Impact Behavior of Sawdust/Recycled–PP Composites

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ABSTRACT: The instrumented Izod and Charpy tests were performed on the sawdust–polypropylene (PP) composites to study the nature of impact resistance in the thermoplastic wood composites. The notched Izod strength was found to increase with filler content in composites containing the Maleic–anhydride–grafted PP (MAPP) additive. The Izod strength even exceeds that of the neat matrix resin at higher filler contents, indicating a methodology for applying the recycled PP and also for reinforcing the thermoplastics with wood powders. While the static tensile results show evidences for the reinforcing role of the wood filler, a fracture mechanics characterization through the Charpy impact tests effects of MAPP under the impact loading rates. The choice of MAPP as the additive was discussed accordingly for application of the sort of materials.
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Key words: sawdust/recycled-PP composites; Izod test; fracture toughness

INTRODUCTION

With the growing economic competition and the ecological pressure, the past decade has seen a renewed interest in developing more efficient reinforcements based on low-cost fillers. Cellulose-based fibers meet such requirements in most aspects. They are made from recoverable resources themselves, and thus may add degradability to plastics.[1,2] The natural fibers usually have relatively high strength and modulus and low-density compared to traditional reinforcements for composites, such as E-glass fibers.[3] The versatility and abundance of their resources ensure the low cost of the potential reinforcements.

The renaissance of the cellulose fiber composites, however, has been greatly hindered by the available forms of the cellulose fibers. Rather than providing continuous cellulose filaments, which would be the essence of the reinforcement, present techniques produce fibers or fabrics consisted of cellulose microfibrils.[3] The situation makes the wood flour a competitive substitute for the cellulose fibers, especially when extrusion compounding and injection molding are adopted as processing methods. The case is especially true when polypropylene, a family of commodity thermoplastics, the properties of which are the most versatile upon tailoring,[4] is concerned as the matrix resin. The promise of using the cellulosic reinforcement, in combination with the consideration of plastics recycling, has made the subject of polypropylene–wood fiber or flour blends one of the most widely studied area. A commercial recycled PP was selected as matrix resin for this study because it exemplifies an extreme choice of the PP resins.

With the technical importance, dispersion of the hydrophilic wood fillers in the hydrophobic PP and the interfacial structures naturally becomes one of the major concerns. Efforts had been made

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either to improve filler dispersion,[1,5,6] to modify the interfacial bonding,[7–9] or to investigate the interfacial structure.[10] Among the methods to modify interfacial interaction, incorporating the Maleic–anhydride–polypropylene (MAPP) copolymers of various molecular weights and via several methods has proven to be an efficient method to improve tensile properties.[11,12] Through the interfacial modifications, several strength properties of the filled PP can increase significantly, owing to[5,6,11–14] the role of MAPP in promoting better wetting of filler surface.

Another problem involved is the poor impact resistance of the PP/wood composites based on PP homopolymers. Although methods of matrix toughening[14–17] proved to be effective in improving the impact strength, the improvement is at the price of stiffness. A number of the toughening studies had emphasized on balancing the stiffness with toughness through adjusting the compounds.[14,15] However, if the wood particles play a role as reinforcing agent under the impact test rates, an improvement in the impact resistance should be achieved through using cellulosic fibers with larger aspect ratio, as had been achieved in discontinuous glass fiber reinforce PP.[4] On the other hand, the works that aimed at probing into the nature of the impact resistance through a simplified linear elastic fracture mechanism (LEFM) characterization[16,17] were frustrated because of the nonlinear behavior of PP[18] involved in the static loading fracture process. It is thus urgent to perform a fracture mechanics study directly on the dynamic impact fracture behaviors to see the role of the wood filler during the impact tests.

For this purpose, the impact behaviors of the reclaimed PP reinforced with wood flour based on sawdust were studied in this work. The static tensile performance of the composites was compared to examine the role of wood fillers. The standard Izod impact tests were performed to see whether the notched impact strength could be raised without adding any elastomer components. The drop-weight dart Charpy tests were carried out and analyzed based on the principles of LEFM to reveal the effects of the wood fillers under impact loading.

EXPERIMENTAL

Materials and Processing

The polypropylene resin used in the work was a commercial PP reclaimed mainly from postconsumer blown PP bottles obtained locally in Guangzhou, China, respectively. Maleated propylene (MAPP) was the Epolene E-43 manufactured by Eastman Chemical Products Inc. The copolymer has an acid number of 47 and molecular weight by weight average of 9,100.[10] The wood flour adopted here was the sawdust that passed a 20-mesh sieve. The sawdust was given by a local furniture factory, and was mainly from pine and Chinese fir. All wood flours were oven dried before use.

