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Effect of zinc dimethacrylate on compatibilization and reinforcement of peroxide dynamically cured PP/EPDM TPVs

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Thermoplastic vulcanizates (TPVs) based on polypropylene (PP)/ethylene–propylene–diene (EPDM) with a ratio of 40/60 (w/w) were *in situ* compatibilized by zinc dimethacrylate (ZDMA) *via* peroxide induced dynamic vulcanization. The effects of dynamic vulcanization and ZDMA on the compatibility between PP and EPDM phases and the mechanical properties of PP/EPDM TPVs were investigated. The incorporation of ZDMA improved the melt viscosity of the resultant TPVs. Dynamic mechanical analysis (DMA) demonstrated that the increase in ZDMA concentration improved the compatibility between EPDM and PP phases. Scanning electron microscopy (SEM) results showed that the incorporation of ZDMA reduced the size of crosslinked rubber particles. Differential scanning calorimetry (DSC) was conducted to investigate the crystallization behavior and the results indicated that the addition of ZDMA promoted the nucleation process of PP, but a higher ZDMA concentration showed a negative effect on the crystallinity of the PP component. Furthermore, it was found that the mechanical properties of the TPVs were significantly improved by ZDMA. Particularly, with about 18 wt % ZDMA in the PP/EPDM TPV, the tensile strength and elongation at break increased from 7.7MPa and 250% to 13.4MPa and 410%, respectively.

Key words: reinforcement; thermoplastic vulcanizates; compatibility; dynamic vulcanization; mechanical properties

INTRODUCTION

Thermoplastic vulcanizates (TPVs) are an important family of thermoplastic elastomers (TPE) since their introduction in 1970s [1]. They are prepared by melt mixing of thermoplastic and rubber in the presence of a curing agent. TPVs are blends where the rubber component is *in situ* crosslinked during melt mixing with thermoplastics at a certain temperature, using a curing agent. The way to prepare TPVs is called dynamic vulcanization [2-4]. The crosslinking of the rubber component causes a variation of the rubber/plastic viscosity ratio, which results in a change of phase morphology [5, 6]. TPVs combine the elasticity and mechanical properties of traditional rubber vulcanizates with the processing characteristics of thermoplastics [7]. Therefore, TPVs can be processed by injection, extrusion, compression, etc., which improves the productivity and reduces costs. TPVs have experienced a rapid development due to their good mechanical properties and processability.

Among the different kinds of TPVs, PP/EPDM TPVs have received considerable attention because of their low cost and general availability in household, automobiles, appliances, construction industry and so on [8-11]. However, as PP and EPDM are not thermodynamically miscible, it is necessary to improve the compatibility between PP and EPDM phases. There are only a few studies carried out in this field. A. Mirzadeh [12] *et al.* investigated the effects of various dispersion levels of nanoclay on the crosslinking reaction of PP/EPDM TPV. They found that nanoclay affects the crosslinking reaction through its barrier effect which increases the local concentration of curing agent, and dynamic vulcanization improved dispersion and intercalation of nanoclay. De Risi [13] *et al.* introduced zinc dimethacrylate (ZDMA) into peroxide dynamically vulcanized PP/EPDM TPV and found that more peroxide free radicals reacted with the radicals on the ZDMA molecule which limited chain fragmentation of the PP. They paid attention to the importance of ZDMA in minimizing the chain fragmentation rather than the potential improvement of interface adhesion and improved mechanical properties of the TPVs.

In recent years, it was found that some types of rubbers can be reinforced by a high loading of ZDMA (chemical structure is shown below) improving crosslink densities. On the other hand, ZDMA can also form polymerized ZDMA (PZDMA) which dispersed in the matrix or grafted onto rubber chains to enhance the crosslink network in the presence of peroxide free radicals [14-16].

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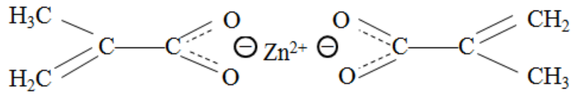
Therefore, it may be a feasible way to prepare PP/EPDM/ZDMA TPVs *via* peroxide induced dynamic vulcanization to enhance the interface adhesion and the mechanical properties in order to extend their application.



