Microelectronic Engineering 110 (2013) 441–445

Contents lists available at SciVerse ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Fast and simple fabrication procedure of whole-glass microfluidic devices with metal electrodes

Jaroslav Kotowski [a,*], Vít Navrátil [a], Zdeněk Slouka [b], Dalimil Šnita [c]

[a] Institute of Chemical Technology Prague, Department of Chemical Engineering, Technická 5, 166 28 Prague, Czech Republic
[b] University of Notre Dame, Notre Dame, IN 46556, United States
[c] University of West Bohemia, Research Centre New Technologies, Univerzitní 8, 306 14 Plzeň, Czech Republic

ARTICLE INFO

Article history:
Available online 29 March 2013

Keywords:
Microfluidic system
Glass chip
Integrated electrodes
Micro-device
Fabrication

ABSTRACT

We developed a new process for fabrication of whole-glass microfluidic chips with integrated metal electrode arrays. Our process is based on a novel technique that enables reliable bonding of two glass substrates, one with a microfluidic channel and the other with an electrode array. The technique uses sodium silicate as an intermediate layer between the two glass substrates; this layer provides very tight bonding while preserving full functionality of the metal electrodes. Functionality of the electrode array was confirmed by impedance spectroscopy measurements of KCl solutions with different concentrations. To confirm the quality of bonding, we used a solution of fluorescein as a tracer of any poorly bonded areas. Our results showed that there was no fluorescein leakage out of the microfluidic channel and that the electrodes were free of sodium silicate where desired. Our method can be applied to fast and cost-effective prototyping of whole-glass microfluidic chips with integrated electrode arrays.

1. Introduction

Microfluidic systems (Lab-On-a-Chip) are widely used in biological/analytical applications such as blood cell sorting [1], DNA analysis [2], or immuno-diagnostics [3] because they combine complex laboratory functions with the low consumption of samples. One of the key components of microfluidic chips is a microelectrode array which can be used either as an active (e.g., source electrodes for AC or DC electro-osmosis [4]) or passive element (e.g., electrical potential sensing [5]). The first Lab-On-a-Chip devices were made of glass or silicon substrates [6] by fabrication techniques commonly used in the semiconductor industry.

Nowadays, polymeric materials such as PDMS, PMMA, and PC are often used for microfluidic system fabrication since they enable the low-cost and mass production of these systems through commercially available processes such as casting, hot-embossing, imprinting etc. Polymeric microfluidic systems are easy to fabricate, but they lack thermal stability and resistance to organic solvents. Problems can be also caused by the gas permeability of some polymeric materials (e.g., PDMS [7]).

Unlike to polymeric materials, glass possesses very high thermal stability and a broad range of available surface chemistries enables to tailor its surface properties to be tailored to specific needs. These properties are advantageously used in many applications e.g., chemical synthesis, the production of nanoparticles, reaction of supercritical solvents etc. [8–11]. Glass microfluidic systems in which integrated electrodes are usually made as two-layer systems where one layer bears microelectrodes (made by the deposition of metal layers followed by lithography) and the other layer contains microfluidic channels (made by etching in HF, laser machining, mechanical machining or sandblasting). A comprehensive summary of the fabrication processes used for glass microfluidic systems was published by Iliescu et al. [12]. The aforementioned manufacturing of microfluidic channels and electrode arrays on glass substrates through processes mentioned above is routinely conducted in many microfluidic laboratories. But the reliable, defect-free and leakage-free bonding of those glass substrates that preserve full functionality of electrodes is still quite challenging. The reason for this is the presence of a thin layer of electrodes, that makes the glass substrate surface rather uneven and heterogeneous. Heterogeneity and surface unevenness cause serious issues during the bonding process during which proper contacting of bonded substrates plays a crucial role.

Several techniques of glass-to-glass bonding have been published: high temperature bonding [13], low temperature bonding [14,15], thermal anodic bonding [16], and two-step activation bonding [17]. But they practically always fail to provide a tight bond and good channel sealing in the case of glass substrates with integrated electrodes. An interesting solution for this issue was published by Henry et al. [18]. The authors created small grooves

* Corresponding author. Address: ICT Prague, Technická 5, 166 28 Prague, Czech Republic. Tel.: +420 22044 3239; fax: +420 220 444 320.
E-mail address: jaroslav.kotowski@vscht.cz (J. Kotowski).

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http://dx.doi.org/10.1016/j.mee.2013.03.123

on a glass substrate which were later filled with a metal by a number of sputtering processes. However, repeated metal deposition requiring precise control of metal layer thickness makes the process quite difficult, costly, time consuming and not suitable for not well equipped microfluidics labs.

Due to fabrication difficulties in bonding glass substrates, several hybrid glass/PDMS microfluidic chips with integrated electrodes were developed [19–21]. In those microfluidic chips, glass and PDMS are used as a carrier of an electrode array and microfluidic channels, respectively. Bonding of those materials relies on traditional surface plasma activation which along with the flexibility of PDMS provides a good way of sealing microfluidic channels on glass substrates with uneven surface caused by the presence of electrodes. The group of Li [19] used a layer of SU8 photoresist coated on a silicon substrate to facilitate the bonding of PDMS by activation in ozone. Group of Shu-Ming prepared a hybrid glass/PDMS chip by using UV curable glue as an intermediate layer [20]. The use of these hybrid chips, however, is limited to low temperature and low pressure applications. Also different surface properties of materials, e.g., a wettable glass and a non-wettable PDMS might introduce unexpected issues in some applications.

