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Low-temperature, Simple and Fast Integration Technique of Microfluidic Chips by using a UV-curable Adhesive

Supplementary Information

Channel fabrication

Glass microchannels. A borosilicate glass wafer (Borofloat 33, 4-inch, 500-µm thick, Schott Technical Glasses Germany) was first thoroughly cleaned in fuming 100% HNO_3 solution for 10 min and subsequently rinsed in DI water and dried. Subsequently, a gold-chromium layer was sputtered (Au 150 nm, Cr 30 nm) on the cleaned glass substrate and patterned using positive photoresist (OIR 907-17) by using a standard photolithography process. In this process, the photoresist was first spin-coated on the metal bilayer (4000 rpm for 20 s) to give a layer of 1.7 µm. Afterward, the spin-coated resist was pre-baked at 120°C for 2 min, exposed to UV irradiation (Mask aligner EVG-620, 4 s exposure at 12 mW/cm^2), post-baked at 120°C for 1 min, and developed in a developer (OIR 907-17 developer) for 1 min and rinsed in demineralized water before hard-baking (120°C for 15 min) on a hotplate. Subsequently, the Au/Cr layer was patterned using Au-etchant (105043, Merck) and Cr-etchant (111547.2500, Merck), respectively, for a few seconds. This patterned Au/Cr layer behaved as a mask layer for the fabrication of the channels in glass. Microchannels were produced by wet-etching technique by immersion for 20 min in 25% hydrofluoric acid (VLSI Selectipur, BASF). It should be mentioned that as wet-etching is isotropic, the glass was also etched laterally and it subsequently formed undercuts below the photoresist mask. As a consequence narrower microchannels had to be designed to compensate for these undercuts. The resulting wafer was rinsed in DI

water before stripping of the remaining photoresist in acetone (15 min; VLSI 100038, BASF) and removal of the Au/Cr layer using the same etchants as before. After rinsing in DI water, the process was finished.

SU-8 channels. SU-8 microchannels were fabricated using photolithography techniques on a borosilicate glass substrate (borofloat 33, 4-inch, 500-μm thick, Schott Technical Glasses Germany). The fabrication process also started with similar cleaning of the glass wafer (100% HNO_3 immersion for 10 min). Following this, the glass substrate was dehydrated at 120°C for one hour and pre-treated by oxygen plasma (Tepla 300E, 350 W, 5 min) to ensure good adhesion of the SU-8 precursor on the substrate. A SU-8 5 precursor (Microchem., MA, USA), a negative photoresist, was firstly spin-coated on the glass substrates (500 rpm for 5 s followed by 1000 rpm for 20 s) to give a 14 μm resist layer. This spin-coated photoresist was subsequently pre-baked at 65°C for 2 min and 95°C for 5 min, patterned by photolithography techniques (Mask aligner EVG-620) at 12 mW/cm^2 for 12 s, post-baked at 65°C for 2 min and 80°C for 5 min, developed in RER600 for 10 min and hard baked for 20 min at 180°C. The resulting microchannels were rinsed for 2 min in isopropanol (technical grade, BASF) and stored until dicing.

UV-curable adhesive microchannels. NOA 81 microchannels were produced using a combination of UV-exposure and casting techniques against a PDMS-based mold, using the same procedure as published elsewhere.[35] The fabrication of NOA microchannels proceeded via the fabrication of a silicon-based master to produce PDMS molds before actual channel fabrication.

