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INVESTIGATION OF THE REGULARITIES OF PHASE TRANSITIONS IN MULTICOMPONENT POLYMER BLENDS

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The influence of the compatibilizers of an ethylene–vinyl acetate copolymer and sodium oleate on the processes of melting and crystallization and the supermolecular structure of polypropylene/copolyamide blends of composition 20/80, 40/60, and 50/50 wt.% has been investigated. It has been shown that a specific fiberization is clearly realized in the compatibilized blends even for compositions corresponding to the region of phase change. Two to three peaks of crystallization of polypropylene in the blends containing an ethylene–vinyl acetate copolymer or sodium oleate have been revealed, which is attributed to the increase in the degree of dispersion of polypropylene as a result of the improvement in the specific fiberization and to the growth in the fraction of polypropylene in the interphase transition layer.

It is well known [1] that polymer blends are being used in a modern, simple, and efficient method of modifying them and obtaining products with completely new properties. Thus, for example, processing of polymer blends opens up a completely new avenue for production of ultrafine synthetic fibers (microfibers). We are dealing with the so-called phenomenon of specific fiberization [2], where one (fiberizing) component forms numerous microfibers strictly oriented in the direction of extrusion in the mass of another (matrix) component in the case of flow of a molten blend. The morphology of a polymer blend is determined not only by the microrheological processes (deformation of droplets to liquid jets, disintegration of the latter into droplets, and migration on the radius of the extruder-die orifice) at the stage of processing but also to a large extent by the capacity of the blended polymers for crystallization and by the conditions under which it occurs. Realization of specific fiberization substantially depends on the degree of compatibility of the components of the blend and the possibility of forming a transition layer transferring the deforming force from the matrix polymer to the fiberizing polymer. Commercially produced polymers are thermodynamically incompatible, as a rule. Therefore, in processing their blends, one adds special substances improving their compatibility; they are called compatibilizers [3]. Physicochemical processes in compatibilized polymer blends were investigated in [4, 5] in detail, but no consideration was given to phase transitions.

This work seeks to study the processes of melting and crystallization in ternary compatibilized polymer blends.

Objects and Methods of Investigation. Blends of polypropylene (PP) and copolyamide (CPA) with a ratio of the components of 20/80, 40/60, and 50/50 were used as the objects of investigation. Copolyamide represented a caprolactam–AH (adipic acid and hexamethylene diamine) salt copolymer of composition 50/50. The characteristics of the starting polymers have been given in [4, 5]. As the compatibilizers we used an ethylene–vinyl acetate copolymer (EVAC) and sodium oleate. We added EVAC in an amount of 5, 10, 20, and 25% of the PP mass. The amount of the sodium amounted to 0.5, 3, and 7 wt.% of the PP mass. The polymers were blended with a combined screw-plate extruder (LGP-25). In this work, we studied phase transitions by using differential thermal analysis and x-ray structural analysis. The differential thermal analysis investigations were carried out on a derivatograph of the MOM Company (Hungary). The conditions of recording of the thermograms were as follows: rate of increase of the temperature 5 deg/min, recording speed 5 mm/min, weight 300 mg, and reference substance Al_2O_3. The error in determination of the transition temperatures was ±1 and $\pm 2^{\circ}C$ for the temperatures at the beginning and end of phase transitions. The heats

