

Morphology and Mechanical Properties of Polyamide-6/K Resin Blends

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ABSTRACT: Mechanical properties and morphological studies of compatibilized blends of polyamide-6 (PA-6)/K resin grafted with maleic anhydride (K-g-MAH) and PA-6/K resin/K-g-MAH were investigated as functions of K resin/K-g-MAH and dispersed phase K resin concentrations, and all the blends were prepared using twin screw extruder followed by injection molding. Scanning electron microscopy (SEM) were used to assess the fracture surface morphology and the dispersion of the K resin in PA-6 continuous phase, the results showing extensive deformation in presence of K-g-MAH, whereas, uncompatibilized PA-6/K resin blends show dislodging of K resin domains from the PA-6 matrix. Dynamic mechanical thermal analysis (DMTA) test reveals the partially miscibility of PA-6 with K-g-MAH, and differential scanning calorimetry

(DSC) results further identified that the introduction of K-g-MAH greatly improved the miscibility between PA-6 and K resin. The mechanical properties of PA-6/K resin blends and K-g-MAH were studied through bending, tensile, and impact properties. The Izod notch impact strength of PA-6/K-g-MAH blends increase with the addition of K-g-MAH, when the K-g-MAH content adds up to 20 wt %, the impact strength is as more than 6.2 times as pure PA-6, and accompanied with small decrease in the tensile and bending strength less than 12.9% and 17.5%, respectively. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 107: 3804–3811, 2008

Key words: polyamide-6; K resin; mechanical properties; morphology; reactive compatibilization

INTRODUCTION

A majority of polymer blends are thermodynamically immiscible in nature due to the low entropy of mixing. Binary blends of two immiscible components form coarse and unstable phase morphology with high interfacial tension and low interfacial adhesion.^{1,2} As a result, the mechanical properties of immiscible polymer blends are inherently inferior in nature. To overcome this problem, reactive compatibilization^{3–7} is very often used to obtain blends with desirable properties. This method is based on the formation of a block or grafted copolymer A-B at the interface between the blend phases during melt blending. Dedecker et al.⁵ reactively compatibilized the polyamide-6/poly(phenylene oxide) blends by means of styrene-maleic anhydride copolymer; Thomas³ blended ethylene-propylene rubber (EPM) with nylon-6, and the blends are immiscible and highly incompatible, therefore a reactive route was employed to compatibilize these blends by addition of maleic anhydride grafted EPM (EPM-g-MA);

Charoensirisomboon et al.^{6,7} blended PA-6 with nonreactive polysulfone (PSU) and small amount of reactive PSU, phthalic anhydride-terminated PSU (PSU-PhAH), which involved *in situ* reactions to form a grafted copolymer and played a role of the emulsifier to prevent particles coalescence and to improve interfacial adhesion; Wei et al.⁸ employed Low-density polyethylene (LDPE) functionalized by melt radical grafting with glycidyl methacrylate (GMA) for reactive blending with polyamide-6 (PA-6)/low density polyethylene (LDPE); Hu et al.⁹ investigated the compatibility of nylon 11/polyethylene (PE) alloys on addition of reactive maleated ethylene-propylene-diene copolymer (EPDM-graft-MAH); and Sailer and Ulrich¹⁰ investigate the influence of reactive compatibilization of SANMA on the rheological properties of polyamide 6/styrene-acrylonitrile (PA 6/SAN) blends in the melt.

Since most polymer blends do not have the appropriate functional groups, it is required to functionalize the components of the blends. In some cases, it is possible to add a third polymer into the blend, which is miscible with one of the blend component and reactive with the other component.

