Morphological origin of toughness in polyethylene–nylon-6 blends

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The effect of compatibilizing polyethylene and nylon-6 on the morphology and mechanical properties of their blends is investigated. A maleic anhydride functionalized styrene–(ethylene-*co*-butylene)–styrene block copolymer (MA/SEBS) is added to the blends as the compatibilizer. The compatibilizer is found to play a dual interfacial function. On the one hand, it reduces the interfacial tension of the system, resulting in reduction of the particle size of the dispersed polyethylene phase. On the other hand, it enhances the interfacial adhesion through the formation of micro-bridges. The fine polyethylene phase thus coupled to the matrix in compatibilized blends can carry load and deform co-continuously along with the matrix. This causes extensive yielding of the blend prior to its failure and high ultimate elongations. Unexpectedly, at MA/SEBS contents higher than 10%, the ductility of the blends drops sharply. This is attributed to flow-induced discontinuities within a large core in the tensile specimens. The lines of instabilities act as large notches within the specimen, causing brittle failure of the otherwise ductile blend.

(Keywords: toughness; morphology; blends; polyethylene; nylon; mechanical properties; compatibilization)

INTRODUCTION

The morphological features of an incompatible blend, such as the size of the dispersed phase domains and the adhesion at the interface, play an important role in determining its mechanical properties[1]. Evidence in the literature suggests that the blend morphology can be controlled by manipulating the interfacial tension and the shear stress of the melt[2–5]. To reduce the interfacial tension, appropriate block copolymers are added to blends as compatibilizers. These reduce the dispersed-phase particle size and may improve the interfacial adhesion[4,6,7]. For instance, incorporation of maleic anhydride grafted polyethylene (PE) (or polypropylene (PP)) in nylon–polyethylene (propylene) blends has been shown to reduce the dispersed-phase particle size[4,6,8,9] and increase the blend's impact strength[8,9].

Related studies have attempted to associate the impact energy of a blend with its morphology[8,10,11]. For blends in which a glassy polymer is toughened with a soft rubbery phase, it is usually recognized that there is an optimum particle size at which the impact strength of the blend is maximized[10,11]. This is rationalized on the basis of rubber-induced multiple crazing of the matrix[10]. For blends with more ductile (shear yielding) matrices, the interparticle distance is proposed to be the determining morphological feature in impact modification[8,11]. Based on the concept of interacting shear fields around neighbouring particles, Wu[11] as well as Hobbs and coworkers[8] developed numerical models that correlate the brittle–tough transition to blend morphology. This approach, useful as it may be, does not reveal the deformation mechanism of the triphase compatibilized blend. For instance, the deformability of the dispersed phase and its significance in the overall behaviour of the toughened blend is not taken into account by these models. The observed correlation between the models and the impact data is probably reflecting the undeformability of the dispersed softer phase at the high speeds encountered in impact tests. In addition, the role of interfacial adhesion in the deformation behaviour of blends at low strain rates is not signified. Even though only a 'minimum adhesion' is invoked as a requirement to cause the rubber particles to remain attached to the matrix upon impact[11], the specific role of the interface in stress transfer remains unclear.

In this paper we study the effect of compatibilization on the morphology and mechanical properties of polyethylene–nylon blends. Analysing the deformation behaviour of this crystalline–crystalline blend allows us to establish a mechanistic rationale for the morphology effect on mechanical properties of similar blends. In a subsequent paper, the role played by the mixing field on the evolution of morphology and the associated properties of the same blends will be presented.

EXPERIMENTAL

Materials

Blends of 75 wt% nylon-6 and 25 wt% low-density polyethylene (LDPE) are studied. The nylon-6 (Capron 8200) was supplied by Allied Chemical Corp. and the LDPE (Norchem PE953) by Northern Petrochemical Co. Kraton FG1901X rubber, a styrene–(ethylene-*co*-butylene)–styrene block copolymer functionalized with 2% maleic anhydride (MA/SEBS), is chosen as the

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compatibilizer. This is produced by Shell Chemical Co. The blends prepared contain 0, 1, 5, 10 and 15 pph MA/SEBS. They are denoted as, for example, N6 75/PE 25/15 pph MA/SEBS.

Blending

Prior to mixing, nylon-6 and MA/SEBS were vacuum dried at 70°C for 10 h. LDPE was dried for 2 h at 80°C in an oven. With our focus on compatibilizer effect in this paper, the results presented are those of the blends compounded in a 1 inch (2.54 cm) Killion single-screw extruder with a Koch static mixer. The extruder was operated at a barrel temperature of 220–240°C and a screw speed of 25 rev min^{-1}. The extrudates were quenched in a water bath, pelletized and dried in a vacuum oven for 18 h. Finally, the dried pellets were injection moulded into dumbbell-shaped ASTM D-638 type I specimens. A Boy 15-S moulding machine was used at a melt temperature of 240°C and a mould temperature of 60°C.

Mechanical tests

At least five specimens from each compound were dried in a vacuum oven for 18 h at 50°C and then brought to the testing temperature in a desiccator. The stress–strain behaviour was determined at 50 mm min^{-1} using a Monsanto mechanical testing machine equipped with a high-extension extensiometer. The elastic modulus computed at 1% strain (E), the yield stress (σ_y) and the elongation at break (ε_u) were determined from the stress–strain curves. For PE and the blends not exhibiting a yield maximum, the yield stress is calculated at the 0.2% offset strain.

Microscopy

In order to study the undeformed phase morphologies developed in the moulded specimens, they were immersed in liquid nitrogen for about 20 min, then fractured perpendicular to the mould fill direction (MFD). Also, to study the mechanisms of irreversible deformation, sections were cut from the necked portion of the tested specimens and, after immersing in liquid nitrogen for 20 min, were then fractured parallel to the tensile direction (also MFD). Samples were gold-coated prior to electron microscopy. Micrographs were taken by a JEOL model 840-A scanning electron microscope (SEM).

The flow-induced birefringence was observed in some specimens by transmission polarized light microscopy. The sections examined were cut from the untested tensile specimens perpendicular to MFD and had 0.25 mm thickness.

Calorimetry

The differential scanning calorimetry (d.s.c.) thermograms from the core and outside the core regions of N6 75/PE 25/15 pph MA/SEBS blend were prepared using a Perkin–Elmer model DSC-7 calorimeter. The thermograms were obtained at a heating rate of 20°C min^{-1}.

