ELSEVIER

Polymer 42 (2001) 1613–1619

polymer

www.elsevier.nl/locate/polymer

# Focused ion beam/lift-out transmission electron microscopy cross sections of block copolymer films ordered on silicon substrates

H. White[a,*], Y. Pu[a], M. Rafailovich[a], J. Sokolov[a], A.H. King[b], L.A. Giannuzzi[c], C. Urbanik-Shannon[c], B.W. Kempshall[c], A. Eisenberg[d], S.A. Schwarz[e], Y.M. Strzhemechny[e]

[a]*Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, NY 11794-2275, USA*
[b]*School of Materials Engineering, Purdue University, West Lafayette, IN 47907-1289, USA*
[c]*Department of Mechanical, Materials, and Aerospace Engineering, University of Central Florida, Orlando, Fl 32816-2450, USA*
[d]*Department of Chemistry, McGill University, 801 Sherbrooke West, Montreal, Que., Canada H3A2K6*
[e]*Department of Physics, CUNY Queens College, Flushing, NY 11367, USA*

Received 27 March 2000; received in revised form 31 May 2000; accepted 12 June 2000

---

## Abstract

Thin poly(styrene$_{210}$-*b*-2-vinylpyridine$_{200}$) and poly(2-vinylpyridine$_{94}$-*b*-styrene$_{760}$-*b*-2-vinylpyridine$_{94}$) films spun cast on silicon and annealed at 180°C for 3 days were directly cross sectioned in less than 1 h using the focused ion beam (FIB) lift-out technique. We show that with the FIB procedure, it is possible to produce cross sections that reveal structure near the silicon interface and hence the surface induced phase transitions could be examined and compared quantitatively with theoretical models. Atomic force microscopy, dynamic secondary ion mass spectrometry, and transmission electron microscopy were used to characterize the films. © 2000 Elsevier Science Ltd. All rights reserved.

*Keywords*: Block copolymer; Focus ion beam/lift-out; Transmission electron microscopy

---

## 1. Introduction

A two component polymer system, such as a homopolymer blend or a block copolymer melt, in the vicinity of an interactive surface is an inherently inhomogeneous system. The difference in chemical affinity between the components and the substrate induces a preferred orientation in the system. For example, for polymer blends, numerous groups [1–10] have demonstrated that spinodal decomposition, and wetting layers with a specific wavelength can be oriented parallel to an interactive surface. More recently studies [11–13] have shown that two dimensional structures can be nucleated on a two dimensional surface chemical pattern. It is also well known [14–16] that surfaces can induce both parallel and perpendicular ordering of block copolymers. In most of these studies silicon has been the surface of choice since it can be obtained in single crystal form with an atomically smooth surface. Furthermore, it is used extensively in the semiconductor industry where numerous procedures have been developed to modify the surface energy and apply both chemical and topographical patterns [17,18].

Most of what is known today on the orientation of polymers at surfaces has been obtained by neutron reflectivity [14–16,19–22], X-ray reflectivity [23–28] and secondary ion mass spectrometry [5–7,29–38]. These methods work best when the orientation occurs perpendicular to the surface and is symmetric in the plane parallel to the surface. Since phase information is lost in reflectivity methods, it is difficult to obtain unique interpretations of the diffuse scattering data from structures where in plane ordering occurs. Cross-sectional transmission electron microscopy (TEM) of specimens ordered on silicon has proved very challenging due to the difficulty of preparing electron transparent sections. It was not possible to directly section hard silicon crystals so several authors relied on reconstructive imaging of the film prepared on silicon via a continuous ion milling technique [39] and preparing films on Ultem followed by ultramicrotomy [40,41] to determine ordering in the bulk away from the polymer/substrate interface. Earlier techniques aimed at determining order near the polymer/silicon interface [6,9] involved surface induced ordering on silicon followed by removal of the film from the silicon substrate, using an etching technique, to a more suitable substrate for ultramicrotomy. Unfortunately, the removal technique destroys the first layers of the film and only works in a

---

* Corresponding author. Tel.: +1-631-632-3234; fax: +1-631-632-5764.
*E-mail address:* hwhite@ic.sunysb.edu (H. White).

PII: S0032-3861(00)00503-6

few limited cases where the film is extremely inert to acid and base degradation.

