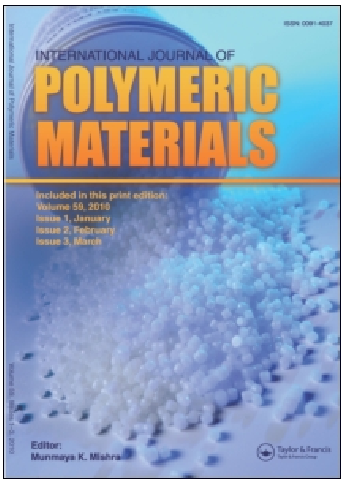

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G. V. Vinogradov[a]; N. P. Krasnikova[a]; V. E. Dreval[a]; E. V. Kotova[a]; E. P. Plotnikova[a]; Z. Pelzbauer[b]
[a] Institute of Petrochemical Synthesis, Moscow GSP-1, USSR [b] Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, Prague, 6, Czechoslovakia

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Effect of Rheological Properties of Compounds on Fiber Formation in Mixtures of Incompatible Polymers†

G. V. VINOGRADOV, N. P. KRASNIKOVA, V. E. DREVAL,
E. V. KOTOVA and E. P. PLOTNIKOVA
Institute of Petrochemical Synthesis, 117912, Moscow GSP-1, (U.S.S.R.)

and

Z. PELZBAUER
Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 16206, Prague, 6 (Czechoslovakia)

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Mixtures of linear polyethylene with polystyrene were used in studying the fiber formation pattern of mixtures of incompatible polymers whose melts were forced through dies. Variables included the viscosity of the components, mixture preparation conditions, and shear stress. It has been established that the process of fiber formation occurs within a definite range of shear stresses, dependent on the viscosity ratio of the fiber-forming polymer and the polymer serving as the dispersion medium. When this ratio is equal to or less than unity, mixtures containing the fiber-forming component in the form of a finely dispersed phase yields continuous fibers several micrometers in diameter within a broad range of shear stresses. At greater values of the component viscosity ratio (tens of units), no fiber formation takes place in the mixtures.

1. INTRODUCTION

Methods of formation of polymer materials with a predetermined structure based on mixtures of incompatible polymers are finding extensive application at present. For example, crystallizable polymers present in an amount of 20 to 40% in a mixture with amorphous polymers form from its melt (forced through capillaries or narrow slots) ultrafine fibers having a diameter of several to tens of micrometers.[1–7] These fibers solidifying when the melt of the mixture is

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cooled down to a level below the crystallization and glass transition temperatures of the components may, for example, perform the function of a reinforcing additive or be used as filtering materials after the polymeric matrix has been removed.

The formation of ultrafine fibers in deformed polymer mixtures is determined by a number of factors: species of the components, mixture composition, fineness of the fiber-forming component, and properties of the interface (particularly, the surface tension at the interphase and the presence of surface-active compounds at the latter). Two of the rheological characteristics are essential: (1) viscosity ratio of both phases, and (2) the shear stress at which fibers are formed. As far as the viscosity ratio is concerned, according to published data,[8–10] equality of the viscosities of both components provides for optimal conditions. Mixtures of linear polyethylene with polystyrene have been studied most extensively. Van Oene has shown,[8] for example, that drops become fibers when the viscosities of both phases are close in value. According to Starita,[9] the less the difference in viscosities, the finer the fibers. The same conclusion has been drawn by Kuleznev[6] with collaborators. The quantitative relations involved in the fiber formation process, namely, diameter distribution of fibers, were presented for the first time by Tsebrenko *et al.*[11] who also stressed the importance of equality of the mixture component viscosities.

This work is concerned with conditions of fiber formation in mixtures of polyethylene and polystyrene in an extremely broad range of viscosity variations, as well as with the effect of fineness of the mixtures on fiber formation. Mixtures of different compositions were studied at shear stresses (τ) ranging from *c.* 10^1 to *c.* 2×10^5 Pa. In these studies, the upper limit of the stress values was determined by the effect of elastic turbulence responsible for the jet losing its stability.

2. EXPERIMENTAL

In the experiment, use was made of three samples of linear polyethylene (PE) and three polystyrene (PS) samples having different molecular weights (Table I). PE contained less than 0.5 CH_3 groups per 1000 carbon atoms in the chain; its degree of polydispersity was $M_w/M_n \approx 2$. PS-1 was a commercial-grade polystyrene produced by emulsion polymerization with a wide molecular weight distribution. PS-2 and PS-3 had been synthesized with a butyllithium catalyst and had $M_w/M_n = 1.7$. Mixtures containing 50, 30, 10, and 1% by weight of PE were examined. In what follows, the results of studying the rheological properties and morphology of mixtures containing 30% by weight of PE are presented, unless otherwise specified.

