

Mechanical and Thermal Properties of Toughened Polypropylene Composites

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ABSTRACT: The mechanical, thermal, and structural properties of a new flexible composite containing polypropylene fiber (PP) in a random poly(propylene-co-ethylene) (PPE) matrix with ethylene-propylene elastomer (EP) was investigated with emphasis on the effect of EP elastomer concentration. The intrinsic composition of the composites, toughening of the matrix with EP and the fiber-matrix interface determined the properties of the composites. Through the incorporation of EP elastomer into the polypropylene-poly(propylene-co-ethylene) (all-PP) composite, tensile and storage modulus (E') decreased, flexural modulus and loss modulus (E'' , damping) increased slightly to 0.15 EP and then decreased. There was an increase in impact resistance for the toughened composites, with about 100% increase in

comparison with an untoughened all-PP composite. The composition corresponding to 0.20 weight fraction EP gave optimum impact and mechanical properties. Creep resistance of the composite decreased with increasing EP content, but recovery showed an increase with increasing EP content up to 0.20. Fracture surfaces of composites after impact tests were studied with scanning electron microscopy. Moreover, the use and limitation of theoretical equations to predict the tensile and flexural modulus of the flexible PP composite is discussed. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 390–397, 2007

Key words: toughness; blends; composites; poly(propylene); morphology

INTRODUCTION

A wide market demand for recyclable, inexpensive, and conveniently processable engineering plastics has made polypropylene (PP) an attractive polymer. Unfortunately, its applications as an engineering thermoplastic are limited because of its poor stiffness and impact strength.^{1–3} This gap between commodity PP and engineering thermoplastics can be filled when effective reinforcement is incorporated in PP such as PP fibers, which give excellent properties without environmental and ecological problems.^{4,5} However, PP reinforcement is accompanied by a reduction in impact toughness. For improved impact toughness and an extension of its application range, much research has been undertaken on PP toughening.^{6–8} Several types of rubber, such as ethylene-propylene (EP) rubber and EP-diene monomer (EPDM) rubber, have been chosen to toughen PP, but EP is the most common. The toughening efficiency on PP was found to depend on the type of rubber, its content, and the operating toughening mechanisms. The physical properties of a polymeric material are dependent on morphology, structure, and relaxation processes corresponding to internal changes and molecular motions.^{7–9}

All engineering polymer applications involve some degree of mechanical loading. Different types of composite are often compared based on their tensile modulus, strength, and elongation. Dynamic mechanical measurements over a wide temperature or load range are useful in characterizing the viscoelastic behavior and provide valuable insights into relationship between structure, morphology, and application properties of polymeric and composite materials.^{10–13} Several studies on fiber-reinforced PP composites based on structure-property relationships by means of dynamic and static stress mechanical analysis have been reported in the literature.^{3,14} However, mechanical and thermal investigations of different aspects on flexible PPE (with EP elastomer) reinforced with PP have not been reported.

The purpose of this work is to investigate the effect of phase morphology, EP and PP fiber inclusion concentrations and interfacial adhesion on the toughness and mechanical properties of all-PP composites. The elastomer (EP) concentration in the composites was optimized. The experimental mechanical properties were predicted using existing theoretical models.

EXPERIMENTAL

Materials

The materials employed in this investigation were random poly(propylene-co-ethylene) matrix (PPE) (density, $\rho = 0.905$, MFI = 0.8 dg/min, melting tempera-

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ture, $T_m = 147.5^\circ\text{C}$, ~ 0.05 wt ethylene, Basell Australia Pty), ethylene-propylene elastomer ($\rho = 0.89 \text{ g cm}^{-3}$, MFI = 2 dg min^{-1} blended with polyethylene to provide extrudable pellets, Exxcelor elastomer from Qenos-Exxon Pty, Australia) and PP fiber (diameter = $50 \mu\text{m}$, tensile modulus = $5.1 \pm 0.3 \text{ MPa}$, formed into a nonwoven mat using a laboratory needle punch machine at Melded Fabrics Pty, Melbourne, Australia). The polypropylene nonwoven fiber mat was washed with acetone to remove any processing lubricants. Fiber diameters were measured using optical microscopy after calibration with a standard graticule slide. The fibers were treated under the same conditions as used for composite preparation to show the effect of these conditions on the fiber stiffness, but in this case, the fibers were clamped between the two clamps (to allow for similar thermal relaxations).