Two master batches of PP/wood blends were prepared using a TE-34 corotating twin-screw extruder (Ke-Ya, Nanjing, China) with a length-to-screw diameter ratio of 40. One of the blends consists of identical amounts of the wood flour and of the PP, and the other was made from the 50% wood, 5% MAPP, and 45% PP. The extrusion parameters were typically 190°C maximum melt temperature and 160 rpm screw rotation. Each of the master batches was subsequently diluted to prepare individual PP blends with varying filler content under similar conditions. Total residence time of the filler in the extruder was around 1.5 min. The extruded pellets were completely dried at 110°C and injection molded into void-free 6 mm-thick plates.

Mechanical Testing

Tensile bars with a width of 6.3 mm and a gauge length of 25.4 mm were machined from the injection-molded plates.[19] Tensile tests were performed using an Instron 4206 machine with a crosshead speed of 1 mm/min. The strain was measured using a clip-gauge type extensiometer with 12.5-mm gauge length. At least six tests were performed for each compound. The tensile modulus, tensile strength, and the elongation at yield were averaged from the six tests. The elongation at break of the filled PP was the average of the specimens with the same failure mode.

Notched Izod impact tests were performed according to ASTM D256-93a using a Ceast instrumented pendulum impact tester. The capacity of the pendulum is 7.5 Joules. Specimens were cut from the middle portion of the plates transverse to the melt flow direction (MFD) with a band saw and then milled to dimensions required by the D256 standard. The test rate at impact was 3.45 m/s for all materials studied here. Five tests were carried out on the compounds with the 100-mesh filler. Six specimens were tested for each of the other compounds.

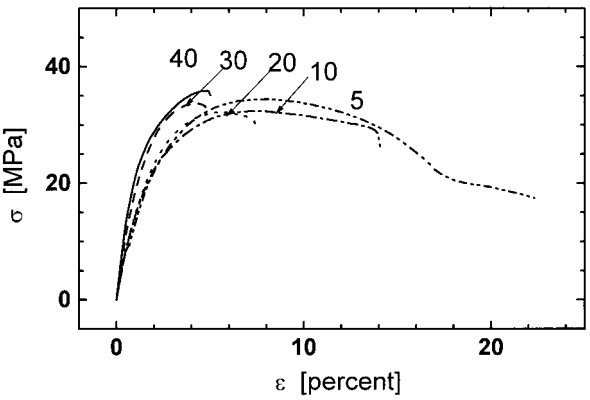

Figure 1 Typical tensile stress–strain curves of the PP/MAPP/wood blends (numbers denotes filler content).

Impact fracture toughness[19,20] was studied through Charpy tests using a Ceast Fractovis instrumented drop-weight impact tester. The specimen have a dimension of 80 × 12.7 mm^2, and were machined from the 6 mm-thick plates. The length of the specimens was also transverse to MFD. The distance between supporting anvils was 51 mm, allowing a span-to-depth ratio of four for the three-point bending tests. A sharp notch was introduced to each specimen by machining a prenotch first and then pressing in a new razor blade.

Fractography

The surface of the broken specimens was examined visually and also with a JSM820 model JEOL Scanning Electron Microscope (SEM). The samples were coated with gold prior to SEM observation.

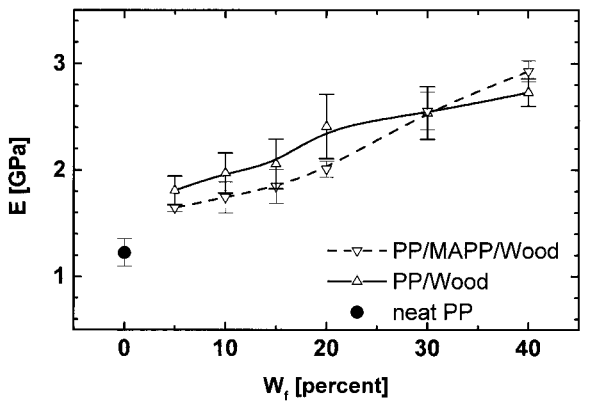

Figure 2 Tensile modulus of PP/wood flour blends with and without MA-*g*-PP.

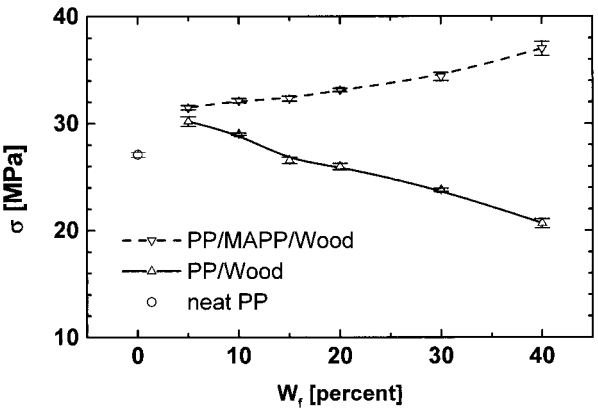

Figure 3 Tensile strength of PP/wood flour blends with and without MA-*g*-PP.

