Study of the Mechanical Properties of Mica-Filled Polypropylene-Based GMT Composite

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ABSTRACT: Mica-filled polypropylene (PP)-based GMT (PP–mica–GMT) was prepared by a double-belt press and its mechanical properties were tested. The effect of the mica content on the mechanical properties of PP–mica–GMT was investigated. It was found that with a lower mica content in matrix the tensile and flexural properties were improved; however, at a high mica content level, the tensile and flexural properties decreased. With respect to the impact strength, there is a maximum value at an approximate 20 wt % mica content level. These results were attributed to the influence of mica on fiber–matrix adhesion. It is most likely that low mica content enhances fiber–matrix adhesion; however, high mica content is unfavorable to fiber–matrix adhesion. Maleic anhydride-grafted PP (MPP) was used to enhance the fiber–matrix adhesion at a higher mica loading level for PP–mica–GMT. With increasing MPP content in the matrix, the tensile properties were significantly improved, whereas the Izod impact strength decreased. In addition, with 5 wt % MPP and 40 wt % mica in the PP matrix formulation, the effect of the mica particle size and glass fiber content on the mechanical properties of PP–mica–GMT was investigated. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 82: 2719–2728, 2001

Key words: glass fiber-reinforced thermoplastic; mica; poly(propylene); mechanical properties; interfaces

INTRODUCTION

Glass mat-reinforced thermoplastics (GMT) has been widely used in automotive applications. Relative to a sheet-molding compound (SMC), which consists of a thermoset resin reinforced with glass fiber, GMT offers considerable advantages such as recyclability, faster processing time, and higher toughness.[1] However, SMC is the major competitor to GMT in automotive applications because it has higher stiffness and good appearance at lower cost due to a higher filler content. The use of a filler can reduce materials costs and increase stiffness and dimensional stability. At the same time, fillers have a tendency to increase resin viscosity. The viscosities of thermoset resins are much lower, so that the addition of mineral fillers does not affect the impregnation of the glass reinforcement. The matrices of GMT are thermoplastics with high melt viscosities, which can impregnate the glass mat only under proper processing conditions. The mineral fillers are not recommended for use in the matrix formulation of GMT since the addition of fillers leads to a higher melt viscosity of the matrix. In general, the matrix formulation is simple, only including small amounts of pigment, stabilizer, and nucleator.

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It has been reported in a patent that highly mineral-filled polypropylene (PP)-based GMT, giving a combination of high stiffness and toughness, has been successfully manufactured by Azdel Inc. (Shelby, NC).[2] According to the patent, the filled GMT is less expensive and exhibits similar or significantly improved mechanical properties over nonfilled GMT. Mica has been shown to provide the highest stiffness relative to other fillers and glass fiber and to reduce warpage effectively due to its platelike shape. A combination of short glass fiber and mica in injection-molded PP composites has been used to enhance stiffness and improve dimensional stability.[3,4] It can expected that if mica can be incorporated with glass mat in the GMT composite successfully the resultant GMT composite will exhibit higher stiffness and good dimensional stability.

In this work, mica-filled PP film was prepared at first, which was then laminated with a random continuous swirled glass mat to prepare PP–mica–GMT. PP–GMT and PP–mica–GMT were prepared by a continuously running double-belt press designed in the lab. The mechanical properties of the prepared GMT sheets were determined. Both the impact fracture mode and the pulled-out fiber surface for PP–GMT and PP–mica–GMT were observed by scanning electric microscopy. The objective of the present work was to study the effect of the addition of mica on the mechanical properties of GMT and to reveal whether the addition of mica will influence the interfacial properties between the fiber and the matrix. Factors affecting the mechanical properties of PP–GMT, such as the addition of an interfacial promoter [maleic anhydride-grafted PP (MPP)], mica particle size, and fiber content, were also investigated.

EXPERIMENTAL

Materials

The homopolymer PP obtained from the Plastices of Shanghai Petrochemical Company (Shanghai, China) was used as the matrix: Y1600 (with melt flow index of 16 g/10 min). MPP was prepared by a solid method as reported in the literature.[5] The stabilizers added to PP prior to processing were Irganox 1010 and Irganox PS 802 FL from the Ciba-Geigy Corp. Ltd. (Hong Kong). A-1100™ organsilane coupling agent for mica surface treatment was obtained from the OSi Co. (Hong Kong).

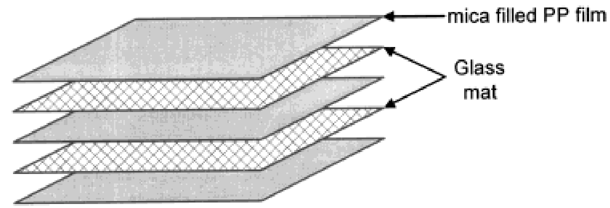


Figure 1 Experimental film-stacking method of two layers of glass mat and three layers of mica-filled PP film.