The polymer mixtures were prepared in different ways: weighed amounts of

TABLE I

Characteristics of polymers

Polymer	Designation	Molecular weight[b]	Average particle size, m km
Polyethylene	PE-1	$1.3 \cdot 10^5$	33
Polyethylene	PE-2	$6.0 \cdot 10^4$	28
Polyethylene	PE-3	$2.9 \cdot 10^4$	26
Polystyrene	PS-1	$4.5 \cdot 10^5$	12
Polystyrene	PS-2[a]	$1.4 \cdot 10^5$	—
Polystyrene	PS-3[a]	$6.6 \cdot 10^4$	—

[a] The PS-2 and PS-3 samples were received from the Voronezh Branch of the All-Union Scientific Research Institute of Synthetic Rubber.

[b] The molecular weight was determined from the intrinsic viscosity value (in decalin at 135°C for PE and in toluene at 25°C for PS).

PE and PS powders were ground in a four-fold amount of ethyl alcohol which was then evaporated at 85°C (method A). A weighed amount of powdered PE was mixed with a predetermined volume of a 20% solution of PS in methylethyl ketone with subsequent evaporation of the solvent at 90°C (method B). The mixture prepared by method A was passed twice at 180°C and 20 rpm through a laboratory extruder with a screw $3 \cdot 10^{-2}$ m in diameter and a head $3 \cdot 10^{-3}$ m in diameter and $1.8 \cdot 10^{-2}$ m long (method C). The mixture of PE and PS prepared by method A was forced twice through a capillary viscometer at 180°C (method D). The above methods have made it possible to prepare mixtures with different fineness of the components.

The viscosities of the PE, PS melts and their mixtures in the range of shear stresses $\tau = (10^1 - 2 \cdot 10^4)$ Pa and shear rates $\dot{\gamma} = (10^{-4} - 10^0)$ s^{-1} were determined with the aid of a rotary viscometer,[12] while in the ranges $\tau = (10^3 - 2 \cdot 10^5)$ Pa and $\dot{\gamma} = 3(10^{-2} - 10^2)$ s^{-1} they were determined by means of a constant-pressure capillary viscometer.[13] The capillary had diameter $d = 1.052 \cdot 10^{-3}$ m and length $l = 4.66 \cdot 10^{-2}$ m. All experiments using the rotary and capillary viscometers were carried out at 180°C, and the mixture of PE and PS flowing from the capillary was cooled in air. Additional experiments conducted by the method of two capillaries have shown that for the systems under examination the end corrections are equal to 6 to 10 l/d. The elastic properties of the polymers, characterized by high-elastic modulus G_e at steady deformation, were determined in the range $\tau = 10^4 - 10^5$ Pa from swelling of the extrudate from the capillary viscometer.[14,15] In this case, the extrudates from the capillary were allowed to relax at 170°C (the extrudate of PE-3 was allowed to relax at 145°C).

The morphology of the mixtures and their extrudates from the capillary viscometer was studied by different methods:

1) Extrudates of the mixtures were soaked in methyl-ethyl ketone in a Soxhlet apparatus for 24 hours to remove the polystyrene, then examined and photographed by an MBI-15 microscope (in polarized or partially polarized light) or by a JSM 35 scanning electron microscope. The average fiber diameter was determined from the negatives to within ±5% at a confidence coefficient of 0.95;

2) Sections were cut from the extrudates in different directions at room temperature, using an LKB 8800 ultramicrotome, and photographed in polarized light by means of an "Opton" microscope;

3) Tranverse chips were taken from the extrudates at room or higher temperatures not exceeding the glass transition temperature of PS. PS underwent brittle failure, while PE exhibited induced high-elastic strains, which permitted the phases to be identified. The chip surfaces were examined by means of a JSM 35 scanning electron microscope.

3. EXPERIMENTAL RESULTS

3.1. Rheological properties of polymer melts and their mixtures

Figure 1a shows the viscosities of PE and PS versus shear stress. It can be seen that all samples (with the exception of PS-3) exhibit pronounced non-Newtonian flow. When the molecular weight of PS and PE is changed, the viscosity varies by two to three orders of magnitude. Unlike viscosity, the high-elastic moduli of the examined polymers are very close and increase with the shear stress (Figure 1b).

Shown in Figure 2 are the viscosities of the PE and PS mixture melts versus shear stress. All mixtures are seen to exhibit non-Newtonian flow which becomes more pronounced with increasing molecular weight of the PE added to PS. The mixture viscosity increases in the same direction. The manner in which the mixture is prepared seems to have little bearing on viscosity (PE-2/PS-1 mixtures). The same applies to mixtures of a different composition (1, 10 and 50% PE).

