Colloid Templated Mulit-sectional Porous Polymeric Fibers

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Experimental Details

Materials. Sulfate terminated polystyrene (PS) colloids with 8% wt. of 2.5 ± 0.1 and 4% wt. of 10 ± 0.7 µm diameter in aqueous form were purchased from Invitrogen. Polymethylmethacrylate (PMMA) capillary of 50 ± 2.5 µm diameter in a spool of 1 m was purchased from Paradigm Optics. (Heptadecafluoro-1,1,2,2-Tetra-Hydrodecyl) Trichlorosilane was purchased from Gelest. Chloroform was purchased from Fisher Scientific. UV curable polyurethane liquid pre-polymer NOA 60 were purchased from Norland Optics. De-ionized (DI) water was obtained from Millipore Milli-Q unit. Anhydrous stabilized Tetrahydrofuran (THF) and Certified ACS Grade Toluene were purchased and used as is from Acros Chemical and Fisher Scientific, respectively.

Glass Slide Fluorination. Plain microscope slides are placed into a staining dish that is filled with a solution formed using 10 μ L (Heptadecafluoro-1,1,2,2-Tetra-Hydrodecyl) Trichlorosilane

and 20 mL Chloroform. Microscope slides are submersed under the solution for approximately 3 hours. Treated slides are removed from the solution and left to dry in ambient condition for 1 hour. Slides are stored up to 7 days before a retreatment.

Fiber Preparation. Polymethylmethacrylate (PMMA) micro-capillaries (from Paradigm Optics) with circular cross sections and an inner-to-outer diameter ratio of 50/100 µm are used as assembly frame and cut to 20 mm length using a scalpel knife. A 10 µL droplet of the 10 µm colloidal solution is placed onto the bottom end of the 20 mm long PMMA capillary fixed on a fluorinated glass slide. After 20 minutes, the capillary is moved to a new fluorinated glass slide and the bottom end of the capillary is placed into a 10 µL droplet of de-ionized (DI) water to complete and keep the assembly hydrated. Once the completion of the assembly is confirmed with optical microscopy (20 min), the capillary is placed onto a new fluorinated glass slide. The process is repeated once with 2.5 µm and once with 10 µm colloids resulting in a multi-sectional colloidal crystal. The bottom end of the PMMA capillary containing the multi-sectional assembly is fixed onto a clean glass slide. 10 μ L of NOA 60 are placed at the bottom end and allowed to infiltrate the colloidal assembly. After 24 hours of infiltration, the pre-polymer is cured under 365nm long-wave UV light (from Spectroline A14-VS) for 2 hours. The cured *micro-capillary/colloid/polymer* matrix is placed into a beaker with tetrahydrofuran for 15 mins to dissolve and remove the PMMA capillary and the PS templating colloids. Repeated solvent dips and washes are performed if needed.

Imaging. Optical images are taken using the Olympus Vanox Microscope and scanning electron microscope images are taken using the Zeiss EVO-40 instrument in variable pressure mode (P = 35-40 Pa). Variable pressure SEM allows imaging of non-conducting materials without the requirement for gold coating enabling the analysis of surface pores.



Figure S1 shows a microscope image of the two interfaces I_1 and I_2 produced during alternating colloidal assembly of 10 µm, 2.5 µm, and 10 µm colloids within a 50 µm PMMA capillary. The strongly tilted angle of the I_1 interface caused by sedimentation of the 10 µm particles is clearly visible. I_2 is much less tilted.



Figure S2 depicts cross sectional SEM views of different sections/interfaces taken from several fibers. The images clearly show that the fibers are porous throughout the fiber cross section. The samples are prepared for cross sectional analysis by dipping the fibers into liquid N_2 and subsequent pulling until the fiber breaks.