RESULTS AND DISCUSSION

Tensile Properties

It was observed that all PP–wood flour composites except some of the specimens with 5% fillers experienced the intensive stress whitening and fail in the semibrittle way after yielding (Fig. 1). No necking and drawing had ever been observed when filler content was equal to or higher than 10%. Figures 2 to 4 summarize the tensile properties of the reclaimed PP (RPP) filled with 20-mesh wood flour of different loading. From Figure 2, it can be seen that the tensile modulus tends to increase with the filler content in the RPP/wood composites either with or without the Epolene additive. The reinforcement by the wood filler is evident in terms of the modulus.

In contrast, the yield stress, σ_y, of a blend changes with wood content in opposite ways, de-

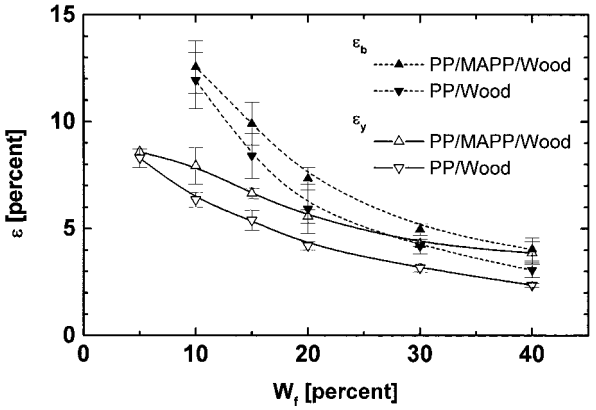

Figure 4 Yield and break strain of PP wood flour blends.

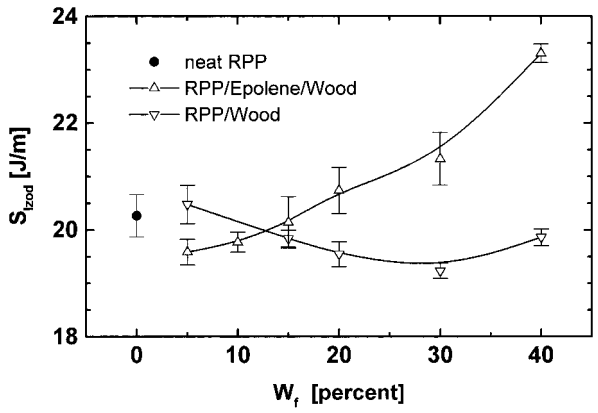

Figure 5 Izod impact strength of PP/wood flour blends with or without MA-*g*-PP.

pending on whether the compound contains MAPP or not. As illustrated in Figure 3, the yielding strength decreases with filler content if the composite contains no MAPP, while σ_y of the PP/MAPP/wood blends does the opposite. The additive MAPP had resulted in larger difference in the yield strength at given filler contents (Fig. 3) than in the modulus (Fig. 2). It is evident that the role of the additive MAPP in terms of the static properties is through enhanced filler dispersion and wetting, rather than forming any interfacial bonding.

The higher strength in PP/MAPP/wood blends may arise from several factors, for example, an increase in filler dispersion, some bonding between PP and wood, and/or modification of matrix PP. A recent work focussing on the potential ester linkage between MAPP and wood fillers[10] had shown negative proof of the interfacial ester bond and indicated the compatibilizing role of MAPP, especially the one with lower molecular weight as used in the present article. As illustrated in Figure 4, the yielding strain and the strain at break had all been higher in blends containing MAPP, agreeing to its role in modifying filler dispersion and interfacial wetting. The weaker interfacial interaction between the MAPP and the wood filler may contribute to strength rather than to modulus.

Izod Impact Strength

Figure 5 shows the Izod impact strength of the RPP filled with the 20-mesh filler, along with the result of the neat resin. As shown in Figure 5, the Izod strength tends to decrease with filler content up to 30% of wood. In contrast, it increases significantly with the filler contents in blends containing MAPP. The opposite filler content dependency suggest that the presence of MAPP had resulted in a filler-related energy-absorbing mechanism. Through the morphology observation, it can be seen that such mechanism included events such as interfacial debonding, filler particle pullout from matrix, individual fiber pullout, and split or defibrillation of the filler particles (Fig. 6).

