The cleanroom-free rapid fabrication of a liquid conductivity sensor for surface water quality monitoring

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Abstract Conductivity is an important issue of water quality detection. Although there are a number of microfluidic liquid conductivity sensors, most of them were difficult to fabricate and required cleanroom facilities, which significantly increased the cost of sensors. Here, we presented a novel liquid conductivity sensor that was built on interdigitated copper electrodes. Microscale sensor electrodes can be fabricated through the modified microfabrication process without any help of cleanroom facilities. In comparison with FEA simulation and commercial conductivity device, measured results showed that the fabricated liquid conductivity sensor was able to measure a wide range of liquids. Considering the ease of fabrication and its low cost, the cleanroom-free fabricated sensors have much potential for water quality monitoring.

1 Introduction

With the rapid development in developing countries, the pollution in such countries is increasing as well. Among all types of pollution, water contamination is one of the most severe problems threatening public health. The water contamination can be divided into ground water contamination and surface water contamination. Both of them have played critical roles in the surface water quality (Pye and Patrick 1983; Rozemeijer and Broers 2007). To examine the water quality, there are many parameters need to be considered. Those parameters include discharge, dissolved oxygen, pH, total suspended solids, nitrate nitrogen, ammoniacal nitrogen and electrical conductivity (Ayers and Westcot 1985; Bolstad and Swank 1997; Shrestha and Kazama 2007). Although there are many existing microfluidic-based devices with the functions of the water quality detection, its fabrication process is relatively complex and requires cleanroom facilities (Glawdel et al. 2009; Lefèvre et al. 2012; Banna et al. 2014), which significantly increases its cost and therefore limit its applications. Even for the single conductivity sensors, most of them still need a cleanroom environment during fabrication (Hyldgård et al. 2008; Li et al. 2011), complex fabrication process (Ramos et al. 2008) or special ceramic materials (Fougere 2000), which brings many difficulties to the realization of the sensors and increases the fabrication cost dramatically.

Here, we present a microfluidics-based device to measure the liquid conductivity that was fabricated outside of the cleanroom facility. This device adopted an interdigitated structure as sensing electrodes to increase the sensitivity. The advantages of such structure include wide measurement range, simple principle and linear response (Ramos et al. 2008; Li et al. 2011; Banna et al. 2014). The proposed device was fabricated by simple photolithography and metal etching in a standard chemical bench without using a spin coater and mask aligner which are usually in a cleanroom. We verified the feasibility of the proposed sensor with finite element analysis (FEA) simulation and a commercial device. Both simulated and measured results are in the same range of the data from a commercial conductivity measurement device. The proposed sensor can measure the liquid conductivity from very conductive liquid (30 % w/v NaCl solution) to purified reverse-osmosis (RO) water. Compared to existing devices, the proposed
conductivity sensors have advantages such as cost effective and easy to fabricate, making the device much accessible for water quality monitoring in developing countries.

2 Materials and methods

2.1 Design and working principle

The general structure of the developed conductivity sensor is shown in Fig. 1a. The whole device includes two main sub-sections, the polydimethylsiloxane (PDMS) microfluidic channel, and the copper-coated glass slide with interdigitated electrodes for the conductivity measurement. The detailed schematic of interdigitated electrodes was presented in Fig. 1b. The liquids under test were injected into a central microfluidic chamber to measure the resistivity. Since there are ions in the liquid, the liquid filled in the chamber can conduct small current between two interdigitated fingers. The value of the current is determined by the resistivity of the liquid. In order to determine the resistivity, a multimeter was used to measure the resistance between two copper electrode fingers. The equivalent circuit of this device is shown in Fig. 1c. The resistance of copper is several degrees smaller than the resistance of the liquid. Thus, the resistance measured by the multimeter is exactly the resistance of the liquid. Divided into such resistance by the cross-sectional area of the electrode fingers, the resistivity can be calculated by the following formula (van der Pauw 1958):

\[
\rho = \frac{R}{l A}
\]  

where \(R\) is the resistance measured, \(l\) the gap between the electrodes, and \(A\) the cross-sectional area of electrodes, and \(\rho\) is the resistivity. The measured resistance values of each liquid solution were used to calculate the resistivity and the conductivity, which is the reciprocal of the resistivity.