Composite preparation

PPE and EP elastomer with varying EP composition from 0.05 to 0.30 weight fraction were compounded in a Brabender twin-screw extruder fitted with a slit die and chilled roller set, to prepare films of the PPE-EP blends. The Brabender twin-screw extruder was used with barrel temperatures of 180°C with die temperature at 160°C . Blend composition pellets were mixed manually and thoroughly before being fed into the extruder hopper.

The composites were prepared using a heated hydraulic press. The processing temperature is important for flexible PP composites to maximize the fiber-matrix adhesion and to keep the original fiber morphology, because the reinforcement and the matrix are of similar materials. Choosing the conditions for the composite preparation was based mainly on the information obtained from differential scanning calorimetry (DSC) experiments,^{15,16} considering that the processing temperature should be higher than the melting temperature of the PPE (147°C , measured by DSC) and lower than the melting temperature of the fibers (164°C). At higher temperatures, the degree of crystalline orientation of the fibers will decrease and the fiber properties would deteriorate. A temperature of between $(152 \pm 5)^\circ\text{C}$ was used. Because of a lower degree of orientation, most of the relaxation was expected to occur in the fiber skin, rather than in the fiber core, resulting in relaxation or partial melting. This in turn produced favorable conditions for bonding at the fiber-matrix interface. The heated press method used for composite preparation consisted of two stages. In the first stage, PP nonwoven fiber mat was placed on top of a film of the blend of PPE and EP ($\sim 0.2\text{--}0.4 \text{ mm}$ thickness) and placed between two Teflon sheets, then pressed at $(152 \pm 5)^\circ\text{C}$ for 5–7 min. After that, an 11–14 kPa pressure was applied for 8–10 min. In the second stage, three layers of the composite pre-

pared according to the previous stage were laminated together to provide a composite with a more random fiber distribution and uniform composition. The resultant PP composite was cooled from the molding temperature to room temperature over 5 min. Three panels of each type of composite were prepared with a fiber volume content of 0.50.^{15,16}

Differential scanning calorimeter

A differential scanning calorimeter (DSC, PerkinElmer Pyris1) was used for thermal analysis of the pure polymer, fibers, and composites. Samples of about 3 mg were sealed in 10- μL aluminum pans. A constant nitrogen flow of 40 mL min^{-1} was used to purge the instrument. The samples were held at 30°C for 2 min, then heated from 30 to 180°C at $10^\circ\text{C min}^{-1}$, held at 180°C for 2 min, cooled to 30°C at the same rate and held for 2 min. A second heating scan to 180°C was then performed. The first heating scan melted both the matrix and the fibers. T_c was measured from the peak of the exotherm during cooling. The second heating cycle provided results that were more consistent for the melting temperatures (T_m) measured from the peak of the endotherms. The instrument was calibrated for temperature using indium and zinc, for enthalpy using indium, and a furnace calibration was performed according to the manufacturer recommendation. A conditioning scan was performed before any data collection scans. A baseline with matched empty pans was used to convert the data to apparent specific heat to allow direct comparison of all curves. In the specific heat convention, both the endotherm on heating and the exotherm on cooling are shown as positive. The overall crystallinity (X_c) of the composites was determined from the enthalpy of the PP endotherms or exotherms using a value of $\Delta H_f^0 = 209 \text{ J g}^{-1}$ for pure αPP crystals. The pure PP fibers melted at $(165.7 \pm 0.9)^\circ\text{C}$ and had a crystallinity of $(0.47 \pm 0.3)^\circ\text{C}$; crystallization was at $(124.5 \pm 1.6)^\circ\text{C}$ with crystallinity of 0.43 ± 0.06 . It was assumed that the PP fibers and the PPE matrix formed the same crystal morphology. For clarity of presentation; successive curves have been shifted by 5 units in Figure 1.

Static mechanical analyses

The static mechanical properties were determined from six measurements of each composite with a PerkinElmer DMA 7e in extension and three-point bend modes. The static force ranged from 100 to 8000 mN at 100 mN min^{-1} and 0.0 to 6400 mN at 400 mN min^{-1} for three-point bend and extension modes respectively. The maximum displacement can be measured with this instrument is $\pm 5 \text{ mm}$. The composite samples were cut along a range of orientation on the sheets, to provide dimensions of $1 \times 12 \times 5 \text{ mm}^3$ and 1×10

