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TECHNICAL PAPER

Hot embossing of microfluidics in cyclic-olefin co-polymer using a wafer aligner-bonder

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Abstract

The fabrication of robust microfluidics can be tedious, often involving the use of numerous cleanroom resources and processes. We propose a process that is easy to apply yet capable of producing precision microfluidics in polymer with high yield and high fidelity at a wafer scale. The process is centered on the use of a wafer aligner-bonder implementing a one-step hot embossing process to transfer microfluidic designs from a Si master mold onto a thermoplastic deformable substrate. The approach has the additional benefit of transferring features directly to the substrate without the need for pre-annealing or other pre-processing. Additionally, the mold used to replicate the microfluidic design can be re-used numerous times. The most important process parameters, embossing temperature, embossing pressure, embossing time and demolding temperature, were optimised. We demonstrate the process by fabricating over 340 microfluidic chips per 4-inch diameter cyclic-olefin co-polymer substrate. The approach should scale to larger wafer diameters using a wafer aligner-bonder of larger diameter platens, suitable for volume manufacturing.

1 Introduction

Microfluidics is an essential technology for controlling and manipulating small volumes of fluids in numerous applications within a lab-on-a-chip context. The use of microfluidic networks and microfluidic components is on the rise and has become essential in many measurement and sensing applications in fields such as medical diagnostics, biology, chemistry, biophysics, and chemical engineering.

Many microfluidic devices were (and still are) made of glass on glass or silicon substrates, presenting the advantages of robustness, but generally requiring fabrication processes that are not trivial, especially with regards to creating interfaces to external fluidic components. Polymer microfluidics offer interesting alternatives in terms of fabrication (O. Rötting et al. 2002). Microfluidic channels can be fabricated in polymers using many different methods and technologies, such as laser writing (Singh et al. 2014), UV embossing (Zhong and Shan 2012), UV lithography (Kim et al. 2011; Karlsson et al. 2016), injection molding (Liou and Chen 2005; McCormick et al. 1997), and hot embossing (Peng et al. 2014; Deshmukh and Goswami 2019; Yi-Je et al. 2002; Guo 2004; Heckele and 2004; Kumar et al. 2009; PinYeo et al. 2009; Zhu et al. 2011).

Numerous technologies have been used for hot embossing of polymer-based devices as reviewed by Rötting et al. (2002). The fabrication of high aspect ratio microstructures using hot embossing was reported (Datta and Goettert 2006), including the optimisation of the process using the force–temperature–deflection method. Polymethyl methacrylate (PMMA) based microchannel devices were also fabricated by hot embossing and direct bonding (Shinohara et al. 2007). Hot embossing of cyclic-olefin co-polymer (COC) based microfluidic devices was reported by Jena et al. (2010), where the embossing conditions were optimised according to the orientation of the

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polymer chains in the film (and/or substrate) in a one-step embossing process. Hot embossing and replica molding processes were applied and compared in the fabrication of microstructures in PMMA and polydimethylsiloxane (PDMS) by Forfang et al. (Forfang et al. 2014), where a brass master mold was used in both processes. The effects of embossing variables such as temperature, time and force were studied by Çoğun et al. (2017), where hot embossing was performed on a PMMA substrate using an aluminum master mold by altering the embossing variables.

Cyclic-olefin co-polymers (COCs) have properties that motivate a wide range of applications in, *e.g.*, electronics, packaging, healthcare, and diagnostic lab-on-a-chip devices (Jena et al. 2012). COCs are available commercially in high purity [e.g., TOPAS®, (Topas Advanced Polymers 2020)]. They are nonpolar, amorphous polymers that have good resistance to acidic and alkaline solutions, and many organic polar solvents such as acetone, methanol and isopropyl alcohol. They also have a low capacity for the absorption of water and a low permeability for water vapour. COCs are biocompatible, transparent, and have very good optical properties. They can be formulated over a wide range of grades with variable heat resistance and flow properties, making them highly attractive thermoplastics for use in heat-driven processes such as hot embossing.

