Use of Zinc-Neutralized Ethylene/Methacrylic Acid Copolymer Ionomers as Blend Compatibilizers for Nylon 6 and Low-Density Polyethylene

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ABSTRACT: The effect of the composition on the morphologies and properties of uncompatibilized and compatibilized blends of nylon 6 and low-density polyethylene were studied over a wide range of weight fractions. The uncompatibilized blends had substantially reduced mechanical properties after mixing, and this was almost certainly due to poor interfacial adhesion between the two polymers. The addition of a zinc-neutralized poly(ethylene-*co*-methacrylic acid) ionomer (Surlyn® 9020) as a compatibilizer improved the mechanical properties in comparison with those of the material blended without the compatibilizer. The clearest evidence of this improvement came from dynamic mechanical studies; for selected blends with high polyethylene contents, the drop in the modulus corresponding to the transition of a solid to a melt occurred at higher temperatures with the added compatibilizer. This improvement in the properties was accompanied by a reduction in the dispersed-phase size due to the interaction between the ionic part of the ionomer and the amide groups of nylon 6, especially when nylon 6 was the dispersed phase of the blend. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 89: 620–629, 2003

Key words: ionomers; compatibilization; blends; polyethylene (PE); polyamides

INTRODUCTION

For the development of new polymeric materials for both scientific and commercial purposes, the blending of two or more different polymers is often used. In industry, the blending process is almost always carried out in the molten state. At equilibrium, the amorphous components of both polymers may exist as a single homogeneous phase. This means that the two polymers are miscible; that is, the two materials are compatible. In most cases, however, the amorphous components of the two polymers separate into distinct phases consisting primarily of the individual components. An incompatible blend can be transformed into a compatible blend by the addition of a compatibilizer, that is, a third compound that may or may not be a polymer. Even if a compatibilizer does not cause true miscibility, these types of materials typically substantially reduce domain sizes in blends and also significantly strengthen interfacial adhesion between the two phases.

This article presents our efforts to improve the miscibility of blends of low-density polyethylene (LDPE) and nylon 6 through the use of an ionomeric compatibilizer. Clearly, nylon 6 and LDPE are very different in terms of backbone structure and polarity, and as expected, blends of these two materials have been shown to be incompatible.[1–3] Interest in blends of these two materials is high, however, because polyamides have good barrier properties and strength while LDPE has good low-temperature toughness and low moisture absorption. In a study of the barrier properties, the incorporation of just a small amount of nylon (<20%) improved the barrier properties of LDPE.[4] As one might expect, this study also found that the performance of this blend as a barrier material depended in large part on the morphology of the system; in particular, the more continuous the nylon phase was, the better the barrier properties were. A study of the rheology of nylon 12/polyethylene (PE) blends showed that the rheology and, therefore, morphology depended on the relative fraction of the two components; at approximately equal fractions of the two components, the morphology was fibrillar, whereas a preponderance of one component or the other led to a dis-

TABLE I
Properties of Neat Resins

Resin	Tensile strength (MPa)	Tensile modulus (MPa)	% Elongation	T_m (°C)	% Crystallinity	Impact strength (J/m)	Hardness (shore D)
LDPE	10.7	112	95	107.6	34.7	Sample did not break	49
Ionomer	26.0	170	475	87	14.8	Sample did not break	53
Nylon	74.3	833	287	222.2	35.5	106	80

persed phase suspended in the continuous phase of the majority component.[5]

The use of compatibilizing agents to improve the miscibility of LDPE and nylon 6 has also been investigated. Miscibility has been improved by the grafting of maleic anhydride and diethyl maleic anhydride onto LDPE.[6–10] Random terpolymers of ethylene, acrylic ester, and maleic anhydride were used to reactively compatibilize nylon 12/LDPE blends.[11] Maleic anhydride attached to a block styrene–(ethylene-*co*-butylene)–styrene copolymer has also been used to compatibilize LDPE/nylon 6 blends.[2,12] The chemical reaction that occurs in all of these systems is a reaction between the maleic anhydride and the terminal amine groups of the polyamide, although a recent article showed that a reaction with amide nitrogens may be occurring as well under certain conditions.[10] Maleic anhydride does not make LDPE and nylon miscible; rather, the domain size is reduced, typically by an order of magnitude or more, and indirect measurements suggest interfacial adhesion is increased dramatically.

Carboxylic acid groups also have the possibility of reacting with terminal amine groups to form amide groups.[13] Such an explanation was advanced to explain the improvement in miscibility of nylon 6 with high-density polyethylene (HDPE)[3,14–16] when a Surlyn® 9020 compatibilizer was used. According to these articles, the compatibilizer was a terpolymer of PE, methacrylic acid, and isobutyl acrylate consisting of roughly 80% ethylene segments. The methacrylic acid component was 70% neutralized with zinc. In blends with HDPE, a maximum reduction in phase size was observed when only 0.5 wt % of the terpolymer was added to the blend at a dispersed-phase concentration of 10% or lower. A more significant reduction of the dispersed-phase size was observed when the minor phase was nylon. More in-depth studies were performed with different ratios of HDPE and nylon at a constant 5 wt % ionomer content. The size of the dispersed phase increased only slightly up to a concentration of approximately 40 wt % dispersed phase. Further, the range of weight concentrations in which the phases were cocontinuous decreased with the addition of the ionomer. These researchers also found a less dramatic dependence of phase size on the viscosity ratio of the blend with added ionomer. Finally, in

injection-molded parts with 75% nylon and 25% PE, a diminution of the thickness of the skin and weld-line regions occurred with interfacial modification, coupled with an order of magnitude reduction in the dispersed-phase size.

