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The use of force modulation microscopy to investigate block copolymer morphology

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Force modulation microscopy (FMM) is used to characterize the external surface and internal fracture surface morphologies of three different block copolymer samples. A roll-cast poly(styrene-butadiene-styrene) triblock copolymer film, spin-coated poly(styrene-*b*-methyl methacrylate) thin films, and an ultrathin poly(styrene-*b*-hexyl isocyanate) rod–coil block copolymer film were investigated. For each sample, height and elasticity images were obtained for the same areas allowing direct comparison. The elasticity images obtained using force modulation microscopy were independent of surface roughness and found to exhibit better contrast and spatial resolution of the respective block copolymer domains than the height images. The lateral resolution of the elasticity images was sufficient to show microphase separated domains having length scales as small as about 10 nm. The poly(styrene-*b*-methyl methacrylate) samples demonstrate that FMM can even be successfully used to study block copolymers in which both blocks are glassy under the conditions of measurement.

1. Introduction

AB block copolymers microphase separate to form ordered structures due to the mutual repulsion of the dissimilar blocks and the constraints imposed by the connectivity of the A and B blocks. Traditionally, transmission electron microscopy (TEM) has been used to study the bulk and near-surface morphologies (in cross-section) of block polymers. For TEM, however, samples normally must be preferentially stained to achieve sufficient electron density contrast between the A and B blocks. The sample preparation needed for TEM can also be quite involved due to the microtomy necessary to achieve thin sections [1]. In addition, many block copolymers can experience significant beam damage which can result in image artefacts. The relatively new technique of low voltage high resolution scanning electron microscopy (LVHRSEM), which requires much less sample preparation than TEM, has also been used to study the near-surface domain structure of block copolymers [2]. While LVHRSEM is a promising tool for directly studying the surface morphology of block copolymers, samples must still be preferentially stained. In addition, because LVHRSEM utilizes an electron beam, samples can only be imaged under high vacuum and beam damage of samples can occur.

More recently, contact mode atomic force microscopy (AFM), which has excellent lateral and height resolution, was used to study the morphology of microphase separated block copolymers at free surfaces [3, 4, 5]. In the studies by Schwark *et al.* [3] and Annis *et al.* [4], solution cast samples of various poly(styrene-*b*-butadiene), P(S-*b*-B), diblock copolymers were investigated. In particular, diblock copolymers which form the lamellar and sphere (PB) morphologies in the bulk were studied. In all the samples, the lower surface tension PB block was found to form a thin (approximately 5 nm) layer which covered the entire free surface of the sample. For samples which exhibited regions of lamellae perpendicular or nearly perpendicular to the free surface, the presence of the PB surface layer resulted in a periodic surface morphology in which the peaks and valleys corresponded to the PS and PB domains, respectively. The peak to valley height as measured by AFM was found to vary between 1.5 and 4 nm dependent on the orientation of the lamellae with respect to the surface. In contrast to these studies, Collin *et al.* [5] studied the evolution of the surface morphology of spin coated lamellar forming poly(styrene-*b*-*n*-butyl methacrylate), P(S-*b*-BMA), diblock copolymer films as a function of annealing time. Previous studies have shown that in these films, the microphase separated lamellae are oriented parallel to the substrate after annealing. As a result, if the initial film thickness is not equal to an integer multiple of the lamellar period (for symmetric boundary conditions), upon annealing, the films form islands or holes having heights equal to the lamellar period. By measuring the time-dependent root-mean-square (r.m.s.) roughness of the films with AFM, Collin and co-workers were able to monitor the kinetics of the ordering process.

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AFM has many advantages over TEM and LVHRSEM. Unstained samples can be imaged under ambient conditions with virtually no preparation. In addition, under proper operating conditions, the force (a few nNs) exerted by the cantilever tip is not great enough to cause permanent plastic deformation of the sample and stable imaging of the same area can be performed for hours. However, with the use of stiff cantilevers and large contact forces, deliberate deformation of the sample can be achieved through ploughing or nanoindentation of the sample surface [6]. Contact mode AFM, however, does have drawbacks. Contact mode can only be used to study relatively smooth block copolymer samples which exhibit height variations commensurate with the morphology of interest. Height variations in block copolymer samples can arise from two sources. The thermodynamics of microphase separation in the presence of a free surface can result in natural variations in the topography which are indicative of the underlying near-surface morphology, as demonstrated in references 3–5. For samples which have been fractured to expose the internal microphase separated morphology, differences in the mechanical properties of the blocks can sometimes give rise to an effective variation in height along the crack plane during deformation which mirrors the block copolymer morphology. In each case, the height variations are small, on the order of 5 nm. As a result, any significant roughness present in the sample with a periodicity similar to the domain spacing of the block copolymer can easily obscure the morphology. As well, there is no *a priori* connection between height features and chemical identity of the blocks.

