ORIENTATION TEXTURE AND GAS TRANSPORT
IN SEMICRYSTALLINE BLOCK COPOLYMER BLENDS

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ABSTRACT

The shear-induced morphologies produced by plane strain compression using a channel die, were investigated in semicrystalline ethylene/ethylene-propylene (E/EP/E and E/EP) block copolymers blended with semicrystalline low density polyethylene and amorphous polyisoprene homopolymers. Two dimensional small angle x-ray scattering (SAXS) was used to determine the lamellar orientation relative to the specimen boundaries. Using a cooling rate of 0.27°C/s and a load of 9.2 MPa, a normal to the plane of shear microstructure orientation texture was produced in the blends. The E homopolymer chains co-crystallize within the confinement of the oriented microphase separated E block copolymer domains. The long period spacing of the oriented microphase separated E/EP block microstructure remained unchanged, while the spacing associated with the crystallization of the E chains increased with increasing weight percent of E homopolymer in the blend. Gas permeability coefficients for He and CO₂ were measured for isotropic and oriented blends. The separation properties of these polymer systems was altered by changing the mechanism of gas transport. A selective solvent was used to develop a uniform porous structure in the oriented blend morphologies. The semicrystalline block copolymer blend structure oriented perpendicular to the direction and normal to the plane of shear presents the opportunity to create a high flux porous structure, while keeping the pores of nanometer dimensions, thus providing some selectivity and retaining mechanical strength.
INTRODUCTION

The gas transport properties of polymeric materials have been extensively studied and applied industrially for the manufacturing of high throughput membranes for gas separations as well as for low permeability (barrier) uses [1, 2, 3]. Polymer blends are of interest because new polymers can be designed or miscible polymers whose properties are already known can be blended to combine their properties. While miscible blends are expected to have properties essentially intermediate between those of the constituents (glass transition temperature, mechanical moduli), incompatible blends are expected to behave as a heterogeneous composite. The presence of a block copolymer in the blend strengthens the interfaces and acts synergistically to improve properties for optimum composite material behavior [4, 5].

Block copolymers are macromolecules composed of sequences or blocks of chemically distinct repeat units. The chemical link between different blocks prevents phase separation on a macroscopic length scale. Microphase separation of diblock copolymers leads to spherical, cylindrical, bi-continuous, and lamellar morphologies [6, 7]. Although we refer to this microphase separated structure of block copolymers as microdomains, typical domain sizes are in the nanometer range. These nanodomains are essentially monodisperse, and the morphology and domain sizes can generally be controlled by adjusting the length of each block and the total molecular weight.

Semicrystalline block copolymers offer a much wider range of possibilities than wholly amorphous materials with regard to increased toughening, resistance to solvents and acids, and higher working temperature applications. Along with these advantages, incorporating crystallinity into a new material presents a variety of challenging problems, both from a syn-
thesis and a processing point of view. The synthetic pathways required to produce semicrystalline polymers are generally more complex than for wholly amorphous systems, and the interaction between the kinetically driven crystallization process and the thermodynamically driven microphase separation has become a topic of several research efforts [8, 9, 10, 11, 12]. In this context, the behavior of a block copolymer with at least one crystallizable moiety is particularly interesting because of the multiplicity of “frozen-in” morphologies which can be expected [13, 14, 15].

The shear-induced morphologies produced above the E block melting point in a series of ethylene/ethylene-propylene (E/EP) diblock and E/EP/E triblock copolymer systems have been reported [16, 17], and were attributed to the proximity of the order-disorder temperature to the processing temperature. The unusual shear-induced perpendicular lamellar morphology for the semicrystalline diblock and triblock systems may have some potential advantages; for example, it provides a “parallel” material from the perspective of transport through a film. Use of this structure was made in earlier studies [17, 18] of gas transport in semicrystalline block copolymer systems. The structure in which the alternating amorphous and semicrystalline lamellae are oriented normal to the film surfaces enables the membrane designer to enjoy the structural and thermal stability offered by the semicrystalline regions without having them interfere with the gas flux through the amorphous lamellae.

