

Impact Modification of Poly(ethylene terephthalate)

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ABSTRACT: The tensile and impact resistance of impact-modified poly(ethylene terephthalate) (PET) is investigated. The impact modifiers are polyolefin-based elastomers or elastomer blends containing glycidyl methacrylate moieties to improve the adhesion with the polyester. The tensile properties are measured on injection molded specimens at room temperature while the Izod impact strength is measured from -40 to 20°C . The blend morphology is observed by scanning electron microscopy and the dispersed phase

average diameter is determined by image analysis. The relation between the impact resistance and the phase morphology is discussed, and the critical ligament size for PET is determined. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 2919–2932, 2003

Key words: polyesters; blends; compatibilization; impact resistance; morphology

INTRODUCTION

The mechanical properties of materials are based on structure or morphology and the micromechanical processes of deformation and fracture. These are influenced by the molecular structures and morphology, as well as loading conditions. The deformation behavior of materials strongly depends on the stress state, temperature, and strain rate. Polymers will fail by shear yielding or cracking/crazing depending on the magnitudes of and the ratio between the applied hydrostatic (mean) and shear (deviatoric) stresses. Stress states that have a large deviatoric to hydrostatic stress ratio often produce ductile behavior, even in so-called brittle materials.¹ Because of the high yield stress relative to their crazing/cracking stress, many polar thermoplastics, such as poly(ethylene terephthalate) (PET), tend to be notch sensitive and can exhibit a sharp ductile–brittle transition. Numerous studies have addressed key parameters affecting the mechanical behavior of rubber-modified thermoplastics, such as the molecular weight of the components; the morphology (rubber particle size, interparticle distance, and rubber content); the testing conditions; and the mechanisms responsible for the improvement in toughness, which include cavitation of the rubbery phase, craze initiation and termination at rubber particles, and shear yielding.^{2–6}

An important factor controlling the impact resistance of polymer blends is the critical interparticle

distance, also referred to as the critical matrix ligament thickness. It was first shown by Wu⁷ on polyamide/ethylene-propylene rubber (EPR) blends that the brittle–tough transition was correlated to a critical interparticle distance. Later studies showed that this critical value was affected by the deformation rate and temperature. The initial interpretation for the role of the interparticle distance involved percolation, but a number of alternative explanations were proposed and this issue has remained controversial to this day. A recent model for interparticle distance–temperature–strain rate dependence has been based on the overlap of the stress field around the rubber inclusions.^{8,9} From this model it is inferred that the critical interparticle distance increases with temperature and decreases with the applied strain rate. The critical ligament size is therefore not a fixed value but depends on testing conditions. An interpretation based on crystallographic observation was also proposed. The critical ligament size was associated with the oriented crystallization within the ligament. It was argued that the polymer crystals in the vicinity of the elastomer inclusion are oriented with their lower energy, easier to shear, crystallographic planes parallel to the interface. If the ligament size is decreased to the point where all the interparticle material is in this preferential orientation state, the result is that the yield stress of the matrix material is decreased, thus enabling plastic deformation instead of brittle fracture. The first evidence for this interpretation was shown by Muratoglu et al.¹⁰ on polyamide/EPR blends. Bartczak et al.¹¹ then demonstrated that the same mechanism could apply to high-density polyethylene (HDPE) modified with polyolefin elastomers. Further evidence of the role of crystallography was given in a

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TABLE I
Properties of Pure Components

Material	GMA Content (wt %)	MA Content (wt %)	T_g (°C)	MFI
Engage 8200	0	0	-50	5
GMA grafted Engage 8200	0.6	0	-50	5
Lotader 8840	8	0	-19	5
Lotader 8920	1	26	-28	4
Lotryl 28MA07	0	27	-28	7
Lotader 8900	8	26	-28	6

TABLE II
Impact Modifiers Investigated

Modifier Designation	Components	GMA Content in Minor Phase (wt %)	MA Content in Minor Phase (wt %)
A	Engage 8200	0	0
B	GMA grafted Engage 8200	0.6	0
C	Engage 8200/Lotader 8840 (7:1)	1	0
D	Lotader 8920	1	26
E	Lotryl 28MA07/Lotader 8900 (7:1)	1	27

subsequent article in which the impact resistance of HDPE was increased by incorporating fine particles of calcium carbonate instead of an elastomer.¹² Detailed wide-angle X-ray scattering and atomic force microscopy analyses showed that PE crystals grow similarly with their low energy (100) plane parallel to the interface whether the substrate is calcite or rubber.¹³

Many aspects related to the influence of a second phase on the toughness improvement in polyester-based blends are still to be elucidated. PET is tough in unnotched situations. However, its behavior changes from ductile to brittle when the stress concentration is induced by the presence of a notch. One way of increasing the impact strength is to incorporate rubbery inclusions that are able to cavitate and lower the overall plastic resistance. Recently, polyolefins produced with metallocene catalysts have been used successfully as impact modifiers. Interfacial modification is

achieved using elastomers containing a functional group that can graft to PET to form a copolymer. Many studies have addressed the effect of functionalized elastomers on the toughening of PET, most of them focusing on styrene-butadiene-styrene copolymers.¹⁴⁻¹⁶ The addition of glycidyl methacrylate (GMA) grafted polyolefins has also been shown to be useful for the reactive compatibilization of blends of polyolefins with PET.¹⁷⁻¹⁹ In such blends, a polyolefin-polyester graft copolymer is generated *in situ* by a reaction involving the grafted epoxy moieties and the carboxyl/hydroxyl polyester end groups.

This study focuses on the mechanical performance of PET containing different polyolefin-based copolymers used as impact modifiers. The effect of the mod-

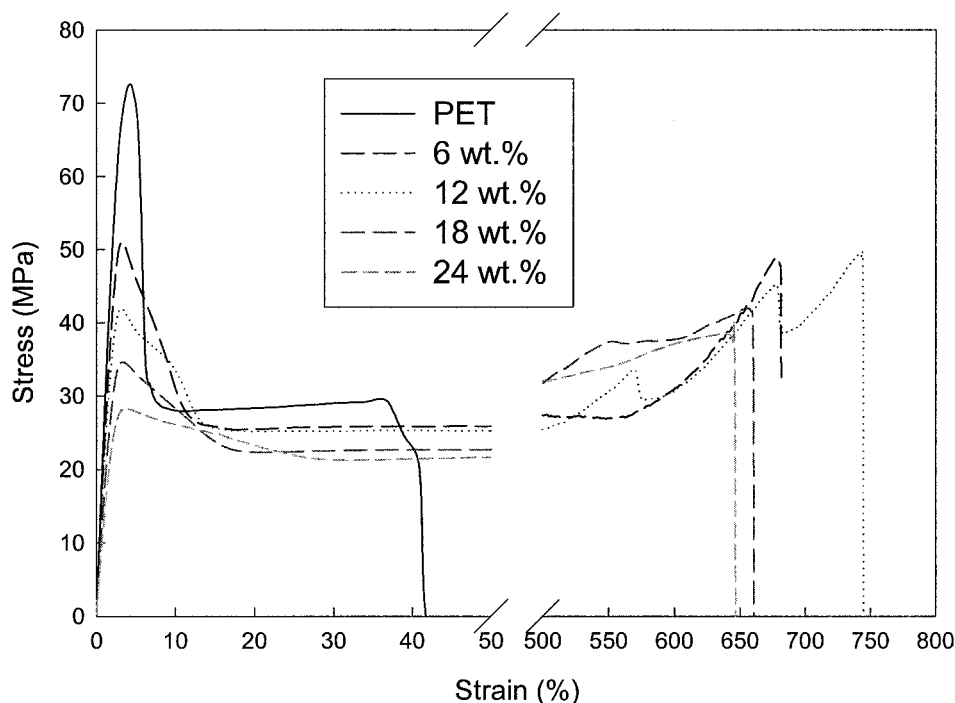


Figure 1 The stress-strain curves for pure PET and blends containing modifier D.