RESULTS

LDPE and nylon-6 are incompatible and exhibit two separate melting endotherms around 110 and 213°C, respectively (*Figure 1*). Regardless of the blend composition, the positions of the peaks remain unchanged. This indicates the absence of any co-crystallization and the presence of micrometre-scale phase separation[12]. Based on an initial survey of various polyethylene/nylon-6 (PE/N6) ratios, only in compositions where PE forms the dispersed phase are the mechanical properties highly sensitive to compatibilization. Accordingly, a PE 25%/N6 75% composition was analysed. D.s.c. analysis shows that the crystallinity of this blend decreases with an increasing amount of compatibilizer (*Figure 2*). Below, we present the mechanical behaviour of the blends, interrelated to the evolving morphology and the resulting deformation mechanisms.

Mechanical properties

Typical stress–strain curves are given in *Figure 3*. *Table 1* gives the measured properties of neat nylon-6 and LDPE (values in parentheses are standard deviations). All the blends exhibit necking associated with a well defined yield point at 4.4% strain, the same yield strain as for neat

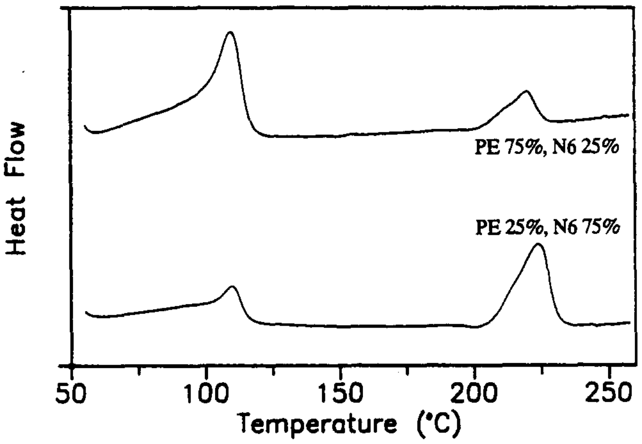

Figure 1 D.s.c. thermograms for PE 25% and PE 75% compositions. Scan rate = 20°C min^{-1}

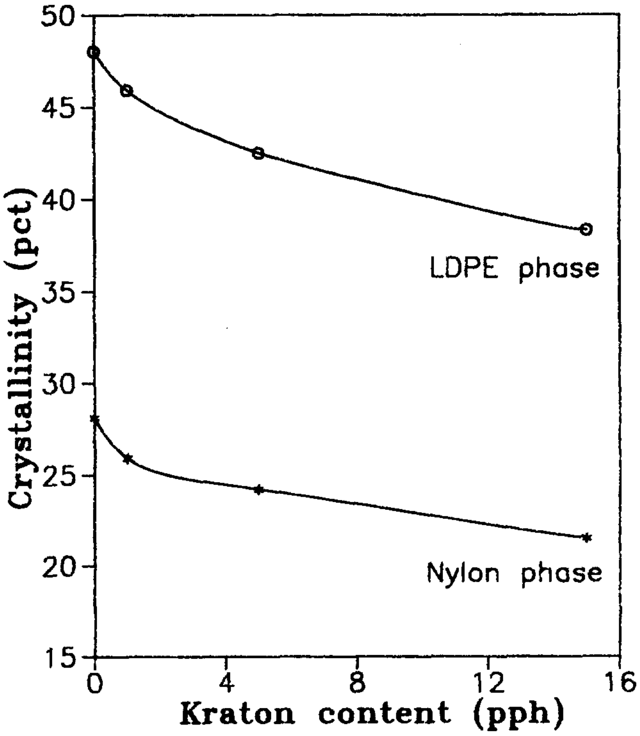

Figure 2 Percentage crystallinity of the N6 75/PE 25 blend as a function of the amount of compatibilizer incorporated

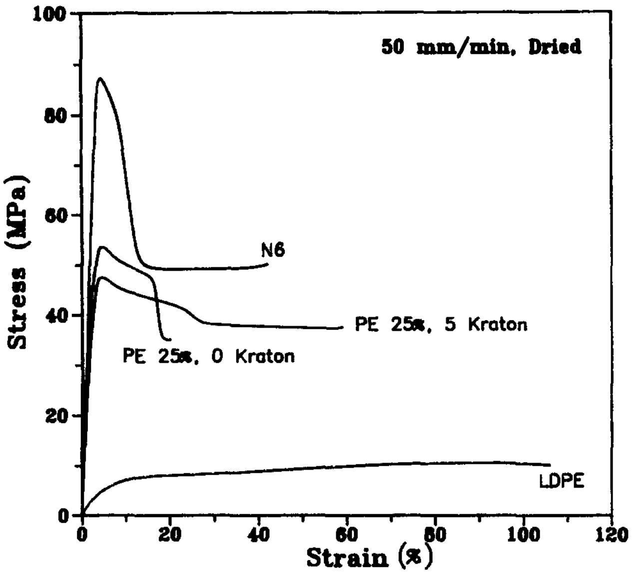

Figure 3 Stress–strain curves obtained for LDPE, N6 and their blends containing 0 and 5 pph MA/SEBS as compatibilizer

Table 1 Tensile properties of the low-density polyethylene (LDPE) and nylon-6 (N6) used for blending

	E (GPa)	σ_y (MPa)	ε_u (%)	σ_u (MPa)
LDPE	0.15 (±0.01)	5.7 (±0.1)	107 (±6.5)	10.8 (±0.2)
Nylon-6	2.95 (±0.53)	87.4 (±0.6)	46.4 (±10.2)	50.0 (±0.7)

nylon resin. This implies that necking in the blends is controlled by a nylon-6 matrix. The extent of necking and cold drawing increases from a very short unstable neck in uncompatibilized compositions to a well stabilized neck in blends with 5 and 10 pph MA/SEBS. Also, the extent of yield drop, i.e. the difference between the peak stress at yield and the plateau stress before fracture, is significantly reduced by the addition of PE to nylon-6. It is further reduced by incorporating MA/SEBS, indicating higher neck stability. Stress-whitening dominated the neck region in all the blends, which is indicative of submicrometre void formation during yielding. Pure LDPE, on the other hand, extends uniformly along the entire gauge length.

