

Structure and Properties of Polyamide 6 Blends with Low-Density Polyethylene Grafted by Itaconic Acid and with Neutralized Carboxyl Groups

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ABSTRACT: A comparative study of the structure and properties of two-phase blends of polyamide 6 (PA6) and low-density polyethylene (LDPE) modified in the course of reactive extrusion, by grafting of itaconic acid (IA) without neutralization of carboxyl groups (LDPE-g-IA) and with neutralized carboxyl groups (LDPE-g-IA^{-M+}) was carried out. It was shown that 30 wt % of LDPE-g-IA^{-M+} introduced to PA6 resulted in blends of higher Charpy impact strength compared with that of PA6/LDPE-g-IA blends. The maximum increase was achieved when Mg(OH)₂ was used as a neutralizing agent. The blend morphology has a two-phase structure with blurred interphases because of increased adhesion between the phases. The neutralization of

carboxyl groups in grafted IA did not lead to two-phase morphology of blends, which had a negative influence on the mechanical properties. It is believed that the differences in the impact strength were caused by the influence of the added neutralizing agents on the structure of interphases, which depends on both the interfaces adhesion and structural effects resulting from the nucleating behavior of the neutralizing agent. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 1702–1708, 2004

Key words: polyamide 6; low-density polyethylene (LDPE); graft copolymers; reactive extrusion; morphology

INTRODUCTION

In recent decades, there has been a rapid increase in the variety of composite materials produced by reactive blending of commercial polymers. The reactive blending is a complex physicochemical process that takes place in polymer melts under the action of thermal fields and shearing stress fields with participation of functional groups of the chains, as well as certain modifiers, mostly compatibilizers, introduced into the blend.^{1–6} Most polymers are immiscible. Therefore, modifying their morphology through compatibilization is needed to create conditions to produce high-performance engineering plastics.

The nonpolar nature of most polyolefins (POs) hampers their direct blending with polar polymers. In this connection, POs are subjected to grafting of monomers containing polar functional groups,³ or by adding certain compatibilizers, which most often are modified polyolefins or copolymers.^{2,5,6} The introduction of a grafted PO or a compatibilizer, on their basis, in a blend of incompatible polymers is accompanied by stronger interfacial adhesion, decreased surface ten-

sion between the phases, and, as a result, better compatibility of components, easier filling of molds at processing, and higher mechanical properties.

In the course of mixing, reactive polymers undergo general reactions common in low molecular weight materials.² It is usually believed that the reactivity of a functional group is independent of the size of the molecule to which it belongs, although steric barriers caused by chain structure of molecules, as a rule, reduce the reactivity to some degree.^{2,5} Therefore, to ensure effective compatibilization of the blend, grafted PO must contain particular functional groups, which could provide for high rate and selectivity of interfacial reactions over the given processing conditions under which a blend is prepared. It is the key problem of selecting a properly grafted PO for a particular blend.

In recent years, a number of investigations have been made in the field of chemical engineering and structural analysis of low-density polyethylene (LDPE), modified by grafting of itaconic acid (LDPE-g-IA) in the course of reactive extrusion^{7–11} as well as with carboxyl groups, which were neutralized by oxides or hydroxides of metals (LDPE-g-IA^{-M+}). Neutralization of carboxyl groups in the grafted polyethylene gives LDPE-g-IA^{-M+} with higher resistance to thermal oxidation compared with that of LDPE-g-IA (Table I).

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TABLE I
Composition and Properties of Grafted LDPE^a