In this paper we report on the application of the focused ion beam (FIB)/lift-out method to thin polymer films ordered on silicon. This method relies on the use of a focused beam of $Ga^+$ ions that are used to mill out electron transparent sections from the specimen for TEM analysis [42]. This method has several advantages that make it potentially suited for the study of polymer films ordered on hard inorganic surfaces. (a) Polymer films need not be removed from their substrates and mounted on embedding media before sectioning since no knife or blade is used. (b) The focused ion beam can be manipulated with high spatial resolution; hence cross sections can be made accurately through diverse features on the surface of inhomogeneous films. (c) Organic/inorganic composite materials can be sectioned without artifacts induced by differences in hardness between the two materials that can potentially deform the sample at the interfaces.

In order to test the FIB method, we chose the diblock copolymer system, poly(styrene-*b*-2-vinylpyridine) (PS-$P_2VP$), which is known [6,7,9] to form highly ordered structures on silicon substrates. In a previous study [6,7] the ordering of PS-$P_2VP$ on silicon was investigated using neutron reflectivity and standard cross section specimen preparation followed by ultramicrotomy. In the case of symmetric diblock copolymers, it was shown [6] that for large degrees of polymerization defects formed around dislocations to minimize chain stretching. In the case of asymmetric diblock copolymers [43], neutron reflection showed that regardless of the bulk morphology, the initial layers at the substrate were lamella. Unfortunately, due to limitations imposed by the floating procedures the layer near the surface region is destroyed, hence, these transitions could not be studied directly by TEM. In this paper we show that with the FIB procedure it is possible to produce cross sections with much smaller structures near the silicon interface and hence the surface induced phase transition could be compared quantitatively with theoretical models [44].

## 2. Experimental section

Monodisperse ($M_w/M_n < 1.1$) diblock and triblock copolymers of PS and $P_2VP$ were synthesized via anionic polymerization and characterized by $^{13}C$ NMR and mass analysis to determine the $P_2VP$ volume fraction [45,46] (see Table 1). Under the experimental conditions, the chain dimensions (i.e. the statistical segment length ($a = 0.67$ nm) [47] and the segment concentration ($\rho_o \sim 9.4 \times 10^{-3}$ mol/cm$^3$) [48]) of both PS and $P_2VP$ are assumed identical, and PS and $P_2VP$ are highly immiscible (Flory interaction parameter ($\chi_{PS-P_2VP} \sim 0.1$) [49].

The diblock and triblock copolymer films were prepared by dissolving 60 mg of polymer in 3 ml of toluene. To remove organic deposits, silicon substrates were cleaned by sonicating in Chromerge solution for 60 min. The solutions were then spun cast at 2500 rpm onto the native oxide covered silicon substrates. In order to orient the block copolymer chains, the specimens were annealed above the glass transition of both polymers, at $T = 180°C$, in vacuum for 3 days. Following annealing, the specimens were stained in iodine vapor for 12 h. Iodine binds preferentially to $P_2VP$ and hence provides electron contrast for TEM analysis. The films were then sectioned via FIB.

Table 1
Block copolymer properties

| | Composition | Polymerization index | *f* |
|---|---|---|---|
| Diblock copolymer | PS-$P_2VP$ | 210-200 | 0.51 |
| Triblock copolymer | $P_2VP$-dPS-$P_2VP$ | 94-760-94 | 0.12 |

The FIB sectioning procedure involved first sputter coating the specimen with gold–palladium to protect the top surface from subsequent TEM specimen preparation damage. TEM specimens were prepared in cross section with an FEI FIB 200TEM Workstation, operating at 30 keV, using the “lift-out” technique [42]. Prior to sectioning, a platinum metal line was ion beam deposited using the FIB. This serves two purposes: (i) the metal line locates the area of interest; and (ii) the metal line prevents the outer surface of the sample from being damaged during subsequent ion milling operations. The finely focused $Ga^+$ ion beam was used to cut out electron transparent membranes from the samples [42,50–53] as illustrated in Fig. 1. A glass rod attached to a hydraulic micromanipulator was then used to lift out the electron transparent membrane and position it on a carbon coated copper mesh grid. In less than 1 h a rigid cross section approximately 20 nm in thickness was prepared for TEM characterization. A scanning electron micrograph of the FIB cross section is shown in Fig. 2. In the figure we can clearly see the protective coatings, the polymer film, and the silicon substrate. Note that there is some minor damage that occurred at the free surface during deposition of the protective coating. In contrast to previous work [6,9] where the polymer/silicon interface is destroyed during the KOH etching process, the polymer/silicon interface produced by the FIB technique is remarkably uniform and strain free.