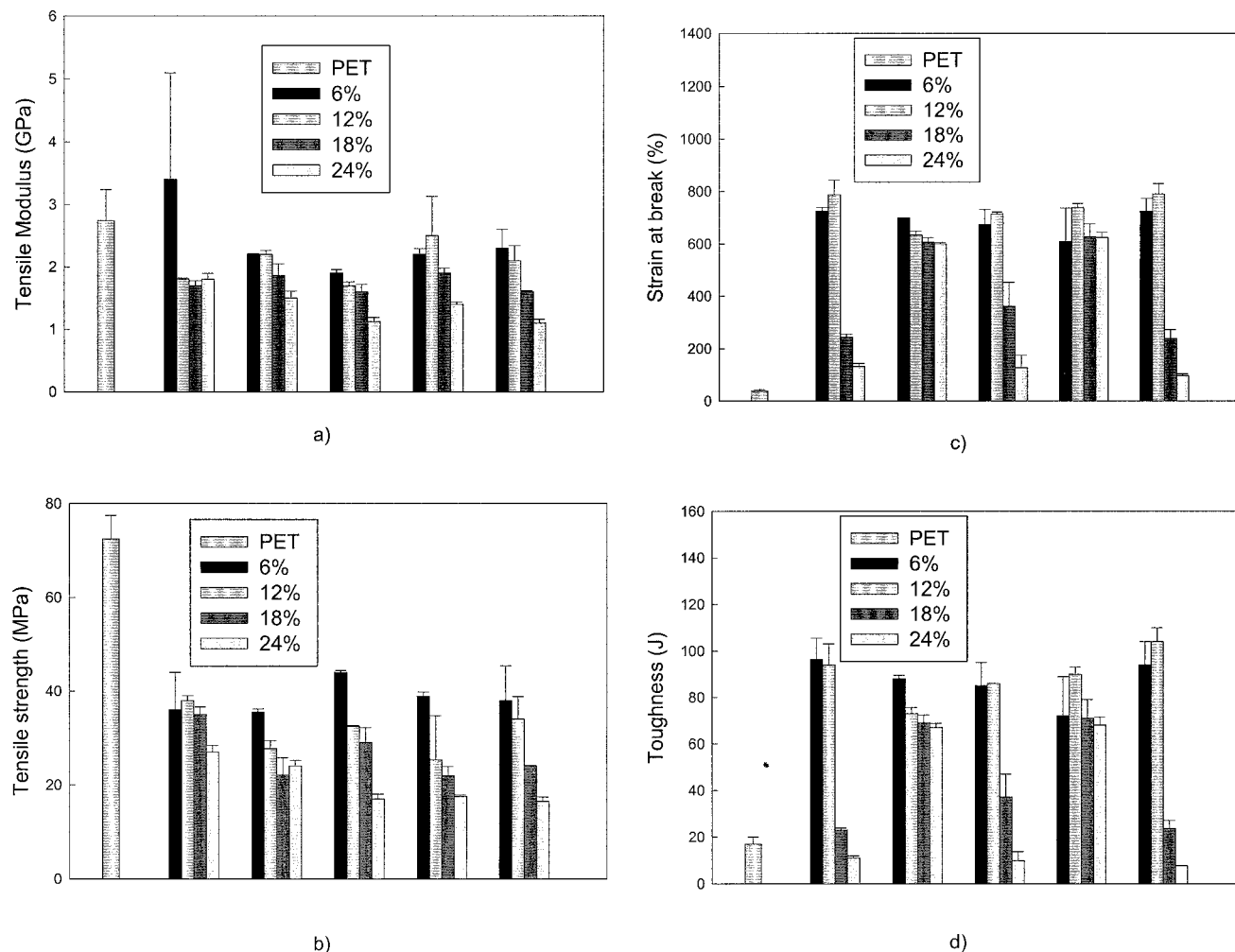


Figure 2 (a) The tensile modulus, (b) tensile strength, (c) elongation at break, and (d) toughness of all blends under study. All properties are obtained at a speed of 500 mm/min, except for modifiers B and D, which are obtained at 50 mm/min.

ifiers on the tensile and impact properties is evaluated. The relationship between modifier properties, blend morphology, and mechanical performance is discussed. The brittle-tough transition of notched Izod specimens is investigated with respect to the morphological parameters and temperature. Finally, the critical ligament size is evaluated.

EXPERIMENTAL

Materials

The PET used in the study was Cleartuf 8006 (Shell) with an intrinsic viscosity of 0.8 dL/g. Six types of modifiers were investigated and their properties are summarized in Table I. Engage 8200 is a metallocene ethylene-octene copolymer that contains 24% octene and has a melt flow index (MFI) of 5 (DuPont Dow Elastomers). A second engage 8200 is the same elastomer that was chemically modified in-house by melt free-radical grafting of GMA. The next four modifiers contain random copolymers called Lotader and Lotryl

(Atofina). Lotader 8840 is a copolymer of ethylene and 8% GMA. Lotader 8920 and 8900 are ethylene, methyl acrylate (MA), and GMA terpolymers that contain 26% MA and 1 and 8 wt % GMA, respectively. The MFI of the three Lotaders are 5, 4, and 6, respectively. Finally, Lotryl 28MA07 is an ethylene-MA copolymer, which contains 28 wt % MA and has an MFI of 7. These materials were used as received or compounded in order to control the GMA and MA content in the final blend. The resulting modifiers are listed in Table II. Engage 8200, its GMA grafted version, and Lotader 8920 were used as received and are referred to as modifiers A, B, and D, respectively. Modifier C is a 7:1 blend of Engage 8200 and Lotader 8840. Modifier E is a blend of Lotryl 28MA07 and Lotader 8900 in a 7:1 proportion.

Modifier A has no chemical affinity for the polyester and is not expected to generate favorable blend morphology or adhesion to the PET. All other modifiers contain GMA, which is known to react with the PET carbonyl end groups to form graft copolymer. This

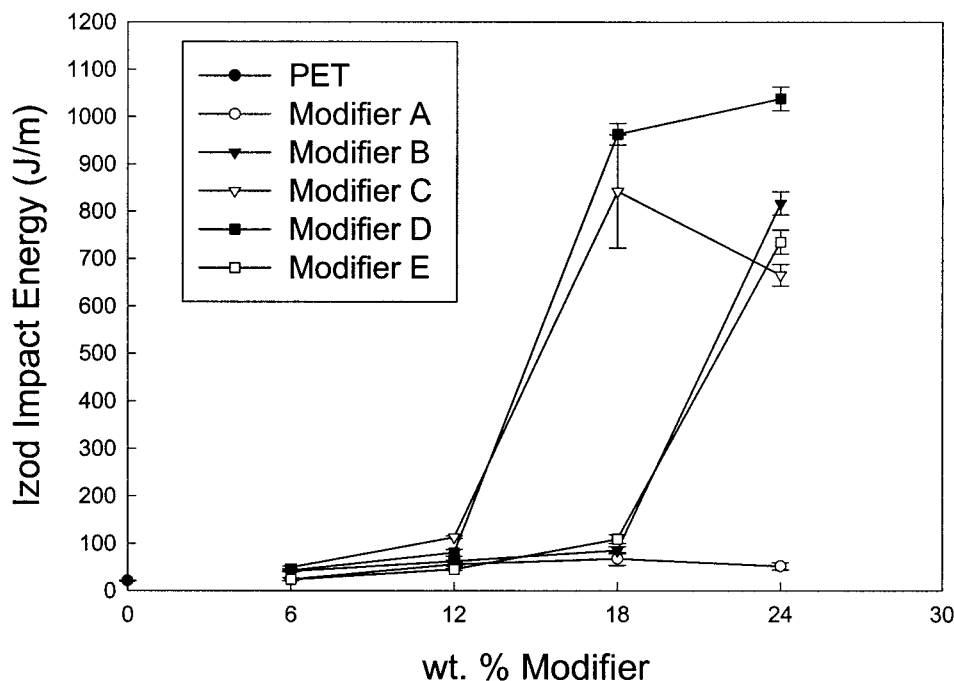


Figure 3 The Izod impact strength at 20°C for all blends.

graft copolymer is expected to locate at the blend interface, decrease the interfacial tension in the molten state, and increase adhesion in the solid state. Modifier B contains 0.6% grafted GMA and modifiers C, D, and E all contain 1% GMA.