Composition	Test material (concentration of NA and DCP, wt %)	α (%)	MFI (g/10 min)	D' (rel unit ¹²)	T_o (°C)
1	LDPE	—	16.2	—	195
2	LDPE-g-IA (0.15% DCP)	65.0	15.5	—	182
3	LDPE-g-IA (0.3% DCP)	87.1	4.7	—	180
4	LDPE-g-IA ⁻ Mg ⁺ (70% MgO, 0.15 DCP)	76.3	14.5	0.13	198
5	LDPE-g-IA ⁻ Mg ⁺ (150% MgO, 0.15 DCP)	79.0	13.7	0.19	192
6	LDPE-g-IA ⁻ Zn ⁺ (70% ZnO, 0.15 DCP)	79.4	11.5	0.39	200
7	LDPE-g-IA ⁻ Zn ⁺ (150% ZnO, 0.15 DCP)	83.5	9.6	0.51	200
8	LDPE-g-IA ⁻ Mg ⁺ (70% Mg(OH) ₂ , 0.15 DCP)	78.2	13.1	0.12	195
9	LDPE-g-IA ⁻ Mg ⁺ (150% Mg(OH) ₂ , 0.15 DCP)	82.0	11.3	0.34	198
10	LDPE-g-IA ⁻ Zn ⁺ (70% Zn(OH) ₂ , 0.15 DCP)	78.9	13.0	0.40	190
11	LDPE-g-IA ⁻ Zn ⁺ (150% Zn(OH) ₂ , 0.15 DCP)	81.7	9.1	0.96	187
12	LDPE-g-IA ⁻ Mg ⁺ (70% MgO, 0.3 DCP)	90.6	3.2	0.08	—
13	LDPE-g-IA ⁻ Mg ⁺ (150% MgO, 0.3 DCP)	94.8	4.4	0.10	—
14	LDPE-g-IA ⁻ Zn ⁺ (70% ZnO, 0.3 DCP)	95.6	4.6	0.28	—
15	LDPE-g-IA ⁻ Zn ⁺ (150% ZnO, 0.3 DCP)	96.1	3.0	0.38	—
16	LDPE-g-IA ⁻ Mg ⁺ (70% Mg(OH) ₂ , 0.3 DCP)	94.4	3.2	0.08	—
17	LDPE-g-IA ⁻ Mg ⁺ (150% Mg(OH) ₂ , 0.3 DCP)	95.9	2.5	0.20	—
18	LDPE-g-IA ⁻ Zn ⁺ (70% Zn(OH) ₂ , 0.3 DCP)	93.2	4.1	0.36	—
19	LDPE-g-IA ⁻ Zn ⁺ (150% Zn(OH) ₂ , 0.3 DCP)	95.2	3.8	0.84	—

^a α is efficiency of grafting (ratio of grafted portion of IA to its total amount added to LDPE); MFI is the melt flow index; D' is relative optical density of absorption maximum in the carboxylate region; T_o is the temperature of onset of oxidation.

In view of the fact that polyethylene grafted with oxygen-containing monomers possesses a satisfactory compatibility toward aliphatic polyamides,^{2,4,13,14} improving the processability and impact strength of the blends,^{4-6,15,16} it is important to study the details of their structure and properties. It is of particular interest to study blends with polyethylene grafted with IA that contains free and neutralized carboxyl groups.

EXPERIMENTAL

Materials

The experiments were conducted using as the basic components of the blend polyamide 6 (PA6; density 1.14 g/cm³, intrinsic viscosity 3.41 dp/g, melting point 220°C), supplied by Khimvolokno (Grodno, Belarus); and low-density polyethylene (LDPE; density 0.92 g/cm³, melting point 105°C), supplied by Polimir Co. (Novopolotsk, Belarus). LDPE-g-IA and LDPE-g-IA⁻M⁺ were prepared following procedures described elsewhere.¹² The neutralizing agents (NA) were the same as those in a previous work,¹² that is, chemically pure oxides and hydroxides of zinc and magnesium as powder having average particle sizes for: ZnO, 0.6 μ m; MgO, 2.0 μ m; Zn(OH)₂, 0.7 μ m; and Mg(OH)₂, 1.6 μ m. The carboxyl groups in the grafted IA were neutralized following a one-stage procedure combining the processes of grafting and neutralization. To do this, LDPE granules covered with peroxide initiator (e.g., dicumyl peroxide) and IA powder were treated with NA powder in an extruder reactor and grafted at melting and shearing. The neutralizing

agents used were arranged as follows in their decreasing neutralizing capacity toward IA: Zn(OH)₂ > ZnO > Mg(OH)₂ > MgO (Table I).