## 3. Results and discussion

The morphology of $(A–B)_n$ block copolymers in the bulk is determined by $\chi N$, *f*, and *n* where $\chi$ is the Flory–Huggins interaction parameter, *N* the total degree of polymerization, *f* volume fraction of the A component, and *n* defines the copolymer architecture ($n = 1$, diblock; $n = 2$, triblock; and $n \geq 3$, starblock). In the strongly segregated regime ($\chi N \gg 10$), lamella, ordered bicontinuous double diamond, cylindrical, and spherical morphologies can exist depending on the value of *f* [54].

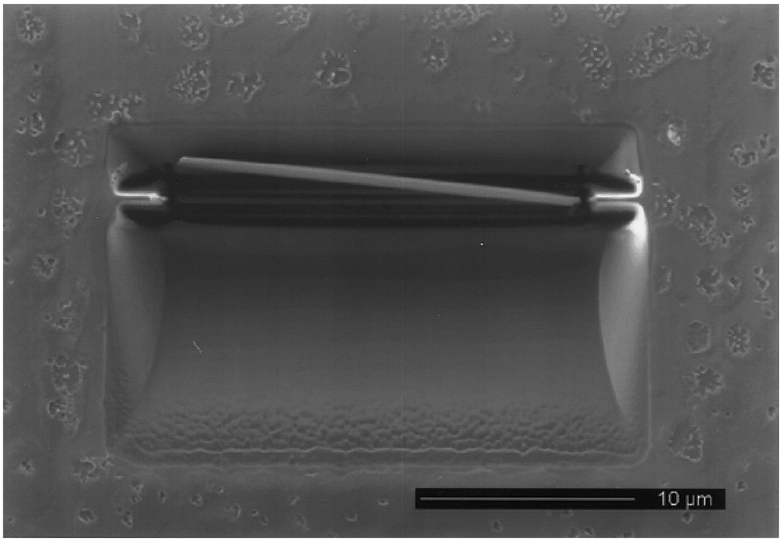

Fig. 1. Scanning electron micrograph of the plan view of the final FIB cut prior to lift-out. An electron transparent cross section (20 μm × 5 μm× ~ 20 nm) is freed within the specimen trench.

For copolymers ordered on hard substrates, the surface energy difference between the components and the chemical affinity of one block for the substrate forces the orientation of lamellae or cylinders parallel to the surface [55]. Cross sectional TEM of specimens prepared by ultramicrotomy have been used to illustrate the surface induced lamella ordering of symmetrical poly(styrene-*b*-2-vinylpyridine) on silicon surfaces [6,9]. The film thickness at any given point can be estimated by $(n + 1/2)L \pm \Delta$ where $n$ is the total number of lamella and $L$, the lamella period. If the total thickness of the film deviates from $(n + 1/2)L$ by a thickness, $\Delta$, a partial layer is formed at the free surface exactly $L$ in height. The morphology of this layer will have islands or holes depending on whether $\Delta$ is smaller or larger than $L/2$, respectively [56,57].

### 3.1. Poly(styrene$_{210}$-b-2-vinylpyridine$_{200}$)

In a previous study [6], plan view TEM micrographs indicated that for symmetric diblocks the lamella ordering is disturbed around the periphery of islands and holes if $N > 500$. No dislocations were observed for $N = 200$. Cross sectional TEM micrographs for symmetric diblock copolymers with $N = 800$ clearly showed the evolution of

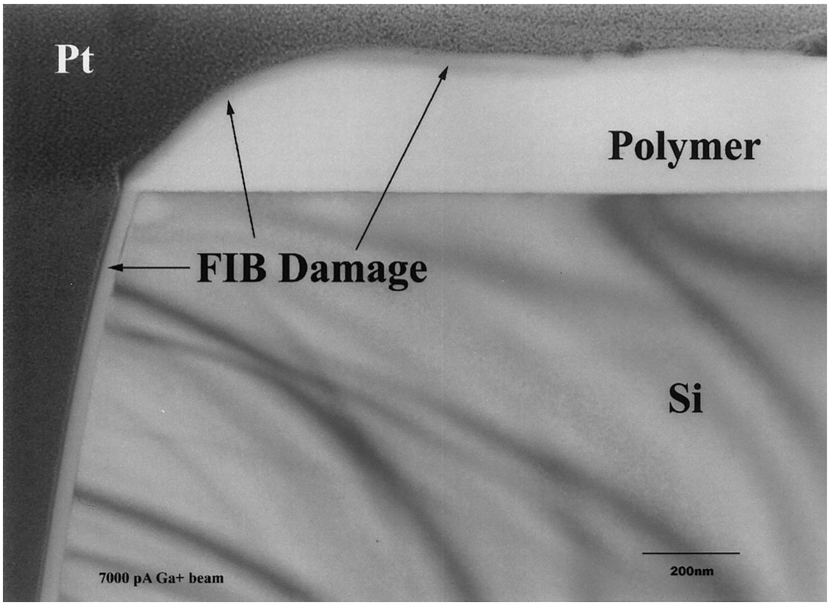

Fig. 2. Scanning electron microscopy image of film damage due to FIB procedure. Damaged areas (slightly below metallized area) are slightly discolored (stained) as indicated by the arrows near the polymer surface.