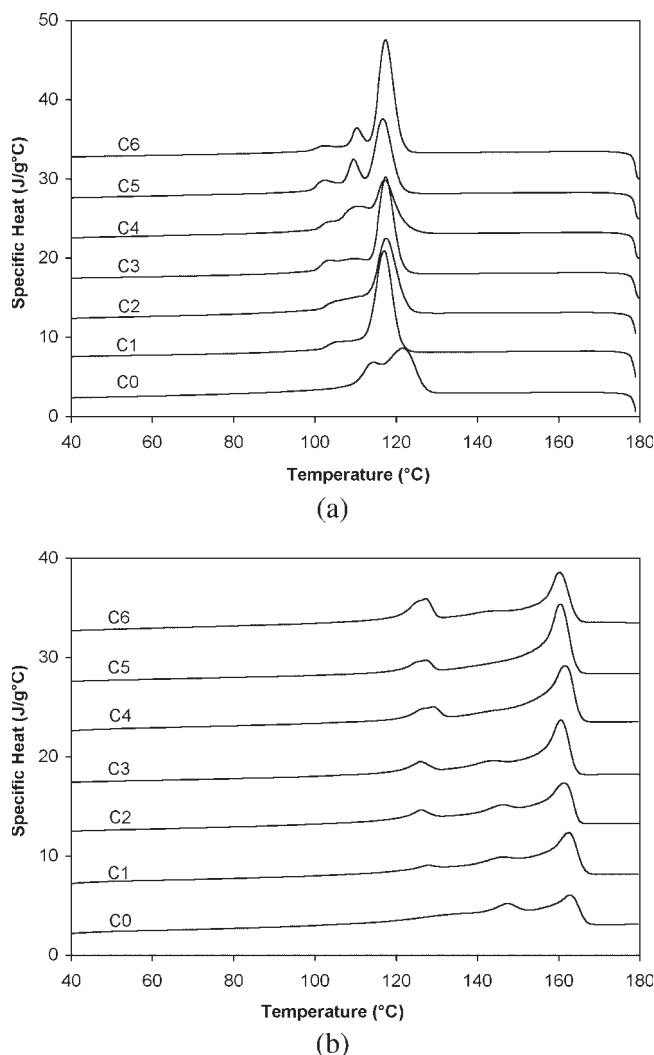


Figure 1 DSC (a) melting and (b) crystallization of polypropylene fiber-matrix composites with various EP concentrations.

$\times 5 \text{ mm}^3$ for three-point bend and extension modes respectively. The sample dimensions were kept as similar as possible to obtain a reliable comparison between the mechanical properties from different composites. The instrument was calibrated for force using a standard mass and distance using a standard steel block.

Dynamic mechanical analyses

Dynamic mechanical analysis (DMA) was performed in three-point bend mode using a PerkinElmer DMA 7e with an Intercooler II. A dynamic force of 50 mN and static force of 125 mN was used with a frequency of 10 Hz and the temperature scan ranged from -50 to 100°C at 2°C min^{-1} . A constant nitrogen flow of 40 mL min^{-1} was used to purge the instrument. The samples were cut from the previously prepared sheets, with the dimensions $1 \times 12 \times 5 \text{ mm}^3$. The sample dimensions were kept as similar as possible to obtain a reliable

comparison between the results from different composites. Storage and loss moduli were recorded as a function of temperature. The instrument was calibrated for temperature using indium, force using a standard mass; probe position, furnace, and eigenvalue calibrations were also performed.

Creep test

Creep recovery was recorded with a Rheometric DMTA VI in dual cantilever mode. All experiments were performed at 25°C . The samples were cut from the sheets with dimensions $1 \times 10 \times 5 \text{ mm}^3$. A stress of 10 MPa and recovery stress of 1 MPa were used with a creep time of 1200 s and a recovery time of 600 s. Each experiment was repeated four or five times and the average results were reported. The instrument was calibrated for force using a standard mass.

Impact tests

Impact testing of notched composite specimens was carried out using a Davenport Izod impact instrument, with notch tip radius of 0.25 mm. The incident kinetic energy was 1.36 or 4.07 J by varying the mass of the pendulum. Specimens were cut from the composites and matrix polymer into rectangular-shapes, to provide dimensions of gauge length 12 mm, width 7 mm, and thickness 3 mm, measured by a digital caliper (Mitutoyo Absolute Digimatic model). Seven measurements were carried out for each material; estimated error was about ± 0.05 .

Fractography

The images of the specimen after the impact test are recorded by using an FEI Quantum 200 Scanning Electron Microscope (SEM). SEM analyses were performed in a low-voltage mode (LVSEM).