Another way to influence the permeability of gases through block copolymer systems is to create a porous membrane by extracting one of the components of a semicrystalline block copolymer blended with an amorphous or semicrystalline homopolymer, or by selectively swelling one of the microphase separated blocks. Porous polymeric materials have a wide variety of applications [19, 20, 21] ranging from microfiltration, ultrafiltration, reverse osmo-
sis and gas separations to catalyst supports, controlled release devices, and artificial cells. These porous structures are exciting for gas separation applications because they represent an opportunity to increase the void content of a polymeric membrane, which has been shown to increase the gas flux, while retaining gas selectivity [22].

We explored a novel pore producing technique in semicrystalline block copolymer and blend systems, referred to as controlled solvent crazing or solvent extraction, depending on the polymer system. This technique has been successfully applied before in completely amorphous block copolymers and their blends with homopolymers [23]. Membranes having oriented nano - morphologies, produced from channel die compression of block copolymer and blend systems, are exposed to a selective solvent, which causes the amorphous phase to expand, while the semicrystalline phase provides mechanical resistance to limit the degree of swelling. In the case of a blend, the selective solvent only dissolves the blended homopolymer. The absorbed solvent is subsequently completely removed by exposure to vacuum, leaving behind a porous polymer film. The permeability in these semicrystalline block copolymers and blend systems increases due to the introduction of the pores. Careful choice of solvent systems is used to reduce the formation of regions of very high free volume, which could significantly reduce selectivity. The block copolymer structure presents the opportunity to create a high flux porous structure, while keeping the pores of nanometer scale, thus providing some selectivity and retaining mechanical strength.

EXPERIMENTAL

Materials: The E/EP and E/EP/E block copolymers were synthesized by hydrogenation of 1,4-poly (butadiene) and 1,4-poly (isoprene) precursors. The butadiene block consists of
10% 1,2, 35% trans 1,4 and 55% cis 1,4 PB, while the isoprene block contains 93% cis 1,4 and 7% 3,4 PI. Hydrogenated butadiene thus resembles low-density polyethylene (E) and hydrogenated polyisoprene is essentially perfectly alternating ethylene propylene rubber (EP). The molecular weights of both blocks of the E/EP diblock are 100,000 g/mole and the E/EP/E triblock blocks are 30,000 g/mole for the E end blocks and 40,000 g/mole for the EP middle block. These values were determined from gel permeation chromatography (GPC) measurements on the polydiene precursors, from knowledge of reactor stoichiometry and conversion, and from a previous demonstration [24] that little or no degradation occurs during the hydrogenation reactions. The 1,4 polyisoprene homopolymer (I) used in the blend samples was anionically synthesized and had a number average molecular weight ($M_n$) of 38,000 g/mole. The E homopolymer was low density polyethylene, having a weight average molecular weight ($M_w$) of 28,000 g/mole.

**Blending:** Homopolymer blending with block copolymer samples was accomplished by dissolving 5 - 7 grams of the block copolymer and 10 weight percent semicrystalline low density polyethylene (E) or amorphous polyisoprene (I) homopolymer in 200 ml of heptane in a 250 ml flask. A Teflon stir bar was added to the solvent/polymer mixture and the flask was placed on a heat/stir pad. The flask was then slowly heated to approximately 90°C while stirring with medium agitation for 30 minutes. When transparent, the solution was precipitated with a (10:1) solvent:polymer solution made up of an even mixture of acetone and methanol. The precipitated blend was collected by vacuum filtration and air dried for 24 hours. The sample was then oven dried for 24 additional hours at 70°C.

**Channel Die Processing:** Isotropic block copolymer and blend samples were heated to 150°C, above the melting point and the order disorder temperature to erase any thermal
history, in a standard laboratory press equipped with digitally controlled heating platens. Load was applied and released to press the sample into a film. The heat was turned off and the film was allowed to cool to room temperature. Once cooled, the polymer film was cut into \( \frac{1}{2} \) inch by \( \frac{1}{2} \) inch squares. The squares were stacked in the center of a channel die. The channel die was placed into the press and heated to the desired temperature, and the polymer was subjected to plane strain compression. A representation of the channel die and its principle deformation directions, the loading (LD), constraint (CD) and flow (FD) directions, are shown in Figure 1.

