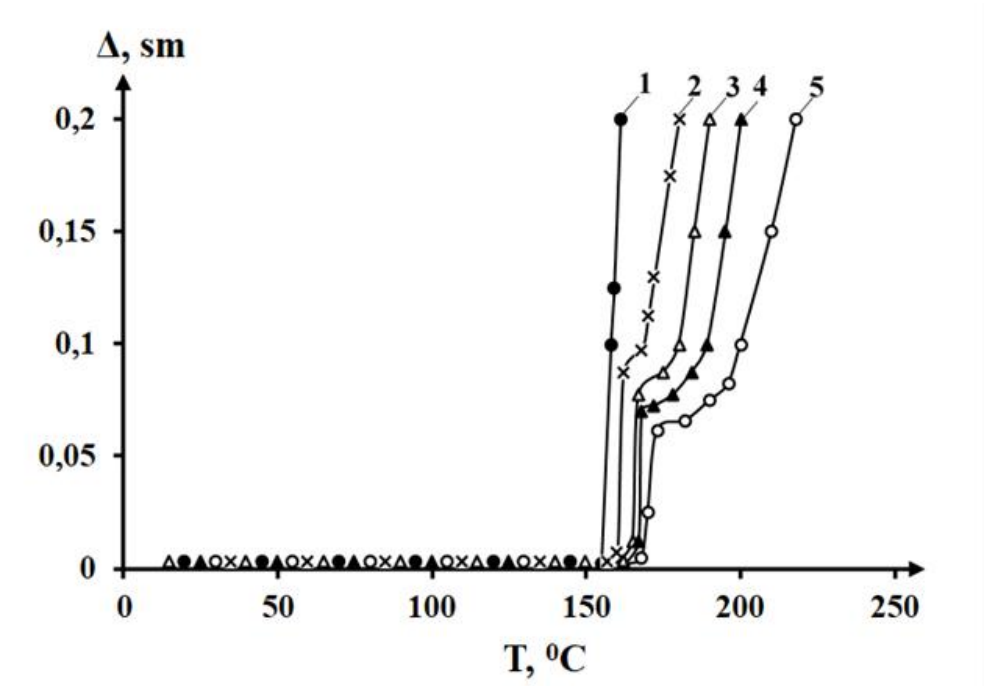
compatibilized PP composites with different quartz content. From a comparative analysis of thermomechanical curves, it can be established that if for compatibilized PP the first-order phase transition occurs at a temperature of 156 °C, then after the loading of quartz in an amount of 5.0 % wt., 10 % wt., 15 % wt., 20 % wt., 25 % wt., the value of this indicator changes in the following sequence: 158 °C, 160 °C, 163 °C, 166 °C, 168 °C. Along with this, with the loading of 5.0 % wt. or more quartz, a kind of plateau appears in the region of the viscous-flow state. Moreover, the width of this plateau increases with an increase in the concentration of quartz in the composite. The observed regularity can be interpreted by the fact that as the quartz concentration increases, the viscosity of the melt increases significantly, which, to a certain extent, contributes to slowing down the deformation processes. This is confirmed by the results of assessing the MFI of composites in Table 1, where at 10 % wt. quartz content and above, zero melt flow is observed.



1 – initial PP; 2 – 5.0 % wt.; 3 – 10 % wt.; 4 – 15 % wt.; 5 – 20 % wt.; 6 – 25 % wt.

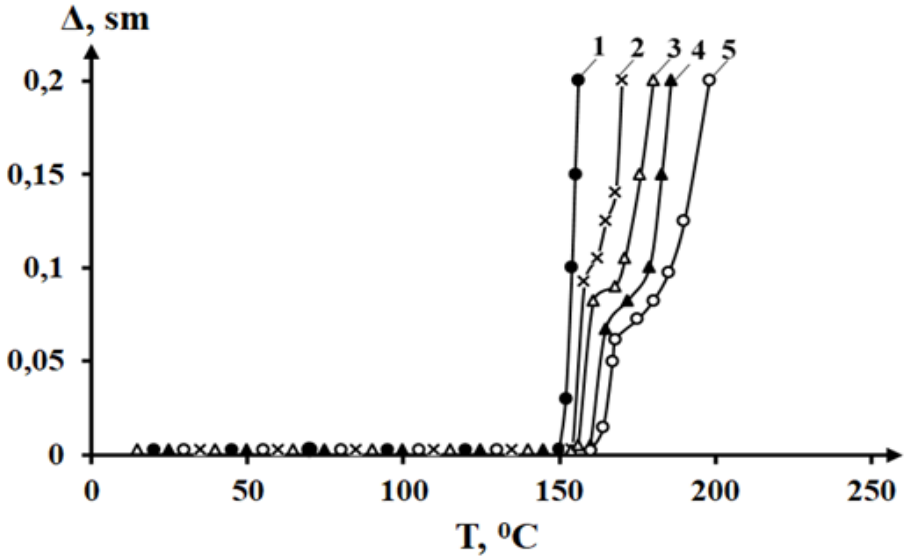
Figure 1. Thermomechanical curves of compatibilized composites based on PP with different quartz content