RESULTS AND DISCUSSION

DSC measurements

The melting and crystallization behavior of all-PP composites with and without EP elastomer were studied and the DSC curves are shown in Figure 1. It can be seen that the melting and crystallization behavior of the composites with and without EP are almost the same except for additional small melting and crystallization peaks at 126.4 and 111.5°C for the composites with EP. These peaks are due to the polyethylene blended with EP in Exxcelor type elastomers. A triplet endotherm and exotherm phenomenon was observed in data before melting or crystallization at 160°C and 120°C . As EP concentration increased, the third of the three peaks gradually appeared; finally, three distinct

TABLE I
Thermal Properties of Polypropylene Fiber–Matrix Composites with Various EP Concentrations

Sample	Designation	EP fraction	T_{C1} (°C)	T_{C2} (°C)	T_{C3} (°C)	X_C	T_{M1} (°C)	T_{M2} (°C)	T_{M2} (°C)	X_M
PPE	PPE	–	116.2	–	–	0.36	–	147	–	0.38
EP	EP	–	111.5	–	–	0.33	126.4	–	–	–
Composite	C ₀	0	–	114.5	121.6	0.45	–	147.1	162.7	0.43
	C ₁	0.05	105.2	–	117.7	0.46	128.0	146.8	160.7	0.50
	C ₂	0.1	104.8	–	117.6	0.50	127.9	147.4	162.2	0.58
	C ₃	0.15	104.2	–	117.6	0.50	127.6	146.1	161.9	0.54
	C ₄	0.2	103.5	110.6	117.2	0.46	127.5	146.0	161.8	0.43
	C ₅	0.25	102.8	109.7	116.9	0.49	128.6	145.4	162.0	0.49
	C ₆	0.3	102.6	110.0	117.5	0.51	128.7	–	161.7	0.53

peaks were observed and this was at a maximum for composites with maximum EP concentration.

The distinct melting temperatures of the matrix and fibers confirm that the PP in the matrix and fibers remained as separate phases. The crystallinity of the composites especially controlled the mechanical properties.^{17,18} According to Figure 1(a), the higher melting peak was influenced by EP concentration, indicating that some crystallinity of PPE may be induced by the presence of EP particles. On addition of 0.10 EP, T_c decreased, but T_m remained the same, while crystallinity increased by about 6%. Overall, the results showed no significant shift in the melting and crystallization temperatures with change in EP. The crystallinity varied slightly with different EP concentrations. The results indicated that change in EP concentration did not significantly affect the crystallization of PPE. Thus, the crystallinity of the PPE component approximately remained unchanged and independent of EP concentration. The crystallinity, X_c , melting temperature, T_m , and crystallization temperature, T_c , are recorded in Table I.

Mechanical properties

Static mechanical properties

Figure 2 shows the tensile and flexural modulus for the different EP concentrations and the mechanical parameters are shown in Table II. The results show that the tensile and flexural properties of the composites were dependant upon EP concentration. The addition of EP elastomer decreased the tensile modulus, but increased elongation at higher stress. The toughened composites show a higher deformation and they have lower tensile strength than the all-PP composites without EP elastomer. The stress–strain curves of composites with EP undergo a short elastic deformation stage when compared with a long plastic deformation stage. Between 0.10 and 0.15 EP content, the flexural modulus and tensile modulus decrease incrementally. It is likely that in this concentration range the critical liga-

ment distance between EP particles was reached, facilitating matrix phase shear deformation to relieve stress.

An increase in EP concentration led to a sharper decrease in tensile modulus, and composites with high