From Figure 5, it can also be seen that the composite without MAPP and containing 5% wood has higher Izod strength than its counterparts containing MAPP. Such a ranking of Izod strength is reversed at higher filler contents. As shown in Figure 6(a) and 6(b), in the 5% wood composite without MAPP, the cleavage between matrix and wood particles is wider, indicating more debonding and thus weaker interfaces. The debonding between the finer cellulosic fibers and their sheath also suggests additional energy-absorbing mechanism that is unique to the blend without MAPP. The larger Izod strength is thus in agreement to the common sense that weak interface can result in higher impact resistance. If the fracture surfaces of composites with higher filler loading is compared [Fig. 6(c) and (d)], it can be found that the wood particles are more integrate in the blend without MAPP, and debonding between the particles and the matrix is significant. In wood composites with the additive, however, there are more evidences of the wood particle defibrillation but few relics of matrix–wood particle debonding.

The different roles of MAPP at the two extreme filler contents are further perceived through the force curves of the blends. At the filler loading of 5%, the blend without MAPP trailed after the maximum but that of the blend with MAPP does the opposite [Fig. 7(a)]. The case is in contrast to the pair of blends with 40% filler shown in Figure 7(b), where the force curve containing MAPP trails because of the contribution from deformation in filler particles. The MAPP has played a role in promoting strong interface in the PP/wood blends. The strong interface causes a decrease in Izod strength at the lowest filler content, and does the opposite when the latter is not too low.

These results are different to the tensile tests, where modulus was lower in MAPP-containing blends for most of the filler contents. In the tensile tests with lower strain rate, lack of the strong bonding, such as ester linkage,[10] prevent MAPP from directly introducing a stronger interface. At the Izod test rate, however, even a weaker bond-