In recent years, force modulation microscopy (FMM), a variation of contact mode AFM, has been developed [7]. FMM relies on differences in elasticity across the sample as the contrast mechanism rather than height differences. FMM has already been successfully demonstrated on mixed Langmuir–Blodgett films, composites, and polymer blends [7–13] which had structures ranging from tens of nanometres to several μm. In contrast, microphase separated block copolymers typically have domain sizes of 10–50 nm. FMM is ideally suited to studying block copolymers because of its unprecedented lateral resolution and sensitivity to elasticity differences. To the best of our knowledge, block copolymer have not been studied before using this technique. In this paper, the technique of FMM is successfully used to reveal the morphology of actual block copolymer samples. These studies show that FMM, which is a non-destructive technique requiring little sample preparation, can be used to study the near-surface morphology of block copolymers with resolutions equal to or better than those obtainable from TEM or LVHRSEM studies.

2. Principles and experiments

In conventional contact mode AFM [14], a sharp tip (radius of curvature of ~10–20 nm) mounted on a flexible cantilever is brought into contact with the sample. The sample which is mounted on a piezoelectric tube is then scanned at a certain rate in a xy raster scan. During the scan, the force between the tip and the sample (or cantilever deflection) is kept constant through a feedback loop which continuously monitors the deflection (input to the feedback loop) of the cantilever (a laser beam is reflected off the back of the cantilever into a segmented photodiode) and adjusts the height of the sample accordingly by applying a z motion (output of the feedback loop) to the piezoelectric tube. These z height adjustments along with their xy coordinates are used to form a topographic image of the sample. In recent years, tapping mode AFM, a variation of contact mode AFM has been developed [15]. In tapping mode AFM, the cantilever is oscillated near its resonant frequency by a small piezoelectric bimorph and has a characteristic r.m.s. amplitude of oscillation. In contrast to contact mode AFM, the tip is only in contact with the sample at the lowest point of its oscillation which reduces sample damage and minimizes the effect of lateral forces on the tip. As the oscillating tip is scanned along the sample, the feedback loop continually adjusts the z position of the piezoelectric tube so that the cantilever maintains a constant r.m.s. amplitude (typically 75 to 85% of the maximum r.m.s. amplitude). The height adjustments along with their xy coordinates are used to form the topographic image of the sample just as in contact mode AFM. In FMM, a sinusoidal modulation, whose amplitude is ~20 nm, is applied to either the sample position [7] or the cantilever position [16] while the tip is scanned across the sample surface. The applied modulation of the tip or sample position results in a modulation of the applied contact force and of the cantilever deflection which is detected by the segmented photodiode. The average cantilever deflection is now used as the input signal into the feedback loop as in conventional contact mode AFM to maintain a constant average applied force. The r.m.s. amplitude of the cantilever deflection, however, can be used to obtain information on the mechanical properties of the sample. For a given amplitude modulation, the resulting r.m.s. cantilever deflection for a soft material will be less than that for a hard material. As a result, the measured r.m.s. amplitude at each point in the scan along with the corresponding xy coordinates can be used to obtain an image of the surface in which contrast is achieved through relative differences in elasticity across the sample surface.

The particular method of FMM used in this study was slightly different from the one described above. A Digital Instruments, Inc. Nanoscope III controller and Multimode AFM was operated in tapping mode using the interleaved mode. When the interleaved mode is used, the piezoelectric scanner performs twice as many scan lines for a given area, displaying separate images for the odd and even numbered scan lines. The scan conditions and feedback parameters, however, can be adjusted independently for the odd (main) and even numbered (interleaved) scan lines. As a result, different sample information can be displayed simultaneously such as height and elasticity. The main scan was used to display the height information obtained using tapping mode AFM (see Fig. 1(a)). To obtain the force modulation image, a feature called lift mode was employed during the interleaved scan. In lift mode, the feedback loop for the interleaved scan line is disabled and the oscillating tip tracks the surface at a user specified distance above or below the topography acquired during the previous main scan line (see Fig. 1(b)). Modulation of the cantilever position is achieved by oscillating the bimorph at its resonant frequency. The resulting r.m.s. deflection of the cantilever is obtained from the normalized photodiode difference signal, (U − L)/(U + L), which is offset to zero, rectified, and low-pass filtered (U = upper detector, L = lower detector). The Nanoscope III software converts this r.m.s. deflection signal into a grey scale value where stiffer regions, which result in a larger r.m.s. cantilever deflection, appear as darker areas. Operating the interleaved force modulation scan using lift mode has the key advantage of decoupling the height from the elasticity information, which is not possible with traditional force modulation techniques.

