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# Fast and inexpensive method for the fabrication of transparent pressure-resistant microfluidic chips

**Alexandre Martin[1] · Sébastien Teychené[1] · Séverine Camy[1] · Joëlle Aubin[1]**

**Abstract** The recent rise of high-pressure applications in microfluidics has led to the development of different types of pressure-resistant microfluidic chips. For the most part, however, the fabrication methods require clean room facilities, as well as specific equipment and expertise. Furthermore, the resulting microfluidic chips are not always well suited to flow visualization and optical measurements. Herein, we present a method that allows rapid and inexpensive prototyping of optically transparent microfluidic chips that resist pressures of at least 200 bar. The fabrication method is based on UV-curable off-stoichiometry thiol-ene epoxy (OSTE+) polymer, which is chemically bonded to glass. The reliability of the device was verified by pressure tests using $CO_2$, showing resistance without failure up to at least 200 bar at ambient temperature. The microchips also resisted operation at high pressure for several hours at a temperature of 40 °C. These results show that the polymer structure and the chemical bond with the glass are not affected by high-pressure $CO_2$. Opportunities for flow visualization are illustrated by high-pressure two-phase flow shadowgraphy experiments. These microfluidic chips are of specific interest for use with supercritical $CO_2$ and for optical characterization of phase transitions and multiphase flow under near-critical and critical $CO_2$ conditions.

**Keywords** Microchip · Fabrication · High pressure · Supercritical $CO_2$ · Visualization

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## 1 Introduction

Pressure-resistant microfluidic chips have become of increasing interest in recent years for flow chemistry (Benito-Lopez et al. 2008; Verboom 2009; Razzaq et al. 2009; Trachsel et al. 2008), processes involving supercritical fluids (Benito-Lopez et al. 2007; Marre et al. 2012; Luther and Braeuer 2012), materials science (Marre et al. 2012) and also as analysis tools (Benito-Lopez et al. 2005; Keybl and Jensen 2011; Dorobantu Bodoc et al. 2012; Pinho et al. 2014; Macedo Portela da Silva et al. 2014; Gothsch et al. 2015; Blanch-Ojea et al. 2012; Zhao et al. 2013; Ogden et al. 2014). Indeed, it is well known that operating processes at high pressure modify fluid properties, which may enable enhanced kinetics, conversion and selectivity (Verboom 2009; Benito Lopez et al. 2007; Kobayashi et al. 2005). In addition to this, miniaturization of these chemical systems further enhances mass and heat transfer and provides improved control of in situ conditions and process safety, which is particularly important for high-pressure processes (Hessel et al. 2004).

With the emergence of high-pressure applications in microfluidics, the development of highly resistant microfluidic chips has been the focus of a number of studies. Among these, glass/glass (Tiggelaar et al. 2007) and silicon/glass microreactors (Trachsel et al. 2008; Murphy et al. 2007; Marre et al. 2010) have shown to resist pressures of up to 400 bar. The microchannels are constructed using either wet or dry etching methods and then are sealed to glass via anodic bonding. These microchips have the advantage of resisting high pressures and also allow optical access to the flow for in situ characterization. In addition, silicon/glass chips have the asset of enabling very good temperature control due to the high thermal conductivity of the silicon wafer (Marre et al. 2010). However, the fabrication methods employed require clean room facilities,

specific equipment and expertise, and entail relatively high operating and fabrication costs, which are limiting when rapid prototyping is required. Furthermore, silicon/glass chips may be limited for some optical measurement techniques due to the opaque and reflective nature of the silicon wafer.

Some simpler glass devices based on fused silica capillaries have also been proposed for high-pressure applications (Macedo Portela da Silva et al. 2014; Marre et al. 2009). Marre et al. (2009) showed the use of silica capillaries under pressure conditions ranging from 80 to 180 bar. Macedo Portela da Silva et al. (2014) demonstrated that operating pressures up to 300 bar could be achieved by embedding silicon capillaries in epoxy resin. Both of these studies also showed that two-phase flow can be easily visualized and characterized by shadowgraphy in such capillaries. However, more elaborate optical characterization techniques, such as micro particle image velocimetry, would be limited in these devices because of problems related to optical distortion, poor light transmission and focal depth.