At high values of τ marked by an arrow near the abscissa the shape and surface of the extrudate are distorted, the distortion increasing with shear stress and being caused by the mixture approaching high elastic turbulent flow. The stress values are in the neighborhood of 10^5 Pa, just as in the case of other polymer systems.[16]

Normally, the flow of single-phase polymers is represented as a plot of η/η_0 versus $\eta_0\dot{\gamma}$, where η_0 is the initial viscosity.[17] Figure 3 shows that data for PE and PS mixtures studied. In cases where the Newtonian flow of the mixtures was not attained, the values of η_0 were determined from the data of Figure 2, by extrapolating the log $\eta-\tau$ curve to $\tau = 0$.[17] As can be inferred from Figure 3,

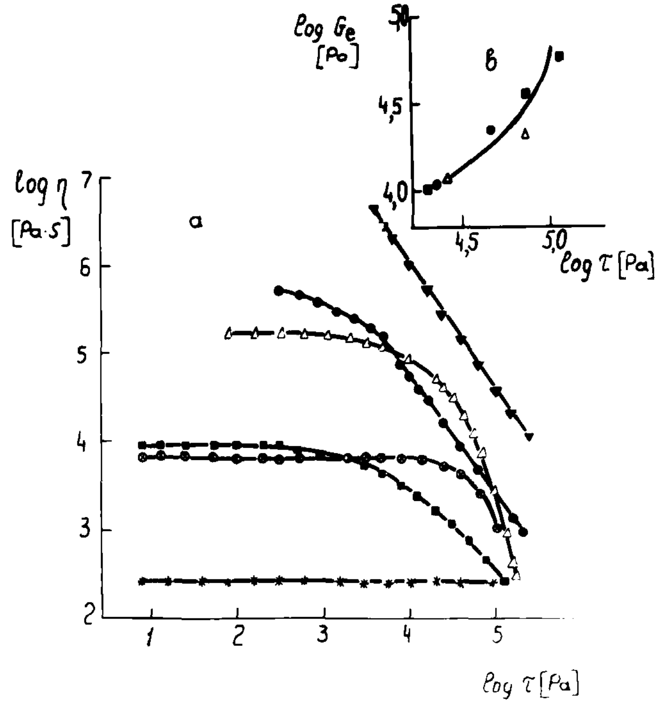

FIGURE 1 Viscosity (a) and high-elastic modulus (b) of the examined polymers versus shear stress: ▼, PE-1; ●, PE-2; ■, PE-3; △, PS-1; ⊗, PS-2; *, PS-3.

in the case of one PS being used with different PE, the data obtained are describable in invariant terms regardless of the PE concentration. For example, the values of $\eta/\eta_0 - \eta_0\dot{\gamma}$ for mixtures based on PS-1 are similar (lower curve). As one goes to mixtures based on PS-2 and PS-3, the dependence is described by the upper curve. This seems to stem from the narrower MWD of these PS samples as opposed to PS-1. These data suggest a possibility of invariant description of the flow of melts of polymeric two-phase mixtures, proceeding from the assumption of a characteristic system relaxation time, which underlies the method under consideration.[17]

3.2. Morphology of mixture extrudates

Morphological studies of the extrudates indicate that at low shear stresses typical of each examined mixture PE is present in the PS matrix in the form of particles. An increase in τ gives rise to PE fibers of limited length and having a variable cross-section over the fiber length ("imperfect fibers"). The photomicrograph of Figure 4a was taken in the scanning electron microscope after

