# Interfacial Aspects of Latex IPNs for Toughening Polycarbonate. I. Synthesis and Characterization

R. HU,[1,3] V. L. DIMONIE,[1] M. S. EL-AASSER,[1,2,5,6] R. A. PEARSON,[4-7] L. H. SPERLING,[2,4-7,*] A. HILTNER,[8] and S. G. MYLONAKIS[9]

[1] Emulsion Polymers Institute, [2]Department of Chemical Engineering, [3]Department of Chemistry, [4]Department of Materials Science and Engineering, [5]Center for Polymer Science and Engineering, [6]Polymer Interfaces Center, [7]Materials Research Center, Lehigh University, Bethlehem, Pennsylvania 18015; [8]Department of Macromolecular Science and Center for Applied Polymer Research, Case Western Reserve University, Cleveland, Ohio 44106; [9]54 Wilshire Drive, Belle Mead, New Jersey 08502

## SYNOPSIS

Core/shell structured latex particles with an interpenetrating polymer network (IPN) core (polymer A and polymer B) and glassy shell (polymer C) were synthesized by emulsion polymerization techniques. Polymer A was poly(butadiene-*stat*-styrene) (90/10) (P(Bd/S)). Polymer B was either poly(butyl acrylate-*stat*-methyl methacrylate) (60/40) or poly(butyl acrylate-*stat*-styrene) (60/40) prepared by a sequential IPN technique to form the cores. The glass transition temperatures, $T_g$, of polymer A and polymer B were around −70 and 10°C, respectively. The shell, polymer C, poly(styrene-*stat*-acrylonitrile) (72/28), was polymerized with grafting onto the IPN core by a semicontinuous process. The particle size and particle size distribution were measured by light-scattering, capillary hydrodynamic fractionation, and transmission electron microscopy. The glass transition temperatures were determined by differential scanning calorimetry. The polymer A (particle sizes between 200–270 nm) with different gel fractions was used to characterize the effect of gel fraction on IPN morphology. A core/shell type phase separation was observed for IPN particles when using a polymer A with > 90% gel fraction. A distribution of polymer B domains in polymer A was obtained when using polymer A with a 40% gel fraction. A good coverage of polymer C on the IPN core particles was obtained at a core/shell ratio of one to one. A broad glass transition range for the core/shell structured latex particles was observed. In article II of this series, these latex particles will be used to toughen such engineering thermoplastics as polycarbonates to understand the particular toughening theories of these polymers. © 1995 John Wiley & Sons, Inc.

## INTRODUCTION

Good engineering plastics of the future should be light, cheap, and exhibit great toughness to withstand high stress and impact loading, in order to be used in the automotive, aerospace, electronics, appliance, and other industries.

It is well known that the toughness of thermoplastics can be improved by adding the rubber.[1] It has been found that a discrete rubbery phase in a continuous plastic matrix can significantly improve the toughness, often without deterioration of the desirable inherent loading-bearing-strength properties.[2] Rubbery core/glassy shell type latex particles can be individually dispersed in a matrix phase, forming a discrete rubbery phase. The big advantage of using core/shell type particles includes the convenient synthesis technique through emulsion polymerization. Important variables include the effect of rubber particle size, particle size distribution,
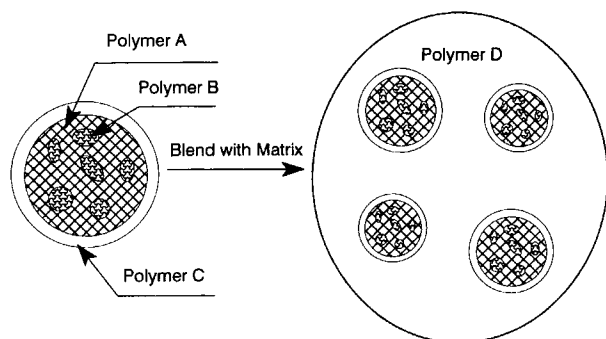
**Figure 1**   Possible morphology of the IPN latex rubber particle-toughened thermoplastic.

crosslink density of the rubber phase, degree of grafting of shell polymer on core polymer, and the shell polymer composition on the toughening mechanism. The multilayered core/shell morphology can be finely tailored for a particular plastic matrix, and such latexes also lead to easy processing procedures.[3]

So far, much work has been done on commercial rubber-toughened plastics. Although many commercial core/shell particles are available and used in practical application, only limited work has been done on using interpenetrating polymer network (IPN) particle as a core in rubber toughening application. An IPN is defined as a combination of two or more polymers in network form, with at least one synthesized and/or crosslinked in the presence of the other.[4] The IPNs may be prepared either by sequential or by simultaneous routes.[5,6] The latex IPNs can be prepared through emulsion polymerization, forming both networks in each latex particle.[6-11]

The objective of this work is to design a new kind of impact and damping modifier for thermoplastics to study toughening mechanisms of modified thermoplastics. The modifier is a core/shell structured particle with an IPN core and a glassy shell. The

IPN core consists of low and high glass transition temperatures, polymer A and polymer B in Figure 1. Polymer A must have a $T_g$ at least 60°C below the ambient temperature (i.e., $T_g < -40$°C) to act as a good toughening agent, and polymer B will have a $T_g$ between $-30$°C and $10$°C to provide effective damping, absorbing energy through the onset of coordinated molecular motion, which is similar to the action of sound and vibration damping materials,[12] and also to offer controlled resistance to cavitation. The shell material, polymer C, should provide good interfacial bonding between the core and the matrix plastic, polymer D.