The NA concentration was calculated proceeding from the stoichiometry of neutralizing reactions between IA and the oxides and hydroxides used. NA was added either in somewhat deficient quantities to obtain partially neutralized IA (up to 70%) or in somewhat excessive quantities to obtain completely neutralized NA. In the latter instance, NA constituted 150% of the calculated value required to totally neutralize the carboxyl groups. Because the properties of polymer blends greatly depend on the melt viscosity of the blend components when grafted LDPE is prepared, the concentrations of peroxide initiator were as low as 0.15 and 0.3 wt %. The viscosity of the molten LDPE-g-IA differed several times from that of LDPE-g-IA⁻M⁺ (Table I). The concentration of IA added to the reaction blend for grafting was 1 wt %. Table I lists effective values of grafting for the LDPE-g-IA and LDPE-g-IA⁻M⁺ used in the experiments.

Preparation of blends

Blends of PA6/grafted polyethylene were prepared by melt mixing of granules in a single-screw plasticating extruder (screw diameter 45 mm; L/D = 20). The melt temperature in the die was 240°C. The extruded material was water-cooled and granulated. The contents of the grafted LDPE in blends were 15 or 30 wt %. Before mixing, PA6 was dried in an oven at 100°C for 14 h.

TABLE II
Properties of PA6-based Blends with Grafted LDPE^a

Composition	Material (wt %)	σ_T (MPa)	ε_P (%)	Charpy impact strength (kJ/m ²)			MFI (g/10 min)
				S_n	R_n	S_n (-40°C)	
1	PA6	60	240	7	4	3	8.2
2	PA6/LDPE-g-IA—15	52	210	22	18	—	2.5
3	PA6/LDPE-g-IA—30	42	255	28	43	18	0.8
4	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—15	64	249	17	22	7.3	3.5
5	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—15	57	240	17	20	6.6	4.1
6	PA6/[LDPE-g-IA ⁻ M ⁺ (70% Mg(OH) ₂)]—15	55	167	17	22	7.7	3.1
7	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—15	58	240	21	24	7.2	4.1
8	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—30	43	148	23	26	9.8	2.6
9	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—30	48	217	30	39	15.1	1.6
10	PA6/[LDPE-g-IA ⁻ M ⁺ (70% Mg(OH) ₂)]—30	49	218	34	57	15.0	2.3
11	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—30	46	214	43	52	13.8	1.6

^a σ_T and ε_P are the yield point in elongation and relative elongation at break, respectively; S_n , R_n , and S_n (-40°C) stand for Charpy impact strength measured on specimens sharp-notched and rectangular-notched at 23°C and sharp-notched at -40°C, respectively.

Testing of specimens

The melt flow index (MFI) of the grafted LDPE melt was determined as described in the literature⁷⁻⁹ using an IIRT-AM instrument (Ekodatchik, Tula, Russia). For grafted LDPE it was measured at 190°C under 10-kg loads; for polymer blends it was determined at 250°C and 2.16 kg.

The structural transitions were analyzed by differential scanning calorimetry (DSM-3A, Institute for Biological Instruments, RAS, Russia). The rate of heating or cooling was 16°C/min; the specimen weighed 5 mg. Variations in the crystallinity of LDPE as a result of grafting were evaluated by the crystallinity index (ΔI_{cr}), which was determined as the ratio of an area below crystallinity peaks of the modified and neat polymers, or those for the blend components, taking into account their concentration in that blend. To eliminate the influence of thermal history of specimens on the structure, measurements were conducted on the specimens initially heated in the DSC cell up to 190°C in the case of grafted LDPE and to 250°C for the blends. The specimens were maintained at these temperatures for 1 min, cooled to 40°C, and analyzed.

The scanning electron microscopy (SEM) instrument JSM 50A (JEOL, Tokyo, Japan) was used to examine morphologies of the test materials. The accelerating voltage was 25 kV. The specimens were fractured after dipping in liquid nitrogen, and then the fractured surfaces were coated with a layer of gold about 20 nm thick. The method of thermal vacuum deposition was used. In addition, we examined topography of the surface of the specimens, which were fractured by impact loading at 23°C. The specimens were notched at a sharp angle.

The relaxation properties of polymer materials were tested on the prototype reversible torsion pendulum

device with increased sensitivity of measurements that provided an operating frequency of 1 cps.¹⁷ The test specimens were injection molded in the form of plates measuring 50 × 5 × 0.5 mm. The temperature was controlled and maintained within an accuracy of ±0.1°C.