Because of the inherent chemical functionality, polyamide-6 is an attractive candidate for modifica-

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TABLE I
Composition Ratios for PA-6 and K-g-MAH Blends

	1	2	3	4	5	6	7	8	9	10	11
PA-6	100	95	90	85	80	70	80	75	70	60	80
K-g-MAH	0	5	10	15	20	30	10	10	10	10	
K resin							10	15	20	30	20

tion. Numerous authors describe the approaches that improve the toughness of this material by reacting a polymer containing an appropriate chemical functionality with the amino end groups of the PA-6 during melt processing.^{11–29} Wan has used HDPE-g-MAH¹² to toughening PA-6. The carboxy end groups of MAH-g-HDPE compatibilized the blend during melt blending and formed PA-6-MAH-g-HDPE copolymer. Others such as LDPE-g-MAH,¹³ SEBS-MAH¹⁴ or engineering plastic PC,^{22–25} PPO,²⁶ inorganic pellets,³⁰ and nanoparticles^{31,32} have been used to toughening PA-6.

K resin is a copolymer with high content styrene and high modulus. Though the compatibility of PA-6 and K resin is very poor, it has been claimed that the grafted K resin-g-maleic anhydride (MAH) which prepared by means of melt grafting method, exhibits good miscibility with PA-6. It is also to be pointed out that there are very few literatures available on PA-6/K resin blends where K-g-MAH has been utilized as a reactive compatibilizer. The aim of this article is to address the effects of reactive compatibilization using K-g-MAH on the mechanical properties and morphology of the binary and ternary blends of PA-6, K resin, and K-g-MAH.

EXPERIMENTAL

Materials

Polyamide-6 was supplied by YueYang Juli Engineering Plastic Co. Hunan. The melt flow rate (MFR) at 230°C and 0.325 kg loading) is 0.128 g/10 min. K resin was obtained from Philip Corp., Korea (styrene/butadiene = 7/3) (MFR at 220°C and 2.16 kg loading is 3.825 kg/10min). Maleic anhydride and styrene were provided by Santou Xilong Chemical Factory, Guangdong.

Blending and preparation of test specimen

K resin grafted with maleic anhydride (K-g-MAH) was prepared by means of melt grafting method. The temperature settings along the extruder barrel ranged from 180 to 195°C with the higher temperature zone close to the extrusion die. The grafting ratio of this kind of K-g-MAH is 0.54 wt % measured by chemical titrimetry,³³ and the MFR of the graft

copolymer (at 220°C and 10 kg loading) is 2.692 kg/10 min.

Granules were dry-mixed in appropriate ratios to keep the predominant mechanical properties of PA-6, and the binary as well as ternary blends of PA-6, K resin and K-g-MAH were prepared in a twin screw extruder ($L/D = 25$) 225°C. The detailed blend compositions are given in Table I. The extruded strands were quenched immediately after extrusion in a water bath kept at room temperature, and then chopped into granules and finally dried in a vacuum oven at 100°C for over 8 h. Test specimens for determining the mechanical properties were prepared by injection molding.

Morphology examination

Morphological studies were conducted by scanning electron microscopic (SEM) (JEOL. JSM-5600LV, Japan). The impact fractured specimens were sputter coated with gold prior to SEM examination. Cryogenically fractured etched impact specimens were used for phase morphological analysis. The etching was carried out by styrene to remove K resin (or K-g-MAH) selectively from the respective blends samples.

Dynamic mechanical thermal analysis

The mechanical loss factor ($\tan \delta = E''/E'$) as a function of temperature (T) was assessed by dynamic mechanical thermal analysis (DMTA) using a DMTA-V system. DMTA spectra were taken at a fixed frequency of 1 Hz, and heating rate of 2°C/min in a broad temperature range ($T = -80$ to 150°C). Specimen dimensions were 25 mm \times 4 mm \times 2 mm.

Thermal analysis

The melting and crystallization behavior of pure PA-6 and all the blends were studied under nitrogen atmosphere by differential scanning calorimetry (DSC-Q10), using 8–10 mg sample sealed into aluminum pans. To avoid any effect of moisture, all the test specimens were dried using vacuum oven at 80°C prior to the measurements. The temperature was raised from 50 to 250°C at a heating rate of

