

Structure and Mechanical Properties of Heterogeneous Polymer Blends

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SYNOPSIS

Material response under uniaxial stress has been reported for heterogeneous systems: low density poly(ethylene)-poly(styrene). The systems differed in a component ratio and in a content of the macromolecular modifier type compatibilizer. Tensile properties have been determined for the systems in a molten state (473 K) and below T_g as a function of elongation rate. The modifier which diminishes the interfacial tension is advantageous for mechanical properties of the system—both in the melt and the solid state. The differences in mechanical characteristics have been related to a specific phase structure of polymer systems. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

Polymer blends have recently comprised ca. 20% of the overall plastics production. The high dynamics of 10% annual increase lasting already over 10 years allows the prediction that in the near future most products made of plastics will be manufactured from multicomponent polymer systems.¹⁻³ Among the reasons for such high interest in polymer blends at least the most important should be mentioned:

- properties adjustment is the needs by a proper choice of the particular kind of components and their ratio in the blends;
- modelling of the phase structure of heterogeneous systems has recently become accessible;
- better economy (lower costs and shorter time-scale) of the new polymeric material designed through blending rather than searching of new monomers for the new (co)polymers synthesis;
- reduction of the price of the blends based on "engineering polymers"; and
- ecological aspects (utilization of the plastic wastes by blending).

The above-mentioned advantages can be of practical worth only in the case of thorough knowledge of the

polymer mixing process theory and of the proper application of empirical rules which determine the interrelations of melt rheology, the phase structure formation, and the end-use properties of the finished products.³⁻¹³

Most polymer blends are heterogeneous because of the thermodynamically substantiated immiscibility of the components. These systems are usually of poor mechanical strength resulting from weak interfacial adhesion. For example, the relationships between the empirical Young modulus or tensile strength and the component ratio represent a negative deviation (NDB) from the theoretically predicted values. Significant differences in the mechanical properties of polymer blends of identical component ratios suggest an important role of the phase structure mode, size of the morphology elements, and interfacial characteristics.

Among the factors influencing the morphology of polymer systems, the most significant are^{3,7-20}

- composition;
- viscosity ratio of the molten components;
- melt elasticity ratio;
- interfacial tension; and
- processing history.

The usually designed morphology comprises microdispersions of the minor phase component in the matrix polymer, because of favorable properties of

such systems. By increasing the dispersed phase content in the system, one can enlarge the size of the domains until the phase inversion occurs. Under specific conditions, the characteristic interpenetrating structure is formed.^{15,16,21,22} The domain size is dependent also on the interfacial tension, i.e., the size usually decreases as the interfacial tension diminishes. Such effects can be attained by the addition of macromolecular modifiers of compatibilizer type.²³⁻³¹ Usually these are block- or grafted copolymers which contain blocks of a high affinity to the blended polymers. Such units commonly form an interfacial layer which enhances the adhesion between components. Such structural modification involves differences in mechanical properties. Throughout this paper the correlation of the compatibilizer influence on the tensile strength of polymer systems, both in the molten and solid state, was studied.

EXPERIMENTAL

Materials

The polymers used were: low density poly(ethylene) (LDPE) and poly(styrene) (PS) from BP Chemicals and partially hydrogenated di-block copolymer (styrene-*b*-isoprene) Kraton G1701 (SEBS) from Shell Chemicals. These polymers were used within the IUPAC Working Party IV.2.1 by participants in the research project "Melt Rheology and Comitant Morphology of Polyblends," performed during 1985-1992 under the coordination of A. P. Plochocki.³²

Samples Preparation

Multicomponent polymer systems composed of various volume content (ϕ) of particular polymers with compatibilizer (alloys are coded A in the Tables) or without it (blends) were prepared within the framework of the IUPAC research project, using the two-screw compounder type ZSK 30 from Werner Pflöderer.

The granules were used for rheological measurements and for studying the melt elongational strength. Tensile strength at room temperature was examined using the dog-bone-shaped tensile bars produced by injection molding.

Melt Rheology

Rheological characteristics were obtained by using the capillary rheometer type MCR 3210 mounted

into the Instron 1122 machine. The measurements were performed at a temperature of 473 K using a capillary $L/D = 40$. Results were computed in a routine manner receiving the relationship of viscosity (η) versus shear stress (σ_{12}), the exponent n of the Ostwald deWaele equation, and the exponent m of the elasticity equation.³³ The viscoelastic characteristics of the polymers, polymer blends, and alloys are presented in Table I.