Figure 4 depicts the effect of compatibilizer content on tensile modulus. The broken lines represent the additive moduli, accounting for the standard deviation of measurements in neat resins. These moduli are calculated from the rule of mixtures, i.e. they are the volume-fraction weighted-average moduli for the blend composition. The rule of mixtures predicts an upper limit for the modulus of a blend based on a parallel structure of the phases in the tensile direction. Therefore, its proximity to our experimental values indicates a fairly uniform dispersion of the phases in the tensile direction and the continuity of the harder phase as the matrix[13]. Accordingly, in our blends with nylon-6 as the harder phase, the blending extruder appears to produce fine dispersions of PE particles in a nylon matrix. This will be shown later in this section. The deviation seen for 15 pph MA/SEBS blend, as will be discussed later, is due to the large-scale discontinuities experienced during moulding, which invalidates the basic assumptions underlying the rule of mixtures.

The yield stress, on the other hand, appears to defy additivity (*Figure 5*). The negative deviation observed, which also increases on increasing the MA/SEBS content, may be explained by the stress concentration induced by the PE particles in the nylon matrix. In calculating an additive yield stress, the weighted contribution of each phase to the total yield stress is simply assumed to be proportional to the yield stress of the phase in its neat state. However, numerous stress concentration sites in the matrix (PE particles) cause the matrix to yield locally at a stress lower than expected. This explanation for the initial stages of deformation in the blend parallels those of Hobbs[8] and Wu[11] for rubber- and polyethylene-toughened nylon blends. These authors attribute the high impact strength of the blends to the enhanced plastic flow in the matrix, caused by extensive interaction of shear stress fields around individual neighbouring rubber (or PE) particles.

The ultimate elongation is more sensitive to compatibilization than the modulus and yield stress (*Figure 6*).

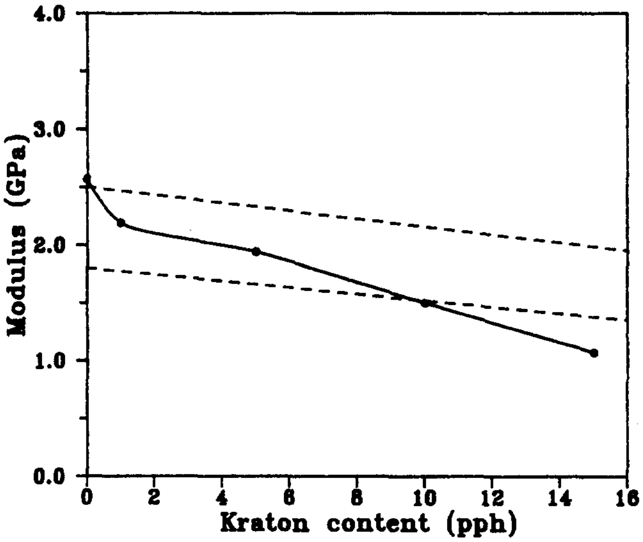

Figure 4 Tensile modulus of the blends as a function of MA/SEBS content (pph). Broken lines represent additive values obtained from the rule of mixtures, accounting for the standard deviation

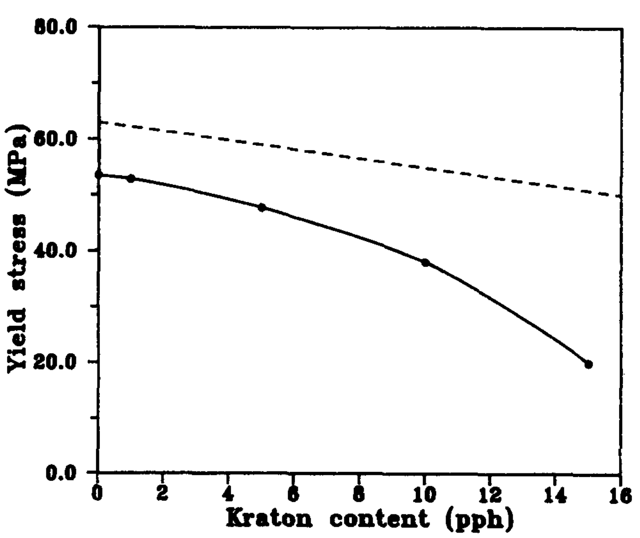

Figure 5 Yield stress of the blends as a function of MA/SEBS content (pph). The broken line represents the additivity (weighted-average) values

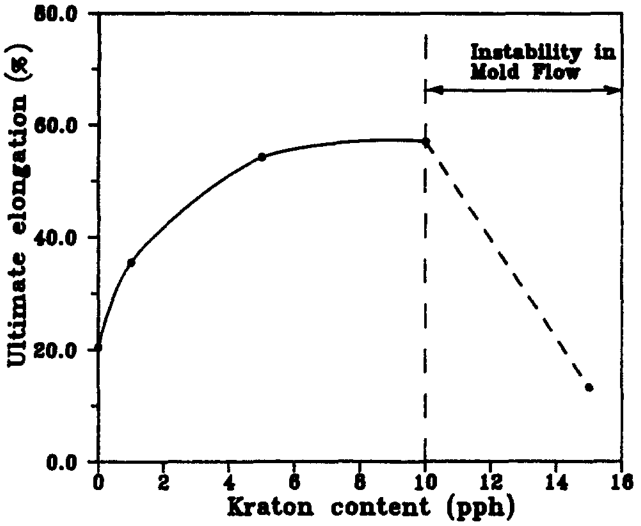

Figure 6 Ultimate elongation of blends as a function of MA/SEBS content

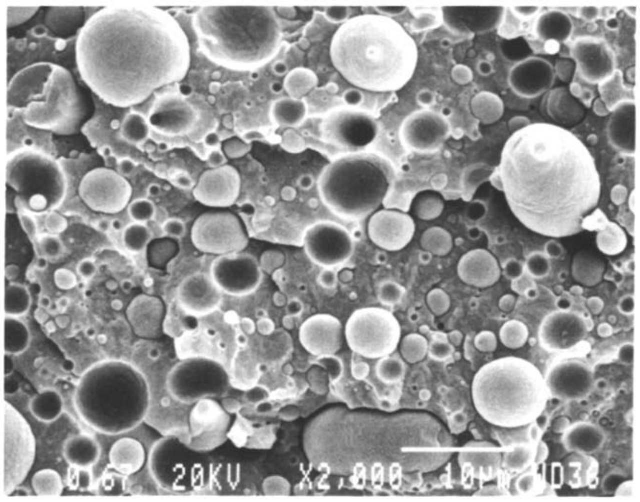

Figure 7 Phase morphology in N6 75/PE 25 blend with no MA/SEBS, indicating high dispersity in particle size distribution and no interphase adhesion

Noticeable increase is observed upon addition of up to 5 pph MA/SEBS. This toughening effect reaches saturation at 10 pph MA/SEBS. Increasing the MA/SEBS content to 15 pph, however, results in a sharp drop in ultimate elongation to 13.3%. To rationalize the observed mechanical behaviour, we examine the deformation mechanisms by following morphological changes upon deformation.