The channel die was maintained at a selected constant temperature during the compression flow. An applied load of 9.2 MPa was applied continuously using the press. The compressed specimens were quenched under load to room temperature at a mean rate of 3.50°C/s. After the samples were cooled to room temperature, the load was released. The final compression ratio was determined from the reduction of the thickness of the samples. The channel die experiments were conducted at a range of compression ratios of \( \lambda = 10 \) to \( \lambda = 20 \). The temperature data was obtained by placing a thermocouple into the polymer melt during channel die compression. Data was recorded every 5 seconds while the cooling rate was applied.

*Small Angle X-Ray Scattering Experiments:* The change in orientation of the block copolymer blend morphology due to deformation was studied by means of small-angle x-ray scattering (SAXS). The SAXS data was obtained using the 10-meter digital camera in the National Institute of Standards and Technology (NIST) Polymers Division [25]. This SAXS instrument is modeled after the one designed by Hendricks[26] at Oak Ridge National Lab. The 2-D data sets obtained in these experiments were corrected for dark current and
unscattered primary beam. Depending on whether the scattering was isotropic or oriented, 
the data was reduced to circular or sector averages by averaging over the detector picture 
elements within a series of annuli located at a fixed distance from the beam center. Sector 
averages were done at azimuthal angles of ±45° with a width of ±22.5°. All intensity values 
were normalized to a NIST Lupolen sample to provide absolute intensity. Uncertainties were 
calculated from the standard deviation of the pixel statistics in the averaged annulus and 
were plotted only when the uncertainty limits were larger than the size of the plotted data 
points [26, 27].

*Gas Permeability Measurements:* Permeability measurements were taken on an ASTM 
Dek-Tron Scientific DCS135 Permeability cell [28]. One inch diameter membranes were 
prepared from blend samples oriented using the channel die and processed under the desired 
deformation, temperature, and cooling rate conditions. Solvent swollen permeation samples 
were produced by exposing a permeation sample to a good solvent for the EP block and the 
I homopolymer. The solvents used in this study were xylene and heptane. The sample was 
soaked in the solvent for 24 hours at 35°C with low agitation. The samples were then removed 
and placed in a beaker with a perforated cover to allow the solvent to evaporate slowly over 
a five day period. The sample thickness was measured and the sample was mounted between 
two neoprene rubber gaskets on the permeability cell. The system was purged with the 
permeating gas for a period of 15 minutes. The apparatus was then sealed and submerged in 
a water bath to maintain constant temperature. The system was checked for leaks and then 
allowed to come to equilibrium over a minimum of twelve hours at the desired gas pressure. 
Permeability measurements were taken over a period of 4 hours for CO₂ and He gas. These 
values were then averaged.
RESULTS

The addition of E homopolymer into a random spherulitic 30/40/30 E/EP/E triblock copolymer was studied using SAXS. Figure 2 contrasts the 2-D SAXS patterns for an isotropic sample of an (a) unblended triblock and an isotropic (b) 10 wt% E homopolymer triblock blend. Two concentric rings are evident in the SAXS patterns. The inner ring is associated with the microphase separated long period of the E and EP blocks and the outer ring with the crystalline - amorphous long period of the semicrystalline E chains. The addition of semicrystalline E homopolymer causes the intensity of the outer ring to increase. A comparison of the circular averaged IQ² vs Q plots obtained from these samples is shown in Figure 2c and shows the inner ring peak (D₁) decreases in intensity a and there is a slight increase in the intensity of the outer ring peak (D₂) as result of blending the semicrystalline homopolymer.

The Bragg spacings obtained for the isotropic triblock and 10 wt% E homopolymer triblock blend are presented in Table 1. The shift in position of the peaks results in a increase of 7.6 nm in the Bragg spacing corresponding to the crystalline - amorphous long period of the E chains. There is no observed change in the Bragg spacing of the E/EP microphase separated block copolymer long period.