Compatibilization in the HDPE/nylon 6/Surlyn 9020 system has been focused on the interaction between the polyamide and ionomer. However, HDPE has been used, and one should not expect the ionomer to be very compatible with HDPE. Ionomers are synthesized in a process similar to that used to make LDPE, and so the two materials should be much more compatible than HDPE and the ionomer. The sodium-neutralized form of ethylene/methacrylic acid was somewhat successful in compatibilizing polyamide and LDPE;[17] however, the qualitative difference in the environment of the unneutralized acid groups in the sodium and zinc ionomers could lead to differences in the compatibilization behavior for the two species.[18] The purpose of this study was to explore the compatibilization of LDPE with nylon 6 with a zinc-neutralized ionomeric compatibilizer.

EXPERIMENTAL

An ethylene/methacrylic acid copolymer neutralized with zinc was graciously supplied by DuPont. This particular material contains an additional comonomer used to facilitate toughening with nylon and is sold under the trademark Surlyn® 9020. The LDPE was an injection-molding grade (LD1450J) from Thai Polyethylene Co., Ltd., graciously supplied by MC Industrial Chemical, Ltd. Finally, the nylon 6 employed in this study was also an injection-molding grade (1013B), supplied by UBE Nylon (Thailand). Table I shows the relevant properties for the pure resins used in this

TABLE II
Extruder Temperatures

Region	Temperature (°C)
I	75
II	200
III	215
IV	220
V	220
VI	230

TABLE III
Tensile Properties

	Nylon/LDPE ratio			
% Surlyn	0.8/0.2	0.6/0.4	0.4/0.6	0.2/0.8
Tensile strength				
0.0	32.65 ± 1.07	10.72 ± 0.80	11.23 ± 0.26	11.42 ± 0.38
0.1	33.27 ± 1.56	14.22 ± 0.97	12.97 ± 0.79	11.92 ± 0.73
0.5	32.92 ± 0.80	12.91 ± 0.44	12.36 ± 0.12	11.88 ± 0.72
1.0	37.00 ± 1.05	15.89 ± 1.02	13.27 ± 0.43	12.05 ± 0.36
2.5	38.06 ± 0.79	18.28 ± 0.82	13.04 ± 0.33	12.2 ± 0.44
5.0	40.02 ± 1.61	15.4 ± 0.39	14.54 ± 0.44	12.59 ± 0.38
10.0	36.17 ± 0.80	15.62 ± 0.98	13.96 ± 0.31	12.55 ± 0.76
15.0	36.69 ± 3.05	15.46 ± 0.87	14.03 ± 0.75	11.7 ± 0.06
35.0	25.44 ± 1.40	14.58 ± 0.27	13.26 ± 0.68	11.29 ± 0.74
Percent elongation				
0.0	82.44 ± 3.35	6.02 ± 0.59	8.11 ± 1.16	20.76 ± 3.00
0.1	65.82 ± 1.25	10.78 ± 0.71	10.02 ± 0.63	17.9 ± 1.70
0.5	54.79 ± 1.04	7.48 ± 1.50	6.67 ± 0.93	19.93 ± 2.28
1.0	96.28 ± 8.87	7.21 ± 1.15	9.45 ± 0.53	27.32 ± 1.86
2.5	118.06 ± 7.94	10.45 ± 2.38	11.71 ± 1.03	32.04 ± 2.05
5.0	145.6 ± 3.04	10.54 ± 1.33	14.69 ± 2.03	33.11 ± 2.43
10.0	155.67 ± 1.97	11.8 ± 0.78	21.24 ± 2.47	35.64 ± 1.68
15.0	130.43 ± 2.40	19.24 ± 1.50	28.24 ± 4.45	39.36 ± 2.41
35.0	98.22 ± 1.51	38.24 ± 4.10	40.92 ± 6.68	52.39 ± 5.53
Young's modulus				
0.0	564.9 ± 19.1	548.1 ± 53.6	430.4 ± 20.5	393.3 ± 35.6
0.1	655 ± 19.1	657.5 ± 10.7	440.8 ± 21.8	431.7 ± 38.3
0.5	678.8 ± 56.4	695 ± 28.0	577.8 ± 40.3	436.3 ± 36.0
1.0	730.9 ± 40.1	673.1 ± 42.6	654.7 ± 63.4	401.6 ± 28.7
2.5	663.8 ± 74.7	585.8 ± 32.0	468.9 ± 27.3	328.8 ± 28.7
5.0	552.5 ± 33.2	563.2 ± 17.8	438.8 ± 18.3	379.2 ± 9.4
10.0	572.3 ± 28.5	550.5 ± 8.1	447.8 ± 21.8	404.1 ± 31.9
15.0	452.3 ± 22.4	418.8 ± 24.4	337.4 ± 18.3	326.9 ± 10.9
35.0	381.4 ± 6.5	328.9 ± 23.0	287.1 ± 25.6	289.6 ± 26.2

study. These properties were generated with the same processing conditions used for the blended materials.

