Wafer scale fabrication of glass-FEP-glass microfluidic devices for lipid bilayer experimentation

Device fabrication

For the greatest part, the fabrication proceeds as already reported.[1] Microfluidic channels are wet-etched in glass substrates using a 33% HF solution and a patterned Cr/Au layer as a mask. Fluidic connections in the top wafer are made by powder blasting with a patterned Ordyl BF410 foil as a mask. For dry-etching of the FEP foil, a silicon-based shadow mask is utilized, and fabricated as previously described.[2]

For the novel bonding procedure, first, a silicon wafer is coated with a 80 nm fluorocarbon anti-sticking layer, grown in a home-made parallel plate Reactive Ion Etcher for 5 min (with 20 W 13.56 MHz RF power, 120 mT pressure, 20 sccm CHF_3 gas and 10ºC substrate temperature).[3] On top of this substrate, 15 g of PDMS (10:1 ratio, Sylgard 184, Dow Corning) is applied and subsequently cured in a leveled furnace at 80ºC for 1 h, to yield a 1.6-mm thick PDMS slab. The contact angle of the PDMS surface is 110º, as measured with a Dataphysics OCA-20 surface contact angle equipment, which is is too high to get a good wettability for the gluing material. PDMS is treated with oxygen plasma using the aforementioned equipment (25 W 13.56 MHz RF power, 100 mT pressure, 50 sccm O_2 and 10 sccm N_2 gas and 10ºC substrate temperature), to reduce the contact angle to about 27º. A thin layer of NOA81 is applied to the bottom wafer by means of a 'spin & roll' procedure, whereby NOA81 is spin-coated on a flexible substrate and transferred to the object by 'rolling' the flexible substrate onto the surface to be bonded.

The NOA81 is spun onto the PDMS surface for 60 s at 6000 rpm rotation speed and an acceleration of 1000 rpm/s. By weighing before and after spinning, the thickness of the NOA81 layer is determined to be 4.2 µm, knowing the density of NOA81 to be 1.2 gr/cm^3

(https://www.norlandprod.com/literature/81tds.pdf) and the 100 mm wafer surface area to be 77.9 cm^2. An experiment on a dummy wafer by spinning NOA81 under the same conditions, covering the NOA with a sheet of PET foil and subsequent UV exposure, shows a layer thickness of 3.9 µm, as measured by a Veeco Dektak 8 surface profiler, which is in good agreement with our estimation, taking into account that curing of the NOA might reduce its volume.

After spinning NOA81, the PDMS is peeled off the substrate wafer, as shown in Fig. S1. Next, the PDMS slab is turned upside down and carefully rolled over the cleaned Borofloat bottom wafer, without shifting, as shown in Fig. S2. The stack is checked for the presence of air inclusions, which are easily wiped out. After careful removal of the slab, part of the NOA81 is left on the Borofloat wafer. The thickness of this NOA layer is determined to be ca. 1.8 µm using the same weighing procedure on a blank Borofloat wafer.

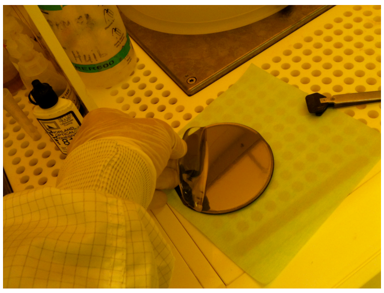

Figure S1: Peeling off the PDMS slab with NOA81 coating from a dummy silicon wafer

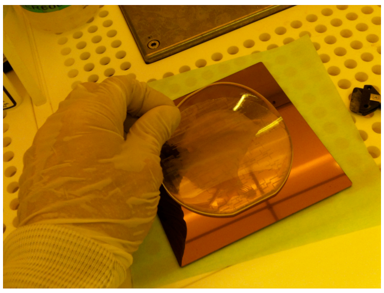

Fig. S2: Rolling over the NOA81-coated PDMS slab over the bottom glass wafer with wet-etched channels.

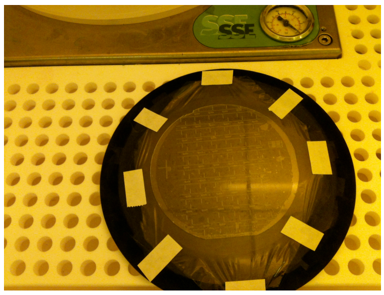

Fig. S3: NOA81-coated bottom wafer attached to the FEP foil secured on a dark substrate.