Depending on the cooling rate and temperature of channel die processing in semicrystalline block copolymers, microstructure orientations parallel, perpendicular or transverse to the direction of shear can be achieved. It has been reported that a perpendicular to the direction and normal to the plane of shear microphase separated block copolymer lamellar orientation [29] is developed in a E/EP/E 30/40/30 triblock sample using a 3.50°C/s cooling
rate during plane strain compression processing above the E block melting point, and an applied load of 9.2 MPa. Blending this triblock with semicrystalline E homopolymer up to 10% by weight, and subsequently processing the blend in the channel die using the same processing conditions was explored. It was found that blending up to 10 wt% semicrystalline E homopolymer did not disrupt the oriented microphase separated morphology and the same normal to the plane of shear microstructure orientation texture as in the unblended triblock was achieved. Figure 3 provides the 2-D SAXS patterns for a series of triblocks blended with (a) 2.5 wt% E, (b) 5.0 wt% E, and (c) 10.0 wt% E which have microstructures oriented normal to the plane of shear. In each SAXS pattern, two sharp dots at 43.6 nm are observed in the constraint direction corresponding to the orientation of the block copolymer microstructure. A circular ring is also observed for each sample, associated with the crystallization of the E chains. The IQ² vs Q sector average plots for the three oriented block copolymer blend samples shown in Figure 3 are presented in Figure 4. In each of the three blends, the first peak from the oriented E/EP block copolymer microphase separated domains have a peak centered at Q=0.144 nm⁻¹. The second peak at higher Q is attributed to the crystalline - amorphous spacing within the E block copolymer domain. Figure 4 is an enlargement of the semicrystalline E chain portion of the SAXS data where the change in Bragg spacing is occurring. With increasing weight percent of semicrystalline E homopolymer added, the maximum peak Q value of this peak attributed to the crystallized E chains in each blend, can be seen to decrease, and broaden from Q=0.433 nm⁻¹ for the oriented unblended sample. The resulting Bragg spacings from these oriented block copolymer blend systems are presented in Table 2. An increase in the Bragg spacing of the semicrystalline E domain is observed with increasing weight percent E homopolymer. The E/EP microphase separated
block long period, however, shows no change in Bragg spacing upon addition of up to 10% E homopolymer, and subsequent processing in the channel die.

Permeability measurements of these samples were taken for both isotropic and perpendicular orientation morphologies which were blended with a semicrystalline E homopolymer. The permeability results for the random spherulitic blend samples are shown in Table 3. A decrease in permeability can be seen for both gas species with the addition of 10 wt% E homopolymer. The permeation results obtained for the homopolymer blended samples which were oriented perpendicular to the plane of shear, using the channel die, are shown in Table 4. With the addition of E homopolymer, the permeability of both gas species decreased.

The room temperature gas permeability values of isotropic E/EP 100/100 diblock samples blended with 10 wt% PI, which were exposed to xylene or heptane to dissolve the PI homopolymer, are shown in Table 5. The results indicate the formation of a porous morphology upon the dissolution of the PI homopolymer, where the increase in permeability was dependent on the degree of swelling and type of solvent. The largest increase in permeability was observed in the xylene treated sample which produced the largest degree of swelling. An increase of 25 barrer for CO$_2$, 32 barrer for He, and a decrease in the selectivity of the polymeric membrane were observed after the xylene treatment. The heptane treatment resulted in a less pronounced increase in the permeability of the membrane for both gases.

DISCUSSION

Permeation of gases through membranes is usually described by the solution - diffusion model developed by van Amerongen, Barrer and others [30, 31, 32, 33]. In this model, it is
assumed that gas on the high - pressure side of the membrane dissolves in the membrane ma-
terial and diffuses down a concentration gradient to the low - pressure side of the membrane,  
where the gas is desorbed. Gas permeation in semicrystalline polymers is essentially confined  
to the amorphous regions since gas molecules are generally taken to be insoluble in polymer  
crystallites and, therefore, are unable to permeate through them [34]. The crystallites reduce  
the permeability by decreasing the volume of polymer available for penetrant solution and  
by constraining the transport along irregular tortuous paths between the crystallites. The  
reduction in permeability ($P$), which is the product of the effective diffusion ($D$) and solubil-
ity ($S$) coefficients ($P = D \cdot S$), will be proportional to the volume fraction of the crystalline  
phase [35] when all samples have a random misoriented morphology.  