Rapid prototyping methods based on polymer molding and milling are very popular in the academic microfluidics community because they are inexpensive, fast and easy to implement. They also allow the fabrication of planar geometries in transparent materials, which facilitate the use of optical visualization and measurement techniques. Polydimethylsiloxane (PDMS) is the most widely used polymer for such applications; however, its low Young's modulus means that it is very elastic and as a result, the microchannels are deformed under high-pressure or high-flow rates. It has also poor solvent compatibility so applications are limited. Recently Sollier et al. (2011) have analyzed the performance of other rapid prototyping polymers, including thermoset polyester (TPE), polyurethane methacrylate (PUMA) and Norland Adhesive 81 (NOA81), for high-pressure injections. They showed that PUMA and NOA81 chips resisted pressures up to approximately 8 bar and 5 bar, respectively, before leakage or delamination. The maximum pressure resistance of their setup made of NOA81 is considerably lower than the 15 bar reported by Bartolo et al. (2008). The TPE chips, on the other hand, resisted the maximum pressure tested of approximately 10 bar without any mechanical failure. TPE, PUMA and NOA81 were also shown to have optical transmission similar to glass and have good solvent compatibility compared with PDMS.

Recently, Carlborg et al. (2011) have developed a rapid prototyping method based on off-stoichiometry thiol-enes (OSTEs), which allow excellent adhesion to silicon, glass, many metals or even another layer of this polymer, due to covalent bonds and offer very good optical transmission. It also allows surface modifications, tunable mechanical properties and reliable sealing with other substrates, and the authors have since demonstrated a number of custom specificities (Sandström et al. 2015; Saharil et al. 2012; Haraldsson et al. 2014). OSTE has also shown compatibility with solvents such as toluene, water, acetone, ethanol, methanol and dimethyl sulfoxide (www.ostemers.com). However, up until now, pressure resistance has not been the objective of their studies and the chips have only been shown to resist pressures in the range of 4–7 bar (Sandström et al. 2015; Saharil et al. 2013).

In this article, a new method for the fabrication of microchannels made of UV-curable OSTE (OSTEMER Crystal Clear) and glass, compatible with possible utilization with supercritical carbon dioxide, is presented. The pressure resistance of the microfluidic chips is demonstrated by flowing $CO_2$ at high pressure through the microchannel. The method enables inexpensive rapid prototyping without the requirement of any highly specific microfabrication equipment. The resulting microfluidic chips have the advantage of being optically transparent and planar, thereby being well suited to visualization via shadowgraphy, and particularly to laser-based methods like micro particle image velocimetry and optical measurement techniques based on light transmission or reflection, such as spectrophotometry (UV–Vis, IR), that benefit from planar surfaces. Indeed, this fabrication method provides the possibility to perform multiphase flow visualization studies in microchannels—that have been largely investigated at ambient pressures (e.g., Garstecki et al. 2006; Gupta and Kumar 2010; Abadie et al. 2012; Zaloha et al. 2012; King et al. 2007; Wang et al. 2015)—under high-pressure conditions.

## 2 Experimental

### 2.1 Microchannel geometry

The microchannel fabricated in this study is a 200 μm square cross-sectional channel with a meandering topology that has a total length of 12.5 cm as shown in Fig. 1. The two inlets are connected by a T-junction inside the microchip structure.

### 2.2 Fabrication method of microchannels and assembly of chips

The microfluidics chips are fabricated with a soft lithography method, which uses OSTEMER Crystal Clear. OSTEMER Crystal Clear is a polymer used for quickly building optically transparent, hard and robust microstructures using inexpensive laboratory equipment. This polymer is based on the off-stoichiometry thiol-ene-epoxies and features a

