Lasers in Manufacturing Conference 2021

Laser-manufactured glass microfluidic devices with embedded sensors

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Abstract

We describe a laser-based process that allows the rapid manufacturing of custom microfluidic devices from transparent borosilicate glass slides, as well as an inexpensive method that enables the integration of commercially-available fiber optic pH and pressure sensors with microfluidic devices. For this purpose, we fabricated a microfluidic device with bespoke ports in the inlet and outlet channels that were deliberately designed to embed the sensors. The microfluidic device was manufactured using an ultrashort pulsed picosecond laser (TruMicro 5x50, Trumpf), which was used to: (a) generate a microfluidic pattern on the glass surface by ablating the material; (b) drill an inlet, outlet and sensor ports in a second glass plate; and (c) close the microfluidic pattern from the top with a second glass plate by creating weld seams at the glass-glass interface and permanently bonding the two glass slides together. The fiber optic sensors were attached to the microfluidic device using custom connectors that were manufactured from transparent UV-curable resin using a desktop, stereolithography 3D printer (Form 2, Formlabs). The pH sensors ("pH SensorPlugs", manufactured by PreSens Precision Sensing GmgH) were tested with pH calibration buffers, while the pressure sensors (FOP-MIV, manufactured by FISO Technologies Inc.) were used to measure pressure directly in the ports during the flow of water through the microfluidic pattern, providing quantitative information on the dynamic events occurring in the microfluidic channels.

Keywords: Microfludiic devices; Ultrafast lasers; Laser abalation; Microwelding; Glass; Fibre optic sensors;

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1. Introduction

Microfluidic devices play an important role in different branches of life sciences, especially in biomedical-related fields, as well as in petroleum engineering and carbon dioxide (CO_2) research. In biomedicine, as described by Winkler et al., 2021, they are used as tools for cell manipulation and analysis, drug delivery and development, pathogen detection, tissue engineering, DNA sequencing, and many more. In petroleum engineering and CO_2 sequestration research, microfluidic devices are often used as physical models of porous media to investigate different phenomena related to flow, transport and reactive processes at microscale (Jahanbakhsh et al., 2020). In many of these applications, an in-situ measurement of the physical and chemical parameters in a microfluidic device is highly desirable because this allows better monitoring and hence understanding of the processes within the device. For instance, real-time measurement of pH, O_2 and/or CO_2 allows us to monitor chemical reactions, detect various pathogens or obtain in-situ conditions suitable for cell culture. The measurement of pressure at specific locations in the microfluidic device is important because it allows us to use these systems as models to better understand different mechanisms that govern the flow and transport of fluids in porous media, e.g. oil, CO_2 and brine in hydrocarbon reservoirs.

In this paper, we describe a custom glass microfluidic device that was manufactured using an ultrashort pulsed picosecond laser and equipped with miniature, commercially-available fiber optic sensors.

2. Materials and Methods

The microfluidic device shown in Fig 1 was manufactured using two 1.1 mm thick borosilicate glass slides (Borofloat 33, Schott AG) that were machined and welded together using a 50 W picosecond laser (TruMicro 5x50, Trumpf) following the procedure described by Wlodarczyk et al., 2019. This laser-based manufacturing technique enables rapid prototyping of glass microfluidic devices without the use of projection masks and hazardous chemicals to etch glass. The microfluidic device has an inlet, outlet and three round ports with a diameter of 2.8 mm, which were fabricated to attach the commercially-available fiber optic sensors. The microfluidic pattern has a depth of 40 ± 4 µm, as measured using an Alicona 3D surface profilometer, and contains a uniform structure in the center (see the insert in Fig 1) that models a porous medium with a porosity of approximately 44 %. On both sides of the structure, there are bifurcation channels to enable uniform injection of fluids into the pores.