Sample preparation and characterization

The blends were prepared in a Leistritz 34-mm corotating twin-screw extruder at a screw speed of 150 rpm, a throughput of 15 kg/h, and a barrel temperature of 290°C. The PET was dried at 170°C for 4 h prior to extrusion runs. Blends containing 6, 12, 18, and 24 wt % modifier were extruded into strands and pelletized. These were dried again and then injection molded into ASTM type I specimens using a mold temperature of 10°C.

The tensile mechanical properties were measured according to the ASTM D 638 standard on injection molded specimens. Because of the high elongation at break typical of rubber-modified blends, type IV dog-bones were used. The drawing speed used for the tests was 50 mm/min and the initial deformation rate was measured independently using an extensometer in order to provide accurate tensile modulus measurements. The notched Izod impact resistance was measured according to ASTM D 256 using the central portion of the injection molded type I specimens (62.5 × 12.7 × 3.17 mm, length × width × thickness). These specimens were notched according to the same standard. The mechanical properties reported are the average of five tests.

The morphology of the blends was investigated by scanning electron microscopy (SEM). Injection molded specimens were cut and annealed at 120°C for 4 h. The annealing is necessary to increase the crystallinity of the PET matrix and to enable the subsequent selective dissolution of the elastomer phase. The central part of the specimens was microtomed at room temperature in the transverse direction (perpendicular to the flow). The elastomer phase was then dissolved by immersing the samples in boiling toluene for 3 h. Impact fractured surfaces were also investigated by SEM.

The crystallinity of the materials was measured by modulated differential scanning calorimetry (mDSC) using a TA Instruments Q1000. A scan rate of 2°C/min was used with a temperature modulation amplitude of 0.32°C and an oscillation period of 60 s. The initial crystallinity was calculated by subtracting the values of the integrated crystallization and melting peaks obtained from the nonreversing and reversing components, respectively.

RESULTS AND DISCUSSION

Mechanical properties

The tensile properties of pure PET and all blends were determined and are reported in Figures 1 and 2. Figure 1 presents typical stress-strain curves for pure PET and for PET/modifier D. PET is a ductile material with an elongation at break of about 40%. The addition of the modifier decreased the elastic modulus and the yield stress. The elongation at break increased

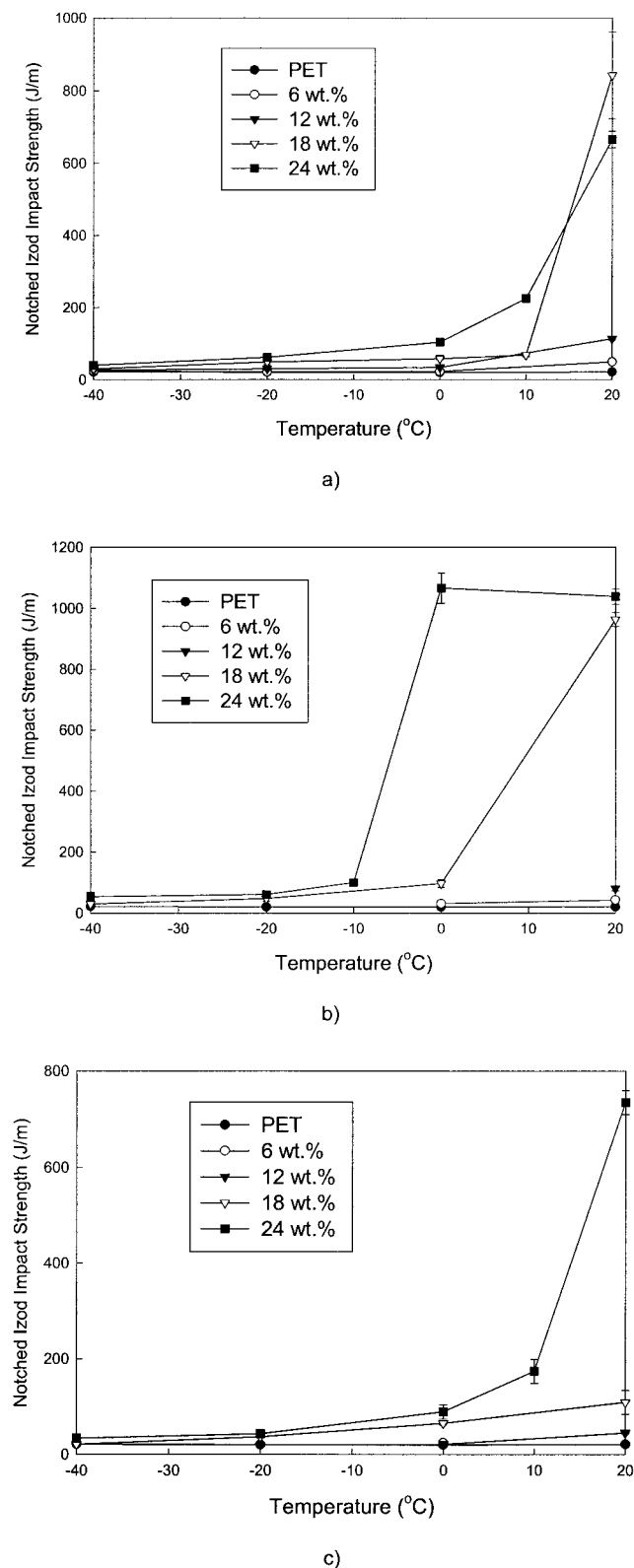


Figure 4 The Izod impact strength as a function of the temperature for blends containing from 6 to 24 wt % (a) modifier C, (b) modifier D, and (c) modifier E.

significantly, jumping from 40% for pure PET to between 650 and 750% for the modified PET, depending on the modifier content. This is typical of rubber-

modified materials. Similar observations were made for all modifiers under study. The tensile modulus, tensile strength, elongation at break, and toughness of all investigated blends are reported in Figure 2. The tensile modulus and tensile strength decreased relatively linearly with the elastomer content [Fig. 2(a,b)]. The property reduction is expected because of the modifiers' lower modulus and strength. The tensile modulus of blends is usually unaffected by the interfacial adhesion because it is measured at low deformation when the blend components are still in contact. However, the tensile strength can benefit from increased adhesion by enabling stress transfer from the matrix to the dispersed phase. In our case, the blends with modifier A that does not have any reactive moieties, and thus no affinity for PET, performs as well as modifiers B–E. This is due to the important rigidity mismatch between the PET and the elastomers. The material essentially behaves as a cellular material. From a mechanical point of view, the elastomers act as voids, reducing the effective matrix area bearing the tensile load. The ultimate tensile properties do not obey simple mixing rules. Adding 6 wt % modifier significantly increased the elongation at break and toughness (area under the force–displacement curve) of all the blends, as shown in Figure 2(c,d). At 12 wt %, the elongation at break and toughness values for all blends remained high but further increases to 18 and 24 wt % resulted in an important reduction for blends with modifiers A, C, and E to values close to those of unmodified PET. In contrast, the ultimate tensile properties of blends containing high contents of modifiers B and D remained essentially constant. These differences will be discussed in light of morphological differences in a subsequent section.