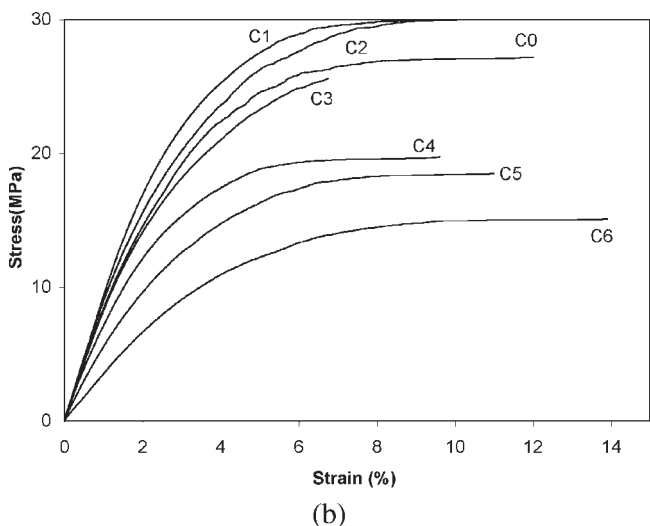
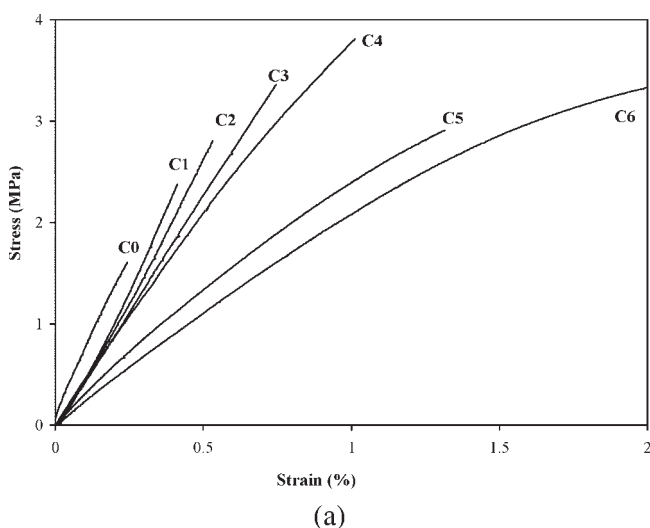


Figure 2 Stress–strain curves used to obtain (a) initial tensile modulus and (b) flexural stress–strain curve of the composites with various EP concentrations.

TABLE II
Experimental and Theoretical Tensile Modulus for the Composite
with Various EP Concentrations

Sample	EP fraction	Designation	Flexural modulus at 25°C (GPa)	Tensile modulus at 25°C (GPa)	
				Experimental	Theoretical
Composites	0	C ₀	1.01	0.89	1.05
	0.05	C ₁	1.05	0.59	1.01
	0.10	C ₂	1.10	0.53	1.00
	0.15	C ₃	0.95	0.42	0.99
	0.20	C ₄	0.87	0.39	0.97
	0.25	C ₅	0.64	0.32	0.96
	0.30	C ₆	0.45	0.27	0.95

EP concentration showed high plastic deformation. The effectiveness of EP on the tensile modulus of the composites can be represented by the rule of mixtures. While,

$$E_m = V_e E_e + V_p E_p \quad (1)$$

$$E_c = \eta_1 \eta_0 V_f E_f + V_e E_e + V_p E_p \quad (2)$$

where e , p , f , m , and c represent EP elastomer, PPE, PP fiber, matrix, and all-PP composite, respectively.^{19,20} The value of $\eta_1 \eta_0 V_f E_f$ is constant for all composites but varying EP concentration in the matrix varies $V_e E_e$ and $V_p E_p$. The values obtained for different systems at a frequency 10 Hz are given in Table II. In this case, the lowest value of E_m has been obtained for the composite with 0.30 EP elastomer and the highest value of E_m for 0.00 EP. The effects of EP on the composite are (a) debonding between fiber and matrix, (b) breakage of the matrix resulting in a high energy absorption level. Thus, the EP composite materials possess higher energy absorption during deformation, compared with all-PP composites without EP.

The flexural properties showed the same behavior, except that the composites with EP showed more deformation with low load. The flexural modulus of the composites increased with low EP concentration in the composite to 0.10 EP, with 0.2 EP being still greater than that of the composite with no EP [Table II,

Fig. 2(b)]. In this range of low EP concentration, the increased crystallinity of the matrix overcame the flexibilizing effect of the EP. Then, there was a reduction in the flexural modulus when EP concentration exceeded this critical value for ligament distance. Increasing EP to 0.20 resulted in an increase in the flexural modulus by about 10%, but composites with more than 0.20 EP showed a decrease, for example, the composite with 0.30 EP had 50% of the flexural modulus of the composite without EP, because at this concentration PPE and EP phases may become cocontinuous. This was because the composites can then absorb more energy, but composites with more than 0.20 EP also became softer due to the rubbery behavior of the elastomer in the matrix. It is expected that blends of PPE and EP would have decreased flexural modulus, but in the composites there was an increase in the crystallinity of PPE that would increase the rigidity.

