ESRF	**Experiment title: Self-assembly of grapho-epitaxied block copolymers studied by GISAXS**	**Experiment number**: 02-01-840
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Report:

In order to characterize the self-assembly of PS-*b*-PMMA block copolymers (BCP), GISAXS experiments were performed using a photon energy of 11 keV and a beam focused at the sample position with horizontal and vertical widths equal to 300µm and 150µm.. The GISAXS patterns were recorded under an incidence angle of 0.14° (i.e. just above the critical angle of BCP), using a XPAD pixel detector (960*560 pixels, 130 µm pixel size) mounted on the SAXS bench and located at a distance of 2.99 m from samples. The BCP samples were spin-coated on top of a brush PS-r-PMMA BCP prepared on both unpatterned and prepatterned Si wafers, the pattern consists of a HSQ line grating generated by e-beam lithography. An annealing treatment at 240°C for 10min leads to the formation of a microdomain hexagonal array with perpendicularly oriented PMMA cylinders in a PS matrix. The total BCP layer thicknesses were about 60nm.

We first studied the efficiency of different chemical treatments preceded by UV beam exposure for removing PMMA, required to prepare templates for CMOS industry . Fig.1 shows the effect of UV exposure time. The increase of the intensities of the Bragg rods stemming from the 2D hexagonal array with the UV exposure time is due to the electronic density contrast between the matrix and cylinders which increases with PMMA removal. Along the rods, the intensity oscillations come from the average form factor of cylinders, their period leads to the depth of the empty cylinders. Therefore the best PMMA removal process would be the one leading to the largest depth. Fits of the vertical cuts calculated along the (10) rods using the FitGISAXS software lead to the values of depths. The parameter of the 2D hexagonal array corresponds to an average distance between cylinders of 36.20 nm.

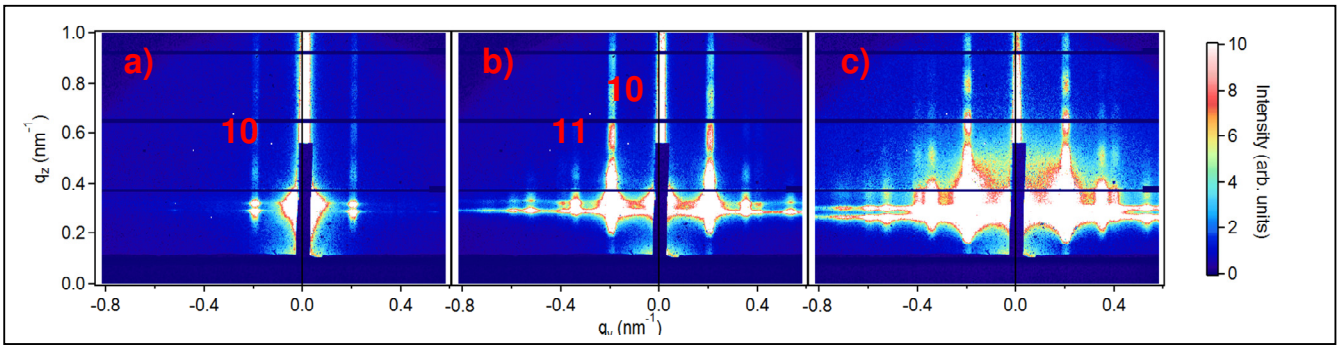

Figure 1: GISAXS patterns of self-assembled (PS-*b*-PMMA) copolymers obtained after different UV exposure times (0s (a), 2s (b) and 10s (c)) followed by a chemical treatment

Other measurements were performed on BCP samples exhibiting different morphologies (lamellae, cylinders) spin-coated on prepatterned surfaces consisting of HSQ line gratings.

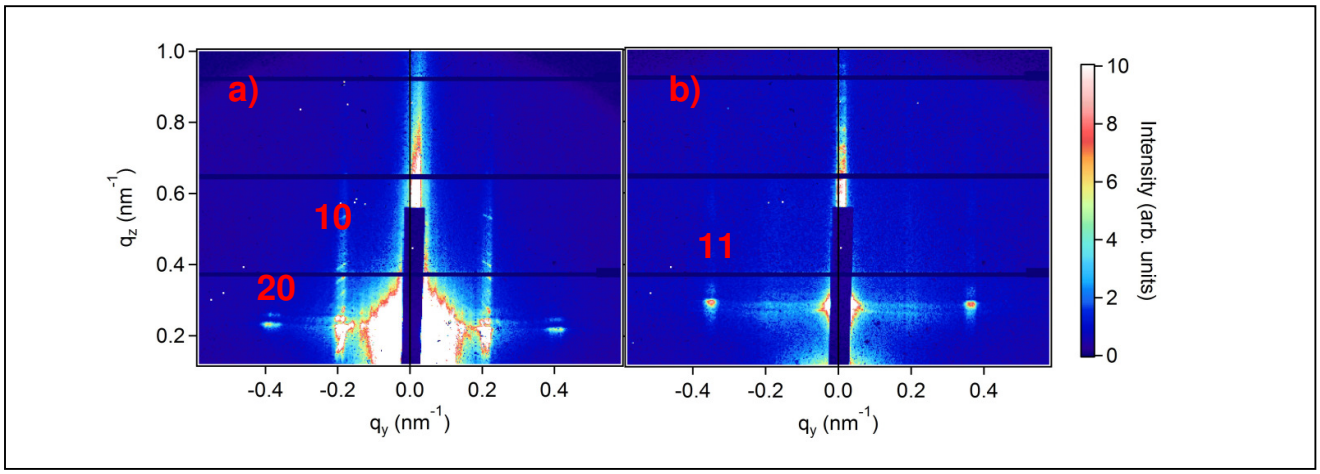

Figure 2: GISAXS patterns of self-assembled diblock (PS-b-PMMA) copolymers on a prepatterned surface of HSQ line grated: a) parallel; b) perpendicular to the incident beam.

To illustrate this series of experiments, figure 2 shows the GISAXS patterns obtained on a cylindrical morphology BCP when the beam direction was parallel to the lines (a) and perpendicular to the lines (b) (i.e. after rotating the sample from 90deg). The sample size was 1x10mm^2, 10mm being the length of the grating lines. The pattern (a) exhibits only the l0 and 20 rods, and the pattern (b) only the 11 rod, confirming the formation of a single microdomain, such the dense rods of cylinders are aligned with the grating lines.