We describe in this paper a hot embossing process that makes use of a wafer aligner-bonder. Wafer aligner-bonders are precision tools found in cleanrooms, useful to accurately align wafers, initiate wafer contact in a controlled fashion under vacuum conditions or with a process gas, and enable bonding or embossing processes through the controlled application of heat and force to the contacted wafers. We describe first the fabrication of a silicon master embossing mold of the desired microfluidic design, the embossing of the patterns from the mold into the surface of a COC substrate using a wafer aligner-bonder, and the subsequent release of the COC substrate from the mold. Once fabricated the master embossing mold can re-utilised many times. We demonstrate our hot embossing process by simultaneously producing hundreds of precision microfluidic chips per COC wafer.

2 Experimental

2.1 Materials

In this study, the cyclic-olefin co-polymer TOPAS® (Topas Advanced Polymers 2020) was used in the form of substrates supplied by Microfluidic ChipShop (product # mcs-COC-13, [Microfluidic ChipShop 2020)]. The COC substrates have a thickness of 1.5 mm and a diameter of 4 inches (115 mm). The refractive index and glass transition temperature of the grade used are $n = 1.53$ and $T_g = 142$ °C, respectively. Its coefficient of thermal expansion (CTE) is of the order of 10^{-6}/K, which is in the range of well-known and studied materials such as Si (1.5×10^{-6}/K) and borofloat glass, available in substrate form and suitable as embossing molds (once patterned) in a wafer bonding system. Matching the CTE of the substrate used for the mold to that of the material to be embossed is important to minimize thermally induced stress in the material and limit replication errors (Becker and Heim 2000).

2.2 Silicon master embossing molds

Four master embossing molds were fabricated on four 4-inch Si wafers using standard Si processing. The microfluidic channels defined on a biosensor mask were patterned on Si wafers using lithographic steps, then etched via a deep reactive ion etching (DRIE) process using SiO_2 as a hard etch mask. Upon completion, the molds have raised channels (protruding features) as the negative of the microfluidic layout to be embossed. The raised channels on the four molds have heights of 21.4, 26.6, 28.5 and 29.6 µm, achieved by varying the DRIE time. The channel widths range from 60 to 280 µm depending on the design. A thin SiO_2 layer forms naturally on Si upon exposure to lab air (native oxide), which defines the contact surface of the mold. The fabrication details of the Si molds is described in the supplementary file which also collects various images of fabrication results.

Figure 1 shows a collection of scanning electron microscope (SEM) images of raised channels achieved by etching Si, taken after completion of the DRIE process described in the supplementary file. The features are of very good quality exhibiting vertical sidewalls and very low roughness. Figure 1(c) shows curtaining on sidewalls, which has been observed to occur on certain channels for the deeper etches. The roughness of the measured etched surface on the Si wafer was less than 0.5 nm (RMS) as measured by AFM.

2.3 Hot embossing process

Figure 2 shows the 5 main process steps involved in our hot embossing process using a Si mold and a COC substrate: (a) Alignment of the mold and substrate in the wafer aligner-bonder; (b) heating the mold and substrate to the embossing temperature, which is slightly above T_g of the polymer; (c) isothermal molding by embossing into the substrate the raised microfluidic patterns on the mold under controlled force; (d) hold time to allow the polymer substrate to flow and infill the embossing pattern; (e) cooling

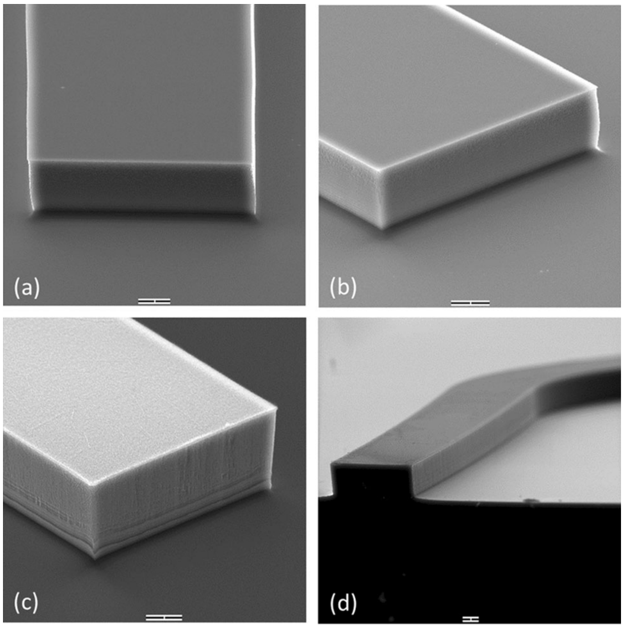

Fig. 1 SEM images of raised channels achieved by etching Si, taken after completion of the fabrication process described in the supplementary file; **a** and **b** 21.4 μm high raised channels; **c** and **d** 26.6 μm high raised channels. The scale bar on all images is 10 μm long