In the meantime a piece of FEP foil is stretched over a dark substrate plate, on which it is fixed using tape. The FEP foil is treated using oxygen plasma in the same way as the PDMS, to enhance the wettability of NOA81. The NOA81 coated bottom wafer is turned upside down and attached to the FEP foil (Fig. S3), and air inclusions are wiped out. NOA81 is cured for 2-3 min in an Electrovision mask aligner type EVG 620, with a 12 mW/cm^2 UV-A source.

After cutting out the wafer from the superfluous Teflon foil with a surgeon knife, the wafer is aligned to a silicon shadow mask by hand with the help of a stereomicroscope. The alignment can be relatively accurate, and more accurate than at the chip level, because the alignment marks are far away, near the opposite edges of the wafer. Thereafter, the shadow mask is fixed with tape to the glass wafer-FEP stack, and loaded in the RIE equipment, to dry etch the structures in the FEP foil (50 W, 13.56 MHz RF power, 100 mT pressure, 50 sccm O_2 / 20 sccm N_2 / 2 sccm CHF_3 gas and 10ºC substrate temperature, for 70 min).

The shadow mask is removed and the top wafer is glued on the glass-FEP stack in almost the same way as described before. First, the top glass substrate is covered with a layer of NOA81 using the same 'spin & roll' procedure, but, here, a thicker layer of NOA is used to level out irregularities, since now two stiff wafers have to be bonded, while in the previous step the FEP foil could level out irregularities. Specifically, the NOA layer is spin-coated for 30 s (instead of 60 s before) using the same conditions as before (6000 rpm rotation speed and an acceleration of 1000 rpm/s). The resulting NOA81 layer is of ca. 3.1 µm after completion of the bonding procedure. The top glass wafer is loaded in the Electrovision mask aligner, and the anodic bond program and accompanying tool of the machine are used to align both wafers accurately and clamp them together, while keeping them separated by the tools' flags. Next, the bond tool is loaded in the Electrovision Anodic Bonder EV-501, where vacuum is applied to the secured pieces to prevent air inclusions. Subsequently, the wafers are pressed together in the center, and the flags are withdrawn while the whole wafer surface is pressed together for 5 min with a 2500 N force. Next, NOA81 is pre-cured in the Spectrolinker XL-1500 UV Crosslinker for 5 min (3 mW/cm^2) followed by final 2 min curing in the mask aligner type EVG 620, with a 12 mW/cm^2 UV-A source. Finally, the devices are released through dicing (Loadpoint MicroAce 3).

Device characterization

The thickness of the gluing layer is measured using profilometry (Veeco Dektat 8) across a whole wafer, after removal of the FEP film and creation of scratches in the NOA layer. Values determined are indicated on the sketch in Fig. S4.

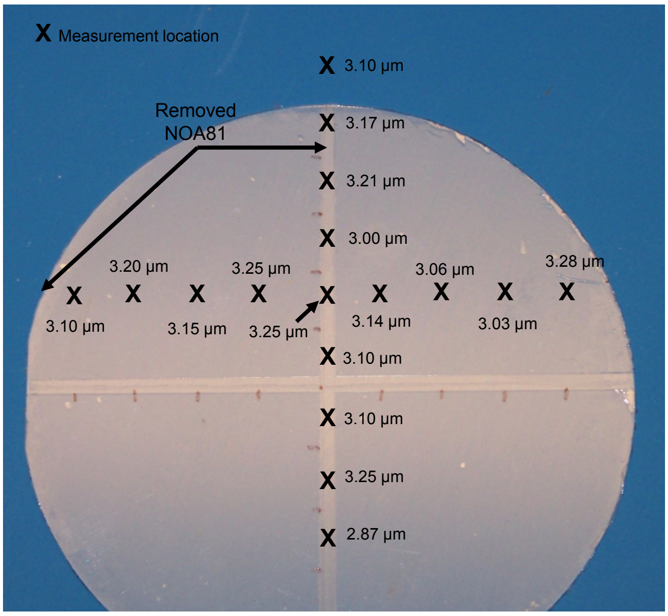

Figure S4: Assessment of the uniformity of the NOA81 gluing layer across a whole wafer after stripping of the FEP foil and creation of two stripes. The thickness of the NOA81 layer is indicated at different measurement positions, as measured by profilometry (Dektak).

References

1. V. C. Stimberg, J. G. Bomer, I. van Uitert, A. van den Berg and S. Le Gac, *Small*, 2013, **9**, 1076-1085.
2. M. Apanius, P. B. Kaul and A. R. Abramson, *Sensor Actuat a-Phys*, 2007, **140**, 168-175.

3. H. V. Jansen, J. G. E. Gardeniers, J. Elders, H. A. C. Tilmans and M. Elwenspoek, *Sensor Actuat a-Phys*, 1994, **41**, 136-140.