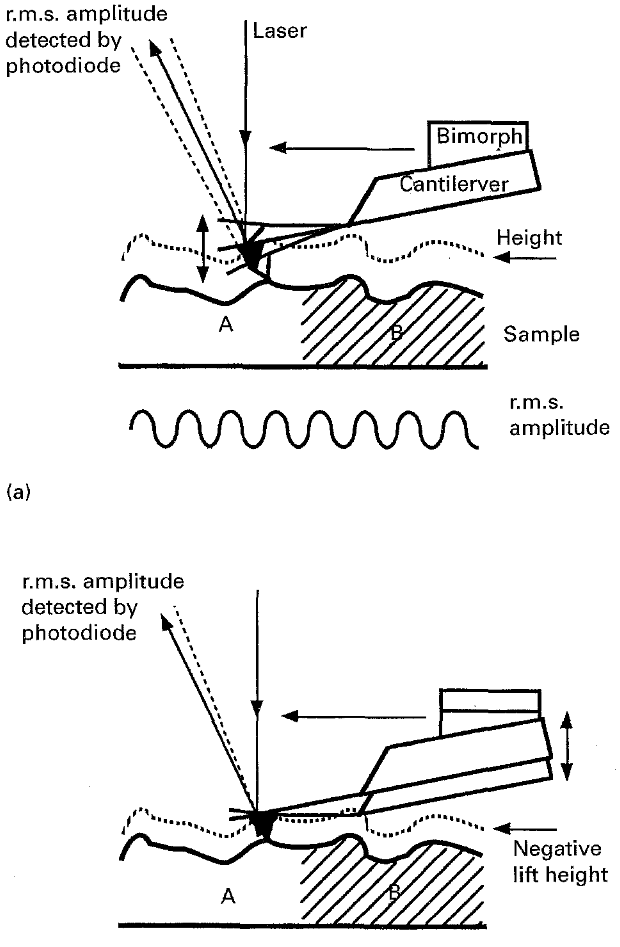


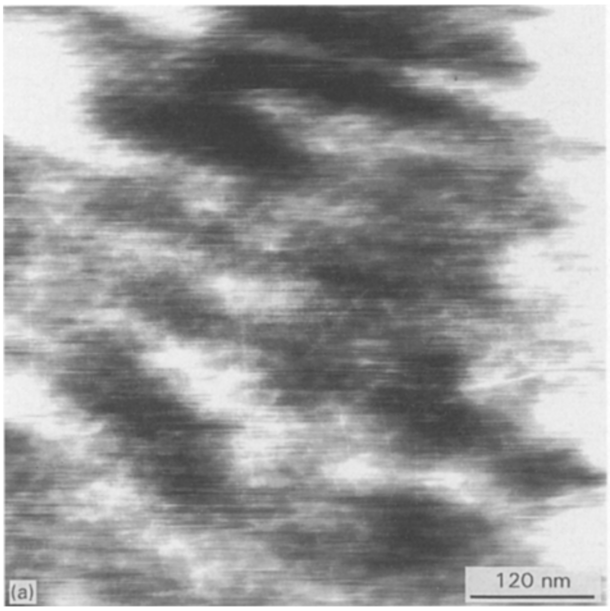
Figure 1 (a) Schematic diagram illustrating how the height images are acquired using tapping mode atomic force microscopy during the main scan. In tapping mode, the cantilever is oscillated at its resonant frequency of ~70 kHz while the feedback loop keeps the r.m.s. amplitude constant. (b) Schematic diagram illustrating how the elasticity images are acquired in the interleaved scan using FMM with negative lift mode. During the interleaved scan, the cantilever is oscillated at the bimorph resonant frequency of ~8 kHz. The detected r.m.s. amplitude of the cantilever is a measure of the local surface elasticity of the sample. In the diagram, material B is stiffer and results in a greater r.m.s. amplitude.

The FMM was done under ambient conditions using 226 μm long etched silicon cantilever substrates having spring constants ranging from 1.7 to 4.3 N m^{-1}. To obtain the height and force modulation images, the cantilever was oscillated at ~70 kHz near its resonant frequency and the bimorph at ~8 kHz near its resonant frequency. During the interleaved force modulation scans, negative lifts ranging from − 12 to − 20 nm were employed. Under these conditions, the average contact force exerted on the sample by the tip was approximately 15 nN. Height and force modulation images ranging from $1 \times 1\ \mu m^2$ to $2.5 \times 2.5\ \mu m^2$ in size were obtained at scan rates per line of 1–2 Hz.