the mold and substrate to the demolding temperature, which is slightly below T_g of the polymer, then demolding by removing the force and separating the mold from the substrate. The polymer substrate experiences two phases of deformation in the complete process; the first phase is stress concentration and strain hardening that occurs in the heating and embossing steps, and the second phase is stress relaxation and deformation recovery that occurs in the cooling and demolding stages (Liu, et al. 2009).

Hot embossing was performed in the AML-AWB-04 wafer aligner-bonder system (Applied Microengineering Ltd. 2020)—the opened system is shown in Fig. 3. The wafer aligner-bonder has two platens, upper and lower, 100 mm in diameter. The platens are enclosed in a vacuum chamber. Both platens can be heated independently at a controlled rate. The system cools naturally when heating is turned off.

There are a number of variables that may be tuned to develop an optimized embossing process. The optimal process was deduced empirically through trial-and-error by determining the best set of embossing parameters to obtain the desired geometries and results. Thus, embossing experiments were carried out in two stages. In the first stage, the most sensitive parameters affecting embossing were explored. As reported by Cameron et al. (2006), the embossing temperature, pressure, demolding temperature and hold time are important parameters for embossing polymers. In the second stage, the parameters determined in the first stage were applied and tuned to achieve the best results.

To perform hot embossing in the wafer aligner-bonder, the Si mold and COC substrate were loaded onto the upper

