| ESRF | **Experiment title:**<br>Tracking sub-micron assembly in single-domain polymer opals | **Experiment number**:<br>26-02-658 |
|---|---|---|
| **Beamline**:<br>BM26B | Date of experiment:<br>from: 27.09.2013 to: 30.09.2013 | **Date of report**:<br>27.04.2014 |
| **Shifts:**<br>9 | **Local contact(s)**:<br>dr. Giuseppe Portale | *Received at ESRF:* |

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The measurements are performed using the photon energy of 12.4 keV. Due to problems with the lens setup and rather low scattering contrast of the opal films, no compound refractive lenses were used in this experiment. Instead, careful beam focusing at the detector position was performed by bending the second crystal of the monochromator and the mirror.

Both single and multi-component polymer opal films processed with different degrees of biaxial bending induced oscillatory shearing (B-BIOS) and uniaxial bending induced oscillatory shearing (U-BIOS) methods have been characterized. In agreement with our preious results (Exp. No. 26-02-573), clear hexagonal patterns were observed in all the sheared samples with different degrees of structure order. Asymmetric intensity distribution between (10) and (1-1) spots was also observed, and it varies depending on different shearing methods and degrees of shearing.

By fitting the diffraction patterns with 2D Gaussian model, detailed analysis (Fig. 1) has been done, which provides insight into how spheres are rearranged with different shearing methods. Both B-BIOS and U-BIOS methods improved the structural order of polymer opals significantly. While integrated intensity of (1-1) spot remains approximately constant, amplitude increases drastically with increasing amount of shearing which is also accompanied with a rapid drop in FWHM. With same amount of shearing, structure order improves more effectively with U-BIOS. FWHM in radial direction (narrow axis of the spot) drops much slower than the hoop direction (long axis of the spot), which indicates orientational order imprves much fast than positional order. Significant difference between (10) spot and (1-1) spot has been observed, which needs further invesitgation. Overall order of the films reaches a saturated level after approximately 40 passes of shearing with both methods, while structural order improves most effectively in the first ten passes.

BIOS method can also be used in fabricating binary or ternary opal structures. Fig. 2 showes clear hcp arrangement in binary polymer opals comprised of spheres with different sizes. Furture work includes characterization of stacking order of different opal films by grazing angle SAXS, *etc*.