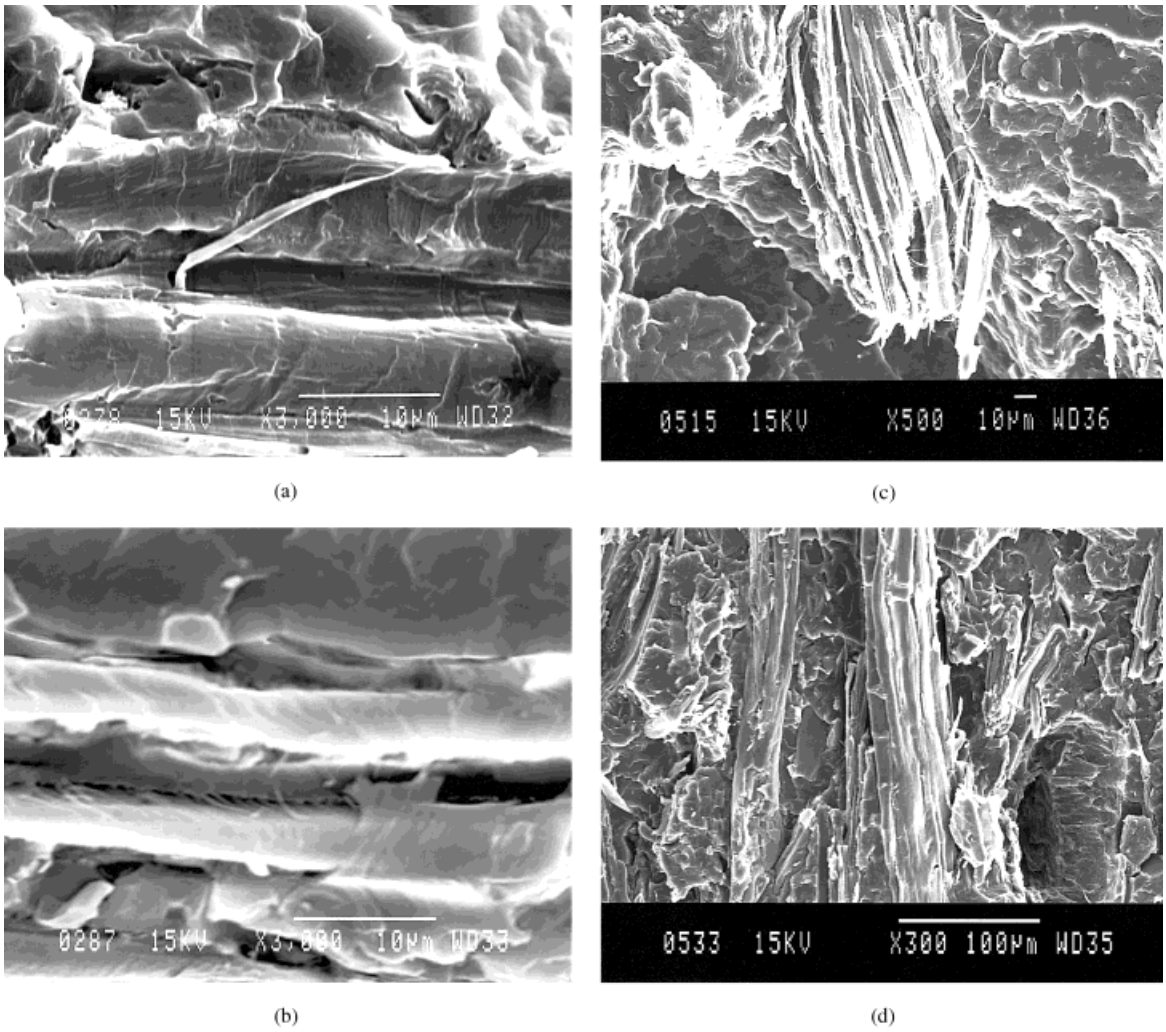

Figure 6 SEM photos of PP/wood blends containing 5% (a and b) and 40% (c and d) filler. Compounds in (a) and (c) had included MAPP, while specimens in (b) and (d) had not.

ing, such as hydrogen bonds, which are most reasonable to be at the interface, can actively transfer load across the interface. Higher unnotched Izod strength in similar blends[14,17] should also originate from such interfacial mechanism.

Impact Fracture Toughness

The better reinforcement under dynamic conditions through incorporating MAPP is more directly revealed through the following analyses based on the Linear Fracture Mechanics (LEFM).

Figure 8 shows the typical force vs. time, $F(t)$, plot of the neat polypropylene. As shown in Figure 8, the force grows through consecutive oscillations to reach its maximum in about a thousandth second and then decreases abruptly to zero. The $F(t)$ curve resembles that of the PP

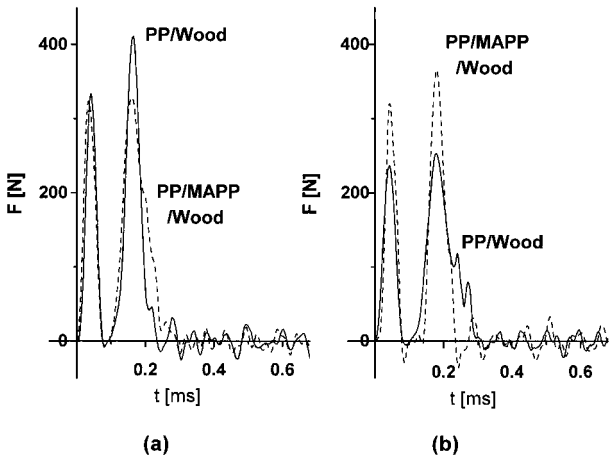

Figure 7 Typical force vs. time curves during the Izod test. Wood content is (a) 5% and (b) 40%, respectively.

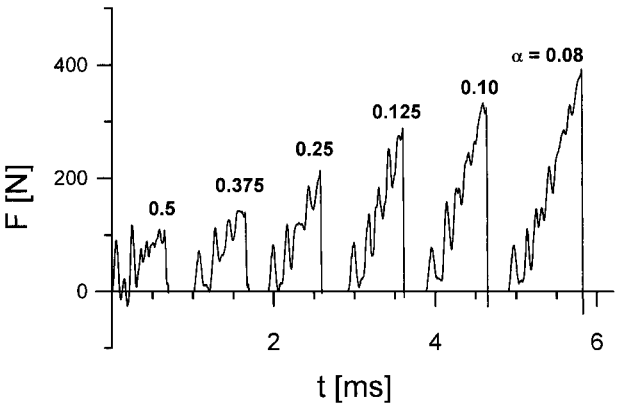

Figure 8 Representative load history of neat resin in Charpy tests. The curves have been translated along the time axis for clearance.

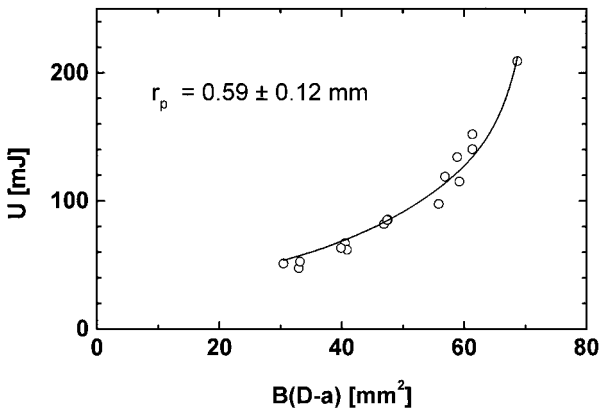

Figure 9 U vs. $B(D - a)$ plot of neat resin.

homopolymer, suggesting a brittle propagation after the initiation.[19] The energy up to the force maximum, U, was then used to calculate the critical strain energy release rate, G_c, according to LEFM.[20]