The needled continuous swirled fiber bundle glass mat, from the Nanjing Glass Fiber Research & Design Institute (Nanjing, China), was used as a reinforcement. The glass mat was sized for compatibility with PP. Three commercial grades of muscovite mica (mesh size of the flakes: 100S, 325S, 600S) supplied by the Shanghai Jianghu Chemical Company (Shanghai, China) have no chemical surface treatment.

Preparation of PP–Mica–GMT

The mica was coated in a high-speed mixer with a 1.0 wt % (calculated for the amount of mica) silane coupling agent A-1100™ which was prehydrolyzed in distilled water, then dried in a oven at 110°C. The mica, stabilizer, and other aids were dry-blended in the high-speed mixer. PP and mica were melt-blended using a twin-screw compounding machine. The ratio of the raw materials and the compounding conditions such as temperature, screw speed, and throughput were monitored. The extrudate from a sheet extruder using the dried pelletized compounds of mica-filled PP was rolled by a roller press into thermoplastic film of 0.8 mm thickness.

The samples of mica/PP GMT were prepared by the method of impregnation using a double-belt press (DBP). Three layers of PP film and two layers of glass mat were alternatively combined to a stack (as depicted in Fig. 1), which was then pulled into the press by the belts. The glass fiber content of GMT was determined by the basis weight of the used glass mat. To reduce the value scatter of the mechanical properties, the glass mats were carefully selected to ensure their homogeneity in the glass fiber distribution. The glass fiber content was controlled at approximately 12.5 vol % for PP–GMT and the same volume content for PP–mica–GMT. The prepared laminates were on the order of 4.0 mm thick and with a width of 0.3 m and a length of 0.5 m.

Testing

The tensile testing was performed on a CMT 4204 universal testing machine manufactured by Shenzhen SANS Experimental Equipment Corp. Ltd. at a crosshead speed of 2 mm min^{-1}. Tensile samples were prepared according to ASTM D638, type I specification. An extensometer with gauge length of 50 mm was used for modulus measurements.

The flexural testing was conducted using a CMT 4204 universal testing machine in the three-point loading mode. Flexural specimens with a width of 13 mm and length of 100 mm were prepared. The span length between two supporting noses was 80 mm and a crosshead speed of 2 mm min^{-1} was used. The procedure ASTM D 790 was followed. The notched Izod impact test was performed on a Cantilever Beam impact machine. The test method was consistent to D265. All mechanical tests were performed at 23°C, and seven specimens for each sample were tested.

The void content of mica/PP GMT was measured using eq. (1) according to ASTM D2734:

$$V_d = 100(T_d - M_d)/T_d \quad (1)$$

where V_d is the void content (%); T_d, the theoretical density (g/cm^3); and M_d, the measured density (g/cm^3), which was measured from the nominal volume and the mass of the specimen. T_d was calculated using eq. (2):

$$T_d = \frac{1 + (W_{mica}/W_{PP}) + (W_{glass}/W_{PP})}{1/\rho_{PP} + (W_{mica}/W_{PP}\rho_{mica}) + (W_{glass}/W_{PP}\rho_{glass})} \quad (2)$$

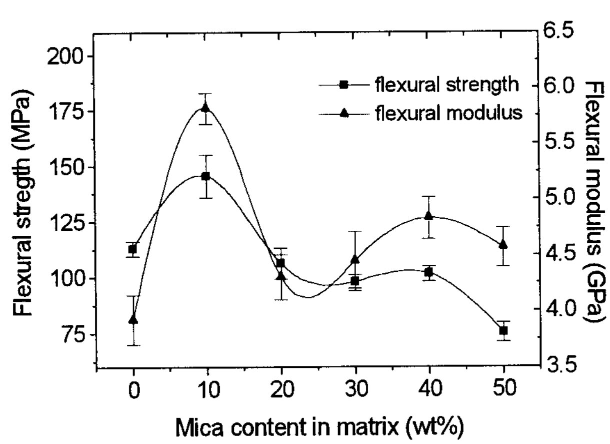


Figure 2 Flexural properties of PP–mica–GMT as a function of mica content in the matrix.

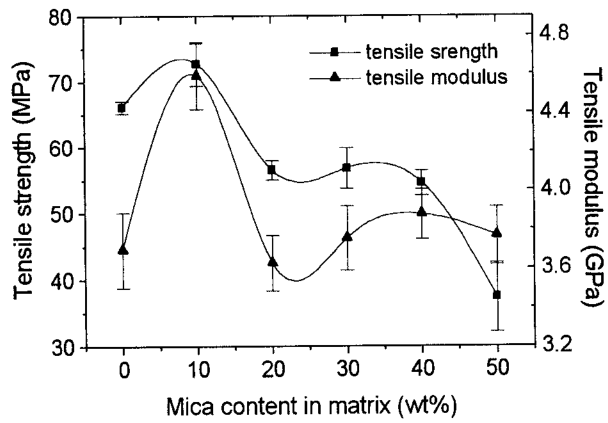


Figure 3 Tensile properties of PP–mica–GMT as a function of mica content in the matrix.

where ρ is the density obtained from the raw material supplier, and weight ratios W_{mica}/W_{PP} and W_{glass}/W_{PP} were obtained by burning off mica-filled PP film and PP–mica–GMT. Both the film specimen and the GMT sheet specimen was burnt off for 4 h at 600°C.