The IPN particles were prepared by the sequential technique. Polymer A was poly(butadiene-stat-styrene) (P(Bd/S)) (90/10 by wt). Two kinds of polymer B were designed, poly(butyl acrylate-stat-methyl methacrylate) (P(BA/MMA)) and poly(butyl acrylate-stat-styrene) (P(BA/S)).

The IPN latex particles of this study have been designed for rubber modification of polycarbonate (PC), polymer D. PC is an engineering thermoplastic with excellent clarity, high heat deflection temperature, and high toughness. The impact resistance, however, of PC is notch sensitive, especially in thicker sections and at low temperatures. The notch sensitivity of PC can be improved by blending with a small amount of an elastomer.

**Table I   Recipe for Preparation of P(Bd/S) Latex Particles with High and Medium Gel Fractions by Emulsion Polymerization**

| Component | Recipe I Parts, g | Recipe II Parts, g |
|---|---|---|
| Distilled deionized water (DDI) | 90 | 90 |
| Bd/S (90/10) | 50 | 50 |
| Stearic acid | 0.92 | 0.345 |
| LiOH | 0.078 | 0.029 |
| $K_2S_2O_8$ (KPS) | 0.25 | 0.20 |
| Dodecyl mercaptan (DDM) | 0.05 | 0.05 |
| $Li_2CO_3$ | — | 0.028 |

**Table II   Recipe for Preparation of IPN Particles by Emulsion Polymerization**

| Component | Parts, g |
|---|---|
| P(Bd/S) particles | 2.25, 1.5, 0.75 |
| BA/MMA (60/40) or BA/S (60/40) | 0.75, 1.5, 2.25 |
| AIBN | 0.6% on monomer |
| t-EGDMA | 1% on monomer |
| Distilled deionized water | 12 |

**Table III   Recipe for Preparation of IPN/SAN Structured Latex Particles by Semicontinuous Emulsion Polymerization**

| Component | Parts, g |
|---|---|
| IPN seed | 3.0 |
| 72/28 S/AN | 3.0 |
| KPS | 1.0% on monomer |
| $NaHCO_3$ | 1.0% on monomer |
| Dowfax 2A1[a] | 6.0% on monomer |
| Distilled deionized water | 24 |

[a] Dowfax 2A1: sodium disulfonated dodecyl alkylated diphenyl oxide.

**Table IV    Particle Size of P(Bd/S) Latex Particles**

| Sample | G.F.[a] % | Conv. % | Nicomp $D_n$, nm | Nicomp $D_v$, nm | CHDF $D_n$, nm | CHDF PDI | TEM $D_n$, nm | TEM PDI |
|---|---|---|---|---|---|---|---|---|
| I | 95 | 100 | 208 | 230 | 203 | 1.02 | 180 | 1.02 |
| II | 40 | 50 | 259 | 276 | 270 | 1.01 | 230 | 1.02 |

[a] Gel fraction.

Polymer C was poly(styrene-*stat*-acrylonitrile) (SAN), which is compatible with the PC matrix at certain range of AN concentration.[13–18]

A lot of work has been done in rubber-toughened PC systems with commercial polymers. Paul et al.[19] investigated the PC blends with different acrylonitrile–butadiene–styrene (ABS) materials. A blending of PC with maleated ABS (ABS-g-MA) system was studied by Dong and co-workers.[20] The deformation mechanism of rubber modified PC has been reported previously.[21–24] The importance of such parameters as cavitation resistance, debonding of the rubber particles from the PC matrix, particle size, and interparticle distance were thoroughly investigated for MBS type modifiers.[21,23] Cavitation of rubber particles is recognized as a requirement for toughness enhancement because it relieves hydrostatic strain energy, which allows the buildup of shear strain energy. The buildup of shear strain energy is dissipated by the processes of matrix dilation and shear banding. To optimize toughness, it has been proposed that the rubber particles should cavitate at a specified stress level (matrix dependent) in order to maximize the size of the plastic zone.[25] The IPN technology developed in this study will provide a means for controlling the cavitation phenomena in the rubber particles to provide maximum impact resistance of the PC matrix.

