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Phase Behavior of Binary Homopolymer/Diblock Blends: Temperature and Chain Length Dependence

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ABSTRACT: The phase behavior of binary blends of A homopolymer and symmetric AB diblock copolymer is studied within mean-field theory. The occurrence of lamellar, hexagonal, and body-centered-cubic phases is examined in the weak to intermediate segregation regime and over a wide range of homopolymer length relative to that of the diblock. This ratio is found to be a crucial parameter for the topology of the phase diagram. Several different classes of behavior are established, and comparisons are made with experiment.

Introduction

Systems containing block copolymers are of great interest because of the ability of the copolymer to bring about a variety of structured, ordered, or partially ordered morphologies. As shown by Leibler,[1] the phase diagram in mean-field theory of the one-component diblock system depends upon the fraction f of the copolymer that is of type A, and χN, the product of the dimensionless parameter χ, which measures the interaction between A and B monomers, and the total number of segments per chain, N. Copolymers for which f is approximately 0.5 form lamellae, while more asymmetric diblocks form cylinders that are arranged in a hexagonal array, or spheres in a body-centered-cubic lattice. Between lamellar and hexagonal phases, there is a narrow region in which the bicontinuous gyroid phase (space group $Ia\bar{3}d$) occurs at intermediate segregations. The order/disorder transition is continuous for the particular value $f = 0.5$, and first-order otherwise. The mean-field phase diagram for this system[2,3] has been calculated and is in general agreement with experiments.[4] Fluctuations affect the phase behavior mainly in the weak segregation limit. The isolated continuous transition is driven first-order, and transitions from the disordered phase, which in the absence of fluctuations occurred generally to the body-centered-cubic phase, now occur to the other phases as well.[5,6]

The behavior of AB diblock copolymers blended with A or B copolymer is more complicated than that of pure diblocks. For each homopolymer blended with the diblock, there are *two* additional independent variables, the volume fraction of the homopolymer and the chain length ratio $\alpha = N_A/N_{AB}$. These additional variables permit behaviors in the blend that are not encountered in the simpler system, such as macrophase separation and the swelling of microphase structure. In the extreme case, swelling continues indefinitely, giving rise to a complete unbinding transition in which the wave vector of the ordered phase approaches zero continuously with increasing homopolymer concentration.[7,8]

It is currently not possible to predict with reliability the exact phase diagram of a particular homopolymer/copolymer blend. But it is possible to calculate a representative set of phase diagrams to illuminate the general trends in the phase behavior and to provide a guide to experiment. We endeavor to do so here for the binary system of *symmetric* AB copolymer blended with A homopolymer. We are able to compare our phase diagrams with experiment as there have been numerous investigations of binary homopolymer/diblock blends.[9−25] Calculation of the behavior of the binary system also provides a valuable limit on the behavior of the ternary system of symmetric AB diblock and both A and B homopolymers, a system that we have investigated previously.[26,32]

We employ the standard Gaussian chain model[27] and solve the mean-field equations in Fourier space[2] in the grand-canonical ensemble.[7] All our calculations are for a diblock with A and B blocks of equal length (i.e., $f = 0.5$), but the relative chain length of the A homopolymer, $\alpha = N_A/N_{AB}$, is varied from $\alpha = 0.1$ to $\alpha = 1.5$. The incompatibility is varied from the order/disorder transition, $\chi N \approx 8$, to $\chi N = 17.5$. We consider only the three classical phases of lamellae (LAM), cylinders arranged on a hexagonal lattice (HEX), and spheres arranged on a body-centered-cubic lattice (BCC). Our calculations complement those of Matsen[7,8] and of Kielhorn and Muthukumar.[28] Matsen studied, within mean-field theory, not only the classical phases but also complex ones, such as the bicontinuous gyroid and double-diamond. He considered primarily how they were brought about by varying the architecture of the diblock, f, as well as the concentration of homopolymer. The greater numerical effort necessary for the satisfactory description of the complex phases restricted the range of χN in which they could be studied. They were found only in a narrow region between the lamellar and hexagonal phases, just as in the pure diblock system. In the present study, we focus on the evolution of the topology of the phase diagram over a wide range of homopolymer lengths α, and incompatibilities χN, and therefore consider only the three classical phases. Kielhorn and Muthukumar[28] go beyond mean-field theory and consider the effect of fluctuations within a self-consistent Hartree theory. As noted earlier, this brings about direct transitions between the disordered and hexagonal phases. However, these authors include only a single star of wave vectors to describe the ordered phases, which limits the reliability of their calculations to the weak segregation regime. Furthermore, it precludes the possibility of studying unbinding transitions,