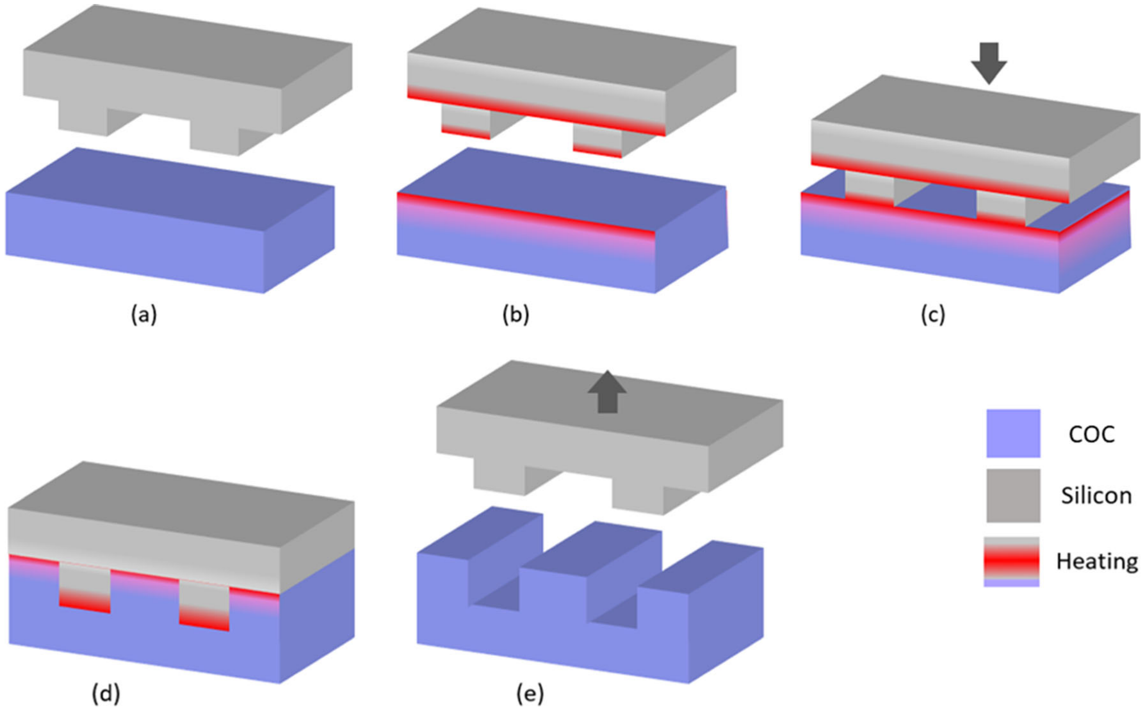

Fig. 2 Embossing process flow using a Si mold and a COC substrate: **a** Mold and substrate alignment in wafer bonder-aligner; **b** heating of mold and substrate; **c** embossing through the application of force; **d** hold time; **e** cooling and demolding by separation of the mold from the substrate

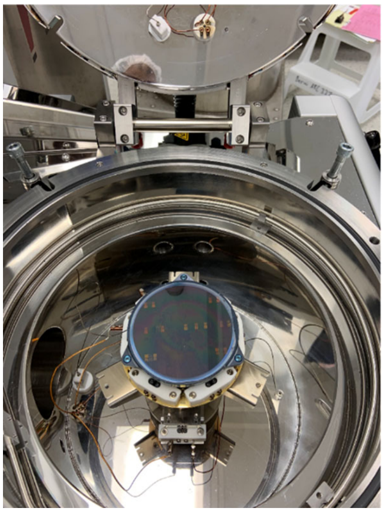

Fig. 3 AML wafer aligner-bonder with a wafer centered on the bottom platen

and lower platens, respectively. Si master embossing molds and COC substrates 4-inches in diameter were used throughout the experiments. After reaching the desired vacuum, the mold and substrate were aligned, then heated to the embossing temperature, which is slightly above the T_g of our COC (T_g = 142 °C). The embossing temperature was varied from $T_g + 10$ °C to $T_g + 25$ °C in our experiments. After setting the platen temperature, 5 to 15 min were required for the mold and substrate to reach thermal equilibrium. Force was then applied slowly to ensure good control and good contact of the mold to the substrate. The embossing pressure was varied in the range from 190 to 740 kPa in our experiments. The applied heat and force were kept constant for a time—the *embossing time* – to allow COC to flow and fill in the mold features. Then heat was turned off, and the wafers allowed to cool to the demolding temperature under applied force. The demolding temperature was varied in the range from T_g—10 °C to T_g—25 °C in our experiments. The *hold time* is then is the time between exerting the required pressure at the embossing temperature, and the release of pressure at the demolding temperature—the hold time thus depends on the demolding temperature. Cooling was allowed to proceed naturally after turning off the platen heat sources. Finally, the mold and substrate were removed from the wafer aligner-bonder and carefully separated. The detailed embossing process steps are summarised in Table 1.

3 Results and discussion

Trials were carried out using the four Si molds fabricated as described in the supplementary file as the embossing process was developed. The embossed structures were examined by optical microscopy, atomic force microscopy, stylus profilometry, and scanning electron microscopy to assess quality and guide process optimisation. We show results achieved using the mold with raised channels 29 µm in height, as this represents the most challenging case in our set of trials.

The sensitivity of important process parameters such as embossing temperature, force, demolding temperature and hold time were examined initially. For this purpose, the embossing temperature was varied in the range of 152–167 °C, the embossing pressure was varied in the range of 190 kPa to 740 kPa, and the demolding temperature was varied from 117 to 132 °C. It was determined early on that the structures embossed showed low sensitivity to the demolding temperature. Thus, in subsequent iterations, the embossing temperature and force were varied, while keeping the demolding temperature constant to 117 °C. Furthermore, no difficulties were encountered in separating the Si mold from COC substrates in our experiments, indicating that the process does not result in the adhesion of COC to the native oxide (SiO_2) that formed on our Si molds. Table 2 summarises experimental parameters for three sets of trials. Each set of trials consisted of many experiments exploring specific combinations of embossing parameters.