A somewhat different change occurs in the deformation of samples in plasticized PP composites, the results of which are shown in Figure 2 and Figure 3. So, for example, Figure 2 shows the dependence curves of the deformation of plasticized composites PP + quartz + PPMA with the same quartz content, which differ in that the samples additionally contain 2.0 % wt. SEV in their composition. From a comparative analysis of the thermomechanical curves in Figure 2, it can be established that they differ significantly from non-plasticized PP composites. The phase transition of the first order for the considered composites changes in the region of 157–166 °C. The main difference is manifested in the fact that the plateau region is almost completely absent in samples with a 5.0 % wt. plateau content (curve 1). For composites with a higher quartz content (10–25 % wt.), the plateau region is strongly smoothed out.



1 – 5.0 % wt.; 2 – 10 % wt.; 3 – 15 % wt.; 4 – 20% wt.; 5 – 25 % wt.

Figure 2. Thermomechanical curves of plasticized 2.0 % wt. PEV compatibilized composites based on PP with different quartz content



1 – 5.0 % wt.; 2 – 10 % wt.; 3 – 15 % wt.; 4 – 20% wt.; 5 – 25 % wt.

Figure 3. Thermomechanical curves of plasticized 3.0 % wt. PEV compatibilized composites based on PP with different quartz content

According to Figure 3, when 3.0 % wt. SEV is loaded into the composition of the PP + quartz + PPMA composite, an even greater narrowing of the plateau region is observed. In addition, the fundamental difference between plasticized composites is not only the narrowing of the region of the first-order phase transition, but also a decrease in the temperature region of deformation in the viscous-flow state (up to 2 mm mark). So, for example, according to Figure 1, at 2 mm deformation, with an increase in the quartz concentration from 5.0 % wt. to 25 % wt., the temperature range in the viscous-flowing state changed within 160–232 °C. The loading of 2.0 % wt. and 3.0 % wt. SEV promotes an increase in the temperature range of deformation from 159 °C to 218 °C and from 159 °C to 198 °C, respectively. With the loading of 3.0 % wt. PEV into the composition of composites with a quartz content of 5.0 % wt., 10 % wt., 15 % wt., 20 % wt., and 25 % wt., the maximum deformation temperature on the thermomechanical curves decreases by 10 °C; 16 °C; 18 °C; 23 °C and 32 °C respectively. On the basis of

the results obtained, it can be argued that the plasticizing effect of PEV is most pronounced on composites with a high content of quartz. It becomes obvious that theloading of SEV into the composition of the compatibilized PP + quartz composite significantly affects the decrease in the viscosity of the melt and, as a consequence, the decrease in the width of the plateau on the thermomechanical curves. This circumstance is important, since it allows us to state that plasticized samples should be processed in a narrower temperature range.

One of the promising possibilities for increasing the heat resistance of polymeric materials is the use of the possibility of their chemical modification in the process of peroxide crosslinking [33–35]. This method of modification of polymer composites allows not only to increase their heat resistance, but also has a significant effect on a sharp decrease in their MFI. The latter circumstance creates a situation where the very process of their processing by injection molding and extrusion becomes impossible. In such cases, either the pressing method is used to obtain sheet products, or by reducing the cross-linking agent (dicumyl peroxide) to the minimum allowable concentration, the MFI of the composites is maintained at the proper level. Moreover, the decrease in the concentration of the vulcanization agent should be accompanied by a simultaneous increase in the content of the surfactant. Therefore, the MFI in the vulcanizates was increased by loading 3.0 % wt. PEV.

Table 3 shows the physical-mechanical properties of vulcanized compatibilized and plasticized filled composites based on PP with 10 % wt. quartz content. The concentration of dicumyl peroxide (DP) was varied within 0.25 % wt., 0.5 % wt., 1.0 % wt., 2.0 % wt. Analyzing the data in this Table 3, it can be established that with an increase in the concentration of dicumyl peroxide from 0.25 % wt. to 2.0 % wt., a rather noticeable increase in strength characteristics is observed with a maximum at 1.0 % wt. content of the vulcanizing agent. It is characteristic that the heat resistance constantly increases from 158 °C to 195 °C as the DP content increases. As expected, the MFI of