## EXPERIMENTAL

### Materials

Butadiene (Bd) (Matheson Gas Products, Inc.) and styrene (S) (Aldrich) were purified by passing them through the Ascarite II (Thomas Scientific) and the aluminum oxide (Aldrich) columns, respectively. n-Butyl acrylate (BA) and methyl methacrylate (MMA) were purified by passing through the inhibitor-remover columns. Acrylonitrile (AN) was distilled before use. Potassium persulfate ($K_2S_2O_8$ or KPS) and 2,2'-azobisisobutyronitrile (AIBN) were used as initiators, without further purification. Dodecyl mercaptan (DDM) and tetraethylene glycol dimethylacrylate (t-EGDMA) were used as a chain transfer and crosslinking agents, respectively, without further purification. Lithium hydroxide (LiOH), lithium carbonate ($Li_2CO_3$), stearic acid, and Dowfax 2A1 (sodium disulfonated dodecyl alkylated diphenyl oxide, Dow Chemical Co.) were used as received.
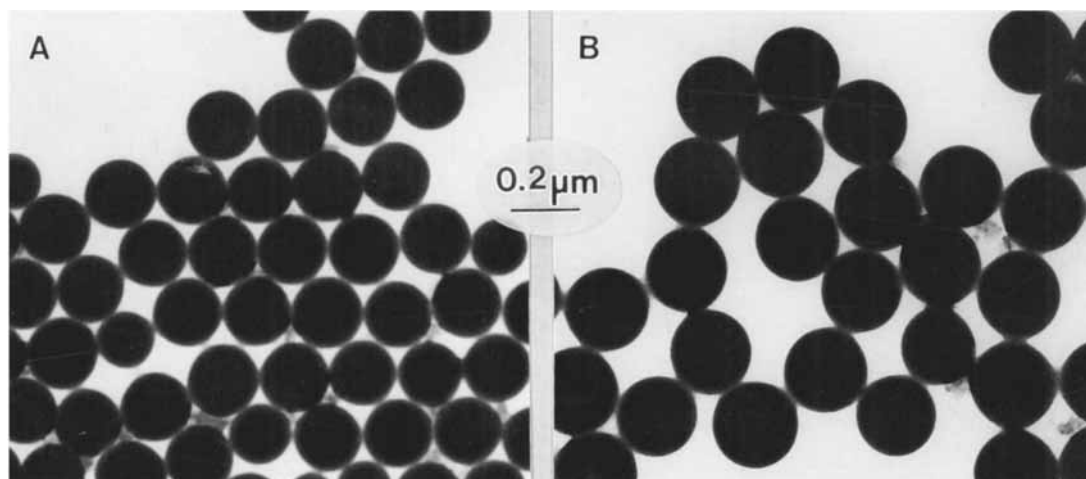


**Figure 2**    TEM micrographs of P(Bd/S) latex particles: (a) 95% gel fraction, 180 nm; (b) 40% gel fraction, 230 nm.
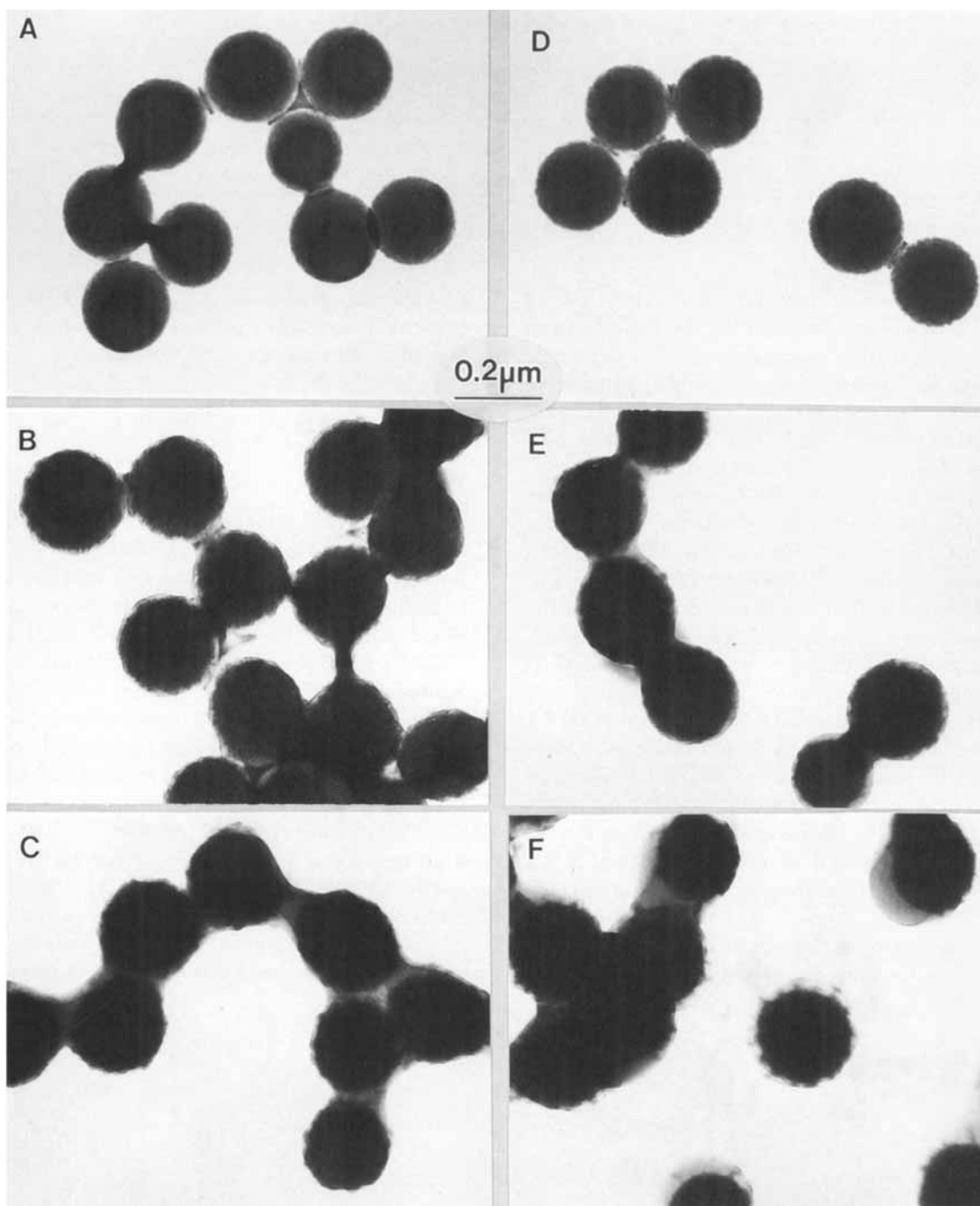
**Figure 3** TEM micrographs of IPN latex particles: (A) P(Bd/S)/P(BA/MMA) = 3/1; (B) P(Bd/S)/P(BA/MMA) = 1/1; (C) P(Bd/S)/P(BA/MMA) = 1/3; (D) P(Bd/S)/P(BA/S) = 3/1; (E) P(Bd/S)/P(BA/S) = 1/1; (F) P(Bd/S)/P(BA/S) = 1/3. P(Bd/S) cores with 95% gel fraction. Latex particles were stained with $OsO_4$ and PTA.

