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Gas Permeation in PDMS Monitored by On-site Pressure Sensors

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***Abstract*—This paper proposes a new methodology to detect the gas-leakage and the corresponding diffusion coefficient of PDMS. The authors use PDMS instead of Pyrex #7740 glass to seal the backside V-grooves of pressure sensor chips. The packaged sensor is put into a pressure testing machine and pressurized with CO_2 at 300 psi. By observing the output voltage, the time history for CO_2 permeating into cavity of the sensor was easily to be found. In this paper, the authors use several PDMS membranes with different thickness, from 45 to 2000 μm, to package the sensors and investigate the gas-leakage of PDMS. The gas leaking through PDMS is shown to be governed by the diffusion mechanism, and the diffusion coefficients derived from CO_2 leaking history of PDMS chip frames is 2.2×10^{-9} m^2 /s, matched with the previous work. The thickness effect of PDMS on the diffusion mechanism is also addressed.**

***Index Terms*—PDMS, permeation, pressure sensor, CO_2**

Nomenclature

D	=	diffusion coefficient
n	=	the number of moles of gas
P	=	instantaneous pressure
R	=	gas constant
T	=	temperature
t	=	diffusion time
V	=	volume
V	=	output voltage
x	=	diffusion length

I. Introduction

In recent years, Polydimethylsiloxane (PDMS) has become a very popular material in the fabrication of micro-scale devices [1-3], especially for microfluidics research like micro-pumps [4-6], micro-mixers and micro-channels [7]. The main reason for PDMS becoming so popular is based on its hyper-elastic toughness, transparency, easy in fabrication and low cost. Another very important advantage is that PDMS can be suitable in low-temperature process and bonded with variable materials [8]. Yang *et al.* used PDMS instead of Pyrex #7740 glass to seal the backside V-grooves of pressure sensor chips to substantially lower the package cost and to maintain the output performance of pressure sensors in a quite acceptable manner [9, 10].

Another intrinsic characteristic of PDMS is its porous structure. Although it has a very good isolation for liquid, PDMS often causes the infiltration phenomenon for gas molecules. This limitation causes the PDMS-packaged pressure sensors [9] to be applied only to dynamic monitoring of pressure rather than the precise measurement of static pressure. The gas permeability issue of PDMS has been brought up in 1997. Merkel *et al.* designed experiments to determine the permeability coefficients of different gases [11]. There are two more groups studied the permeation behavior of various gases through poly PDMS [12, 13]. Others even used such selective permeability to produce vacuum-driven pumps [14].

Additionally, the PDMS-based microfluidic devices integrated with micro incubation systems on a chip for cell culture appeared in 2007 [15]. In the general procedure of cell culture, the cells are posited in 37°C, 5% CO_2 humidified ambient. Based on the fact that CO_2 plays an important role to balance the pH value in the cell-culture medium, the authors proposed a novel approach of using backside V-groove of a pressure sensor chip to bond on a PDMS chip frame for investigating CO_2 gas-leakage through PDMS. By this way, the properties of CO_2 transporting through the PDMS can be studied via the output of pressure sensors and it benefits the design of PDMS-based microfluidic chip integrated in a micro-incubation system specifically.

II. Experiment and Discussions

A. Experimental setup

In order to explore the gas permeation of PDMS, the authors put the sensor into a pressure testing machine and pressurize it with CO_2 at 300 psi. In this work, the authors first use two PDMS membranes with different thicknesses, 45 μm and 500 μm, to package the sensors as Fig. 1 and explore the gas-leakage phenomena of PDMS membrane.