According to LEFM, the critical strain energy release rate G_c can be obtained through a linear U vs. $BD\phi$ regression because

$$U = E_k + G_c BD\phi \tag{1}$$

where E_k is the kinetic energy, B and D are specimen thickness and width, respectively, and ϕ is the correction for specimen compliance based on LEFM.[20–22] The calibration ϕ is the function only of the specimen geometry and crack length in fully elastic cases, that is, it is α, the ratio of crack length over specimen depth, a/D, at a given span.[20] When the LEFM method is to be used in the polymer systems with a confined plastic zone ahead of the crack tip, a correction[21,22] has to be made to α and thus to ϕ. The correction can be performed by adding a quantity r_p, that is, the size of the plastic process zone, to the initial crack length a

$$\alpha = (a + r_p)/D \tag{2}$$

For convenience of calculation, the numeric solution of the LEFM compliance correction $\phi(\alpha)$[20,21] can be rewritten as a two-order exponential decay function in terms of α

$$\phi = C_0 + e^{-\alpha/\tau_1} + e^{-\alpha/\tau_2} \tag{3}$$

where the constants are $C_0 = 0.06766$, $\tau_1 = 0.04003$, and $\tau_2 = 0.35124$, respectively. Substituting eqs. (2) and (3) into eq. (1) yields a nonlinear U vs. a function. The $U(a)$ relationship can thus be expressed in terms of the ligament area $B(D - a)$

$$U = E_k + G_c B(D - a)\,\frac{C_0 + e^{-(a+r_p)/D/\tau_1} + e^{-(a+r_p)/D/\tau_2}}{1 - a/D} \tag{4}$$

where parameters E_k, G_c, and r_p can be obtained through a nonlinear regression of U vs. a data. Such a $U(a)$ function had been illustrated in terms of a U vs. $B(D - a)$ nonlinear curve.[21,22] The value of r_p was then obtained for the impact fracture. As shown in Figure 9, the r_p value for the neat PP is 0.59 ± 0.16 mm. The plastic zone size is far less than either of the specimen dimen-

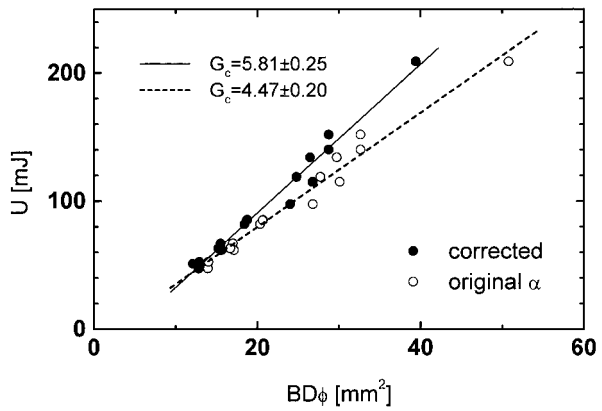

Figure 10 U vs. $BD\phi$ plot of the neat resin before (open circles) and after (solid symbols) the plastic zone correction.

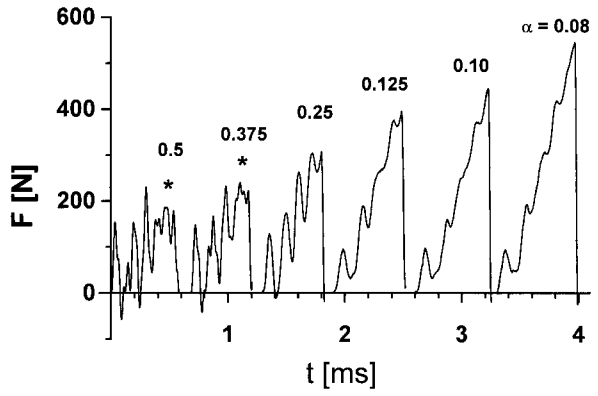

Figure 11 Typical load history of the PP/MAPP/wood blend with 40% filler.

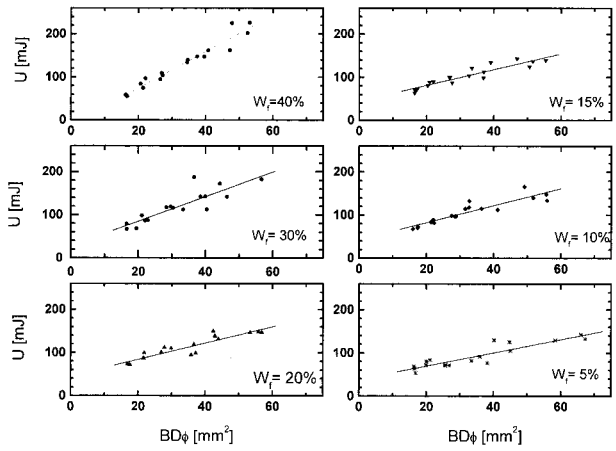

Figure 12 U vs. corrected $BD\phi$ plots of the blends containing MAPP.

sions, ensuring the validity to apply LEFM in characterizing the impact fracture. The initiation energy U of the neat PP vs. $BD\phi$, calculated from ϕ either corrected or not, was plotted in Figure 10. It is evident that the corrected data follows better linear U vs. $BD\phi$ relationship. The corrected data yields relatively a high value

$$G_c = 5.81 \pm 0.25 \text{ kJ/m}^2$$

which coincides with the fact that the resin had been reclaimed from the blown bottles.