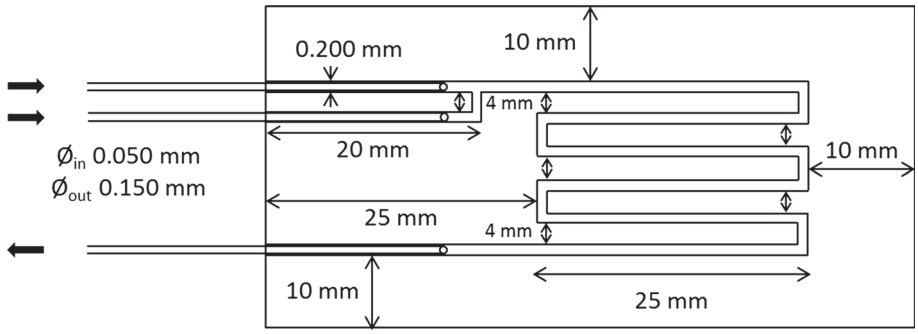


**Fig. 1** Schematic diagram with dimensions of the microchannels fabricated in this study. $Ø_{in}$ and $Ø_{out}$ denote the diameters of the inlet and outlet capillaries, respectively

two-step curing process. It is initially shaped using UV-casting and is then hardened and bonded to itself—or other materials—via a second thermal cure. The first cure geometrically defines the polymer into a micropatterned intermediate stage, but a predetermined amount of epoxy groups remain unreacted. In a second curing step, all the remaining epoxy groups are reacted to form an inert polymer, which stiffens and bonds covalently to most other surfaces that it is in contact with, similarly to epoxy glues.

The fabrication method presented here is based on that previously published by Carlborg et al. (2011) and has been modified in order to create microfluidic chips that allow high-pressure operation. The fabrication method is comprised of several steps, as shown in Fig. 2.

The principal differences with this new method, compared with the original method provided by Carlborg et al. (2011), are:

- The use of a 2-mm-thick glass slide as a base to which all of the other chip layers are strongly bonded;
- The use of a thin (and hence slightly flexible) silanized cover slip to seal the microchannel. This ensures very good contact between the surfaces and improved bonding strength.
- The reinforcement of the sealed microchannel with an additional layer of OSTEMER and a final 2-mm glass slide which also provides a planar top surface for possible optical measurements.

The specific details of the fabrication procedure are provided below in Fig. 2. Note that since some of the materials used in this protocol (i.e., photoresist dry films and OSTEMER Crystal Clear) are UV sensitive, the microchip fabrication was carried out in a UV-protected room.

*Dry film fabrication (steps 1–4)* Two 100-μm layers of a dry film photoresist (WBR2100 DuPont, France) are laminated one-by-one on a glass slide using a heated roll laminator (MEGA Electronics). The glass slide with the dry film is then heated for 20 min at 60 °C. The multilevel dry film is then exposed to UV LED light through