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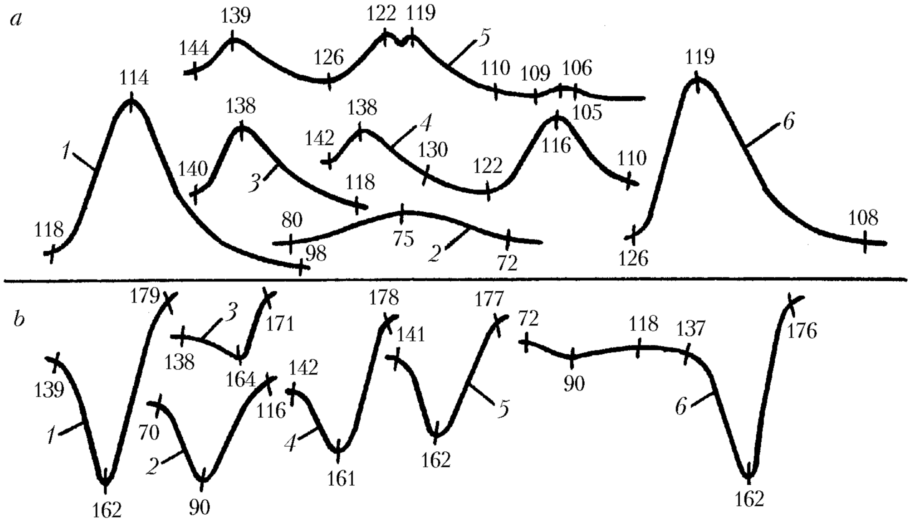

Fig. 1. Thermograms of crystallization (a) and melting (b) of the starting polymers and PP/CPA/EVAC blends: 1) PP; 2) EVAC; 3) CPA; 4) 40/60 PP/CPA; 5) 40/60/10 PP/CPA/EVAC; 6) microfiber after the extraction of CPA from sample No. 5.

of phase transitions were determined with an error of $\pm$3.5%. In calculating the thermal effects, we recorded the thermogram of melting of *n*-aminobenzoic acid with a known thermal effect of 151.9 J/g [6] for calibration.

The supermolecular structure of the PP/CPA blends and the influence of the additions of EVAC and sodium oleate on it were investigated by the method of scattering of x rays at large angles [7] with a DRON-4-07 x-ray diffractomer (Cu$_{k\alpha}$ radiation monochromated by an Ni filter). On the diffractograms obtained, we determined the angular positions (angle 2θ) of the diffraction maxima which are a consequence of the reflection of the x rays from the crystal planes with a given value of the interplane distance. Since the reflexes of the components of the blend overlap, we could determine only the total conventional degree of crystallinity (X_{cr}) by the Matthews method [8]:

$$X_{cr} = S_{cr}/(S_a + S_{cr}) ,$$

where $S_a + S_{cr}$ is the area occupied by the entire diffraction curve minus the background scattering by the collimation system and S_{cr} is the area occupied by the crystal reflexes minus the contribution of the amorphous part of the polymer (amorphous halo) to the diffraction pattern. The error of determination of X_{cr} amounted to $\pm$3%.

From the thermograms obtained in diffraction thermal analysis, we determined the temperatures and heats of phase transitions and the difference between the temperatures of their beginning and end. We calculated for PP the degree of crystallinity from the melting heat DC$_m$ and from the crystallization heat DC$_{cr}$. From the data of thermogravimetry and differential thermogravimetry, we determined the mass loss (Δm) and the corresponding temperature at which this occurs. The results of the investigations are presented in Tables 1 and 2 and in Figs. 1–6.

Results and Discussion. *Influence of EVAC Additions on Phase Transitions in PP/CPA Blends.* It is well known that the morphology of a polymer blend is not only formed by the microrheological processes at the stage of processing but is largely determined by the capacity of the blended polymers for crystallization and by the conditions under which it occurs. From Fig. 1 it is clear that PP is a crystallizing polymer with a high DC, whereas CPA, having an irregular chemical structure, is characterized by threefold lower melting and crystallization heats (Table 1). No mass loss occurs, in practice, in the interval of melting temperatures for PP, whereas the starting CPA loses 0.9% of its mass and, after letting it pass through an LGP-25 combined screw-plate extruder, 3.7% of its mass. The latter is due to the fact that low-molecular-weight compounds removed in the temperature interval 139–210°C can be accumulated in the process of melting. The presence of such compounds can have a plasticizing action on a molten CPA. Ethylene–vinyl acetate copolymer is also a weakly crystallizing low-melting polymer.