Based on the same principles, G_c values can be calculated based on the same procedure for the filled systems. As shown in Figure 11, the force curves are similar to that of the resin, allowing the LEFM method to be applied. When the nonlinear regression was performed for estimating r_p, however, the scatter is relatively large for most of these compounds, as illustrated in Table I. The larger scattering makes it less confidant to calculate the G_c based on the r_p value. A fixed r_p = 0.635 (hence α = 0.05), which is the nominal average of all these filled PP compounds, has to be chosen to correct α and ϕ, leading to the U vs. $BD\phi$ plots shown in Figure 12. Through the linear $U - BD\phi$ regressions, G_c was obtained for both group of wood blends and illustrated in Figure 13.

From Figure 13, it can be seen that G_c values of all filled compounds are lower than that of the neat resin. In agreement with the behavior of tough PP polymers filled with inorganic fillers,[4] this result reveals the lower level of plastic flow ahead of the crack tips. For blends containing no MAPP, G_c is always far less than the value of neat PP and depends little on filler content. When MAPP had been included in the compounds, G_c tends to increase generally with the increasing filler content, although the increasing trend seems to be more evident at higher filler contents. It increases to about 73% of the neat resin value.

Table I Radium of Plastic Process Zone, r_p, in mm, Through $U - B(D - a)$ Nonlinear Regression

W_f %	PP/Wood	PP/MAPP/Wood
40	0.534 ± 0.598	0.000 ± 0.167
30	0.000 ± 0.202	0.740 ± 1.206
20	0.237 ± 0.316	0.368 ± 0.543
15	0.000 ± 0.186	1.078 ± 1.342
10	0.468 ± 0.942	1.821 ± 4.751
5	0.568 ± 0.946	0.110 ± 0.422

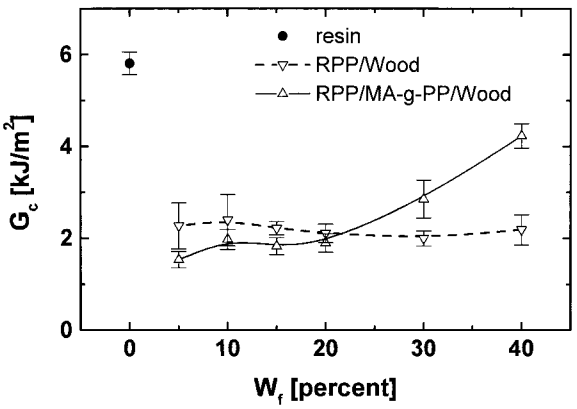

Figure 13 Critical strain energy release rate G_c of PP/wood flour blends.

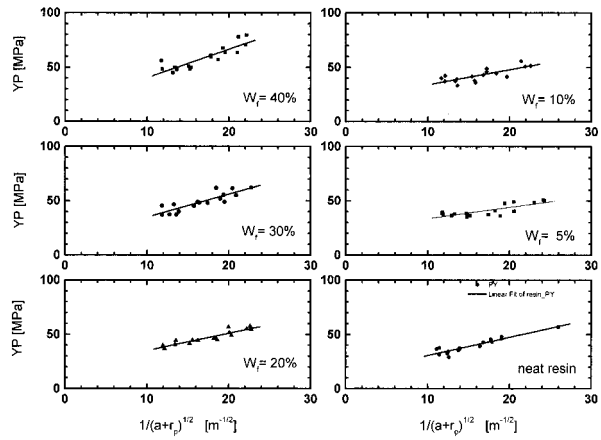

Figure 14 $YP - (a + r_p)^{-1/2}$ plots of the blends containing MAPP and of the neat resin.

The effect of MAPP on G_c at given filler contents is similar to the Izod test results (Fig. 5) to a certain degree. It can be seen from Figure 13 that the effect of MAPP is also different in two ranges of filler content. G_c values of the blends without MAPP are higher at lower filler contents and vice versa when filler content is high. The similarity between the Izod strength and the Charpy G_c is reasonable, for the Izod strength measure, in fact, the fracture energy at the specific condition, and the loading rates had not been very different.