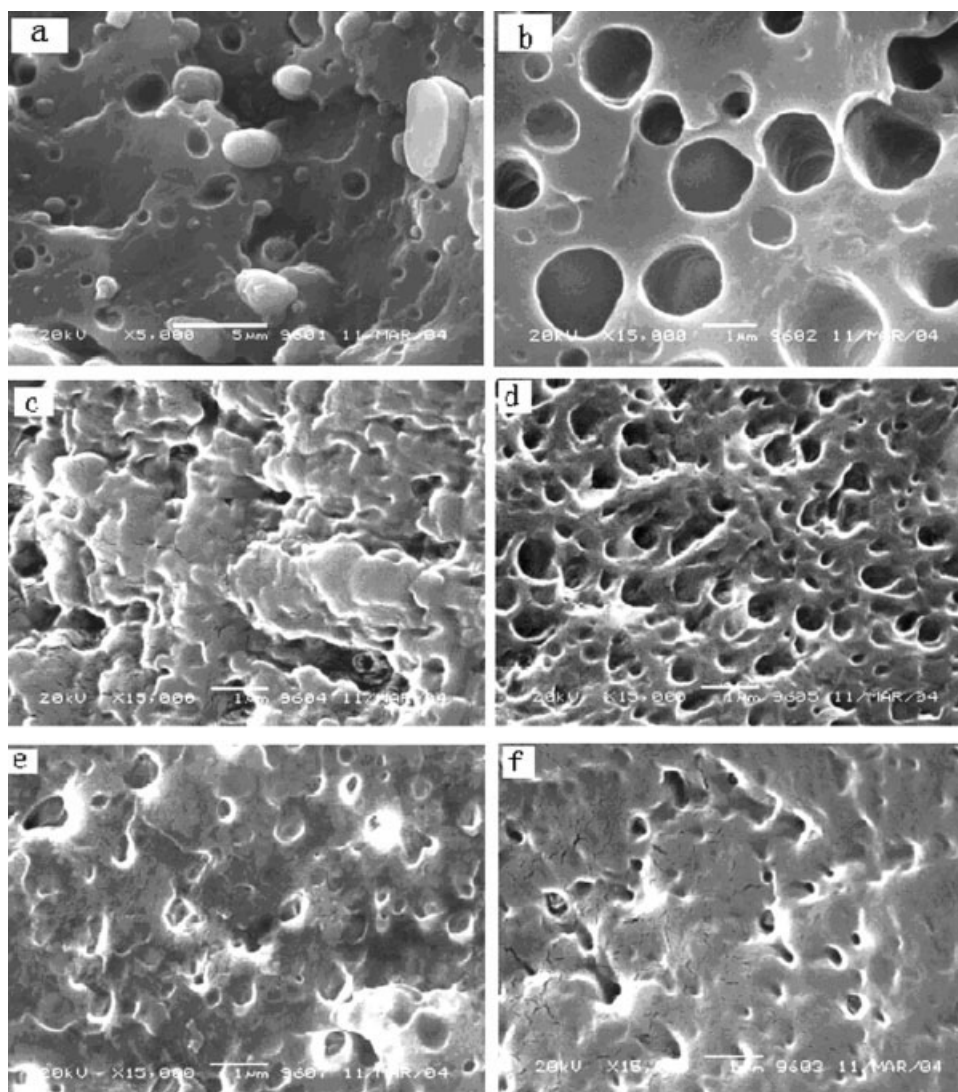


Figure 1 SEM micrographs of impact fractured surfaces of PA-6/K-g-MAH/K resin blends; (b, d, f) were etched by styrene and (a, c, e) were not etched: (a, b) PA-6/K resin 80/20; (c, d) PA-6/K-g-MAH 80/20; (e, f) PA-6/K-g-MAH/K resin 80/10/10.

10°C/min, and kept for 5 min at this temperature to eliminate the effect of previous thermal history, then it was swept back at $-10^{\circ}\text{C}/\text{min}$. Second heating similar to the first was then performed to erase the thermal history. The melting thermogram was recorded from the second heating.

Mechanical properties

Izod notch impact strength was measured on XJJ-50 impact tester following GB1043-93. All impact strength measurements were carried out under dry states. Tensile and bending properties were measured on a microcomputer-controlled Universal Tester (RG.T-5A) in accordance with GB1042-92 and GB9341-88, respectively. The test results are the average values five times tested in each case.