The mechanical properties were measured at room temperature and at -40°C by impact testing following the standard procedure. For low-temperature measurements, a cryogenic chamber described in Pesetskii et al.¹⁷ was used.

RESULTS AND DISCUSSION

PA6/LDPE-g-IA⁻M⁺ blends (Table II) have a much lower melt viscosity than that of PA6/LDPE-g-IA. Because of this, the MFI of PA6/LDPE-g-IA⁻M⁺ blends exceeds 1.4 to 1.6 times the values for PA6/LDPE-g-IA blends with 15 wt % grafted polyethylene, and between 2 and 3.2 times with 30 wt % grafted polyethylene (Table II). An increase in MFI of the blends was not an obvious fact when a more viscous component (LDPE-g-IA⁻M⁺) was introduced into PA6 compared with the less viscous LDPE-g-IA.

This finding is not fully understood and shows that the logarithmic law of additivity (often applied to estimate the MFI of polymer blends by summing the viscosities of individual components, taking into account their concentrations)¹⁸ appears inapplicable to the blends under consideration. It is because, in the one-stage procedure of grafting and neutralization used in our work, MFI values are lower for LDPE-g-IA⁻M⁺ than those for LDPE-g-IA (Table I). It can be anticipated, therefore, that the higher MFI for PA6/LDPE-g-IA⁻M⁺ compared with that of PA6/LDPE-g-IA is a result of the influence of NA on the intensity

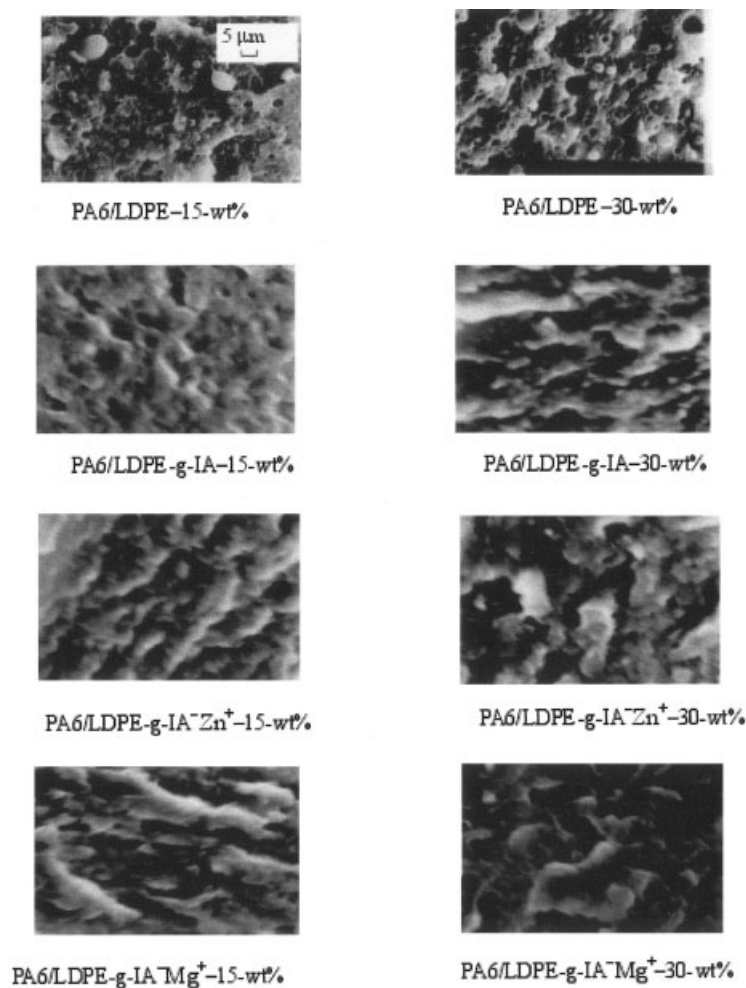


Figure 1 SEM micrographs of PA6 blended with neat and grafted LDPE.

of interactions between phases in molten polymer blends. It seems that NA binds carboxyl groups in the grafted IA and amide groups as well. This action decreases the energy of interphase interactions in the molten blends and is inevitably followed by a decrease in their viscosity.¹³ From these phenomena it may be concluded that the major result of neutralization of carboxyl groups in LDPE-g-IA⁻M⁺ is, most likely, that on blending with PA6, intermolecular associates cannot be formed at the expense of hydrogen bonds between molecules of the functionalized polyethylene and polyamide. In addition, under the influence of NA, hydrogen bonds between molecules of PA6 break down and the portion of associated macromolecules of polyamide decreases in the molten polymer blend.