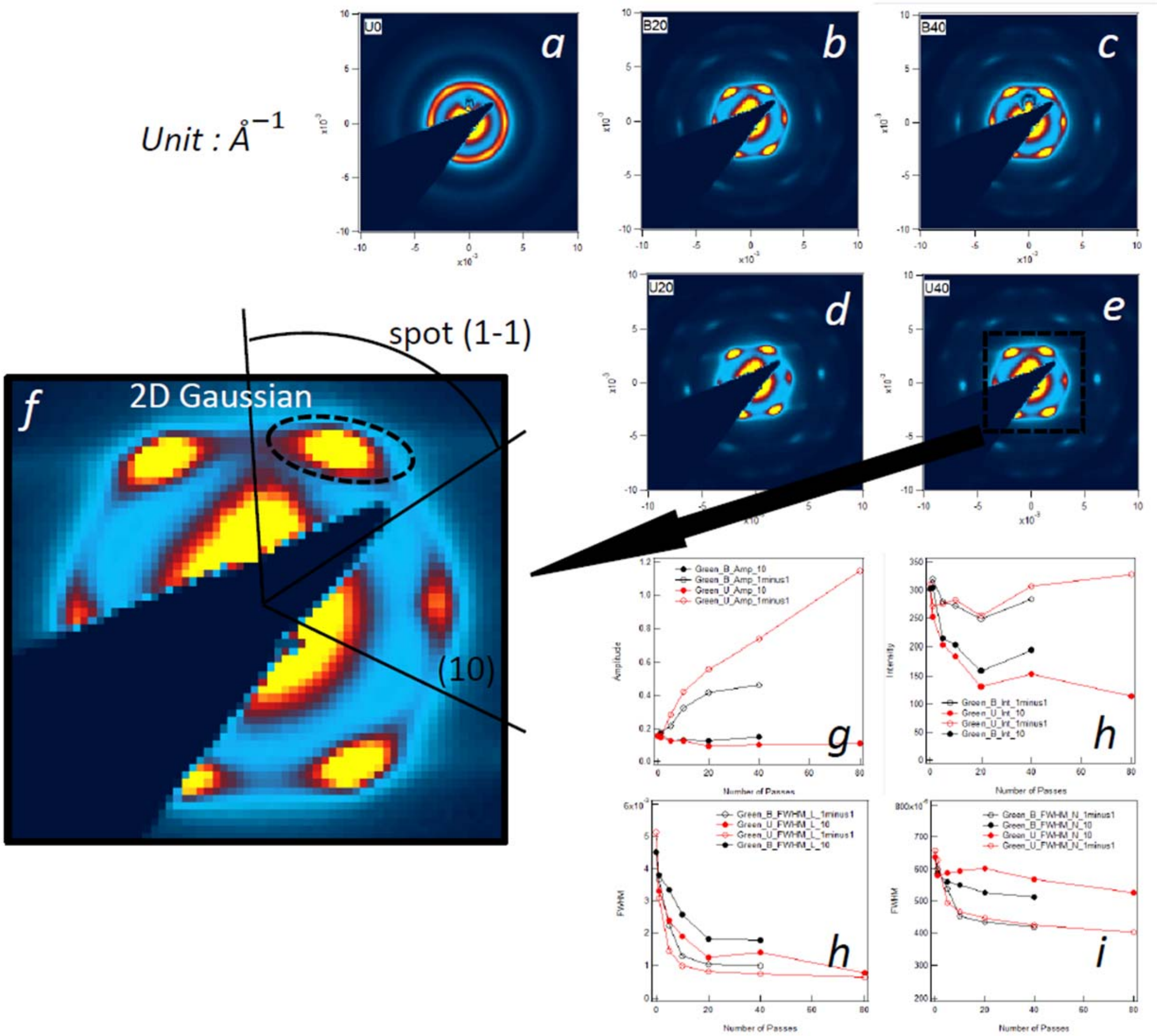


**Figure 1.** 2D SAXS patterns of polymer opals with different shear ordering methods. (a), before shearing, (b), biaxial-BIOS 20 passes, (c), biaxial-BIOS 40 passes, (d), uniaxial-BIOS 20 passes, (e), uniaxial-BIOS 40 passes, (f), zoom in of the first ring in (e), spots are analyzed with 2D Gaussian fit, (g)-(i) amplitude and integrated intensity of (10) spot and (01) spot for different samples with different shearing methods and number of shearing.

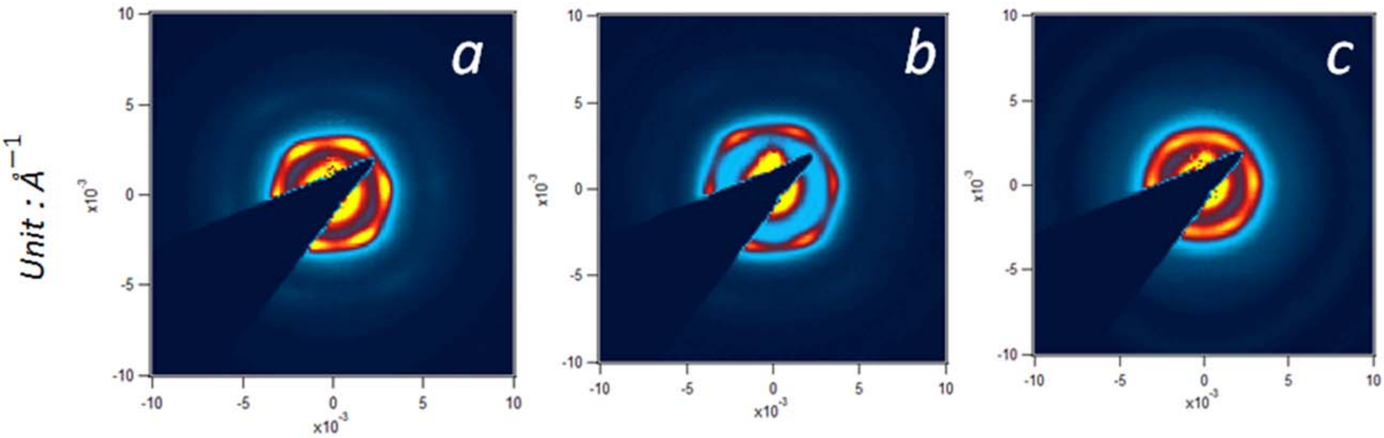


**Figure2.** 2D SAXS patterns of binary polymer opal films ordered with uniaxial 20 passes. (a), $R_L$: $R_s$ $= 1.2$, (b), $R_M$: $R_s$ $= 1.2$, (c), $R_L$: $R_s$ $= 1.4$