Supporting Information for

Oxygen K Edge Scattering from Bulk Comb Diblock Copolymer
Reveals Extended, Ordered Backbones above Lamellar Order-Disorder Transition

Jeffrey B. Kortright,1,* Jing Sun,2,+ Ryan K. Spencer,3 Xi Jiang,1 and Ronald N. Zuckermann1,2,*

1Materials Sciences Division, Lawrence Berkeley National Laboratory,
1 Cyclotron Road, Berkeley, California 94720, United States
2Molecular Foundry, Lawrence Berkeley National Laboratory,
1 Cyclotron Road, Berkeley, California 94720, United States
3Department of Chemical Engineering & Materials Sciences, University of California, Irvine,
California 92697, United States

†Current address: School of Polymer Science and Engineering, Qingdao University of Science
and Technology, 53 Zhengzhou Road, Qingdao 266042, China

*Corresponding Authors:
jbkortright@lbl.gov (510-486-5960), rnzuckermann@lbl.gov (510-486-7091)
SI-1. Azimuthal scattering intensity dependence for linear polarization

**Figure S1.** The top CCD images show dark-corrected scattering intensity measured at 30°C and 532 eV using linear horizontal and vertical polarization as indicated. An azimuthal asymmetry of the lamellar peak intensity at 0.6 nm\(^{-1}\) clearly tracks the direction of the electric field. The sum of these two images (bottom) shows no azimuthal asymmetry, confirming isotropic texture of the phase separated lamellar microstructure yielding in the 1 micron thick microtomed slices as observed for bulk material observed with SAXS.


We start with the dark-corrected, azimuthally averaged intensity/pixel data from the sample-plus-membrane assembly and from a blank silicon nitride (SiNx) membrane as provided by the Nika program. In the geometry used the sample was facing the CCD camera, so that background intensity from the SiNx membrane is attenuated as it passes through the sample. Figure S2 below shows this raw data from the sample, prior to any further normalization as described below.
Figure S2. Raw intensity from the sample plus membrane.

The blank membrane intensity is assumed to contain an elastic component that is smoothly varying (generally decreasing) with q and a fluorescence component from nitrogen in the SiNx membrane support that is constant with q. We arbitrarily set the flat fluorescence component to be 0.9 of the maximum possible fluorescence consistent with the data. We then scaled the elastic component by the measured sample transmission and the fluorescence component by the calculated transmission for nitrogen fluorescence at 400 eV through a 1 micron sample prior to subtracting them from the measured intensity from the sample. The remainder is the estimated intensity coming solely from the sample.

This membrane-corrected sample intensity also contains both the desired elastic component and a fluorescence component from the sample. We consider explicitly just the non-resonant fluorescent contribution that we assume is dominated by the carbon in the 1 micron thick sample. Again we arbitrarily set the flat fluorescence component to be some fraction of the maximum value consistent with the data and subtract it from the data. The remainder is our estimate of the elastic scattering from internal sample structure that has been attenuated by resonant absorption in the sample. Dividing this remainder by the sample transmission yields the corrected elastic scattering data presented in manuscript Figure 4. Figure S3 shows the corrected elastic intensity evaluated assuming that the flat non-resonant fluorescence is 90% (black), 50% (red), and 0% (green) of its maximum possible value. In the main text, 90% of maximum is assumed. Subtracting more flat background increases the dynamic range in the data.
along both the $q$ and $h\nu$ axes, although the differences between trends at the carbonyl and ether oxygen resonances at 532 and 538 eV persist.

This procedure does not attempt to correct for any resonant oxygen fluorescence from the sample. Changing the flat non-resonant fluorescence values essentially yields a constant offset that, when normalized by the transmission appropriate for the elastic energy, yields a spectral shape resembling the sample absorption spectrum. While there is evidence of such shape in the results in Figure 4, it is not obvious that this is the source of this shape, since resonant scattering from just the oxygen can exhibit a similar shape. Pronounced $q$ dependence signals elastic scattering.

**Figure S3.** Estimates of corrected elastic intensity from the sample shown here are evaluated assuming that the flat non-resonant fluorescence background is 90% (black), 50% (red), and 0% of its maximum value.

**Figure S4.** Projected form factors $P_i(q, h\nu)$ at 532 (a) and 538 eV (b) are obtained as the normalized square of the Fourier transform of the oxygen-only scattering amplitude along the principle axes $i = x, y, z$ of the extended molecule in Figure 1. Each panel shows the scattering power along the 3 directions and their equally weighted average. Differences between the two energies result from the different distributions of carbonyl and ether oxygens within the molecule.