The dimension of the buckmachined pressure sensor is also shown in Fig. 1. The bottom of the packaged pressure sensor fixed to a printed circuit board (PCB), so gas would only enter the pressure cavity by the side-walls of the PDMS membranes (Fig. 2). The authors put the packaged pressure sensors in the pressure testing machine for enough time to monitor the output signal changing. The experimental setup (Fig. 3) is accessed to retrieve output signals of pressure

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sensors with packaging by Pyrex #7740 glass and PDMS at the same time. The time step of data retrieve for the data acquisition system (SPARTAN-L, Integrated Measurement & Control, Inc.) is 500 μs, and each exporting data takes average for every 2000 points. Therefore, the averaged data show one time per sec.

The authors use a power supply to provide 5 V DC bias voltage for the silicon piezoresistive pressure sensors. The pressure would slowly increase to 300 psi in 5 minutes for avoiding huge temperature changing in the test chamber and maintain over there to observe the situation of gas permeation. Figs. 4 and 5 are the output performance of pressure sensors packaged with Pyrex #7740 glass versus 500 μm thick PDMS, and with Pyrex #7740 glass versus 45 μm thick PDMS, respectively.

B. Background pressure leakage

In order to maintain the pressure stability of the pressure chamber in pressure testing machine and guaranteeing the safety during the measurement, twelve sets of bolts will be set up around the pressure chamber. Moreover in the pressure chamber of the contact surface between the upper and lower cover, the authors will use the oxygen free copper (OFC) ceramic washers to enhance air tightness. However, even the pressure of chamber is hoped to continuously maintain at 300 psi, nearly twenty times larger than the atmospheric pressure, it's still difficult to exactly keep the pressure of 300 psi in the testing machine. In Figs. 4 and 5, there is a small voltage drop in output signal of Pyrex #7740 glass packaging pressure sensors, and it's regarded as the background pressure leakage (baseline) of the experimental result.

Fig. 4 shows that there is no more output signal changing after 2500 sec in the test of the pressure sensor packaged with 500 μm thick PDMS, whereas the sensor packaged with 45 μm thick PDMS has no output signal changing after 8800 sec. This is because the pressure inside the sensor cavity is balanced with the high-pressure of machine chamber, and there is no pressure difference across the PDMS membrane. Additionally, the pressure of whole test environment decreases from 300 psi to about 280 psi due to the gas-leakage from pressure chamber by observing the signal output of pressure sensors packaged with Pyrex #7740 glass and the pressure gauge of the testing machine.

C. 1-D Gas leakage model

In this work, the authors try to explain the gas-leakage model from the perspective of gas diffusion depicted in Fig. 6. The CO_2 gas would diffuse from higher pressure P_1 to lower pressure P_2, where P_1 is the testing environment with 300 psi, and P_2 is the pressure inside the sensor cavity. The mathematical model derived from the one-dimensional (1-D) diffusion equation for the leaking process could be simplified to Eq. (1) [16].

$$\frac{P(\mathrm{x},t)}{P_{\max}} = erfc\left(\frac{\mathrm{x}}{2\sqrt{Dt}}\right) \tag{1}$$

where $P(\mathrm{x},t)$ denotes the instantaneous pressure occurred at any point x of PDMS and at anytime t since the initial pressure setting of P_{max}. The function *erfc* is the complementary error function. D is the (equivalent) diffusion coefficient of this PDMS problem.

The output voltage of the pressure sensor is regulated by a linear equation [17]:

$$\frac{V_{out}}{V_{\max}} = \frac{\Delta R}{R_0} = kP \tag{2}$$

where V_{max} is bias voltage; V_{out} is the output voltage, which varies with the applied pressure P. Combined with the equation of state for ideal gas (which assumption deviates from the real case of air), $PV = nRT$, the authors could conclude Eq. (3) to fit the gas-leakage phenomena of PDMS in a theoretical matter:

$$\left\{\frac{V_{out}(\mathrm{x},t)}{V_{\max}} = \frac{P(\mathrm{x},t)}{P_{\max}} = erfc\left(\frac{\mathrm{x}}{2\sqrt{Dt}}\right) = \frac{\mathrm{maximummass - leakagemass}}{\mathrm{maximummass}}\right. \tag{3}$$