The notched Izod impact strengths of all blends at room temperature are summarized in Figure 3. Pure PET has poor impact resistance with a value of 21 J/m. Except for the blend containing modifier A, the addition of the elastomers resulted in an impressive jump of the Izod impact energy. Except for those with modifier A, all blends presented a brittle to ductile transition when increasing the modifier concentration and exhibited high toughness between 700 and 1040 J/m when used at 24 wt %. Modifiers C and D showed an earlier transition leading to high toughness even down to 18 wt %. In all cases, adding 12 wt % or less elastomer did not significantly improve the impact resistance of the blend. It must be specified that, although the pure PET specimens always broke completely, the behavior of the tough blends was of the “nonbreak” type, according to standard ASTM D 256 specifications (i.e., that the fracture extended less than 90% of the distance between the vertex of the notch and the opposite side). For these blends, a plastic deformation zone could clearly be identified and the thickness of the specimens was slightly narrower near

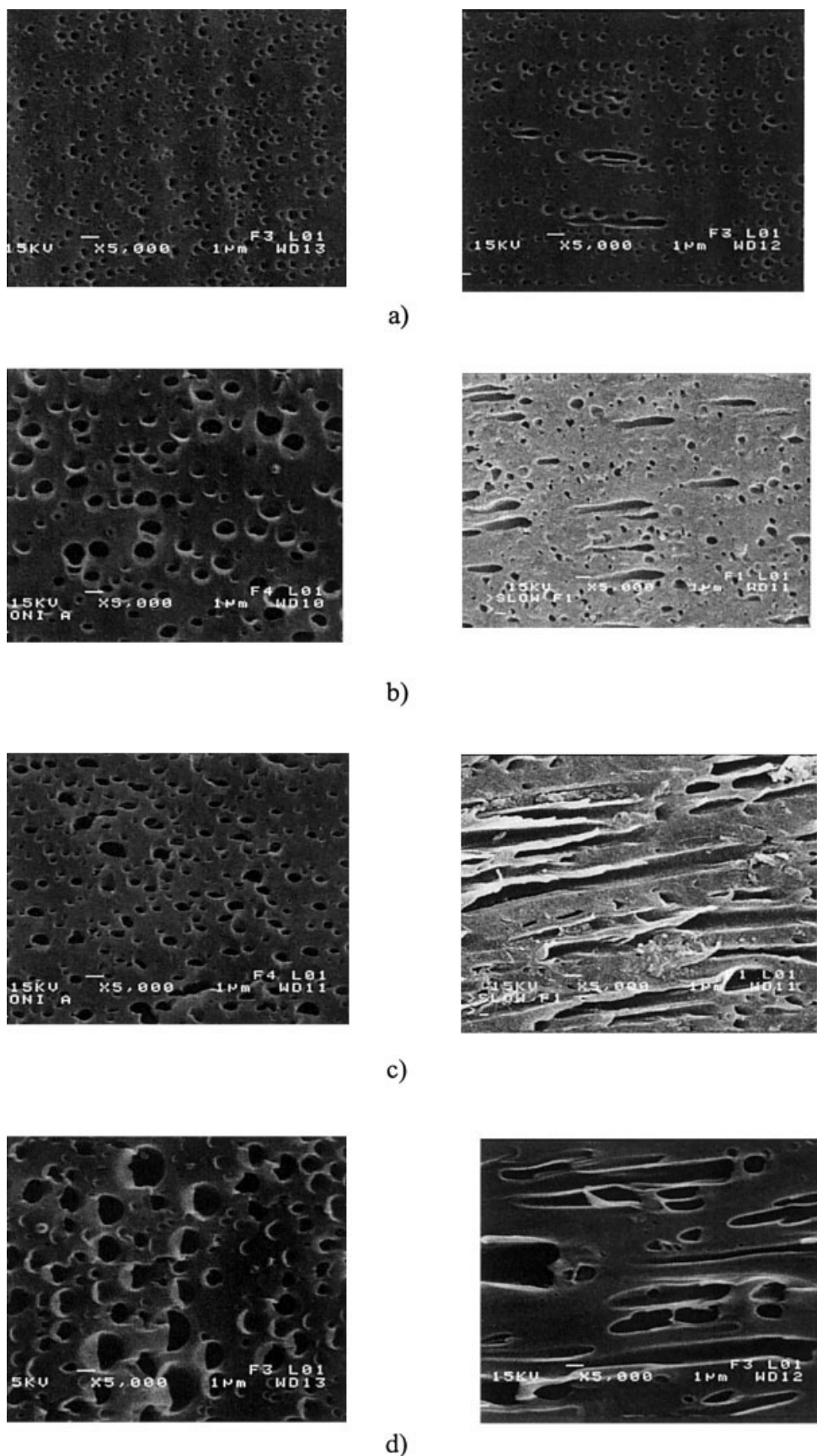


Figure 5 Scanning electron micrographs of uncompatibilized (modifier A) blends at (a) 6, (b) 12, (c) 18, and (d) 24 wt %. Left column: transverse direction. Right column: longitudinal direction.

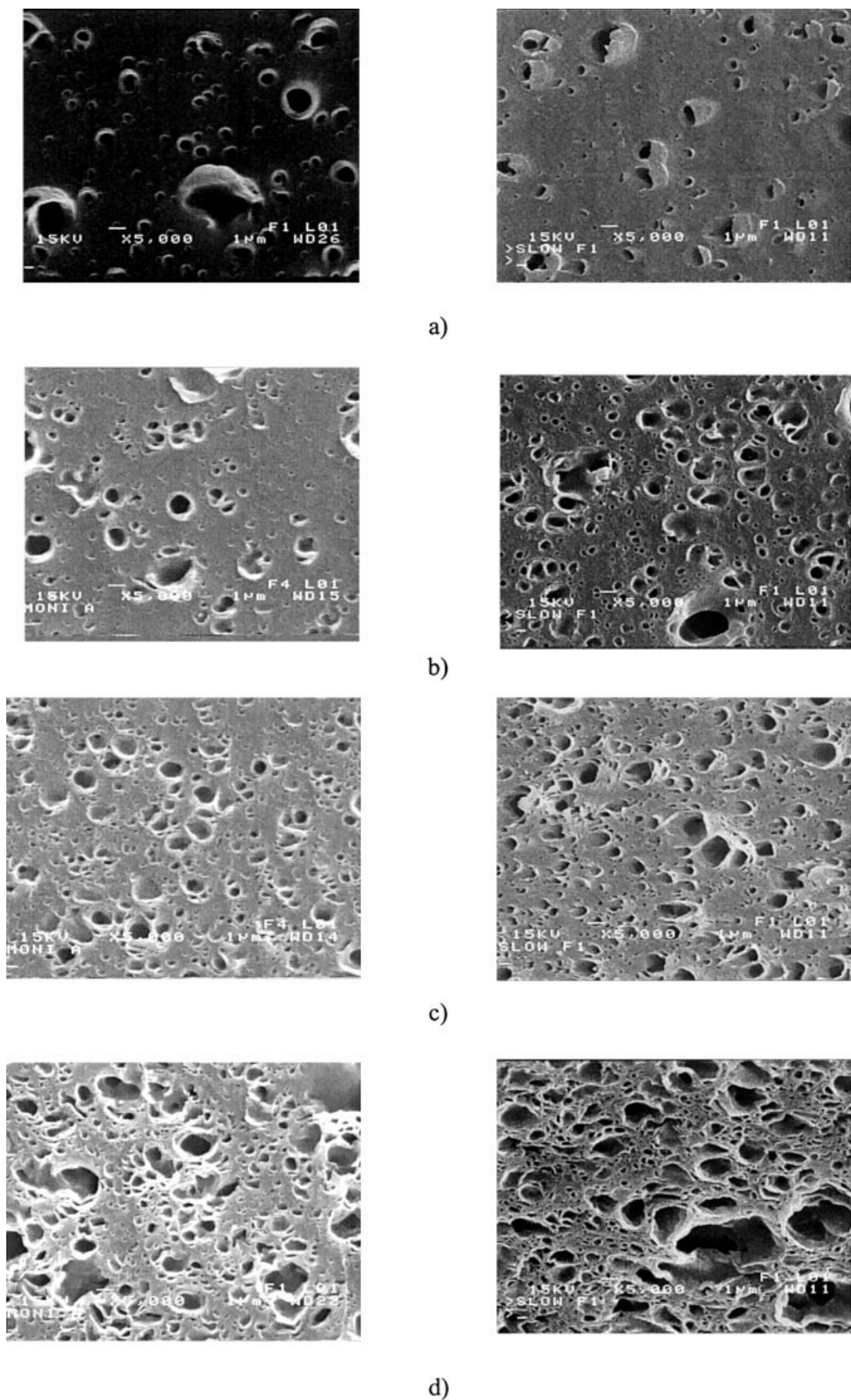
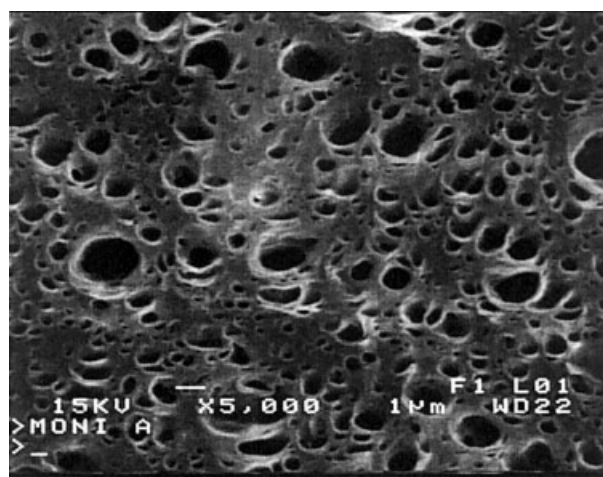
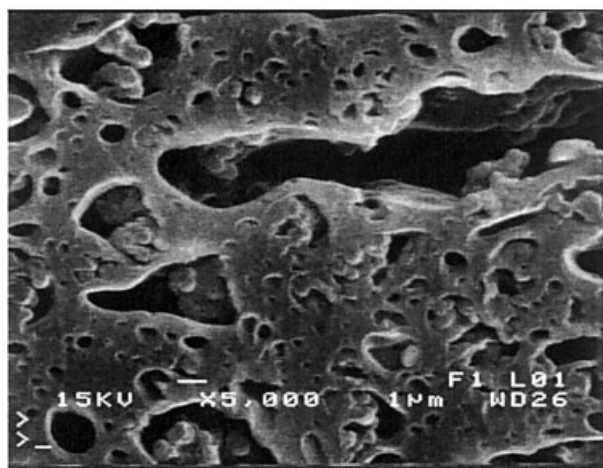


