

Supplemental Information for “Control of the length of microfibers”

Janine K. Nunes, Krzysztof Sadlej, Jee Ian Tam and Howard A. Stone*

Microfluidic Device Fabrication:

A 15:1 PDMS base to crosslinker ratio was used to ensure that the elastomeric membranes (approximately 38 μm in width) separating the air and monomer channels were sufficiently flexible for actuation. The PDMS microchannel replica was bonded to PDMS-coated cover glass slides so that all four walls of the microchannels were PDMS, which provided an inhibition layer that reduced polymer sticking and channel clogging.¹⁻² The channel height was 120 μm , which we found produced cylindrical fibers without channel clogging.

Viscosity and Interfacial tension measurements:

The viscosities of the monomer solution (54 vol% poly(ethylene glycol) 400 diacrylate (PEG-DA, Polysciences), 42 vol% de-ionized water and 4 vol% Darocur 1173 photoinitiator (Ciba)) and the oil phase (hexadecane with 4 vol% Span 80) were measured using Cannon-Fenske glass viscometers (#50 for the oil phase and #100 for the monomer solution). The results were confirmed by measuring the viscosities with an Anton Paar Physica MCR 301 rheometer. The viscosities were found to be 3.85 ± 0.03 and 12.96 ± 0.06 mPa.s for the oil and monomer phases, respectively. The interfacial tension, γ , between the oil and monomer phases was measured using the pendant drop method. γ was measured to be 0.61 ± 0.01 mN/m.

Valve conditions:

The minimum air pressure necessary to break the monomer jet was found to be approximately 30 psi, but this value was found to vary from device to device due to small variations in the modulus of the PDMS used to make the devices. Below 30 psi, the monomer jet did not break, but did exhibit a decrease in diameter when air filled the chambers. For the experiments presented, the compressed air regulator was set to a value of 40 psi to ensure a clean pinch off between monomer segments.

The solenoid air control valve (McMaster-Carr, product # 4916K64) was controlled by a function generator (Stanford Research System, Model D5345, 30 MHz Synthesized Function Generator). A sawtooth waveform was used, and the peak to peak voltage was set to 4.5 V, with an offset of 2.25 V. The frequency was varied from 1 – 5 Hz. These conditions produced sharp rapid pulses that were required to break the monomer jet.

Fiber Collection:

To clearly measure the length of the fibers, it was necessary to keep the individual fibers fairly straight and untangled. For this purpose we designed long serpentine microchannels with periodic constrictions, which aided in keeping most of the fibers reasonably elongated (Fig. S1). The output from the valve-controlled microchannel (fibers, oil and any unreacted monomer) was collected in the 8 mm well of the collection microchannel (Fig. S1A), which contained a small amount of ethanol. The fiber suspension was pulled through the device to align the fibers in the microchannel. The bright-field image in Fig. S1B

shows how fibers (valve frequency: 3.5 Hz) were collected for imaging. For each valve frequency (1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5 Hz), the lengths of the fibers were measured. The fiber length, which was taken as the mean of about 20 measurements of different fibers for a given frequency, was then plotted as a function of 1/valve frequency. The standard deviation for each length was also determined for error bars.