Scanning Electron Microscopy (SEM)

The Izod impact fracture surface of PP–GMT and PP–mica–GMT was observed using a Cambrige Stereoscann S200 SEM. The observation direction is perpendicular to the thickness direction of the GMT sheet. In addition, the pulled-out fiber surface and the dispersion state of mica particles at higher mica content was studied by SEM.

RESULTS AND DISCUSSION

Effect of Mica Content on the Mechanical Properties of PP–Mica–GMT

The influence of the mica content in the PP matrix on the flexural properties of PP–mica–GMT is illustrated in Figure 2. It can be seen that with 10% mica content by weight both the flexural strength and the flexural modulus are significantly increased relative to PP–GMT. With a further increasing mica content, the flexural strength decreases and the flexural modulus shows a drastic decrease followed by a slight increase up to 40% mica content. At the content of 50%, the flexural properties are decreased to some extent. Figure 3 represents the tensile strength and the tensile modulus as a function of the mica content, which shows the same trend as

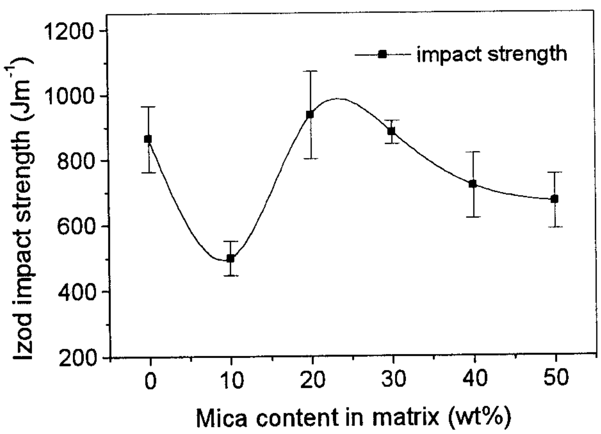


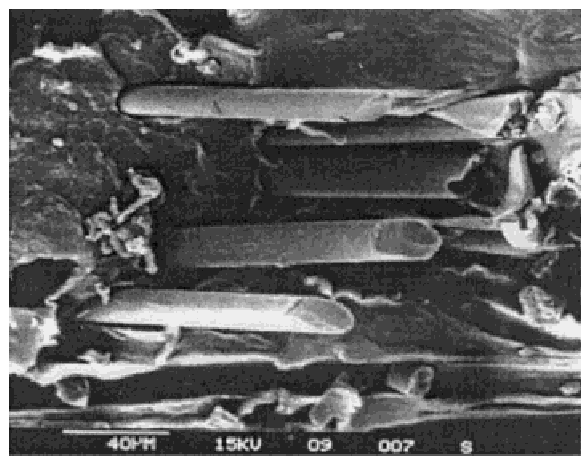
Figure 4 Impact strength of PP–mica–GMT as a function of mica content in the matrix.

that of the flexural properties. The impact strength of PP–mica–GMT is shown in Figure 4. A minimum value at 10% mica content and a maximum value at approximate 20% mica content can be observed.

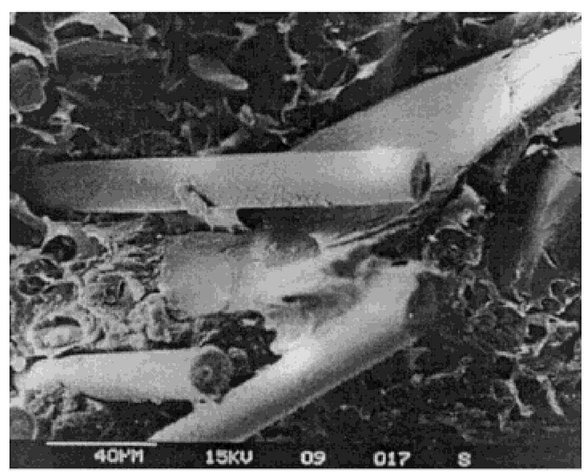
It is most likely that the addition of mica influences fiber–matrix adhesion, which is responsible for the mechanical property variation with mica content for PP–mica–GMT. The interfacial shear strength (τ) is one of the most important parameters controlling the strength and toughness of composites.[6,7] It has been proposed that, in the case of glass fiber in PP, the principal factor governing τ is the static friction between the fiber and the matrix, in which case, $\tau = \rho_s \sigma_R$, where ρ_s is the static friction coefficient, and σ_R, the radial stress due to thermal shrinkage of the matrix.[7,8] Dilandro proposed that σ_R can be calculated from[9]