In the 1st set of trials, the embossing temperature was set to 10 °C above the T_g of our COC to 152 °C, and the embossing pressure was varied between 190 and 255 kPa. The embossed structures had the required channel depth (29 µm as verified by profilometry), but the channel walls were not vertical, and were wider along the channel top than along the channel bottom, indicating that the polymer did not completely fill the mold during embossing. In the 2nd set of trials, the embossing temperature was set to 15 °C above the T_g of our COC to 157 °C, while keeping the embossing pressure in the same range. The embossed structures had channel walls of improved vertically, however, the top edges were rounded, again indicating limited filling of the mold. In the 3rd set of trials, the embossing temperature was kept to between 18 °C and 25 °C above the T_g of our COC and the embossing pressure was varied between 370 to 740 kPa. The embossed channels from this trial exhibit sharp and precisely-defined edges along the top and vertical walls, indicating good infilling of mold features.

A typical embossing process cycle is shown in Fig. 4, which shows force and temperature *vs*. time, as measured and logged in real-time by the wafer aligner-bonder. In this example, the embossing temperature was set to 162 °C and the embossing force to 1.5 kN (190 kPa) (Wafer MA44, similar to Trial Set 2 of Table 2). Interestingly, the force applied initially (1.5 kN) drops almost immediately after

Table 1 Hot embossing process steps in relation to Fig. 2

Figure 2	Activity	Parameter
a	Mount wafers on platens	Silicon mold mounted to top platen and COC substrate mounted to lower platen
	Establish vacuum	Set vacuum level in bonding chamber to 10^{-2} mbar
b	Align and heat platens	Ramp platens to embossing temperature, slightly above T_g = 142 °C; embossing temperatures were varied from 152 °C to 167 °C in trials
	Thermal equilibrium	Hold time of 5 to 15 min for mold and substrate to reach thermal equilibrium
c	Contacting	Bring mold and substrate into contact
d	Apply force	Force was varied from 1.5 to 6 kN in trials
	Emboss	Hold temperature and applied force for embossing time of 30 s
	Set platen heaters to demolding temperature	Allow mold and substrate to cool to demolding temperature, slightly below T_g = 142 °C; demolding temperatures were varied from 132 °C to 117 °C in trials
e	Remove force	Completely remove force
	Turn off platen heaters	Allow mold and substrate to cool to lab temperature (about 25 °C)
	Remove wafers from platens	Separate mold and substrate with care

Table 2 Summary of hot embossing process parameters for 3 trial sets

Parameter	Trial Set 1	Trial Set 2	Trial Set 3
Vacuum pressure	2 × 10^{-2} mbar	2 × 10^{-2} mbar	2 × 10^{-2} mbar
Embossing temperature	152 °C	157 °C	160 °C–167 °C Optimal value: 160 °C
Thermal equilibrium time	5 min	5 min	5 min
Applied force/pressure	1.5–2 kN / 190–255 kPa	1.5–2 kN / 190–255 kPa	3–6 kN/370–740 kPa Optimal value: 5kN / 620 kPa
Embossing time	30 s	30 s	30 s
Demolding temperature	117 °C	117 °C	117 °C

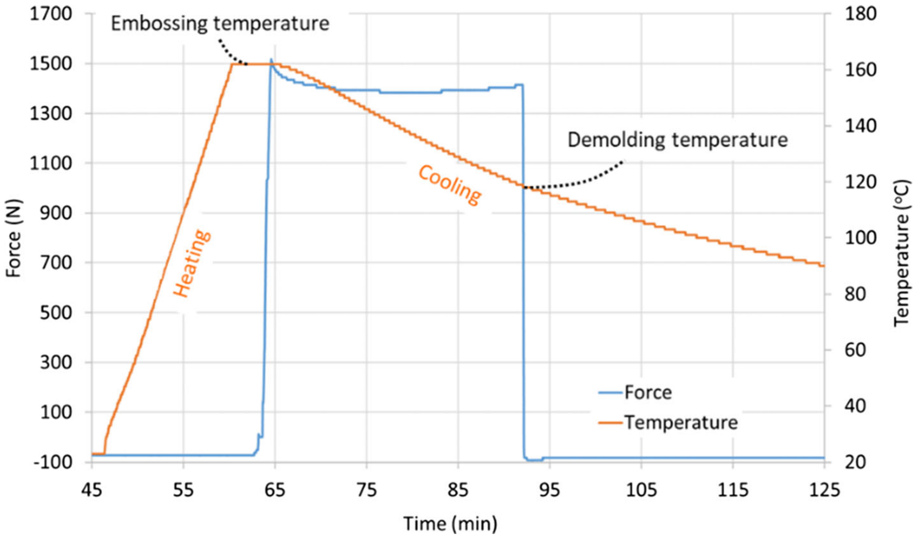

Fig. 4 Hot embossing process cycle applied to a COC substrate (Wafer MA44, similar to Trial Set 2 of Table 2)

contact indicating polymer flow and reforming around the raised channels on the mold.