Materials were dried in a hot-air oven at 60°C for 5 h before use for the removal of absorbed moisture. Polymer blends were prepared in a Model T-20 corotating twin-screw extruder (Dr. Collin) with a length/diameter ratio of 30:1 and with 25-mm-diameter screws. There were two kneading sections along the screw and a total of eight different sections altogether. At the operating speed of 35 rpm, the residence time of the resin in the extruder was approximately 1 min. The temperatures of each zone of the extruder are shown in Table II and were chosen to minimize the possibility of degradation on the basis of thermogravimetric analysis. The listed weight percentages are on a per nylon 6 + LDPE basis; that is, 5 wt % means that 5 g of the ionomer was mixed with 100 g of nylon 6 + LDPE. The melt was extruded through a single-strand die, solidified with cold water (35°C), and pelletized. Pellets were dried in a hot-air oven at 60°C for 2 days and kept in sealed plastic bags before compression molding so that moisture absorption would be minimized.

Samples for mechanical and physical property tests were prepared from compression-molded sheet with a V 50 H compression press (Wabash). The pellets were placed in a picture frame mold, and the mold was preheated at 240°C for 3 min. The mold was then compressed under a force of 10 tons for 3 min. The compression-molded sheet was cooled to 40°C at a cooling rate that was fit well by an exponential decay with a time constant of 3 min. Test specimens for each test were cut from the molded sheets with a die cutter.

An RSA II solids analyzer (Rheometric Scientific) was used to measure the storage and loss moduli as a function of temperature. The film and fiber fixture was used to mount the samples, and 3 K temperature steps were used. All experiments were performed with a 10-Hz frequency, a 0.1% strain, and a static force tracking dynamic force.

A DSC 7 (PerkinElmer) was used to measure the melting temperature (T_m) and the fractional crystallinity of the blended materials at a heating rate of 10°C/min. Crystallinities were calculated on a component basis; that is, a PE crystallinity percentage of 50% means that 50% of the PE in the given blend existed as PE crystallites. The tensile properties, impact strength, and hardness of the blends were determined from averages of 10 samples. A D1708 microtensile die was
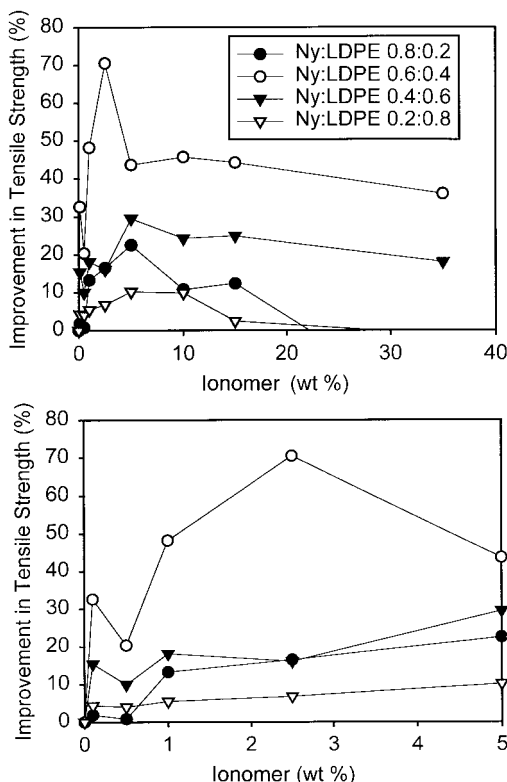
Figure 1 Fractional improvement in the tensile strength. The y axis is the tensile strength at weight fraction w with no compatibilizer divided by the tensile strength with no compatibilizer.

used to cut the samples for tensile testing, and an Instron universal testing machine was used to measure the tensile modulus, tensile strength, and elongation at break with a crosshead speed of 1.30 mm/min. The Izod impact strength was measured with a Zwick impact tester according to ASTM D 256 standard with a 2.7 J pendulum. A Shore D durometer was used to
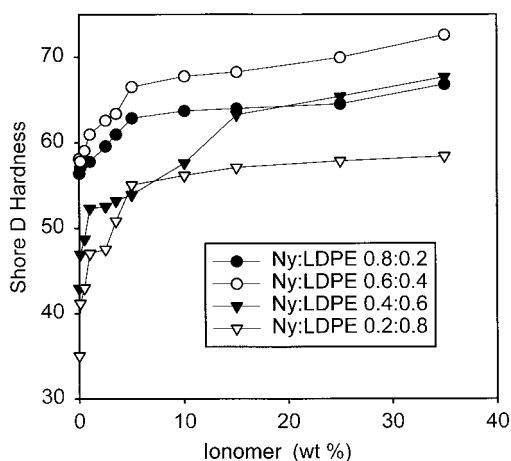
Figure 2 Hardness as a function of the compatibilizer content for different nylon/LDPE ratios.
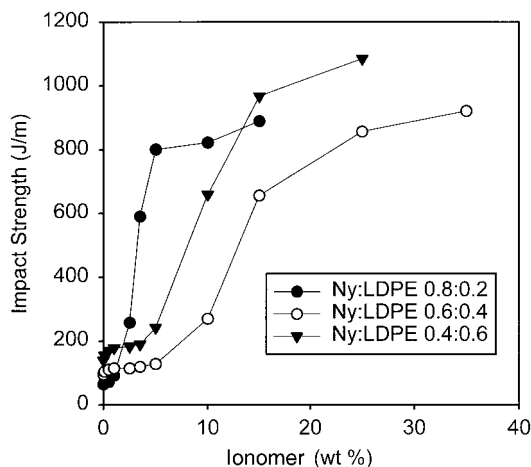

Figure 3 Impact strength as a function of the compatibilizer content for different nylon/LDPE ratios.

measure the hardness of the blends according to the ASTM D 2240 test procedure.