$$\sigma_R = \frac{(\alpha_m - \alpha_f)\Delta T E_f E_m}{(1 + \nu_f + 2V_f)E_f + (1 + \nu_m)E_m} \tag{3}$$

where α is the thermal expansion coefficient; ΔT, the difference between the matrix solidification temperature and the testing temperature; ν, Poisson's ratio; and E, the Young's modulus. The subscripts f and m refer fiber and matrix, respectively. It is obvious from eq. (3) that the fiber radial compressive stress is dependent on the modulus and thermal expansion coefficient of the matrix. The addition of mica into PP increases the Young's modulus of mica-filled PP to a considerable extent, whereas the thermal expansion coefficient is decreased only to a slight degree relative to E_m. With 10% mica content, the measured tensile modulus of PP increases from 1.7 to 2.7 GPa, whereas the thermal expansion coefficient decreases by a value of 3% calculated by the rule of mixture. So, it can be expected that the fiber compressive radial stress is increased with the addition of mica, thereby leading to higher interfacial shear strength. In Figure 5, it can be seen that the pulled-out fiber surface for PP–mica–GMT with 10% mica content is somewhat rough relative to PP–GMT, evidencing that, in the case of lower mica content, the fiber–matrix adhesion is strong for PP–mica–GMT relative to PP–GMT. Consequently, the tensile and the flexural properties of the PP–mica–GMT with 10% mica content are significantly improved.



(a)



(b)

Figure 5 SEM micrographs of the pulled-out fibers and matrix for (a) PP–GMT and (b) PP–mica–GMT with 10 wt % mica content in the matrix.

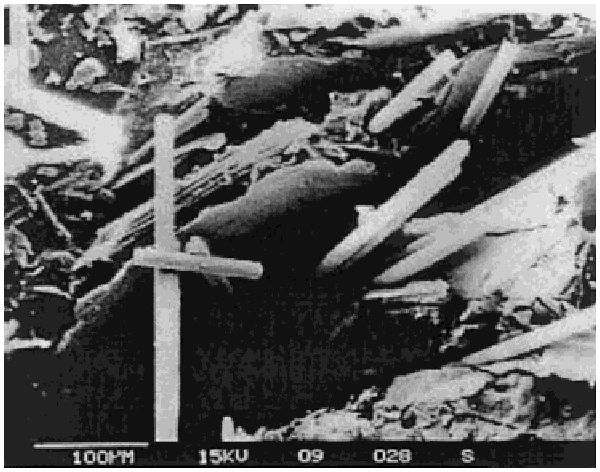


Figure 6 SEM micrograph of the dispersion state of mica flakes and glass fibers in PP–mica–GMT with 20 wt % mica in the matrix.

In the PP–mica–GMT system, the mica flakes have opportunities to contact with the glass fibers. In the case of lower mica content, only a small amount of mica will, contact with the glass fibers, as shown in Figure 5(b). However, in the case of higher mica content, the glass fibers will be contacted by a large amount of mica flakes (Fig. 6), thus resulting in insufficient wetting of the glass fiber and therefore poor fiber–matrix adhesion. For this reason, when the mica content is to 20%, the tensile and flexural properties are remarkably decreased. Referring to Figures 2 and 3, the tensile and flexural properties show a slight improvement with the mica content up to 40%. These results can be attributed to two trends with increasing mica content: With increasing mica content, it is believed that the reinforcing efficiency of the glass fiber decreases due to fiber–matrix adhesion weakening. On the other hand, the reinforcing effect of the mica in the composite is enhanced. When the mica content is to 30%, the later trend weighs the former one, so that the tensile and flexural properties show a slight improvement.

When the mica content is too high, to 50%, the total inorganic reinforcement content is so high that the polymer matrix cannot fully wet and impregnate the reinforcements and, also, fiber-matrix adhesion is too poor due to the disturbance of mica flakes. From Table I, it can be seen that with increasing mica content the void content increases. In the case of 50% mica content, the void content is too high. Voids may toughen the polymer in some cases.[10] The toughening mechanism is that the presence of voids allows some matrix shear yielding to occur around the voids. For PP–mica–GMT at a high filler content level, due to the contact of mica flakes and fibers, the matrix can not wet and impregnate the fibers completely and, hence, the voids are formed mainly at the fiber–matrix interface. Voids in the matrix can enhance matrix shear yielding; however, voids at the fiber–matrix interface cannot initiate matrix shear yielding, but weakens interfacial adhesion further, as well as the mechanical properties.