Morphological evolution

Figure 7 shows the undeformed phase morphology in the uncompatibilized blend. The dispersed PE particles range in size from 1 to 10 μm and appear either separated from, or loosely attached to, the nylon matrix. The lack of interfacial adhesion is evidenced by the absence of adhered nylon on the surface of PE particles and by the large number of craters produced by pulled-out particles during sample preparation by freeze-fracture.

Incorporating 1 and 5 pph MA/SEBS not only reduces the LDPE particle size in the blend, but produces a more uniform particle size distribution as well (*Figure 8a*). The gaps between the particles and the matrix are now wider, but bridged by tie fibrils of the order of 100 nm (*Figure 8b*), which appear to be due to MA/SEBS. The fibrillar nature of the interface reflects the interaction of MA/SEBS with PE and nylon-6 in the melt, and the solidification history of two crystalline domains with melting temperatures more than 100°C apart (*Figure 1*). In the melt, the MA/SEBS interacts with the evolving PE droplets through van der Waals bonding between the PE chains and the ethylenic segments of the MA/SEBS. Its interaction with nylon probably occurs due to hydrogen bonding involving the amide of the nylon and the carbonyl of the anhydride. The nylon phase solidifies first, containing molten PE droplets, coated with MA/SEBS, which remains coupled to the inner surface of the nylon cavities. As the PE droplets solidify, they shrink, causing the formation of gaps flanked with fibrillated MA/SEBS nano-bridges (*Figure 8b*). In the next section, we present evidence to substantiate this mechanism.

The number-average particle diameter of the dispersed PE phase (D_{avg}) is estimated from similar micrographs and is presented in *Figure 9*. The results are similar to the observations made by Hobbs and coworkers[8] in blends of nylon-6,6 and maleic anhydride grafted polyethylene. They report a rather sharp drop of D_{avg} at low percentages of maleic anhydride grafted to PE, and markedly less

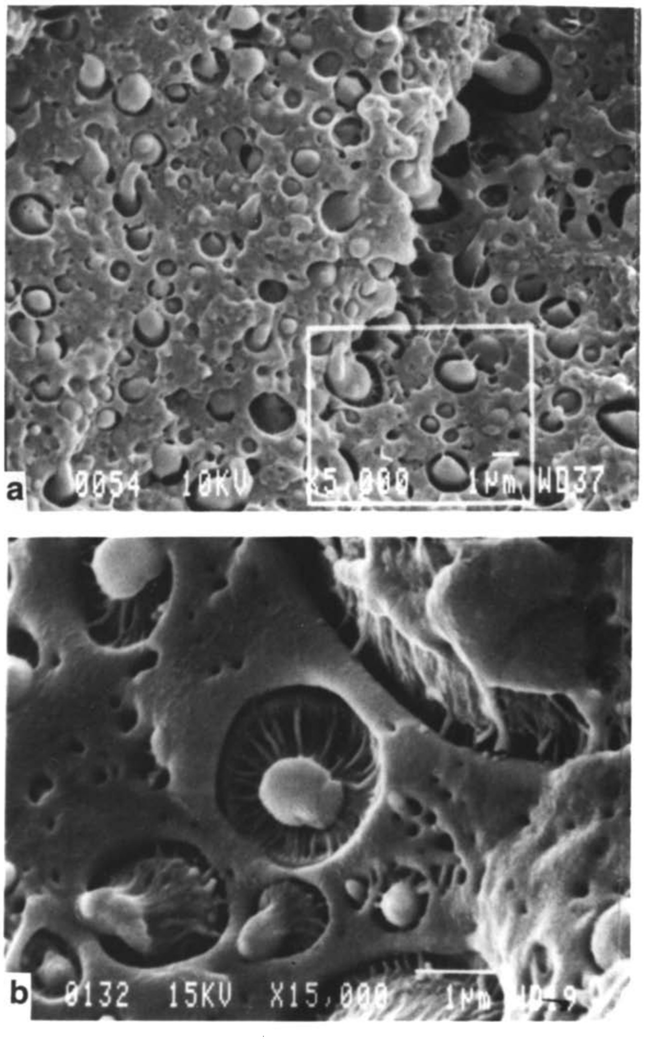

Figure 8 (a) Phase morphology in N6 75/PE 25/5 pph MA/SEBS; the marked rectangle shows the fine particles and good interphase bonding. (b) Phase morphology in N6 75/PE 25/1 pph MA/SEBS blend. Interphase micro-bridges link the phases (a different blending extruder)