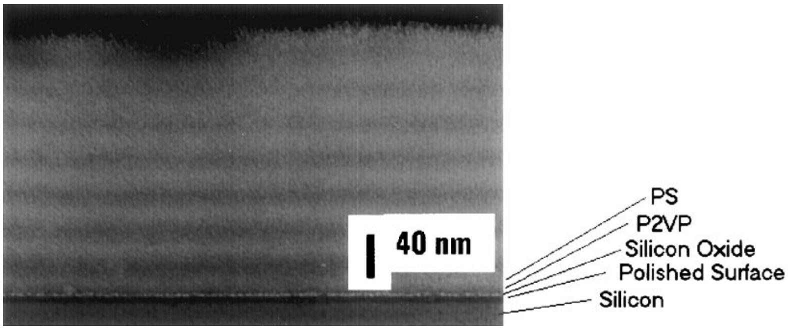

Fig. 3. TEM micrograph of FIB lift-out cross section of a poly(styrene$_{210}$-*b*-2-vinylpyridine$_{200}$) film ordered on a silicon substrate. The lamella period is 40 ± 1 nm.

dislocations that eventually led to a surface hole morphology. From cross sectional TEM a lamella to cylindrical transition was apparent when $L$ was large and the deformation imposed by the high surface tension induced an appreciable amount of chain stretching (greater than 40%). For $N < 500$, a small value of $L$ allows holes to form without chain stretching.

A TEM image of the FIB lift-out cross section of PS$_{210}$-*b*-P$_2$VP$_{200}$ ($N < 500$) is shown in Fig. 3. As expected, the symmetric diblock copolymer ($f \sim 0.5$), is microphase segregated in a lamella structure that is oriented normal to the silicon interface. From the silicon substrate, the polished silicon surface (dark line); the oxide layer (white line); and a partial P$_2$VP layer are clearly seen. The lamella height ($L$) is found to be 40 ± 1 nm and the film consists of well-ordered layers. The polymer/free surface layer is not as distinct as the subsequent layers due to the diffuse interface produced when the protective layer was applied.

Fig. 4 shows a contact mode atomic force micrograph from the surface topography of the PS$_{210}$-*b*-P$_2$VP$_{200}$ film taken prior to the FIB procedure. The presence of holes, uniform in depth, indicates that the film is well ordered with respect to the silicon substrate and that $\Delta$ is larger than $L/2$. From the depth of the holes we obtain $L = 41 \pm 5$ nm, which is in good agreement with the TEM micrograph and delta approximation.

### 3.2. Poly(2-vinylpyridine$_{94}$-b-d-styrene$_{760}$-b-2-vinylpyridine$_{94}$)

In order to investigate the surface induced ordering in a non-lamella system a film of the triblock copolymer, P$_2$VP$_{94}$-dPS$_{760}$-P$_2$VP$_{94}$, was spun cast onto a silicon substrate and annealed at 180°C for 3 days. For this system $f = 0.12$ and hence a spherical micelle morphology is expected. Using a Landau Ginzberg mean field treatment, the profile of highly ordered block copolymers near an interface that is attractive for one of the components was calculated. When the attraction of the interface to the minority component is large, the energy gained by covering the interface with only one component is sufficient to induce a phase transition from the lower energy bulk spherical micelle phase to a surface lamella phase. The decay length of the lamella phase to the bulk spherical phase is obtained by balancing the interfacial tension between the two blocks, $\gamma_{ab} \sim 6.7\chi^{1/2}$, with the bending and compressional energy of deforming the lamella phase. Hence for small $\chi$, the decay length ($\xi$) is long, spreading the interface over many lamellae and minimizing the distortion of the chains in the individual lamella. If $\chi$ is large, or greater than 0.1, as is the case for PS and P$_2$VP, then the interfacial region is narrow and large undulations occur over the space of a few layers (Fig. 3 in Ref. 44) as the spherical interface is forced to mesh with the lamella interface. In this limit we can estimate the value for the decay length of the lamella layer to the bulk spherical structure. In the strong segregation