The addition of mica leads to a decrease of the impact strength of PP to some extent. In our study, the addition of 40% mica into PP caused a slight decrease of notch Izod impact strength from 23.5 to 17.2 J/m^2. Taking into account this fact, the influence of mica itself on the impact strength of PP–mica–GMT can be neglected. It was suggested previously that the addition of mica influences the fiber–matrix adhesion. It is believed that the impact resistance variation of PP–mica–GMT should be associated with the influence of mica on the fiber–matrix adhesion.

The influence of the mica content on the impact resistance for PP–mica–GMT is discussed. Karger-Kocsis et al. demonstrated that for GMT the energy-absorbing mechanisms include matrix deformation, mat mesh-type deformation, fiber strand debonding, split-up, pullout, and fiber fracture.[11,12] It is shown in Figure 7(a,c) that there is a large amount of fibers with a long fiber length protruding from the fracture surface, resulting from the fiber strand split-up and fiber pull-out during the impact process. The poor fi-

Table I Void Content of PP–Mica–GMT with Various Mica Content

	Mica content (wt %)					
Parameter	0	10	20	30	40	50
Void content (vol %)	1.47	1.65	2.36	2.54	3.17	8.02

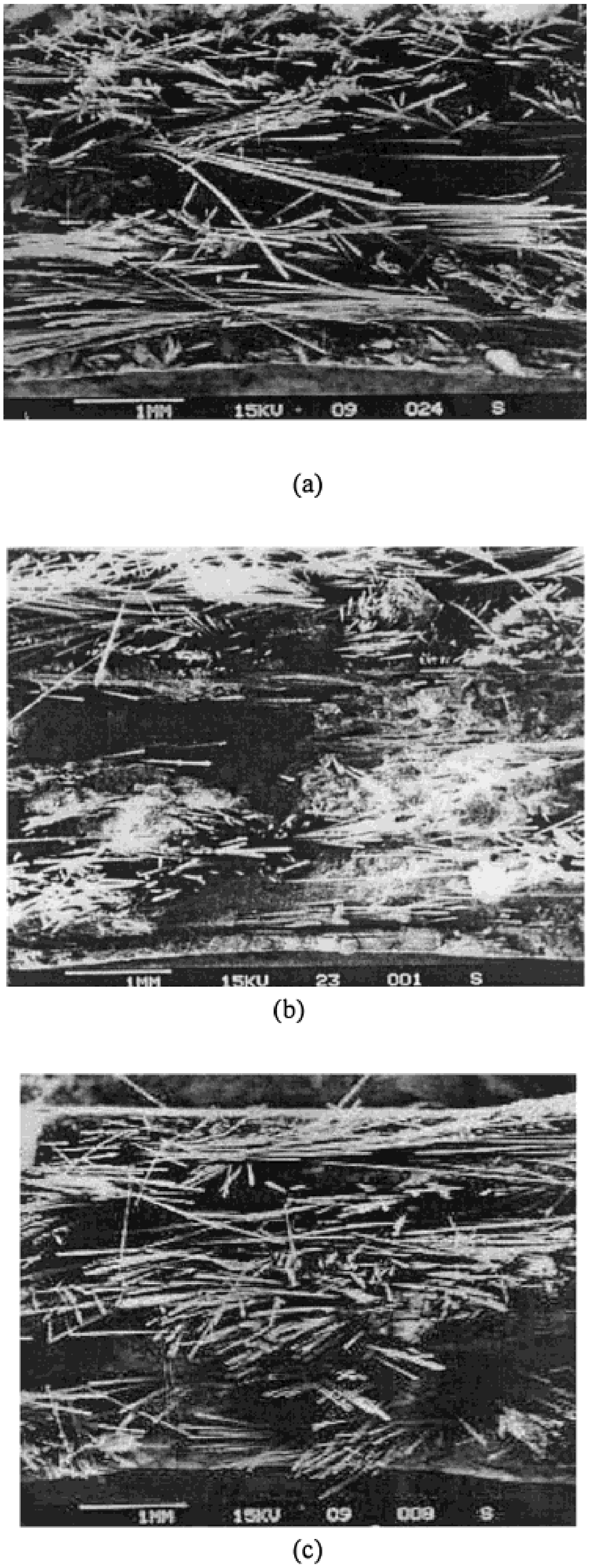


Figure 7 SEM micrographs of the impact fracture surfaces of PP–mica–GMT with varous mica contents in the matrix: (a) 0; (b) 10 wt %; (c) 20 wt %.

ber–matrix adhesion for PP–GMT and PP–mica–GMT with a high mica content is responsible for the fracture mode. It can be expected, in this case, that both the fiber strand debonding length and pullout length are long, and, hence, the fiber mat-related energy-absorbing capability is high. In consideration of the fact that the fiber mat-related energy-absorbing mechanisms contribute to the impact strength of GMT to a large extent, it can be understood that the impact resistance for PP–GMT and PP–mica–GMT with a high mica content is superior. In Figure 7(b), it can be seen that there is a small amount of pulled-out fibers with a short fiber length for PP–mica–GMT with a low mica content, indicating a shorter fiber strand debonding length and pull-out length. Also, it is most likely that strong fiber–matrix adhesion restricts the fiber mat mesh-type deformation during the impact process. Therefore, the impact strength is lower for PP–mica–GMT with a low mica content.