RESULTS AND DISCUSSION

Morphology analysis

The micromorphology of the fracture surfaces of PA-6/K-g-MAH/K resin blends were investigated by SEM. The difference between brittle and ductile fracture can be distinguished from the fracture surface of the samples. On a brittle fracture surface (K resin), stress whitening can only be observed at the notch tip, but for a tough fracture (K-g-MAH), all the material around the fracture surface involved in stress whitening, crazing, and formed shear-banding (Fig. 6), which are related to the impact strength.

The SEM micrographs of the Izod notched impact fractured surfaces of PA-6/K-g-MAH and PA-6/K/K-g-MAH blends together with PA-6/K resin blends are presented in Figure 1. The samples in Figure

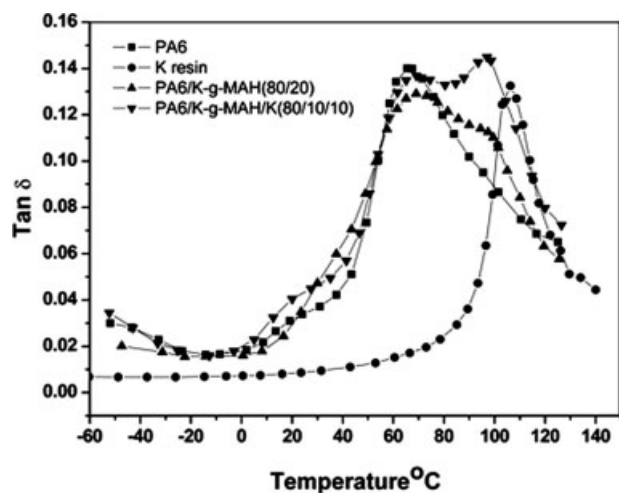


Figure 2 The relationship between $\tan \delta$ and temperature for PA-6 blends.

1(b,d,f) were etched by styrene and in Figure 1(a,c,e) were not. Figure 1(a,b) present the micromorphology fracture surface of PA-6/K resin blend and show debonding of K resin particles with hemispherical bumps indicating little adhesion between PA-6 and K resin. The size of the dispersed K resin particles is about 2–3 μm , some of them are as large as 5 μm , which will influence the mechanical properties of PA-6/K resin blend. The 80/20 PA-6/K resin blend is characterized by brittle fracture and there is no sign of plastic deformation or cavitation of K resin particles. On impact, only notch tip undergoes stress whitening. On the contrary, the entire fracture surface undergoes stress whitening in the reactively compatibilized blends. Figure 1(c–f) show that reactively compatibilized blends exhibit rumples with shear bands formation in some compositions. This suggests that high rate of plastic deformation is associated with the fracture. The SEM micrographs of PA-6/K-g-MAH blends are alike with little variety of particle size. Figure 1(c,d) display SEM micromorphology of the fracture surface of PA-6/K-g-MAH blends, the fracture surface deformed seriously like cellucotton and it is impossible to distinguish two phases [Fig. 1(c)], and the cavities formed after etching are irregular and around the boundary, there appears grave stringing and great shear-band, this phenomenon is typical of tough fracture, and it indicates that PA-6 has partial compatibility with K-g-MAH, it may be attributed to the active reaction between the anhydride groups of K-MAH graft and amino groups^{34,35} of polyamide-6 during the melt-blending process. Figure 1(e,f) reveal the micromorphology of the fracture surface of PA-6/K resin after the addition of 10 wt % K-g-MAH. The sizes of dispersed K resin pellets decreased distinctly comparing with Figure 1(a,b) and dispersed homogeneously, the interfaces between two phases were am-

biguous, and the cavities formed after being etched by styrene [Fig. 1(f)] were not smooth and regular and with stress whitening around them. So, it is concluded that there are considerable plastic deformation of the dispersed K resin particles occurred during the impact test and intensive stress delivery appeared between the dispersed and continuous phases. This is believed to be due to better interfacial adhesion between PA-6 and K resin in the presence of maleic anhydride. Similar kind of observations has also been reported in other blends system by Arup and Anup.³⁶ This strongly suggests that on addition of K-g-MAH, the interfacial adhesion between PA-6 and K-g-MAH improved significantly.