TABLE 1. Temperatures and Heats of Phase Transitions in PP/CPA/EVAC Blends

Sample No.	Composition of the blend, wt.%	Melting					Crystallization			
		T_m, °C	ΔT_m, °C	$Q_m \cdot 10^{-3}$, J/kg	DC_m, %	Δm, %	T_{cr}, °C	ΔT_{cr}, °C	$Q_{cr} \cdot 10^{-3}$, J/kg	DC_{cr}, %
1	Starting PP, granules	162	139—178	91	36	0.03	111	118—96	187	72
2	PP, granules after LGP-25	162	139—179	104	40	0.4	114	118—98	175	68
3	Starting PP, granules	150, 170	139—183	30		0.9	132	143—126	64	
4	CPA, granules, after LGP-25	164	138—171	28		3.7	138	140—118	57	
5	Starting EVAC, granules	95	80—109	28		0	78	80—75	71	58
6	EVAC, granules after LGP-25	90	70—116	50		0	75	80—72	41	
7	40/60 PP/CPA, granules after LGP-25	161	142—178	57 (total)		1.8	PP 116 CPA 138	122—110 142—130	148 37	57
8	40/60/10 PP/CPA/EVAC, granules after LGP-25	162	141—177	87 (total)		3.0	PP 122, 119, 106 CPA 139	126—104 144—126	158 57	61
9	Microfibers after the extraction of CPA from the granules of a 40/60/10 PP/CPA/EVAC blend	PP 162 EVAC 90	PP 137—176 EVAC 72—118	114	39		PP 119 EVAC 70	126—108 72—66	204	79
10	40/60/10 PP/CPA/EVAC, an extrudate after the MV-2 viscosimeter *	164	146—181	75		2	PP 123 CPA 142	129—111 148—129	195	75

* The time of storage of the molten blend in the viscosimeter's chamber is 9 min.

The data for 40/60-PP/CPA granules (sample No. 7, Table 1) demonstrate that in the PP/CPA blend, the processes of melting of the components overlap, and crystallization occurs in such a manner that CPA crystallizes first and then PP does in the presence of a solid CPA. This is the reason for the certain increase in T_{cr} of PP (Table 1). Crystallization in the presence of the crystals of the other component is carried out by heterogeneous nucleation. The crystallization rate is higher than that in homogeneous nucleation. Enhanced nucleation also leads to an increase in the crystallization temperature. The decrease in the crystallization heat and DC_{cr} of polypropylene is related to the disperse state. Thus, it has been established [9] that the rate of crystallization of a crystallizing polymer in a blend depends on the degree of its dispersity: it decreases with increase in the degree of dispersity, which is caused by the influence of the interphase layer formed. Using the low-pressure polyethylene/polyoxymethylene (LIPE/POM) and high-pressure polyethylene/polyoxymethylene (HPPE/POM) blends as an example, it has been shown in [10] that if the particles of the dispersed phase are finely divided, this contributes to an increase in the fraction of the material incorporated into the transition amorphous interphase layer between the components and consequently to a decrease in the degree of crystallinity. According to the data of [11], when the degree of dispersity is very high (the size of the polyolefin particles in polyisoprene is 0.1–0.2 μm), the crystallizing component remains amorphous in the blend. We have crystallization for larger polyolefin particles, but the DC is determined by the particle size. An unusual kind of distribution of the particles of the dispersed-phase polymer not only by size but also by the degree of crystallinity occurs. In investigation of the processes of melting of such polymer dispersions by differential thermal analysis, we can observe several peaks of melting rather than one peak [12].

With addition of 10% EVAC to a 40/60 PP/CPA binary blend, the processes of melting and crystallization change (Table 1 and Fig. 1): the heats and temperatures of phase transitions increase. Thus, for example, the total Q_m grows from 57 for the binary blend to 87 J/g for the ternary blend. In our opinion, the following processes occur in the ternary blend.

