“All-in-One” Chip Fabrication by 3D Femtosecond Laser Microprocessing for Biophotonics

K Sugioka¹, Y Cheng and K Midorikawa
RIKEN – The institute of Physical and Chemical Research, Wako, Saitama 351-0198, Japan
¹ksugioka@riken.jp

Abstract. We developed a novel technique fabricating 3D hollow microstructures embedded in photosensitive glass. The fabrication procedure consists of the following three steps: (1) femtosecond laser direct writing, (2) thermal treatment, and (3) wet etching in a diluted HF solution. The developed technique can fabricate various microcomponents such as microfluidics, microvalve, micromirror, microsplitter, freestanding fibre, and microfluidic laser. In this paper, integration of these microcomponents in a single glass chip by a single procedure is demonstrated for “all-in-one” lab-on-a-chip device manufacture.

1. Introduction
Today, a microchip, so called, a lab-on-a-chip device, is becoming a common tool for chemical analysis, medical inspection, genomic and proteomic science, and drug-discovery research [1]. Simple devices have been already realized, with development now shifting to “all-in-one” microchips in which the microfluidics, micromechanics and microdetector systems are integrated in a single chip for the next generation. However, the integration of such microcomponents using today’s technology, based on semiconductor processing including photolithography, alignment, assembly, and packing processes, faces great challenges because of the significant labour spent on multiple manufacturing tools. Therefore, it is preferable to develop a new technique by which both the fabrication and integration of the microcomponents can be completed in a single procedure. Femtosecond (fs) laser micromachining is a good choice for three-dimensional (3D) integration due to its ability to form internal structures in transparent materials.

In recent years, we have developed the rapid prototyping of 3D hollow structures buried in a commercially available photosensitive glass using fs laser micromachining [2-7]. Currently, microfluidic, micromechanical and microoptical components can be easily fabricated in the glass by the developed technique. One of advantages of the developed technique is that each type of microcomponent can be fabricated by the same process, which means that they can be integrated in a single glass chip by a single procedure without cumbersome alignment processes. Naturally, the next step is to integrate these microcomponents in a single glass chip and then to fabricate an “all-in-one” lab-on-a-chip device like a micro-total analysis system (µ-TAS). For example, in such a device as shown in figure 1, first, a laser beam is coupled into a waveguide, and then, the waveguide guides the beam to a liquid sample in microfluidics to perform optical absorption spectroscopy or fluorescence...
detection. Lastly, the second waveguide, which is connected to an optical spectrometer or a photodetector, collects the light transmitted from or emitted by the liquid sample for information acquisition.

In this paper, integration of several microcomponents in a single glass chip by a single procedure using 3D fs laser micromachining is demonstrated, and followed with demonstration of several functional microdevices for lab-on-a-chip applications.

2. Experimental

The glass used in our work is a photosensitive glass which is commercially available under the trade name of Foturan from Schott Glass Corporation. This glass is composed of lithium aluminosilicate doped with trace amounts of silver, cerium, and antimony [8]. The experiments were carried out at a commercial fs laser workstation, as described elsewhere [7]. The repetition rate, wavelength and pulse width of the fs laser were 1 kHz, 775 nm and 145±5 fs, respectively. The focusing system was a 20x microscope objective with a numerical aperture (NA) of 0.46. Samples under fabrication were

Figure 1. One example of “all-in-one” lab-on-a-chip devices.

Figure 2. Fabrication procedure of 3D hollow microstructures embedded in the photosensitive glass by (1) fs laser direct writing followed by (2) thermal treatment and (3) successive wet etching in a 10% HF solution. Microscope images of Foturan samples are shown on the right.
translated by a PC controlled x-y-z stage with a resolution of 0.5 μm. The fabrication process was viewed on a PC monitor by a charge-coupled device (CCD). Our technique of fabricating 3D hollow microstructures embedded in the glass consisted of three steps as shown in figure 2. The right-side photos show images of Foturan samples processed at each step. (1) First, three-dimensional latent modification zones are written inside the photosensitive glass by the fs laser direct writing. At this stage, silver atoms are precipitated, but no visible change occurs. The detailed irradiation conditions and the modification mechanism of photosensitive glass by the fs laser have been described in Ref. 9. (2) After exposure of the fs laser, the samples undergo a programmed heat treatment with temperature first ramped up to 500 °C at 5 °C/min and held at this temperature for 1 h, and then raised to 605 °C at 3 °C/min and held for another hour. Here, the laser-exposed regions become a crystalline phase of lithium metasilicate which appears as a dark colour. (3) After the sample is cooled to room temperature, it is etched in a solution of 10% hydrofluoric (HF) acid diluted with water in an ultrasonic bath. The formed lithium metasilicate is preferentially etched away with a contrast ratio of 43 in etching rate compared with unmodified volumes that defines the hollow microstructures inside the glass. Every internal structure requires formation of one or more modification channels that open into the sample surface for internal infusion of reagents.