Dynamic mechanical thermal properties

The miscibility property between PA-6/K-g-MAH blends was studied by DMTA, and the turning peak

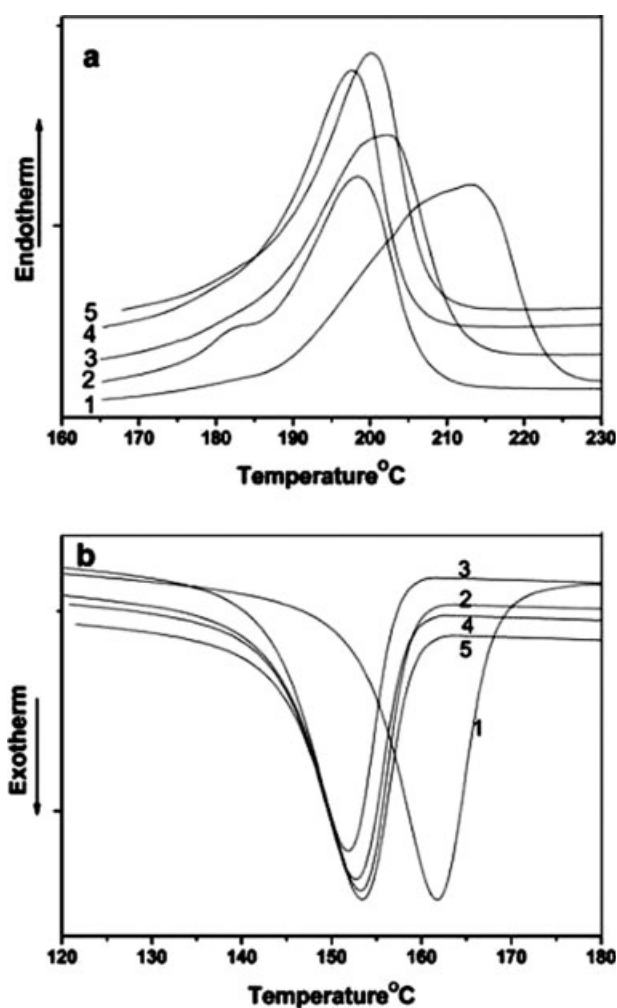


Figure 3 The melting (a) and crystallization (b) behavior of PA-6/K-g-MAH blends with different K-g-MAH contents. (1: PA-6/K-g-MAH 100/0; 2: PA-6/K-g-MAH 95/5; 3: PA-6/K-g-MAH 90/10; 4: PA-6/K-g-MAH 80/20; 5: PA-6/K-g-MAH/K 80/10/10).

TABLE II
DSC Crystallization Parameters PA-6/K-g-MAH/K Blends

PA-6/K-g-MAH/K	100/0/0	95/5/0	90/10/0	80/20/0	80/10/10
T_m (°C)	212.9	202.1	198.6	197.7	200.2
T_c (°C)	161.8	153.2	152.8	152.0	152.6
ΔH_m (J/g)	46.4	43.5	34.9	29.3	30.5
X_c (%)	24.2	24.0	20.4	19.3	20.1

temperature of the loss factor α ($\tan \delta$) is corresponding to the glassy transition temperature (T_g). The loss factor $\tan \delta$ versus temperature are plotted in Figure 2. The peak at 105.3°C is the T_g of K-MAH, and the pure PA-6 exhibits a $\tan \delta$ peak at 68.4°C. Compared with PA-6 with and without K-MAH, the T_g of PA-6 shift to high temperature and the T_g of K-MAH phase shift to low temperature for the PA-6/K-g-MAH blends. As known that if the blend shows a single transition or two transitions at intermediate temperature between those of the pure components, the blend is miscible or partially miscible.³⁷ So, Figure 2 shows increased miscibility of PA-6 with K-g-MAH copolymer.