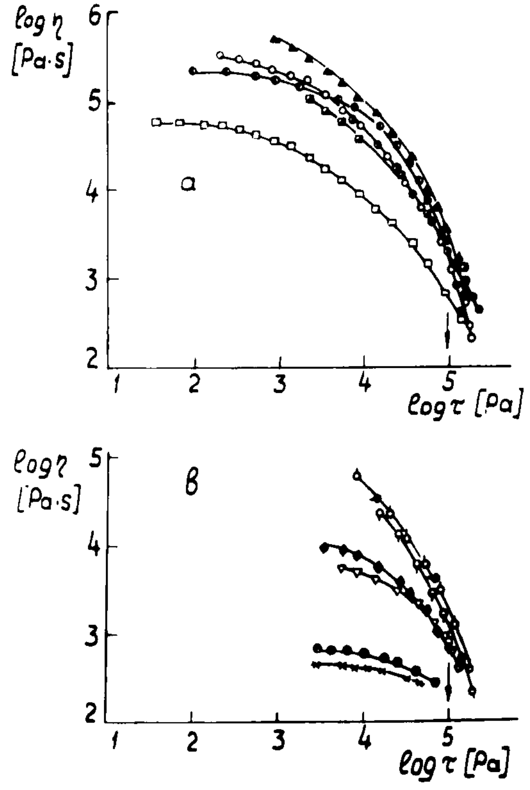

FIGURE 2 Viscosity of PE and PS mixtures versus shear stress (a) prepared by method A: ▲, PE-1/PS-1; ○, PE-2/PS-2; ◑, 50% PE-2/PS-1; ◐, 1% PE-2/PS-1; □, PE-3/PS-1; ◪, 10% PE-3/PS-1. (b) prepared by methods B, C, and D†: ȯ, PE-2/PS-1 (B); -●, PE-2/PS-1 (D); ♀, PE-2/PS-1 (C); ◆, PE-3/PS-2 (B); ▽, PE-2/PS-2 (B); ⊗, PE-2/PS-3 (B); *, PE-3/PS-3 (B).

PS was washed away from the extrudate of the PE-3/PS-1 mixture. Individual PE particles are visible along with PE fibers.

It has been established that for each examined mixture (except for PE-2/PS-3) there is a relatively narrow range of shear stresses, covering 0.1 to 0.2 of an order of magnitude and characterized by an average "critical" value τ_{cr} above which PE fibers of virtually unlimited length are formed in the extrudate. In this case, examination of extrudate sections $(2\text{–}3)\cdot 10^{-3}$ m long containing thousands of fibers, failed to reveal fiber ends. It should be emphasized that these fibers have an invariable cross-section over the entire length ("perfect fibers"). This can be seen from Figure 4b representing the extrudate of the PE-3/PS-3 mixture after removal of PS. A similar picture is also observed at lower

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† Here and in Figures 3 and 5 the mixture preparation method (A, B, C, or D) is given in brackets.

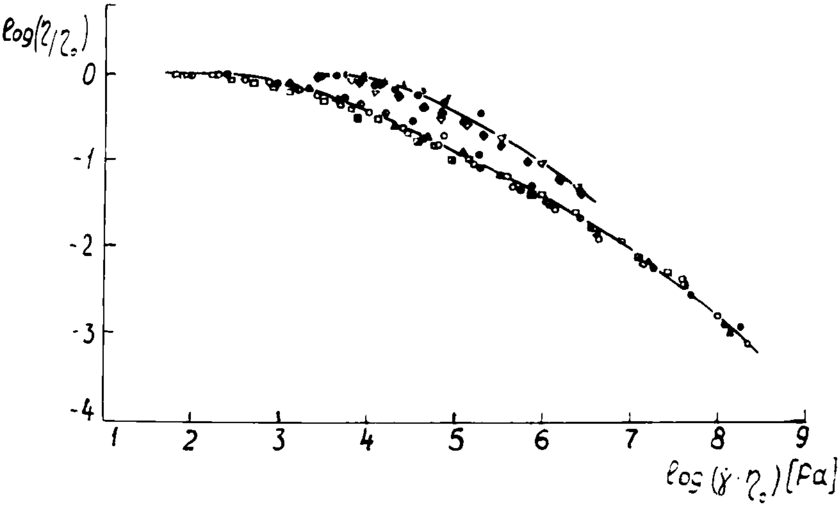


FIGURE 3 $\log(\eta/\eta_0)$ versus $\log \eta_0\dot\gamma$ of the examined mixtures: ▲, PE-1/PS-1 (A); ○, PE-2/PS-1 (A); □, PE-3/PS-1 (A); ◆, PE-3/PS-2 (B); ▽, PE-2/PS-2 (B); ⊗, PE-2/PS-3 (B); *, PE-3/PS-3 (B); ◑, 50% PE-2/PS-1 (A); ◐, 1% PE-2/PS-1 (A); ◪, 10% PE-3/PS-1 (A).