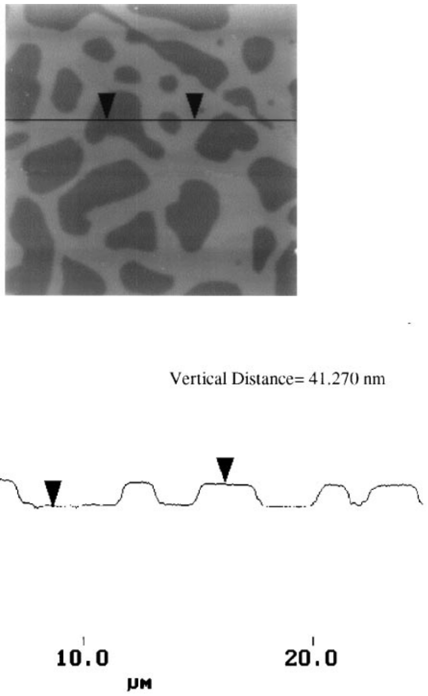

Fig. 4. Contact mode sectional atomic force micrograph of poly(styrene$_{210}$-*b*-2-vinylpyridine$_{200}$) illustrating a film thickness which is not an integer of the lamella height. Holes form with depth, $L$, 40 ± 5 nm.

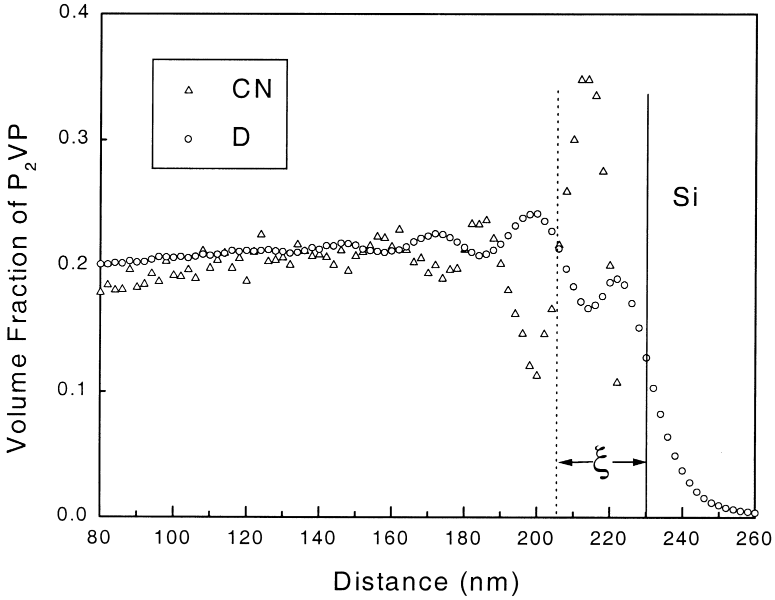

Fig. 5. dSIMS profile of poly(2-vinylpyridine$_{94}$-*b*-d-styrene$_{760}$-*b*-2-vinylpyridine$_{94}$) on silicon (PS profile was superimposed on the $P_2$VP profile to illustrate the decaying lamella morphology). An ordered structure is present near the silicon surface. The correlation length is ~25 nm.

regime, the correlation length is found by minimizing the free energy of the film given by;

$$F_A = \frac{1}{4} a_0^2 (KB)^{1/2} q^2 \coth(\nu\xi) \tag{1}$$

where $B$ is the compression modulus; $K$, the bending modulus; $q$, the wavevector; $a_0$, the amplitude of the deformation; and $\nu$ is $q^2(K/B)^{1/2}$.

Here the gain in enthalpy caused by converting the surface with the minority component is balanced by the stretching and compressional energy of creating the interface between the two structures. Solving for $\xi$ we