Scanning electron microscopy (SEM; JEOL 5200-2AE, MP152001) was used to characterize the microphases after liquid-nitrogen fracturing. The fracture surfaces of the specimens were then etched with decalin and formic acid to remove dispersed PE and polyamide, respectively. The specimens were coated with gold, and magnifications of 2000× and 3500× were used.

RESULTS AND DISCUSSION

Table III shows the improvement in the tensile strength and elongation percentage with the addition of just a small percentage of the ionomer in comparison with those of the blend manufactured with no compatibilizer. Figure 1 recasts the data in Table III, showing the fractional improvement at each ionomer weight percentage w, that is, [property(w) − property (w = 0)]/[property (w = 0)]. The mechanical properties either changed very little or even plateaued at compatibilizer contents above approximately 5. Although the improvement as a percentage of the blend without the compatibilizer was quite high, even in the best case the tensile strength was lower than a simple additive rule would predict. The behavior of the elongation was even worse: the elongation was lower than the smallest elongation for any of the pure materials, except for a few samples. Therefore, blending definitely reduced the ultimate mechanical properties. For many of the samples, the behavior became worse at high compatibilizer contents, and this could indicate some change in the phase behavior of the system.

The behavior of the hardness and impact strength, two mechanical properties with a characteristic timescale much shorter than the ultimate properties, was much more promising. As indicated in Figures 2 and

TABLE IV
Properties of PE Crystallites from DSC Experiments

| | Nylon/LDPE ratio | | | | | | | |
| | 0.8/0.2 | | 0.6/0.4 | | 0.4/0.6 | | 0.2/0.8 | |
% Surlyn	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity
0.0	104.8	51.3	106.7	51.1	105.3	51.6	104.7	46.2
0.1	104.8	51.6	104.8	37.1	105.2	28.5	105.2	35.1
0.5	105.2	46.8	104.7	40.9	105.0	35.3	105.2	38.6
1.0	105.7	47.3	105.7	49.7	104.8	47.7	104.8	42.7
2.5	104.8	60.7	105.0	49.1	104.5	37	104.7	47.6
5.0	104.2	22.4	105.2	30.5	105.3	33.9	105.2	38.0
10.0	104.8	23.2	104.0	31.3	103.8	35.3	105.2	31.6
15.0	105.0	26.2	104.0	36.3	106.3	36.2	105.0	32.8
35.0	104.5	23.7	104.8	43.4	105.6	50.3	105.0	43.2

Measurements for T_m and percentage crystallinity were duplicated five-times. Separate samples were cut for each duplicate measurement; however, these samples were all cut from the same compressed sample.

3, respectively, the hardness and impact strength showed substantial improvement with an increased amount of the compatibilizer. Furthermore, the values did not compare poorly with the values for the unblended homopolymers. The data for high nylon contents, in particular, are quite interesting: a very small percentage of the compatibilizer led to nylon/LDPE blends with very high impact strengths. At a nylon/LDPE ratio of 0.8/0.2 with 5% compatibilizer, the impact strength was many times larger than that for pure nylon, whereas the tensile strength was reduced by less than half versus the pure nylon. This latter value could presumably be increased if a lower weight fraction of LDPE was used with perhaps not much change in the improvement in the impact strength.

Tables IV and V show the properties of the LDPE and nylon 6 crystallites, respectively, as a function of the compatibilizer content. The addition of the compatibilizer had little to no effect on T_m of the PE crystallites, even though T_m of the crystallites in the bulk ionomer was much lower than T_m of LDPE. Although no drop was found in T_m, the fractional crystallinity of the PE component dropped dramatically as the weight percentage of the ionomer changed from

2.5 to 5, especially at high nylon contents. In particular, for the nylon/LDPE ratio of 0.8/0.2, the fractional crystallinity of the PE component became much closer to that of the pure ionomer, but T_m did not change substantially. The change in the fractional crystallinity with an increasing amount of the compatibilizer clearly indicated that the ionomer substantially influenced the crystallization behavior of PE. From these results, we cannot know if the ethylene segments from the ionomer were cocrystallizing with ethylene segments from LDPE; however, there was no indication of a separate ionomer exotherm in the differential scanning calorimetry (DSC) results. For the nylon, the only clear trend was that the nylon T_m dropped slightly with decreasing nylon content. Perhaps there was an increase in the nylon crystallinity at low ionomer contents, but the scatter in the data is so large that this conclusion seems tenuous at best.

In a previous article on binary nylon/ionomer blends, the ionomer was shown to substantially slow crystallization in nylon.[15] In this study, the kinetics were probed indirectly because the cooling rate after molding was the same for all materials. However, the cooling rate was quite slow in comparison with that of

TABLE V
Properties of Nylon Crystallites from DSC Experiments

| | Nylon/LDPE ratio | | | | | | | |
| | 0.8/0.2 | | 0.6/0.4 | | 0.4/0.6 | | 0.2/0.8 | |
% Surlyn	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity	T_m (°C)	% Crystallinity
0.0	221.3	32.9	221.3	24.8	220.0	28.4	220.0	31.7
0.1	221.7	40.0	221.2	38.2	220.2	35.4	220.1	27.6
0.5	221.5	38.6	221.0	28.6	220.3	26.0	219.7	29.0
1.0	221.8	38.9	221.3	37.1	220.3	34.4	219.7	28.3
2.5	221.3	28	220.7	31.9	220.7	29.7	220.5	26.0
5.0	221.3	34.5	221.8	35.5	221.3	35.6	219.5	33.8
10.0	221.3	33.0	221.2	34.1	220.0	41.6	220.5	42.4
15.0	221.7	34.1	220.7	35.6	219.8	33.7	219.5	35.7
35.0	221.2	33	220.7	32.8	221.2	28.5	221.2	27.5