Rheotensometry

Mechanical strength in the molten state³⁴⁻³⁶ was examined using the Gottfert Rheotens at 473 K. The elongation was performed by the cogged wheels rotated with an acceleration of 1.5 cm s^{-2} . Representative diagram: force versus extension has been presented in Figure 1. Table II includes the torque values (M_d) of the extrusimeter upon the extrusion of particular compositions, as well as the breaking force (F) and elongation (Δl) at the break of extrudate.

Mechanical Strength at the Solid State

The uniaxial tension of normalized samples was examined using the FU 100eZ testing machine at room temperature. Cross-head speed was changed within a range $1-100 \text{ mm min}^{-1}$. The tensile modulus of elasticity (E), yield stress (σ_y), tensile strength (σ_r) and the elongation at break (ϵ) were calculated. Results are presented in Table III.

Microscopy

The damage region of the samples was inspected both in parallel and in perpendicular direction to the elongation axis. For in-axis observations the

Table I Viscoelastic Characteristics of Polymers, Polymer Blends (Sample Code Expressed with PS Content, Wt (%) and Polymer Alloys (Additionally Code A)

Sample Code	$n \times 1000$	$\eta, \text{ Pa s}$	$m \times 1000$
LDPE	453	1,780	625
15	472	1,320	555
15A	498	1,390	726
33	367	970	476
33A	457	1,340	543
67	356	780	476
67A	302	790	—
PS	307	590	636
SEBS	370	2,270	194

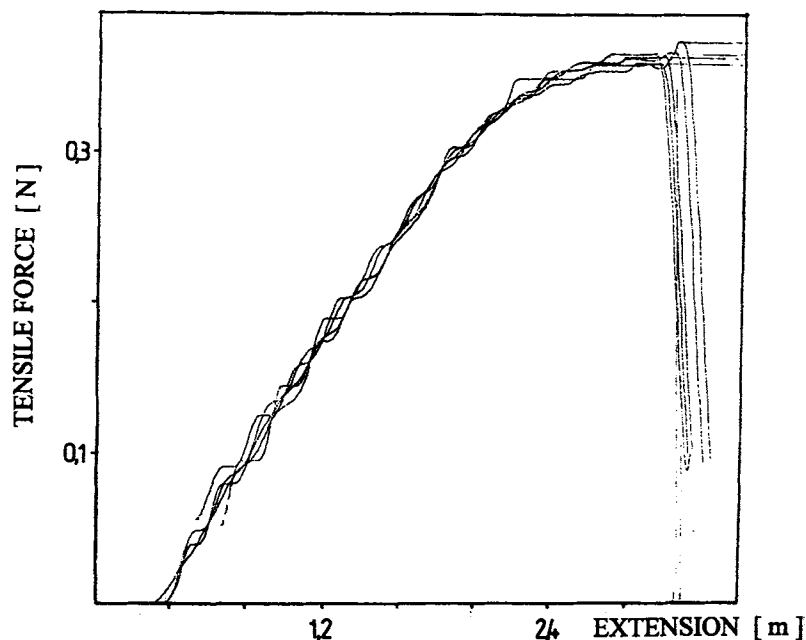


Figure 1 Diagram of uniaxial stretching of LDPE melt.

cold-fractured (liquid N_2) samples were used. The surfaces were vacuum-coated with the Au-Pd layer and then observed using the JEOL JSM840 scanning electron microscope (SEM). The SEM-micrographs are presented in Figures 2-7.

RESULTS AND DISCUSSION

Melt Flow

The rheological characteristics of the polymer systems in the molten state are presented in Table I. Viscosity versus shear rate dependence, both for blends and alloys, was non-Newtonian, and similarly for the homopolymers LDPE and PS. The melt viscosity of multicomponent systems has been comprised between the values measured for poly(ethylene) and poly(styrene). The highest melt viscosity has been found for the block copolymer SEBS, whereas PS showed the lowest viscosity.

Flow instabilities of the "melt fracture" type were observed at high shear rates. As a rule, this feature revealed for blends and for a virgin PS at the shear rates of one order of magnitude higher than for the alloys and for LDPE.

Table I comprises the viscoelastic characteristics at the shear stress $\sigma_{12} = 10^2$ kPa. Melt viscosity increase has been noted as a result of the block copolymer addition—the most distinct for the systems LDPE-PS of the weight fraction ratio of 2 : 1. The

elastic behavior of alloys has been more pronounced in comparison to blends of the same ratio of the component polymers. The viscoelastic characteristics suggest enhanced interactions between the components, resulting from the block copolymer addition. Simultaneously, the microheterogeneity level was increased in this system composed of two high-viscosity fluids (LDPE and PS at temperature 473 K). SEBS behaves like emulsifier, indeed.