In our previous works we found that the incorporation of ZDMA into PP/EPDM blend *via* peroxide-induced dynamic vulcanization largely improved the toughness of PP-rich blends due to the *in situ* reactive compatibilization of ZDMA [17-19]. We also found that ZDMA showed excellent reinforcement on EPDM-rich TPVs with various PP/EPDM ratios [20, 21]. However, the effect of ZDMA concentrations on the mechanical properties of EPDM-rich PP/EPDM TPV has been not studied yet. In this paper, we fixed the PP/EPDM ratio at 40/60 and prepared TPVs in the presence of ZDMA. The effect of ZDMA concentrations on the processing was evaluated by the torque of mixing. The morphology, dynamic mechanical properties, crystallization behavior, and mechanical properties of the resultant TPVs were studied in detail.

EXPERIMENTAL

Materials

Polypropylene (PP, HP500N, MFI=12g/10 min (210°C, 2.16 kg)) was purchased from CNOOC & Shell Petrochemicals Company Limited., China. Ethylene-propylene-diene monomer (EPDM4045, density=0.87g/cm^3, 56% ethylene content, M_L (1+4, 100°C) = 40, 7.5% the third monomer: (5-ethylidene-2-norbornene) was obtained from Jilin Chemical Co., China. ZDMA was purchased from Xian Organic Chemical Technology Plant (Shanxi, China). Dicumyl peroxide (DCP) was obtained from Sinopharm Chemical Reagent Co. Ltd., China.

Compositions and Preparation of TPVs

Three weight ratios of EPDM/ZDMA were employed in this study: 100/10, 100/20 and 100/30 (w/w) [18]. Dynamically vulcanized PP/EPDM/ZDMA TPVs were prepared with a PP/EPDM weight ratio of 40/60 (w/w). The weight ratio of DCP was maintained constant relative to the amount of EPDM. The compositions in terms of the weight ratios of components for PP/EPDM/ZDMA TPVs are presented in Table 1.

Two-step processing method was employed to prepare the PP/EPDM/ZDMA TPVs, in which EPDM and ZDMA were mixed to get rubber compounds, and then the rubber compounds were blended with PP melt. The detailed process can be found in our previous reports [20, 21].

Table 1. Formulations of the prepared samples (weight ratio)

Coding	PP	EPDM	ZDMA	DCP
B46	40	60	0	0
TZ46-0	40	60	0	0.6
TZ46-1	40	60	6	0.6
TZ46-2	40	60	12	0.6
TZ46-3	40	60	18	0.6

Dynamic Mechanical Analysis

Dynamic mechanical behavior of the samples was investigated using a dynamic mechanical analyzer (DMA 242C NETZSCH, Germany) in tensile mode at 10Hz with a heating rate of 3°C /min in the temperature range of -100 to 80°C.

Morphology Analysis

Nova NanoSEM 430 (FEI Company, Holland) was used to investigate the phase morphology of the samples. Before morphological observation, the cryogenically fractured surface of the specimens was pre-treated as follows: etching by boiling cyclohexane to remove non-crosslinked EPDM phase or etching by boiling xylene to remove PP and non-crosslinked EPDM phase.

Differential scanning calorimetry (DSC)

The crystallization behavior of the blends was studied by means of DSC (NETZSCH DSC 204 F1, Germany). In the tests, samples of about 5 mg were heated to 200°C with a rate of 10°C/min in nitrogen atmosphere and held at 200°C for 5 min to eliminate the thermal history. Afterward, the samples were cooled to 20°C with a rate of 20°C/min, let at 20°C for about 3 min, and then heated again to 200°C with a rate of 10°C /min. The degree of crystallinity (Xc%) was calculated according to Eq. (1):

$$X_c=\Delta H_c/(\Delta H_o*\varphi)\times 100\% \qquad (1)$$

where ΔH_c is the heat fusion of PP during crystallization, ΔH_o=209 J/g, represents the heat fusion of 100% isotactic PP and φ is the mass fraction of PP in the composites.

Mechanical properties

Standard tests of tensile and tear strength were conducted on a Computerized Tensile Strength Tester (UT-2080, U-CAN Dynatex Inc, Taiwan) at room temperature according to ISO 37–2005 and ISO 34–2004, respectively. All tests were repeated at least 5 times, and the results were averaged.