## Synthesis of Polymer A (P(Bd/S)) with Different Gel Fractions

Poly(butadiene-*stat*-styrene) (90/10 by wt) latex particles with high and medium gel fractions were prepared by emulsion polymerization based on the recipes given in Table I. Recipe I gave P(Bd/S) particles with high gel fraction at substantially 100% conversion. Because branching reactions cause gelation, especially in the later stages of polymerization
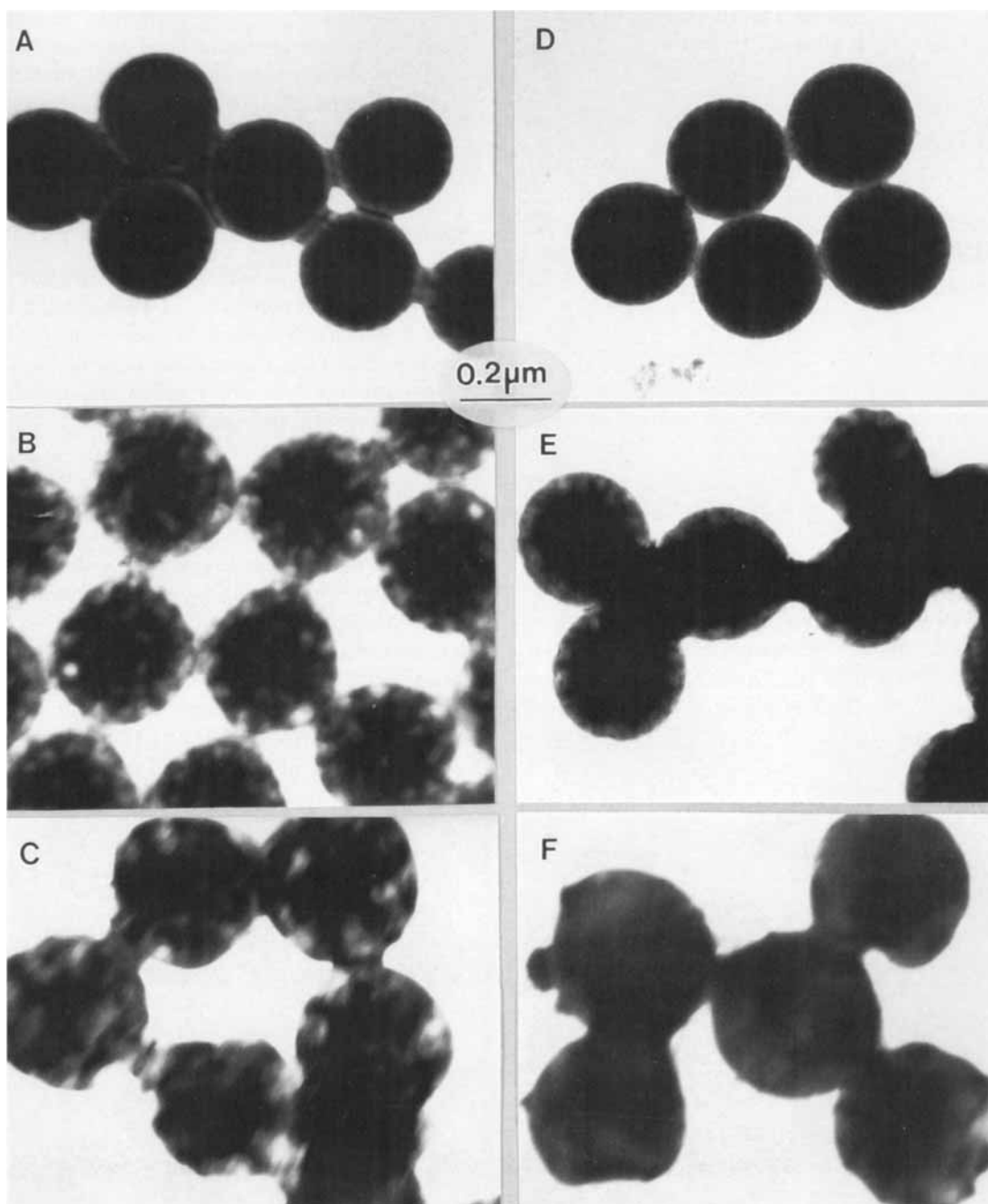
**Figure 4**   TEM micrographs of IPN latex particles: (A) P(Bd/S)/P(BA/MMA) = 3/1; (B) P(Bd/S)/P(BA/MMA) = 1/1; (C) P(Bd/S)/P(BA/MMA) = 1/3; (D) P(Bd/S)/P(BA/S) = 3/1; (E) P(Bd/S)/P(BA/S) = 1/1; (F) P(Bd/S)/P(BA/S) = 1/3. P(Bd/S) cores with 40% gel fraction. Latex particles were stained with $OsO_4$ and PTA.