Melt Stretching

The results of rheotensometry (Figure 1, Table II) show only small differences between the tensile

Table II Rheotensometry Results

Sample Code	Torque M_d , J	Melt Strength F , mN	Melt Elongation at Break Δl , m
LDPE	40.2	361	2.78
15	36.8	371	2.47
15A	40.2	417	6.22
33	36.5	386	2.22
33A	38.3	352	3.64
67	35.3	276	2.80
67A	34.3	362	6.01
PS	38.2	323	7.28
SEBS	14.7	352	1.30

strength of particular systems. Addition of SEBS is favorable for a tensile strength of the melts, excluding the system LDPE/PS 2/1. Much higher differences have been found for the elongation at break, both between the neat polymers and between blends and alloys. The PS strand was extended 2.6 times more than PE under the same conditions. This can be attributed to a different viscosity of the polymer melts (viscosity of PS melt is 3 times higher than that of PE). There exists an interesting relationship between the results of the uniaxial melt elongation and the previously reported "melt fracture" appearance, which can explain the above findings. Melt defects that deteriorate the material strength arise at lower stress in LDPE, therefore poly(ethylene)-dominated systems break at a lower extension ratio.

The melt elongation at break for all blends was similar to that of the LDPE melt. This allows for a supposition that this polymer forms an intermeshed (network) phase structure, according to the percolation model. This tendency was diminished with the addition of the component which decreases the interfacial tension (compatibilizer), resulting in distinctly higher extensibility of the alloys.

Solid State Tensile

Diverse relationships were found as far as the results of tensile tests performed at 293 K and reported in Table III are concerned. Poly(ethylene) exhibits much higher elongation at break and distinctly lower tensile modulus than poly(styrene). This can be related to the semicrystallinity of LDPE and to the much higher brittleness of PS. Essential differences of the neat polymers influence the fracture mode and therefore the tensile results for LDPE/PS blends and alloys.

In the discussion below, we consider the heterogeneous polymer systems as the matrix-fiber composites. In the course of stretching, the following phenomena may take place³⁷⁻⁴⁸:

- detachment of the fiber/matrix connection;
- slippage in this interfacial area;
- pulling-out of fibers and the stress-relaxation in a matrix;
- fracture of a brittle phase; and/or
- fracture of a ductile phase.

The total fracture energy W consists of three components:

$$W = W_f + W_m + \sum W_{fm}^{\alpha} \quad (1)$$

where W_f and W_m are the fracture energy of the fibers and matrix, respectively; and $\sum W_{fm}^{\alpha}$ is the whole spectrum of interactions on the fibers/matrix interface.

After Wu¹³, we correlated the molecular parameters to the fracture modes of LDPE and PS. Semicrystalline LDPE was considered as a composite of crystallites distributed in an amorphous matrix. The matrix behavior is more particularly dominant as the growth of crystallites is disturbed by the second polymer. According to Wu classification, polymers are brittle (tending to craze under stress) or pseudoductile (tending to yield). Crazing starts with the chain breaking, which is related to the entanglement density ν_e . The entanglement density is defined by:

$$\nu_e = \rho_a/M_e \quad (2)$$

where ρ_a is the amorphous phase density and M_e the molecular weight of an entanglement.

On the other hand, yielding is a function of the intrinsic chain flexibility measured by the characteristic ratio of chain C_{∞} :

Table III Tensile Properties Versus Tensile Rate

Sample Code	Crosshead Rate mm/min	Tensile Modulus MPa	Yield Stress MPa	Tensile Stress MPa	Elongation at Break %
LDPE	1	201	14.4	13.8	73
	10	226	15.0	14.2	73
	100	—	16.5	16.0	65
15	1	348	14.8	13.8	38
	10	404	15.7	14.5	38
	100	—	16.8	16.1	34
15A	1	266	15.5	14.7	61
	10	346	15.6	14.9	58
	100	—	16.7	16.1	55
33	1	720	15.5	15.3	3
	10	690	18.7	18.7	3
	100	—	20.3	20.3	4
33A	1	488	15.0	14.1	38
	10	567	15.8	14.8	36
	100	—	15.5	15.3	31
67	1	1137	28.7	28.7	2
	10	1214	29.5	29.5	2
	100	—	27.6	27.6	3
67A	1	1424	29.1	29.1	2
	10	1186	31.1	31.1	2
	100	—	28.2	28.2	3
PS	1	2695	46.5	46.5	3
	10	2205	50.5	50.5	4
	100	—	47.2	47.2	3
SEBS	1	88	1.8	1.4	2

$$C_{\infty} = \lim(R_o^2/nl^2) \quad (3)$$

where R_o^2 is the mean-square end-to-end distance of an unperturbed chain, n is the number of the statistical skeletal units, and l^2 is the mean-square length of a statistical unit.