RESULTS AND DISCUSSION

Torques during dynamic vulcanization

Fig. 1 shows the torque curves *versus* mixing time for all samples. The first and second peaks correspond to the loading and melting of PP and EPDM (or EPDM/ZDMA compound), respectively. After EPDM is completely melted, the torque reaches a rather constant value, indicating the complete melting of PP and the full homogenization of the blends. The torque of the blends abruptly increases when DCP is added to the melt. This is related to the drastic changes in the viscosity and elasticity of the EPDM phase due to crosslinking. After passing through a maximum, the torque decreases until reaching a new constant value at the end of mixing. The decrease in torque is partly due to the deagglomeration of the physical networks formed by the crosslinked rubber droplets. Considering the strong reduction of torque, we believe that the DCP still induces the degradation of PP even in presence of ZDMA. However, note that for the PP/EPDM TPV (TZ46-0), the final stable torque is lower than that of PP/EPDM blend (B46), which indicates that degradation of PP occurs in the presence of peroxide free radicals. While for all the PP/EPDM/ZDMA TPVs, the final stable torques are higher than those before adding DCP, indicating that ZDMA limits the chain fragmentation of PP by consuming the free radicals, and thus the final stable torque shows an increase with increasing ZDMA concentrations, as expected.

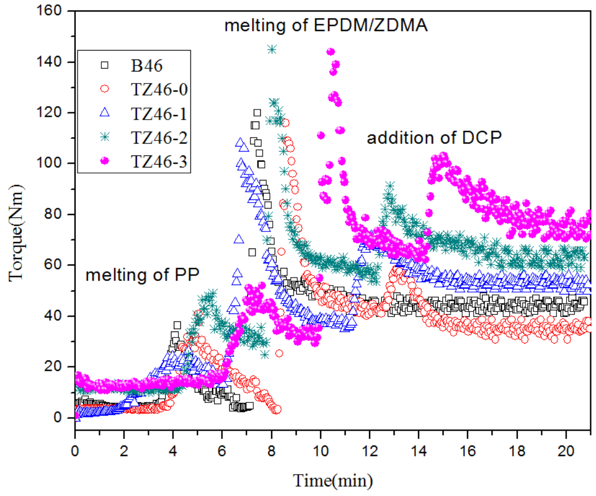


Fig. 1. Torques *versus* mixing time for all specimens.

DMA Analysis

Fig. 2a shows the effect of temperature on the storage modulus (E'). Each specimen shows a two-step decrease of E' value at temperatures ranging between -70~-30°C and -20~40°C, which corresponds to the glass transition of the EPDM phase and PP phase, respectively.

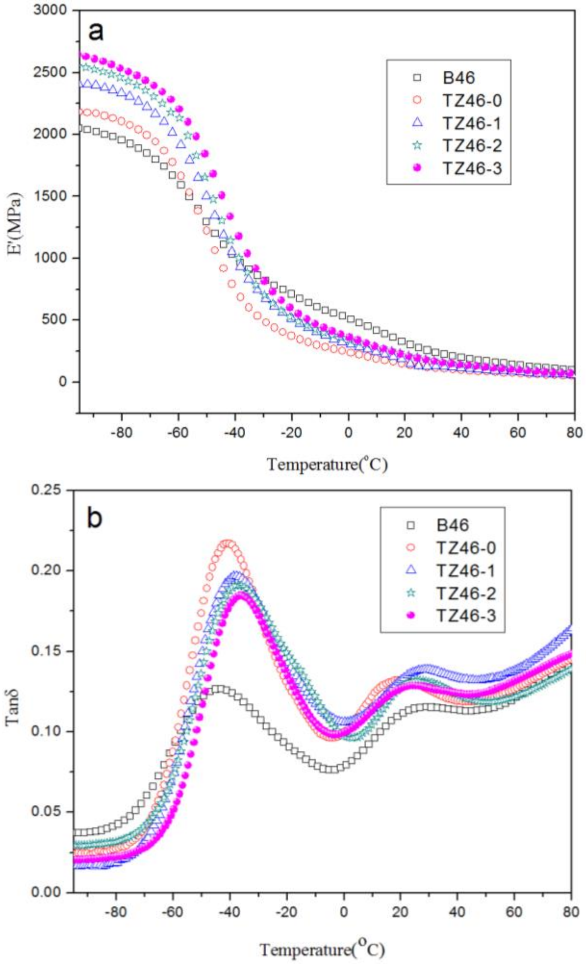


Fig. 2. Storage modulus (a) and loss tangent (b) *versus* temperature for all specimens.