Dynamic mechanical properties

The dynamic moduli of a composite depend on matrix and reinforcement type. Dynamic mechanical properties were measured and results are listed in Table III. Figure 3(a,b) illustrates the storage and loss modulus of the composites as a function of temperature at a frequency of 10 Hz for all-PP composites with different EP concentrations. It is interesting to note that E' showed a sharp decrease on introduction of EP into

TABLE III
Mechanical Properties of Polypropylene Fiber–Matrix Composites with Various
EP Concentrations (Storage and Loss Modulus at 25°C, T_g)

Sample	Designation	EP fraction	Storage modulus at 25°C (GPa)	Loss modulus at 25°C (GPa)	T_g (°C)
Composite	C ₀	0	1.79	0.24	5.2
	C ₁	0.05	1.49	0.28	3.9
	C ₂	0.10	1.29	0.32	2.9
	C ₃	0.15	1.30	0.27	1.5
	C ₄	0.20	1.27	0.22	-3.3
	C ₅	0.25	0.98	0.18	-3.5
	C ₆	0.30	0.77	0.16	-3.6

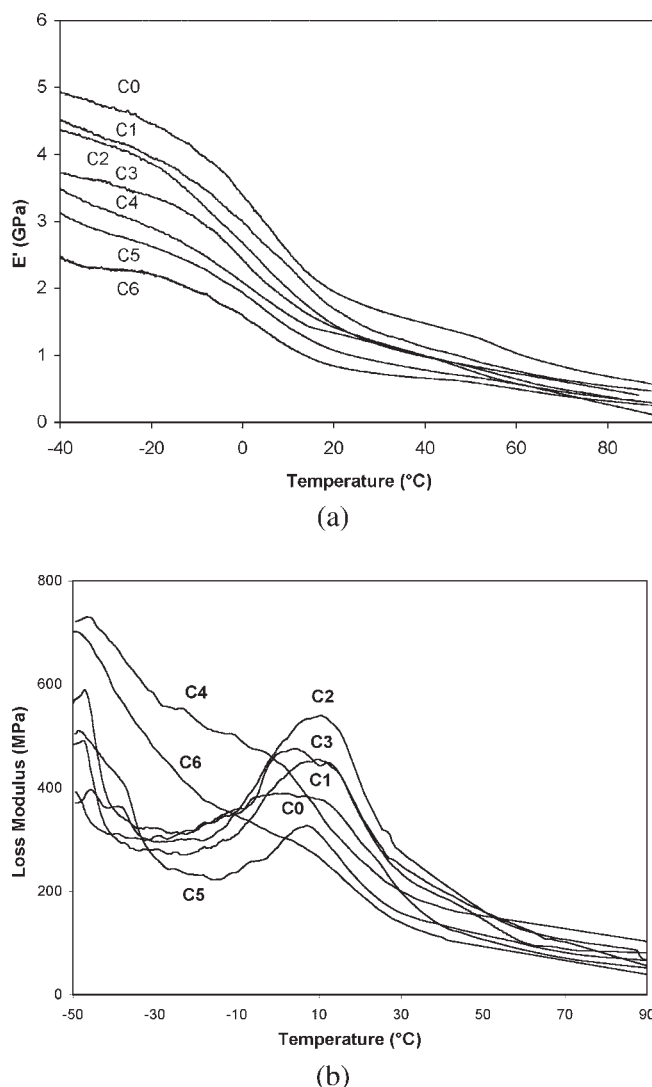


Figure 3 Dynamic mechanical analysis of polypropylene fiber-matrix composites with various EP concentrations using three-point bend configuration, (a) storage modulus, (b) loss modulus.

the matrix with an addition of 0.20 EP rubber, followed by a leveling beyond 0.030 EP. Increasing EP concentration provided further reduction, so that for the matrix with 0.30 EP, the reduction was about 45%. This means EP leads to an increase of the mobility of the macromolecular chains. The stiffness at high temperature was determined by the amorphous region, which was compliant above the relaxation transition temperature. However, the drop in the modulus on passing through the glass transition temperature was higher in case of composites containing high EP elastomer concentration. Composites with high EP concentration at room temperature showed lower storage modulus in comparison with the other systems.