Wu criteria predict crazing for the polymers having $\nu_e < 0.15$ mmole/cc and $C_{\infty} > 7.5$. Polymers characterized by $\nu_e > 0.15$ mmole/cc and $C_{\infty} < 7.5$ should yield. Respective values for poly(ethylene) are $\nu_e = 0.613$ mmole/cc and $C_{\infty} = 6.8$; whereas for poly(styrene), $\nu_e = 0.056$ mmole/cc and $C_{\infty} = 10.8$. Therefore, we expected crazing as the predominant fracture mechanism of PS and yielding as typical for LDPE. Our anticipation was strengthened by the comparison of the test temperature with relaxation temperatures. Following Wu³⁷ we used thereafter the notations $\{\alpha\}$ and $\{T_{\alpha}\}$ for the primary (glass) transition as well as $\{\beta\}$ and $\{T_{\beta}\}$ for the secondary transition (subglass relaxation related to localized skeletal motions). The $\{\beta\}$ relaxation corresponds to the brittle/ductile transition related to the crazing/yielding behavior under the stress.

The primary relaxation $\{\alpha\}$ which is for amorphous polymers the glass transition, occurs for poly(styrene) at $\{T_{\alpha}\} = T_g = 373$ K, whereas the brittle/ductile relaxation $\{\beta\}$ at $\{T_{\beta}\} = T_b = 363$ K. In poly(ethylene) the glass transition is at 275 K and $\{\beta\}$ relaxation at 165 K. According to Wu,^{13,37} for chains with tetrahedral skeletal bonds, the highest intrinsic flexibility or the "maximum" ductility is attained when $C_{\infty} = 2$ and $\{T_{\beta}\}/\{T_{\alpha}\} = T_b/T_g = 0.3$.

For the chains having $C_{\infty} > 10.5$ there is $\{T_{\beta}\}/\{T_{\alpha}\} = T_b/T_g = 1$. This ratio for poly(ethylene) is 0.60 and for poly(styrene), 0.97. In other words, in LDPE, yielding which arises from the onset of conformational rotation of skeletal bonds under the stress can occur at much lower temperature than that at which the tensile test was performed. Moreover, because the test temperature was above $\{T_{\alpha}\}$, segmental motions (C_{10} – C_{100}), alignments, and mutual sliding of chains, as well as motions in the crystalline domains or at domain boundaries, should be taken into account. To the contrary, in PS the brittle/ductile transition occurs only near the glass transition, therefore brittle behavior (crazing) must be expected at the tensile test temperature.

The analysis of the data presented in Table III shows that the tensile modulus of LDPE-rich systems is dominated by the E-value for poly(ethylene), especially as far as the alloys are concerned. PS-rich systems are characterized by a high tensile modulus, close to that predicted by a linear mixing rule. In

the elastic region of deformation, the chain coils and van der Waals distances are reversibly strained. In poly(ethylene), which is of high entanglement density, the stretching of polymer chains occurs under low stress value, whereas the corresponding strain value is pretty high. Therefore LDPE and LDPE-dominated systems are of low elasticity modulus. To the contrary, the poly(styrene), having one order of magnitude lower ν_e -value is being stretched to the yield point at low strain but with considerable stress. Interestingly, the elasticity modulus of PS is one order of magnitude higher than that of LDPE.

The tensile strength of LDPE/PS systems represents similar correlations; the values are determined by the component dominating the system. Therefore, the predominant mode of fracture (crazing/yielding) should be the same as that of the matrix.

The analysis of the elongation at break versus the content of specific polymers led to interesting