It can be seen that TZ46-0 shows a higher E' value in the glass region than B46, which may result from the crosslinking of EPDM during dynamic vulcanization. Incorporation of ZDMA further improves the E' value showing an increase with increasing ZDMA concentrations below the glass transition temperature. The *in situ* polymerization of ZDMA can form homopolymer particles (h-PZDMA) and graft products (g-PZDMA). The h-PZDMA particles dispersed in the EPDM phase strongly reinforce the rubber droplets [19], contributing to the increased E'. The *in situ* compatibilization of ZDMA occurs at the interfacial layer between the PP and EPDM phases through the reaction between the double bonds of ZDMA and the free radicals generated in both EPDM and PP chains, forming graft products which improve the interface adhesion between PP phase and reinforced EPDM phase [18]. However, note that the E' of the B46 shows a higher value than other TPVs at temperatures above -30°C because E' is mainly contributed by the PP phase.

Fig. 2b shows the temperature dependence of tan δ. The strong one at around -37°C corresponds to the glass transition of the EPDM phase and the weak one at around 22°C is related to the glass transition of the PP phase. It is clearly seen that dynamic vulcanization apparently improves the Tg of EPDM phase due to the crosslinking of EPDM restricting the mobility of rubber chains. Moreover, Tg of the EPDM phase increases from -41.3°C to -38.3°C with a ZDMA concentration of 10 wt% relative to the amount of EPDM. The increasing ZDMA concentration further increases the Tg of EPDM phase, which can be well explained by the resultant denser rubber network restricting the mobility of rubber chains. In addition, B46 shows higher tan δ values than TPVs in the lower temperature range due to the entanglement of EPDM chains under dynamic vulcanization, which makes it more difficult to move.

Morphology Analysis

Fig. 3 shows the morphology of the cryogenically fractured surface for PP/EPDM blend (B46), PP/EPDM TPV (TZ46-0) and PP/EPDM/ZDMA TPV (TZ46-2). Fig. 3a shows a typical cryogenically fractured surface without any plastic deformation signs for PP/EPDM blend, implying the poor compatibility between the two phases. As for the TPVs (Figs. 3b and c), the cryogenically fractured surface becomes rough, indicating improved compatibility between PP and the rubber phase. For TZ46-0 (Fig. 3b), a large number of random fringes formed during the cryogenic fracture process and a relative flat surface with light plastic deformation of the ligaments are observed. While, as shown in Fig. 3c, there are numerous fringes on the cryogenically fractured surface of PP/EPDM/ZDMA TPV, which means that the material undergoes a lighter brittle failure process compared with PP/EPDM TPV. This implies a good *in situ* reactive compatibilization effect of ZDMA on the TPVs. In order to further observe the phase morphology, we used boiling cyclohexane and boiling xylene to etch the cryogenically fractured surface of the simple lends and TPVs, respectively. Fig. 4a shows that PP and EPDM formed a co-continuous phase in the PP/EPDM blend in which the large holes represent EPDM phase. However, for the TPVs (TZ46-0, TZ46-2 and TZ46-3), numerous particles are observed. Since PP and non-crosslinked EPDM phases were removed by boiling xylene, the particles left shown in Figs. 4b, c and d are crosslinked EPDM phase. This phenomenon indicates that dynamic vulcanization changes the phase structure from co-continuous phase into “sea-island” structure (PP as continuous phase and EPDM as dispersed phase). Moreover, it can be clearly seen that incorporation of ZDMA reduces the size of rubber particles, and the increase of ZDMA concentration further reduces the particle size (Figs. 4c and d).