Figure 4 DMA spectra of samples with a nylon/LDPE ratio of 0.8/0.2 at selected compatibilizer levels.
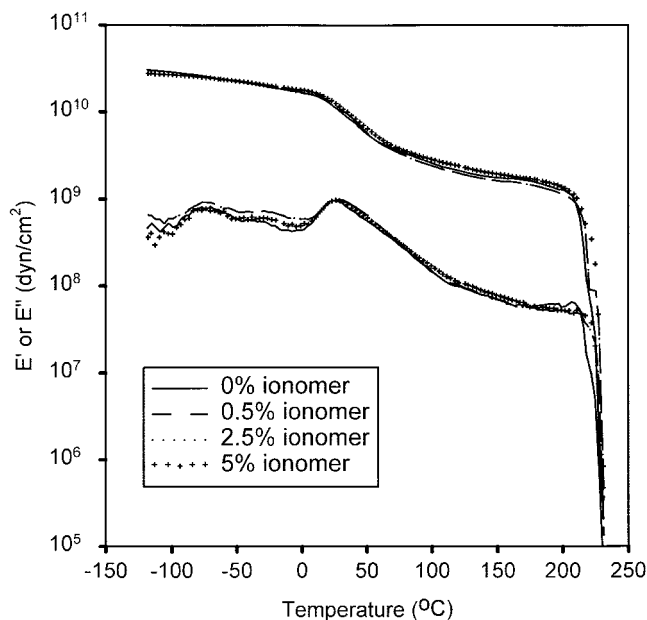

Figure 6 DMA spectra of samples with a nylon/LDPE ratio of 0.6/0.4 at selected compatibilizer levels.

a typical injection-molding application, for example, and this rather slow rate might mitigate any kinetic effects that could be present. Our study did show that the ionomer had a substantial effect on the crystallization behavior of the PE segments.

Dynamic mechanical analysis (DMA) results are shown in Figures 4–8. The small-strain rheological behaviors for the systems at the highest nylon content did not seem to change with the compatibilizer con-

tent; in fact, all of the results seemingly overlap one another at this nylon/LDPE ratio. However, as indicated in Table VI, the E' drop-off corresponding to the melting transition of nylon shifted to a higher temperature as the compatibilizer content changed from 2.5 to 5 wt % for nylon/LDPE at a 0.8/0.2 ratio. The increase in the drop-off modulus that occurred between compatibilizer contents of 2.5 and 5 wt % corresponds to the same weight fraction as the drop-off in the PE fractional crystallinity; this strongly suggests

Figure 5 DMA spectra of pure nylon (dashed lines) and a representative nylon/LDPE sample (0.8/0.2 with 5% ionomer; solid lines), which shows a higher storage modulus in the melting transition region. The inset illustrates this difference more clearly.
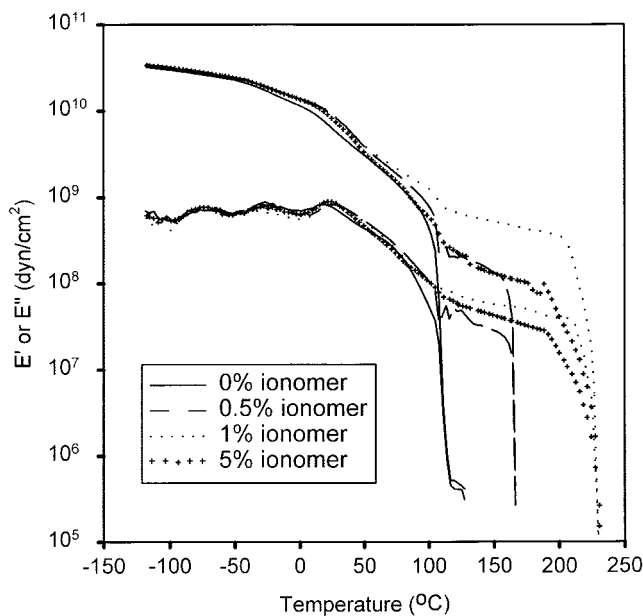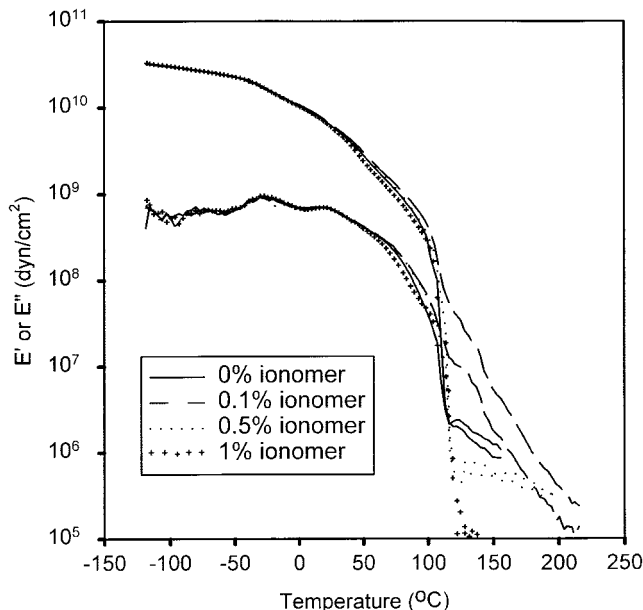