The critical stress intensity factor, K_c, shortened as fracture toughness below, can also be obtained from the measured maximum dart stress. As a general consideration, the stress intensity factor K_I depends on the crack length a and the far-field stress p

$$K_I^2 = Y^2 p^2 a \tag{5.a}$$

where Y is a finite-width correction.[21] In the present case of dynamic loading, an analogy to eq. (1) would yield a linear relationship between the stress and the square root of crack length, with the K_c as the slope

$$YP = (YP)_k + K_c/\sqrt{a + r_p} \tag{5.b}$$

where P is the stress based on the maximum force measured via the impact dart and the ligament cross-section, the subscript k stands for the kinetic case as in eq. (1), and $(a + r_p)$ is the crack length including the plastic zone. The r_p value is the same with the one obtained through $U - BD\phi$ curve fitting. It is evident that the corrected stress PY depends on $(a + r_p)^{-1/2}$ linearly for each blend and also for neat resin (Fig. 14). The K_c values were thus obtained and listed in Figure 15.

As illustrated in Figure 15, the fracture toughness K_c increases with filler content for blends either with or without the MAPP, indicating the reinforcing effects of the wood filler. The filler content dependence is much stronger for the MAPP-containing blends than without, leading to a larger and larger difference between the blend containing MAPP and without. For blends without MAPP, the increase in filler content from 5 to 40% results in an increases in K_c by only 32%. The fracture toughness values of all these blends containing no MAPP are lower than the neat resin. In contrast, the same increase in filler content causes K_c to increase by 1.3 times, higher than that of the neat resin when filler content is 30 or 40%. As the higher G_c in MAPP-containing composite was observed only for higher filler contents, the always larger K_c clearly indicates that the MAPP had enhanced the interaction across the interface during the impact loading and thus improved the reinforcing role of the wood filler. The effects of MAPP as "coupling agent" at the dynamic loading was factually also revealed in Table I, where higher r_p can be found for composites with MAPP except for the 40% compound. The larger area of the plastic process zone ahead of the crack tip indicate that the impact energy had been dissipated by materials including more filler particles, again revealing a strong interfacial bonding at the loading rates. Obviously, the stronger interfacial interaction that accompanied the better wetting had played the role of interfacial bonding. The "strong" chemical bonding such

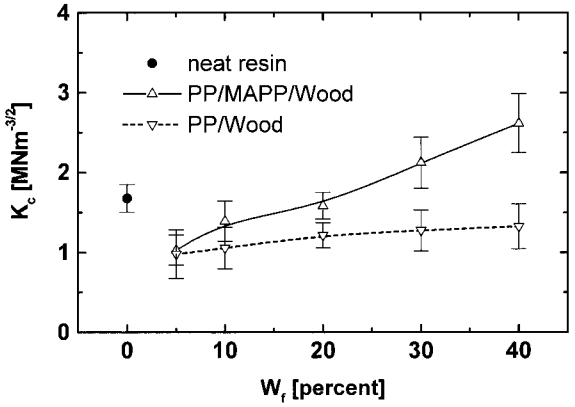

Figure 15 K_c of blends containing MAPP and without the additive.

as ester linkage and hydrogen bonding may not be necessary for these higher loading rates.

CONCLUSIONS

The impact resistance of the sawdust-recycled PP composites has been characterized through both the standard Izod test and the Charpy experiments. It is observed that the notched Izod strength of the MAPP-containing composites tends to increase with the filler content, and may exceed that of the neat matrix resin. Without the MAPP additive, however, the Izod strength decreases with the filler content and is always lower than the strength of the resin. The difference between the two groups of compounds reveals the reinforcing potential of the sawdust filler and the positive effect of the MAPP compatibilizer.

The nature of the impact resistance of the recycled PP/wood blends was revealed more clearly through the successful application of LEFM. It is found that the fracture energy G_c of the studied compounds are all lower than that of the neat matrix resin. The fracture energy increases significantly with filler content in composites containing MAPP, but decreases slightly in composites without the additive. The fracture toughness, K_c, however, increases with filler content for both groups of compounds. When the filler content increasing from 5 to 40%, K_c increases by 1.3 times in MAPP-containing composites but rises merely by 32% in blends without. With the presence of the MAPP, K_c becomes considerably higher than that of the neat resin at the higher filler loading of either 30 or 40%. With the presence of the MAPP, the impact resistance tends to increase generally with filler content in terms of both the stiffness and energy absorption. Therefore, the higher notched Izod strength through including the MAPP revealed the toughening effects of the wood powder reinforcement that has a relatively large aspect ratio.

It is important to note that the strong interfacial bonding has not been observed in terms of a significant rise in the static tensile modulus. The rate-dependent interfacial interaction is readily explained if the interfacial structure between PP and wood inclusions are predominantly electrodispersive. Therefore, the dynamic modulii data and the direct morphology observation are to be characterized further for a more convincing fracture resistance mechanism of the thermoplastic wood composites.

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