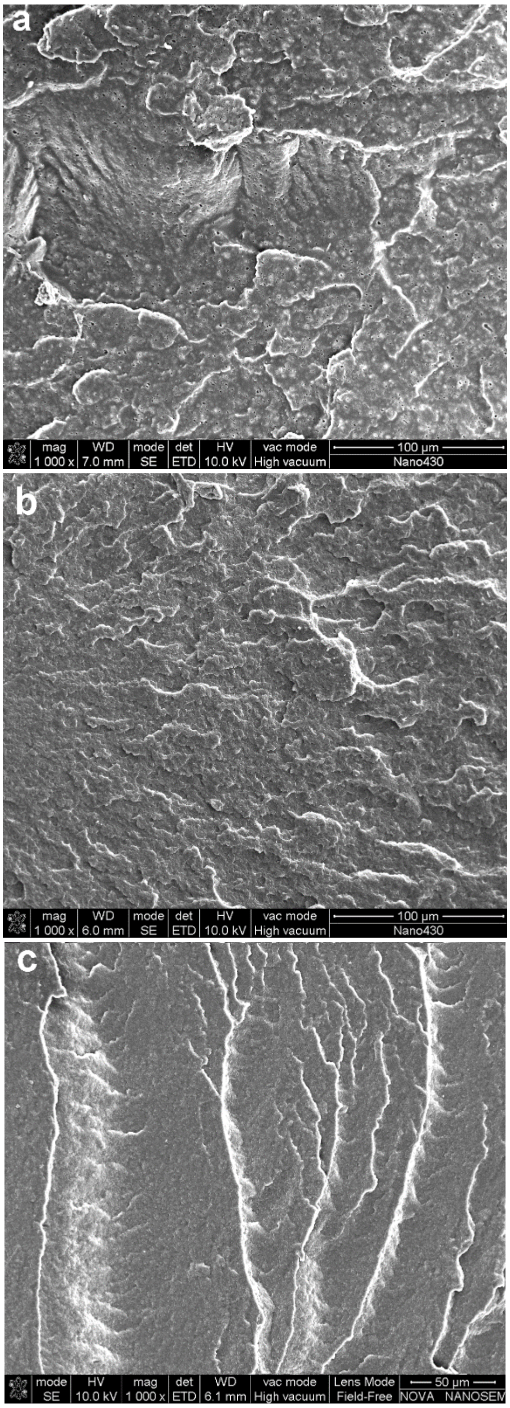


Fig. 3. SEM image for the cryogenically fractured surface of the TPVs: (a) B46; (b) TZ46-0; (c) TZ46-2.

It has been reported that the dispersion of EPDM during melt mixing with PP mainly depends on the viscosity difference between PP and EPDM [22]. When a curing agent is added, the viscosity of the rubber phase raises abruptly as dynamic vulcanization, the viscosity difference between EPDM and PP increased. At this time, the effect of shear on the morphology of the blends may be dominant and the intense shear results in the dispersion of EPDM particles.

Afterwards, the viscosity difference between EPDM and PP plays the critical role, leading to the EPDM particles being immobilized by crosslinking and breaking down into small droplets under the applied shear field [23]. Since the ZDMA largely promoted the crosslinking process and the crosslink density of EPDM phase in the presence of peroxide free radicals, this leads to the immobilization of the rubber particles and therefore breaking down into smaller sizes. In addition, the size reduction also enlarges the contact area which may be beneficialfor the interface interaction between PP and EPDM phases.

Crystallization behavior

Fig. 5 shows the DSC curves of all TPVs during non-isothermal crystallization. It can be seen that all PP/EPDM/ZDMA TPVs show a higher crystallization peak temperature (Tc) than PP/EPDM TPV, and Tc increases with increasing ZDMA concentrations. The effects of dynamic vulcanization and ZDMA concentration on Tc, onset crystallization temperature (To) and crystallinity (Xc) of the TPVs are summarized in Table 2. PP/EPDM/ZDMA TPVs show higher To and Tc values than those of PP/EPDM TPV, which indicates that the addition of ZDMA promotes the nucleation process of PP, leading to an increased crystallization rate and improved crystallization temperature. For PP/EPDM/ZDMA TPVs it can be seen that both To and Tc increase with increasing ZDMA concentrations. However, higher ZDMA concentration seems to have a negative effect on Xc of the PP phase. Note that To and Tc of TZ46-1 and TZ46-3 increase from 118.4°C to 121.3°C and 110.3°C to 112.5°C, respectively, while Xc decreases from 48.10% of TZ46-1 to 45.31% of TZ46-3. Here, the possible explanation of this crystallization behaviour is given below: The formation of polymerized ZDMA (PZDMA) in peroxide free radicals has been verified, it may have formed homopolymer (h-PZDMA particles) or grafted onto polymer chains to form graft products (g-PZDMA) [19].

Therefore, the h-PZDMA particles may serve as nucleating agents for the crystallization of PP phase, thus promoting the crystallization process of the PP component. On the other hand, *in situ* compatibilization of ZDMA occurs at the interfacial layer between PP and EPDM through the reaction