As discussed above, poor fiber–matrix adhesion is favorable to impact resistance. However, it is impossible that the impact strength of GMT increases continuously with the fiber–matrix adhesion strength decreasing. When the fiber–matrix adhesion is so poor that the stress cannot be effectively transferred from the matrix to the reinforcing fibers, only a small amount of energy will be exhausted by the fiber strand debonding and pull-out and other fiber mat-related energy-absorbing mechanisms. As a result, the impact strength of GMT decreases in the case of too poor fiber–matrix adhesion. This is the case for PP–mica–GMT at a higher mica content level. When the mica content is higher than 30%, the fiber–matrix is so poor that the impact strength decreases with increasing mica content.

In addition, referring to Figure 4, it can be found that the notch Izod impact strength is very high (from 400 to 1000 J/m^{-2}) for PP–GMT and PP–mica–GMT relative to PP–GF. It is well known that GMT exhibits superior impact resistance, which arises from its high fiber mat-related energy-absorbing capability and the associated matrix deformation. The matrix deformation for PP–GMT and PP–mica–GMT can be observed in Figure 6. From the above discussion, it is believed that the addition of mica just influences the energy-absorbing capability to some extent instead of changing the energy-absorbing energy mechanism for GMT. So, the notch Izod impact strength of PP–mica–GMT is still high.

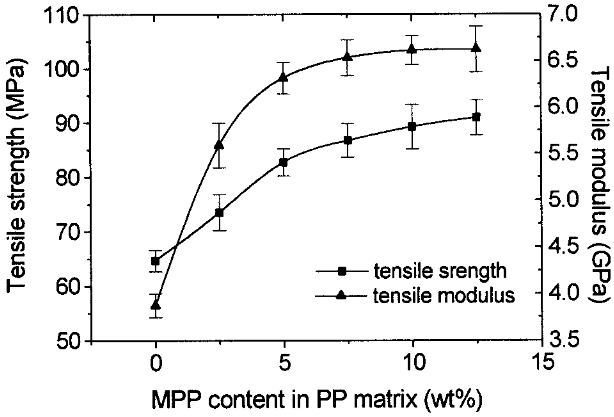


Figure 8 Effect of MPP content in the matrix on the tensile properties of PP–mica–GMT with 40 wt % mica content in the matrix.

Effect of the Addition of MPP

As discussed earlier in the first section, at a higher mica content level, the fiber–matrix adhesion is very poor for PP–mica–GMT. MPP has proven to be the most effective compatibilizer in enhancing interfacial bonding in both mineral filler-filled and glass fiber-reinforced PP composites.[13–17] Therefore, in this experiment, MPP prepared by the solid method was used as an interfacial adhesion promoter. The maleic anhydride (MAH) content of MPP was 1.5 wt %, detected by titration. To study the effect of the MPP content on the mechanical properties, the mica content was kept at 40 wt %.

The tensile properties as a function of the MPP content in the matrix are shown in Figure 8. It is found that both the tensile modulus and the tensile strength increase to a significant extent with the MPP content up to 5 wt %. Figure 9 shows the effect of MPP content on the impact strength, from which we can see that, with increasing MPP content, the impact strength gradually decreases.