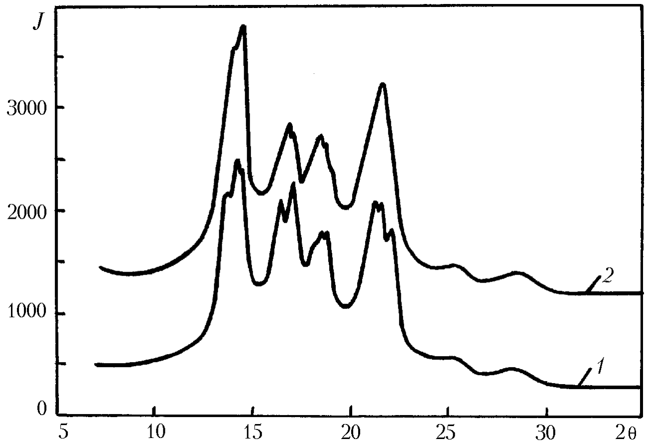

Fig. 2. Diffractograms of scattering of x rays by the PP sample: 1) along the sample's axis; 2) perpendicularly to the sample's axis.

When the ternary blend is prepared on a screw-plate extruder (LGP-25 line), CPA (139^{o}C) is the first to crystallize in the process of cooling of the melt; this substance forms dipole–dipole bonds with EVAC (specific interactions). In so doing, a molten CPA is additionally cross-linked by the latter. The above two factors enhance nucleation in crystallization of PP, which increases Q_{m}, T_{cr}, and DC_{cr}. On the other hand, PP in the ternary blend forms predominantly ultrafine fibers, i.e., the degree of its dispersity grows as compared to that of the binary blend. Therefore, two peaks — at 119 and 106^{o}C (Fig. 1) — that correspond to the crystallization of PP in the form of microfibers of different diameters appear on the thermogram of crystallization of PP. The lowest-temperature peak (at 106^{o}C) probably corresponds to the crystallization of the PP of the transition layer.

To remove CPA from the blend we extract it with ethanol. The extraction of CPA from the ternary blend takes 10–12 h at the boiling point of ethyl alcohol (T = 78^{o}C). Thereafter, the crystalline structure of PP is improved, which results in a growth in the heats of phase transitions and DCs (Table 1). To confirm the role of specific interactions in realization of the phenomenon of fiberization in the region of phase change (the PP/CPA ratio is 40/60) we conducted the following experiment.

Granules of a 40/60/10 PP/CPA/EVAC blend, obtained using the LGP-25 line of granulation of polymers, were charged into the chamber of an MV-2 viscosimeter at T = 190^{o}C. The melt was stored for 9 min, after which we obtained extrudates at a shear stress of $\tau = 5.69 \cdot 10^{4}$ Pa. The experiments have shown that the process of extraction of CPA from the extrudates is difficult and becomes much longer. The washed-off residue is hard and consists of numerous films. The entire fibrous structure has become film-type. The thermograms recorded demonstrate an increase in T_{m}, T_{cr}, Q_{cr}, and DC_{cr} of PP (Table 1). Thus, with such a long storage of the molten blend under static conditions, we have breaking of the dipole–dipole bonds, aggregation of PP, and phase separation. A consequence of these processes is the formation of a film-type structure. Thus, in the technology of processing of a ternary blend into a composite film, one must monitor the time of the processing and avoid the presence of dead zones.

Influence of EVAC Additions on the Supermolecular Structure of PP/CPA Blends. Direct information on the supermolecular structure of polymers is obtained from the results of x-ray investigations. In this work, we have recorded diffractograms of scattering of x rays at large angles (Figs. 2–4). A confirmation of a pronounced crystalline structure in the polymers is the presence of several distinct maxima (at least, of two). Polypropylene is characterized by four of the most intense maxima: $2\theta = 13^{o}55'$, $16^{o}52'$, $18^{o}45'$, and $21^{o}30'$. The "arms" on the first and fourth maxima and the manifestation of a doublet (splitting) in the region of the second maximum ($2\theta = 16^{o}38'$ and $17^{o}12'$) must demonstrate the high degree of order of macromolecules in PP crystallites. The phenomena of splitting of peaks and the presence of "arms" on the diffractograms of PP have not been described in the literature [7]. The above features of the character of scattering of x rays, observed experimentally, are related to the processes of orientation of the PP macromolecules in the plate zone and in the zone of spinneret extrusion. The aforesaid is confirmed by the differences in the diffractograms of the PP samples located along the x-ray beam and across it (Fig. 2). There is splitting in the first case and no splitting in the second case. One broad peak at $2\theta = 21^{o}20'$ is prescribed for copolyamide, which