Three model block copolymer samples were investigated in this study. The first sample was an unannealed poly(styrene-butadiene-styrene), P(S-B-S), triblock copolymer film fabricated from a 40 wt % toluene solution using the "roll-casting" technique [17, 18]. The cylinder-forming triblock copolymer was synthesized by Dexco Polymers and had a total molecular weight of 73 400 g mol^{-1} and a PS fraction of 29.2%. The roll-casting process yields nearly single crystal films in which the hexagonally packed PS cylinders are macroscopically oriented in the flow direction. The roll-cast samples were cut perpendicular to the oriented cylinders with a razor blade prior to imaging to expose a fresh surface of the internal morphology. The second sample consisted of 290 nm thick unannealed and annealed poly(styrene-*b*-methyl methacrylate), P(S-*b*-MMA), diblock copolymer thin films spin-coated from a 6.2 wt % solution in toluene at 5000 r.p.m. onto glass cover slips. For annealing, samples were placed in a vacuum oven at 170 °C for five days. The lamellar-forming P(S-*b*-MMA) diblock copolymer was synthesized by Polymer Labs and had a total molecular weight of 107 800 g mol^{-1} and a PS fraction of 47.3%. The third sample was an ultrathin film of a poly(styrene-*b*-hexyl isocyanate), P(S-*b*-HIC), rod–coil diblock copolymer cast from a dilute 0.05 wt % solution in toluene onto a carbon coated mica sheet. The synthesis and morphology of this rod–coil diblock copolymer have been discussed previously [19].

3. Results and discussion

The typical lateral resolution achievable with FMM on block copolymer samples is shown (see Fig. 2) in the $0.6 \times 0.6\ \mu m^2$ height and force modulation images obtained for the unstained P(S-B-S) roll-cast samples. The left and right images, Fig. 2(a and b), correspond to the height image obtained with tapping mode AFM and to the force modulation image obtained from the interleaved scan using negative lift, respectively. The particular triblock copolymer studied is known to form a near-single-crystal morphology consisting of PS cylinders in a PB matrix from previous cross-sectional TEM [20] and small-angle X-ray scattering (SAXS) [21] experiments. Fig. 2(a) shows that the exposed fresh cut surface of the roll-cast sample is



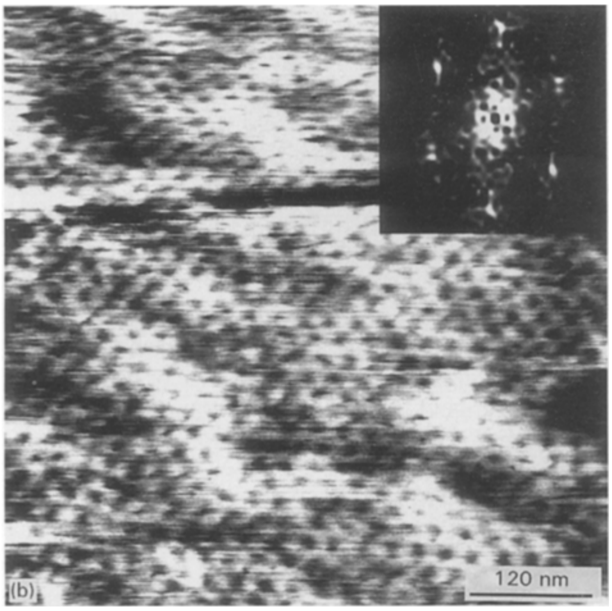


Figure 2 (a) Height image of the unannealed P(S-B-S) roll-cast film cut perpendicular to the PS cylinder axis. Faint indications of the PS cylinders are visible along with overall height variations on the surface. (b) Elasticity image for the same sample. The two dimensional packing of the PS cylinders is clearly visible. The inset shows the digital FFT of a smaller area which confirms the hexagonal packing of the cylinders.

relatively rough and has height variations of ~16 nm over the area of the scan. A closer inspection, however, reveals the presence of a faint periodic hexagonal lattice of PS cylinders which are seen to protrude above the surface of the film by about 1.5–3 nm. The different mechanical properties of PS and PB most likely give rise to this topography when the sample was cut with the razor blade. In comparison to the height image, the regular two dimensional array of cylinders is more easily recognizable in the elasticity image Fig. 2(b), due to the large stiffness contrast between PS and PB. As expected, the glassy PS cylinders appear dark in Fig. 2(b) indicating that they are stiffer than the surrounding rubbery PB matrix. The inset of Fig. 2(b) is a digital fast Fourier transform (FFT) of a $0.3 \times 0.3\,\mu\mathrm{m}^2$ area within the image and confirms the hexagonal packing of the PS cylinders. In particular, the FFT is a hexagonal pattern which has been stretched in the vertical direction. In unannealed roll-cast films, asymmetry in the cylinder morphology has been shown to be a direct result of the roll-casting process. Additional asymmetry can also result from either plastic deformation experienced during cutting and/or a fracture surface not exactly perpendicular to the cylinder axis. The 23.3 ± 1.1 nm *d*-spacing obtained from the smallest Bragg peak agrees well with the 24 nm *d*-spacing measured with SAXS. In addition, the measured average PS cylinder diameter of 13 nm is the same within error to the cylinder diameter obtained from cross-sectional TEM measurements and that calculated from simple volume fraction considerations assuming the 24 nm *d*-spacing. The roll-cast sample demonstrates that FMM can be used to study the internal morphologies of traditional coil–coil block copolymers in which one block is glassy and one is rubbery. In addition, the method of FMM used in this study successfully decouples topographic information from elasticity information.

