Controlling the coassembly of highly amphiphilic block copolymers with a hydrolytic sol by solvent exchange – Supporting Information –

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Full experimental details

Material fabrication. The poly(isoprene-*b*-ethylene oxide) (PI-*b*-PEO) BCP was synthesised by anionic polymerisation as described in reference[1]. The polymer characteristics were studied by gel permeation chromatography and NMR and determined as follows: molecular weight $M_n = 33.5\,\mathrm{kg\,mol^{-1}}$, 23 wt% PEO, polydispersity index PDI = 1.03.

Titanium containing sol was synthesised by quickly adding 0.69 ml of HCl (37 %) into 1 ml of titanium ethoxide (purum) under vigorous stirring at ambient conditions and left age for 90 min.[2] Depending on the inorganic to organic weight ratio, the adequate amount of sol was subsequently mixed with PI-*b*-PEO copolymer in 7 ml THF (99.97 %). This ratio was determined by the weight of the resulting TiO_2 compared to the initial weight of the polymer in solution. For a weight ratio of 2:1 between the resulting TiO_2, 0.87 ml sol was added to 0.1 g BCP. After solvent evaporation in a Petri dish at 50 °C , the dried hybrid material was redissolved in an azeotrope solvent mixture of toluene (apolar, 99.8 %) and 1-butanol (polar, 99 %). Typically a solid weight content of 70 mg polymer per ml was used. The solution was then solvent casted in a covered Petri dish at 40 °C and then left to dry for over 24 hours. The dry hybrid material was subsequently annealed by slow ramping to 130 °C (180 min linear ramp, 30 min dwell).

Transmission Electron Microscopy (TEM). TEM samples of the PI-*b*-PEO/TiO_2 mesostructure were prepared by sectioning the bulk materials to 50 nm with a Leica Ultracut UCT cryo-ultramicrotome and transferring the slices onto copper TEM grids. These samples were imaged in a Tecnai T12 Spirit TEM with an acceleration voltage of 120 keV.

Small angle X-ray scattering (SAXS). SAXS patterns were recorded in the G1 beamline in Cornell High Energy Synchrotron Source (CHESS) with a Flicam 2D CCD detector, with the beam energy at 10.5 keV and a sample-to-detector distance of 2.3 m.

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Supplementary Table S1 | Bragg spacings for investigated morphologies.[3–5].

Sample	assigned morphology	q^*	q_{hkl}			
1:1	inverse hexagonal	0.192	$\sqrt{3}q^*$	$\sqrt{4}q^*$	$\sqrt{7}q^*$	$\sqrt{9}q^*$
1:2	lamellar	0.175	$2q^*$	$3q^*$	$4q^*$	
1:3	hexagonal	0.201	$\sqrt{3}q^*$	$\sqrt{4}q^*$	$\sqrt{7}q^*$	

Supplementary Table S2 | Characteristic properties of employed BCPs.

M_n PI-*b*-PEO	N_TOT [kg/mol]	M_n PEO	N_PEO [kg/mol]	f_PEO	PDI ($= \frac{M_\mathrm{w}}{M_\mathrm{n}}$) [%]
33.5	554	7.7	175	25.8	1.03

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