From the above it may be concluded that incorporation of modified LDPE containing grafted IA with neutralized carboxyl groups to PA6 improves the processability of the blends and eases control of their processing.

The PA6 blends containing neat LDPE or grafted LDPE consist of two phases that substantially differ in

morphology (Fig. 1). PA6/LDPE blends show that the disperse phase of LDPE creates aggregates as smooth droplets (ellipsoids) of a size between 5 and 8 μm . Such a structure is typical for systems in which polymers are rather incompatible and show weak adhesion between phases.^{1,18} Despite the two-phase character of the blends, no distinct boundary between the phases was revealed. Blending of PA6 with grafted LDPE leads to a highly dispersed polyolefin phase ($<0.5 \mu\text{m}$), in which the particle areas were hardly discernible because of blurred interphases (Fig. 1). This can be explained by intensive interactions between the phases, supported by numerous published data. However, there were but small differences in the morphologies of blends prepared with PA6/LDPE-g-IA and PA6/LDPE-g-IA⁻M⁺. Consequently, the neutralization of carboxyl groups in the grafted IA does not lead to a morphology that could impair the mechanical properties of PA6-based blends.

All PA6/LDPE-g-IA⁻M⁺ blends showed (Table II) quite high tensile strength (σ_T). Their impact strength, however, greatly depends on the type and concentra-

TABLE III
Results of DSC Analysis of the Blends^a

Composition	Test material (wt %)	Polyamide component			Polyethylene component		
		T_m (°C)	T_{cr} (°C)	ΔI_{cr}	T_m (°C)	T_{cr} (°C)	ΔI_{cr}
1	PA6	218.7	164.5	1.0	—	—	—
2	LDPE	—	—	—	105.0	88.0	1.0
3	PA6/LDPE—15	217.0	180.3	1.2	102.2	86.8	0.24
4	PA6/LDPE—30	218.6	181.2	1.2	102.8	87.8	0.36
5	PA6/LDPE-g-IA—15	220.2	183.7	1.38	103.5	89.5	0.3
6	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—15	219.5	183.8	1.34	102.0	88.8	0.36
7	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—15	218.8	185.2	1.4	102.4	91.0	0.5
8	PA6/[LDPE-g-IA ⁻ M ⁺ (70% Mg(OH) ₂)]—15	218.8	183.1	1.32	102.4	89.5	0.3
9	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—15	218.8	185.2	1.3	102.4	91.0	0.35
10	PA6/[LDPE-g-IA ⁻ M ⁺ —30	218.8	182.5	1.38	102.4	88.8	0.38
11	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—30	218.3	182.2	1.2	103.0	88.1	0.33
12	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—30	218.1	183.4	1.3	102.4	88.8	0.42
13	PA6/[LDPE-g-IA ⁻ M ⁺ (70% Mg(OH) ₂)]—30	218.4	183.4	1.34	102.0	89.5	0.24
14	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—30	218.8	183.7	1.3	102.4	88.1	0.6

^a T_m and T_{cr} are melting and crystallization temperatures, respectively; ΔI_{cr} is the crystallinity index.

tion of NA used. We suppose that the observed increase in σ_T of the blends could result (Table III) from the somewhat higher crystallinity of both the polyamide and polyolefin phases.