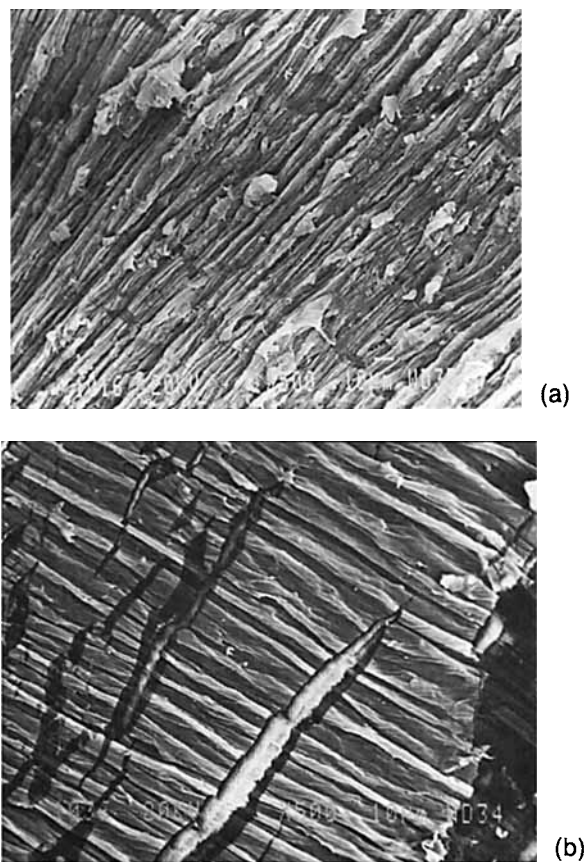


Figure 2 SEM micrographs of LDPE/PS 85/15 blend: failure surface (a) and in-axis cold-fracture in a damage region (b).

findings. The blend containing 15 wt % of PS shows elongation equal to approximately 50% that of LDPE itself. The corresponding ϵ -value for the alloy is equal to that theoretically predicted according to:

$$\epsilon = \varnothing_1 \epsilon_1 + \varnothing_2 \epsilon_2 \quad (4)$$

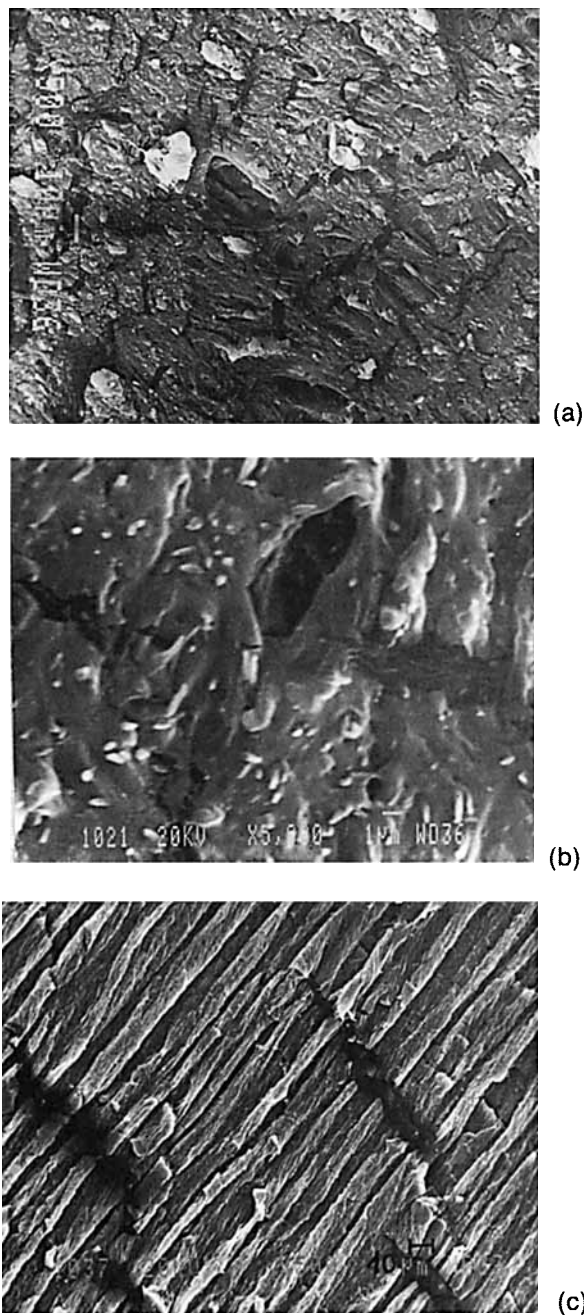


Figure 3 Morphology of LDPE/PS 85/15 alloy: failure surface (a) and (b)—higher magnification. In-axis cold fracture, (c).