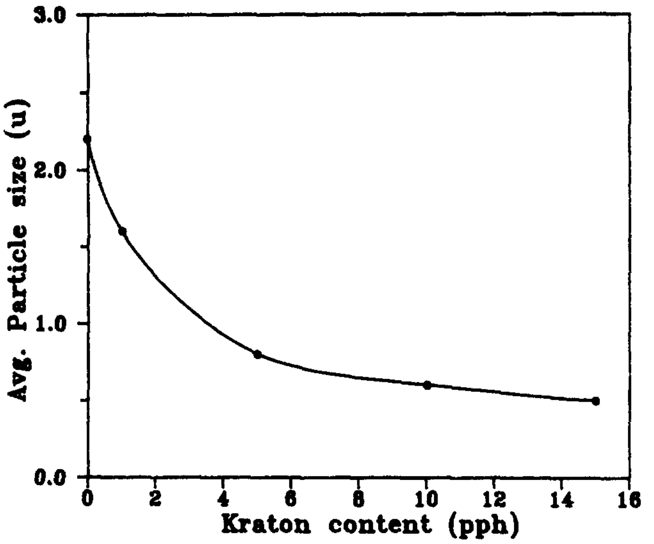

Figure 9 Number-average particle diameter of the dispersed PE phase in N6 75/PE 25 blends as a function of MA/SEBS content

reduction at higher graft levels. The same effect of compatibilizer on particle size is also reported for blends of PE (or PP) and nylon-6, compatibilized by ionomers[14]. The conformity between our results and those of Hobbs and coworkers[8] indicates that the maleic anhydride moieties grafted to a separately added MA/SEBS compatibilizer act in a similar way, in particle size reduction, as if they were directly grafted onto the PE chains. Nevertheless, our results indicate that compatibilization by a third phase (MA/SEBS) produces physical links between the two major domains, in addition to reducing interfacial tension, which causes particle size reduction.

Deformation mechanisms

To understand the toughening mechanisms of a two-phase blend, it is essential to examine how each phase responds to mechanical stress. Hence, in this section we examine the deformed morphology of our blends, compare it with the undeformed morphology (*Figures 7* and *8*), and correlate the observed deformation mechanisms to the elongation at break. The morphology of the phases in a deformed state (in the neck) is viewed parallel to the tensile direction. Upon deforming the uncompatibilized blends, the coarse PE particles, unattached to the matrix (*Figure 7*), are seen to display negligible deformation and are decoupled from the yielded nylon matrix (*Figure 10a*). This decoupling results in formation of relatively large cavities in the matrix, of the order of several micrometres in diameter (*Figure 10b*). The same cavitation is believed to be responsible for the stress-whitening observed within the neck prior to ultimate failure.

In compatibilized blends the bridged interfaces (*Figure 8b*) provide good load transfer from the nylon matrix to the dispersed PE particles. Hence they are deformed along with the matrix as the latter deforms (*Figure 11a*). As a result of this mechanical coupling, both domains deform co-continuously to a high draw ratio, and therefore absorb considerable strain energy. Comparing *Figures 10a* and *11b*, one notes that in uncompatibilized blends only the nylon matrix draws to domains that are a few micrometres thick, and in compatibilized blends both domains draw to less than a half-micrometre thickness. This reduction in the size of the local deformation naturally resulted in a much reduced void content. It is also noticed (*Figure 11b*) that the interconnecting bridges remain intact at this high level of deformation.

To ascertain the composition of the bridging material, a specimen similar to that of *Figure 12a* was treated with hot tetrahydrofuran (THF), which preferentially etches out MA/SEBS[15]. As shown in *Figure 12b*, the interfacial bridges have been etched out, causing the attached PE domains to be washed away. This is an indication of their MA/SEBS composition.

As seen in *Figure 6*, going from 10 to 15 pph MA/SEBS causes a marked reduction in blend ductility. The morphology of the blend, however, still shows a sub-micrometre-scale dispersed phase (*Figure 13*). The embrittlement occurs regardless of the type of mixing machine used for blending[16]. *Figures 14–16* are intended to explain the origin of this anomaly. The tensile fracture surface of the 15 pph MA/SEBS blend (*Figure 14*) shows a distinct core inclusion, which acted as the initiation site for brittle fracture. This inclusion appears to be associated with a gross segregation of a core material as the melt flows into the mould. Since the dispersion of the phases in the moulded specimen is a function of the flow field induced during the moulding operation, transmitted polarized

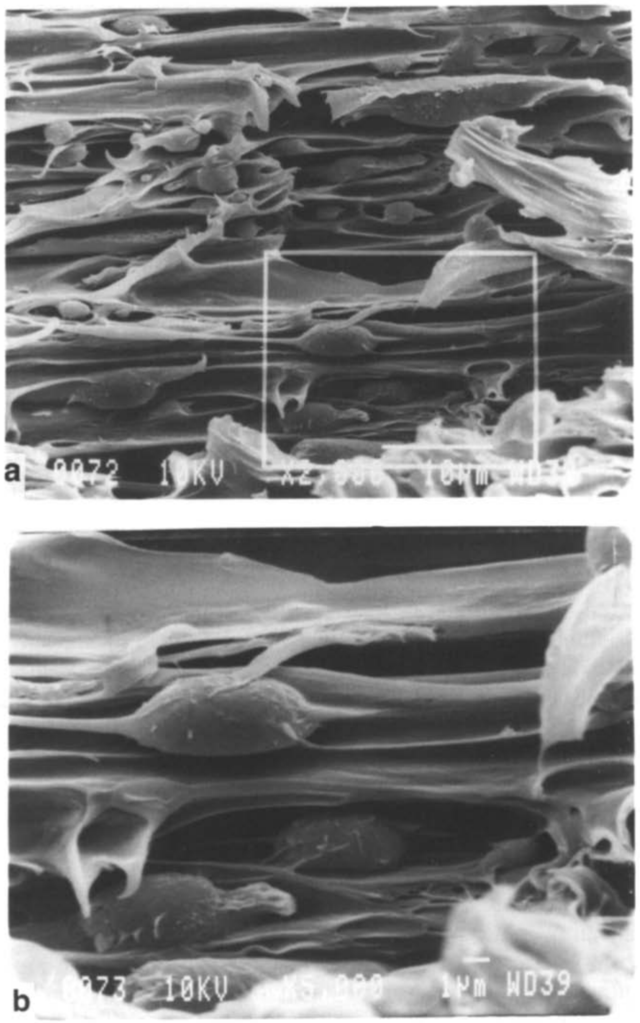

Figure 10 (a) Deformed phase morphology in uncompatibilized N6 75/PE 25 blends, observed in the necked section of the tensile specimen. Most PE particles are seen undeformed. Tensile direction = horizontal. (b) Magnified micrograph of the marked rectangle in (a). PE particles are not fully elongated and are loosely attached to the yielded matrix