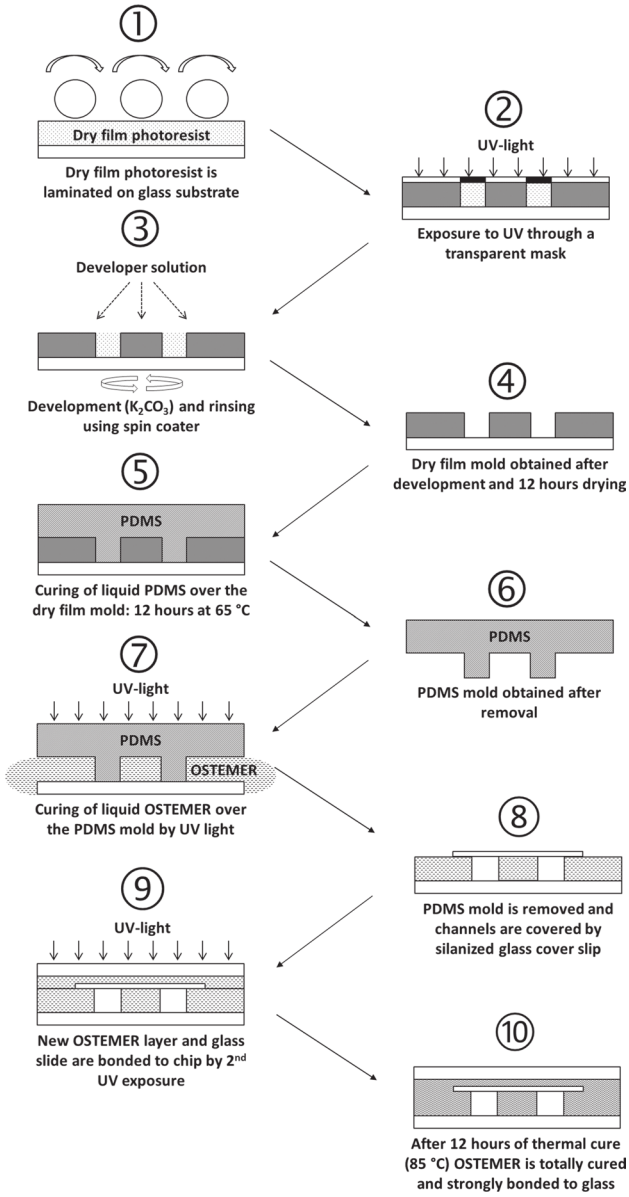


**Fig. 2** Schematic overview of microchannel fabrication. *1–4* Dry film mold fabrication. *5–6* PDMS mold fabrication. *7–10* Molding of OSTEMER and sealing of the microchannel

a photolithography mask using a UV-KUB 2 exposure masking system (Kloe, France) for 19 s at 25 mW/cm$^2$ to obtain the desired channel topology. This time is sufficient to cure the thickness of the dry film without degrading it due to over exposure. Following this, the glass slide is put on a heated plate for 60 s at 100 °C. The dry film mold is developed using a potassium carbonate solution (1 % w/w), which is applied continuously using a spin coater (SPS Spin 150) for 6 min at 900 rpm. The mold is then rinsed with distilled water using the same spin coating procedure as for the developer. The mold is thoroughly dried with compressed air and baked for 60 s on heated plate at 100 °C. The mold is then dipped in a bath of toluene for 1 min. This helps in removing the PDMS mold in the next step. The glass slide with the dry film is then left for 12 h in a warm oven (65 °C) to remove all toluene residuals.

*Fabrication of the PDMS mold (steps 5–6)* Sylgard 184 silicone elastomer kit (Dow Corning, USA) is used to prepare a mixture of PDMS and curing agent with a 10:1 ratio. The mixture is degassed in a desiccator and then poured over the dry film mold, which is degassed again. The PDMS is then cured for 12 h at 65 °C. The PDMS mold is peeled off the dry film mold, resulting in a negative microchannel structure printed on the PDMS (step 6).

*Molding and curing of the OSTEMER Crystal Clear (step 7)* 3 g of degassed liquid OSTEMER Crystal Clear are placed on a 2-mm-thick (60 mm × 38 mm) glass side. The PDMS mold is degassed and immediately placed over the top of the liquid OSTEMER for 15 min so that any bubbles trapped between the OSTEMER liquid and the PDMS mold have time to be absorbed into the PDMS structure. The ensemble is then exposed to UV light for 35 s at 25 mW/cm$^2$ using the UV-KUB 2 masking system in order to partially cure the OSTEMER before the sealing step. The PDMS mold is then cautiously peeled off the OSTEMER layer.

*Sealing the chip (steps 8–10)* Before sealing the microchannel, fused silica capillaries (ID 50 µm, OD 150 µm, C.I.L. TSP050150M010) are cautiously inserted at the inlet and outlet of the microchannel. Here, capillary connections are preferred to nanoports or modular connections (e.g., those used in Marre et al. 2010) due to better resistance under high-pressure flows. A glass cover slip (Menzel-Gläser #1, 0.13–0.16 mm-thick), which has previously been silanized in a mercapto silane solution [(3-Mercaptopropyl)-trimethoxysilane in ethanol 2 % w/w] for 15 min, blown with nitrogen and dried for 30 min in a 100 °C oven, is then positioned over the OSTEMER polymer layer to cover the channel. The silanization of the cover slip enables better bonding of OSTEMER epoxies groups to glass surface. Rubbing a flat blade over the top of the cover slip ensures good contact between the cover slip and the OSTEMER without trapping any air bubbles, which could weaken the bonding. Note that a thin cover slip is preferred over a 1- or 2-mm glass slide since it is slightly more flexible and therefore improves the contact between the surfaces and hence the adherence. Tests were carried out using 1- and 2-mm glass slides to directly seal the channels, but due to the rigidity of the glass slides, the bonding was poor, resulting in leakages at moderate pressures (around 30 bar). Two grams of uncured liquid OSTEMER are then poured over the cover slip, and a 2-mm glass slide is placed on top. The chip is then exposed to UV light for 35 s at 25 mW/cm$^2$, followed by a heating for 12 h at 85 °C to complete the reticulation process of the OSTEMER and to ensure that it is strongly bonded to the glass. The purpose of this second OSTEMER layer and the final glass slide is to reinforce the glass cover slip and increase the pressure resistance of the microfluidic chip while keeping a planar top surface for possible optical measurements. It also ensures that the capillaries are kept securely in place within the square cross-sectional microchannel and that no leaks occur. The microfluidic chip resulting from this fabrication process is presented in Fig. 3.