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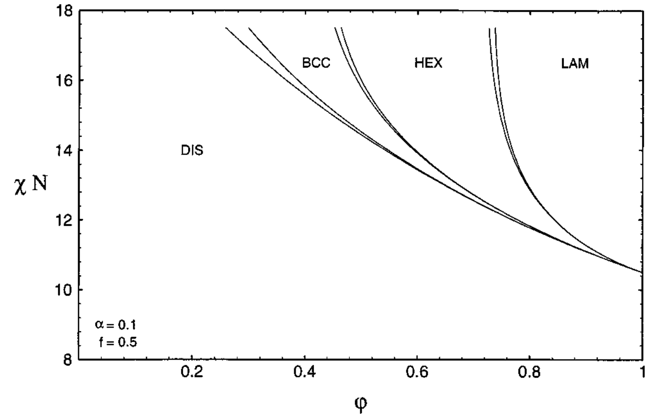

Figure 1. Phase diagram for a symmetric AB diblock of N monomers blended with a homopolymer. The copolymer volume fraction is denoted φ; the Flory–Huggins parameter χ. The ratio of the chain lengths $\alpha = N_A/N$ is $\alpha = 0.1$. The order/disorder transition for the pure diblock at $\chi N = 10.495$ is continuous; all other transitions are first-order. The lamellar phase is denoted LAM, the phase of hexagonally arranged cylinders HEX, and the phase of spherical micelles on a body-centered-cubic lattice by BCC. The disordered DIS phase is assumed homogeneous. Regions of two-phase coexistence are unlabeled. Notice how the order/disorder transition occurs at larger values of χN as homopolymer is added. This disordering effect is due to the large entropy of mixing of such short chains.

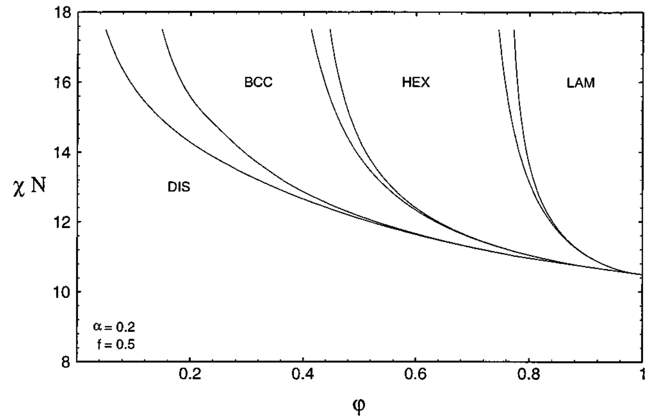

Figure 2. As in Figure 1, but with $\alpha = 0.2$. Notice that the homopolymer still has a disordering effect, but much less so than in Figure 1.

which can neither be characterized by a single length scale nor a small order parameter. By including a large number of wavevector stars, we are able to study these transitions, which comprise one of the most interesting features of the binary system. We also consider a much wider range of homopolymer size α, than do they.

Results

It is convenient to discuss the various systems in order of increasing value of the parameter $\alpha = N_A/N_{AB}$, the ratio of the index of polymerization of the homopolymer to that of the diblock. All diagrams are given in terms of the relative interaction strength χN and the diblock volume fraction φ.

Phase diagrams for blends containing homopolymers shorter than a quarter the length of the diblock (i.e., $\alpha < 0.25$) are shown in Figures 1 and 2. This value was shown previously[29] to be the threshold at which homopolymers have a disordering effect on any microstructure. We see clearly that the transition to an ordered structure occurs at higher values of χN as more homopolymer is added. This is due to the relatively large entropy of mixing that these short chain molecules gain by distributing themselves randomly throughout the system. A similar effect was also observed in ternary systems containing homopolymers of this length.[26] Since the homopolymers are much shorter than the diblock, they enter the brushes formed by the copolymer at the internal interfaces and bring about transitions to phases characterized by nonvanishing mean curvature of the interfaces.