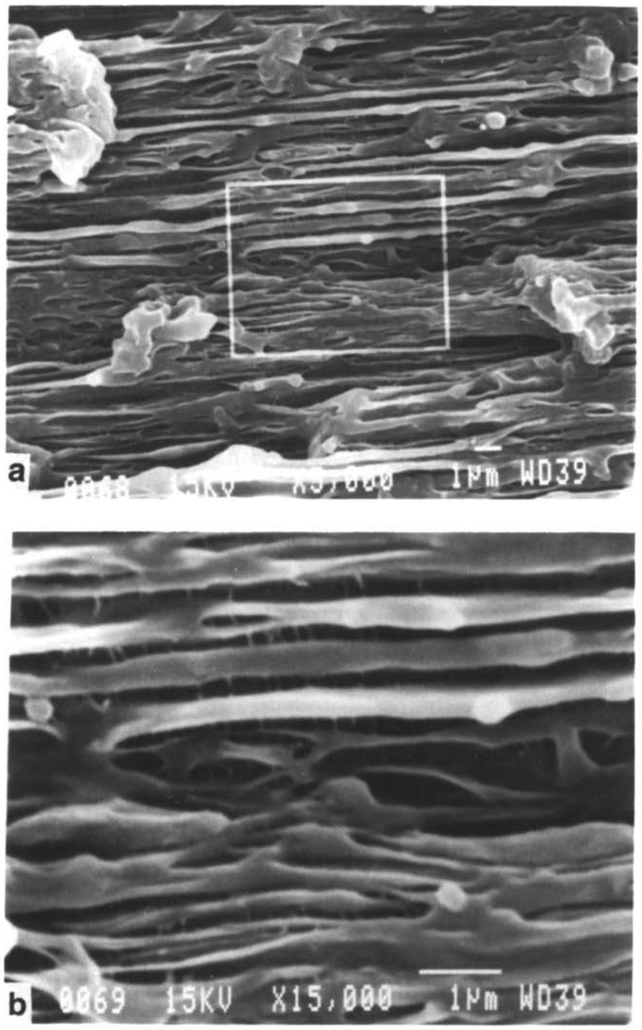

Figure 11 (a) Deformed morphology in N6 75/PE 25/5 pph MA/SEBS in the necked section of the tensile specimen. Tensile direction = horizontal. (b) Magnified micrograph of the marked rectangle in (a). Phases are grafted and both highly extended (yielded) parallel-wise

microscopy was performed on thin sections perpendicular to the MFD to visualize the flow field pattern. It was observed that only in the blends containing 15 pph MA/SEBS does a sharp birefringence pattern exist between the core and the surroundings (*Figure 15*). This phenomenon may be the result of a core melt stream flowing with a different velocity profile during moulding. The discontinuities observed in another section prepared parallel to the MFD from an undeformed specimen seemingly reflect flow instabilities of the core material (*Figure 16*). These flow-induced discontinuities act as stress concentration sites and are responsible for an early fracture initiation from the core, leading to brittle propagation across the specimen width. This mechanism may be further confirmed by the curved shape of the fractured cores, with their concavity always towards the injection gate (*Figure 15*). Cursory examination of a cross-section of the sample using infra-red microscopy indicates no major compositional difference between the core inclusion and its surroundings. In addition, no difference could be detected in the average particle size of the dispersed domains as examined by SEM. This phenomenon of flow instability was noticed to persist in samples moulded under identical conditions from blends having the same composition but mixed in two other equipments, namely, a single-screw extruder without static mixer and a co-rotating twin-screw extruder[16]. No attempts were made to study the effect of moulding conditions, the mould geometry or operational conditions of the mixing devices on the instability. Such a study could

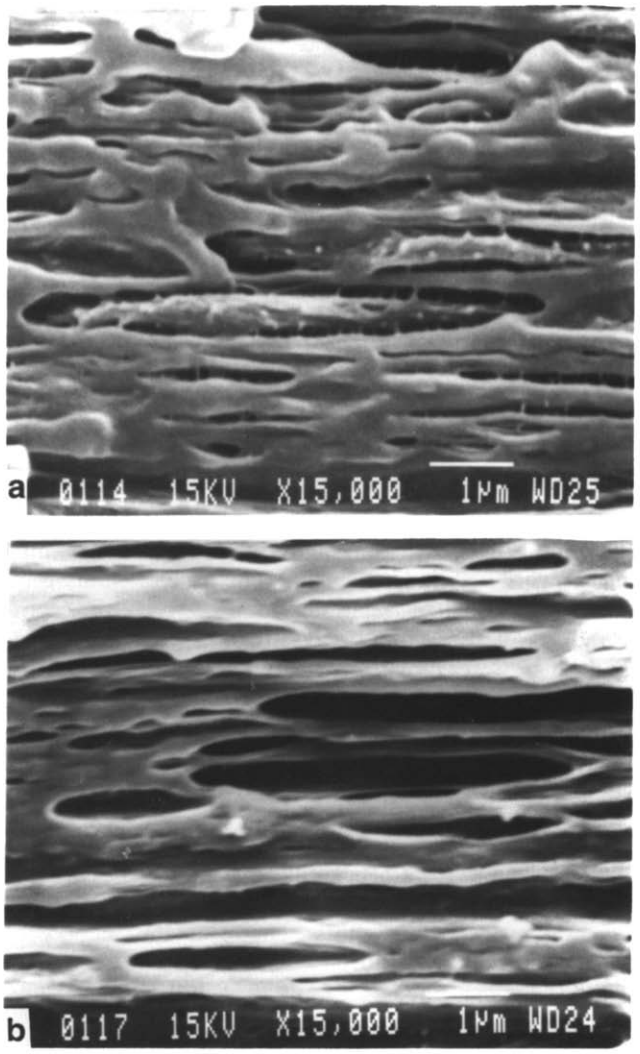

Figure 12 (a) Deformed morphology in compatibilized blends prior to etching out MA/SEBS by THF. Tensile direction = horizontal. (b) Deformed morphology in compatibilized blends after etching out MA/SEBS by THF. Tensile direction = horizontal