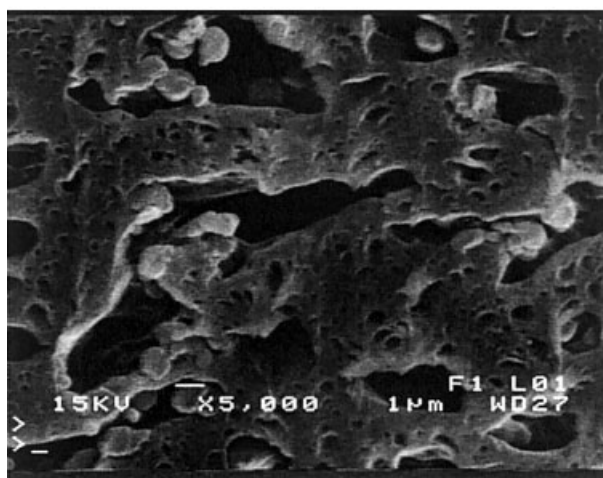
Figure 6 Scanning electron micrographs of blends containing modifier D at (a) 6, (b) 12, (c) 18, and (d) 24 wt %. Left column: transverse direction. Right column: longitudinal direction.



a)



b)



c)

Figure 7 Scanning electron micrographs on transverse cuts of blends containing 24 wt % (a) modifier B, (b) modifier C, and (c) modifier E.

TABLE III
Modifier Number Averaged Diameters

Modifier (wt %)	Particle Diameter (μm)			
	6	12	18	24
A	0.18 ^a	0.63 ^a	0.36 ^a	0.85 ^a
B	0.29	0.45	0.20	0.41
C	0.13	0.31	0.24	Irregular shapes
D	0.36	0.36	0.22	0.21
E	0.20	0.13	0.39	Irregular shapes

^a Fibers observed in the longitudinal direction.

the fracture flank. These features were not observed on pure PET.

The blends containing 24 wt % modifiers C, D, and E, which exhibited the best impact resistance at 20°C, were tested at lower temperatures. The results are presented in Figure 4. Blends with modifiers C and E have their brittle to ductile transition between 10 and 20°C whereas for modifier D the ductility is maintained down to 0°C [Fig. 4(b)]. The brittle-tough transition temperature is often attributed to the glass transition of the elastomeric modifier. In this work, the GMA terpolymers (C, D, and E) have similar glass transitions and therefore the performance difference will have to be discussed in light of the blend morphology.

Blend morphology

The morphologies were characterized for all injection molded specimens used for impact testing. Micrographs were taken in the center of the specimens. The micrographs for the blends with modifiers A (no chemical affinity with PET) and D (containing both GMA and MA) are shown in Figures 5 and 6, respectively. These two series are the most different in terms of toughness, modifier D being the most efficient and modifier A having little or no effect on the impact resistance. The blends were microtomed in both the transverse and longitudinal (i.e., flow) directions. Surprisingly, at 6 wt %, modifier A is better dispersed than the chemically active modifier D. This may be due to viscosity effects as the terpolymer reaction with PET yields branching and even possibly some crosslinking, rendering dispersion more difficult. As the concentration is increased, however, the modifier A phase becomes coarser. In the case of modifier D, the number of dispersed phases obviously increases but their size remains relatively constant. The most striking difference is found when observing the morphology in the longitudinal direction. Modifier A has formed very long fibrils parallel to the flow direction whereas the reactive terpolymer morphology, even at 24 wt %, is nearly isotropic with most particles having a low aspect ratio. This emphasizes the efficiency of this modifier to form a graft copolymer of PET and of

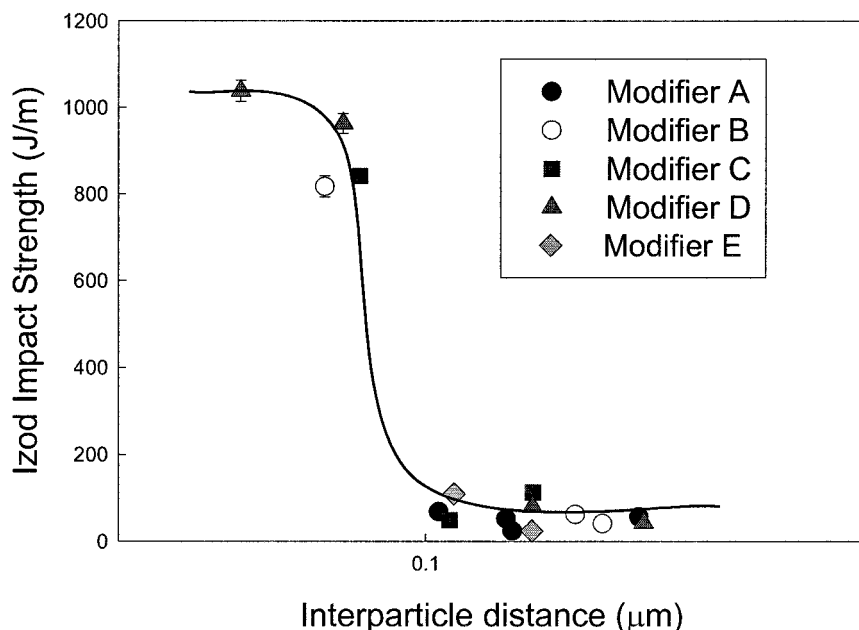


Figure 8 The Izod impact strength as a function of the interparticle distance for blends at 20°C.

the GMA terpolymer that will locate at the interface during blending, reduce the interfacial tension, and minimize coalescence.

