Supporting Information

Stabilizing the Ordered Bicontinuous Double Diamond Structure of Diblock Copolymer by Configurational Regularity

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GPC curves of iPP-b-PS and the corresponding homopolymers

The average molecular weights and PDI (M_w/M_n) were determined by hightemperature gel permeation chromatography (Waters 150-CALAC/GPC) with a refractive index (RI) detector and a set of U-Styragel HT columns of 106, 105, 104, and 103 pore sizes in series. The measurements were conducted at 135 °C using 1, 2, 4trichlorobenzene as the solvent. PS samples with narrow PDI were used as the standards for calibration. The successful preparation of a structurally well-defined iPP-*b*-PS ($M_{n,iPP} = 5500$ g/mol, $M_{n,PS} = 11500$ g/mol) sample was demonstrated by the GPC analysis shown in Figure S1, which compares the GPC elution curves of the tosyl group end-capped iPP ($M_n = 5500$; $M_w/M_n = 1.39$) and the living anionic PS ($M_n = 11500$; $M_w/M_n = 1.04$) with that of the iPP-*b*-aPS ($M_n = 17000$ g/mol, $M_w/M_n = 1.19$).



GPC curves of (a) iPP ($M_n = 5500 \text{ g/mol}$; $M_w/M_n = 1.39$), (b) PS ($M_n = 11500 \text{ g/mol}$; $M_w/M_n = 1.04$), (c) iPP-block-PS ($M_n = 17000 \text{ g/mol}$; $M_w/M_n = 1.19$) (solvent: 1, 2, 4-trichlorobenzene; temperature: 135 °C).



Figure S2 Temperature-dependent WAXS profiles collected simultaneously with the SAXS data in (a) heating cycle and (b) subsequent cooling cycle. The corresponding degrees of crystallinity are shown in (c). The iPP crystallites melted at 140 °C in the heating cycle, while the crystallization occurred at ca. 100 °C in the cooling process.



Figure S3 SAXS profile of iPP-*b*-PS having been treated by annealing the as-cast film at 150 °C for 24 hrs.