of the P(Bd/S) particles, the medium gel fraction particles were obtained from recipe II by stopping the reaction at 50% conversion. The polymerizations were carried out in 250 mL pressure bottles by a batch process. The aqueous phase (distilled deionized water, KPS, LiOH, and $Li_2CO_3$ if used) was mixed with the oil phase (styrene, stearic acid, and $n$-dodecyl mercaptan) to form an emulsion. The bottles were purged with $N_2$ and then cooled in a $-10°C$ freezer for about 30 min. Finally, the condensed butadiene was charged into the bottles. The bottles were capped with a crown cap with a rubber

**Table V Particle Size and Gel Fraction of IPN Cores (P(Bd/S) with 40% gel fraction)**

| Monomer B | A/B | Nicomp $D_n$, nm | CHDF $D_n$, nm | Gel fraction % |
|---|---|---|---|---|
| BA/MMA | 3/1 | 279 | 294 | 99 |
| BA/MMA | 1/1 | 339 | 318 | 100 |
| BA/MMA | 1/3 | 402 | 401 | 100 |
| BA/S | 3/1 | 279 | 298 | 98 |
| BA/S | 1/1 | 341 | 322 | 97 |
| BA/S | 1/3 | 438 | 411 | 98 |

gasket, and placed in a bottle polymerizer at 70°C for 24 h.

## IPN Formation

Poly($n$-butyl acrylate-$stat$-methyl methacrylate) (P(BA/MMA)) and poly($n$-butyl acrylate-$stat$-styrene) (P(BA/S)) were polymerized as polymer B into the polymer A to form IPN latex particles. $t$-EGDMA was used as the crosslinker. The recipe is shown in Table II. The ratios of polymer A to polymer B were 3/1, 1/1, 1/3. The P(Bd/S) par-

ticles were swollen by the monomer mixture for 24 h at room temperature and then polymerized at 70°C for 24 h at 32 rpm in a bottle polymerizer. Both P(Bd/S) particles with high and medium gel fractions were used to see the effect of gel fraction of the P(Bd/S) polymer on IPN morphology.

## Synthesis of IPN/SAN Structured Latex Particles

Semicontinuous emulsion polymerization was carried out for the synthesis of IPN core/SAN shell type structured latex particles according to the recipe given in Table III. The additional emulsifier, Dowfax 2A1, was added in order to obtain a stable latex without forming secondary particles.

## Characterization

The average particle size and particle size distribution of the latex particles were determined using light scattering (Nicomp), capillary hydrodynamic fractionation (CHDF), and transmission electron microscopy (TEM, Phillips 300). The polydispersity index (PDI) was given as $D_w/D_n$. For TEM measurements, the latex samples were stained with osmium tetroxide (OsO$_4$). The particle sizes and par-
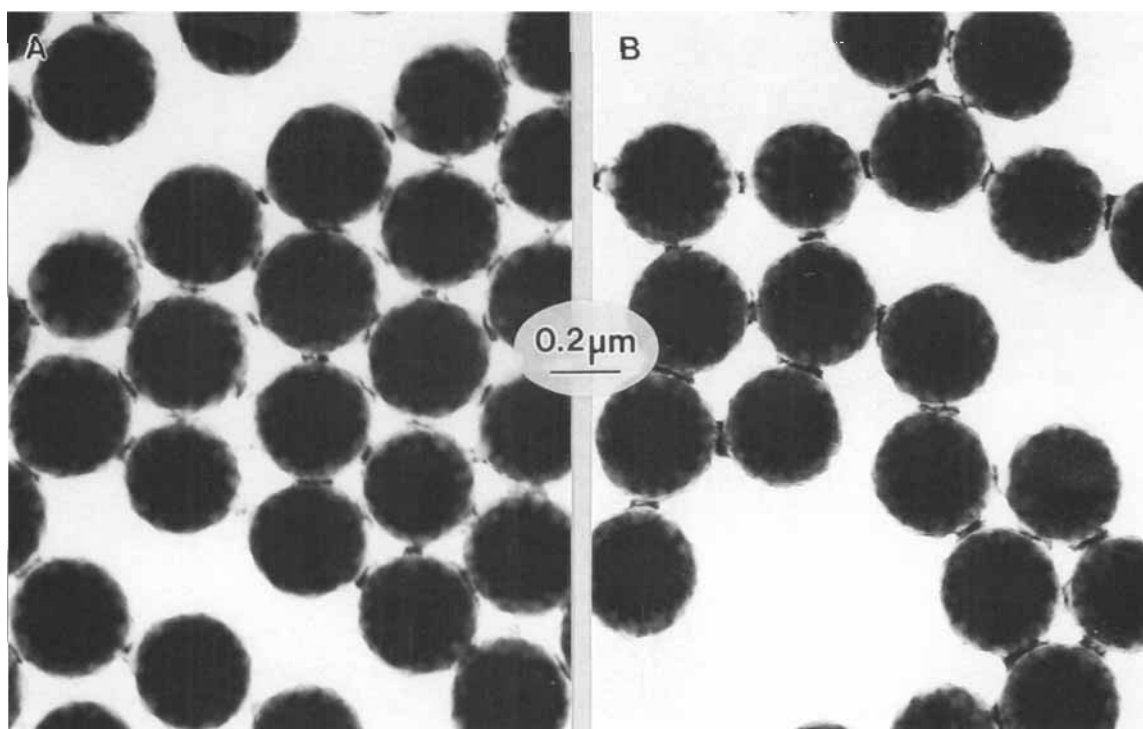