Figure 5 shows SEM images of embossed channels of various geometries on a COC substrate. These channels were embossed as part of Trial Set 3 (Table 2). The features are replicated from the Si master mold with high fidelity.

Further optimisation of the process led to our best results, obtained at an embossing temperature of 160 °C (18 °C above the T_g of our COC) and 5 kN of applied force (620 kPa of applied pressure), achieved during Trial Set 3 (Table 2). These parameters produced highly vertical sidewalls and sharp edges as shown in the SEM images of Fig. 6a, b. Close inspection reveals curtaining along the bottom of the sidewalls – this curtaining originates from the etched sidewalls along some of the raised channels of the Si mold (*cf*. Fig. 1c) and was transferred during embossing.

Figure 6(c) shows a 4-inch diameter COC wafer bearing over 340 high-precision microfluidic chips embossed simultaneously and replicated with high fidelity using our optimal parameters. The circular depressions (at left and right from centre) are artefacts due to COC flow in holes located in the bottom platen of the aligner-bonder. They can be avoided by placing an unpatterned Si wafer below the COC wafer for support. The approach should scale in a straightforward way to larger wafer diameters using a wafer aligner-bonder of larger diameter platens.

4 Concluding remarks

We developed and demonstrated a one-step hot embossing process based on the use of a wafer aligner-bonder to replicate precision microfluidics in polymer with high yield and high fidelity at a wafer scale. A Si master embossing