Figure 7 presents micrographs for blends containing 24 wt % modifiers B, C, and E taken on a cross-section perpendicular to the flow direction. The morphology of the blend containing 24 wt % modifier B consists of well-dispersed elastomer droplets and it is very similar to the one observed for the blend containing modifier D [Fig. 6(d)]. This type of morphology, which is homogeneous and consists of a discrete dispersed phase, resulted in high impact toughness. Better ultimate tensile properties were also observed, as reported earlier in Figure 2(c,d). In contrast, the blends containing modifiers C and E exhibit a coarse and nonhomogeneous morphology. Large domains of irregular shapes were observed, which could act as defects and explain the significant decrease of the elongation at break and tensile toughness. Despite similar content in the GMA moieties, the morphologies are very different and it seems that the blending of more reactive and inert species is not as efficient as using a low GMA content terpolymer.

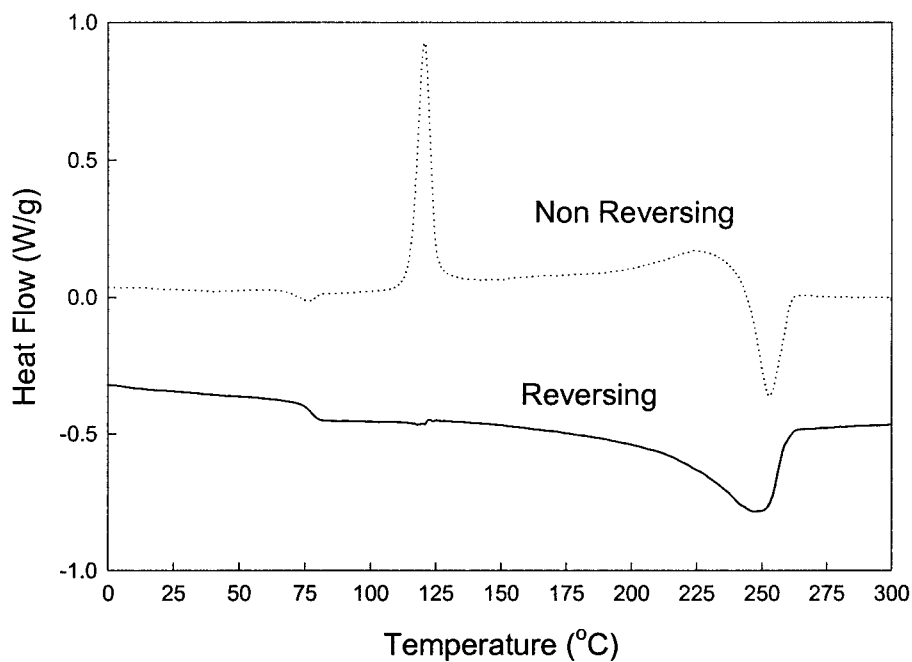
The average particle sizes were determined by image analysis based on micrographs taken on sections perpendicular to the flow. The values are given in Table III. It should be noted that, because of their complex morphology, no attempts were made to quantify the particle size for the blends containing 24 wt % modifiers C and E. The data for modifier A should also be regarded with care because, as reported previously, the observed diameter is that of a fiber, not a sphere. All average particle sizes are below the 1- μm mark. The particle size does not increase systemati-

cally as is usually expected in polymer blends, probably because of the complex flow patterns occurring in injection molding. Larger particles can be deformed more readily into elongated particles or fibrils, thus appearing as smaller features on a perpendicular cross section. This emphasizes the importance of looking at the surface topology using different cross-cuts.

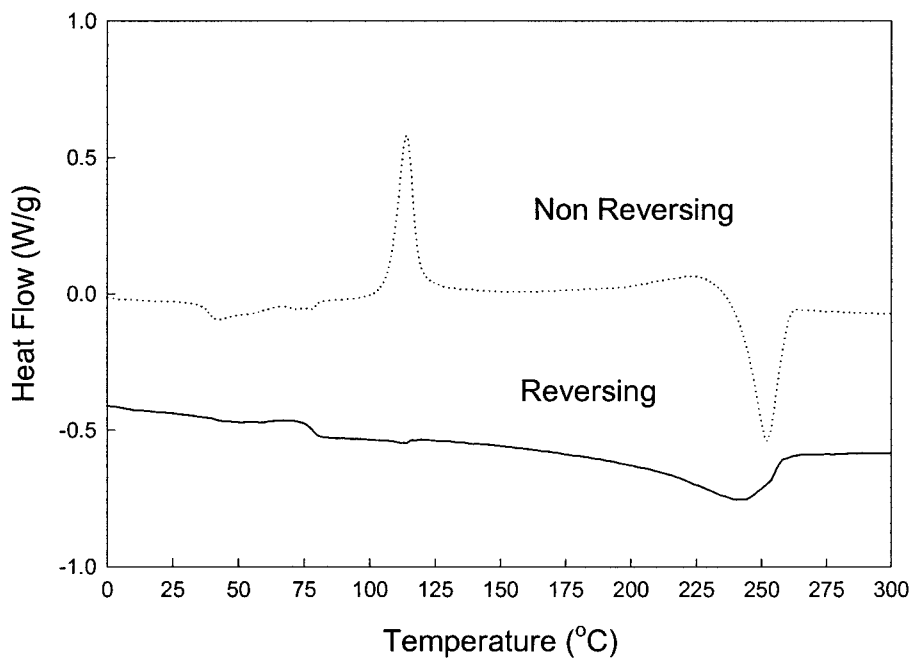
Morphology and impact resistance relationship

The average diameters observed on transverse cuts are all within a relatively narrow range and do not correlate readily with the observed mechanical data. The shape of the dispersed phase however seems to modulate the modifier efficiency. In the case on the nonreactive modifier A, the combination of high aspect ratio fibrils and poor adhesion result in easy paths for crack propagation, leading to catastrophic failure even at 24 wt % modifier. For the other modifiers, all 24 wt % blends exhibited similar room temperature high impact strength (above 600 J/m). At a lower concentration or lower temperature, the isotropic morphology observed with modifier D yields better performance than fibrillar or irregular ones obtained with the elastomer blends. The increasing particle aspect ratio and orientation increase the actual average distance between particles, resulting in an apparent modifier concentration loss.

The critical interparticle distance (or ligament thickness) for the investigated blends has been calculated from the volume concentration (ϕ) and volume averaged diameter (d) of the dispersed phase according to the relation proposed by Wu⁷:



a)



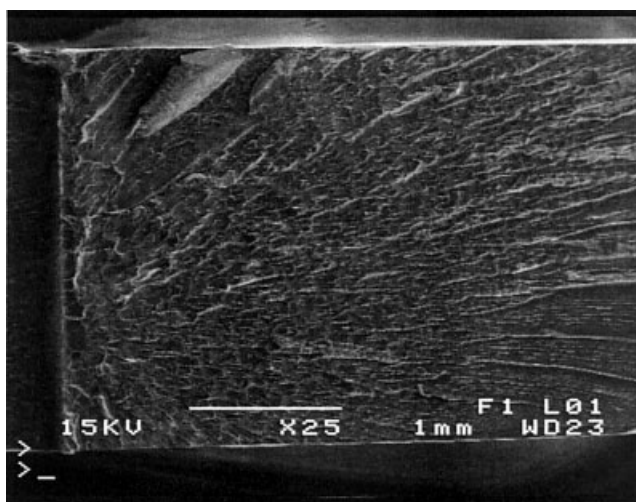
b)

Figure 9 Modulated DSC thermograms of (a) pure PET and (b) a blend containing 24 wt % modifier D.