**Figure 5** TEM micrographs of IPN/SAN structured latex particles prepared by semicontinuous emulsion polymerization: (A) IPN-I = 1/1 P(Bd/S)/P(BA/MMA); (B)IPN-II = 1/1 P(Bd/S)/P(BA/S). P(Bd/S) with 40% gel fraction.

**Table VI   Particle Size of IPN/SAN Latex Particles (P(Bd/S)
with 40% Gel Fraction**

| Sample | IPN core | Theoretical $D_n$, nm | CHDF $D_n$, nm | CHDF PDI |
|---|---|---|---|---|
| IPN-I | 1/1 P (Bd/S)/P(BA/MMA) | — | 318 | 1.01 |
| IPN-I/SAN | 1/1 P(Bd/S)/P(BA/MMA) | 401 | 387 | 1.01 |
| IPN-II | 1/1 P(Bd/S)/P(BA/S) | — | 322 | 1.01 |
| IPN-II/SAN | 1/1 P(Bd/S)/P(BA/S) | 406 | 390 | 1.01 |

ticle size distributions were determined by measuring the diameters of at least 500 particles for each sample from the micrographs using a Zeiss MOP-3 analyzer. The morphologies of the IPN and core/shell latex particles were determined by TEM (Phillips EM 400T), with the particles stained both positively with OsO₄ for P(Bd/S) polymer and negatively with phosphotungstic acid (PTA) for the background.

The gel fraction of the P(Bd/S) particles was determined by a solvent extraction method. The latex particles were first dried at room temperature. Then 0.2 g of the polymer film was swollen by an excess of toluene for 48 h, followed by centrifugation for 1 h at 3000 rpm. Then, 2.5 g of the supernatant containing the soluble P(Bd/S) was dried in an oven at 75°C and weighed. The gel fraction was determined by difference.

The glass transition temperatures were measured by differential scanning calorimetry (DSC, Mettler DSC30) on 20 mg samples. The heating rate was 10°C/min, from −120 to 150°C.

## RESULTS AND DISCUSSION

### Particle Size and Morphology

Using the recipes in Table I, P(Bd/S) particles with narrow particle size distributions were obtained, see Table IV. Figure 2 shows the TEM micrographs for these two kinds of P(Bd/S) particles. In both recipes, stearic acid reacts with lithium hydroxide to form lithium stearate, which forms soap micelles. Lithium carbonate can be added to control the solubility of lithium stearate, and hence, the conversion and the particle size (recipe II in Table I).

Two types of polymer B were synthesized into polymer A particles to form IPN latex particles. The ratio of BA/MMA or BA/S was kept at 60/40 in
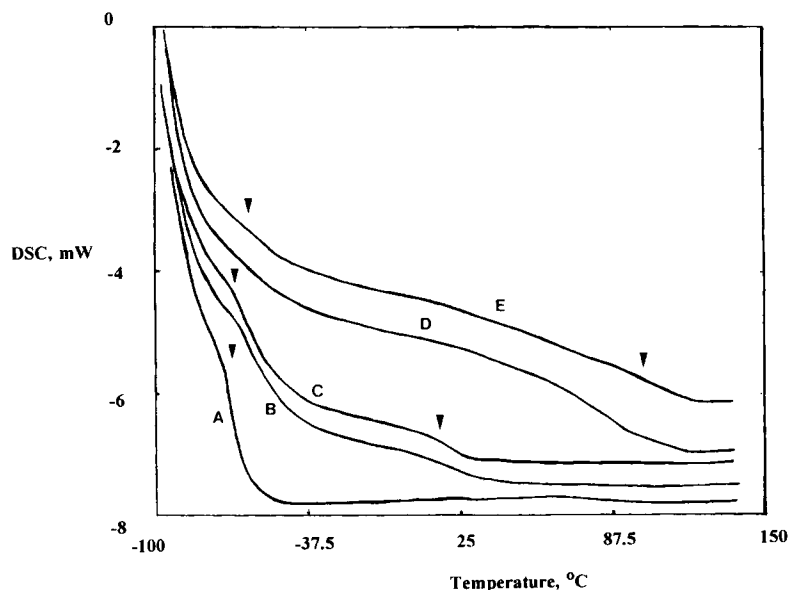