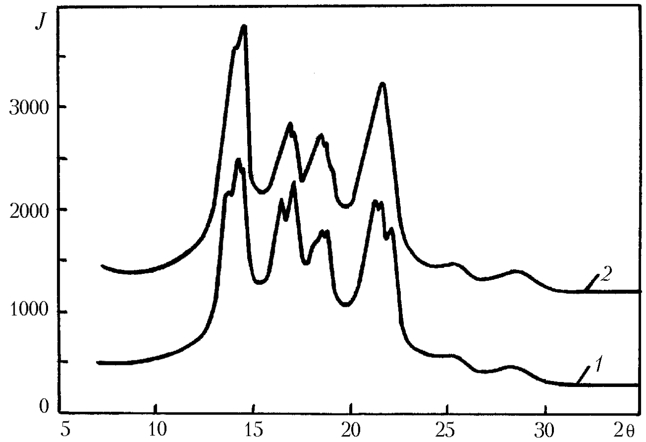

Fig. 3. Diffractograms of scattering of x rays: 1) PP; 2) additive curve; 3) 50/50 PP/CPA; 4) residue after washing-off CPA from sample No. 3.

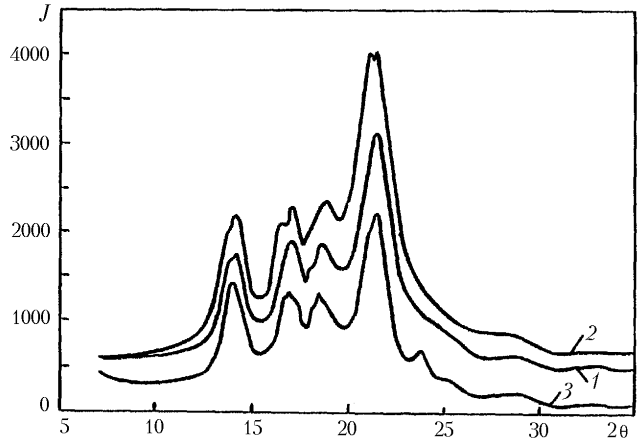

Fig. 4. Diffractograms of scattering of x rays: 1) 30/50/20 PP/CPA/EVAC after LGP-25; 2) 30/50/20 PP/CPA/EVAC after LGP + MV-2; 3) microfibers after the extraction of CPA from sample No. 1.

demonstrates an imperfect structural order of its macromolecules. The latter has already been confirmed by the data of differential thermal analysis.

A 50/50 PP/CPA blend is close to phase reversal; PP in this blend is in a coarsely divided state in the CPA mass. It is known from the literature [13] that, unlike the radial spherulites of the starting POM, in crystallization of POM in the form of microfibers, spherically degenerated spherulites in which the rays are directed predominantly along the extrusion axis are formed. From the x-ray data (Fig. 3), we have independent crystallization of the components in the 50/50 PP/CPA blend, which is confirmed by the same intensity of the experimental and additive bands of scattering of the x rays. The splitting (already discussed above) of the diffraction maxima of PP in the blend of composition 50/50 is absent, which demonstrates both the more imperfect crystalline structure and the decrease in the degree of orientation of the crystal formations of PP and macromolecules in them. The presence of the transition layer in the blends also contributes to the total amorphism of the structure: X_{cr} decreases from 65% for the starting PP to 58% for the blend. The same regularities have been revealed by the method of differential thermal analysis for the 40/60 PP/CPA blend.