Figure 7 DMA spectra of samples with a nylon/LDPE ratio of 0.4/0.6 at selected compatibilizer levels.
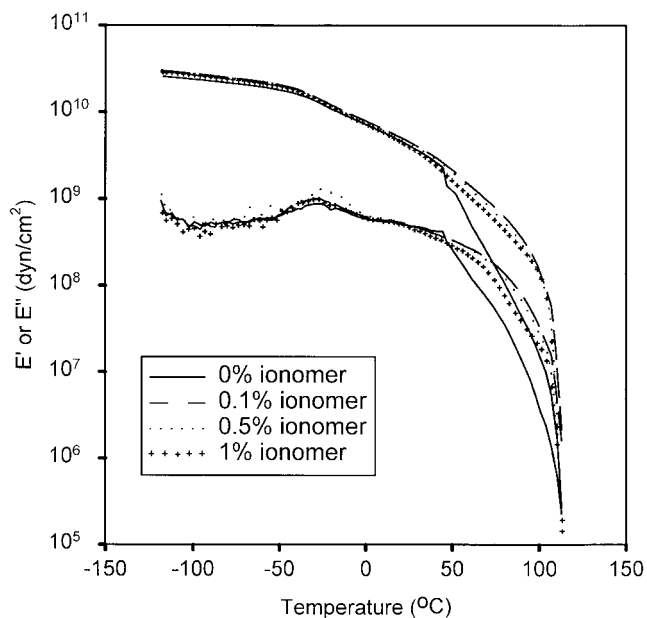
Figure 8 DMA spectra of samples with a nylon/LDPE ratio of 0.2/0.8 at selected compatibilizer levels.

TABLE VI
Temperature (°C) where $E' = 10^8$ dyn/cm^2

	Nylon/LDPE ratio			
% Surlyn	0.8/0.2	0.6/0.4	0.4/0.6[a]	0.2/0.8
0.0	219	106	110	80
0.1	219	107	142	104.5
0.5	219	158.5	114	102
1.0	215	213	114	102
2.5	219.5	222	114	101
5.0	226	173	114	101
10.0	226	105	113	
15.0	225.5	106	117	95.5
35.0	225.5	92.5	93	89

[a] For these data, the temperature corresponded to a modulus of 10^7.

that both effects were real and due to the same phenomena. Because the PE crystallites could not directly influence the modulus at 220°C, what we are suggesting is that the drop-off in crystallinity was a result of some complicated morphological change. In fact, some T_m's listed in Table VI are higher even than the corresponding temperature for pure nylon 6 (218°C); a direct comparison of the DMA spectra is shown in Figure 5. The increase in the drop-off modulus versus pure nylon must be due to differences in the spatial arrangement of nylon crystallites in the sample because DSC results indicated no substantial differences in the melting temperature or the percentage of crystallinity with the addition of the compatibilizing agent.
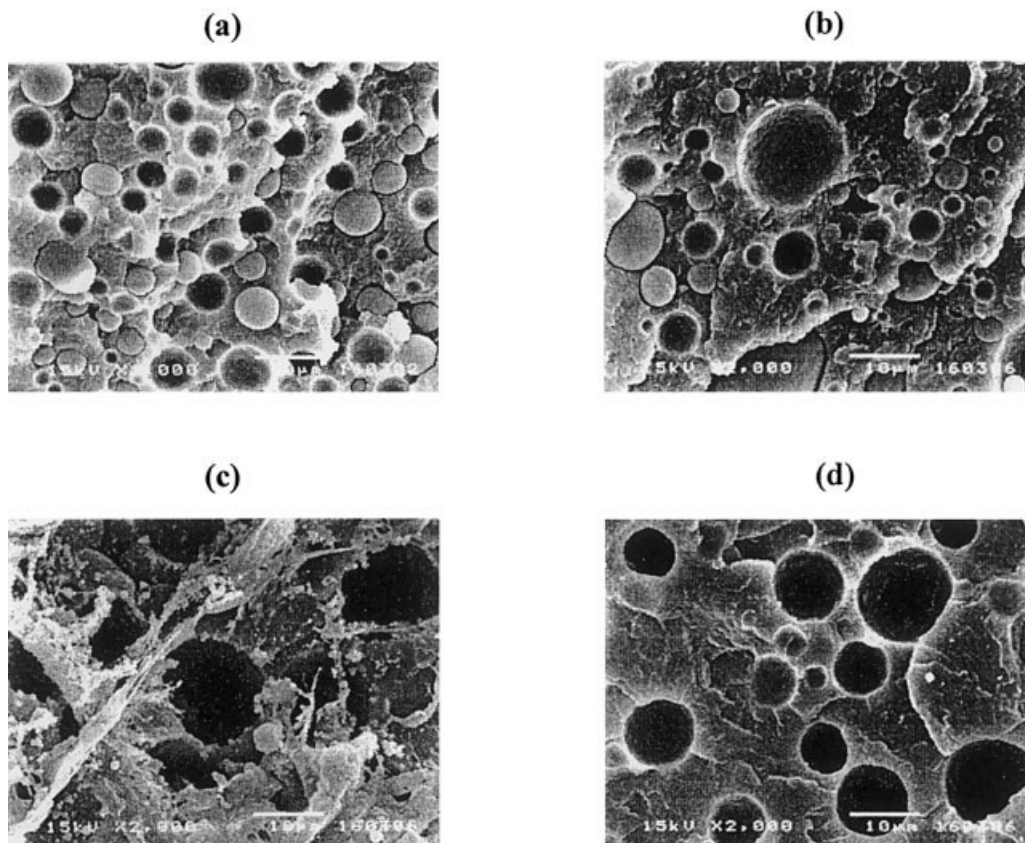