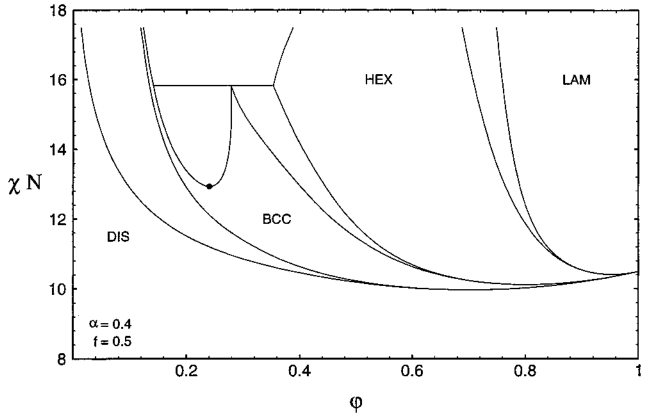

Figure 3. As in Figure 1, but with $\alpha = 0.4$. Notice the BCC/BCC phase coexistence for low copolymer content.

As the polymerization index of the homopolymer increases, $\alpha > 0.25$, several new features appear. First, the tendency of the homopolymer to disorder the microstructure by increasing the entropy of mixing now competes with its tendency to bring about *order* by relieving stress within the microstructure. The latter tendency dominates[29] for small homopolymer concentrations so that the value of χN at the order/disorder transition initially decreases as the homopolymer content increases, as shown in Figure 3 for $\alpha = 0.4$. The region of phase coexistence exhibits an azeotropic point at which the concentrations of the coexisting phases are equal.[30] Second, a region of coexistence appears between two body-centered cubic phases that differ in their homopolymer content and their lattice parameter. Phase coexistence between ordered phases differing in their repeat distances has been discussed previously,[31,32] but for lamellar phases only. The region of coexistence ends at a critical point. There is a line of such critical points within the approximate range $0.3 < \alpha < 0.9$. Presumably, this line terminates at critical end points. Certainly it seems from Figure 4 for $\alpha = 0.6$ that one is about to appear as the line of critical points encounters the plane of BCC–HEX coexistence.

Figure 4 also shows another new behavior characteristic of homopolymers of about this length and longer, $0.6 < \alpha < 1.0$: the complete unbinding of the hexagonal phase to the disordered state at *large* homopolymer concentration.[7,8] As homopolymer is added, the periodicity of the ordered phase increases without limit, leading to the appearance of the disordered phase without a region of phase coexistence being encountered. This is completely analogous to the behavior of the Abrikosov flux lattice[33] in a type II superconductor as the magnetic field approaches H_{c1} from above.

Although the hexagonal phase undergoes an unbinding transition, as does the lamellar phase at a larger value of α (see below), the body-centered-cubic phase does not. For $\alpha \geq 0.6$, we find the BCC phase to be stable for weak segregations, but unstable to the

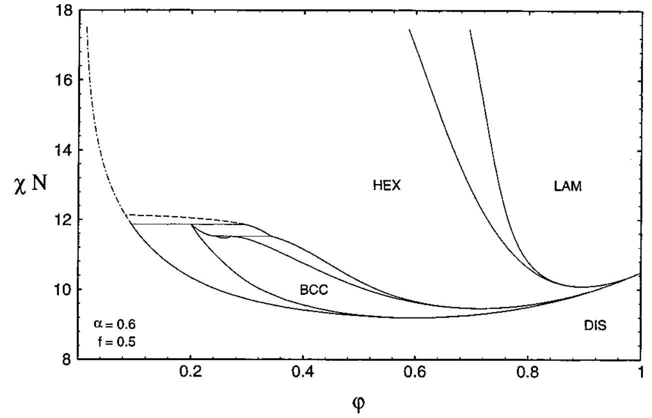

Figure 4. As in Figure 1, but with $\alpha = 0.6$. The unbinding line, at which the periodicity of the hexagonal phase becomes infinite is indicated by a dot–dashed line. Notice that the BCC/BCC phase coexistence region has shrunk as compared to Figure 3. Dashes indicate extrapolated phase boundaries.

