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Report: The directed self-assembly of polystyrene-*b*-polymethylmethacrylate (PS-*b*-PMMA) block copolymers (BCPs) is a potential approach to shrink the dimension of contact holes and multiply the number of contacts¹. In-depth structural characterization of *i*)the guiding pattern (a 2D square lattice of cylindrical holes), *ii*) the cylindrical morphology BCP self-assembled inside the guiding pattern and *iii*) the final contact hole after transfer in a hard mask is crucial to validate the contact shrink (CS) process. For it, grazing incidence small angle x-ray scattering measurements were performed on the CRG-BM02 beamline using a photon energy of 11keV. The probed samples of 20x7mm² size are composed of four juxtaposed areas (5x7mm²) with different hole pitchs (successively 500, 120, 180 and 250nm). The samples were positioned on a Kappa goniometer at a distance of 3080mm from a XPAD 2D pixel detector (960x560 pixels, 130mm pixel size). Accurate alignment of the [10] direction of the square lattice with respect to the direct beam was done. GISAXS patterns were thus recorded for a series of samples representative of the main steps of the CS process with the 4 different pitchs. By way of examples, Fig. 1 shows the GISAXS images obtained for a initial guiding pattern (120 nm pitch and 52nm hole diameter) and after direct self assembling of BCP (intrinsic period of 35nm) and PMMA removal, together with the corresponding SEM images. The sample cross-sections of these two samples are depicted in Fig.2 a and d, respectively.

pitch= 120nm



Figure 1 : (a,b) SEM and GISAXS images for the guiding pattern, at a low incidence angle ($\alpha_i=0.115$ deg) the oscillations of the hole form factors expected along the 10 are blurred by enhanced Bragg rod waveguiding interference effects leading to high frequency beats (in red ellipse). (c,d) after self-SEM and GISAXS image assembling of BCP in guiding holes and for $\alpha_i = 0.22 \text{deg}$, the PMMA removal, modulations (in red ellipse) observed along the rods below the intense dots (intersects between the rods and Ewald sphere) are characteristic of the hole depth and come from grazing incidence transmission small angle x-ray scattering (GTSAXS) effects².

Such GISAXS images reveal complex peculiar features in which the signature of the hole form factors is not straightforward and in particular information about the hole diameter is really difficult to solve due to the contribution of the perfect 2D square guiding array. However, from the intensity profiles drawn along the 10 rod (q_z direction), some information about the different embedded stacked layers in the CS process can be extracted. Figure 2 shows such profiles for a series of samples (pitch:120nm) corresponding to the main stages of the process.



Figure 2: Intensity profiles along the 10 rod extracted from GISAXS images recorded for an incidence angle of 0.22°, i.e. slightly smaller than the critical angle of TiN (0.24°) but larger than those of other layers:SiARC (0.15°), SOC (0.13°), LTO and Si (~0.16°). In inset, schematic cross-sections of the layer stacking around the different holes. The blue arrow indicates the Yoneda peak of the TiN layer.

In presence of the SiARC uppermost top layer, GTSAXS is enhanced and leads to the regular oscillations between 0.15 and 0.25nm⁻¹ stemming from the total thickness of the two upper layers (170nm). From a to e profiles, the features observed on the plateau before the Yoneda peak of TiN correspond to exit angles which are equal to the values of the incident angles at which resonance-enhanced x-ray scattering occurs³. Beyond the Yoneda peak of TiN, slow oscillations characterizing the 20nm-thick TiN layer are only observed, and it is also true in the GISAXS patterns measured with α_i =0.26°. Simulations performed using the IsGISAXS software⁴ have corroborated the absence of the interference fringes stemming from the the form factor of 170nm-deep holes for incidence angles up to 0.24°, but using larger incidence angles, a signature of the cylinder form factor along the Bragg rods should be reached.

Further GISAXS experiments are already planned to perform measurements under incidence angles at least up to twice the critical angle of TiN.

- 1. I. Servin ET al, Jap Journal of Apl; Phys., 53, 06JC05 (2014)
- 2. Lu et al, J. Appl. Cryst., 46, 165 (2013)
- 3. Narayanan et al, Phys. Rev. Lett., 94, 145504 (2005)
- 4. R. Lazzari, J. Appl. Cryst. 35, 406-421 (2002).