Capillary-Assembled Straight Microfluidic Devices †

Mert Arca a, Xuhui Feng a, Anthony J. C. Ladd a, and Jason E. Butler * a

Received Xth XXXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
First published on the web Xth XXXXXXXXXX 200X
DOI:

A lithography-free method to produce microfluidic channels for imaging applications is described. Silica microcapillaries, with either circular or square cross-sections, are embedded in the surface of a polydimethylsiloxane (PDMS) base for imaging studies of flow and electrokinetics within a straight channel. Successful production of the devices relies on precisely timing the assembly process. This timing is identified by an in-situ elasto-capillary thinning experiment on PDMS; a rheological study supports the use of these measurements. The assembled devices are characterized using atomic force microscopy (AFM) and bright field microscopy.

Straight microfluidic devices are commonly used for imaging single molecules, particulates and, droplets under pressure driven flow 1 and can include electric fields for generating electrophoretic and electroosmotic flows 2. Many methods for fabricating microfluidic channels for imaging applications exist 3,4, though these methods can be expensive and require significant expertise. However, if the goal is to study flows in straight channels, complex methods for constructing microfluidic devices may be unnecessary.

We present a simple method to assemble straight microfluidic devices by embedding glass capillaries in the surface of a polydimethylsiloxane (PDMS) base. The approach takes advantage of inexpensive materials to produce a straight channel suitable for imaging applications. Assembly can be performed using commonly available equipment and the final device has several advantageous features, including mechanical stability.

Primary materials used in constructing microfluidic devices include glasses (borate silica, fused silica) or polymeric materials such as PDMS, polymethyl methacrylate, and polycarbonate 3,5. Soft lithography is frequently used for fabricating polymeric-based devices because of the relative ease and low cost of production. However, devices produced by soft lithography suffer from mechanical instability, as the bond between the glass substrate and polymer can fail 6. Additional shortcomings include poor compatibility of the polymers with organic solvents 7,8, clogging due to absorption of organic material on the hydrophobic surfaces 7,8, and deformation of the channel dimensions and flow fields upon application of pressure gradients 9. In contrast to PDMS and other polymers, silica-based devices offer better mechanical stability and surface properties, but at a substantially increased cost due to the complex methods of production 3.

Silica based microfluidic devices can be produced with laser etching 10, which requires significant capital investments, but bare silica capillaries can also be used 1. However, positioning these microcapillaries and making connections for fluid pumping and electrokinetics experiments is difficult due to their small size. Securing capillaries with an adhesive can result in bends and misorientation of the final assembly.

The concept of stabilizing silica-based microcapillaries by embedding them within a PDMS network was introduced in 2004 11, when two functional capillaries were placed into a PDMS lattice. These capillary-assembled methods require a PDMS template made by soft lithography that would match the outer dimensions of the capillaries. Embedding circular cross-sections in a PDMS template is not achievable by the
The rheological phase angle of PDMS with respect to time during polymerization at 50°C. The rheology of PDMS transitions from viscous (region a) to strongly elastic (region c) due to polymerization. Introducing the capillary during the transition period (region b) results in the final depth and alignment shown in Fig. 1, without the need to manually control either parameter.

Here we introduce a simple way to construct silica-capillary based microfluidic devices without the need for soft lithography. Inexpensive silica microcapillaries are readily available in different geometries (square, rectangular, and cylindrical) with inner dimensions as small as 20 μm. Embedding microcapillaries on the surface of a block of PDMS facilitates their use in imaging experiments without introducing bending or orientation problems.

Figure 1 shows a schematic of the device: a glass slide, a PDMS mount, a silica microcapillary, and tubing. The assembly process begins by preparing the PDMS (Slygard 184 silicone elastomer kit). After mixing the base and curing agent with a 10:1 ratio, the mixture is degassed in a vacuum chamber at 0.1 torr to remove any air bubbles and then poured onto a flat plate to a depth of approximately 2 mm. The flat plate should be preheated and maintained at a temperature of 50°C to promote polymerization. The silica microcapillary is placed on top of the PDMS mixture 25 ± 5 minutes after initiating the polymerization process. There is no need to actively control the alignment or other details of the placement of the capillaries. The channels used here included square and circular cross-sections (Wale Apparatus, PA). Square channels used in these experiments have inner dimensions of 50 and 80 μm and outer dimensions of 100 and 160 μm, respectively; the cylindrical channels have a 100 μm inner and 170 μm outer diameter. The system is left to polymerize for an additional six hours after placing the microcapillary on the PDMS surface. Then the PDMS block is lifted from the heated plate and attached to a silica microscope slide with plasma treatment. Inlet and outlet tubing is attached by cutting 1 cm of PDMS and sealing the connection with silicone.

The channel will end up embedded at the PDMS-air interface, as diagrammed in Fig. 1. It is strongly affixed to the PDMS base, but the position enables unobstructed imaging experiments. To attain this configuration, the channel must be put on the surface of the PDMS while the rheology transitions from a viscous (liquid-like) to elastic (solid-like) behavior. Placing the microcapillaries on the PDMS too soon, when the material is viscous, will result in the sedimentation of the microcapillaries through the PDMS. In this case, PDMS will fully cover the channel and fill the capillary. If placed on the PDMS after the material has become strongly elastic, the microcapillary will remain on top of the surface and will not be attached to the PDMS.