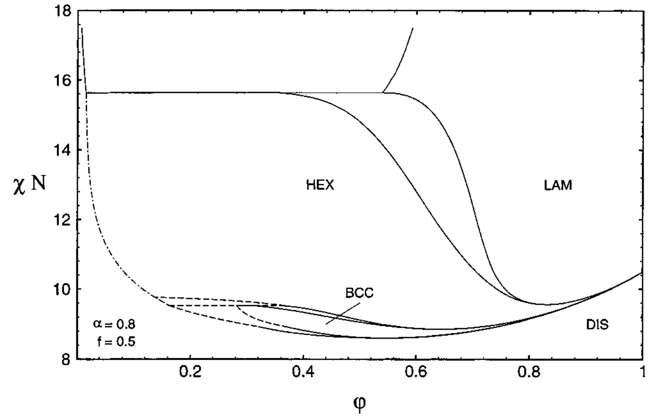

Figure 5. As in Figure 1, but with $\alpha = 0.8$. Notice the large two-phase region between the lamellar phase and an almost pure homopolymer phase. The BCC/BCC phase coexistence has vanished. Notation as in Figure 4.

hexagonal phase at larger χN. This can easily be understood. The relatively short homopolymers enter the spherical micelles making up the body-centered-cubic lattice and relieve the stress of stretching experienced by the symmetric diblocks that straddle the curved interface of the spheres. However, this increases the number of unfavorable AB contacts, an effect that becomes more costly as χN is increased. The homopolymers also become less efficient in reducing stress as they become longer. Consequently, the transition to the hexagonal phase occurs at lower values of χN as the homopolymer length increases.

For $\alpha \geq 0.8$ (Figure 5) the homopolymers phase separate from the lamellar microphase at large χN and within the range that we have studied. The line of complete unbinding transitions of the hexagonal phase, which exists at lesser segregations, ends at a form of critical end point at which it encounters the planes of first-order transitions separating the lamellar phase from the disordered and hexagonal phases. The value of χN where this occurs is the largest at which microphase unbinding can occur. As α increases, the region of coexistence between lamellar and disordered phases extends toward smaller values of χN, indicating that the ability of the homopolymer to swell the mesophase decreases. This is due to the considerable loss of configurational entropy suffered by homopolymers that are comparable in length to the microdomain spacing on being confined to the microstructure.

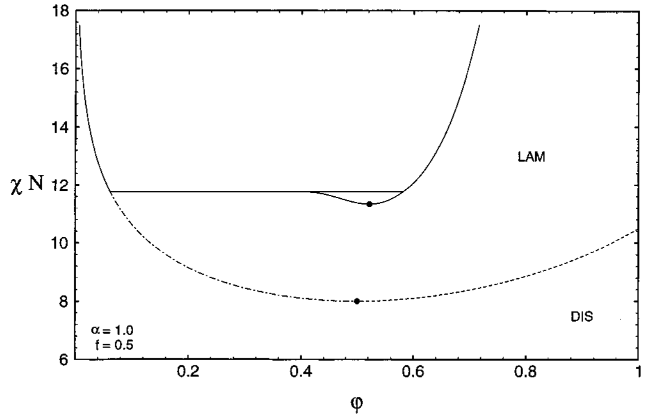

Figure 6. Binary phase diagram with $\alpha = 1.0$. The ordered phases, except for the lamellar phase, have disappeared; the order/disorder transition is continuous for $\varphi > 0.5$ (short dashed line). The Lifshitz point at $\varphi = 0.5$, $\chi N = 8.0$ is indicated by a dot, as is the critical point of the preunbinding region. Notation as in Figure 4. After ref 32.