2.2 Fabrication process

Figure 2 shows the cleanroom-free photolithography fabrication process. The mask of interdigitated electrodes was designed using DraftSight software (Dassault Systèmes, Vélizy-Villacoublay, France) and printed on a PVC film in a local printing store. Glass slides coated with a 100 nm thick copper layer were purchased from EMF Co. (Ithaca, NY, USA). Photoresist (Microposit S1813, Rohm and Hass Electronic Materials LLC, Marlborough, MA, USA) was pipetted on the whole surface of the copper-coated slide. Then the slide was held vertically several minutes to flow the excessive photoresist down and uniformly coat the photoresist on the copper surface. Subsequently, the slide was baked at 90 °C for 2 min on a hot plate. The photoresist was exposed to 365 nm ultraviolet light (ENF-260C UV Lamp, Spectronics Co., Westbury, NY, USA) through a mask for 12 min to transfer the pattern. The mask was then removed from the slide, and the slide was placed in the photoresist developer (MF319, Rohm and Hass Electronic Materials LLC, Marlborough, MA, USA).
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Fig. 2 Schematic of the clean-room-free fabrication process. a Photolithography was used to pattern copper electrodes. b PDMS channel was fabricated by the soft-lithography replica molding technique and tubing holes were punched. c Bond PDMS over the interdigitated electrodes and assemble tubes

LLC, Marlborough, MA, USA) for 2 min to remove the exposed photoresist from the slide. The slide was washed with reverse osmosis (RO) water. The photoresist pattern was transferred to the copper layer by the wet copper etchant (Ferric chloride, M.G. Chemicals Ltd., Burlington, ON, Canada) for sixty-seconds. Then the slide was washed by RO water and dried. The photoresist was kept covering the surface of the electrode finger but the side wall electrode fingers was exposed for measuring the resistance of liquids.

The PDMS channel was fabricated via 3D printed mold (CONNEX 500, Stratasys, Eden Prairie, MN, USA). The mold was cleaned with isopropanol and coated with Trichloro (1H,1H,2H,2H-perfluoro-octyl) silane (Sigma-Aldrich, St. Louis, MO, USA). Salinization was performed by adding 50 µl of the silane solution to a petri dish and placing the petri dish and the molds into a desiccator for 30 min. These processes are necessary to easily separate 3D printed molds with PDMS (King et al. 2014). PDMS elastomers (Sylgard 184 silicone elastomer base, Dow Corning Co., Midland, MI, USA) were mixed evenly with curing agents (Sylgard 184 silicone elastomer curing agent, Dow Corning Co., Midland, MI, USA) with the ratio of 20:1 and then poured over the mold placed in a petri dish. Subsequently, the petri dish was placed in a vacuum desiccator to remove air bubbles for 2 h. After all bubbles had been removed, the petri dish was put into the oven to bake the PDMS over the mold at 70 °C for 8 h. The petri dish was taken out from the oven, and then PDMS was peeled off from the mold and cut into the proper size. After that, the through-holes for inflow and outflow were punched.

The PDMS channel was treated by the oxygen plasma using a hand-held corona device (BD-20, Electro-Technic Products, Chicago, IL, USA) for 2 min. Subsequently, the PDMS channel was bonded to the glass slide. Then the whole device was baked in the oven at 75 °C for 3 h. After that, the steel tubes were assembled into the holes of inflow and outflow.

2.3 Computational simulation

The simulation of the conductivity sensor was performed by finite element analysis (FEA) using COMSOL 4.4 (COMSOL Inc., Palo Alto, CA, USA). The two-dimensional electrode structure model in the simulation was directly imported from the mask layout. Detailed simulation parameters utilized in the simulation were presented in Table 1.
3 Results and discussion

Figure 3 shows the fabricated device and the optical microscopic picture of interdigitated electrodes. It is clear that although the edge of the pattern was a little bit over etched, it still kept acceptable pattern resolution. According to the formula (1) above, the resistivity is not very sensitive to the small edge erosion in the microstructure when the erosion is small compared to the width of the fingers. Therefore, even though the cleanroom-free method slightly reduced the pattern resolution, the feasibility of the conductivity sensor remained good. Since the interdigitated electrode fingers on the conductivity sensor are in microscale without the facility in cleanroom, it is hard to fabricate the microstructure clearly. However, we can achieve 200 µm of the gap between interdigitated fingers.