The loss modulus (E'') is indicative of the ability of the material to dissipate mechanical energy, is proportional to viscoelastic behavior and sensitive to molecu-

lar motions. E'' is a measure of the energy dissipated or lost as heat per cycle of sinusoidal deformation, when different systems are compared with the same strain amplitude.^{1,15,20-23} The loss modulus provides much information on the overall flexibility and interactions between the components of composite materials. Figure 3(b) shows the variation of loss modulus as a function of temperature for the systems with different EP concentration at frequency 10 Hz. The plasticity of EP composites at room temperature was slightly higher than the composite without EP, due to the increased crystallinity of the matrix. The loss modulus peak temperatures, compared with the composite without EP matrix, increased with increase of EP concentration up to 0.20 and then decreased, where the rubbery nature of the EP elastomer overcame the crystalline properties of the PPE matrix and an increased mobility of the matrix became apparent. This means the composites with EP absorbed more energy. However, more than 0.20 EP in the composites made the composites more flexible as the critical ligament distance of EP was exceeded. The E'' curve of the composites with more than 0.20 EP became flatter and extended to lower temperature.

Creep behavior

The deformation and creep response as a function of time for composites with different EP concentration is shown in Figure 4. The creep behavior of the composites is a combination of several factors; EP in the matrix acts by increasing the matrix mobility of the composite and molecules can move more easily causing more deformation at lower stress. The systems appear to follow the general additively rule.^{1,24} As the EP concentration was increased, the response became dominated by the EP elastomer. The creep properties of the composites are reported in Table IV.

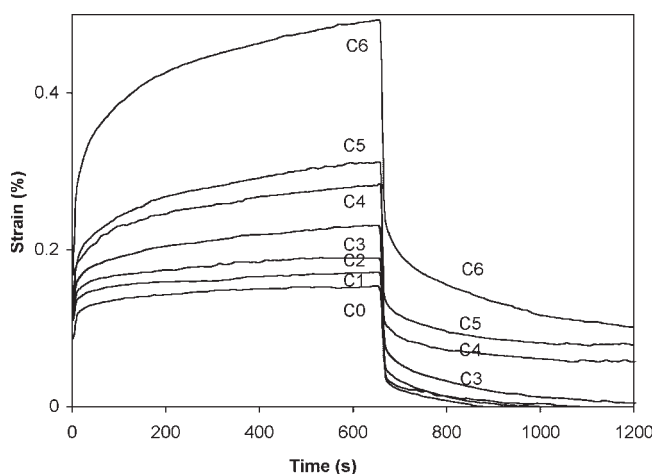


Figure 4 Creep response at room temperature of the composite with various EP concentrations.

TABLE IV
Creep Properties of the Composite with Various EP Concentrations, Modulus, Viscosity, Shear Viscosity, and Unrecoverable Strain at Room Temperature

Sample	EP fraction	Designation	Modulus (MPa)	Viscosity ($\text{Pa s}^{-1} 10^{-12}$)	Shear viscosity ($\text{Pa s}^{-1} 10^{-12}$)	Recoverable strain (%)
Composite	0	C ₀	0.73	2.48	0.83	0.95
	0.05	C1	0.64	3.28	1.09	0.98
	0.10	C2	0.59	5.83	1.94	0.99
	0.15	C3	0.43	7.29	2.43	0.93
	0.20	C4	0.42	7.38	2.46	0.78
	0.25	C5	0.40	8.75	2.92	0.74
	0.30	C6	0.26	1.55	0.52	0.72

Impact properties

Figure 5 shows the impact resistance as a function of EP concentration. Overall, the data show similar trends to results previously observed for stiffness. As can be seen in Figure 5, the relative impact strength shows an increase with respect to that of the matrix. Elastomer-toughened polymers provide stress redistribution in the composite, thereby resulting in a more efficient energy dissipation mechanism including matrix phase shearing. By increasing the EP concentration, the impact resistance increased due to the reduction in the ligament distance between rubbery EP particles. By increasing EP to 0.30 there was a sharp increase in impact strength observed for the composite; because below a critical ligament distance the rubber increased energy dissipation in the matrix. While by increasing EP to more than 0.30, the impact resistance showed only a small further increase.

Figure 6 shows scanning electron micrographs of the fracture zone of a broken impact specimen, with the pendulum and crack growth direction from the left. It can be seen that most of the damage is on the left of