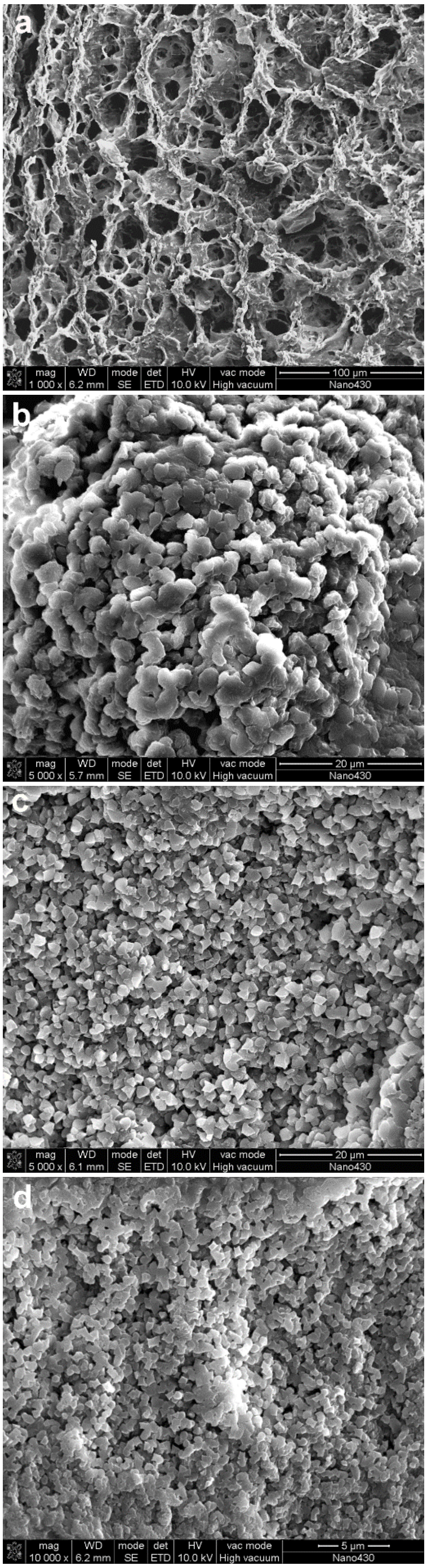


Fig. 4. SEM image for the etched cryogenically fractured surface: (a) B46 (cyclohexane-etched); (b) TZ46-0 (xylene-etched); (c) TZ46-2 (xylene-etched); (d) TZ46-3 (xylene-etched).

between the double bonds of ZDMA and the free radicals generated in both EPDM and PP chains, restricting the rearrangement of PP molecular segments during the crystallization process [17]. This leads to the decreased Xc at a higher ZDMA concentration.

Stress-strain behavior of PP/EPDM/ZDMA TPVs

Mechanical properties

To better understand the influence of ZDMA on the peroxide cured PP/EPDM TPVs, the mechanical properties for all specimens are summarized in Table 3. Representative stress-strain curves of the TPVs are shown in Fig. 6. The PP/EPDM/ZDMA TPVs display good combination of stress and strain properties: a rapid increase in tensile stress can be observed at the initial tensile and then the stress increases gradually with the larger extension of strain. All stress-strain curves show the representative elastomer character of soft and tough. Compared with the stress-strain curves of PP/EPDM blend and PP/EPDM TPV, improved tensile strength and elongation at break can be observed, due to the crosslinking of the rubber phase. The incorporation of ZDMA further improves tensile strength and elongation at break. For example, TZ46-1 exhibits tensile strength and elongation at break of about 11.8MPa and 421%, which is much higher than 7.7MPa and 250% of TZ46-0, indicating the strong reinforcing effect of ZDMA [24]. The improved mechanical properties indicate the good interaction between the ZDMA reinforced EPDM phase and the PP phase. Meanwhile, the higher crosslink density of EPDM also contributes to the improved mechanical properties.

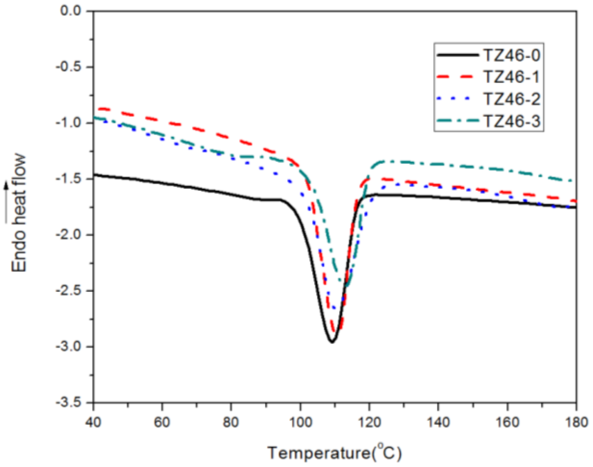


Fig. 5. DSC thermograms of the TPVs during non-isothermal crystallization

Table 2. Crystallization parameters for all specimens.