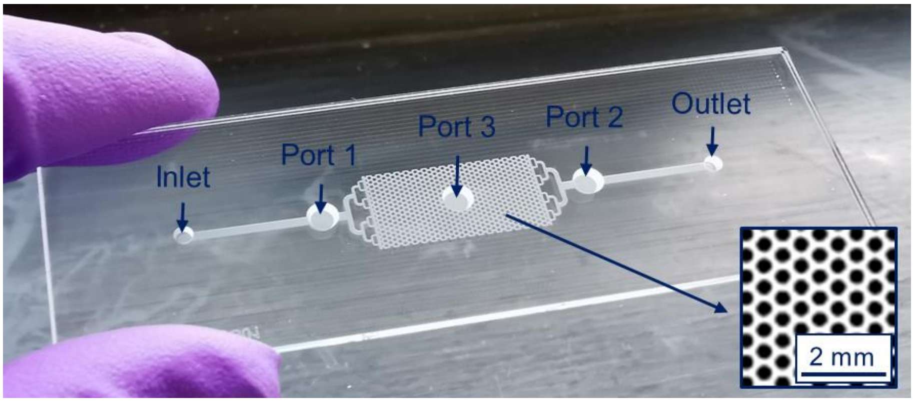

Fig. 1. Laser-manufactured glass microfluidic device used to embed commercially-available fiber optic pH and pressure sensors.

Two different types of sensors were embedded and tested with the microfluidic device shown in Fig 1. The first were pH SensorPlugs (model SPL-ML-HP5) that were provided by PreSens Precision Sensing GmbH (Germany). The second were miniature fiber optic pressure transducers (FOP-MIV) manufactured by FISO Technologies Inc. (Canada).

The pH SensorPlugs allow the measurement of pH in the range of 5.5 to 8.5 with an accuracy of ± 0.05. The time response of these sensors is less than 120 s. The pH SensorPlugs utilize male mini Luer connectors to which a thin, pH sensitive disc (fluorescent optode) with a diameter of approximately 2 mm is attached (see Fig 2a). In order to embed the sensors into the ports of the microfluidic device, custom connectors were designed and manufactured using a desktop, stereolithography 3D printer (Form 2, FormLabs). A 3D drawing of these connectors is shown in Fig 2a. Each part consists of a short thread, as can be seen in Fig 2b, so that the two parts can be easily connected together and disconnected if the inserted sensor must be removed from the microfluidic device. Parts A were attached to all three ports of the microfluidic device using epoxy adhesive (Araldite Standard, UK). Then the pH SensorPlugs were inserted into Ports 1 and 2 and gently secured with Parts A, as shown in Fig 2d. Due to the lack of a third pH sensor readout unit, the pH measurement could only be performed simultaneously in two ports, therefore in Fig 2d we show an example where the middle port (Port 3) was closed using a commercially-available male mini Luer plug. To ensure absolute tightness of the ports, the edges of the connectors were wrapped with PTFE tape before inserting them into the ports.

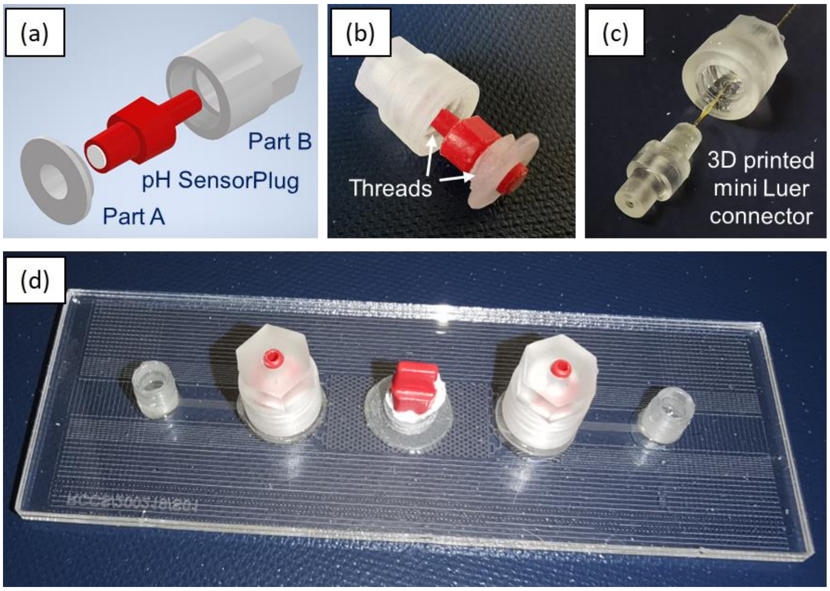

Fig. 2. Custom connectors manufactured using a Form 2 3D printer: (a) 3D drawing, (b) 3D printed parts with a fitted pH SensorPlug, (c) 3D printed mini Luer connector with a built-in FOP-MIV sensor, and (d) glass microfluidic devices with connected pH SensorPlugs.

