Development of Pressure-Sensitive Channel Chip for Micro Gas Flows

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Abstract. Optical measurement techniques are useful for experimental studies on micro gas flows, which enable us to non-intrusively measure the flows with a high spatial resolution. The pressure-sensitive paint (PSP) technique, which is based on the emission of photons from luminophore, is a potential diagnostic tool for pressure measurement of micro gas flows. However, measurements by conventional PSPs are limited to the sub-millimeter order spatial resolution of ca. 200 μm, indicating the difficulty of the micro scale measurements. The present study proposes pressure-sensitive channel chip (PSCC) which is a micro channel with the capability of measuring pressure. We focused on the poly(dimethylsiloxane) (PDMS) micro-molding technique, which is one of the most popular techniques to fabricate a micro channel easily. Moreover, PDMS is a polymer used as a binder in PSP because of high optical transparency, gas permeability, and gas diffusivity. Thus, we developed a micro channel by the PDMS micro-molding technique with mixing a pressure-sensitive luminophore into PDMS: i.e. a micro channel fabricated by PSP, which is named PSCC. A flow through a micro converging-diverging nozzle with the throat width of 120 μm was demonstrated. The pressure distribution on the nozzle surface was successfully obtained by PSCC.

1. Introduction
It has been strongly desired to understand thermo-fluid phenomena around micro- and nano-systems such as a magnetic head slider of hard disk drive, a micro thruster for a micro satellite, and a semiconductor thin film fabrication system and so on. Flows through a micro system should be treated as high Knudsen number flows (Kn = λ/L: λ is the mean free path for gas molecules and L is the characteristic length of the flow). There are no appropriate techniques for the measurement of gas pressure on solid surfaces inside micro-systems. To measure pressure distributions in high Knudsen number regimes, we have focused on a pressure sensitive paint (PSP) technique [1, 2]. Because PSP works as a so-called “molecular sensor”, it seems suitable for the analyses of high Knudsen number flows, which require diagnostic tools in molecular level. The pressure measurements of the flows in a small object were carried out by Huang et al. [3] and Nagai et al.[4]. However, the spatial resolutions of their measurements are limited to sub-millimeter order of ca. 200 μm due to the aggregation of luminescent molecules in polymer binders [5]. Moreover, conventional PSPs are very thick [2] (> 5 μm) compared to the dimension of micro-devices owing to the use of polymer binders. In the previous study, we developed pressure-sensitive molecular film (PSMF) by the Langmuir-Blodgett (LB) technique to construct ordered molecular assemblies, and tested PSMF to clarify the feasibility of the
pressure measurement around micro-devices \[6, 7, 8\]. Because the fabrication of PSMF takes a long time and the application of PSMF is restricted only to a flat chemically-modified surface, the measurement by PSMF is not easy to be applied to practical measurements. We proposed pressure-sensitive channel chip (PSCC) which is a micro channel with the capability of measuring pressure \[9\]. We focused on the poly(dimethylsiloxane) (PDMS) micro-molding technique, which is one of the most popular techniques to fabricate a micro channel easily. Moreover, PDMS is a polymer used as a binder in PSP because of high optical transparency, gas permeability, and gas diffusivity. Thus, we developed a micro channel by the PDMS micro-molding technique with mixing a pressure-sensitive luminophore into PDMS: i.e. a micro channel fabricated by PSP, which is named PSCC.

2. Fabrication of PSCC
The fabrication process of PSCC is very simple, because most of the processes are the same as the PDMS molding technique. PSCC consists of three parts: (i) a sensor layer; (ii) a gas barrier layer; (iii) a supporting PDMS block. A sensor layer is fabricated by a pressure-sensitive luminophore and PDMS with high gas permeability. The high gas permeability of a binder is required to achieve high pressure sensitivity. A gas barrier layer is necessary to prevent the permeation of oxygen molecule from a sensor layer to a supporting block and vice versa, because oxygen molecules through a supporting block to a sensor layer changes the luminescent intensity of PSCC regardless of the pressure in the channel and will cause measurement error. The high gas permeability and diffusivity of PDMS are drawbacks from this standpoint. A supporting block is fabricated by pure PDMS to fix these layers.