Sample	ΔH$_c$ (J/g)	X$_c$ (%)	T$_c$ (°C)	T$_o$ (°C)
B46	40.86	48.88	108.2	114.9
TZ46-0	36.26	43.63	109.5	116.6
TZ46-1	37.72	48.10	110.3	118.4
TZ46-2	35.06	47.22	110.9	119.2
TZ46-3	31.94	45.31	112.5	121.3

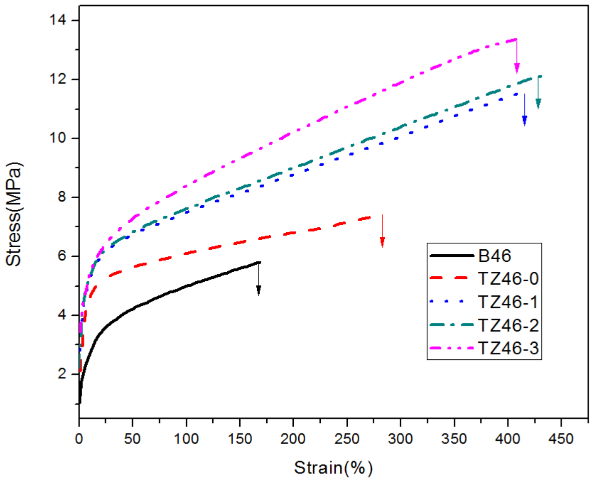


Fig. 6. Stress-strain behavior of all specimens.

Obviously, dynamic vulcanization improved the mechanical properties of the PP/EPDM blend. Incorporation of ZDMA largely improved the mechanical properties of the PP/EPDM TPVs. Especially, tensile strength and tear strength increased from 7.7MPa and 46.9kN/m to 11.8MPa and 72.3kN/m, respectively, with only 6wt% ZDMA added to the composites. This is attributed to the improved compatibility between EPDM and PP phases and the reinforced EPDM phase by ZDMA mentioned above.

In addition, ZDMA has caused a reduction of EPDM particles, as shown in the SEM images (Fig. 4), which may also contribute to the improved mechanical properties. Further increase in ZDMA concentration leads to an increased tensile strength, tear strength and tensile set at 100% elongation. The elongation at break reached maximum for ZDMA concentration of about 12wt%. The improved mechanical properties indicate that ZDMA is not only an effective *in situ* reactive compatibilizer for peroxide curing PP/EPDM TPVs, but also a strong reinforcing filler.

The effect of DCP concentration on the mechanical properties of PP/EPDM/ZDMA TPV (40/60, ZDMA concentration is 20wt% of EPDM) is also studied and shown in Fig. 7. It is clearly seen that both tensile strength and elongation at break increase with the increase of DCP concentration, which may be caused by the improved crosslink density of the rubber phase. However, further

Table 3. Mechanical properties of all specimens

Samples	B46	TZ46-0	TZ46-1	TZ46-2	TZ46-3
Tensile strength (MPa)	5.8	7.7	11.8	12.4	13.4
tensile set at 100% elongation (MPa)	5.0	6.0	7.5	7.9	8.3
Tear strength (kN/m)	42.0	46.9	72.3	77.1	86.2
Elongation at break (%)	168	250	421	439	410