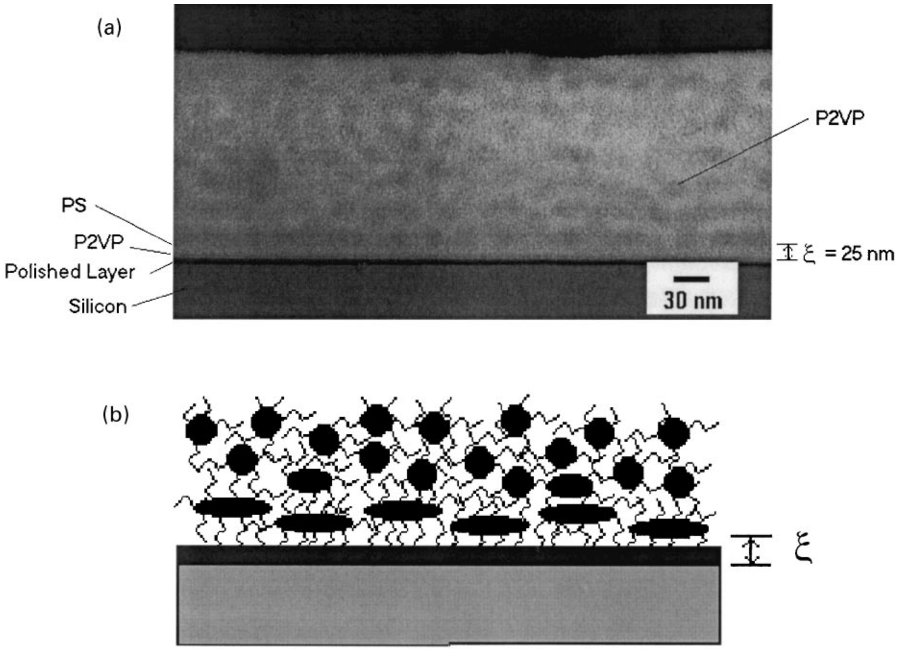

Fig. 6. (a) TEM micrograph of FIB/lift-out cross section of a poly(2-vinylpyridine$_{94}$-*b*-d-styrene$_{760}$-*b*-2-vinylpyridine$_{94}$) film ordered on a silicon substrate. A disordered (i.e. projection of spherical domains) morphology is present within the film and a continuous PS brush is present near the silicon surface. (b) Lamella to spherical phase transition. The lamella ordering is initially surface induced due to the differences in free energies of the two blocks dominating the creation of a lamella layer and bending and compressional energy penalties. The lamella layer begins to undulate causing a break down in the lamella layer and the formation of a disordered structure near the free surface. Poly-2-vinylpyridine is black.

find [44];

$$\xi/h \cong 3.05 + \log[\gamma_{AB}/h\Delta F] \quad (2)$$

where $h$ is the thickness of a single lamella layer ($h = (\gamma_{AB}/3)^{1/3}N^{2/3}$), and $\Delta F$ is the free energy difference between the lamella and spherical phases given by

$$\Delta F = 3.05(\gamma_{AB}/N)^{2/3}(0.28 - f) \quad (3)$$

Substituting $\chi = 0.1$, $N = 854$, $\gamma_{AB} = 2.1$ dyn/cm, $h = 8$ nm and $f = 0.12$ into Eq. (2) for the triblock copolymer we obtain $\xi = 28$ nm.

Dynamic Secondary Ion Mass Spectroscopy (dSIMS) data obtained from the film prior to the FIB procedure is shown in Fig. 5. The $P_2VP$ ($CN^-$) and dPS ($D^-$) traces are plotted as a function of distance from the free surface. The dSIMS data suggest that a partial layer of $P_2VP$ exist at the silicon surface followed by a PS layer that interfaces with a disordered structure. The correlation length is approximately 25 nm.

The FIB cross section of the $P_2VP_{94}$-$dPS_{760}$-$P_2VP_{94}$ film is shown in Fig. 6a. From the silicon substrate, we can see the polished silicon surface (dark line); a very thin oxide layer (white line); a lamella layer of $P_2VP$ (very thin) followed by PS; and a disordered structure (i.e. projection of spherical domains) in the bulk of the film. The PS layer interfaces gradually with the bulk disordered morphology as illustrated in the schematic in Fig. 6b. The correlation length of the lamella layer (~25 nm) is in good agreement with the values determined by the mean field theory and by dSIMS. This result is also in agreement with previous neutron reflection studies on asymmetric diblock copolymers [43], which showed that regardless of the bulk morphology, the initial layers at the substrate were lamella.

## 4. Conclusions

In this paper, we report the first direct cross sectioning of copolymer films ordered on silicon substrate using the FIB/lift-out technique. The motivation for this study is to create interest in using the FIB technique as an alternative to ultramicrotomy to prepare (directly, precisely and quickly) cross sections of interesting polymer morphologies ordered on “hard” surfaces. The resulting diblock morphology showed a dislocation free perfect ordering of lamellae throughout the film thickness. Atomic force microscopy (AFM) measurements showed holes at the free surface. These results were previously obtained [6] using plan view transmission electron micrographs to illustrate the formation of surface features (islands and holes) without a lamella to cylindrical phase reorientation.

The triblock morphology resulted in a surface induced lamella ordering (i.e. “brush”) near the silicon surface gradually losing order into the bulk of the film. This phenomenon was predicted by neutron reflection experiments [43] and is similar to the lamella to spherical phase transitions reported in the literature [44].