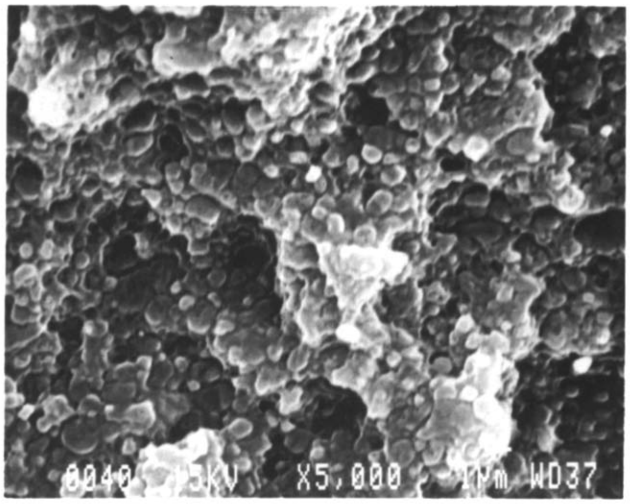

Figure 13 Phase morphology in N6 75/PE 25/15 pph MA/SEBS blend

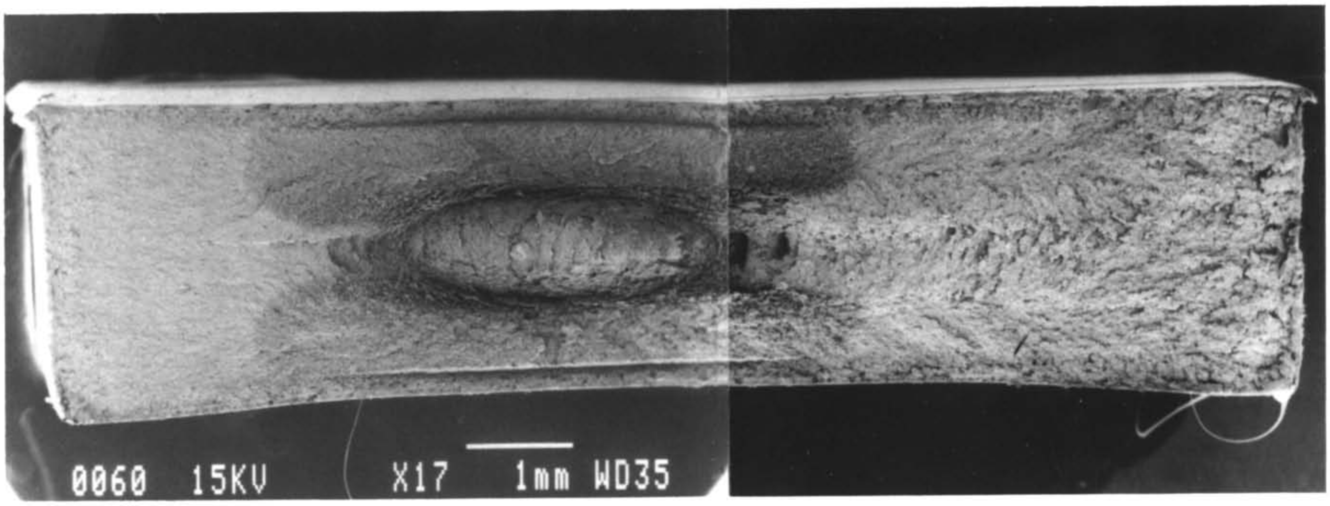

Figure 14 The tensile fracture surface of the N6 75/PE 25/15 pph MA/SEBS blends. Mould gate direction = in the plane of the paper

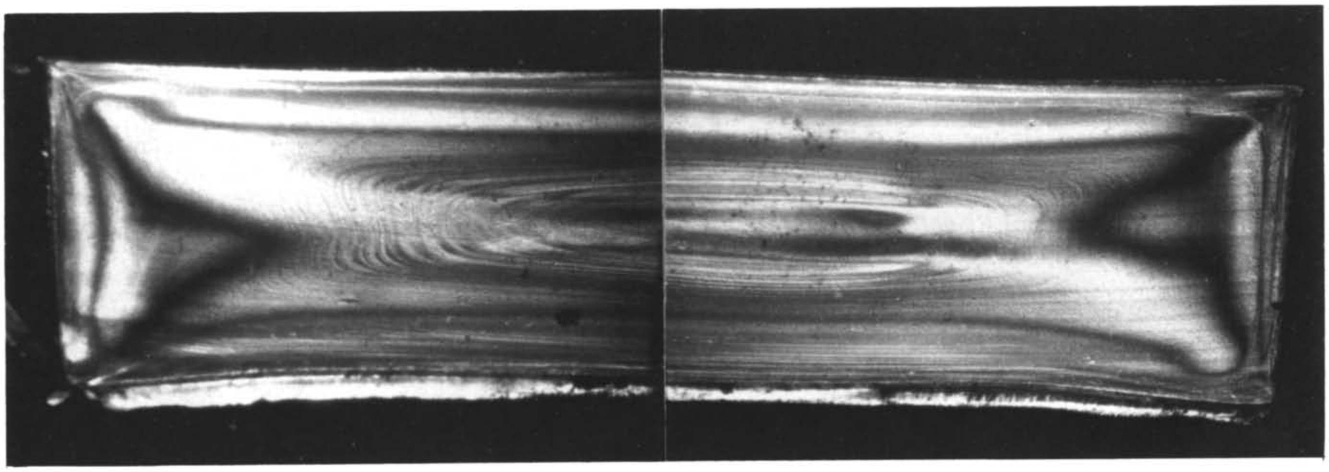

Figure 15 The birefringence in a transmission polarized light micrograph from a 0.25 mm thick section, perpendicular to MFD in 15 pph MA/SEBS blend

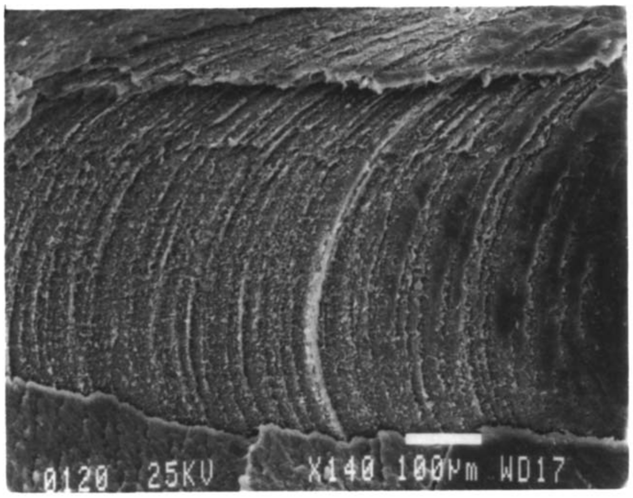

Figure 16 The parabolic velocity profile and the discontinuous flow lines in the core of 15 pph MA/SEBS blend. Mould fill direction = from right to left