The height and force modulation images shown in Fig. 2 were obtained less than ~10 minutes after the internal surface of the roll-cast samples were exposed to the air using the razor blade. Both the elasticity contrast and the height contrast arising from the oriented PS cylinders were found to disappear after prolonged imaging with FMM. Images of previously unscanned areas on the same sample also showed an absence of height or elasticity contrast which ruled out the possibility of sample damage as a source of the loss in contrast. PB has a lower surface tension with the air than PS. Consequently, the evolution of the fracture surface with time indicates a migration of the PB block over the surface which covered the exposed PS domains and obscured the presence of the underlying morphology. The migration of the PB block is thermodynamically driven and reduces the free energy of the higher energy PS–air surfaces [3]. The process is further facilitated by the fact that PB is rubbery at room temperature and is the majority component. Typically, the total loss of elasticity contrast between the PS cylinders and the PB matrix took place within 30 minutes after cutting. Although not carried out in this study, the technique of FMM is ideally suited to studying migration phenomena of this type.

While FMM is ideally suited to studying glassy/rubbery block copolymer morphology, the technique is also able to detect much smaller elasticity differences. Height and force modulation images of the as spun P(S-*b*-MMA) thin film are shown in Fig. 3 for a scan size of $1 \times 1\,\mu\mathrm{m}^2$. Both images reveal a chaotic bicontinuous morphology consisting of interconnected PS and PMMA channels which results from the rapid solvent evaporation caused by the spin coating process. The two images, however, are significantly different in a number of ways. The height image shows both a gradual variation in the height of the P(S-*b*-MMA) film, which causes some areas of the scan to appear brighter and others darker, and ~4.5 nm pseudo-periodic height variations due to the microphase

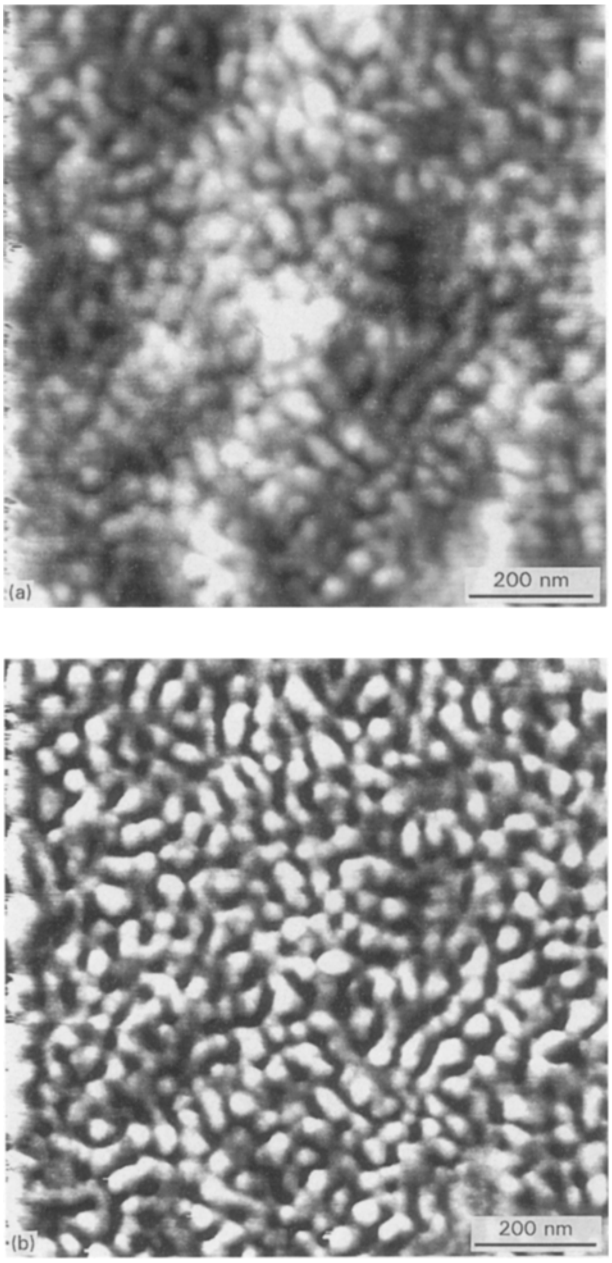


Figure 3 (a) Height image of the unannealed spin coated P(S-*b*-MMA) thin film which shows both surface roughness and height variations due to the microphase separated morphology. (b) Elasticity image of the same area. The contrast arises solely from the differences in stiffness between PS and PMMA.