Crystallization behavior: DSC analysis

DSC was used to study the melting and crystallization behavior of PA-6 blends and the corresponding results are shown in Figure 3. It is evident from Table II that the melting point of PA-6 shifts to lower temperature with the increase of K-g-MAH content in PA-6/K-g-MAH/K blends. Therefore, the miscibility of PA-6 and K-g-MAH was further improved according to the T_m depression criterion,³⁸ which is originated from the compatibilization of K-g-MAH. The chemical reactions taking place in the PA-6/K-g-MAH/K blends will inevitably affect the

crystallization process. The crystallization peak temperature of PA-6 decreased significantly on increasing K-g-MAH content. This suggests the hindrance of PA-6 chains in presence of K-g-MAH copolymer. The lowering of heat of crystallization of PA-6 in the blends of PA-6/K-g-MAH/K with higher K-g-MAH content suggests the formation of graft copolymer between the amino end group of PA-6 and anhydride functionality in the interface. This graft copolymer, in turn, affects the mobility of PA-6 chain and lows the crystallization. Such effect has been reported by Atchara and Manit for PA-6/LDPE blends compatibilized with Na-EMAA³⁹ or an ethylene-acrylic acid copolymer and a low molar mass bisoxazoline,⁴⁰ and by Arup and Anup for PA-6/EVA blends compatibilized with MAH.³⁶

Mechanical properties

Figures 4 and 5 show the Izod notch impact strength, tensile and bending strength PA-6/K-g-MAH blends as functions of K-g-MAH content range from 0 to 30 wt % in these blends, respectively. It is seen from these two figures (Figs. 4 and 5) that Izod notch impact strength of pure PA-6 is about 11.2 kJ/m²; however, when 5 wt % K-g-MAH was added, the impact strength greatly increased, while the tensile and bending strength just decreased slightly, and with the increasing addition of K-g-MAH, the impact

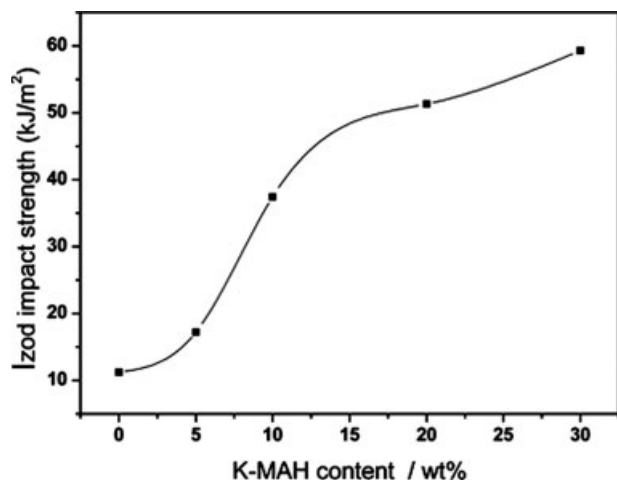


Figure 4 Effect of K-g-MAH content on Izod notch impact strength of PA-6/K-g-MAH blends.

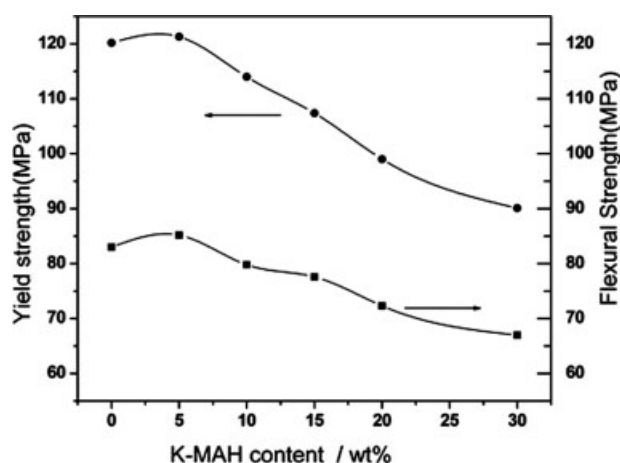


Figure 5 Relationship between tensile and bending strengths of PA-6 blends and K-g-MAH content.