The simulation result of electric potential distribution was shown in Fig. 4. The color change in the picture indicated the voltage distribution in the device during the measurement. From the picture, it is clear that voltage drops were occurred mostly in the chamber, which proves that the resistance of copper connections can be ignored. By monitoring the power consumed by the devices and applying ohm’s law, we can have the resistance of the liquid inside. FEA simulation was able to derive the resistance of the same configuration of the device directly via built-in 1D plot function. The gap between the electrodes and the cross-sectional area of electrodes were used in the simulation. Obtained resistance values from the simulation were used to calculate the resistivity using formula (1) and subsequently calculate the conductivity plotted in Fig. 5.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multi physics</td>
<td>Electric currents (EC)</td>
</tr>
<tr>
<td>General boundary condition</td>
<td>Electric Insulation</td>
</tr>
<tr>
<td>Initial voltage</td>
<td>0 V</td>
</tr>
<tr>
<td>Terminal setup</td>
<td>Left bottom line was with 0.01 A current source Right bottom line was connected to ground</td>
</tr>
<tr>
<td>Mesh</td>
<td>Physics-controlled mesh: normal</td>
</tr>
<tr>
<td>Solver</td>
<td>Stationary</td>
</tr>
</tbody>
</table>

Fig. 3 Pictures of the fabricated device. a An assembled device using the cleanroom-free fabrication method. b The microscope images of the interdigitated microstructure electrodes. (Scale bar 200 µm)

Fig. 4 Simulation result of electric potential distribution in the conductivity sensor. a General distribution (Scale bar 2 mm). b Details in the interdigitated electrodes (Scale bar 0.5 mm)
To verify the feasibility of this device, we utilized the fabricated device to measure the conductivity of various liquids, such as 30% w/v NaCl solution, 10% w/v NaCl solution, tap water and RO water. Also, the measured data were compared to reference data measured by a commercial conductivity sensor (Thermo-scientific Orion Star, Thermo-scientific, Fort Collins, CO, USA). Both measurements were conducted five times for each liquid under the room temperature around 20 °C. 5 mL of each solution to be measured was prepared in a syringe that was connected to the inflow of the sensor. The liquids were injected with the velocity of 200 µL/min by a syringe pump (Kent Scientific Corp., Torrington, CT, USA). The multimeter was connected to the two copper pads to measure the resistance between two pads. The output of the multimeter was recorded until its value reaches its stable point. Subsequently, the injection continued for at least thirty-seconds before the next measurement. The measurement was conducted five times for each liquid. After the test of each solution, the channel was washed with 10 mL RO water. At the end of the measurement, all the liquid in the microfluidic channel was taken out by the syringe manually.

As shown in Fig. 5 and Table 2, the measured data using the fabricated liquid conductivity sensor was in the same range of the result from FEA simulation and commercial sensor. The result indicated that although the resolution of a micro pattern was affected slightly by the cleanroom-free method, the conductivity sensor was working reliably to distinguish the conductivity level of various liquid solutions. The relatively large discrepancy between the fabricated device and commercial device has occurred in RO water due to that the resistance of the RO water was reached very close the maximum measurable resistance value of the multimeter (50 MΩ). Therefore, the multimeter was not capable of measuring the high resistance accurately. On the other hand, the difference between tap water and RO water was still significant, indicating that this device was able to distinguish very clean and slightly contaminated water. From this perspective, the measurement range of the device can cover from the purified RO water to extremely contaminated 30% salt water. This proves that the great applicability of this cleanroom-free device as the water quality monitoring sensor. Using this low-cost sensor chip, the surface water quality can be monitored in a better way. In the sacrifice of little accuracy, the cleanroom-free method significantly reduced the complexity and facility requirements during fabrication, providing an easy-to-fabricate and low-cost solution for making liquid conductivity sensors. Thus, the cleanroom-free fabrication method is an interesting technology and has the many potential applications for researchers who do not have access to expensive cleanroom facilities.

4 Conclusion

In this paper, a liquid conductivity sensor was designed and fabricated using the cleanroom-free fabrication method. The method could achieve highly sensitive microstructured interdigitated finger electrodes without using any cleanroom facilities. The measurement results from the fabricated device, commercial equipment, and FEA simulation were matched, demonstrating that this low-cost and easy-to-fabricate sensor was well suitable for monitoring the surface water quality. The measurement ranges were wide enough to measure from the salty liquid to the purified RO water. Furthermore, because there is no cleanroom facility requirement, and fabrication process is easy to follow, this fabrication technique opens the door for researchers who do not have access to expensive cleanroom facilities to build microscale devices.

References

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