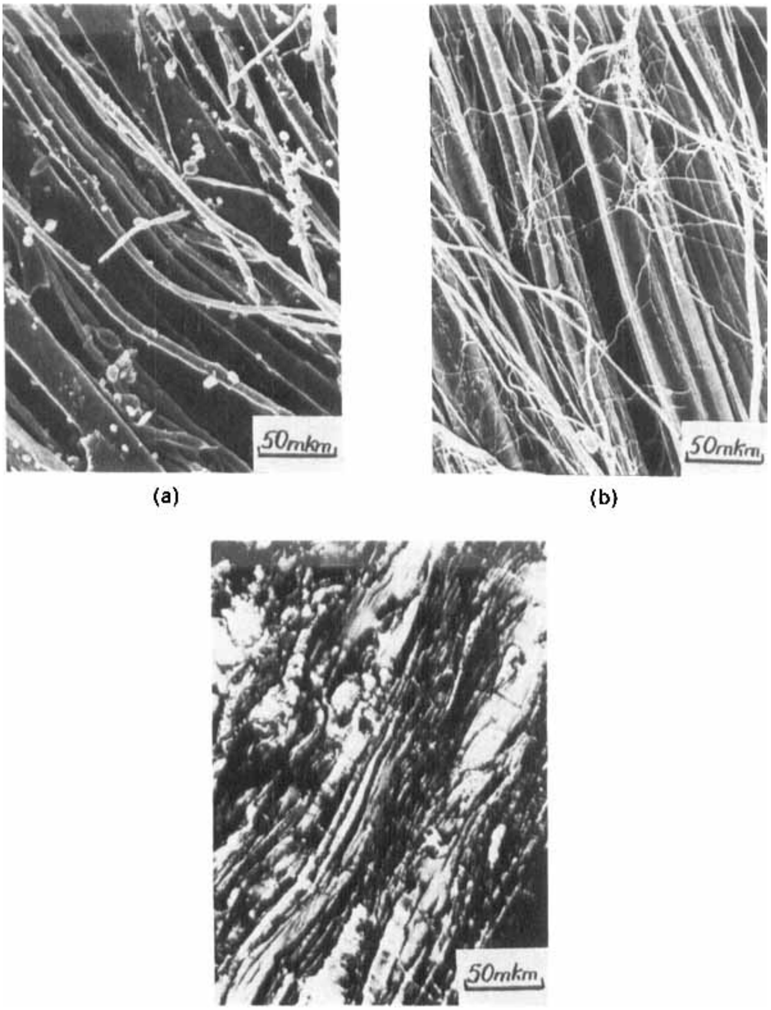
PE contents in the mixture. For example, we could detect formation of continuous PE fibers when the PE content in the mixture was 10% (PE-3/PS-1) and 1% (PE-2/PS-1).

In most PE and PS mixtures a limiting shear stress (τ_{lim}) was found, above which fiber formation is disturbed in the mixtures because of their approaching high elastic turbulent flow. This is manifest from the photomicrograph of a longitudinal section of the extrudate of PE-3/PS-1, taken in polarized light (Figure 4c), where the light areas indicate the PE phase. The value of τ_{lim} varies somewhat from one mixture to another and lies within the range $\log \tau_{\text{lim}} = (5.0 \pm 0.10)$ Pa, which is close to the stresses at which the surface and shape of the extrudate are distorted.

4. DISCUSSION OF THE RESULTS

Although the formation of PE fibers is associated with deformation and coalescence of its particles in a mixture with PS, as the mixture is being pressed through a capillary, the value of the PE viscosity does not play a decisive role in the possible fiber formation. Indeed, PE-2 does not form fibers in a mixture with the low-viscosity PS-3 practically at any shear stress, but forms fibers in a sufficiently wide range of τ in a mixture with the high-viscosity PS-1.

According to Taylor,[18] the deformability of a Newtonian liquid drop in a Newtonian medium is determined by two parameters: the viscosity ratio of the drop to the medium and the ratio of the shear stress (unvariable in transition from one phase to the other) to interfacial tension. The behavior of polymer mixtures is complicated by their exhibiting non-Newtonian flow and high elasticity.[8] Some investigators[19] contend that in this case viscosity does play a significant role, too, since it varies to a much greater extent than high elasticity.



(a) (b)

(c)

FIGURE 4 Photomicrograph of PE-3 fibers formed in a mixture with PS-1 at $\tau = 1.0 \cdot 10^3$ Pa (a) and in a mixture with PS-1 at $\tau = 3.2 \cdot 10^4$ Pa (b); longitudinal section from the PE-3/PS-1 extrudate at $\tau = 1.7 \cdot 10^5$ Pa (c).

This is particularly true in the case under consideration, where the high elasticities of the polymers (determined from the values of G_e) are similar (Figure 1b).