The actual fabrication procedure is as follows (see Figure 1): firstly, a master of SU-8 photoresist (MicroChem, SU-8 3050) on a silicon wafer was fabricated using standard photolithography procedures (Figure 1(1)). \(\text{Pt(II) tetrakis(pentafluorophenyl)porphyrin (PtTFPP)}\) was adopted as a pressure-sensitive luminophore, and a PDMS prepolymer (Dow corning TORAY, Silpot 184) was prepared by mixing PtTFPP in the mass ratio of \(\text{PtTFPP:PDMS = 0.1 mg : 1.0 g}\). Then the degassed PDMS prepolymer containing PtTFPP was poured over the master, and was spun at 1000 rpm to make thin layer of the PDMS with luminophore (sensor layer, see Figure 1(2)). The parylene (KISCO, dix C) layer was deposited on the sensor layer using parylene coater (KISCO, DACS-LAB) as a gas barrier layer \[10\] with the thickness of ca. \(0.5 \mu\text{m}\) (Figure 1(3)). A pure PDMS prepolymer (without PtTFPP) was poured over the sample and was cured at \(70^\circ\text{C}\) for \(60\) min to form a supporting block (Figure 1(4)). This three-layer structure was peeled off from the master, and the PSCC was obtained. For the measurement, the PSCC was holed for tube connections and was placed on a glass slide (Figure 1(5)).

3. Stern-Volmer relation
The pressure measurement technique using PSCC is based on the oxygen quenching of luminescence. When PSCC is illuminated by UV/blue light \((350 – 420\) nm), the luminescent molecules are excited by absorption of photon energy, and then the molecules emit phosphorescence. On the other hand, oxygen molecule with triplet ground state acts as a quencher of the phosphorescence \((630 – 700\) nm for PtTFPP\[1, 4\]). As a result, the phosphorescence intensity decreases as an increase in the partial pressure of oxygen gas. Pressure on the PSCC surface can be deduced from the relationship between pressure \(p\) and the luminescence intensity \(I\) (Stern-Volmer plot \[1, 2\]),

\[
\frac{I_{\text{ref}}}{I} = A + B \frac{p}{P_{\text{ref}}}, \tag{1}
\]
Figure 1. A schematic process of PSCC.

Figure 2. Stern-Volmer plot for the PSCC used in this study, where the reference pressure $p_{\text{ref}} = 15$ kPa. Fitting line: $I_{\text{ref}}/I = 0.61 + 0.39p/p_{\text{ref}}$

where the subscript “ref” describes the known (reference) condition. The coefficients $A$ and $B$ are the Stern-Volmer coefficients which are determined by a calibration test, and show temperature dependencies. In this study, the temperature dependencies of the Stern-Volmer coefficients was neglected, because our experiment was carried out in a constant temperature condition.

The Stern-Volmer plot of our PSCC is shown in Figure 2, where the reference pressure $p_{\text{ref}} = 15$ kPa. It is shown that the experimental data was well fitted by Equation 1 with the constraint condition of $A + B = 1$ at $(I_{\text{ref}}/I, p/p_{\text{ref}}) = (1, 1)$. The pressure sensitivity of PSCC of 0.39 is similar to that of conventional PSPs [1], and is applicable for pressure measurements.

4. Experimental Setup
The PSCC employed in this study was a converging-diverging nozzle. As shown in Figure 3, the converging and the diverging angle were $90^\circ$ and $24^\circ$, respectively. The throat width and the diverging length were 120 and 485 $\mu$m, respectively. The depth of the nozzle was 59 $\mu$m. The micro-nozzle was put on a holder with an inlet and an outlet port (see Figure 4). Oxygen (21.0 %) gas balanced with nitrogen gas was supplied from the inlet port, and the outlet port was evacuated by a scroll pump (Ulvac, DVS-631). The pressures of the inlet and the outlet were monitored by capacitance manometers (Keyence, AP-44) connected to pressure taps. Since both the inlet and the outlet port were attached far from the nozzle throat (1.2 cm), the effects of inlet and outlet conditions can be negligible.