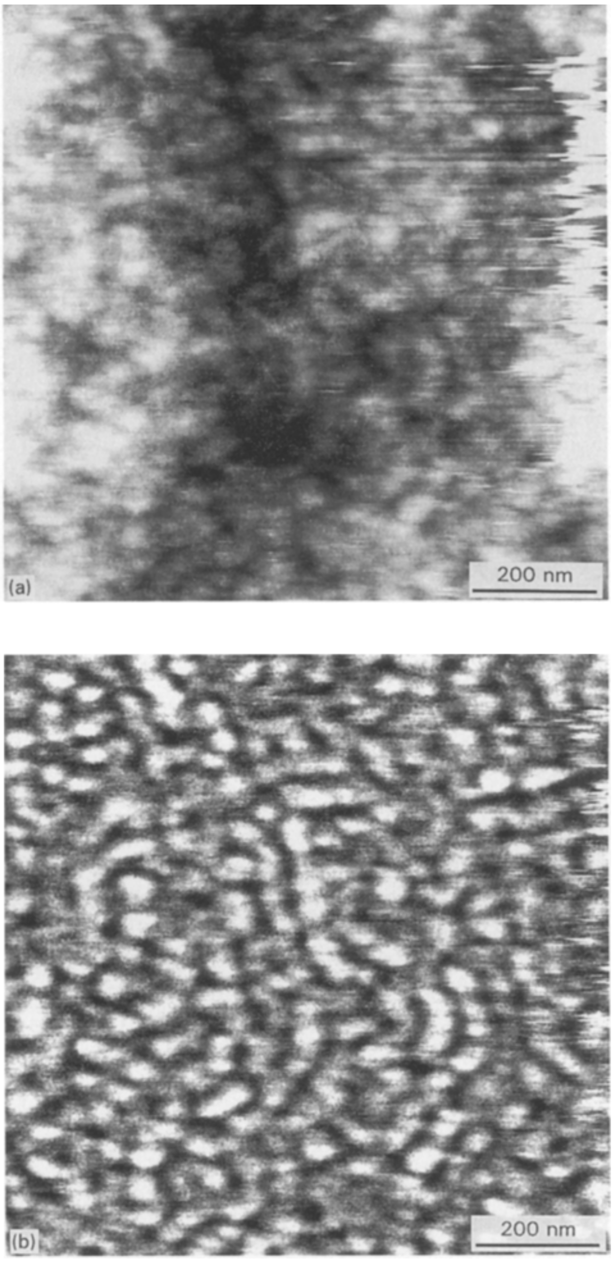


Figure 4 (a) Height image of the annealed spin coated P(S-*b*-MMA) thin film which mainly shows the presence of surface roughness. Surface roughness and migration of the PS have obscured the block copolymer morphology. (b) Elasticity image for the same sample. Although the elasticity contrast is 1/3 that of the unannealed sample, the short-range lamellae are still visible.

separated morphology which have an approximate domain size of 47 nm. In contrast, only the variation in elasticity across the sample resulting from the relative differences in the stiffness of the microphase separated PS and PMMA domains is apparent in the elasticity image. Although under ambient conditions and at the 8 kHz frequency of loading used in this study both PMMA and PS are glassy polymers, PMMA has been shown in a number of studies [22–25] to be stiffer than PS by a factor ranging from 1.1 to 1.5. Consequently, the dark areas in the elasticity image and the lower areas in the height image correspond to the stiffer PMMA domains.

Fig. 4 shows similar height and force modulation images obtained for the annealed P(S-*b*-MMA) film for a scan size of $1 \times 1\ \mu m^2$. Because PS has a lower surface tension with air, annealing causes the PS block to migrate over the surface of the sample covering the PMMA domains [26]. In comparison to the unannealed sample, the height features present in Fig. 4(a) are due mainly to the overall surface roughness of the film. In addition, the faint height variations associated with the microphase separated morphology are only 0.8 nm in amplitude. As a result, the surface roughness tends to obscure the microstructure making the block copolymer morphology not readily apparent. The elasticity image (see Fig. 4(b)), however, does show the alternating PS and PMMA domains of the lamellar morphology. The elasticity contrast present in the annealed sample is only about 1/3 that of the unannealed sample which is consistent with a thin PS surface layer. The characteristic domain spacing of the annealed morphology obtained from Fig. 4(b) is found to be 55 nm. Although this value is significantly larger than that measured for the unannealed sample, spin coating of diblock copolymers has been shown to produce chaotic morphologies with domain sizes much smaller than their equilibrium spacings [27]. The domain spacing of the annealed sample, however, does agree well with the equilibrium bulk domain spacing measured with SAXS of 55.3 nm. The P(S-*b*-MMA) samples show that FMM is well suited to studying the near-surface morphologies of block copolymer thin films even when both blocks of the copolymer are glassy.