As the length of the homopolymer approaches that of the diblock, all ordered phases except the lamellar one disappear. In Figure 6, which shows the phase diagram for $\alpha = 1.0$, only the lamellar phase remains, and it undergoes an unbinding transition. The unbinding line, at which the lamellar wavelength diverges, extends from the first-order unbinding transition to the Lifshitz point at $\chi N = 8.0$, $\varphi = 0.5$.[29] That the unbinding transition is first-order is clearly indicated by the existence of a preunbinding region in which two lamellar phases of different wavelength coexist. The preunbinding region ends at a critical point.[31] The way in which the other ordered phases disappear in the narrow region $0.9 < \alpha < 1.0$ is presumably as follows. As α increases beyond 0.9, a narrow channel of lamellar phase opens up between the lamellar/disordered coexistence and the hexagonal region. This lamellar phase undergoes a complete unbinding as homopolymer is added. As α approaches 1.0, this channel widens, while the regions of hexagonal and body-centered-cubic phases shrink and are shifted toward lower values of χN. At $\alpha = 1.0$ they have collapsed completely, and the transition from the remaining lamellar phase to the disordered phase is continuous.

For values of α greater than 1.0, the Lifshitz point is pre-empted by a region of disorder/disorder phase coexistence that ends in an ordinary (Ising) critical point. The order/disorder transition at high copolymer concentration is presumably still continuous within mean-field theory.[32] A narrow channel of lamellar unbinding remains for $\alpha = 1.05$ (Figure 7) between the disorder/disorder phase coexistence at weak segregation and the lamellar/disorder coexistence at stronger segregation.

The channel of complete lamellar unbinding closes rapidly with increasing α. For $\alpha = 1.1$, the lamellar phase is bound at all temperatures (Figure 8), although the signature of the nearby unbinding transition is still visible in a nonmonotonic behavior of the composition and periodicity of the lamellar phase in coexistence with the disordered one. Such nonmonotonic behavior of the composition was also observed by Matsen.[8] As α continues to increase, this effect diminishes. At $\alpha = 1.5$ (Figure 9) the periodicity of the lamellar phase coexist-

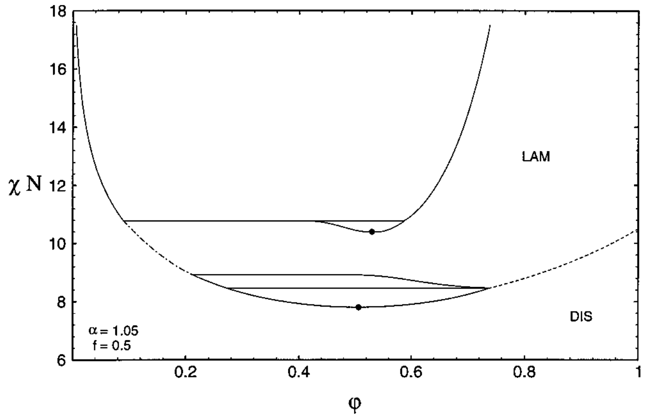

Figure 7. As in Figure 6, but with α = 1.05. The order/disorder transition is still continuous for high copolymer content. In the weak segregation limit, there is a phase coexistence between different disordered phases. Notice the narrow channel of lamellar unbinding.

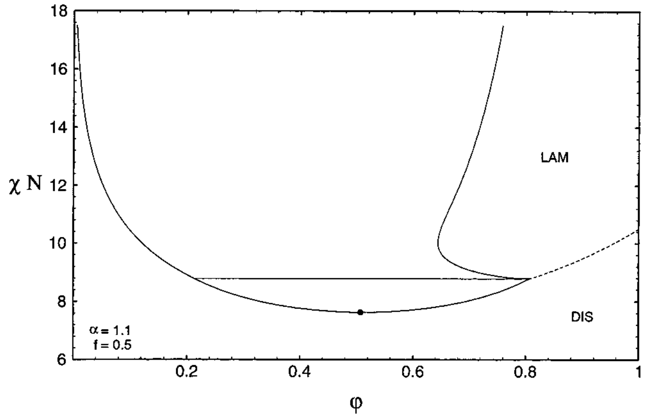

Figure 8. As in Figure 6, but with α = 1.1. The channel of lamellar unbinding has closed, although its signature is still visible in the strongly nonmonotonic behavior of the phase boundary.