The effect of nanodomain orientation on gas permeability is significant. Permeability  
coefficients for a film whose nanodomains are oriented normal to the film surface (parallel to  
the permeation direction), are much higher than for a film having its nanodomains oriented  
in the same plane as the film surface (in series with respect to the permeation direction)[17].  
Figure 5 shows the many possible orientation morphologies that have been achieved [18, 17,  
29] with channel die processing of semicrystalline block copolymers and their expected gas  
transport properties.  

The permeability results obtained for samples oriented normal to the direction and per-
pendicular to the plane of shear, and then exposed to a selective solvent indicate an increase  
in the measured gas permeability. The polyethylene block and E blended homopolymer is  
resistant to the xylene solvent, but is expected to swell to a small extent and may be sus-
ceptible to a small degree of recrystallization. The solvent is then evaporated slowly to allow  
the swollen amorphous EP block to relax, while creating pores throughout the amorphous
material, due to the constraint imposed by the crystallized E chains which lock-in the porous microstructure. The permeation behavior is not entirely governed by transport through a porous structure in these semicrystalline block copolymer blend systems. In a sample with porosity dominating the permeation mechanism, selectivity would approach unity. An unusual result to note is, that for the case of the amorphous I homopolymer blending (Table 5) the separation factor (column 3) for the heptane swollen sample can be maintained at a constant level while the permeability of the CO$_2$ gas increases by a factor of about 50%. There is clearly not a complete change to a porosity dominated permeation mechanism, because CO$_2$, the larger molecule, remains the faster permeating species. In the case of the xylene swollen sample however, selectivity is lost.

Rangarajan et al [36], have published studies on homogeneous semicrystalline E/EP diblocks, which were blended with 10 to 75 weight percent of semicrystalline ethylene homopolymer. They observed the Bragg spacing for the E-EP domain thickness to significantly decrease with homopolymer addition. They accounted for the odd result of decreasing domain spacing with increasing amount of homopolymer as the result of relaxation of chain stretching in the EP block. In our experiments with much higher molecular weight diblock E/EP and triblock E/EP/E copolymers, which have been shown to be heterogeneous in the melt [17], we found that upon blending with up to 10% semicrystalline E homopolymer, the lamellar periodicity corresponding to the E/EP microphase separated domain did not change with increasing wt% of E. The lamellar periodicity associated with the semicrystalline E domain increased with increasing E wt%. This phenomenon is believed to occur due to the change in chain packing of the amorphous block to accommodate the added E homopolymer. Since weight fractions were small relative to the overall block sizes, overall block copolymer
domain thickness was not effected.

Novel results also include the orientation of these semicrystalline block copolymer blend morphologies perpendicular to the direction and normal to the plane of shear. The addition of homopolymer can again be seen to have no effect of the E/EP domain thickness in these oriented block copolymer blend systems. An increase of the crystal / amorphous spacing within the E block domain is observed with increasing homopolymer. The addition of E homopolymer decreased the permeability, in the perpendicular oriented samples. This is an expected result because E homopolymer addition should decrease the measured permeability values due to the increased volume fraction of the semicrystalline low permeability material.

**CONCLUSIONS**

This study successfully demonstrated that changes in morphology observed by SAXS in semicrystalline block copolymer blends can be directly related to corresponding observations in the measured gas transport properties. Control over gas transport properties was achieved through variations in processing conditions, which affect the microstructure orientation texture of the blend system, and solvent treatments, which create porous structures. Blending semicrystalline E homopolymer with E/EP/E triblock copolymers and subsequently orienting the blend system using a channel die, achieved a microstructure orientation texture normal to the direction and perpendicular to the plane of shear, as well as anisotropic gas transport properties. The addition of semicrystalline E homopolymer up to 10 weight percent in the E/EP/E triblock resulted in an increase in the lamellar spacing associated with the crystallization of the E chains, but no observed changes in the spacing associated with the microphase separation between the E and EP block copolymer nanodomains. Amorphous PI
homopolymer blending with an E/EP diblock and subsequent swelling in selective solvents created a porous structure, whose gas permeability could be altered by swelling the blend system to different extents. The semicrystalline block copolymer blend structure oriented perpendicular to the plane of shear presents the opportunity to create a high flux porous structure, while keeping the pores of nanometer dimensions, thus providing some selectivity and retaining mechanical strength. This research brought to light advances in the complex microstructure orientation textures exhibited by semicrystalline block copolymer blends, and their relation to membrane gas transport properties.