Measuring the sizes of individual domains using conventional contact mode AFM or TEM is not always a straightforward process. For AFM, the local topography does not always correspond directly to the microphase separated morphology. For TEM, staining, microtomy, beam damage and sample tilt can all lead to incorrect domain spacing measurements. FMM, however, is much better suited to measurements of domain size. In a strongly segregated block copolymer, the elasticity measured at any given point is directly correlated to the composition of the material being probed by the tip. As a result, the force modulation image gives a true representation of the microphase separated morphology. Height and force modulation images for the P(S-*b*-HIC) rod–coil block copolymer thin film are shown in Fig. 5 for a scan size of 2.5 × 2.5 μm^2. Fig. 5(a and b) shows the alternating "zig-zag" lamellar structure of PS and PHIC domains observed previously by Chen *et al.* [19] using bright field TEM and electron diffraction. From the height image, the amorphous PS domains are found to be ~7 nm lower than the surrounding PHIC rod domains. Height profiles obtained from slices vertical to the sample plane show that many of the height depressions present throughout the scan result from local packing defects within the PHIC domains and are only ~3 nm in depth. Unlike the previous two samples, the morphology of the P(S-*b*-HIC) thin film in Fig. 5(a) is not significantly obscured by any surface roughness across the scan area. In this case, the elasticity image, Fig. 5(b), shows essentially the same morphology. While PHIC is known to be a rigid rod [28, 29], it adopts an 8_3 helical conformation [19] in which the paraffin-like pendent hexyl groups surround the PHIC backbone. Consequently, the glassy PS domains are stiffer than the PHIC domains and appear dark in the elasticity image. The average PHIC domain spacing measured from the elasticity image, 177 nm, agrees well with the 180 nm value obtained from TEM. In contrast, the average width of the PS domains obtained from the elasticity image is found to be 15 nm, which is significantly lower than the average value obtained previously using TEM of 25 nm. The value of the PS domain size obtained using FMM is likely a more accurate value of the actual domain size. Swelling effects resulting from the RuO_4 staining of the PS and the effect of sample tilt on the finite thickness samples could easily explain the larger values of the PS domain size measured previously. These results show a major advantage of FMM over other existing microscopy techniques. Namely, for a given sample, block copolymer domain sizes can be accurately measured using FMM entirely bypassing the need for heavy metal staining or for microtomy which can lead to errors induced through sample preparation.