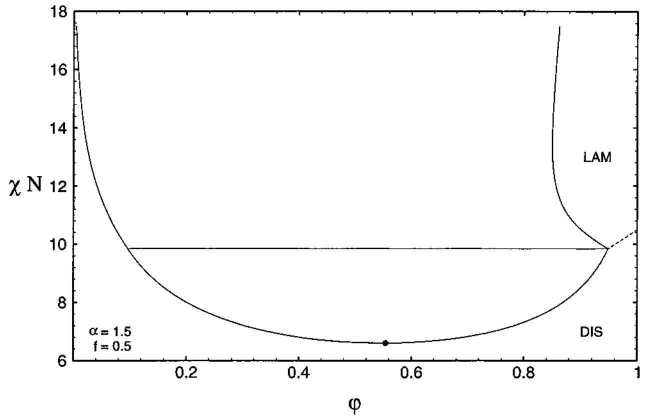

Figure 9. As in Figure 6, but with α = 1.5. Only the slightest trace remains of the unbinding transition in the nonmonotonic behavior of the lamellar phase boundary. The maximum wavelength to which the lamellar phase can be swollen before macrophase separation occurs increases monotonically with χN.

ing with the disordered phase is monotonic in χN, although there is still a trace of the unbinding transition in the nonmonotonic behavior of the composition.

Discussion

There have been a large number of experiments examining the behavior of diblocks blended with homopolymer.[9−25] Here we shall focus on those that specifically address microphase behavior in systems containing a symmetric ($f = 0.5$) diblock.[10−12,14−19,23−25]

One serious difficulty encountered in comparing calculated phase diagrams with those obtained from experiments is the proper identification of the Flory–Huggins parameter χ. Various values are reported in the literature.[34,35] Hashimoto and co-workers find, for a blend of polystyrene-*block*-polyisoprene with polystyrene homopolymer, a very strong dependence of this parameter on φ *and* on α, in addition to the expected dependence upon temperature.[34] Further, the variation with α is not monotonic. Therefore we will not attempt to identify a specific value of χN for a given experiment but simply distinguish between strong, intermediate, and weak segregation regimes. We also compare our results with other calculations.

Experiments on very short homopolymers ($\alpha < 0.25$) show good agreement with our calculations for the most part. Results of two different groups[10,12,17,19] on homopolymers with $\alpha \approx 0.07$ and $\alpha \approx 0.125$ find the ordered phases to be stable at compositions for which they are stable in our calculations at intermediate segregation (i.e., χN in the range 15–20). Particularly noteworthy is that the order exists for rather large dilution (up to $\varphi = 0.2$).[12] For $\varphi < 0.2$, however, the body-centered-cubic crystal melts and is replaced by a disordered micellar solution.[19] As expected, our result for $\alpha = 0.1$ is very similar to that of Kielhorn and Muthukumar[28] for the same value of α and $N = 10^6$. The major difference, as noted earlier, is that the fluctuations they include permit direct transitions from the disordered phase to the lamellar phase over a range of homopolymer concentrations, and to the hexagonal phase as well. Fluctuations also change the critical point at $\varphi = 1.0$ to a first-order transition. The stability region of the BCC phase is reduced by fluctuations, so that it occurs at higher segregation and a lower value of φ than in mean-field theory.

Our results for systems with α in the range between 0.25 and 0.5 can be compared with experiments employing homopolymers with $\alpha \approx 0.25$ and $\alpha \approx 0.3$.[10,17,19] Again, ordered phases are observed at those compositions at which we find them to be stable. Winey et al.[18] also report a complex phase, allegedly of ordered bicontinuous double diamond symmetry (OBDD, space group $Pn3m$) in the composition range $\varphi = 0.66–0.7$. Since we consider only the classical phases, we cannot comment on the stability of the OBDD phase in this region as compared to the gyroid phase (space group $Ia\bar{3}d$). The stability region of the ordered phases seems to be smaller for $\alpha > 0.25$ than for blends containing shorter homopolymers. For example, one finds a disordered micellar fluid,[19] presumably of spherical micelles, for concentrations of copolymer as great as $\varphi = 0.4$. This would indicate that the entire region of BCC phase in Figure 3 has been destroyed by fluctuations. A similar result was found in a Monte Carlo simulation of short chain surfactants with $\alpha = 0.25$.[36] The entire region of BCC phase was replaced by a disordered phase, while the LAM and HEX phases were found at essentially the same compositions as we find them for that value of α. A different phenomenon was calculated by Kielhorn and Muthukumar[28] for $\alpha = 0.5$. They found a large region of HEX/DIS phase coexistence in the weak segregation limit, $\chi N < 13.0$, which is replaced by LAM/DIS coexistence for $13.0 < \chi N$. This result, in accord with neither experiment nor the full mean-field theory used here, probably signifies the breakdown of their description of the ordered phase by the first harmonic. Such a description prevents them from finding strongly swollen phases, which are characterized by two very different length scales.