The results of DSC analysis confirm the above conclusion that all blends studied have a distinct two-phase structure (Fig. 2). The melting points of the components only slightly depend on their ratios in a

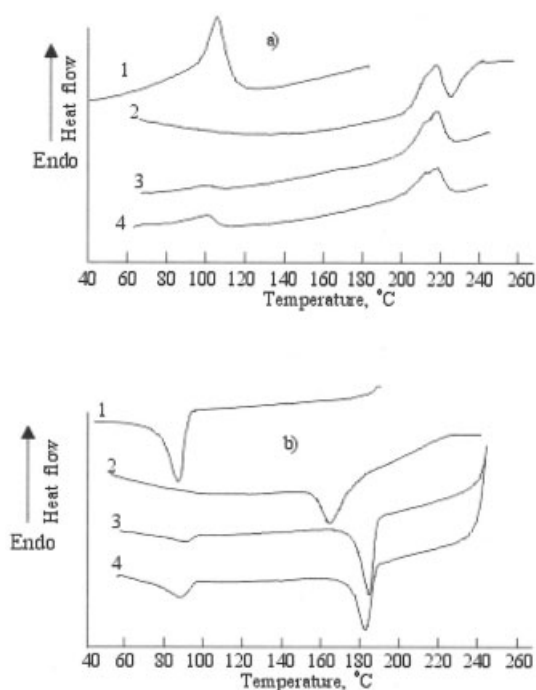


Figure 2 DSC curves for (1) LDPE, (2) PA6, and blends (3) PA6/[LDPE-g-IA⁻M⁺ (150% Mg(OH)₂)]—15 wt % and (4) PA6/[LDPE-g-IA⁻M⁺ (150% Mg(OH)₂)]—30 wt % at (a) melting and (b) crystallization.

blend. Therefore, crystallization of every component gives crystallites that are free of a foreign polymer phase. However, the crystallization temperature of components (especially of PA6) increases significantly (Table III) in the case of blends prepared with NA. Therefore, NA used to prepare LDPE-g-IA⁻M⁺ acts simultaneously as a nucleating agent for crystallization of the two components of the system. Because of this, a higher crystallinity was found for both components in the blends containing grafted polyethylene, unlike the blends of PA6 and neat polyethylene. This can be explained by stronger interfacial adhesion attributed to grafting.

The two-phase structure of the blends was also proved by relaxation spectrometry (Fig. 3, Table IV). The glass-transition temperature (T_g) of the PA6 amorphous phase in the blends did not vary (55–58°C), irrespective of the type of grafted LDPE or the type and concentration of NA. The β -transition temperature (T_β), however, being –60°C for neat PA6, shifts to

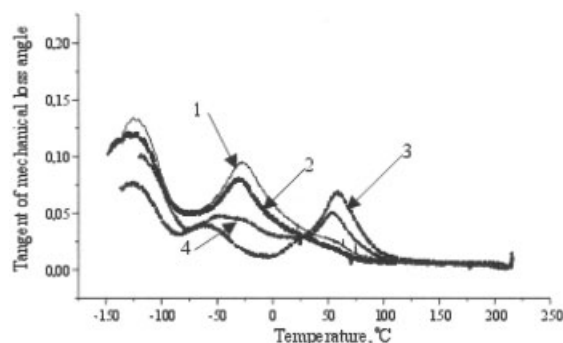


Figure 3 Temperature dependencies of the tangent of mechanical loss angle: (1) LDPE, (2) LDPE-g-IA⁻Mg⁺, (3) PA6, and (4) PA6/[LDPE-g-IA⁻M⁺ (150% Mg(OH)₂)]—30 wt %.

TABLE IV
Relaxation Transition Temperatures for Amorphous Phase of Neat PA6 and That in PA6/LDPE-g-IA⁻ and PA6/LDPE-g-IA⁻M⁺ Blends

Composition	Material (wt %)	β -transition (°C)	α -transition (°C)
1	PA6	-60	58
2	PA6/LDPE-g-IA—15	-55	58
3	PA6/LDPE-g-IA—30	-36.5	58
4	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—15	-52	55
5	PA6/[LDPE-g-IA ⁻ M ⁺ (70% ZnO)]—30	-40	56
6	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—15	-56	58
7	PA6/[LDPE-g-IA ⁻ M ⁺ (150% ZnO)]—30	-42	58.5
8	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—15	-55	55
9	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Mg(OH) ₂)]—30	-48	52
10	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Zn(OH) ₂)]—15	-62	55
11	PA6/[LDPE-g-IA ⁻ M ⁺ (150% Zn(OH) ₂)]—30	-45	58

the high-temperature region for blends with grafted LDPE. The β -relaxation peak for PA6 is combined with the glass-transition peak for grafted PE at $T_g = -28^\circ\text{C}$. The shift extent of $T_{\beta\text{PA6}}$ depends on the concentration of grafted LDPE in the blend and the type of NA used. For 15 wt % polyolefin, this shift value reaches 4–8°C and for 30 wt % it reaches 12–24°C. The shift is more significant for blends prepared without NA (Table IV). This can be explained by interactions between phases in the blends that involve segments belonging to polyolefin structure as well as structural units of smaller size (responsible for β -relaxation) in PA6.