Figure 5 represents the viscosity ratio of PE to PS (η_{PE}/η_{PS}) versus shear stress. The Roman numerals stand for the intervals in which the extrudates display different morphologies (I, no fibers; II, formation of imperfect PE fibers; III, formation of perfect PE fibers; IV, disintegration of fibers). It can be seen that there is a certain relationship between η_{PE}/η_{PS} and the width of the τ interval in which fiber formation is pronounced, that is in the perfect PE fiber formation region (III). This relationship is such that the minimum values of τ corresponding to region III, for all examined mixtures are associated with the following relation of τ_{cr} to η_{PE}/η_{PS}:

$$\tau_{cr} = 8 \cdot 10^4 (\eta_{PE}/\eta_{PS})^{0.7} \text{ Pa} \tag{1}$$

Equation (1) is represented by the thick line in Figure 5. The maximum values of τ in the perfect fiber formation region, indicated by the dashed line in Figure 5, correspond to the relationship between τ_{lim} and η_{PE}/η_{PS} associated with transition to unsteady high elastic turbulent flow. Thus, in the case of small values of $\eta_{PE}/\eta_{PS} \approx 2.5 \cdot 10^{-2}$, τ_{cr} is about $2 \cdot 10^3$ Pa and pronounced fiber

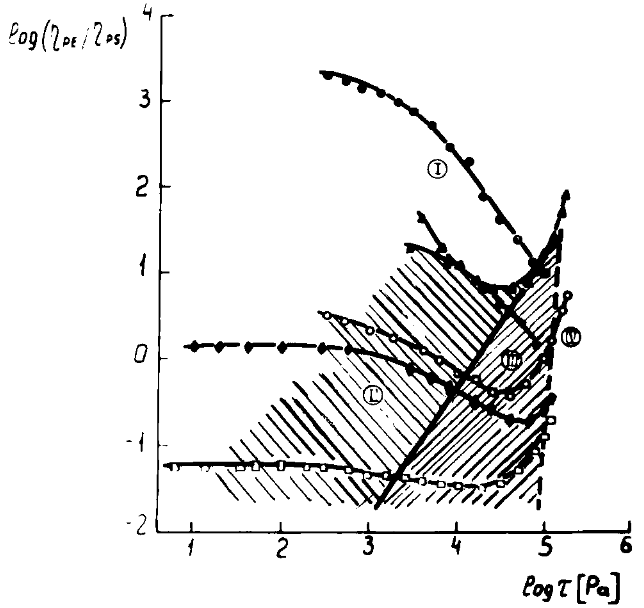


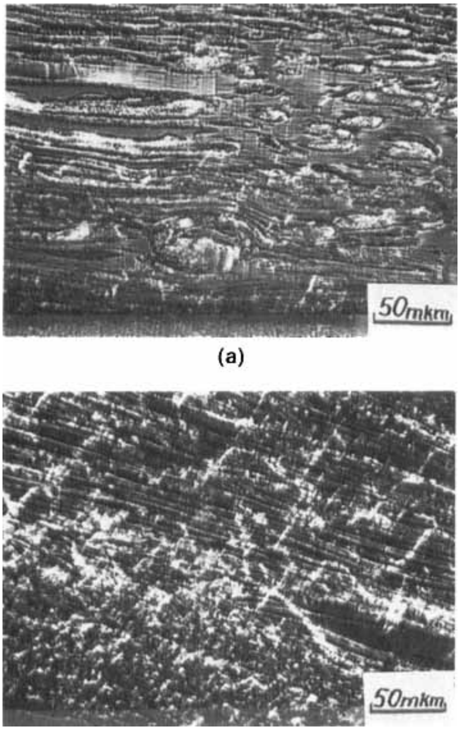
FIGURE 5 η_{PE}/η_{PS} versus shear stress: ⊗, $\eta_{PE\text{-}2}/\eta_{PS\text{-}3}$ (B); ▲, $\eta_{PE\text{-}1}/\eta_{PS\text{-}1}$(A); *, $\eta_{PE\text{-}3}/\eta_{PS\text{-}3}$ (B); ○, $\eta_{PE\text{-}2}/\eta_{PS\text{-}1}$ (A); ◆, $\eta_{PE\text{-}3}/\eta_{PS\text{-}2}$ (B); □, $\eta_{PE\text{-}3}/\eta_{PS\text{-}1}$ (A). Boundaries of the pronounced fiber formation region: thick line, τ_{cr}; dashed line, τ_{lim}. Roman numerals indicate the regions of fiber formation in mixtures: I, no fibers; II, imperfect PE fibers; III, perfect fibers; IV, disintegrated fibers.