Figure 9 Morphology of blends without an ionomeric compatibilizer at the following nylon/LDPE ratios: (a) 0.8/0.2, (b) 0.6/0.4, (c) 0.4/0.6, and (d) 0.2/0.8.
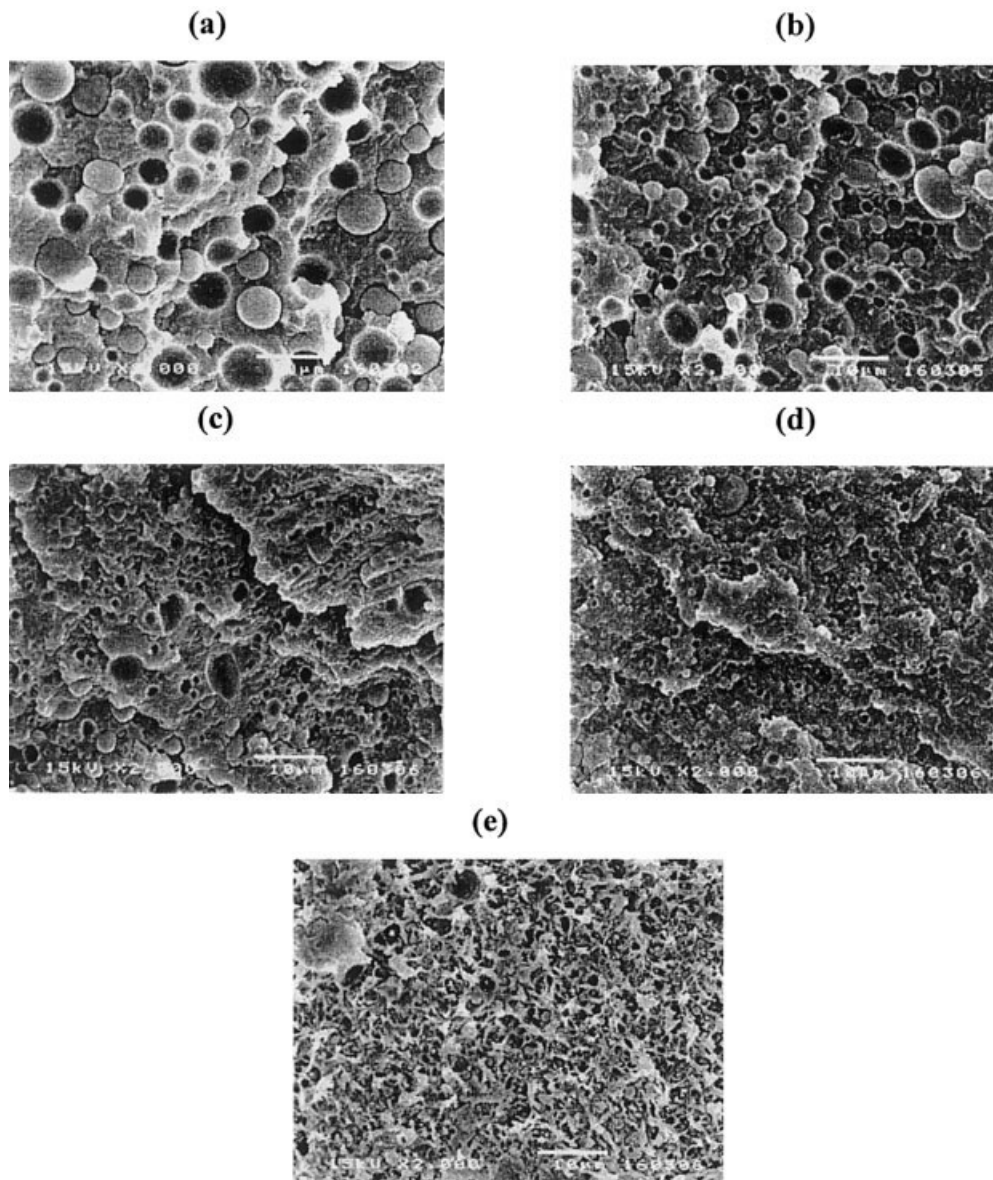
Figure 10 Morphology of 0.8/0.2 nylon/LDPE blends with an added ionomeric compatibilizer at the following weight percentages: (a) 0, (b) 0.1, (c) 0.5, (d) 1.0, and (e) 2.5%.