In this study the FIB/lift-out technique was used to successfully produce cross sections (~20 nm thickness) of interesting surface induced lamella morphologies. The investigation however lacked care in protecting the free surface. Future work in this area will should directed towards protecting the free surface/polymer interface prior to sectioning with the FIB/lift-out technique.

## Acknowledgements

This work was funded by the NSF (DMR 9632525, DGE 955452 and DMR 9703281) and the I4-UCF/Cirent Partnership. We are grateful to Paul Anzalone of FEI for his assistance with the FEI FIB 200TEM workstation.

## References

[1] Bruder F, Brenn R. Phys Rev Lett 1992;69:624.

[2] Steiner U, Klein J, Fetters LJ. Phys Rev Lett 1994;72:1498.

[3] Liu Y, Rafailovich MH, Sokolov J, Schwarz SA, Zhong X, Eisenberg A, Kramer EJ, Sauer BB, Satija S. Phys Rev Lett 1994;73:440.

[4] Genzer J, Composto RJ. Europhys Lett 1997;38:171.

[5] Karim A, Slawecki TM, Kumar SK, Douglas JF, Satija SK, Han CC, Russell TP, Liu Y, Overney R, Sokolov J, Rafailovich MH. Macromolecules 1998;31:857.

[6] Liu Y, Rafailovich MH, Sokolov J, Schwarz SA, Bahal S. Macromolecules 1996;29:899.

[7] Liu Y, Zhao W, Zheng X, King A, Singh A, Rafailovich MH, Sokolov J, Dai KH, Kramer EJ, Schwarz SA, Gebizlioglu O, Sinha SK. Macromolecules 1994;27:4000.

[8] Dai CA, Jandt KD, Iyengar DR, Slack NL, Dai KH, Davidson WB, Kramer EJ, Hui C-Y. Macromolecules 1997;30:549.

[9] Liu Y, Rafailovich MH, Sokolov J, Schwarz SA, Zhong X, Eisenberg A, Kramer EJ, Sauer BB, Satija S. Phys Rev Lett 1994;73:440.

[10] Hariharan A, Kumar SK, Rafailovich MH, Sokolov J, Zheng X, Duong DH, Schwarz SA, Russell TP. J Chem Phys 1993;99:656.

[11] Karim A, Douglas JF, Nisato G, Liu DW, Amis EJ. Macromolecules 1999;32:5917.

[12] Boltau M, Walheim S, Mlynek J, Krausch G, Steiner U. Nature 1998;391:877.

[13] Rockford L, Liu Y, Mansky P, Russell TP, Yoon M, Mochrie SG. J Phys Rev Lett 1999;82:2602.

[14] Russell TP. Curr Opin Colloid Interface Sci 1996;1:107.

[15] Russell TP, Lambooy P, Kellogg GJ, Mayes AM. Physica B 1995;213:22.

[16] Mansky P, Russell TP. Macromolecules 1995;28:8092.

[17] Higashi GS, Chabal YJ, Trucks GW, Raghavacharik K. Appl Phys Lett 1990;56:656.

[18] Trucks GW, Raghavacharik K, Higashi GS, Chabal YJ. Phys Rev Lett 1990;65:504.

[19] Huang E, Russell TP, Harrison C, Chaikin PM, Register RA, Hawker CJ, Mays J. Macromolecules 1998;31:7641.

[20] Mansky P, Russell TP, Hawker CJ, Pitsikalis M, Mays J. Macromolecules 1997;30:6810.

[21] Russell TP. Physica B 1996;221:267.

[22] Russell TP. MRS Bulletin 1996;21:49.

[23] Wang J, Tolan M, Seeck OH, Sinha SK, Bahr O, Rafailovich MH, Sokolov J. Phys Rev Lett 1999;83:564.

[24] Li Z, Tolan M, Hohr T, Kharas D, Qu S, Sokolov J, Rafailovich MH,

Lorenz H, Kotthaus JP, Wang J, Sinha SK, Gibaud A. Macromolecules 1998;31:1915.

[25] Vignaud G, Gibaud A, Wang J, Sinha SK, Daillant J, Grubel G, Gallot Y. J Phys: Condens Matter 1997;9:L125.

[26] Sinha SK. Curr Opin Solid State Mater Sci 1996;1:645.

[27] Sinha SK, Feng YP, Melendres CA, Lee DD, Russell TP, Satija SK, Sirota EB, Sanyal MK. Physica A 1996;231:99.