formation occurs in a wide range of stresses, covering about two orders of magnitude. The increase in η_{PE}/η_{PS}, as one passes from one pair of components to another, is followed by an increase in τ_{cr} and narrowing of the stress range in which fiber formation is possible. At η_{PE}/η_{PS} 10 to 30, fiber formation in the PE/PS mixture becomes impossible because τ_{cr} approaches the limiting value τ_{lim}. Characteristically, the relationship between τ_{cr} and η_{PE}/η_{PS} does not depend significantly on the way in which the mixture is prepared. Variations in the PE concentration in the mixture do not affect τ_{cr} either. It has been found that the values of τ_{cr} coincide for PE-3/PS-1 mixtures containing 30% and 10% of PE. It should be noted that the lower limit of applicability of Eq. (1) has not been established within the framework of this study. In any event, it must be confined by low viscosities of the components, leading to disintegration of the fibers in the mixture into separate drops while the melt of the mixture flows through the capillary.[10]

Another interesting feature is the fiber formation in the examined mixtures within the region defined by the thick solid and dashed lines, irrespective of whether the value of η_{PE}/η_{PS} increases or decreases as a function of shear stress. This fact indicates that in the region of high values of τ a large contribution to the fiber formation process may be made not by tangential but tensile stresses commensurate with those occurring at the capillary inlet.[2,20]

Analysis of the data obtained suggests that the value of η_{PE}/η_{PS} determines the range of shear stresses, in which imperfect short PE fibers are observed as well (region II). This range extends toward small values of τ with decreasing η_{PE}/η_{PS}. For example, in the case of the PE-1/PS-1 mixture, it covers only about 0.3 of an order of magnitude to the left of the thick line. As far as other mixtures (e.g., PE-3/PS-1) are concerned, the minimum values of τ at which perceptible fiber formation begins are so small that they could not be detected in our experiments.

It has been found that in the range of stresses corresponding to pronounced fiber formation, the extrudate morphology depends to a great extent not only on η_{PE}/η_{PS} but also on the absolute value of viscosity of the components, τ, and uniformity of distribution of the PE particles in the initial mixture. For example, mixing of the high-viscosity PE-1 and PS-1 leads to formation of comparatively thick fibers of widely differing diameters. The lower PE viscosity in the PE-2/PS-1 mixture yields thinner fibers of closer diameters, which becomes evident from comparison of the photomicrographs of longitudinal sections of the extrudates of these mixtures (Figures 6a,b). Similar diameters were also observed in the fibers in the PE-3/PS-1 mixture (the least viscous PE), where the value of τ_{cr} approaches that of τ_{cr} of the PE-1/PS-1 mixture (Figure 5). The increase in the shear stress also results in thinner fibers forming in this mixture. In general, depending on various factors, PE/PS mixtures yield fibers with an average diameter of 2 to 7 m km.



(a)

(b)

FIGURE 6 Longitudinal sections cut from extrudates of PE-1/PS-1 (a) and PE-2/PS-1 (b) mixtures at $\tau = 6.8 \cdot 10^4$ Pa.

As a rule, PE fibers are unevenly distributed across the extrudate: they group into concentric layers, and a PE "jacket" is formed on the extrudate surface. A similar extrudate morphology has been observed in other fiber-forming polymer mixtures.[1–5] This results from the radial migration of the mixture components while the melts are being pressed through the capillary, due to different rheological characteristics of the components and a wider size distribution of their particles. The latter factor seems to be more important. In fact, we used a capillary with a high length-to-diameter ratio, which must contribute to the radial migration of the mixture components.[19] It has been found that there are no significant differences in the morphology of mixtures based on different PE-PS pairs, when the mixtures are prepared by method A. At the same time, additional homogenization of a mixture prepared by method A by pressing it twice through the capillary (method D) leads to a more even distribution of the PE phase across the extrudate. This follows from the photomicrographs of transverse sections of PE-2/PS-1 mixture extrudates