After conducting experiments with the pH SensorPlugs, they were removed from the microfluidic device and replaced with FOP-MIV pressure sensors. Since the FOP-MIV sensors are significantly smaller than the pH SensorPlugs, first they were embedded into custom 3D printed mini Luer connectors (see Fig 2c) before insertion into Ports 1 and 2 of the microfluidic device. The FOP-MIV sensors have a diameter of only 0.55 mm, and they can measure pressures in the range of ± 0.4 bar with an accuracy of ± 1.3 mbar. The key component of the FOP-MIV sensors is a Fabry-Pérot (FP) cavity that contains a thin diaphragm that deflects under the influence of external pressure. When the diaphragm deflects, it reduces the length of the FP cavity and changes the distance between characteristic peaks in the so-called Free Spectral Range (FSR) of the FP cavity. To acquire the FSR from two FOP-MIV sensors, an in-house designed bespoke interrogation system and interrogation algorithm were used.

Before inserting the FOP-MIV sensors into the ports of the microfluidic device, they were calibrated against hydrostatic pressure. This was done by immersing the sensors in a 145 cm high glass cylinder filled with water and recording the FSR of the sensors cavities for different depths. Immersion of the sensors at the depth of 100 cm corresponded to a hydrostatic pressure of about 100 mbar, giving a calibration resolution of 1 mbar per cm.

3. Results

3.1. pH SensorPlugs

The pH SensorPlugs were attached to the microfluidic device, as shown in Fig 3a, and connected to commercially-available readout units (pH-1 SMA HP5-v2, supplied by PreSens) using polymer optical fibers (POF). To measure the temperature of the pH calibration buffer, which could affect its actual pH value, a Pt 100 temperature probe (also supplied by PreSens) was connected to one of the readout units to monitor the ambient temperature during the experiments. The pH values were recorded with a sampling rate of 0.33 Hz.

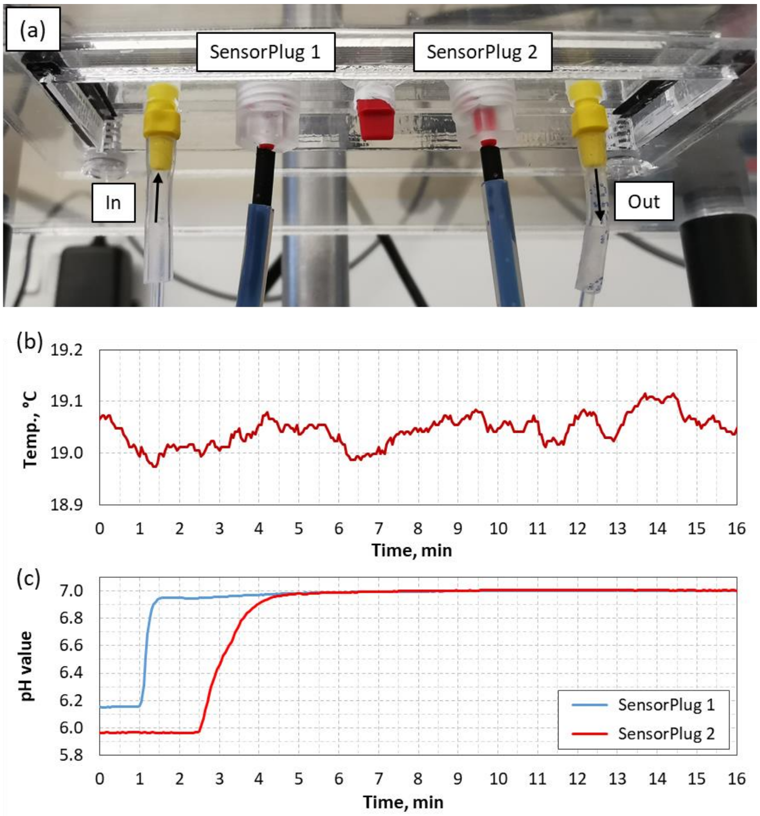

Fig. 3. (a) Microfluidic device with connected pH SensorPlugs, (b) temperature and (c) pH values measured during the injection of pH7 calibration buffer with a rate of 2 µL/min.