In our study, microoptical components like a micromirror and a freestanding fibre were fabricated by the above technique. To achieve high performance of the microoptical components, surface smoothness of the components is strongly required. However, because the surface is somewhat rough after the HF etching, the samples were annealed again at 570°C to smooth the channel walls from 80 nm to 0.8 nm (average roughness) [4].

3. Integration of microfluidic and micromechanical components

To control flow direction of chemical reagents in the microfluidic circuit, a freely movable glass microplate, to serve as a microvalve, was fabricated in the hollow microchamber. The scheme for fabricating the movable microplate is similar to that described in Ref. 7.

Figure 3 (left) shows the schematic configuration of a microfluidic device integrated with the freely movable microplate that can switch the flow direction of reagents. The dark colour regions correspond to the hollow parts to be defined by the laser procedure inside the photosensitive glass. A photograph of the fabricated device is shown on the right. The microplate indicated by the arrow can move inside the glass by air infused from the side openings connected with syringes. When compressed air is applied to the left-side opening, the microplate moves to right side to seal the entrance of right-side outlet, and the reagent injected from the inlet will only flow into the left-side outlet, or vice versa.

Figure 4 shows microscope images that demonstrate the flow direction control of a liquid solution using the laser-fabricated structure: (1) Air was infused from the left-side opening connected with a silicone tube and a syringe (not shown in the photo), moving the microplate to the right side to seal the right-side outlet. To aid in the sealing of the microplate, the right-side outlet was pumped out by the syringe. Then, the liquid sample, in this case, red ink, was injected from the reservoir. (2) The
introduced liquid sample was pumped from the left-side inlet by the syringe, (3) so that the sample flowed to the direction of the left-side inlet. (4) Finally, the entire sample flowed into the left-side outlet without leakage into the right-side outlet. Thus, the fabricated structure can control the flow direction of liquid sample in the microfluidic circuit.

Figure 4. Demonstration of the flow direction control of a liquid sample using the fabricated structure shown in figure 3.

Figure 5. (a) Fabricated structure that comprises two series of freestanding fibres intercepted by a microwell fabricated in the glass chip. (b) Optical micrograph of part of the fabricated microstructure. (c) Optical micrograph of the cross-section of the freestanding fibre.

4. Integration of microfluidic and microoptical components
It is desirable in some lab-on-a-chip devices to directly analyze the on-chip reactants as they form and are carried through the microfluidic network. For the analysis, optical means such as absorption spectroscopy or fluorescence measurements are commonly employed. Therefore, microoptical
components like a micromirror, a microlens, a microbeam splitter, a microoptical sensor, and a microlight source should be integrated in the glass chip.

The present technique of fabricating the hollow microstructure has been applied to the fabrication of the micromirror and the microbeam splitter embedded in Foturan glass [4]. Use of the same technique makes it possible to fabricate and integrate both of the microfluidic and microoptical components in a single procedure, which eliminates cumbersome alignment processes of each microcomponent and also reduces the number of manufacturing steps.

The microoptics and the microfluidics have been integrated in a single glass chip for fabricating a microfluidic dye laser which is useful as a light source for optical analysis like fluorescence detection or photoabsorption spectroscopy in the lab-on-a-chip device [6]. The fabricated microlaser can cover any wavelength in visible range by changing the kind of laser dye and controlling its concentration.

For the optical analysis, an additional microoptical component to guide the laser beam to the microfluidic network is necessary. For this purpose, we fabricated a freestanding solid glass fibre within the glass chip by the present laser technique [10]. The optical propagation loss of fibre was estimated to be 0.7 dB/cm. The freestanding fibres can easily be incorporated into the microfluidic circuits. Here, we fabricated two collinear freestanding fibres that were intercepted by a microwell as illustrated in figure 5 (a). Figures 5 (b) and (c) show the optical micrographs of a part of the fabricated microstructure and the cross-section of the freestanding fibre, respectively. The coupling loss between the two fibres intercepted by the microwell was approximately 1 dB.

The fabricated fibre structure was applied to fluorescence detection of a liquid sample. The microwell was filled with laser dye Rh640 and then excited by a probe laser beam introduced into the left-side fibre. Figure 6 shows a photo of the device undergoing fluorescence detection. Bright fluorescence from the dye is clearly observed from the microwell. A small part of the fluorescence light is collected and guided by the second freestanding fibre arranged at right side of the microwell and could be detected by a spectrometer.

5. Conclusions

We developed a novel technique fabricating 3D hollow microstructures embedded in the photosensitive glass by fs laser direct writing followed by the thermal treatment and successive wet etching in a HF solution. The developed technique can fabricate various microcomponents such as microfluidics, microvalve, micromirror, microsplitter, freestanding fibre, and microfluidic laser. These components can easily be integrated in a single chip. The most important feature is that every component can be fabricated by a single procedure. This feature can eliminate the highly accurate repositioning of the sample on the stage for integrating each component, thereby reducing the number of manufacturing steps and the cost. Our next step is to integrate these microcomponents in a single chip for manufacture of “all-in-one” lab-on-a-chip devices as shown in figure 1 for biochemical microanalysis.
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