TABLE III
Mechanical Properties of PA-6/K Resin Blends Toughened by K-g-MAH

PA-6/K-g-MAH/K (wt%)	100/0/0	80/10/10	75/10/15	70/10/20	60/10/30	80/0/20
Tensile strength (Mpa)	74.0	64	61.1	56.1	48.3	60.8
Bending strength (Mpa)	112.2	93	86.3	82.5	72.6	105.4
Bending modulus (Mpa)	1976.3	1825.1	1721.7	1629.7	1448.1	1821.3
Impact strength (kJ/m ²)	11.1	35.6	34.1	15.7	15.0	9.6
Elongation at break (%)	92.6	213.3	208.6	167.1	71.5	48.1

strength of PA-6/K-g-MAH blends were greatly increased concomitant with slight decrease of tensile and bending strength. Generally speaking, the Izod notch impact strength increases while the tensile and bending strength decrease with the continue increasing of K-g-MAH content in the experiment range (0–30 wt %). Comparing the two figures (Figs. 4 and 5), it reveals that when the K-g-MAH content adds up to 20 wt %, the Izod notched impact strength increased to as 6.2 times higher as pure PA-6, and tensile and bending strength decreased by 12.9% and 17.5%, respectively.

The mechanical properties of PA-6/K resin blend compatibilized with and without K-g-MAH are detailed as Table III. It is obvious that the mechanical properties of PA-6/K-g-MAH (80/20) without K-g-MAH were inferior to pure PA-6, while after adding K-g-MAH to PA-6/K resin blends, the mechanical properties of these blends system were greatly improved. But it is also seen that the application of K-g-MAH in compatibilizing PA-6 blends has limita-

tion. When the composition ratio of K-g-MAH/K resin is larger than 1 : 1.5 (K-g-MAH 10 wt %), the tensile and bending strength, bending modulus and elongation at break decrease greatly while impact strength increases a little, and the compatibilization of K-g-MAH is poor in this composition. So there is an optimum K-g-MAH content (wt %) for well compatibilization effect in this experiment, when the composition ratio of PA-6/K-g-MAH/K resin blend is 80/10/10, the blend gets the optimal mechanical properties.

Fracture mechanism

The micrographs of the fracture surface of PA-6/K-g-MAH show that crazing and shear-banding of the matrix are the major toughening mechanism in these impact modified PA-6. The crazing and shear-banding are initiated by the stress concentrations of K-g-MAH particles. And the toughening mechanism can be modeled as Figure 6.

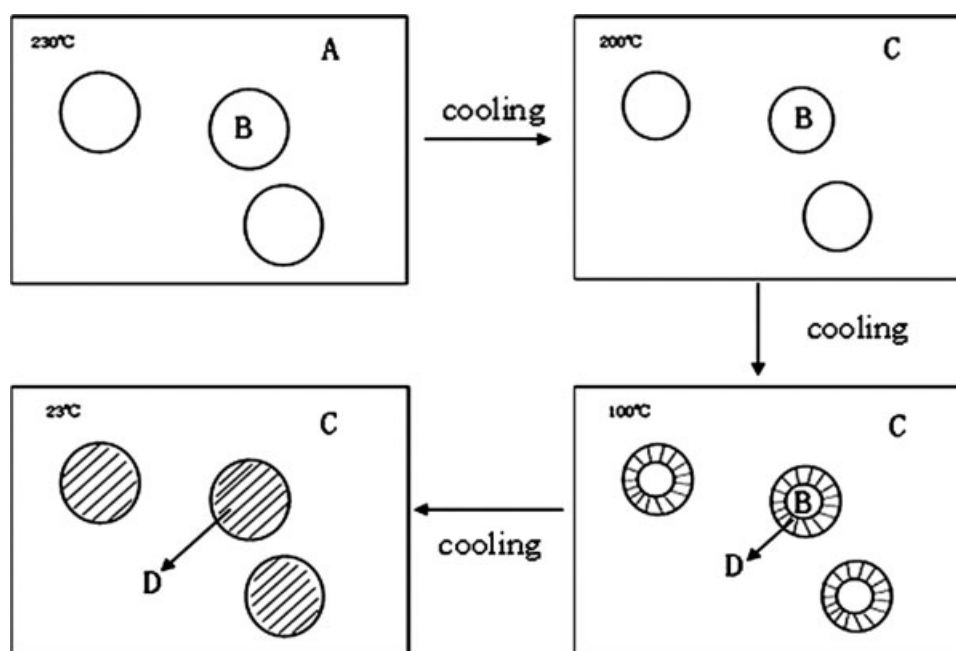


Figure 6 Schematic diagram for the formation of crazing of PA-6/K-g-MAH blends (A: Melting PA-6; B: Melting K-g-MAH; C: Solid PA-6; D: Glass phase K-g-MAH).