As $erfc\left(\frac{\mathrm{x}}{2\sqrt{Dt}}\right) = 0.5$, the similarity variable $\left(\frac{\mathrm{x}}{2\sqrt{Dt}}\right)$ is also near to 0.5. Therefore the diffusion time t subject to $(V_{out}/V_{max}) = 0.5$ or the half pressure is shown in Eq. (4).

$$\frac{V_{out}}{V_{\max}} = 0.5 \Leftrightarrow \frac{\mathrm{x}}{2\sqrt{Dt}} = 0.5 \Leftrightarrow \mathrm{x} = \sqrt{Dt} \tag{4}$$

For the convenient interpretation of the experimental data with different thickness of PDMS, the authors retrieve data from Figs. 4 and 5 and plot them in Fig. 7 by normalizing their maximum voltage. For providing more investigation and evidence of the thickness effect of PDMS in the next section, the authors add another set of pressure leaking data tested from a pressure sensor with PDMS of 2000 μm thick.

D. Thickness effect of PDMS

Due to the two-dimensional configuration of the more realistic pressure sensor chip frame in Fig. 2, the thickness effect of PDMS beyond the prediction of Eq. (3) should be addressed more herein. With the clarification of the distinct gas diffusion pathways and the "equivalent" diffusion length depicted in Fig. 8, the one-dimensional diffusion model of Eq. (3) still works. (However, the two-dimensional diffusion equation is not the governing equation for the pressure field $P(x,y,t)$ in this study. Even not shown herein, the finite difference simulation has been done by the authors and showed the contradictory result trend of the half pressure time in TABLE I.)

The diffusion path or the thickness of PDMS in Fig. 8(a) is only 45 μm. Such a thickness is much smaller than the distance (1072.5 μm) from V-groove edge point A to outer interface point B. Therefore, the gas molecules at the middle of V-groove cavity cannot but take the longer way than AB

(1072.5 μm) for penetrating the PDMS from the ambient. In other words, the "equivalent" diffusion length of Fig. 8(a) should be larger than AB (1072.5 μm), even though the theoretical (computational) value still waits for verification.

Fig. 8(b) shows the extreme case of very thick PDMS, near to or even larger than 1072.5 μm. All the gas molecules take the shortest path AB for its smallest impeding resistance to gas diffusion.

Fig. 7 collects the time history of the reduced voltage ratio or reduced pressure ratio in the V-groove cavity. The authors take the half voltage point (V_{out}/V_{max})=0.5 to define the diffusion length and diffusion time in Eq. (4).

For the case of very thick PDMS, the diffusion length x is regarded as AB (1072.5 μm) in Fig. 8(b). The question is how thick PDMS should be? Without knowing the proper thickness of PDMS as the lower limit, the authors prepared another pressure sensor packaged with a PDMS of 2000 μm thick for additional leakage testing. Therefore, three reduced pressure history curves corresponding to different PDMS thickness were combined in Fig. 7. Fortunately the authors found the two curves of 500 μm and 2000 μm PDMS are very close to each other. The measured diffusion time *t* of 464 s corresponding to 500 μm thick PDMS or 455 s corresponding to 2000 μm thick PDMS via Eq. (4) verifies the fitted diffusion coefficient D as 2.2×10^{-9} m^2/s for CO_2 herein. This data matches with the diffusion coefficient of Merkel's work [11] very well.

TABLE I summarizes that the expected "equivalent" diffusion length of PDMS is much thinner than the extremely thick case predicted by Eq. (4) with the presumed diffusion coefficient *D* of 2.2×10^{-9} m^2/s. The equivalent diffusion lengths of 2000 μm and 500 μm are actually close to the ideal length AB of 1072.5 μm with the maximum deviation of 6.8 % only. On the contrary, the equivalent diffusion length of 45 μm PDMS is computed as 1838 μm and reasonably less than the half chip size of 2000 μm. All the equivalent diffusion lengths could be regarded as reference values for further theoretical investigation.