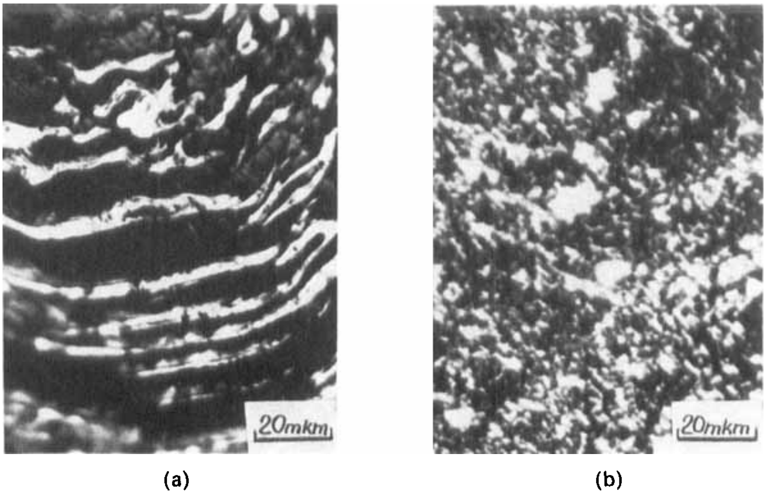
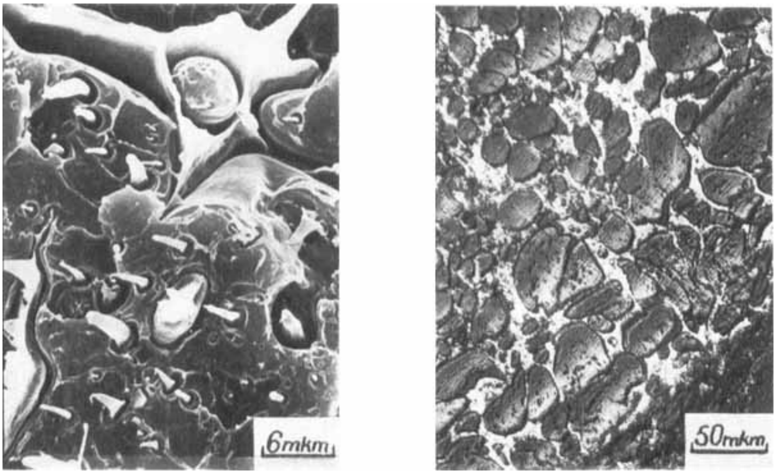


FIGURE 7 Transverse sections cut from extrudates of PE-2/PS-1 mixtures prepared by method A (a) and D (b) at $\tau = 6.8 \cdot 10^4$ Pa.

(Figures 7a,b). A similar phenomenon is observed when the morphologies of mixtures prepared by methods A and C are compared.

It is important to point out that a considerable decrease in the value of η_{PE}/η_{PS} at a given PE content and mixture preparation procedure may lead to a change in the phase state of the mixture, which is true as regards other polymer mixtures as well.[21] As a consequence, changes are observed in the extrudate morphology which becomes highly sensitive also to the amount of applied stress. For example, in the case of the PE-3/PS-1 mixture prepared by method A and characterized by $\eta_{PE}/\eta_{PS} \approx (3\text{–}10) \cdot 10^{-2}$, PE with PS forms mutually penetrating phases when the mixture is heated and subjected to pressure in the viscometer. As a result, along with PE fibers the extrudate also contains its films enveloping the PS domains which extend coaxially with the extrudate and have a diameter of about 8 m km (Figure 8a). Such a morphology of the extrudate may in some cases be preferable to that when the PE phase contains only PE fibers.[7] Then, an increase in shear stress brings about a higher content of films and a lower content of PE fibers in the mixture, and at shear stresses approaching τ_{lim} PE becomes a dispersion medium and PS becomes a dispersed phase. This can be seen from the photomicrograph of a transverse section cut from the extrudate, taken in polarized light, where dark areas correspond to PS (Figure 8b). Obviously, the films can be eliminated by reducing the concentration of the fiber-forming component to such a level where it will form a dispersed phase. This concentration must diminish with η_{PE}/η_{PS} and can be estimated from the literature.[21] It has been established that



(a) (b)

FIGURE 8 Transverse chip from an extrudate of PE-3/PS-1 mixture (a) at $\tau = 6.8 \cdot 10^4$ Pa and transverse section (b) at $\tau = 1.0 \cdot 10^5$ Pa.

as one goes from a 30% to 10% PE-3/PS-1 mixture in which the formation of mutually penetrating phases is less probable, the extrudate with PS washed away almost completely consists of PE fibers.

Thus, fiber formation in polymer mixtures whose melts are pressed through capillaries or narrow slots is a complex process determined by a combination of many factors sometimes producing opposite effects. In this case, of primary importance is the viscosity ratio of the mixture components and the state of the mixture before its melt is pressed through the capillary. High viscosity ratios of the fiber-forming and second components (more than 10–30 units) render fiber formation impossible. If this ratio is reduced to fractions of a unit, fibers may form in a broad range of shear stresses beginning with relatively low values of the latter. Preparation of homogeneous polymer mixtures with the fiber-forming component clearly forming the dispersed phase allows producing extrudates containing primarily fibers of the dispersed component. Such mixtures are more easily obtained if the difference in viscosity between the components is minimum. A considerable decrease in the viscosity of the fiber-forming component as compared to that of the second component may result in a mixture consisting of mutually penetrating phases of both polymers. A generalized plot of η/η_0 versus $\eta_0\dot{\gamma}$ indicates that changes in the size and shape of the PE particles within several to tens of micrometers affect both insignificantly the viscosity of mixtures. The phase state of the mixture seems to be the decisive factor.

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