In this paper, we present a fast and inexpensive method for the fabrication of whole-glass microfluidic chips with integrated metal electrode arrays. Our fabrication method requires minimum number of expensive devices and can be performed with standard laboratory equipment.

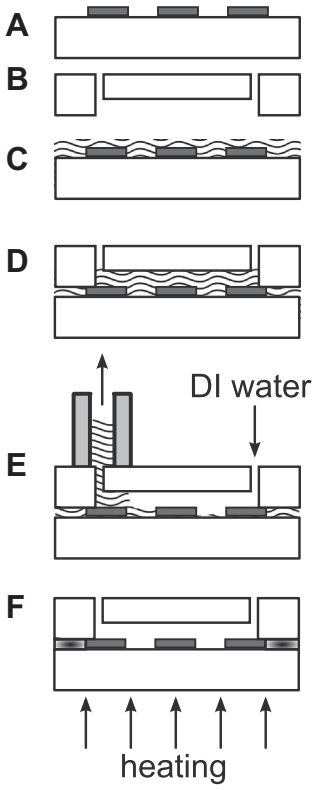

Fig. 1. Scheme of the whole fabrication process of the glass microfluidic chip with integrated electrode array: (A) glass slide with an electrodes array, (B) glass slide with a microfluidic channel, (C) coating of the glass slide with sodium silicate, (D) attachment of the mating glass slide, (E) removal of the excess of sodium silicate by vacuum suction and rinsing with DI water and (F) drying of sodium silicate at 95 °C for 60 min.

2. Experimental

2.1. Fabrication procedure

The entire fabrication procedure is illustrated in Fig. 1. Microscopic glass slides (75 by 25 mm) were used as glass substrates. Electrode arrays were made by the standard process based on the combination of metal sputtering and photolithography (photoresist P-ma 1275 by Microresist) (Fig. 1A). In the first stage, electrodes made of inexpensive nickel were used to develop and optimize the whole fabrication procedure. Nickel was later replaced by more expensive gold. The electrode array was designed as a four electrode array with the following parameters: width of the electrodes 200 μm, the gap between the electrodes 400 μm. In order to investigate the influence of the metal layer thickness on the quality of bonding, we prepared electrode arrays with different thicknesses ranging from 200 to 700 nm.

Microfluidic channels were etched into a second glass slide (Fig. 1B) through a structured polyester tape (type M42 by Europack Chrudim) in a solution made up of hydrofluoric acid, sulfuric acid, and DI water mixed in the volumetric ratio 1:2:1 [22]. The polyester tape serves as a mask for etching of the required structures. The tape was cut manually using a scalpel. Alternatively, a cutting plotter can be used [23]. The etching was performed in a laboratory shaker (IKA KS125) set to 300 cycles/min at room temperature. The etching rate was 5 μm/min. Via-holes located at the beginning and at the end of the microchannel were drilled by diamond coated ball burrs (by Dremel Motoflex) under water to prevent the local overheating and possible breakage of the glass substrate.

The glass substrate bearing the microelectrodes is coated with a non-diluted solution of sodium silicate (10% NaOH and 26% SiO_2 – known as water glass, by Sigma Aldrich) (Fig. 1C), and immediately covered with the glass substrate containing the microfluidic channel (Fig. 1D). The excess of sodium silicate located along the perimeter of the contacted glass substrates is rinsed off by DI water. The microchannel filled with the water glass is then connected to a vacuum pump (KNF N816.3KT) that removes the water glass from the channel.

To ensure that all water glass is removed from the channel and the surface of the electrodes is free of any sodium silicate solution, we flush the channel with DI water (approximately 2 ml) while the channel is still hooked up to the vacuum (Fig. 1E). The assembled chip is then put in an oven set at 95 °C for 60 min to dry the remaining water glass which bonds the two glass substrates together. Fig. 2A and B shows the fabricated glass chip.

2.2. Confirmation of glass bonding

To prove that the bond in glass/metal/glass sandwich is defect-free, we filled the micro channel with a fluorescein solution to trace any possible defects. Inspection was done visually after the chip illumination by UV light (using microscope OLYMPUS BX51WI/U-RFL-T).

2.3. Electrochemical impedance spectroscopy measurements

An important step in the fabrication procedure is flushing of the microfluidic channel. Flushing assures that the surface of the electrode array is free of any sodium silicate. To test that the electrodes are fully functional, we measured electrochemical impedance spectra of KCl solutions with different concentrations. Electrochemical impedance spectroscopy (EIS) measures the impedance of the electrochemical cell in the dependence on frequency of a low amplitude AC electrical signal. The results from EIS are traditionally plotted as a dependence of total impedance on the frequency (Bode plot) or real and imaginary parts of impedance are