Calibration buffer solution (HP-70007P, Hanna Instruments Ltd.) with a nominal pH value of 7.03 at 20°C was injected into the microfluidic pattern with a rate of 2 µL/min. The ambient temperature was 19.0 ± 0.1 °C, as shown in Fig 3b. At the beginning, when the pattern was filled only with air, the pH values measured by SensorPlug 1 and SensorPlug 2 were 6.15 ± 0.01 and 5.96 ± 0.01, respectively, as shown in Fig 3c. When the buffer started to pass through Port 1, SensorPlug 1 immediately responded (in less than 3 s) and the pH value began to increase. A pH value of 6.95 was obtained in about 30 s, reaching a final value of 7.0 after several minutes. In turn, SensorPlug 2 detected a change in pH with a 90 s delay with respect to SensorPlug 1. This delay is in agreement with our expectations because the volume of the pattern between Ports 1 and 2 is approximately 3 µL and is expected to take this time to fill based upon the injection rate. As shown in Fig 3c, SensorPlug 2 responded differently from SensorPlug 1 because the rise time is slower (approximately 2 min). Nevertheless, after this time the sensor recorded the same pH value as SensorPlug 1. At this point, however, it should be emphasized that the accuracy of the pH measurement strongly depends on the physical contact established between the measured fluid and the sensing element (optode) of the SensorPlug. The appearance of trapped air (bubble) in the ports has been observed, which in some cases may cause the measured pH values to be underestimated and the time response of the sensors to be extended.

3.2. FOP-MIV pressure sensors

The FOP-MIV sensors were inserted and secured in Ports 1 and 2 (see Fig 4a). Then deionized (DI) water was injected into the microfluidic pattern (initially filled with air) with a rate of 5 µL/min. When the DI water arrived to Port 1, FOP-MIV #2 began to register an increase of pressure above the ambient pressure (see Point A in Fig 4b). The pressure in Port 1 increased to 12.5 ± 0.8 mbar (see Point B in Fig 4b) and then sharply dropped to 9.3 ± 0.8 mbar (after 15 s) when the fluid passed through the mini-Luer plug in Port 3 and reached Port 2. The arrival of water to Port 2 was immediately detected by FOP-MIV #1, which started to show an increase of pressure (see Point C in Fig 4b). The next 90 s show that the pressures in Ports 1 and 2 were changing over time until the DI water reached the outlet (see Point D in Fig 4b). Then, the flow of water through the microfluidic pattern reached equilibrium and both sensors began to show almost constant pressures. The pressure difference (ΔP) between Port 1 and 2 during the steady flow of water was measured to be 6.0 ± 0.8 mbar (see Fig 4c). As can be seen in Fig 4d, the DI water saturated almost the entire microfluidic pattern. The exceptions are Ports 1 and 2, some channels in the outlet branch and the periphery (edges) of the main pattern, where the flow velocity was expected to be the lowest. These areas have some air that was trapped during the flow of water.