The images of the PSCC emission were recorded with an EM-CCD camera (Princeton...
Figure 3. Geometry of PSCC (converging-diverging nozzle).

Figure 4. Schematic image of micro-nozzle assemblies.

Instruments, ProEM:1024B) mounted on a fluorescent microscope (Olympus, BX61). We adopted a band-pass filter (400 ± 20 nm), a long-pass filter (600 nm), and a dichroic mirror (reflection at 400–450 nm, transmission at 535–700 nm) in the microscope. An objective lens, Olympus MPlanFLN 10× with a numerical aperture of 0.30, was used for the measurement. The images were taken by a camera with a binning of 2 × 2 pixel, 1 pixel of the image corresponds to 2.6 μm.

5. Results and discussions

Figure 5 shows the pressure distribution of the nozzle with the inlet port pressure $p_{\text{in}} = 30.1$ kPa and the outlet port pressure $p_{\text{out}} = 2.9$ kPa. The images were taken with exposure time of 0.05s, and 32 images were averaged to reduce the noise for wind-on and wind-off (reference) images. The pressure distribution on the nozzle surface was successfully obtained without any aggregations of luminophore observed in conventional sprayed PSPs[5, 8]. The PSCC captured the sharp pressure drop near the throat 160 < $x$ < 250 μm as shown in Figure 6. As shown in Figure 7, the pressure distribution at the nozzle exit was a parabolic curve. The pressure near the wall was measured, while the pressures at the edge of the wall contain considerable errors due to the scattered light. The fluctuation of the measured pressure values were ±0.5 kPa at the lowest SN ratio area (ex. $x = 10$μm) and this is much smaller than that by PSMF [8]. The pressures at $x = 0$ and 850 μm were 20.5 ± 0.3 kPa and 11.7 ± 0.5 kPa, respectively. These measured pressures were considerably different from $p_{\text{in}} = 30.1$ kPa and $p_{\text{out}} = 2.9$ kPa at the pressure taps, which were attached far from the channel (ca. 1 cm). These pressure drops would be caused by wall friction and the acceleration at the long converging area of the nozzle (see Figure 3).
Figure 5. Pressure distribution of the nozzle.

Figure 6. Pressure distribution along the centerline of the nozzle.

Figure 7. Pressure distribution along the crossline of the nozzle exit.
6. Conclusion
We have developed a new pressure sensing tool named pressure-sensitive channel chip (PSCC) by combining the pressure-sensitive paint (PSP) technique with the poly(dimethylsiloxane) (PDMS) micro-molding technique. PSCC is a micro channel which itself works as a pressure "distribution sensor. In this study, a micro converging-diverging nozzle with the throat width of 120 μm was prepared and the pressure distribution on the nozzle surface was successfully obtained by PSCC. It is shown that PSCC is feasible to measure the pressure in micro gas flows.

As a future work, we are going to apply PSCC to dissolved oxygen (DO) measurement. The measurement of DO in water is very important in many research fields, because oxygen plays an important role in biochemical reactions, chemical reactions and so on. The DO distribution measurement will facilitate understanding of the mechanism of such reactions. We have proposed a novel utilization of PSP as a DO sensor film for macro scale unsteady DO distribution measurements [11]; thus it is considered that PSCC can be applied to “micro scale” DO measurement. For DO measurement, Equation 1 is written as follows:

\[
\frac{I_{\text{ref}}}{I} = A_c + B_c \frac{[O_2]}{[O_2]_{\text{ref}}},
\]

where \([O_2]\) is DO concentration. Since the measurement system is the same as presented this paper, this new method easy to be developed. PSCC will be a promising measurement method for micro DO measurement method.

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