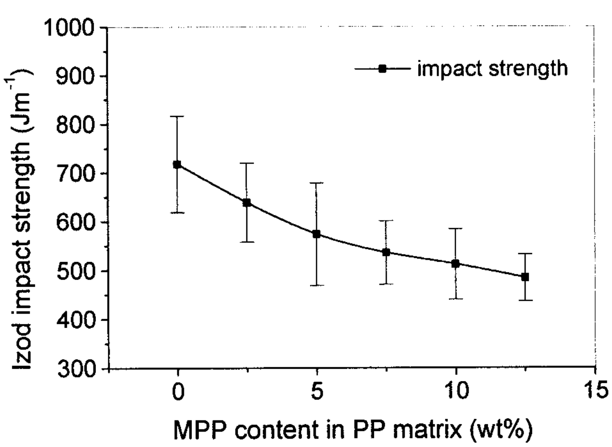
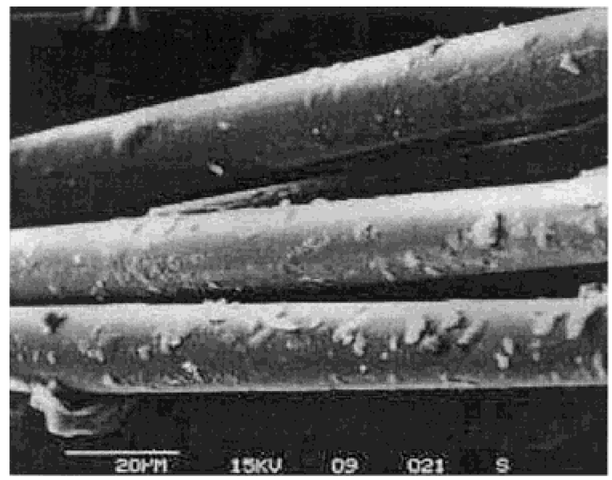
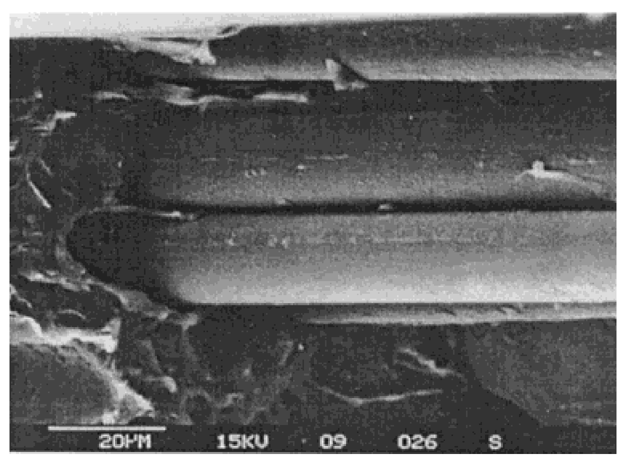


Figure 9 Effect of MPP content in the matrix on the impact property of PP–mica–GMT with 40 wt % mica content in the matrix.

Representative scanning electron micrographs of the pulled-out fiber surface for PP–mica–GMT containing MPP or not are presented in Figure 10. Figure 10(a) shows that, in the case of with 5 wt % MPP in the matrix, many platelike pieces adhere on the pulled-out fiber surface, indicating strong fiber–matrix adhesion. With respect to



(a)



(b)

Figure 10 SEM micrographs of the pulled-out glass fiber surface during the impact process for PP–mica–GMT with 40 wt % mica in the matrix: (a) with 5 wt % MPP in the matrix; (b) without MPP.

Table II Effect of Particle Size of Mica on the Properties of PP–Mica–GMT

	Tensile Properties		Flexural Properties		
Mesh Size of Mica (S)	Tensile Modulus (MPa)	Tensile Strength (MPa)	Flexural Modulus (MPa)	Flexural Strength (MPa)	Izod Impact Strength (Jm^{-1})
100	6313	82.7	8200	148.3	574
325	5338	67.8	8035	145.7	593
600	4693	61.1	6400	134.0	655

PP–mica–GMT without MPP, the pulled-out fiber surface is smooth and clean as shown in Figure 10(b), indicating poor fiber–matrix adhesion. It can be concluded that the addition of MPP effectively improves the interfacial adhesion between the fiber–matrix for PP–mica–GMT at a high mica content level, thus resulting in higher tensile properties. However, a strong interface is unfavorable to the fiber mat-related energy-absorbing capability for GMT. As a result, the Izod impact strength of PP–mica–GMT decreases with increasing MPP content.

Effect of Particle Size of Mica

With 5 wt % MPP and 40 wt % mica in the PP matrix, the effect of mica size on the mechanical properties of PP–mica–GMT was studied. Table II shows the effect of the particle size of mica on the mechanical properties of PP–mica–GMT. It is found that the tensile and flexural properties decrease as mica size decreases, whereas the impact strength shows a certain increase.

The effect of the particle size of mica on the mechanical properties of PP–mica–GMT can be related to the distribution of mica particles in GMT and the influence of mica particles on the fiber–matrix adhesion. The molten liquid of the mica-filled PP blend is a suspension of mica particles. During the impregnation process, this suspension penetrates into the glass mat. At the same time, the glass mat, which can be treated as a fiber bed, should have a filtration effect on the suspension.[18] Thus, the mica particles cannot easily penetrate into the glass mat, and, hence, the mica content inside the glass mat is lower than in the matrix. It is obvious that smaller mica particles can penetrate into the glass mat more easily than can large particles. Consequently, a decrease of mica size leads to an increase of the mica content inside the glass mat. So, the contact possibilities of glass fibers and mica particles at the fiber–matrix interface should be increased and fiber–matrix adhesion strength decreases to some extent. As a result, the impact strength increases and the tensile and flexural properties decrease as the mica size decreases. In addition, the reinforcing effect of mica decreases with decrease of the mica size, which also contributes to the decrease of the tensile and flexural properties. In the other sections of this article, the large mica particles with mesh size 100S were used to fill PP.