[28] Tolan M, Vacca G, Wang J, Sinha SK, Li Z, Rafailovich MH, Sokolov J, Gibaud A, Lorena H, Kotthaus JP. Physica B 1996;221:53.

[29] Ge S, Guo LT, Rafailovich MH, Sokolov J, Peiffer DG, Schwarz SA, Colby RH, Dozier WD. Langmuir 1999;15:2911.

[30] Yokoyama H, Kramer EJ, Rafailovich MH, Sokolov J, Schwarz SA. Macromolecules 1998;31:8826.

[31] Li Z, Qu S, Rafailovich MH, Sokolov J, Tolan M, Turner MS, Wang J, Schwarz SA, Lorenz H, Kotthaus JP. Macromolecules 1997;30:8410.

[32] Liu Y, Schwarz SA, Zhao W, Quinn J, Sokolov J, Rafailovich MH, Iyengar D, Kramer EJ, Dozier W, Fetters LJ, Dickman R. Europhys Lett 1995;32:211.

[33] Liu Y, Zhao W, Zheng X, Singh A, Rafailovich MH, Sokolov J, King AH, Dai K, Washiyama J, Kramer EJ, Schwarz SA. Interfaces II 1995;189:161.

[34] Liu Y, Rafailovich MH, Sokolov J, Schwarz SA, Zhong X, Eisenberg A, Kramer EJ, Sauer BB, Satija S. Phys Rev Lett 1994;73:440.

[35] Mansfield T, Stein RS, Composto RJ, Rafailovich MH, Sokolov J, Schwarz SA. Physica B 1991;173:207.

[36] Agrawal G, Wool RP, Dozier WD, Felcher GP, Zhou J, Pispas S, Mays JW, Russell TP. J Polym Sci B: Polym Phys 1996;34:2919.

[37] Stoffel NC, Dai CA, Kramer EJ, Russell TP, Deline V, Volksen W, Wu WL, Satija S. Macromolecules 1996;29:6880.

[38] Yokoyama H, Mates TE, Kramer EJ. Macromolecules 2000;33:1888.

[39] Harrison C, Park M, Chaikin P, Register RA, Adamson DH, Yao N. Macromolecules 1998;31:2185.

[40] Kunz MS, Shull KR, Kellock AJ. J Appl Phys 1992;72:4458.

[41] Kunz MS, Shull KR, Kellock AJ. J Colloid Interface Sci 1993;156:240.

[42] Giannuzzi LA, Drown JL, Brown SR, Irwin RB, Stevie FA. Focused ion beam milling and micromanipulation lift-out for site specific cross-section tem specimen preparation. In: Anderson RM, Walck SD, editors. Materials Research Society Symposium Proceedings, vol. 480. Pittsburgh, PA: Materials Research Society, 1997.

[43] Zhao W, Zhao X, Rafailovich MH, Sokolov J, Composto RJ, Smith SD, Satkowski M, Russell TP, Dozier WD, Mansfield T. Macromolecules 1993;26:561.

[44] Turner MS, Rubinstein M, Marques CM. Macromolecules 1994;27:4986.

[45] O'Malley JJ, Crystal RC, Erhardt PF. Polym Prepr (Am Chem Soc, Div Polym Chem) 1969;10:796.

[46] Dai KH, Kramer EJ. Polymer 1994;35:157.

[47] Tangari C, King JS, Summerfield GC. Macromolecules 1982;15:132.

[48] Hellmege K, Knappe W, Lehmann P. Kolloid Z 1963;183:110.

[49] Shull KR, Kramer EJ, Hadziioannou G, Tang W. Macromolecules 1990;23:4780.

[50] Giannuzzi LA, Drown JL, Brown SR, Irwin RB, Stevie FA. Microsc Res Tech 1998;41:285.

[51] Prenitzer BI, Giannuzzi LA, Newman K, Brown SR, Shofner TL, Irwin RB, Stevie FA. Metall Mater Trans A 1998;29:2399.

[52] Giannuzzi LA, Prenitzer BI, Drown JL, Shofner TL, Brown SR, Irwin RB, Stevie FA. J Process Anal Chem 1999;4:162.

[53] Giannuzzi LA, Stevie FA. Micron 1999;30:197.

[54] Bates SB. Annu Rev Phys Chem 1990;41:525.

[55] Mayes AM, Russell TP, Bassereau P, Baker SM, Smith GS. Macromolecules 1994;27:749.

[56] Coulon G, Ausserre D, Russell TP. J Phys (Paris) 1990;51:777.

[57] Coulon G, Collin B, Ausserre D, Chatenay D, Russell TP. J Phys (Paris) 1990;51:2801.