By microscopic investigations it has been shown that, when PP in the 50/50 blend is replaced by EVAC, specific fiberization is sharply improved as a result of the realization of dipole–dipole interactions between the carbonyl of EVAC and the amide group of CPA. From this viewpoint (fine dispersion of PP and EVAC), crystallization conditions become worse. On the other hand, nucleation is enhanced in the ternary blend: PP crystallizes on CPA crystallites. The enhancement of nucleation contributes to the crystallization processes. It is the above two opposite factors

TABLE 2. Temperatures and Heats of Phase Transitions in PP/CPA/Sodium Oleate Blends

Sample No.	Composition of the blend, wt.%	Melting					Crystallization			
		T_m, °C	ΔT_m, °C	$Q_m \cdot 10^{-3}$, J/kg	DC_m, %	Δm, %	T_{cr}, °C	ΔT_{cr}, °C	$Q_{cr} \cdot 10^{-3}$, J/kg	DC_{cr}, %
1	20/80 PP/CPA, granules after LGP-25	162	183—180	71 (total)		3.7	PP 116, 104; CPA 136	119—110 110—96 139—119	111 57	43
2	20/80/3/ PP/CPA/Na oleate, granules after LGP-25	160	136—178	70 (total)		3.0	PP 114, 106; CPA 134	118—108 108—98 140—118	108 55	41
3	Microfibers after the extraction of CPA from a 20/80/3 PP/CPA/Na oleate blend	163	136—177	100	38	0	122	127—104	202	78
4	Composite film from a 20/80/3 PP/CPA/Na oleate blend	160 167 arm	147—176	39 (total)		3.8	PP 118, 103; CPA 139	124—113 107—97 143—124	8766	34
5	Composite film (sample No. 4) extruded with a multiplicity of 2.5	160	149—180	38 (total)		3.6	PP 120, 106 CPA 140	124—114 111—96 145—124	127 66	49
6	Filter material (after the extraction of CPA from sample No. 4)	162	139—177	95	37	0	120	125—106	194	75
7	Composite film from a 20/80/3 PP/CPA/EVAC/PEG blend	159 PEG 167 arm	149—173	24 (total)		3.4	PP 118, 106 CPA 134	124—111 111—100 143—127	104 61	40
8	Filter material (after the extraction of CPA from sample No. 7)	PP 162 EVAC 103	141—172 96—122	105, 26	41	0	PP 116, 91 EVAC 81	122—101 94—87 84—80	186	72

that determine the final crystalline structure of the PP/CPA/EVAC ternary blend. On the diffractograms of the latter, there is no splitting of the crystal peaks, but the intensity of the diffraction maxima is redistributed: the intensity of the maximum at $2\theta = 16^{\circ}52'$ has become higher than the intensity of that at $2\theta = 13^{\circ}55'$ (Fig. 4).

Additional passage of the 30/50/20 PP/CPA/EVAC blend through the capillary of the MV-2 viscosimeter (with a 9-min storage of the melt in the viscosimeter's chamber) leads to an aggregation of the PP regions, an enhancement of phase separation in the blend, and, as a result, an improvement of the PP crystalline structure. The latter is expressed in the appearance of the splitting of the maximum at $2\theta = 16^{\circ}52'$, whereas such a splitting is absent in the sample of the blend of the same composition, obtained with the use of only LGP-25 (Fig. 2). The regularity found is in good agreement with the data of the differential thermal analysis.

In the microfibers obtained after the extraction of CPA from the 30/50/20 PP/CPA/EVAC blend, the degree of crystallinity is the same as that of the starting PP but the reflexes at $2\theta = 13^{\circ}55'$ and $16^{\circ}52'$ are not split (Fig. 4). Thus, we can speak of the improvement of the PP crystalline structure in the process of extraction. However, by virtue of the disperse state of PP, it is nonetheless more imperfect than that in the starting PP.