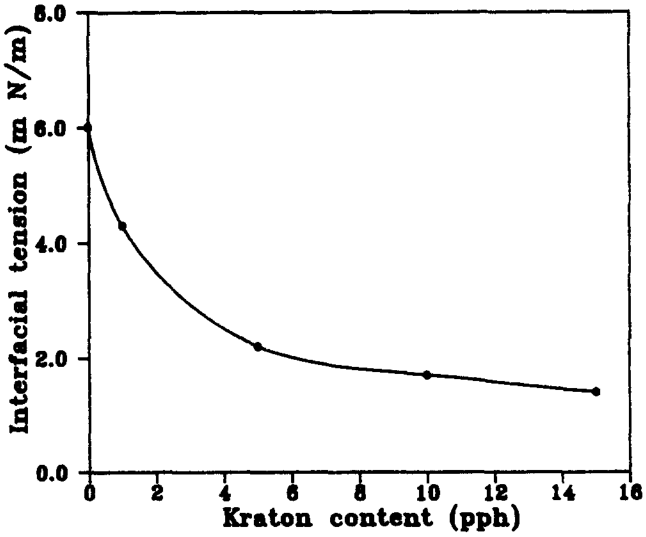

Figure 17 The calculated interfacial tension values for N6 75/PE 25 blends as a function of MA/SEBS content

define the operational flow conditions responsible for the instability. Exploring the origin of this type of melt instability is indeed interesting; however, it embodies a task that extends far beyond the scope of the present work.

DISCUSSION

The results of the present study confirm certain accepted phenomena in the field of incompatible blends, and reveal several new points that are worthy of discussion. The new points encompass the specific mechanism by which interfacial bonding forms and responds to mechanical loading, the toughening function of the dispersed phase, and the possible adverse effects of over-compatibilization.

As presented in the previous section, the study reaffirms the inferior mechanical performance of two-phase blends lacking proper interfacial adhesion[8–11] and demonstrates the usefulness of block copolymers possessing physical and chemical affinity to the blended phases in improving properties[8,9,17,18]. Compatibilizers added as a third component to blends have been reported to migrate to the interface and form layers 10–50 nm thick[18]. In our case, however, microscopic evidence demonstrates, for the first time, that the compatibilizer evolves into a third phase constituting mechanical nano-bridges. The interface strands that have been suggested to consist of MA/SEBS (*Figures 12a* and *12b*) bridge interfacial gaps as large as 800 nm (*Figure 8b*). As such, the micro-bridges perform a load transfer function leading to blend toughening. In this regard, it should be emphasized that such information would be concealed in impact-fractured specimens. Owing to the high test speeds in impact, the role of the stress transfer to the dispersed phase and its deformation in overall deformation of the blend is often ignored[8,11].

Our results also endorse the accepted views that the mechanical properties of a two-phase blend improve as the size of the dispersed domains is reduced to an optimum level[10,11], and that compatibilizers affect this desired reduction[4,6,8,14]. In the present case, where the mixing field remains unchanged, particle size reduction was achieved by increasing the compatibilizer content (*Figure 9*). The compatibilizer, styrene–(ethylene-*co*-butylene)–styrene block copolymer functionalized with maleic anhydride (MA/SEBS), couples to nylon through an assumed reaction between the terminal amine groups and the anhydride moieties[9,17], and to the polyethylene by 'dissolving' the olefinic moieties. The observation that MA/SEBS bridges are etched (dissolved) in THF (*Figures 12a* and *12b*) reinforces the likelihood that the compatibilizer couples to the nylon phase through non-covalent bonds. At present, a hydrogen-bonding scenario is more acceptable. Residing at the interface by migration[18], the compatibilizer forms the observed micro-bridges upon cooling from the melt. The foundation of this phenomenon is physicochemical, and its extent may be assessed from known relationships interrelating the average particle diameter of the dispersed domains D_{avg} to the mixing field parameters. That is[2–4,19,20]:

$$D = F(p)\gamma/(G\eta) \qquad (1)$$

where $F(p)$ is a function of the relative viscosity of the dispersed and the continuous phase, γ is the interfacial tension, G is the shear rate and η is the viscosity of the continuous phase. Using the particle size measured in our experiments (*Figure 9*), and assuming the interfacial tension for PE/N6 as $6\times10^{3}\,\mathrm{N\,m^{-1}}$ (ref. 4), the corresponding reduction in interfacial tension due to the incorporated compatibilizer is approximated (*Figure 17*).

The inverse relationship between the toughness of a two-phase blend and the average particle size of the dispersed domains appears nesessary from a micromechanical point of view. The dispersed domain should be large enough to bear and transfer stress, but should be small enough not to cause deleterious stress concentration effects. However, at least two other conditions must be fulfilled. The presence of sufficient interdomain bonding is required for effective load transfer. In addition, flow instabilities that may be used by excess compatibilizer and can result in moulding defects ought to be avoided. Large-scale discontinuities do not allow the micrometre-scale deformation processes to occur in the blend prior to its failure. As such, the micro-morphology of the blend does not play any role in determining its mechanical properties in defective specimens.

CONCLUSIONS

The results of an experimental study of the tensile behaviour of PE/N6 blends demonstrate that incompatibility of the phases results in their decoupling upon the application of strain. This causes relatively large voids to be formed in the matrix, and, accordingly, a suppressed ductility even below that of neat nylon-6.

Incorporating an olefinic chain thermoplastic elastomer functionalized with maleic anhydride (MA/SEBS) proves to compatibilize the blends. It stabilizes the cold-drawing of the blends by coupling the deforming phases through a 'micro-bridging' mechanism. Formation of these micro-bridges, as well as the fine PE particles, is suggested to be due to the reduced interfacial tension upon addition of MA/SEBS.

The ultimate elongation of the blend is maximized when a fine dispersion is produced with polyethylene particles that are well coupled to the matrix and are of the order of 0.8 μm in diameter. Incorporating MA/SEBS in excess of 10% causes a flow-induced segregation of a large core in tensile bars. The resulting core, with numerous flow line discontinuities, initiates a brittle fracture in tensile specimens.

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