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Fabrication of AD/DA microfluidic converter using deep reactive ion etching of silicon and low temperature wafer bonding

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ABSTRACT

The purpose of this work is to describe an original process that has been designed for the fabrication of a microfluidic converter. The fabrication is based on deep reactive ion etching of silicon and low temperature full wafer adhesive bonding. The technology development includes an improvement of the bonding process in order to produce an adaptive strength of SU-8 bond which not only ensures absence of debonding failures during the silicon deep etching procedure and the subsequent dicing procedure, but also avoids the potential SU-8 overflow leakage into channels due to the bonding step. Besides, the originality of the work is not only in the process but also in the design of the device. Common actuation method for microfluidic system is either based on closed-channel continuous-flow microfluidic (CMF) or dropletbased microfluidic (DMF). Both of them have advantages and disadvantages, and their integration on a single system is in dire need. In this paper, we briefly discuss the concept of microfluidic converter, integrating CMF with DMF, which can: (i) continuously preload reagents, (ii) independently manipulate several droplets, (iii) recombine and export samples into closed-channel continuous flow, making it ideal for interfacing to liquid-handling instruments and micro-analytical instruments.

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1. Introduction

Microfluidic has revolutionized biomedical and healthcare applications, such as clinical diagnosis, DNA sequencing, other laboratory procedures and so on [\[1–4\].](#page--1-0) Currently, microfluidic mainly relies on closed-channel continuous-flow microfluidic (CMF) and droplet-based (digital) microfluidic (DMF). CMF is based on continuous liquid flow through micro-channels driven by electrokinetics or by pressure gradient on or off chip [\[5\]](#page--1-0), which is adequate for routine fluids handling and simple biochemical applications. However, it is less suitable for tasks requiring a high degree of flexibility in the manipulation of tiny liquid volumes. For achieving reconfigurability and scalability, DMF is extensively investigated [\[6,7\].](#page--1-0) One common actuation method for digital microfluidic is electrowetting-on-dielectric (EWOD) where an electric field can control of the wettability of liquids on a dielectric solid surface [\[8,9\].](#page--1-0) However, current implementation of digital microfluidic suffers from the lack of continuous-supply of reagents and the impossibility to export sample in continuous flow format. Thus the development of a fabrication method to integrate micro-channels and digital

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microfluidics in a single system plays a critical role in lab-on-chip systems.

Our work joins a small group of studies that have combine micro-channels and digital microfluidics in a common system introducing the concept of AD and DA conversions. Conversion of a continuous flow into discrete droplet is called by analogy with microelectronics 'Analog to Digital' (AD) conversion. Reciprocally, conversion of a train of discrete droplets into continuous flow is called 'Digital to Analog' (DA) conversion. Both operations are necessary steps towards fully automated lab-on-chip systems. The studies on this topic can broadly be classified into two groups. In the first one, they develop AD interface using EWOD within channel to facilitate droplet generation [\[10\]](#page--1-0) and in the second one, they develop DA interface using EWOD to precisely manage many different reagents and then channels to export the resulting products [\[6,11,12\]](#page--1-0). These works show a better match for pre-separation or post-separation applications. Here we propose a hybrid device, ''AD/DA microfluidics converter'', integrating both AD interface and DA interface. [Fig. 1](#page-1-0) schematically illustrates the configuration of AD/DA converter. The system consists in the following procedures: (i) continuously preload of reagents, (ii) independent manipulation of several droplets, (iii) recombination and export of samples into closed-channel continuous flow, making it ideal for interfacing to micro-analytical instruments.

Our AD/DA microfluidic converter is based on the assembly of a single-plate EWOD platform (base) and an electrode-free top plate

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Fig. 1. Schematic diagram of AD/DA microfluidic converter: generating and manipulating droplets, and ensuring washing.

with a network of micro-channel paths (cover). Using this configuration, two bonding processes are involved: (i) the cover is based on the assembly of silicon and Pyrex wafers by adhesive bonding, then deep reactive ion etching (DRIE) is used to define the microchannel paths, (ii) the base and the cover bind with precise alignment. In order to repeatably achieve high-quality bonding results, the bonding process and parameters, as well as wafer-to-wafer alignment, must be precisely controlled. In the following, we describe the fabrication and primary characterization of AD/DA microfluidic converter.

2. Methods

2.1. Materials

In this paper, SU-8 is used as bonding intermediate layer, because of its good properties, such as photopatternable, high mechanical strength, optical transparency, versatility, good adhesion on many different substrate materials and superior chemical stability [\[13,14\]](#page--1-0). Two-side polished 3-in. (100) Si wafers with 200 um thickness and 3-in. Pyrex wafers with 700 um thickness were employed. The photomasks were generated using a layout software (CleWin) and were printed on a chromium photomask.

2.2. Device fabrication

The fabrication process consists of three steps (as shown in Fig. 2): (a) the base with EWOD platform; (b) the cover with micro-channel paths; (c) the adhesive bonding of the base and cover. The micro-channel paths are patterned in silicon instead of SU-8 because in the process we have to fabricate very thick (200 nm) channel walls and the operation is more reproducible and reliable with silicon. Furthermore, having silicon in the system allows us to plan the addition of other fluidic functions such as filtering or sorting that need very high lithographic resolutions.

2.2.1. The base with EWOD platform

After cleaning procedure, a first 20 nm nickel layer is deposited on Pyrex substrate wafer by electron beam evaporation. Then, the nickel electrode array is photopatterned and formed by using positive AZ1518 photoresister. Whereafter, the first $1.5 \mu m$ thick SU-8 dielectric film is spun coated, at 4500 rpm with acceleration of 2000 rpm/s for 30 s. With these parameters, the uniformity of SU-8 film thickness can be realized. To further crosslinking, hard bake at 180 \degree C on a hot plate for 10 min is performed. Then, a second 50 nm nickel layer (the ground line) is formed by lift-off technology.

The second SU-8 film has two functions: protecting the ground line layer and used as an intermediate bonding material. We have studied the effect of the second SU-8 thickness on the displacement of the conductive droplets. We have analyzed two different thicknesses (450 nm and 1.5 μ m) and we have found (see Section [3\)](#page--1-0) that $1.5 \mu m$ is the right thickness. After that, a CYTOP film is spun coating without softbake. Finally, the crosslinking reactions will be continued during the bond thermal treatment, obtaining strong adhesion.

2.2.2. The cover with micro-channel paths

Fabrication of the cover is based on the assembly of silicon and Pyrex wafers by SU-8 adhesive bonding. To obtain a good adhesion of the SU-8 film, both the Pyrex and the silicon wafers need a dehydrating treatment on a hotplate at 200 $^{\circ}$ C. Based on the process described for the first SU-8 dielectric layer in Section 2.2.1, a photolithographic patterned 1.5 µm thick SU-8 layer is fabricated. Afterward, the two wafers are brought into contact and pressed against each other during the hardbake process (on a hotplate at 85 °C for 3 min) [\[16\]](#page--1-0), to enable moisture evaporation and prevent any void formation. Finally, they are bonded in WSB2 Wafer Substrate Bonding Unit. Deep reactive ion etching (DRIE) of the silicon layer is then used to define the micro-channel paths (inlet and outlet of the converter), followed by spin coating of a thin hydrophobic layer (CYTOP).

Fig. 2. Fabrication process: (a) base, (b) cover, (c) bonded cover and base, and (d) schematics of the process flow.

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