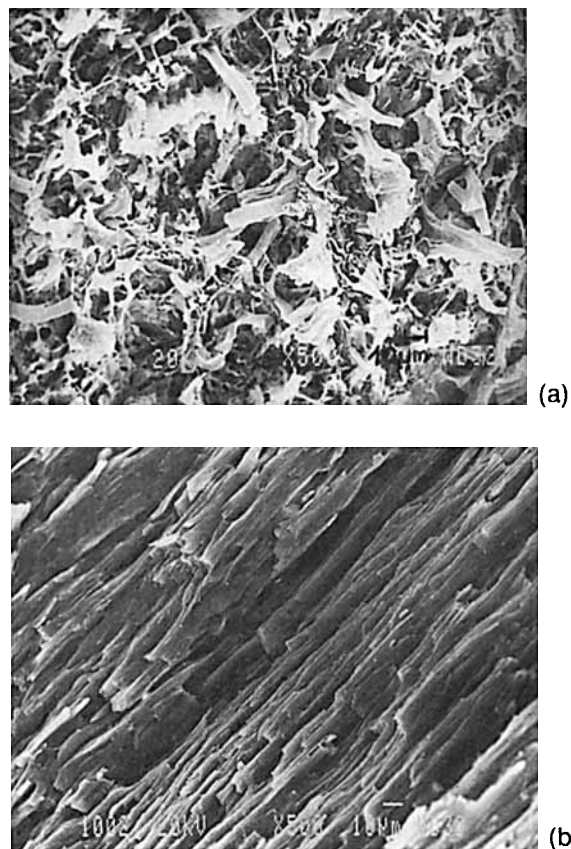


Figure 4 SEM micrographs of LDPE/PS 2 : 1 blend: failure surface (a) and longitudinal cold fracture (b).

where indexes 1 and 2 refer, respectively, to the component polymers.

In turn, the elongation of the blend LDPE/PS 2 : 1 is like that of poly(styrene), whereas the elongation of the system containing an additional 5% of SEBS copolymer is one order of magnitude higher. It has been found that both the blend and alloy dominated by PS exhibit the elongation at break typical of this polymer.

Such characteristic changes of the parameters describing the tensile tests course suggest specific phase structure differences for individual systems. Considering micromechanics and the microstructural and molecular analysis, one can expect an interpenetrating type of the blend and alloy LDPE/PS 1 : 2 and of the blend LDPE/PS 2 : 1. Moreover, similar characteristics of the blend containing 85 wt % of LDPE and the alloy of 67 wt % of LDPE allow us to expect similarities in their morphology. The above expectations have been confronted with the actual phase structure of LDPE/PS systems, observed by means of scanning electron microscopy.

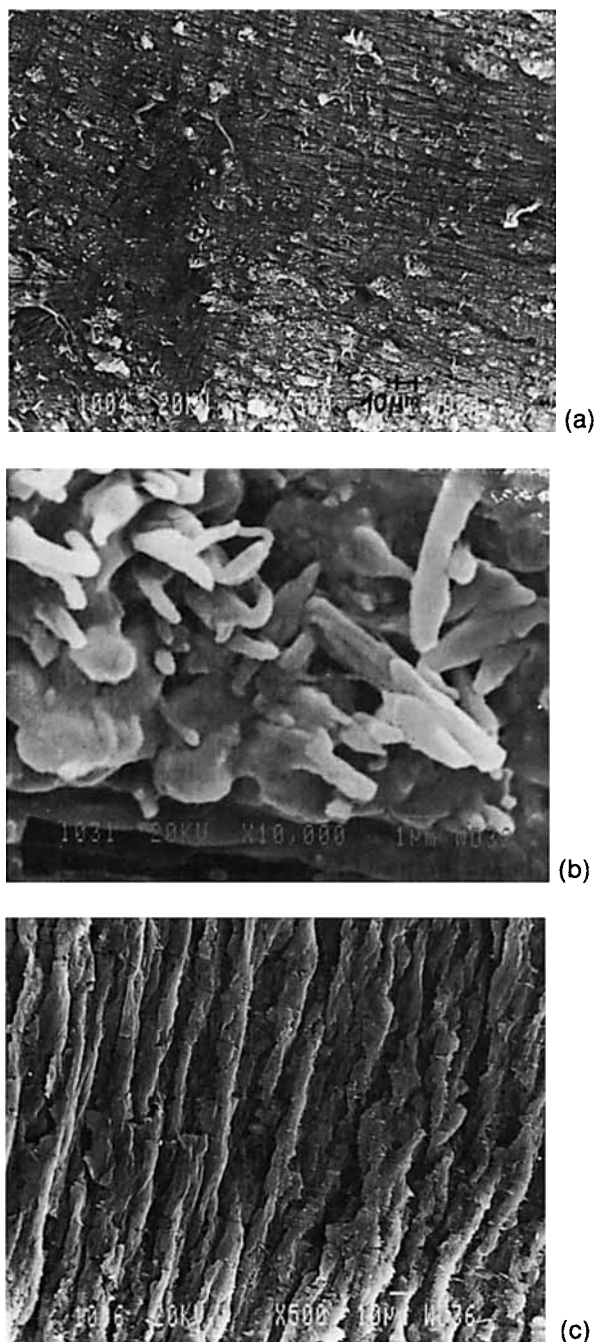


Figure 5 Morphology of LDPE/PS 2 : 1 alloy: failure surface (a) and (b)—higher magnification. In-axis cold fracture, (c).