**Figure 6**   DSC curves for IPN and IPN/SAN latex particles: (A) P(Bd/S); (B)P(Bd/S)/P(BA/MMA) (IPN-I); (C) P(Bd/S)/P(BA/S) (IPN-II); (D) IPN-I/SAN; (E) IPN-II/SAN.

**Table VII    Glass Transition Data for IPN Cores and IPN/SAN Structured Latex Particles**

| Composition | $T_g$, °C | $\Delta Cp$, J/gK |
|---|---|---|
| P(Bd/S) (40% gel fraction) | −70 | 0.573 |
| P(BA/MMA) (60/40 by wt) | 10 | 0.050 |
| P(BA/S) (60/40 by wt) | 13 | 0.119 |
| SAN | 113 | 0.369 |
| 1/1 P(Bd/S)/P(BA/MMA) (IPN-I) | −62, 17 | 0.152, 0.043 |
| 1/1 P(Bd/S)/P(BA/S) (IPN-II) | −63, 13 | 0.205, 0.06 |
| 1/1 IPN-I/SAN | −56, 94 | — |
| 1/1 IPN-II/SAN | −55, 95 | — |

order to obtain a $T_g$ of around 10°C (from the Fox equation) in order to have good damping at room temperature. P(Bd/S) particles with high and medium gel fractions were compared to see the effect of gel fraction on particle morphology. In addition, the ratio for P(Bd/S) and polymer B was varied to see the effect of polymer B on the particle morphology. Figures 3 and 4 show the morphologies of these particles. For P(Bd/S) with high gel fraction, phase separation was observed for polymer B surrounding the P(Bd/S) particles [Fig. 3(B), (C), (E), and (F)] (the white or gray area around the dark P(Bd/S) particle) when the ratio of polymer A to polymer B was 1/1 or lower, and the phase separation was reduced at higher polymer A/polymer B ratio (3/1) [Fig. 3(A) and (D)]. This core/shell phase separation is due to the high crosslink density of P(Bd/S) core, which possibly restricts swelling of the core particles by the second-stage monomers, resulting in the exclusion of polymer B to the exterior of the composite particles, making irregular domains. In addition, the phase separation is due to the immiscibility between the P(Bd/S) core and P(BA/MMA) or P(BA/S) polymer. A quite different morphology was observed when a P(Bd/S) core with medium gel fraction was used. At all A/B ratios, no obvious core/shell formation was observed (Fig. 4). When the A/B ratio was 3/1, almost uniform particles were observed, suggesting very small domains of polymer B in polymer A [Fig. 4(A) and (D)]. When the A/B ratios were 1/1 and 1/3, P(BA/MMA) and P(BA/S) domains were uniformly distributed in the P(Bd/S) particles [Fig. 4(B), (C), (E), and (F)], forming a cellular type morphology. The P(Bd/S) cores with medium gel fraction can be swollen to a greater extent, so they can accept more of the second-stage monomers and polymers, forming a domain-type morphology

instead of a core/shell morphology. Because the solubility parameters for polybutadiene, polystyrene, and poly(methyl methacrylate) are 17.2, 18.6, and 19.3 $(MJ/m^3)^{1/2}$, respectively, the domain size of P(BA/MMA) and P(BA/S) in the P(Bd/S) phase could be different. This can also be seen from Figure 4. The lower the A/B ratio the larger the domain size. The expected particle size for A/B ratios of 3/1, 1/1, and 1/3 are 297, 340, and 429 nm, respectively. The measured particle sizes of these IPN particles generally agree well with the expected ones, as shown in Table V.