ACKNOWLEDGEMENTS

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REFERENCES


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<th>% E Homopolymer</th>
<th>D₁ (nm)</th>
<th>D₂ (nm)</th>
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<tr>
<td>0.0</td>
<td>39.3</td>
<td>13.1</td>
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<td>10.0</td>
<td>39.3</td>
<td>20.7</td>
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Table 1: Bragg spacing results of the isotropic unblended E/EP/E 30/40/30 triblock sample and the 10wt% E homopolymer blend.
<table>
<thead>
<tr>
<th>% E homopolymer</th>
<th>D$_1$ (nm)</th>
<th>D$_2$ (nm)</th>
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<tr>
<td>0.0</td>
<td>43.6</td>
<td>14.5</td>
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<td>2.5</td>
<td>43.6</td>
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<td>5.0</td>
<td>43.6</td>
<td>17.1</td>
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<td>10.0</td>
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<td>17.9</td>
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Table 2: Bragg spacing results for the normal to the plane of shear orientation unblended and E blend samples.
<table>
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<th>E/EP/E 30/40/30</th>
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<th>$P_{He}$</th>
<th>$P_{CO_2}/P_{He}$</th>
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<tr>
<td>0% E</td>
<td>39</td>
<td>31</td>
<td>1.2</td>
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<tr>
<td>10.0% E</td>
<td>5</td>
<td>8</td>
<td>0.6</td>
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Table 3: CO$_2$ and He permeability results for isotropic E/EP/E + E blends.
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<th>E/EP/E 30/40/30</th>
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<th>$P_{He}$</th>
<th>$P_{CO_2}/P_{He}$</th>
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<tr>
<td>0.0% E</td>
<td>43</td>
<td>15</td>
<td>2.8</td>
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<tr>
<td>10.0% E</td>
<td>15</td>
<td>12</td>
<td>1.2</td>
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Table 4: CO$_2$ and He permeability results for normal to the plane of shear oriented E/EP/E + E blends.
<table>
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<th>Solvent</th>
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<th>$P_{He}$</th>
<th>$P_{CO_2}/P_{He}$</th>
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<tr>
<td>Homopolymer</td>
<td>44</td>
<td>19</td>
<td>2.3</td>
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<td>47</td>
<td>24</td>
<td>1.9</td>
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<td>Blend Swollen in Heptane</td>
<td>61</td>
<td>30</td>
<td>2.0</td>
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<td>Blend Swollen in Xylene</td>
<td>72</td>
<td>56</td>
<td>1.2</td>
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Table 5: $CO_2$ and He permeability results for isotropic E/EP 100/100 blended with PI and exposed to xylene or heptane.
Figure 1: A representation of the channel die and the three principle deformation directions

Figure 2: (a) Unoriented SAXS patterns for the unblended triblock, (b) 10wt% E homopolymer blend, and (c) a comparison of the $IQ^2$ vs Q plots obtained for each sample.

Figure 3: The oriented SAXS patterns for the (a) 2.5 wt% E, (b) 5.0 wt% E, and (c) 10.0 wt% E triblock samples.

Figure 4: $IQ^2$ vs Q plots for an unblended, 2.5 wt% E, 5.0 wt% E, and 10.0 wt% E triblock blend samples. Graph is enlarged to highlight changes in the semicrystalline scattering peak.

Figure 5: Schematic of the relationship between permeation behavior and microstructure orientation texture in semicrystalline block copolymers.
GAS PERMEATION DIRECTION

Perpendicular

Random

Parallel

Transverse

High Permeability

Low Permeability

Kofinas, Fig. 5