Focused Ion Beam Enabled Characterization of Nanostructured Polymeric Materials

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The internal structures of ordered polymeric materials produced by self-organization (block copolymers) and by multi-beam laser interference lithography were probed by both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) after sectioning in a focused ion beam (FIB) instrument. In addition to detailing the advantages that FIB based sample preparation offers for these types of materials over more common techniques, we also report a systematic study of gallium ion beam induced amorphization, sample heating and structural deformation.

We show how properly optimized conditions for FIB preparation of TEM lamella of polystyrene-poly(dimethylsiloxane) (PS-PDMS), a block copolymer composed of domains with widely contrasting mechanical properties, can lead to more defect free and distortion free lamella when compared to best effort cryo-microtoming. Defect and damage generation mechanisms by both FIB and microtoming techniques are illustrated and characterized. An example of a defect free area in a FIB processed sample is shown in Figure 1.

In order to study the potential effect of ion beam induced heating and damage of copolymer films during FIB processing, a series of polystyrene-polyisoprene (PS-PI) samples were prepared by spin coating onto silicon followed by controlled annealing in a vacuum oven. Samples were chosen with a range of annealing times and TEM samples were prepared both by conventional cryo-microtoming and by FIB techniques. Ion beam induced heating of thin (<100 nm) supported films results in additional annealing resulting in TEM samples that show more complete ordering than the microtomed samples.

Finally, serial sectioning of 3D structured polymeric materials produced by multi-beam laser interference or holographic lithography techniques[1] have been performed in a FIB. The optical and mechanical properties of these structured polymers are highly dependent on the quality of their full three dimensional structure. Again, damage mechanisms and structural deformation issues are addressed. FIB was used to produce and image multiple consecutive cross-sectional samples and the images are used to reconstruct the overall 3D structure.

[1] J-H. Jang, C.K. Ullal, M. Maldovan, T. Gorishnyy, S.E. Kooi, C.Y. Koh, and E.L Thomas, Adv. Funct. Mater. **17**, 3027 (2007).

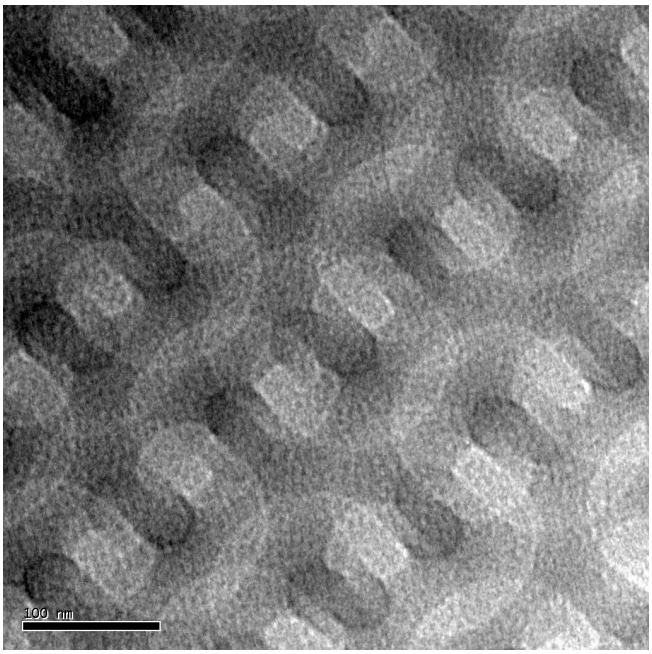

Figure 1: Energy filtered TEM image of a FIB produced lamella from a polystyrene-poly(dimethylsiloxane) (PS-PDMS) block copolymer sample. FIB produced lamella are defect and distortion free compared to cryo-microtomed lamella from the same material.