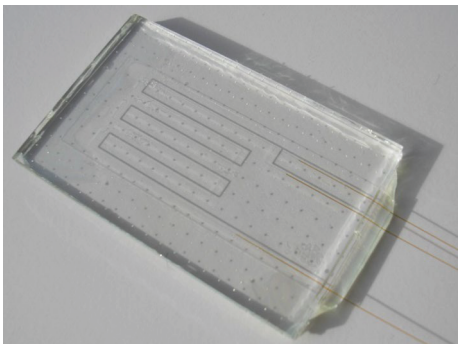

**Fig. 3** OSTEMER chip fabricated according to the described method

### 2.3 Visualization of the chip structure

To verify the different layers of the chip, the cross section was examined using an optical microscope fitted with a digital camera (Nikon ECLIPSE MA 200). A sample was cut from the chip and then embedded in epoxy resin, which acts as a support. The resin was then finely polished using silicon carbide abrasive paper (P4000 grit). Figure 4 illustrates a transverse slice of a chip showing the different layers and a 200 × 200 µm microchannel cross section. The lower layer is the 2-mm glass support. Just above is the OSTEMER layer in which the cross section of the microchannel can be seen. The microchannel dimensions have been correctly reproduced from the PDMS mold, and the sides of channel appear very straight. Above the

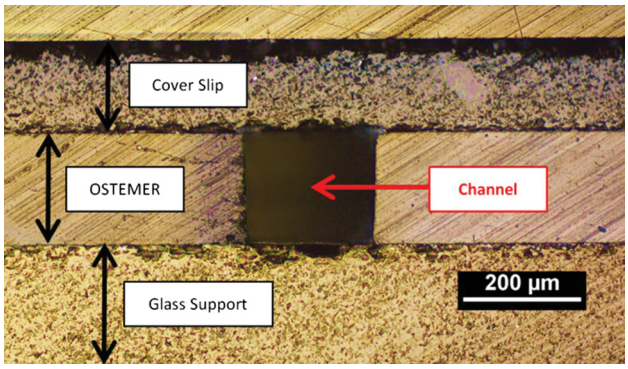


**Fig. 4** Zoom on the cross section of the chip clearly showing the different layers around the 200 × 200 µm channel

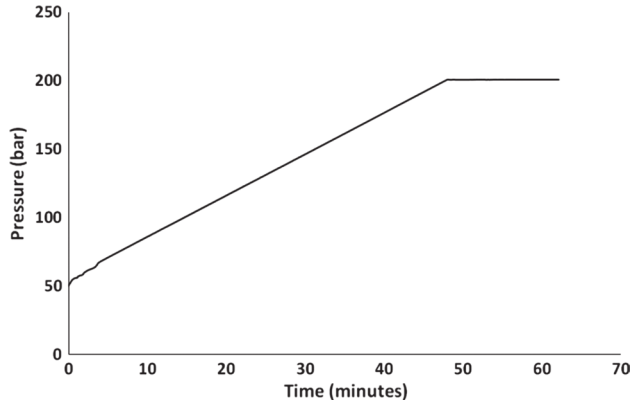


**Fig. 5** Constant gradient pressure test of the OSTEMER/glass microchip. A pressure of 200 bar is achieved without rupture or leakage

OSTEMER layer is the glass cover slip. The irregular edges of the glass support and cover slip are only due to cutting and polishing of the sample that is required for observing the cross section. The bonded surfaces of the OSTEMER polymer with both the glass slide and the cover slip are very satisfactory without imperfections.