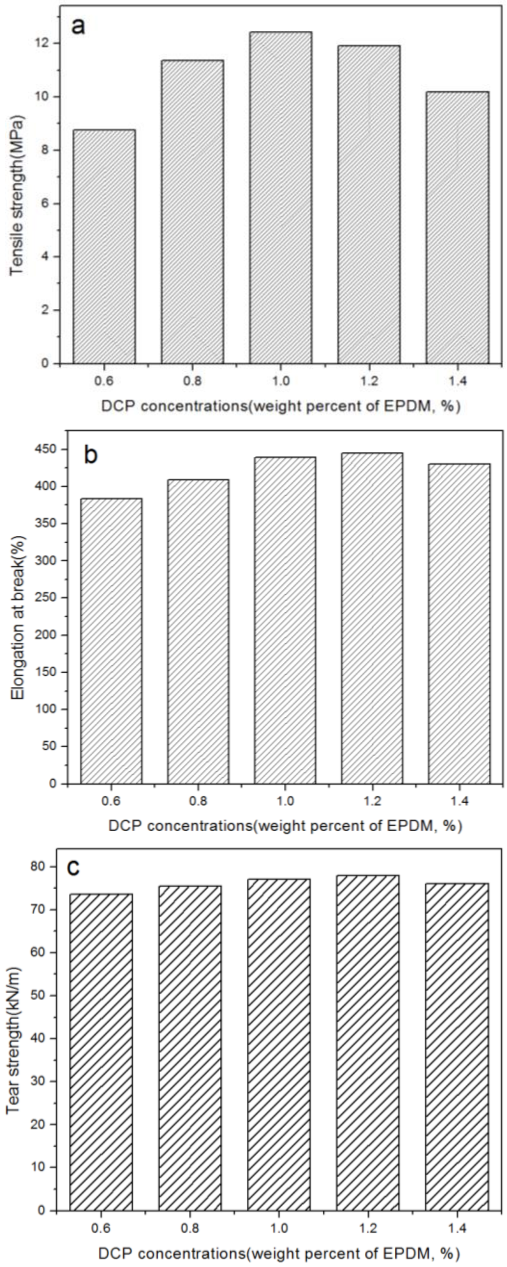


Fig. 7. Effect of DCP concentration on the mechanical properties of PP/EPDM/ZDMA TPV (40/60, ZDMA concentration is 20wt% of EPDM).

strength, which reaches a maximum value when DCP concentration is 1% of EPDM. This is due to the fact that high DCP concentration induced more serious decomposition of PP component. Elongation at break (Fig. 7b) shows the same tendency. Fig. 7c shows that DCP concentration has little influence on the tear strength. From Fig. 7 it can be concluded that PP/EPDM TPV reaches better overall performance when the DCP concentration is 1% of EPDM

CONCLUSION

This work shows that ZDMA can function as an effective compatibilizer and, simultaneously, as a very strong reinforcing agent for PP/EPDM TPVs. The reinforced TPVs based on a PP/EPDM ratio of 40/60 displayed better overall performance when DCP concentration is 1 wt% of the EPDM component. The mechanical properties increased with increasing ZDMA concentrations. Particularly, the tensile strength and elongation at break increased from 7.7MPa and 250% to 13.4MPa and 410%, respectively, with about 18%wt ZDMA. Incorporation of ZDMA resulted in size reduction of the crosslinked EPDM particles and improved compatibility between the PP and EPDM phase, which contributed to the enhanced mechanical properties. In addition, incorporation of ZDMA promoted the crystallization process of PP but had a negative effect on its crystallinity.

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ЕФЕКТ НА ЦИНКОВИЯ ДИМЕТАКРИЛАТ ВЪРХУ СЪВМЕСТИМОСТТА И ЯКОСТТА НА ТЕРМОПЛАСТИЧНИ ПОЛИМЕРИ ОТ ПОЛИПРОПИЛЕН И ЕТИЛЕН-ПРОПИЛЕН ДИЕН, ПОЛУЧЕНИ ЧРЕЗ ПЕРОКСИД-ИНИЦИИРАНА ВУЛКАНИЗАЦИЯ

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(Резюме)

Термопластичните вулканизати (TPVs), основани на полипропилен (PP)/етилен-пропилен-диен (EPDM) с тегловно сьотношение 40/60 са обработени in situ с цинков диметакрилат (ZDMA) чрез динамична вулканизация, индуцирана от пероксид. Изследвани са ефектите на динамичната вулканизация и ZDMA върху съвместимостта между фазите на PP и EPDM и механичните свойства на термопластичните вулканизати PP/EPDM. Включването на ZDMA подобрява вискозитета на стопилката на резултантните TPV. Динамичният механичен анализ (DMA) показва, че нарастването на конценмтрацията на ZDMA подобрява съвместимостта между фазите на EPDM и PP. Сканиращата електрон-микроскопия показва, че включването на ZDMA намалява размера на напречно-свързаните гумени частици. Диференциалната сканираща калориметрия е използвана за изследване на кристалинните отнасяния. Резултатите показват, че добавянето на ZDMA промотира зародишообразуването на PP, но по-високите концентрации показват отрицателен ефект върху кристалинността на PP-компонентата. Освен това е намерено, че механичните свойства на TPVs значително се подобряват от ZDMA. Добавянето на около 18 % (об.) ZDMA в термопластичните вулканизати от PP/EPDM води до повишаване на якостта спрямо усукване и удължаване на скъсване съответно от 7.7MPa и 250% до 13.4MPa и 410%, съответно.