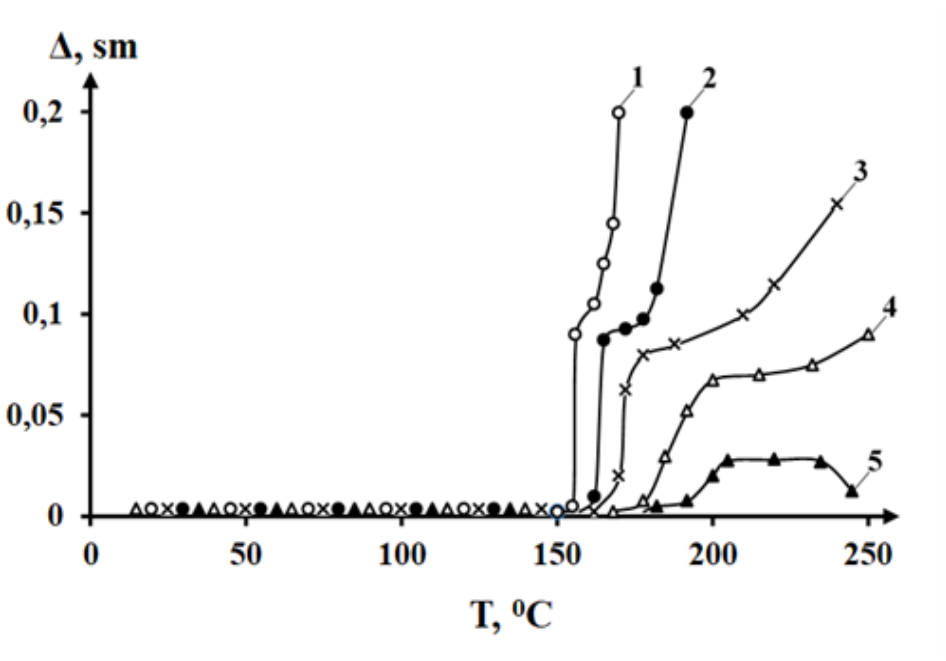
composites deteriorates significantly. So, for example, at 1.0 wt. % DP and above, MFI=0 (samples 4 and 5). With the loading of 0.25 % wt. DP, a certain increase in elongation at break is observed simultaneously with an increase in the ultimate tensile stress. The loading of DP, on the one hand, improves the strength characteristics, heat resistance, and, to a certain extent, elongation at break. Based on the data obtained, the comparatively best indicators in terms of properties are vulcanized composites with a minimum content of DP.

Table 3. Physical-mechanical properties of vulcanized composites based on compatibilized and plasticized quartz-filled PP

№	Composite composition, wt. %	Ultimate tensile stress, MPa	Elongation at break, %	Vicat softening temperature, °C	MFI, g/10min
1	PP+2C+10qu+3P	30.1	40	158	3.6
2	PP+2C+10qu+3P+0.25DP	32.9	55	165	1.9
3	PP+2C+10qu+3P+0.50DP	34.6	35	173	0.3
4	PP+2C+10qu+3P+1.0DP	35.2	10	188	–
5	PP+2C+10qu+3P+2.0DP	32.8	–	195	–
Notes: **C** – PPMA; **qu**- quartz; **P**- plasticizer **PEV**					

To obtain a more complete picture of the processes occurring in vulcanized PP composites, Figure 4 shows thermomechanical curves of the dependence of deformation on temperature for PP composites. The samples presented in Table 3 were used as objects of study. Analyzing the thermomechanical curves in Figure 4, it can be seen that with an increase in the DP content, there is a general tendency to shift the temperature region of the ductile state towards an increase with the simultaneous appearance of a plateau, which in this case characterizes the region of highly elastic deformation. The higher the content of DP, the more clearly the area of highly elastic deformation appears, which is characteristic of rubbers. It can be seen from Figure 4 that at a DP concentration of 1.0 % wt. and higher, the

region of the viscous-fluid state noticeably decreases. This is interpreted by the fact that with an increase in the content of DP, the cross-link density increases to such an extent that the deformation processes noticeably decrease. At the maximum DP concentration of 2.0 % wt. the composite completely loses its ability to flow and vitrifies (Figure 4, curve 5).



1 – % wt. initial uncrosslinked PP composite; 2 – 0.25 % wt.;
3 – 0.5 % wt.; 4 – 1.0 % wt.; 5 – 2.0 % wt.

Figure 4. Thermomechanical curves of vulcanized and plasticized 3.0 % wt. PEV PP composite with 10 % quartz content with different DP content

Conclusions

Thus, based on the foregoing, it becomes obvious that varying the content of quartz, compatibilizer, plasticizer, and cross-linking agent, it seems possible to change the physical-mechanical, thermophysical, and deformation characteristics of PP-based composites in a fairly wide range.

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