The structural and morphological features of blends influence their behavior under impact stresses (Fig. 4, Table II). It is worth mentioning that 30 wt % of grafted LDPE ensures the impact strength typical of numerous commercial impact-resistant materials. The PA6/LDPE-g-IA⁻M⁺ blends prepared with Mg(OH)₂ as a neutral-

izing agent appear most highly impact resistant (Table II). Under impact testing the fractured surface of specimens of these blends appeared whitish because of multiple crazing and numerous tiny pores resulting from crazing on the surface (Fig. 4). Because the impact strength greatly depends on microheterogeneity,^{13,17} it is most likely that the variations in the impact strength of blends prepared with different types of grafted LDPE (Table II) could be caused by NA that influences the structure of interphases, which depends on both the interfacial adhesion and structural features resulting from the nucleating behavior of NA.

CONCLUSIONS

LDPE-g-IA⁻M⁺ used in place of LDPE-g-IA in blends with PA6 could not impair the structural morphology of the blends. On the other hand, compounding PA6

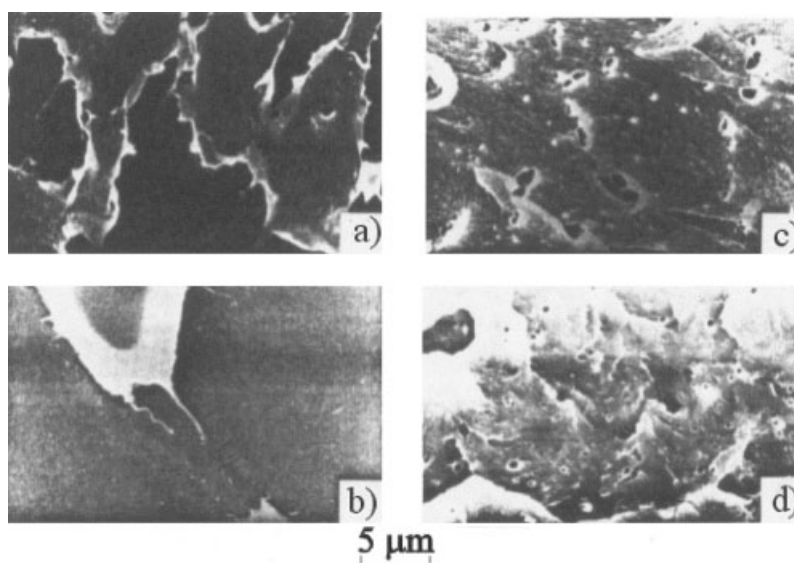


Figure 4 Surface topography of impact-fractured blend compositions at 23°C: (a) PA6/LDPE, (b) PA6, (c) PA6/LDPE-g-IA—30 wt %, (d) PA6/[LDPE-g-IA⁻M⁺ (150% Mg(OH)₂)]—30 wt %.

attributed to higher resistance to oxidation of LDPE-g-IA^{-M+} (Table I) in the melt was accompanied by a lower degree of degradation compared with that of LDPE-g-IA. Simultaneously, the introduction of LDPE-g-IA^{-M+} into PA6 gives blends with higher MFI, which is favorable for their processing.

LDPE containing neutralized carboxyl groups is an efficient modifier of impact strength for PA6-based blends. Because the impact strength greatly depends on the degree of structural heterogeneity of a material, the strength can be increased by introducing LDPE-g-IA^{-M+}, which promotes generation of a microheterogeneous structure of interphases attributed to stronger interfacial adhesion and nucleation of crystallites caused by metal oxides and hydroxides in polymer components. It is assumed that this microheterogeneous structure can easily dissipate the impact energy.

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