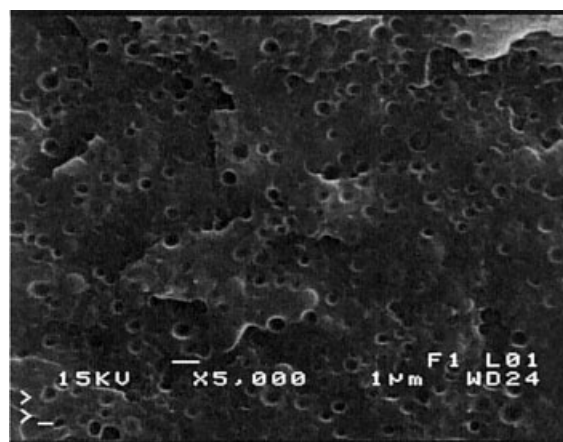
$$\Delta = d \left[\beta \left(\frac{\pi}{6\phi} \right)^{1/3} - 1 \right] \quad (1)$$

where β is a geometric constant equal to 1 for a uniformly distributed simple cubic packing of a sphere.

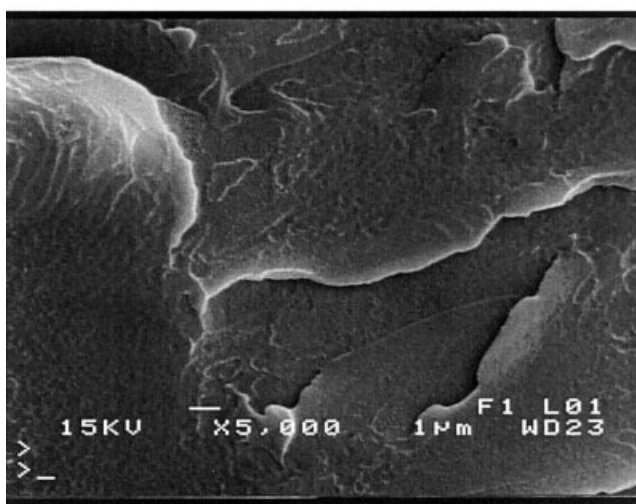
Because there is no simple way to take into account the droplet complex shapes observed at high concentration, eq. (1) was used for calculating the average ligament size; but it should be kept in mind that this does not confer a complete image complex morphol-



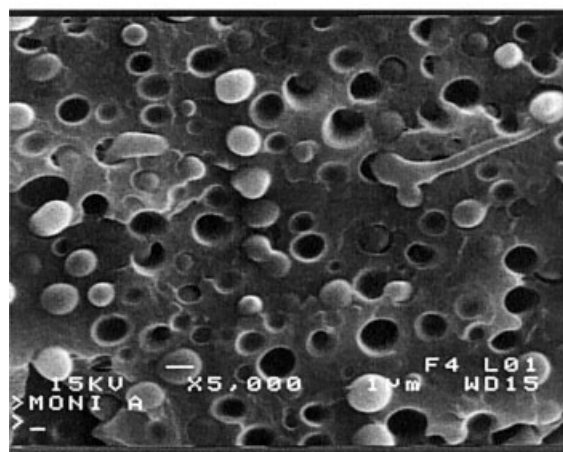
a)



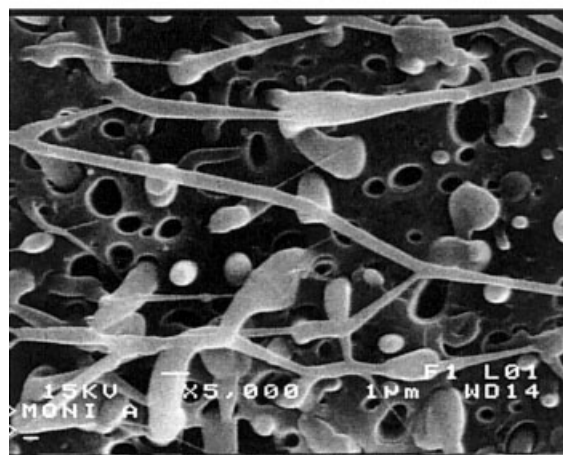
a)



b)



b)



c)

Figure 10 The fractured surfaces of pure PET following Izod impact at 20°C. (a) The notch is on the left side of the picture at original magnification $\times 25$ and (b) the center of specimen at original magnification $\times 5000$.

ogy. Figure 8 shows the dependency of the impact strength on the calculated interparticle distance. Interestingly, all data fall on a single curve. The critical ligament length beyond which the impact properties are poor for all blends is just below $0.1 \mu\text{m}$. To our knowledge, this is the first time the critical interparticle distance is reported for PET. It is smaller than the values reported for polyamide, polypropylene, and PE at 20°C, which are 0.3, 0.15, and $0.6 \mu\text{m}$, respectively,^{7,11,20} and could partially explain why impact modification of PET is not as readily performed. It should be noted that the PET in the blends did not develop

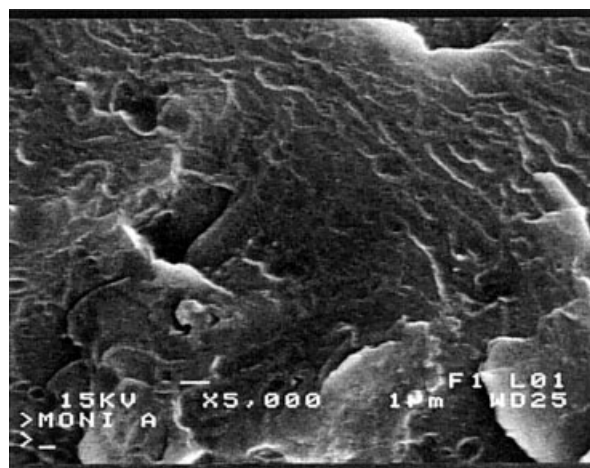
Figure 11 The fractured surfaces of blends containing (a) 6, (b) 18, and (c) 24 wt % modifier A following Izod impact at 20°C. The crack propagates from left to right.

significant crystallinity because of the low mold temperature used for sample fabrication. Figure 9 shows the mDSC thermograms of the pure PET matrix and the blend containing 24 wt % modifier D, which exhibits the highest impact strength. The calculated crystallinities are 0.5 and 1.3% for pure PET and the blend, respectively. Therefore, in this work, crystallographic arguments cannot be used to explain the presence of a critical interparticle distance. In the current blends, the improvement in toughness seems to be correlated with the decrease in yield strength of the material [Fig. 2(b)] and with the blend morphology. Large blend features and lack of homogeneity acted as flaws, decreasing the overall impact performance of the material and resulting in ductility differences, especially for the 18 wt % blends. Most of the theory on impact modification involves monodisperse spherical dispersions. At high concentration, at least for the studied systems, particles adopt irregular shapes and have a wide diameter distribution. Therefore, concepts such as stress overlap and interparticle distance must be rethought.

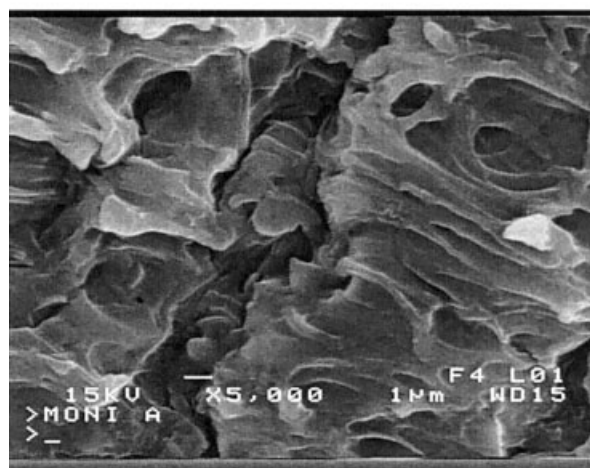
Fracture mechanism

The impact fractured surfaces were investigated by SEM. The pure PET specimens, which exhibited brittle behavior, showed little stress whitening and little deformation, as shown in Figure 10. The same behavior was observed for all brittle blends. The morphology of the blend containing 6 wt % modifier A is shown as an example in Figure 11. There is very little change between the morphological features of the microtomed [Fig. 6(a), left column] and fractured [Fig. 11(a)] surfaces. No deformation of the minor phase has occurred and the diameter of the droplets is similar in both micrographs. In addition, lack of adhesion is obvious, because many particles have been ejected during fracture. The modifier phase of the 18 and 24 wt % blends, which also have poor impact properties, is highly deformed after fracture. These low modulus fibers show no adhesion with the matrix and some were pulled out. The smooth surface of the pure PET matrix indicates no shear yielding occurred.