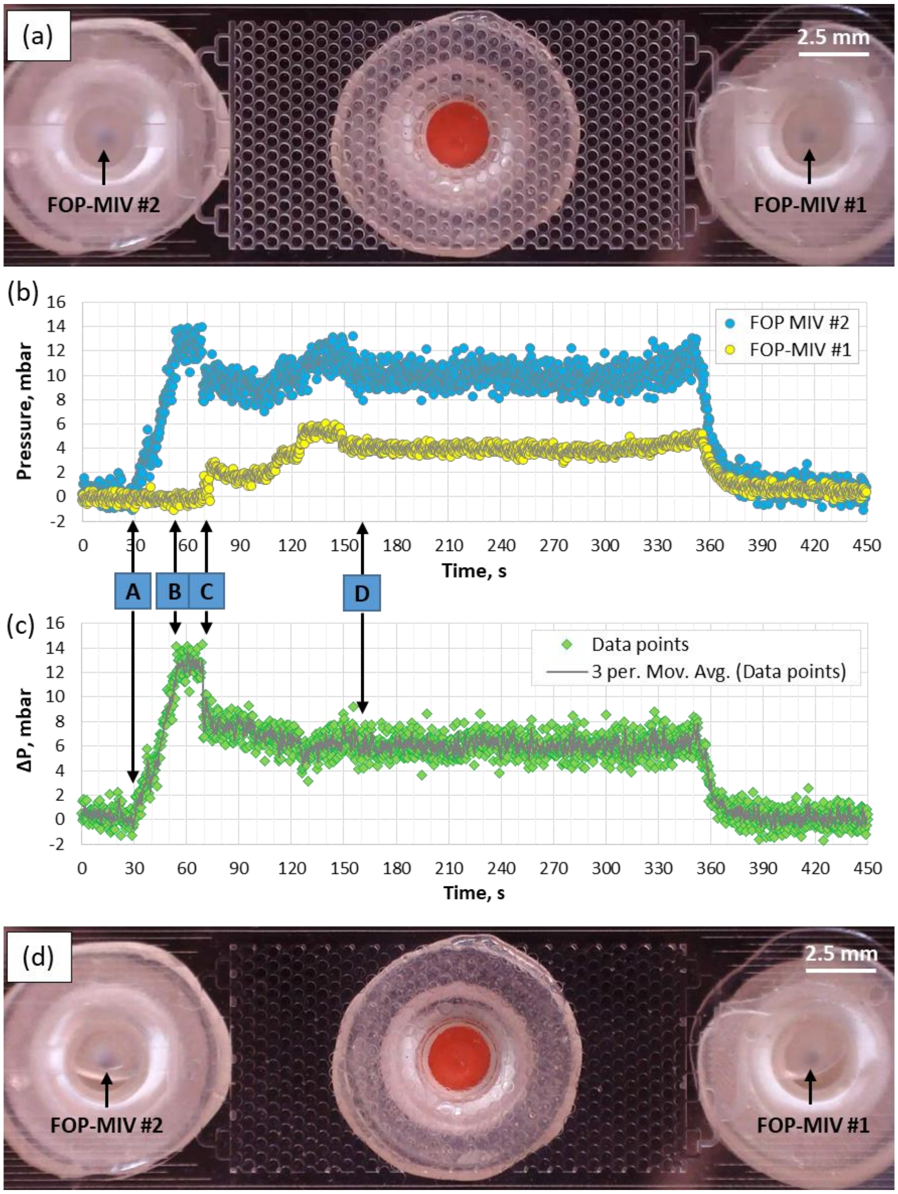

Fig. 4. (a) Microfluidic device with connected FOP-MIV sensors, (b) pressures and (c) pressure difference between Ports 1 and 2 measured during the flow of deionized (DI) water with a rate of 5 µL/min, and (d) microfluidic pattern saturated with water when the fluid reached the outlet (then the ΔP was nearly constant).

4. Conclusions

We have described an inexpensive method that enables the integration of commercially-available pH SensorPlugs and FOP-MIV sensors with custom microfluidic devices. The presented results show that the sensors enable in-situ real-time measurements of pH and pressure inside microfluidic patterns. The pH

SensorPlugs allow real-time monitoring of pH with an accuracy of ± 0.05, provided that sufficient physical contact between the fluid and the sensor optode is achieved, while the FOP-MIV sensors enable the measurement of pressures up to 0.4 bar with an accuracy of 1.3 mbar. This measurement range and resolution is suitable for testing this porous microfluidic model using different flow rates to investigate different phenomena occurring during the flow and transport of different fluids.

Acknowledgements

This research was funded by the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (MILEPOST, Grant agreement no.: 695070). This paper reflects only the authors' view and ERC is not responsible for any use that may be made of the information it contains. The authors also want to thank PreSens Precision Sensing GmbH (Germany) for provision of the pH SensorPlugs and related equipment with the necessary accessories, which were delivered as part of the 2020 SensorPlug competition.

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