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In a first step a silicon-based master was fabricated and structures were produced from SU-8 using a similar process as described above. These structures are identical to the final microchannels fabricated in the UV-curable adhesive NOA 81. The primary master was fabricated on a silicon wafer (100-oriented, p-type, resistivity 5-10 Ω·cm, 100 mm diameter, thickness 525 µm, single side polished, Okmetic, Finland) on which SU-8 5 precursor (Microchem., MA, USA) is spin-coated (500 rpm for 5 s followed by 1000 rpm for 20 s) to give a layer of 14 µm. This SU-8 layer underwent the same treatment as before (pre-bake, UV exposure, post-bake, development in RER 600 and hard-bake). This master was subsequently used for the realization of a PDMS mold. A two-component kit (Sylgard 184, Dow Corning, Midland, Michigan, USA) was used for PDMS fabrication; it includes a pre-polymer of PDMS as well as curing agent which are mixed in a 10:1 weight ratio. The resulting mixture was thoroughly degassed in vacuum, subsequently poured into the silicon-SU8 master, anew degassed in vacuum and cured for 3 h at 80°C. After cooling back to room temperature, the PDMS mould was peeled-off from the master, thoroughly cleaned with ethanol in an ultrasonic bath for 15 min and dried using compressed air before further utilization. As the devices are fabricated at the chip level, this PDMS mould was cut in individual chips using a sharp knife. The resulting chips were subsequently cleaned again in ethanol in an ultrasonic bath for 15 min and carefully dried using compressed air before being used for the channel fabrication. Finally, NOA 81 microstructures were produced using PDMS chips as molds. A small amount of UV-curable adhesive (NOA 81, Norland Product) was placed onto a PDMS mold before pressing against a glass substrate. The adhesive was cured by UV irradiation (5 min, 365 nm) at room temperature. The PDMS chip was peeled-off to

release NOA 81 structures on a glass substrate. It should be noted that the presence of oxygen in the PDMS prevents full curing of the adhesive in the vicinity of the PDMS mold and consequently, a thin layer of uncured material remained on the top of the structures.[35] This can be exploited for straightforward bonding of the NOA 81 structures on a second substrate; the resulting structures were pressed against a cover lid and irradiated again (5 min, 365 nm) to fully cure the adhesive and bond it to a cover lid.

Reservoirs Fabrication

For some chips, we added inlet and outlet reservoirs to the design to facilitate fluidic connections between the chip and the outer world. These reservoirs were fabricated using powder-blasting technique in the glass substrate at the bottom side of the wafers. In a first step, a photosensitive foil (BF410, Ordyl) was laminated on the backside of the glass wafer. The reservoir pattern was defined using standard photolithography technique, and developed in a 0.2% sodium carbonate solution. Afterwards, the front side of the wafer was covered with a Riston foil to protect the microstructures on that side of the substrate during the powder blasting step and while exposing the substrate to Al_2O_3 powder (29 µm grain size). Finally, the photosensitive foil was removed using a 0.2% sodium carbonate solution (room temperature, few hours) and the resulting substrate was ultrasonically rinsed in acetone (VLSI 100038, BASF) for 15 min.

Chip Preparation

Bonding of the chips is performed at the chip level so that individual chips (8 mm x 8 mm) must be released after the channel fabrication before the bonding step. This applies

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to glass and SU-8 channels whereas NOA 81 channels were already fabricated at the chip level. Bonding was demonstrated using two types of substrates (glass and parylene-coated silicon), and these also had to be released as 8 mm x 8 mm chips before bonding. Glass substrates (Borofloat 33, 500-µm thick) were diced using dedicated equipment (Disco DAD-321, blade type TC 300, spindle rev. 25,000 rpm). Parylene-coated silicon substrates were kindly provided by IMEC (Leuven, Belgium). Using Chemical Vapor Deposition (CVD) technique, a 10-micron parylene layer was deposited onto silicon substrate. In this process, the solid-state parylene which was heated up to 690°C from a furnace evaporates and flew into a vacuum chamber (100 mTorr) before depositing onto the silicon substrate. Finally, parylene-coated silicon substrates were diced as individual chips with the dimensions of 8 mm x 8 mm.

Before bonding, all microfluidic chips and cover substrates were ultrasonically cleaned in ethanol for 15 min and dried at 60°C for 30 min. Subsequently, parylene surfaces were activated by plasma treatment (400 mTorr O_2 pressure, 30 W, Plasma Cleaner, Harrick Plasma, USA) for 5 min; this helped spreading the gluing material between the two layers to be bonded.