Morphology

The systems containing 15 wt % of poly(styrene) (Figs. 2 and 3) represent heterogeneous morphology within which characteristic regions of high orientation have been recognized (Figs. 2[b] and 3[c]). We ascribe these regions to poly(ethylene), the flex-

ible phase of which flows under tensile stress. The blend is of stratified morphology, composed with sheets of few μm thickness (Fig. 2[a]). In a failed specimen (Fig. 2[b]), fractures can be observed perpendicular to the stretching axis (“crazes”). The crazes are typical of brittle polymers such as PS. Therefore we suppose that the PS domains are big

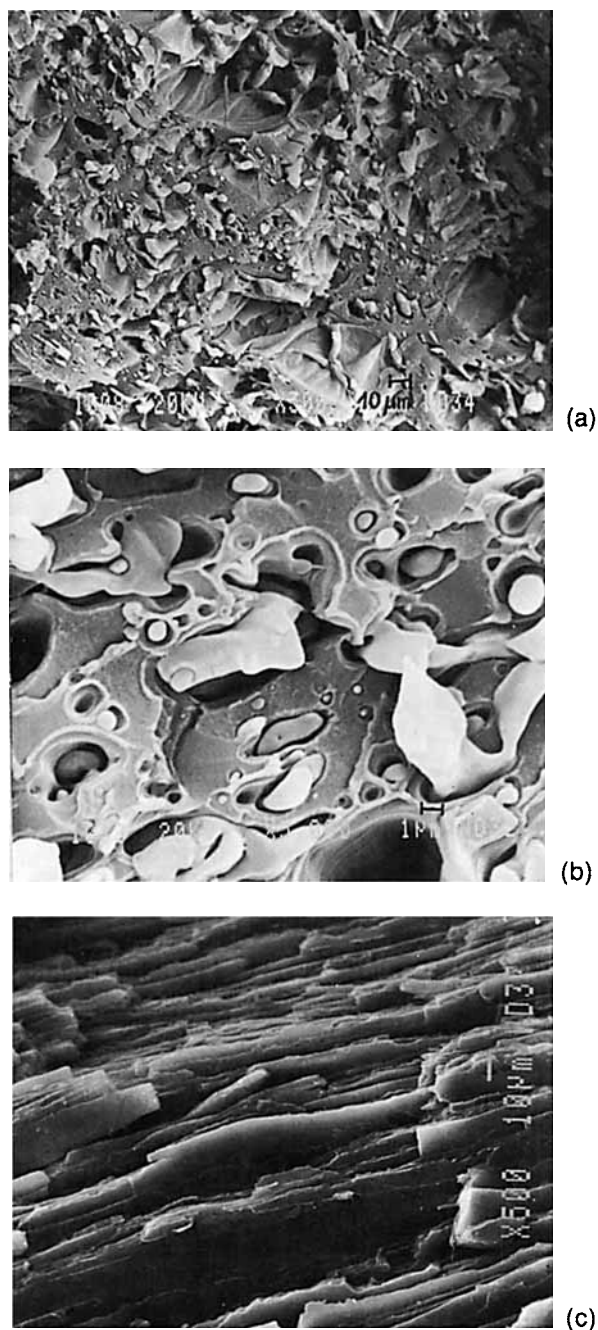


Figure 6 SEM micrographs of LDPE/PS 1 : 2 blend: failure surface (a) and (b)—higher magnification. Longitudinal cold fracture, (c).

enough to initiate microcracks in the system. A part of the overall fracture energy W is consumed for the crack development, including the debonding of the interface LDPE/PS and the yielding of poly(ethylene) bridges. The microfilaments observed in a crack zone have resulted from the plastic deformation of the ductile polymer.

Higher microheterogeneity of the alloy (Figs. 3[a] and 3[b]) and lack of "crazes" (Fig. 3[c]) evidences considerably smaller domains of PS (ca. $0.5 \mu\text{m}$) and diminishing of the interfacial tension. In a macro-scale this has been observed as a much higher elongation at break for the alloy in comparison to the blend.