Restated, this work introduces an on-site integration way of bonding silicon pressure sensors on PDMS to detect CO_2 leakage. It could further interpret the "equivalent" diffusion length through PDMS novelly. For the current 4 mm × 4 mm chip size of silicon pressure sensors, the authors suggested that 500 μm thick PDMS is quite enough to match the designed diffusion length of 1072.5 μm ± 6.8 %. For the practical manner, a thicker PDMS than 500 μm has almost no matter with the diffusion length of gas permeation, but only causes difficulties in the sheet preparation beforehand and the wire bonding process afterwards.

III. Conclusion

This paper presents a novel method to investigate the gas-leakage and the corresponding diffusion coefficient of PDMS. The authors use silicon piezoresistive pressure sensors packaged with different thickness (45, 500 and 2000 μm) of PDMS, and put these sensors in the pressure testing machine with testing pressure up to 300 psi. By observing the output voltage, the half-pressure time of CO_2 permeated into cavity of the sensors are 1535, 464 and 455 sec subject to 45, 500 and 2000 μm of PDMS respectively (when the voltage drops half.) With the 1-D diffusion model for simulating the gas leakage process, the diffusion coefficients *D* can be reversely fitted and verified as 2.2×10^{-9} m^2/s for CO_2 through PDMS. The authors also addressed the thickness effect of PDMS and found that 500 μm is a proper value for the pressure sensor, with cavity edge width of 1072.5 μm as well as the effective diffusion length.

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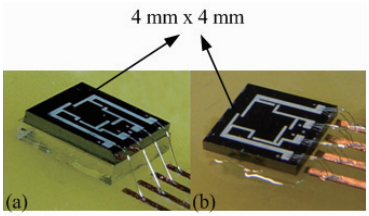

Fig. 1. Pressure sensors with PDMS bases of different thicknesses: (a) 500 μm thick; (b) 45 μm thick [9-10].

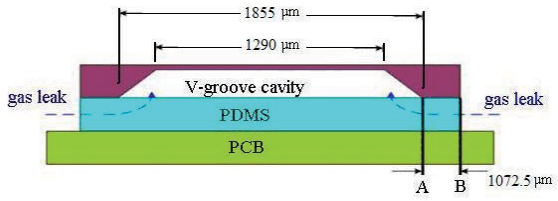

Fig. 2. Sensor configuration and gas leakage through PDMS.

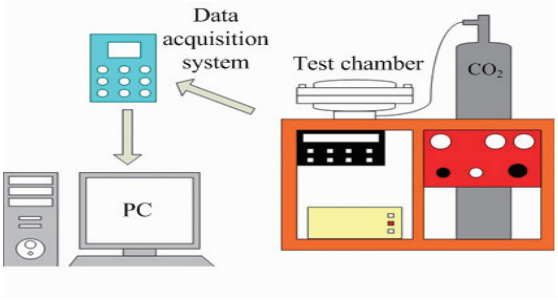

Fig. 3. The experimental setup of gas leakage testing.

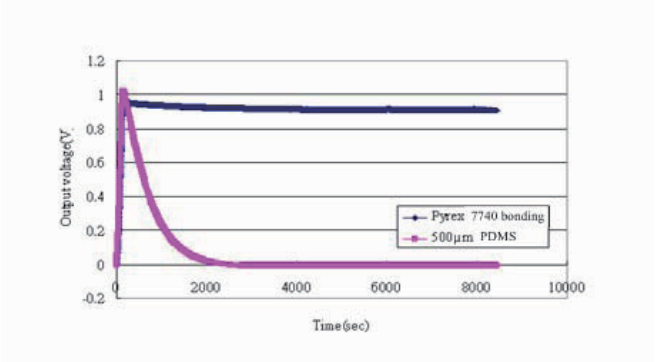

Fig. 4. Comparison of 500 μm thick PDMS-packaged and Pyrex #7740 glass-packaged pressure sensors. The small voltage drop of the Pyrex case denoting the background pressure leakage of the experimental result.