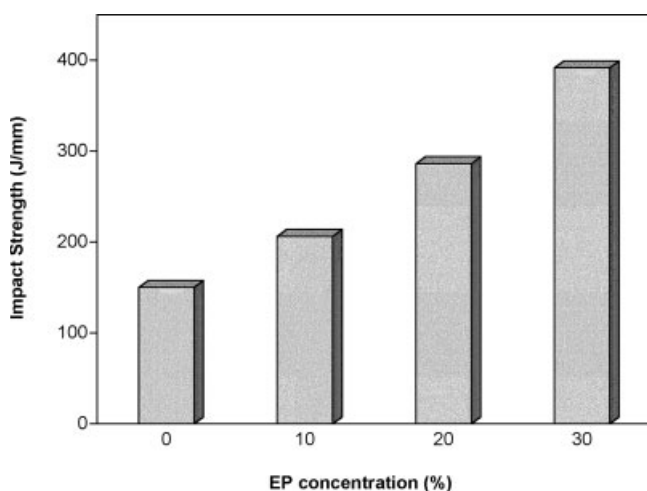
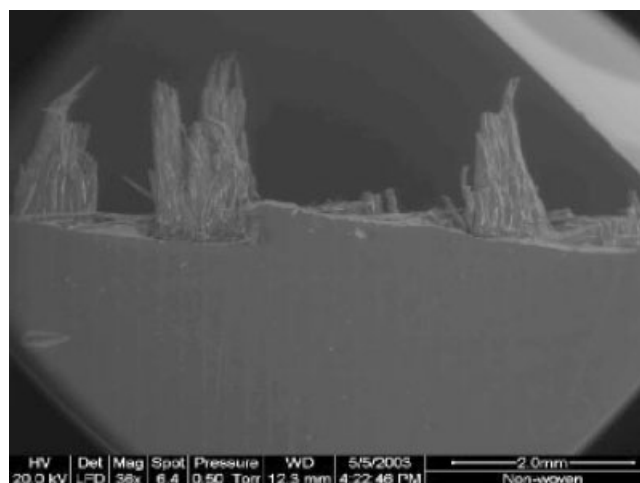
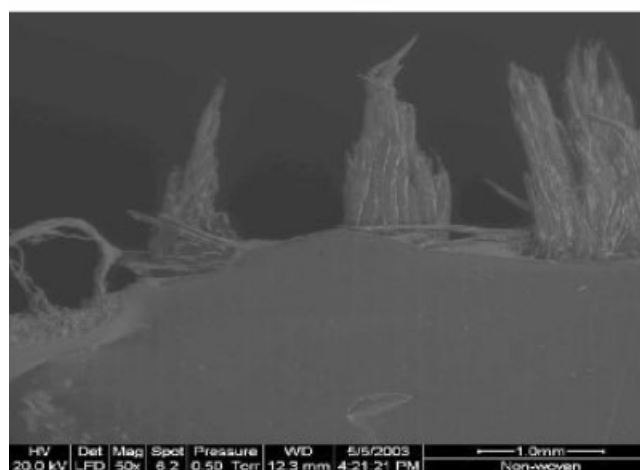


Figure 5 Impact strength of composites with different concentrations of elastomer.

the composite where the pendulum struck, and the regions away from the impact area are less damaged; they appear as though they have been cleanly shear fractured. Figure 6 shows the broken and cut fibers at



(a)



(b)

Figure 6 Scanning electron microscopy of fractured specimens after impact test, (a) 0.10 EP and (b) 0.20 EP.

the middle, fiber pull-out can be seen on the fracture planes. Residual stresses at the interface resulted in an easier fracture path and the crack could disperse energy along interfaces. This created more surface area within the composites and increased the energy absorbed during the impact. When the crack tip approached the reinforcement, there are two possibilities. First, the crack will cross the fibers and break them as well as the matrix; second, the crack will change its direction and move through the matrix parallel to the fibers. The crack proceeds by the path of least energy, which for this composite is passing through the fibers. Thus, it can be concluded that breaking the fibers is an easier mechanism for crack propagation than crossing the energy barrier for debonding. In Figure 6, a small amount of fibers were pulled from the matrix, though all of them were coated with polymer. In this case, there was shearing of the matrix as well as fiber tensile or shear failure modes and considerable fiber rupture during the impact event seems to be the dominating failure mechanism.^{1,22-25}

CONCLUSIONS

The mechanical properties of toughened flexible composites were dependent on the EP concentration in the matrix. EP, which can dissipate energy, enhanced toughness of the composites, while the PP fiber and PPE matrix primarily provided the stiffness and tensile strength. Although flexural strength and loss modulus increased with increasing EP concentration, up to 0.20, the tensile and storage modulus, and creep resistance decreased. This behavior changed for composites with more than 0.20 EP, because the elastomer interparticle distance was decreased. The results showed that impact resistance increased by introducing EP into PPE, due to the ability of EP to provide energy dissipation and facilitate matrix phase shear when the elastomer particles were at or below a critical ligament distance. The optimum composition was observed for a

composite with 0.20 EP, which was chosen as the critical EP concentration.

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