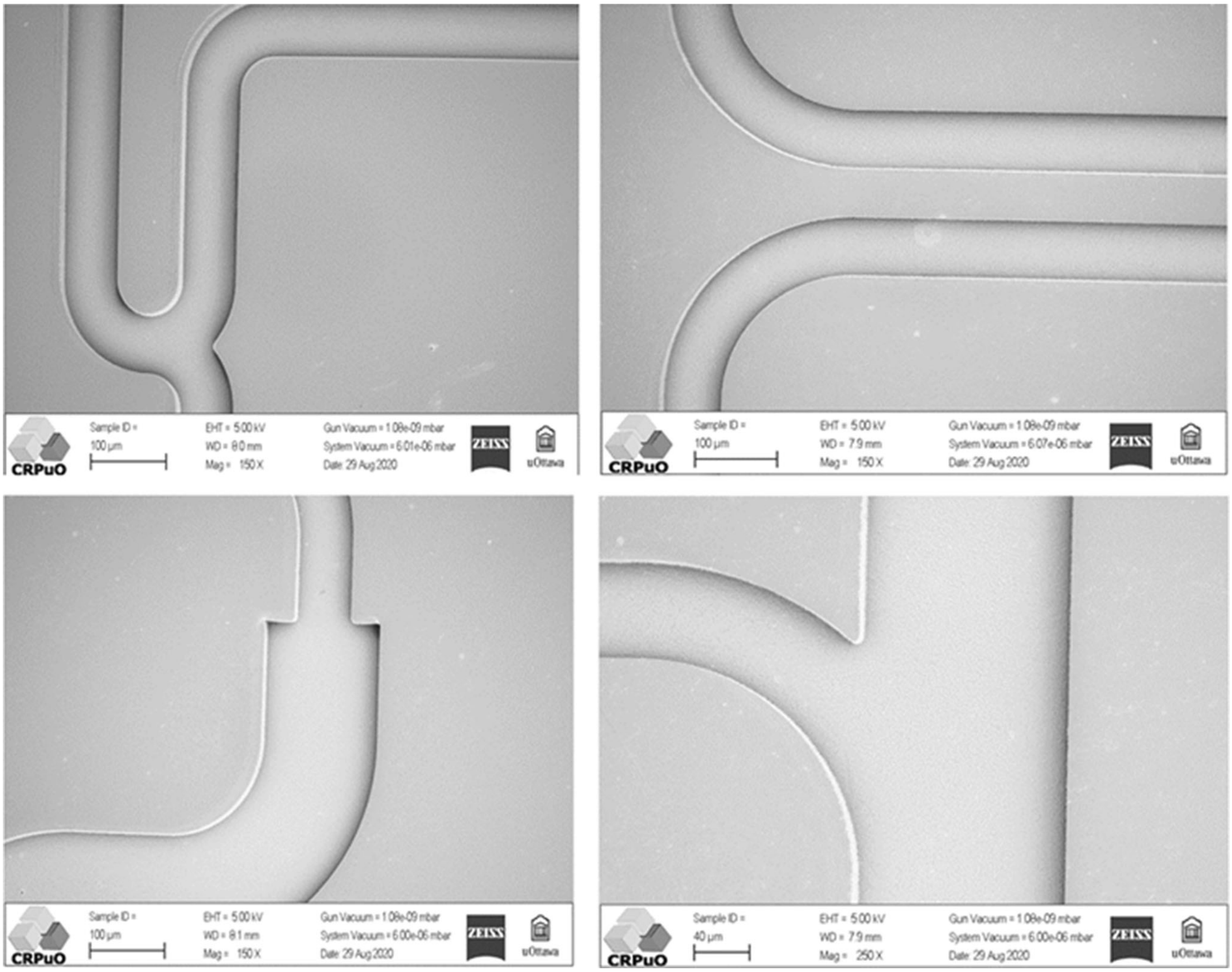

Fig. 5 SEM images of various embossed channels on a 4-inch COC substrate. (Wafer MA 56, Trial Set 3 of Table 2)

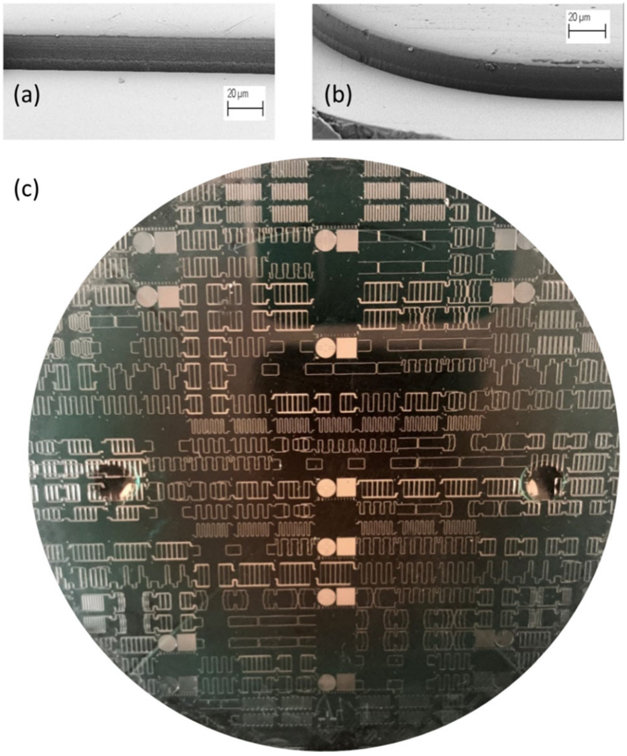

Fig. 6 **a** and **b** SEM images of straight and curved channel walls, respectively. **c** A 4-inch embossed COC wafer showing over 340 chips of precision-embossed microfluidic channels. The COC wafer was placed on a Si wafer to provide optical contrast during imaging. The circular depressions (at left and right from centre) are artefacts

mold was created and re-used many times in our embossing experiments of COC wafers (T_g = 142 °C). Optimisation of the process led to our best results, obtained at an embossing temperature of 160 °C and 5 kN of applied force (620 kPa of applied pressure). These parameters produced highly vertical sidewalls and sharp edges in COC as revealed by SEM imaging. We applied to the process to 4-inch diameter wafers, producing over 340 high-precision microfluidic chips, embossed simultaneously and replicated with high fidelity using our optimal parameters. The approach should scale to larger wafer diameters using a wafer aligner-bonder of larger diameter platens, suitable for volume manufacturing.

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