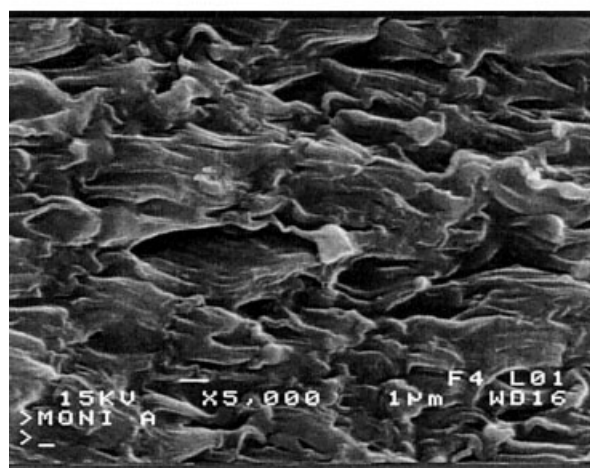
When the blend has tough behavior, cavitation and extensive deformation of the matrix occur. This is shown in Figures 12 and 13 for the blends containing modifiers D and C, respectively. The surface of the blend containing 6 wt % modifier D [Fig. 12(a)] is smooth and is similar to that of pure PET. As the modifier content reaches 18 wt %, the surface becomes rough and strands of highly deformed PET matrix can be observed. The corresponding impact strength then jumps from 43 to 963 J/m. This morphology is similar to those observed on the fractured surfaces of rubber toughened HDPE and nylon.^{10,12} All tough blends exhibited similar features (i.e., a rough surface and/or



a)

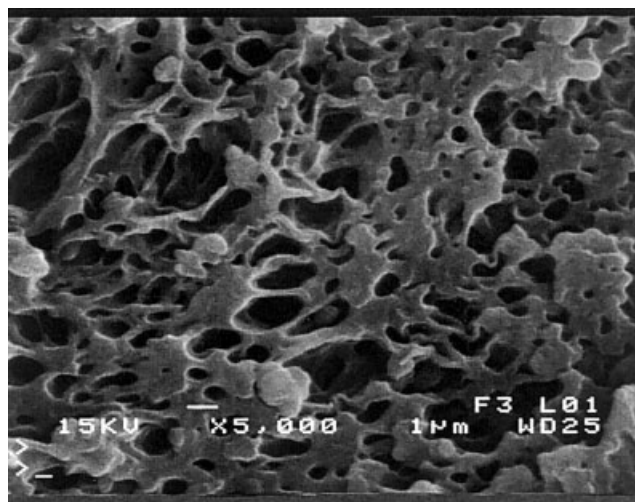


b)

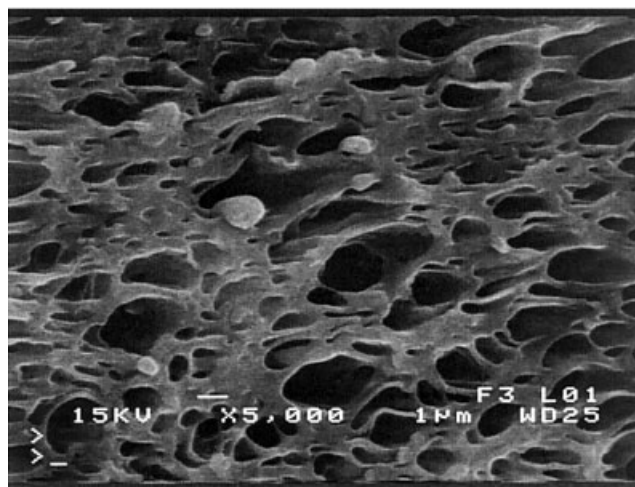


c)

Figure 12 The fractured surfaces of blends containing (a) 6, (b) 18, and (c) 24 wt % modifier D following Izod impact at 20°C. The crack propagates from left to right.



a)



b)

Figure 13 The deformation behavior of a blend containing 18 wt % modifier C as a function of position on a fractured specimen (a) near the notch and (b) at the center of the process zone. The crack propagates from left to right.

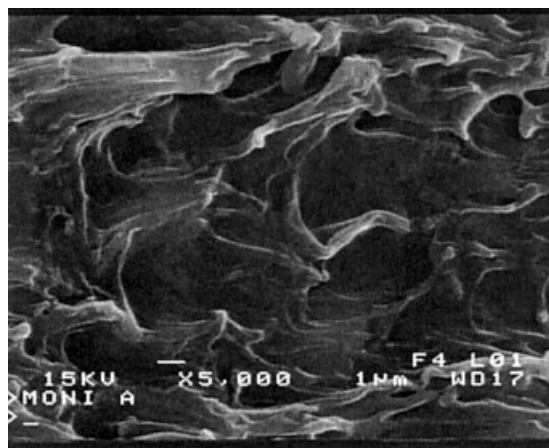
highly deformed matrix ligaments between the minor phase domains). Some differences in morphology were observed, depending on the position on the fractured surface. An example for the blend containing 18 wt % modifier C is given in Figure 13. Highly deformed strands of the matrix are present in the center of the specimen [Fig. 13(b)], a phenomenon less significant near the notch [Fig. 13(a)]. These differences could be attributed to variations of the crack velocity and/or adiabatic heating.

Figure 14(a,b) shows the surface of blends containing 24 wt % modifier D fractured at 0 (ductile) and -40°C (fragile). Again, the surface morphology for the ductile failure exhibits extensive deformation whereas the lower temperature fragile fracture surface is sim-

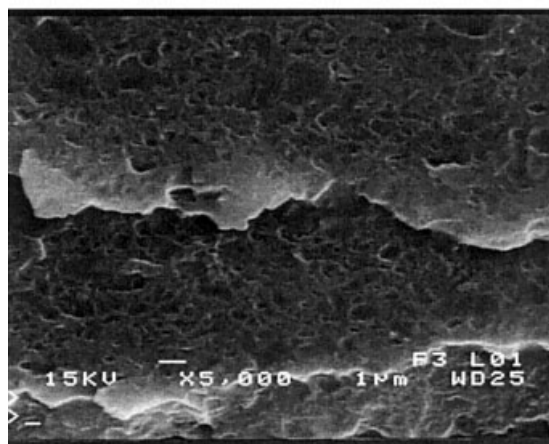
ilar to those observed for brittle specimens tested at 20°C.

CONCLUSIONS

The mechanical and morphological properties of a PET matrix blended with several modifiers were investigated. Adding the modifiers to PET resulted in a decrease of the tensile modulus and tensile strength, whereas the elongation at break and toughness were generally increased compared to pure PET. Impact toughening was obtained only in blends where interface modification was carried out using either grafted or randomly copolymerized GMA moieties. The brittle to tough transition occurred at a lower temperature and lower concentration with the modifier that showed the most uniform and isotropic morphology. The correlation between the brittle-tough transition



a)



b)

Figure 14 The effect of temperature on the fractured surfaces of a blend containing 24 wt % modifier D at (a) 0 and (b) -40°C. The crack propagates from left to right.

and the interparticle distance was shown. At room temperature, the critical ligament size below which ductile behavior occurs was around 0.1 μm . Finally, the investigation of the fracture surfaces of the blends by SEM showed debonding, cavitation, and extensive yielding of the matrix in toughened blends.

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