An in–situ experiment identifies the critical time, within a ten minute window specified above, to place the microcapillary on the surface of the polymerizing PDMS. For this test, a needle is dipped to the bottom of the PDMS and then elevated 1 cm above the surface. PDMS with the right viscoelasticity will form a thin filament that persists for approximately three seconds (see electronic supplementary information†). During the early stages (prior to 20 minutes) of polymerization, no filaments will form; in the later stages, the polymer will exhibit solid-like behavior and the surface will not return to its initial shape after being deformed by the insertion of the needle.

This simplified test of the elasto-capillary thinning behavior suffices to identify the time of placement when assembling the channels. The timing of the microcapillary placement has been done by measuring the viscoelasticity of PDMS during polymerization. An ARES LS-1 strain controlled rheometer (TA Instruments) was used with a cone-and-plate geometry at a temperature of 50±1°C, to match the polymerization temperature during embedding of the capillary. Oscillatory shear was performed at a constant frequency of 50 rad/sec and a strain amplitude of 25%. Figure 2 shows the phase angle, a measure of the overall viscoelastic nature of the sample, as a function of time from the first contact of the polymer with the heated plate. The phase angle remains close to 90° for approximately 20 minutes, indicating that the mixture is primarily viscous. After 30 minutes, the fluid exhibits significant signs of elasticity, when the phase angle declines to approximately 75° and then continues to drop. The channel should be placed on the PDMS surface during the ten minute window (region b in Fig. 2) when the fluid transitions from a viscous fluid to a strongly elastic one. The timing observed...
from the rheological experiments correlates with the timing determined from the needle-test described above.

To confirm the conceptual picture shown in Fig. 1, additional measurements were made to verify the position of the channel with respect to the PDMS-air interface using Atomic Force Microscopy (AFM) and the orientation of the square channels using bright field imaging. The AFM experiments were performed on an Asylum Research MFP3D-Bio instrument with an AC240TS cantilever. Bright field images were collected using a QImaging Retiga SRV CCD camera attached to a Nikon diaphot 200 inverted microscope with a Leitz Wetzlar Extra Flat (63X/0.85) objective.

Figure 3 demonstrates that the microcapillaries are positioned on the PDMS-air interface, with 1 to 4 µm of the silica capillary protruding through the surface. Figures 3a and b show microscope images of a cylindrical and a square channel embedded in PDMS and marks the regions on the surfaces that were interrogated using AFM. Figures 3c and d show the topography and the phase angle; the topography reveals the height of the features and the phase angle differentiates the material (PDMS or silica) exposed on the surface. The curvature of the cylindrical microcapillary (Fig. 3c) extends a distance of 4 µm above the planar surface of the PDMS, where the curved glass region is identified by the larger phase angle as compared to the surrounding PDMS surface. Likewise, Fig. 3d shows that the top plane of the square microcapillary is exposed to the atmosphere and sits approximately 1 µm above the surface of the PDMS.

No active method is necessary to control the alignment of the square microcapillaries, although the quality of the images depends upon the upper face of the capillary laying coplanar with the surface of the PDMS. Figure 4a shows a microscope image and schematic from a channel that was (purposefully) oriented at a relatively large angle of θ = 15°, which obscures the ability to see near-wall phenomena.

The tilt angle and width of sample channels were calculated from ten images each along the length of five capillaries having an inner width of 50 µm and twelve of 80 µm. The angle θ and width H₀ can be determined from measurements of \( M₁ = H₀ \cos θ \) and \( M₂ = H₀ \sin θ \) for each microscope image (see Fig. 4a). The conversion from pixels to physical lengths was made using a calibrated standard, which enables the measured H₀ to be compared to the width H reported by the manufacturer.

The standard deviation in θ, measured along each channel is less than 2°, which is most likely due to measurement errors. The average tilt in the width in Fig. 4c is 7°, which adds only 1% to the error. The standard deviation in the width observed along the channel is less than 2%. The overall uncertainty in the channel width, including the effects of the tilt, is 2% (Fig. 4b dotted line), much less than the tolerance ±10% specified by the manufacturer.

Channels placed with the right timing orient parallel and flush to the surface of the PDMS. A square silica capillary orients with respect to the vertical through buoyancy forces. Recently, we found that a smaller tilt angle (3° on average) can be achieved by more carefully leveling the table on which the device is being assembled. However, the mechanism by which
the capillary is held so that it is just breaking the PDMS surface is not clear. We have observed that channels coated with PDMS prior to the embedding sink to the bottom. Presumably, the low wettability of PDMS on silica prevents it from covering the capillary, and the elastic stress stops it sinking. However, this is not a transient phenomena requiring precise timing of the placement of the capillary. Further attention needs to be given to understanding the physics behind the reproducible positioning of the channel.

Embedding materials during the visco–elastic transition in PDMS polymerization has not been considered before. More complex microfluidics devices can be constructed by this process. For example, multiple channels can be embedded, and connected on a PDMS surface. Interestingly, if the embedded channel is removed from the surface, the form of the channel remains imprinted in the PDMS. This could facilitate manufacture of PDMS channels with semicircular cross sections or silica–PDMS hybrid channels.

A simple and inexpensive method to produce straight silica microcapillary devices for imaging has been introduced, which is suitable for either rectangular or circular cross-sections. The similarities in the refractive index of PDMS and silica enables imaging of cylindrical microcapillaries embedded at the PDMS surface. Placement of the channels at the PDMS-air interface allows the objective to focus at a working distance less than 50 μm. The embedded microchannels have the advantages of silica based devices, but cost less than $2 to make. The method has potential applications to lithography–free PDMS–silica devices.

**Acknowledgments**

The authors acknowledge Dr. Megan A. Hahn at the Particle Engineering Research Center at the University of Florida for conducting the AFM experiments. This work was supported by the National Science Foundation (Grant No. 1067072).

**References**