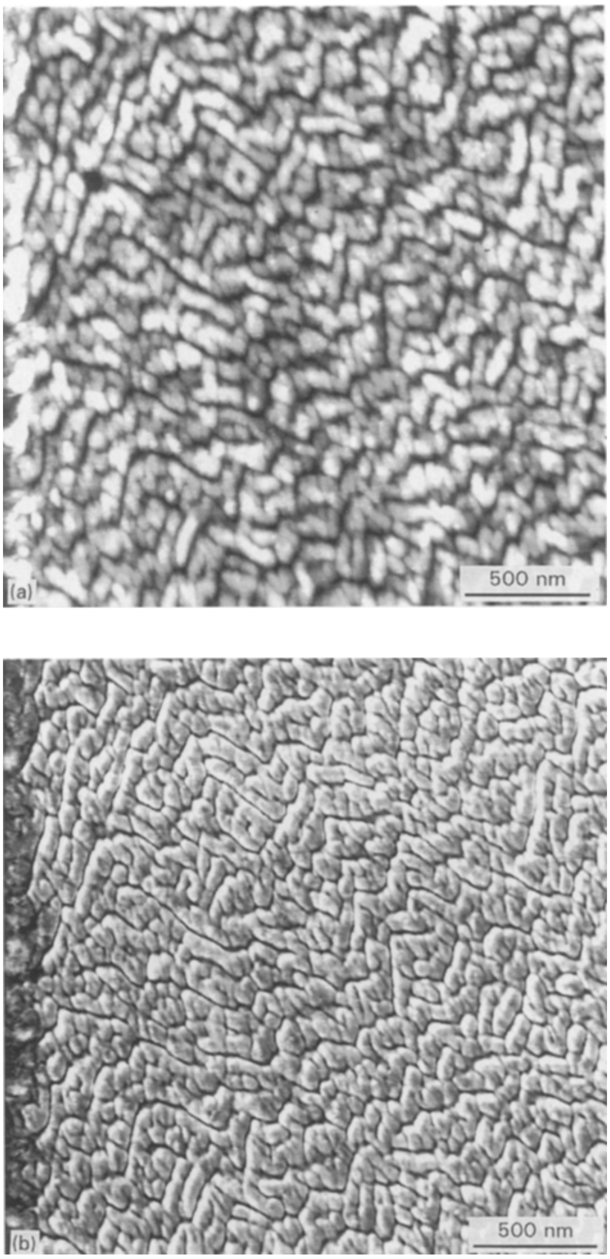


Figure 5 (a) Height image of the unannealed P(S-*b*-HIC) ultrathin film which shows the zig-zag lamellar morphology. Numerous packing defects are present in the higher PHIC domains. (b) Elasticity image for the same sample. The PS domains in the elasticity image are significantly narrower than the corresponding regions in the height image.

In addition to the uses of FMM in studying block copolymer morphology demonstrated in this paper, FMM has the potential to be used in a number of other applications. As mentioned earlier, FMM can be used to study kinetic phenomena such as the migration of polymers over surfaces. Depending on the glass transition temperatures of the individual blocks or homopolymers, temperature stages could be used to conduct *in-situ* migration studies of both pure block copolymers and block copolymer/homopolymer blends. In a pure block copolymer, the lower surface tension block would be tethered to the intermaterial dividing surface (IMDS) and migration would be hindered. In a blend, however, no such anchoring of the homopolymer chains would be present allowing the homopolymer to freely diffuse. In addition, FMM could be used to study surface reconstruction in block copolymers. The presence of a free surface perturbs the free energy of the block copolymer near the surface. As a result, a different equilibrium morphology may be preferred at the surface than is present in the bulk block copolymer. Bulk samples could be fractured to expose the internal morphology and studied with FMM as a function of time or temperature as the surface morphology evolved. The elasticity at a given point on the microphase separated block copolymer sample is dependent on the local composition and the amount of loading. As a result, FMM could be used to study the lateral composition profile near the interfacial region between block copolymer domains (rubbery/glassy, non-crystalline/crystalline, ... etc.). The effect of the degree of segregation on the interfacial width could also be investigated. By varying the amount of modulation applied to the sample, information on the vertical composition could be obtained (i.e. thicknesses of surface layers). In the future, as the complex interaction between the tip and sample is better understood and reliable calibration standards for FMM are developed, exact values of the modulus at any point in the scan may be achievable. Thus, the modulus of a given polymer confined within a strongly segregated block copolymer domain, which is likely to be significantly different from that of the free homopolymer, could be measured. In addition, the influence of architecture, morphology, and molecular weight on the modulus could be studied. With advances in force modulation microscope design, a dynamic mechanical analysis of block copolymers and semicrystalline homopolymers on the molecular scale could also be possible.

4. Summary

Force modulation microscopy (FMM), a type of atomic force microscopy which measures the relative elasticity rather than the height across the surface of a sample has been used to study the microphase separated morphology of an unannealed roll-cast P(S-B-S) film, unannealed and annealed spin-coated P(S-*b*-MMA) thin films, and an unannealed dilute solution-cast P(S-*b*-HIC) rod–coil block copolymer ultrathin film. Height and elasticity images of the same area of each block copolymer sample were obtained simultaneously and compared. In all the samples, the elasticity image was found to produce excellent detail of the actual block copolymer morphology based solely on the elasticity contrast inherently present between the different blocks. The elasticity image, especially for the P(S-B-S) and the P(S-*b*-MMA) samples, was found to be superior to the corresponding height image which showed little or no contrast. Besides imaging a P(S-B-S) sample, in which the elasticity difference between PB and PS is large, P(S-*b*-MMA) samples, in which both blocks were glassy, were successfully imaged. Traditional height scans of block copolymer samples do not always reveal the underlying microphase separated morphology. Topographical scans rely exclusively on morphology-induced height differences which may not always be present. When subtle height features are present, they may easily be obscured by surface roughness. FMM, however, which relies on elasticity differences alone, is directly sensitive to surface composition and thus morphology. In addition, FMM does not suffer from the many artefacts induced by sample preparation in TEM and LVHRSEM. In this study, FMM has been shown to be a valuable new tool which can be used to study the surface and bulk morphologies of block copolymers with a high degree of lateral resolution and local surface modulus sensitivity yielding results equal to or better than those obtainable from existing techniques.

Acknowledgements

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