Measurement of effective channel size reveals a maximum difference of 10 % on each dimension of the channel. The precise replication of the channel shape from the mold depends on the development time of the dry film mold and the duration that the OSTEMER is exposed to UV. The durations provided in the description of the fabrication method have been previously optimized by trial and error. Note that it may be necessary to adapt the duration of some steps if other channel dimensions are desired.

### 2.4 Pressure tests

Before evaluating the pressure resistance of the OSTEMER chips, preliminary tests were conducted to verify that the microchannel was not blocked and enabled the circulation of $CO_2$ at atmospheric pressure. The pressure resistance of the microchips was then evaluated by applying a constant pressure gradient of 3 bar/min by means of a high-pressure syringe pump (Teledyne ISCO, model 260D). The objective was to determine the pressure at which bursting, breakage or any other default of the microchip was observed. Tests were limited to 200 bar to avoid damaging part of the experimental system and flow circuit. Three microchips were tested and gave identical results. Figure 5 shows an example graph of the pressure applied to one of the microchips. As shown, there is a constant increase of the measured pressure until the maximum applied pressure of 200 bar without rupture of the glass or delamination of the different glass and polymer layers within the chip. Once at 200 bar, the tested channels were maintained at this pressure for approximately 20 min.

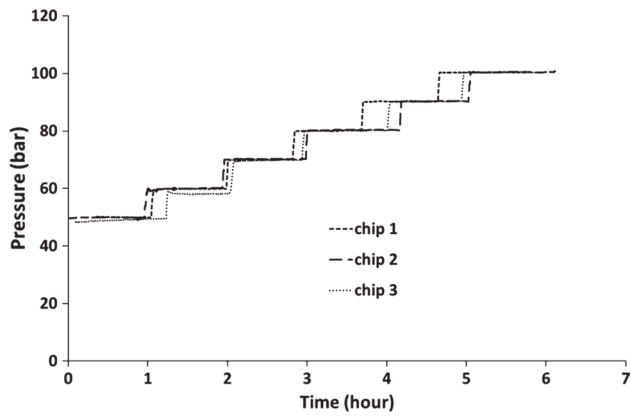


**Fig. 6** Pressure tests of three different OSTEMER–glass microchips. Microchips were fed with $CO_2$ at 50 bar, which was increased by 10 bar every 45–60 min until a pressure of 100 bar was reached

To further test the resistance and reliability of the microchip under high-pressure conditions, the pressure resistance of long operating periods was also verified. The microchannel was fed with $CO_2$ at constant pressure by means of the same high-pressure pump by applying a constant fluid pressure in the chip and then by increasing the pressure with 10 bar steps at regular intervals. The time evolution of pressure at the inlet of the microchannel is recorded. Pressure step experiments were carried out using three different microchips, and the results are shown in Fig. 6. Initially, the chips were fed with $CO_2$ at 50 bar; the pressure was then increased by 10 bar every 45−60 min for a total operating time of more than 6 h. After an hour at 100 bar, the pressure was slowly decreased down to atmospheric pressure.

From the results in Fig. 6, it can be seen that these microchips also resist long operating times at high pressure without any signs of failure.

### 2.5 Flow visualization tests

In order to demonstrate the effectiveness of the hybrid OSTEMER–glass microchips, flow visualization tests were performed by shadowgraphy. To do this, an experimental rig was set up as follows. Fluid is pumped into the microchannel using high-pressure pumps (Teledyne ISCO, models 260D and 100 DX for $CO_2$ and ethanol respectively). To maintain the pressure in the microchannel, a 250-mL tank is used as back-pressure buffer at the outlet of the microchannel. A mass flow controller (mini CORI-FLOW ML120 Bronkhorst) was used to regulate the pump motion to maintain a constant mass flow rate at the inlet of the microchip. The flow rate of ethanol was maintained directly by the pump setting. Fluids can be preheated before the entrance of microchip using a heated bath, and the LED panel (PHLOX White led Backlight 200 × 200) on which the microchip is placed for flow visualization enables the system to be maintained at a constant temperature of 40 °C. A high-speed CCD camera (Mikrotron EoSens CL 1.3 Megapixels) is used to take image flow sequences at 500 frames per second. Examples of two-phase flow images are provided in Figs. 7 and 8.