*Influence of Sodium-Oleate Additions on Phase Transitions in the PP/CPA Blends.*The influence of sodium-oleate additions on the processes of melting and crystallization has been investigated for a PP/CPA ratio of 20/80 (Table 2, Fig. 5). The degree of dispersion of PP in the 20/80 blend is higher than that in the 40/60 blend; therefore, we have a decrease in the values of Q_m, Q_{cr}, and DC_{cr} in the first blend (Table 2) as compared to the second blend.

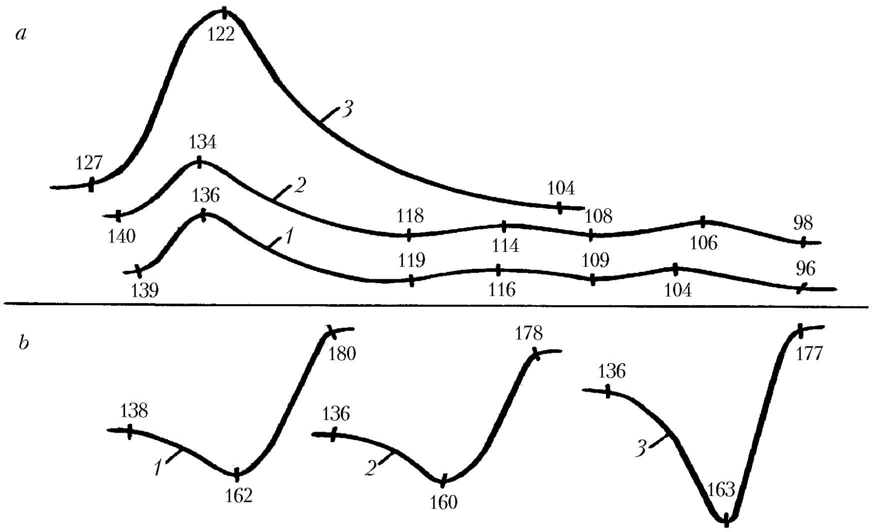

Fig. 5. Thermograms of crystallization (a) and melting (b) of PP/CPA/sodium oleate blends: 1) 20/80 PP/CPA; 2) 20/80/3 PP/CPA/sodium oleate; 3) microfibers from sample No. 2.

Sodium oleate is a polar substance that must show a plasticizing action in a molten blend. However, by virtue of the realization of strong specific interactions (dipole–dipole and ion–dipole bonds), the plasticizing effect is suppressed and the viscosity of the molten blend increases due to its cross-linking [14]. As a consequence, the processes of melting and crystallization in the ternary blend are not different, in practice, from those for the binary blend. For PP microfibers obtained from the extrudates of blend No. 2, we have a sharp increase in the temperatures and heats of phase transitions, which, as has already been discussed, is related to the improvement of the crystalline structure of PP in the process of extraction of CPA.

Noteworthy is the appearance of additional peaks on the thermograms of crystallization of binary and ternary blends at temperatures of 104 and 106^oC (Fig. 5). As has already been discussed, this is due to the disperse state of PP in the blend (PP forms microfibers).

A composite film from a 20/80/3 PP/CPA/sodium oleate blend is formed by repeated extrusion of the blend obtained on LGP-25. A substantial additional dispersion of PP in the form of microfibers occurs, which results in a sharp decrease in the heats of phase transitions and the appearance of an additional peak of crystallization of PP at 103^oC. The increase in T_{cr} of CPA (from 134 to 139^oC) and in Q_{cr} (from $55{\cdot}10^3$ to $66{\cdot}10^3$ J/kg) is related to the enhancement of nucleation in the molten CPA due to the presence of sodium oleate. Thermoorientational extrusion of the composite film leads to an increase in the crystallization temperatures of PP and in Q_{cr} and DC_{cr} (Table 2).

We should emphasize the generality of the regularities of phase transitions for PP microfibers obtained after the extraction of CPA from the extrudates of the blends (sample No. 3, Table 2) and PP microfibers in the form of a filter layer (sample Nos. 6 and 8). In all three cases, we have an improvement in the PP crystalline structure in the process of long extraction at the boiling point of ethyl alcohol, which is expressed in the growth in Q_m of the blend and in T_{cr}, Q_{cr}, and DC_{cr} of PP.