Figures 6–8 show much more dramatic changes than Figure 4. The addition of a small amount of the compatibilizer substantially shifted the transition corresponding to the melting of the material to higher temperatures, whereas even more compatibilizer caused a shift back toward lower temperatures. The change was most dramatic for the material with a nylon/LDPE ratio of 0.6/0.4. Originally, the transition was near that for pure PE, and it rose to that near nylon; eventually, it decreased and became even lower than that of the blend without the compatibilizer. Clearly, the compatibilizer caused phase inversion, first from a continuous LDPE phase to a continuous nylon phase and then back again to a continuous LDPE phase. This double phase inversion was really

quite remarkable. Interesting behavior was also found for the low-content blends, in which the addition of only 0.1% compatibilizer led to the maximum shift in the melting transition to higher temperatures. We suspect very strongly that the specific results detailed and discussed in this paragraph were highly dependent on the processing conditions, but we still suspect that qualitatively at least the results would be consistent.

Figure 9 shows SEM micrographs for the blends with no compatibilizer. The dispersed LDPE phase at high nylon contents had a much higher average domain size than the dispersed nylon phase at high LDPE contents. Presumably, a larger LDPE dispersed phase was found because the viscous force, which opposed drop condensation, was much lower for
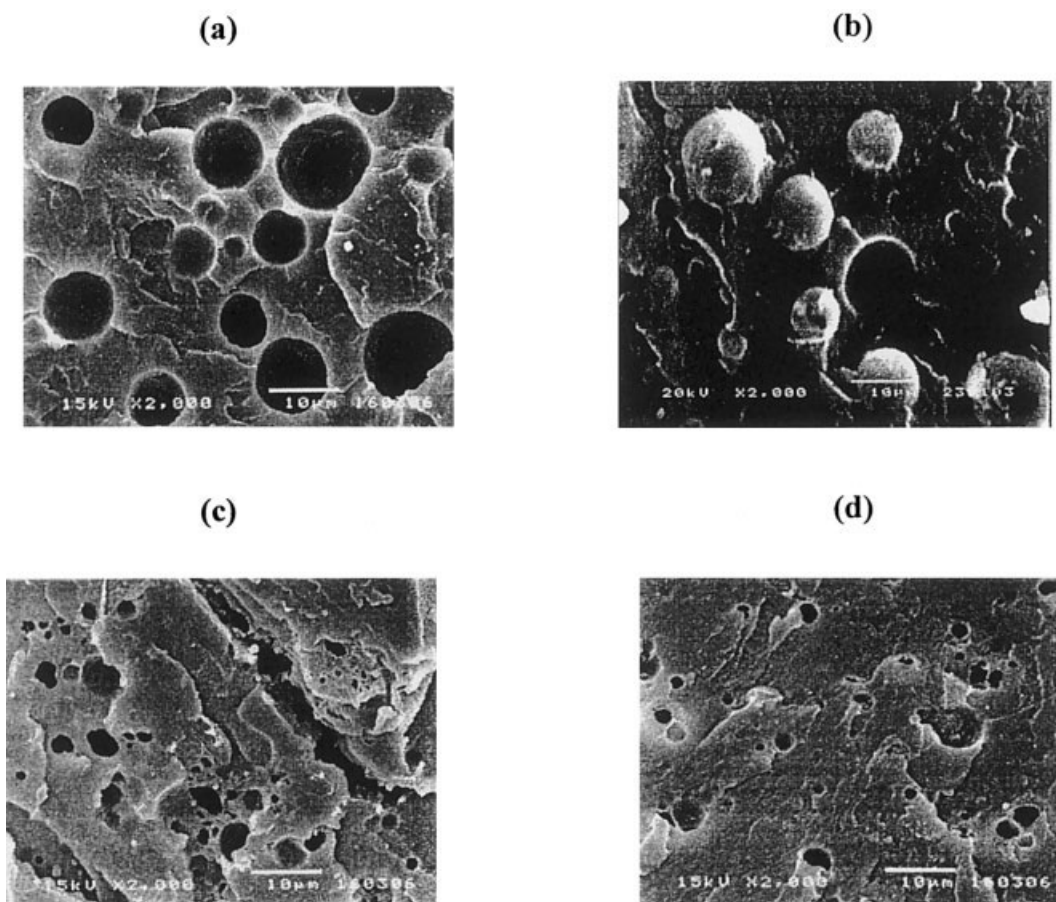
Figure 11 Morphology of 0.2/0.8 nylon/LDPE blends with an added ionomeric compatibilizer at the following weight percentages: (a) 0, (b) 0.1, (c) 0.5, and (d) 1.0%.

LDPE in nylon than for nylon in LDPE. At the two intermediate nylon/LDPE contents, the phase structure did not appear cocontinuous, but neither was one phase dispersed in a continuous phase of the other, as was the case at the two extremes. The addition of the ionomer caused a dramatic decrease in the average domain size, as shown in Figures 10 and 11. Interestingly, the morphology of the material with the dispersed nylon phase seemed to be qualitatively different from the morphology of the material with the dispersed LDPE phase, although this difference could be related to differences in the way in which the material fractured. In any case, these micrographs clearly confirm what the mechanical property measurements indicated, that the addition of an ionomeric compatibilizer dramatically changed the morphology of these nylon/LDPE blends.

CONCLUSIONS

An ethylene/methacrylic acid copolymer partially neutralized with zinc was shown to be an effective compatibilizer for nylon 6/LDPE blends. The ionomeric compatibilizer reduced the domain sizes and improved interfacial adhesion between the incompatible materials. In most cases, the amount of the compatibilizer needed to achieve the maximum improvement in the properties was a few percent or less; in fact, the addition of more compatibilizer could actually lead to unique phase inversion effects, as evidenced by dynamic mechanical results. Interestingly, at high nylon contents, a nylon with high impact strength could be produced that was comparable to nylon in many other respects.

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