For homopolymer lengths approaching that of the diblock ($0.5 < \alpha < 1.0$), the experimental situation seems less clear. Lamellar phases are usually observed only for relatively large copolymer concentrations: $\varphi > 0.5$, for $\alpha \approx 0.5$[10,17,23,24] or $\varphi > 0.7$ for $\alpha \approx 0.7$.[19] As in surfactant systems, other ordered phases do not occupy large regions of the phase diagram,[23,24] and in particular do not exist for high homopolymer concentration. Instead, the blend forms a disordered micellar fluid phase for φ extending to 0.7[19] or 0.5.[24] In contrast, Hashimoto and co-workers[15] studying a system with $\alpha \approx 0.6$, observe a phase of strongly undulating layers, but with clear lamellar order, for a copolymer concentration as low as $\varphi = 0.2$! This is reminiscent of systems of short-chain amphiphiles,[37] in which undulations stabilize the lamellar phase[38] and allow it to be swollen to an extreme degree. The transition to the regime of strongly swollen layers observed by Hashimoto and co-workers[15] should be achievable by varying the effective interaction parameter χN.[31] It would be very interesting to test this prediction.

Comparison of experimental results with our calculations leads to the conclusion that large-scale form fluctuations, which are ignored in our approach, are important for blends with $0.5 < \alpha < 1.0$. The body-centered-cubic phase is probably eliminated entirely, as is the strongly swollen hexagonal phase. Both are replaced by disordered micellar phases. For some, probably narrow, range of external parameters, an undulation-stabilized lamellar phase extends to low copolymer concentrations. It would be very interesting to see if the strongly swollen hexagonal phase could be stabilized by some external field just as the Abrikosov flux phase is stabilized by a magnetic field. Perhaps this could be accomplished by using a copolymer with blocks of very different polarizabilities, in which case the hexagonal phase with the block of larger polarizability in the core might be stabilized by an electric field.

Finally, for blends with $\alpha \geq 1.0$, experiments find a lamellar phase[14,16] at large copolymer volume fractions and phase separation for $\varphi < 0.85$,[23] which is in general agreement with our calculations. This behavior is clearly indicated by the variation of the lamellar wavelength with homopolymer concentration. Initially, the lamellar spacing grows linearly with homopolymer concentration, but then it levels off, indicating that additional homopolymer does not enter the microstructure but separates from it.[23] A temperature/composition phase diagram for a system with $\alpha \approx 1.0$ has resulted from one experiment.[25] The lamellar phase is stable for $\varphi > 0.8$ at all temperatures. At lower diblock concentrations, the lamellar phase becomes unstable at high temperatures (weak segregation) to a disordered micellar fluid, while for low temperatures (strong segregation), it coexists with an almost pure homopolymer phase. Interestingly, between these two regimes, the lamellar phase appears to extend to a very low copolymer concentration ($\varphi = 0.3$). Although the identification of the lamellar phase is not entirely unambiguous from the micrographs provided, the similarity to the behavior shown in Figures 7 and 8 is striking. A closer investigation of this effect, for α very near to unity, would be most interesting. Particular care has to be taken to avoid effects of metastability. For example, Hashimoto and co-workers[14] report a strong dependence of the domain size on the speed of the solvent-casting process.

As we have tried to emphasize, there remain several interesting experiments to be conducted on these systems, particularly ones exploring the systematics of highly swollen lamellar phases and the possible occurrence, or stabilization of, highly swollen hexagonal phases. We hope our study will serve to stimulate such investigations.

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