

# Electronic Supplementary Information (ESI)

## Rapid prototyping of three-dimensional microfluidic mixers in glass by femtosecond laser direct writing

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## 1. Mesoporous glass preparation

The mesoporous glass was fabricated by removing the borate phase from a phase-separated alkali-borosilicate glass in hot acid solution. Reagent-grade chemicals of  $\text{Na}_2\text{CO}_3$ ,  $\text{SiO}_2$  and  $\text{H}_3\text{BO}_3$  were used as starting materials. The chemical composition of initial glass was 9 $\text{Na}_2\text{O}$ -26 $\text{B}_2\text{O}_3$ -65 $\text{SiO}_2$  (mol%). After thorough mixing, the powders were melted in a platinum crucible at 1400 °C for 90 min. The subsequent heat treatment was carried out at 600 °C for 40 h for the development of phase separation. The obtained phase-separated alkali-borosilicate glass was cut into pieces with a size of 10 × 10 × 3 mm<sup>3</sup>, and polished, afterwards they were leached by 1 mol/L  $\text{HNO}_3$  at 100 °C for 72 h and then the mesoporous glasses were obtained. The composition of the porous glass obtained was 95.5 $\text{SiO}_2$ -4 $\text{B}_2\text{O}_3$ -0.5 $\text{Na}_2\text{O}$  (wt.%). The pores with a mean size of ~10 nm are distributed uniformly in the glass and occupy 40% volume of the glass. Particularly, these pores in the porous glass form a 3D connective network which allow liquid to flow through by infiltration.

## 2. Fabrication details of 3D helical microchannel

A regeneratively amplified mode-locked Ti:sapphire laser (Coherent, Inc.) with a pulse duration of ~50 fs, a central wavelength of 800 nm and a repetition rate of 250 kHz was used to produce the microchannels inside the mesoporous glass. The initial 8.8 mm diameter beam was reduced to 5 mm by a circular aperture so as to guarantee a high beam quality. The energy of the beam was controlled by using a combination of polarizer and waveplate and a set of neutral density filters. The sample was fixed in a petri dish filled

with distilled water, which can be translated by a computer-controlled XYZ stage with a resolution of 1  $\mu\text{m}$ . A 100 $\times$  water-immersed objective (NA=1.0) was employed for focusing the beam into the sample at a depth of ~200-300  $\mu\text{m}$  below the top surface. The laser pulse energy was chosen to be 2  $\mu\text{J}$  and the chopper was set at 1 kHz with a duty of 50/50. The hollow helical channel was fabricated by direct writing inside the mesoporous glass following a helical track (see the movie attached in the Electronic Supplementary Information (ESI)). The debris remained in the microchannel was removed by repeated multiple scans. A homogeneous and debris-free channel with a length of ~ 1 cm can be obtained by one slow scan at a translating speed of 10  $\mu\text{m}/\text{s}$  followed by three fast scans at a translating speed of 100  $\mu\text{m}/\text{s}$  over the whole microfluidic channel.

### **3. Fabrication details of 3D micromixer**

In order to obtain larger microchannels, a 50 $\times$  water-immersed objective (NA=0.80) was employed for focusing the beam into the sample at a depth from 200 to 500  $\mu\text{m}$  below the top surface, and the laser pulse energy was chosen to be 20  $\mu\text{J}$ . The outlets and inlet of the mixer was firstly machined from the rear surface of substrate as the exit of the debris. All the vertical channels in the mixer were machined by direct writing parallel to the laser beam, while the horizontal channels was written along the direction vertical to the laser beam. In order to remove the debris in microchannel, three fast scans at a translating speed of 100  $\mu\text{m}/\text{s}$  was performed after the first slow scan at a translating speed of 2  $\mu\text{m}/\text{s}$ . The total machining time is about 60 minutes.

#### **4. Numerical simulations of mixing results**

The numerical simulations of mixing efficiencies of the 1D and 3D mixers were carried out by solving the microfluidic incompressible Navier–Stokes and convection diffusion equations using COMSOL Multiphysics 3.5 (COMSOL Inc., Burlington, MA) software. The geometric modes were set to match the fabricated structures, and the two fluids were assigned to two inlets respectively with a density of  $998 \text{ kg/m}^3$ , a viscosity of  $1.002 \text{ mPa s}$ , and an isotropic diffusivity coefficient of  $10^{-10} \text{ m}^2/\text{s}$ . The boundary conditions were as follows: a flow rate of  $8.8 \text{ mm/s}$  for the inlets, a static pressure of  $0 \text{ Pa}$  for the outlets, and a flow rate of  $0 \text{ mm/s}$  for the channel walls.

#### **5. Movie of the direct writing process**

Movie 1: Real-time video clip showing the process of direct writing of 3D helical microchannel in mesoporous glass