Influence of Sodium-Oleate Additions on the Supermolecular Structure of PP/CPA Blends. By the x-ray method we investigated the structure of the extrudates of a PP/CPA blend of composition 20/80 and a PP/CPA/sodium oleate blend of composition 20/80/3. The results (Fig. 6) confirm the substantial influence of the addition of the salt on the processes of crystallization of the polymers of the PP/CPA blends. This is manifested as a sharp decrease in X_{cr} (to 45%), the splitting of the maximum at $2\theta = 16^o52'$, and the appearance of an additional (weak) maximum at $2\theta = 25^o30'$. Splitting of the bands is absent on the diffractograms of the binary blend, and there is no maximum at $2\theta = 25^o30'$. The decrease in X_{cr} is related to the disperse state of PP in the blend. It is well known that the lower the content of the fiberizing component in the blend, the higher the degree of its dispersion. Sodium oleate strongly cross-links the molten CPA, thus accelerating nucleation and changing the processes of crystallization of CPA and PP.

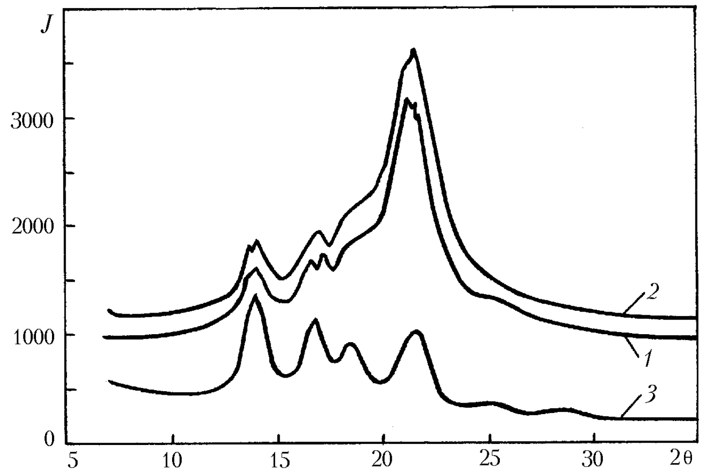

Fig. 6. Diffractograms of scattering of x rays: 1) 20/80/3 PP/CPA/sodium oleate; 2) 20/80 PP/CPA; 3) microfibers after the extraction of CPA from sample No. 1.

It was of interest to evaluate the crystalline structure of a composite film (intermediate product for production of a filter material (FM)) and a finished FM. For this purpose we recorded the diffractograms of a film of 20/80/3 PP/CPA/sodium oleate composition. In the composite film, PP is in the form of very fine microfibers; most of it forms the transition layer. Therefore, X_{cr} of the blend in the film amounts to only 22%; a number of crystal reflexes are absent on the diffractograms. The same regularities have been confirmed by the data of the differential thermal analysis. Four basic reflexes of PP are recorded on the FM diffractograms, but splitting is absent and the intensity of the peaks is lower than that of the starting PP. The same regularities also hold for an FM film from a 50/50 PP/CPA composition with sodium oleate. Thus, the data of the differential thermal analysis and the x-ray data characterizing the crystalline structure of PP/CPA blends and its change under the influence of EVAC and sodium-oleate additions are in good agreement and confirm the change in phase transitions in the ternary blends as compared to the binary blends.

NOTATION

J, number of x-ray pulses; Δm, mass loss in thermogravimetric analysis; Q_{cr}, crystallization heat; Q_m, melting heat; S_a, area occupied by the amorphous reflexes on the diffraction curve; S_{cr}, area occupied by the crystal reflexes on the diffraction curve; T, temperature; ΔT, temperature interval; X_{cr}, degree of crystallinity; θ, angular position of diffraction maxima; τ, shear stress. Subscripts: a, amorphous; cr, crystallization; m, melting.

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