Increasing the PS content to 33 wt % resulted in a significant change of the blend phase structure (Fig. 4). Instead of stratification, the interconnecting structure revealed threads of $2\text{--}10 \mu\text{m}$ width. This finding supposed that the percolation threshold had been surpassed. The failure took place in the poly(styrene) phase with the elongation being characteristic of this polymer. Successive morphology change was observed after the addition of the block copolymer (Fig. 5). The alloy LDPE/PS 2 : 1 represents a dispersive structure of fibrillar type, with fibers of $0.4\text{--}0.8 \mu\text{m}$ diameter (Fig. 5[b]). A similar mode of the phase structure was observed for the alloy containing 15 wt % of PS. The change of interactions between the phases and breaking of the continuity within the PS phase resulted in the increase of the elongation at break of one decade. This suggests that a significant part of the fracture energy W was transmitted by the poly(ethylene) fibrils, being clearly seen at magnification $10,000\times$.

The system LDPE/PS 1 : 2 is again of an interpenetrating type as far as the blend is concerned (Fig. 6) and microdispersive in the case of the alloy (Fig. 7). Width of the threads in polyblend is $1\text{--}10 \mu\text{m}$ (Figs. 6[a] and 6[b]), whereas the fibers in poly-alloy are of $0.4\text{--}0.8 \mu\text{m}$ diameter (Fig. 7[b]). The dominating character of the poly(styrene) matrix is decisive for tensile properties. The continuity of the PS phase caused the samples to fail at a low elongation and under stress twice as high as that observed for other systems.

CONCLUSIONS

1. Properties of heterogeneous LDPE/PS systems depend on the component ratio and on the compatibilizer content.
2. Block copolymer of compatibilizing activity enhanced melt viscosity and elasticity of

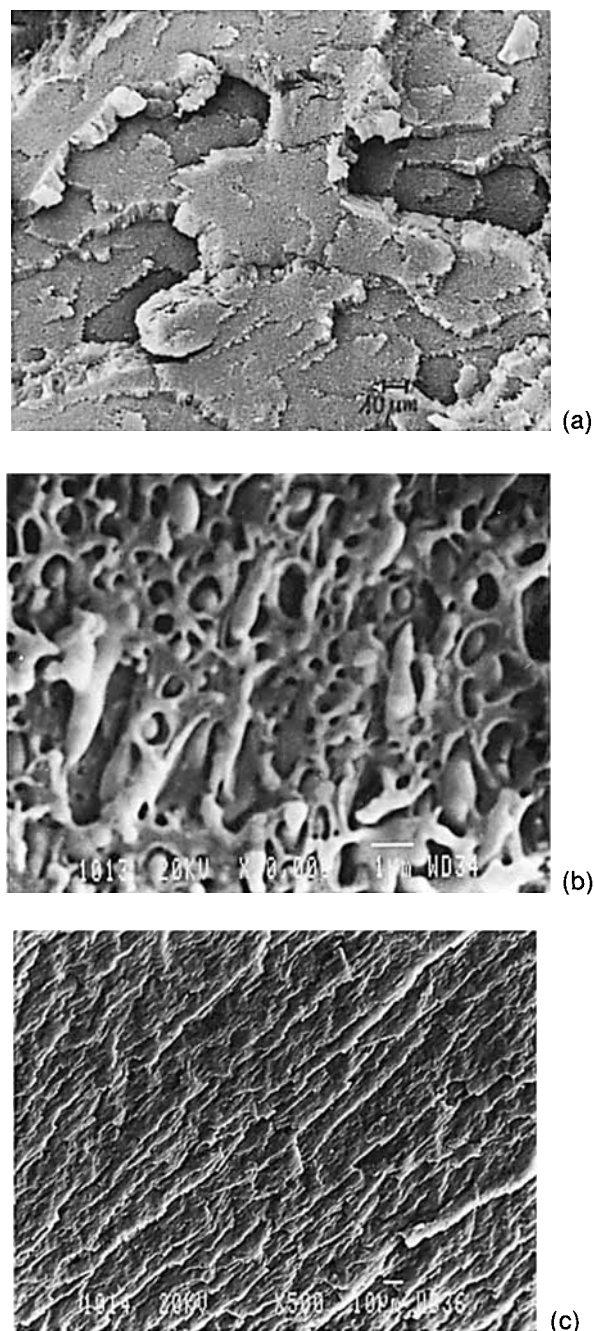


Figure 7 Morphology of LDPE/PS 1 : 2 alloy: failure surface (a) and (b)—higher magnification. In-axis cold fracture, (c).

LDPE/PS systems. Addition of the SEBS copolymer caused decreasing of the dispersed-phase domains.

3. Tensile strength and deformability for the systems of the disperse morphology are determined by the type of the matrix (brittle/ductile) and by the domain size of the dis-

persed phase. In case of interpenetrating-type morphology, the polymer systems fail by the brittle/ductile mode depending on the component of the lowest crazing/yielding stress.

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