Effect of Glass Fiber Content

To study the effect of the glass fiber content on the mechanical properties of PP–mica–GMT, the mica and MPP content in the matrix were kept at 40 and 5%, respectively. Figure 11 shows the effect of the glass fiber content on the tensile strength and modulus of PP–mica–GMT. It can be seen that, with increasing glass fiber content, the tensile modulus exhibits a gradual increase. However, when the fiber content is over 15 vol %, the effect of the fiber content on the tensile modulus

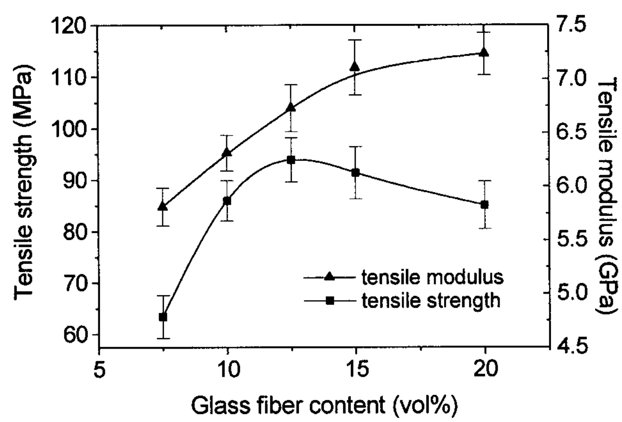


Figure 11 Effect of glass fiber content on the tensile properties of PP–mica–GMT (5 wt % MPP and 40 wt % mica in the matrix).

is no longer significant. With respect to the tensile strength, with increasing fiber content, it increases, then is followed by a decrease beginning at 12.5 vol % fiber content. It has been pointed that when the fiber content exceeds a certain level the reinforcing efficiency of the fibers will begin to decrease.[19,20] This is because of the imperfect impregnation of the reinforcement due to the higher content of the reinforcement. It was reported that the tensile strength of PP–GMT does not increase any longer when the glass fiber content is to 15 vol %.[19] In our work, it can be expected that this critical level of the glass fiber content (12.5 vol %) for PP–mica–GMT should be lower than that of PP–GMT as the total mineral reinforcement content is higher due to the high mica content in PP–mica–GMT. Figure 12 shows the effect of the glass fiber content on the impact strength of PP–mica–GMT. It can be observed that the impact strength increases continuously with the fiber content.

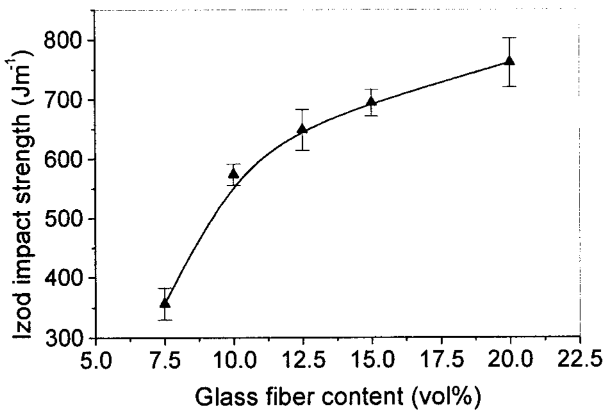


Figure 12 Effect of glass fiber content on the impact strength of PP–mica–GMT (5 wt % MPP and 40 wt % mica in the matrix).

CONCLUSIONS

In this study, mica-filled PP-based GMT was prepared by a double-belt press. From the investigation of the mechanical properties of PP–mica–GMT with different matrix formulations and varied fiber contents, the following results can be concluded:

The addition of mica has an obvious effect on fiber–matrix adhesion for PP–mica–GMT. At lower mica content, the fiber–matrix adhesion is enhanced, indicated by the small amount of fiber pull-out during the impact process. When the mica content is higher, the fiber–matrix adhesion strength is significantly decreased because of the contact of the glass fibers and the mica flakes at the interface evidenced by the SEM micrograph. As a result, at lower mica content, the tensile and flexural properties of PP–mica–GMT are higher; however, at a higher mica content, the tensile and flexural properties are lower. The Izod impact strength of PP–mica–GMT has a maximum value at 20 wt % mica content.

It was found that for PP–mica–GMT at a higher mica content level the addition of MPP significantly improved the tensile properties of PP–mica–GMT, whereas it decreased the impact strength. This is attributed to the fact that the addition of MPP promotes fiber–matrix interfacial adhesion. Large mica flakes are favorable to the tensile and flexural properties of PP–mica–GMT. However, the impact strength increases with decrease of the mica size. The tensile modulus of PP–mica–GMT containing 5% MPP and 40% mica in the matrix increased gradually with an increasing fiber content up to 15 vol %. The tensile strength increased up to 12.5 vol % fiber content and then decreased with more glass fiber content. The impact strength increased continuously with an increasing fiber content.

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