The core/shell type morphology can be obtained by the semicontinuous polymerization mode in the
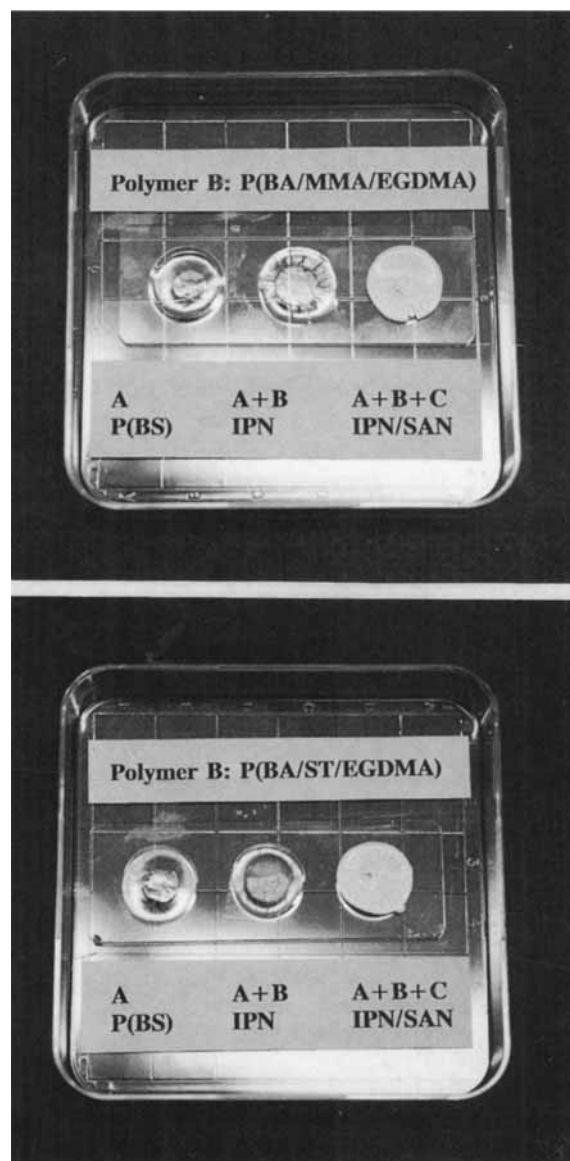


**Figure 7**    Physical appearance of IPN cores and IPN/SAN structured particles.

shell monomer polymerization. Two types of IPN/SAN structured latex particles were synthesized from the two different IPN cores, P(Bd/S)/P(BA/MMA) (referred to as IPN-I) and P(Bd/S)/P(BA/S) (referred to as IPN-II) (1/1 wt ratio, P(Bd/S) with medium gel fraction). A 72/28 (by weight) S/AN monomer mixture was chosen in order to get a maximum adhesion between PC and SAN. Figure 5 shows the morphologies for these two kinds of particles. The IPN cores are black spheres. The black area between the particles is due to the negative staining by PTA and the white area around the IPN cores is the SAN shell. A full coverage of SAN shell on IPN cores is obtained. The particle sizes of both IPN/SAN particles are in good agreement with the theoretical numbers, based on particle A diameters, as given in Table VI.

## DSC Results

An ideal IPN for sound and vibration damping exhibits a single, broad glass transition temperature. However, in many cases, two distinct transitions may be observed in IPN samples attributed to extensive phase separation. The DSC results of the samples in this work are illustrated in Figure 6 and summarized in Table VII. Both IPN-I and IPN-II cores show two $T_g$s, one corresponding to the P(Bd/S) phase and the other to the P(BA/MMA) and P(BA/S) phases. The $T_g$s for the P(Bd/S) phase in the IPN cores were higher than that of the pure P(Bd/S) polymer, suggesting that some P(BA/MMA) or P(BA/S) was molecularly dissolved in the P(Bd/S). The $T_g$s for the P(BA/MMA) and P(BA/S) phases, however, instead of lower, were even higher than those of P(BA/MMA) and P(BA/S) random copolymers. This is possibly because the compressive force exerted by the difference in the expansion coefficients on cooling from the polymerization temperature restricts the polymer chain movements in the P(BA/MMA) or P(BA/S) domain, resulting in higher $T_g$s. For both core/shell particles [(D) and (E) in Fig. 6], the $T_g$ of P(Bd/S) phase was even shifted higher and broad glass transitions between −40 to 90°C were observed, indicating some molecular mixing for the P(BA/MMA) and P(BA/S) phase with the SAN shell polymer forming an interphase zone. A $T_g$ around 100°C for the SAN rich phase was observed for both IPN/SAN particles.

## Physical Appearance

The particle morphology can also be demonstrated by the physical appearance of the dried sample basically from its color and film formation behavior. Figure 7 shows the room temperature-dried IPN cores and IPN/SAN structured latex particles that were prepared from P(Bd/S) particles with medium gel fraction. The pure P(Bd/S) polymer is a continuous transparent film at room temperature due to its low $T_g$ (−70°C). After the formation of the network, the IPN latex particles still form films but look a little bit hazy, with some cracks formed after drying. This indicates that one or both of two phenomena might be important: if polymer B exists to any extent as a shell material, film formation might be nonuniform. Alternately, a significant degree of phase separation is known to occur between polymers A and B. Another factor retarding film formation, of course, is the crosslinked nature of these films. A white, opalescent, powder was formed for the IPN/SAN structured particles because of the high $T_g$ of SAN (113°C from DSC), which confirms the formation of a core/shell type particles with a narrow particle size distribution.

## CONCLUSIONS

P(Bd/S)/P(BA/MMA) and P(Bd/S)/P(BA/S) IPN cores with two glass transition temperatures, −70 and 15°C, were prepared by a sequential emulsion polymerization technique. For lower crosslinked P(Bd/S) seed latexes (40% gel fraction), a greater swelling in of monomer B occurred with concomitant cellular domain formation of polymer B. The IPN core/SAN shell latex particles were synthesized by semicontinuous emulsion polymerization. For the combination of polymer A, B, and C, two shifted glass transitions were observed. The transition of polymer B is either shifted under polymer C transition or combined with it.

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