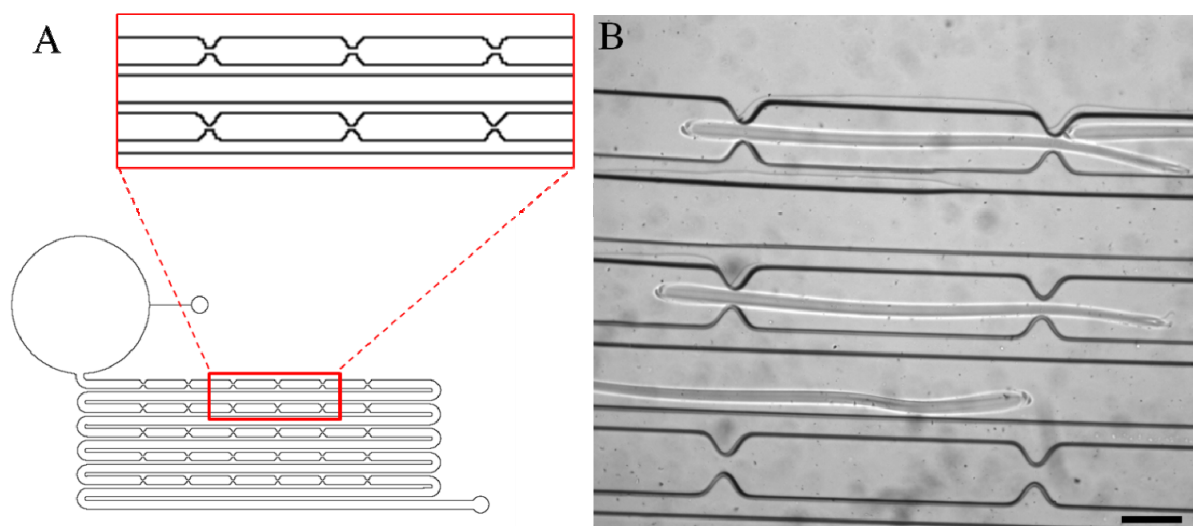


Fig. S1. (A) Schematic of fiber collection microchannel, and (B) micrograph of fibers collected in the microchannel (scale bar: 500 μm).

Small diameter microfibers fabricated with UV pulses:

Small diameter PEG fibers were synthesized from a monomer solution composed of 54 vol% poly(ethylene glycol) 400 diacrylate (PEG-DA, Polysciences), 42 vol% de-ionized water and 4 vol% Darocur 1173 photoinitiator (Ciba). Using PDMS microchannels with main channel dimensions of 300 μm width and 120 μm height, the flow rates were 30 $\mu\text{L}/\text{min}$ for the oil phase and 14 $\mu\text{L}/\text{min}$ for the monomer phase. These conditions produced 30 μm diameter fibers. The shutter was set to open for 100 ms then close for 500 ms. The synthesized fibers are shown in Figure S2A.

Triacrylate fibers were synthesized from a viscous monomer solution composed of 96 vol% trimethylolpropane triacrylate (Sartomer) and 4 vol% Darocur 1173. The viscosity of the monomer solution was measured to be 86.5 mPa.s. Using PDMS microchannels with main channel dimensions of 600 μm width and 120 μm height, the flow rates were 80 $\mu\text{L}/\text{min}$ for the oil phase and 4 $\mu\text{L}/\text{min}$ for the monomer phase. These conditions produced 38 μm diameter fibers. The shutter was set to open for 100 ms then close for 500 ms. The synthesized fibers are shown in Figure S2B.

Tapered ends were observed for both compositions.

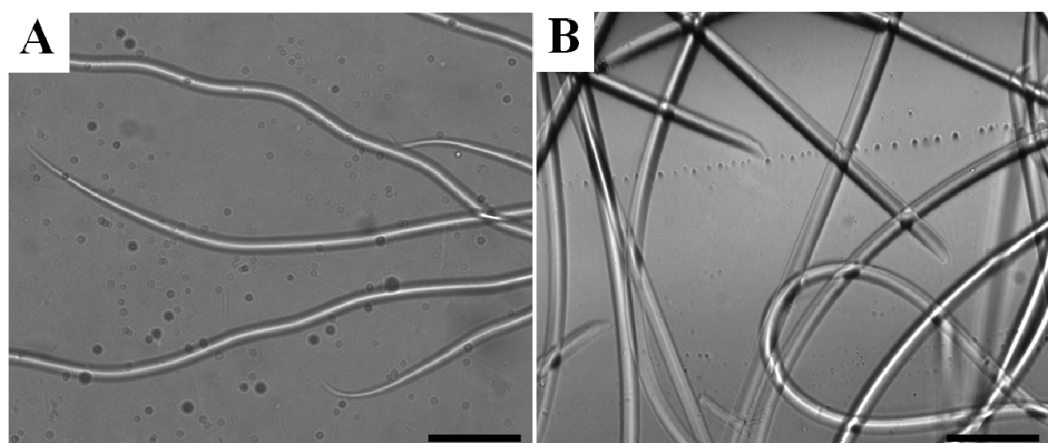


Fig. S2 Bright-field images of the ends of small diameter fibers formed using pulses of UV light: (A) 30 μm diameter cross-linked PEG-DA fibers and (B) 38 μm cross-linked triacrylate fibers (scale bar: 200 μm).

References:

1. D. Dendukuri, P. Panda, R. Haghgooie, J. M. Kim, T. A. Hatton and P. S. Doyle, *Macromolecules*, 2008, **41**, 8547.
2. D. Dendukuri, D. C. Pregibon, J. Collins, T. A. Hatton and P. S. Doyle, *Nat. Mater.*, 2006, **5**, 365.