Figure 7 shows gas–liquid $CO_2$ flow during the transition from the liquid state to the gas state at 22 °C (ambient temperature) and 60 bar (which is indeed the vapor pressure of $CO_2$ at 22 °C). $CO_2$ Taylor bubbles that have been formed within the liquid $CO_2$ flow are clearly visible. This shows the potential of these microchips to be used to observe phase transitions of pure fluids or mixtures.

Figure 8 shows the formation of $CO_2$ bubbles in ethanol (EtOH) at 60 bar and 40 °C. A constant EtOH flow rate of 50 µL/min was fed to the main (top horizontal) channel, and the $CO_2$ gas at 100 µL/min was pinched off from the inlet at the T-junction. In this experiment, both fluids are heated by passing through a heated bath before the entrance of the chip and maintained at temperature by the LED panel used for shadowgraphy. The Taylor bubbles are clearly visible in the EtOH, and the images are of the same quality as those obtained in our previous works using silicon wafer and glass channels (Abadie et al. 2012; Zaloha et al. 2012). This demonstrates the potential of these microchips to be used to study the fundamentals of bubble formation at high pressure.

**Fig. 7** Shadowgraph of $CO_2$ gas bubbles forming in liquid $CO_2$ at the saturation vapor pressure (60 bar at 22 °C) as the pressure is decreased

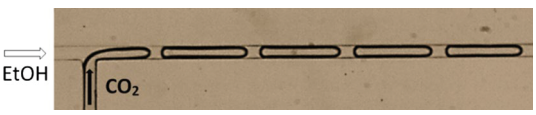


**Fig. 8** Shadowgraph of $CO_2$ Taylor bubbles formation in EtOH at 60 bar and 40 °C. EtOH flow rate is 50 µL/min and gas flow rate is 100 µL/min

### 2.6 Time and cost of the fabrication process

The dry film and PDMS molds are reusable and can be made in less than 2 h each, with an additional 12 h of drying time. These molds can be used several times to make the OSTEMER microchannels, and by employing several PDMS molds, a number of microfluidic chips can be made in parallel.

After investment of the UV light source and the heated laminator, and not including labor, the cost required to fabricate one microfluidic chip following the method described in this paper is less than 20 euros. This corresponds to 5 grams of OSTEMER Crystal Clear, two 2-mm glass slides, one cover slip, 30 cm of fused silica capillaries for the inlets and outlet, as well as the fabrication cost of the reusable dry film and PDMS molds. This cost is ten times less than the inexpensive devices proposed by Macedo Portela da Silva et al. (2014) and is a much more accessible solution than buying commercial microfluidic chips that may cost several hundreds of euros.

## 3 Conclusion

A fast and inexpensive method for the fabrication of transparent pressure-resistant microchannels has been presented. The fabrication method is based on soft lithography using an optically transparent polymer called OSTEMER Crystal Clear. Once the molds are prepared, the microfluidic chips can be prepared in less than 2 h with 12 h drying time for less than 20 euros per chip. Moreover, neither clean room facilities nor specific fabrication equipment (apart from a laminator and a UV radiation source) are required. The microfluidic chips have been shown to resist high pressures for several hours and for pressures at least up to 200 bar.

These entirely transparent pressure-resistant microfluidic chips offer the possibility to observe and measure flow characteristics and transport phenomena in complex multiphase flows operating at high pressure, e.g., supercritical $CO_2$ and liquid or ionic liquid dispersions. The planar and optically transparent nature of the microfluidic chips also enables distortion-free visualization without reflection, and they are therefore better adapted to measurement techniques, such as high-speed imaging, micro particle velocimetry, laser-induced fluorescence and spectrophotometry (UV–Vis, IR), compared with glass capillaries or silicon wafer chips.

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