Melt grafting maleic anhydride with K resin (which with high content styrene and high modulus), the MAH grafted with the butadiene group.⁴¹ K-g-MAH can partially react with PA-6 and form PA-6-MAH-g-K copolymer. For the different glassy transition temperature T_g (the T_g of K-g-MAH higher than PA-6), crazing layer occurred on the surface of K-g-MAH pellets during the injection molding process at the temperature around 90°C with melting and glass K-g-MAH phases coexisting, the K-g-MAH segments in PA-6-MAH-g-K copolymer which cross-linked with K-g-MAH pellets are stretched and plastic deformed. When the temperature decreases to between the glassy transition temperatures of the two materials, PA-6 segments of PA-6-MAH-g-K copolymer have being stretched and deformed till the temperature decreases to below the T_g of PA-6, then PA-6 segments are frozen up, and the craze layer on K-g-MAH pellets surfaces are subdivided into multi-craze. Meanwhile, the two phases' surfaces are partially deformed and when they suffer from exterior forces, K-g-MAH pellets are firstly deformed as its small modulus and induce surrounding matrix producing much crazes and shear-banding^{42,43} which absorb substantive energy. The PA-6-MAH-g-K copolymer plays important roles not only in transmitting stresses between two phases, but also in preventing the cracks' further enlargement, and K-g-MAH pellets and the shear-banding can also hinder and terminate the progress of the existed small crazes. PA-6 blends absorbed considerable energies through induce-deformation, production of multi-crazes, progress and formation of shear-banding, and result in great improvement of the toughness.

CONCLUSIONS

The effect of K-g-MAH on phase morphology and mechanical properties of PA-6/(K resin) blends was investigated in this study. Experimental results show that MAH-grafted K resin (K-g-MAH) particles were very effective to modification the mechanical properties of PA-6/K-g-MAH and PA-6/K/K-g-MAH blends. The major conclusions of the present study are as follows:

1. The reactive grafted copolymer K-g-MAH was prepared by means of melt blending, and act as a compatibilizer to improve the interfacial adhesion between PA-6 and K resin and the properties of PA-6 blends.
2. DMTA tests prove that PA-6 was immiscible with K resin and partially miscible with K-g-MAH copolymer. Compared with PA-6 with and without K-MAH, the T_g of PA-6 shift to high temperature and the T_g of K-MAH phase shift to low temperature for the PA-6/K-g-MAH

blends, which means a increased miscibility of PA-6 with K-g-MAH copolymer.

3. DSC curves show the T_m of PA-6 shift to low temperature with the increase in K-g-MAH content, the overall crystallinity of the reactively compatibilized blends decreased as compared to pure PA-6 which suggests the reduced mobility of crystallizing chains due to the graft copolymer formation at the interface.
4. The Izod notch impact strength was greatly improved with the increase of K-g-MAH content in PA-6/K-g-MAH and PA-6/K/K-g-MAH blends, respectively. The tensile strength of PA-6/K-g-MAH and PA-6/K/K-g-MAH slightly decreased as compared to pure PA-6. The theoretical analysis of impact strength suggests that there is an increase in extent of interaction between PA-6 and K-g-MAH, which is also an indication of better interfacial adhesion between PA-6 and K resin.
5. The micrographs taken in the Izod notch impact fracture surface of PA-6 blends reveal that the stress whitening is a result of cavitation within the K resin particles. The crazing and shear-banding are initiated by the stress concentration associated with K resin or K-g-MAH particles. Consequently, the toughness of PA-6 blends is greatly improved.

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