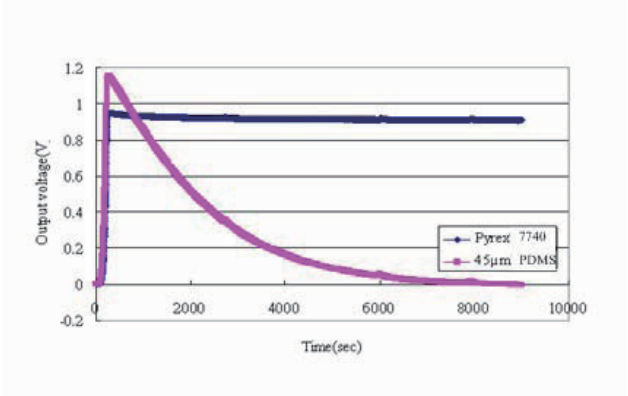

Fig. 5. Comparison of 45 μm thick PDMS-packaged and Pyrex #7740 glass-packaged pressure sensors.

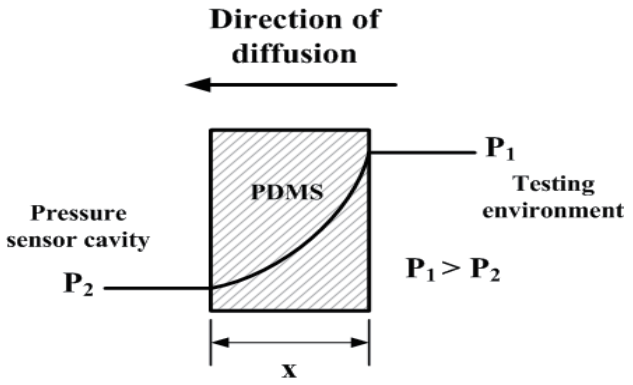

Fig. 6. Direction of gas diffusion in this work.

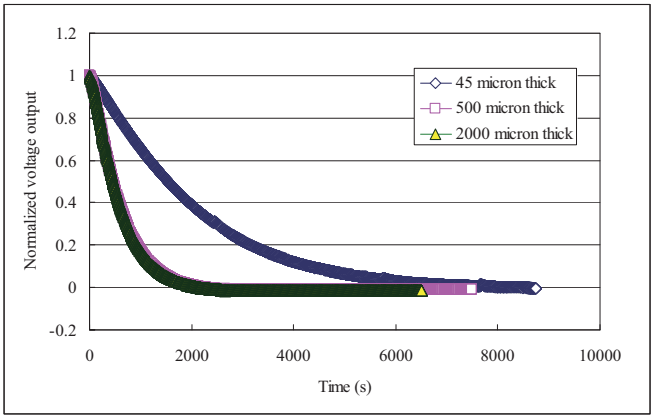

Fig. 7. The normalized voltage or reduced pressure ratio in the V-groove cavity vs. diffusion time.

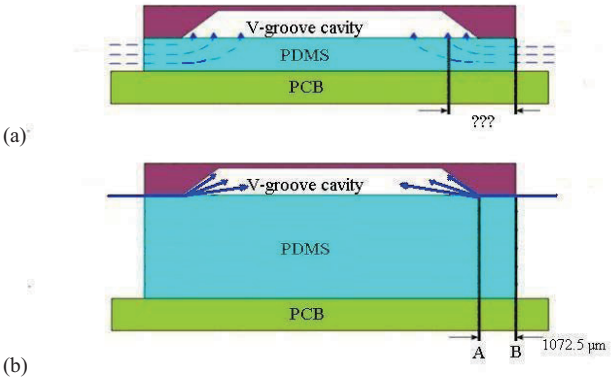

Fig. 8. The diffusion paths of PDMS with different thickness: (a) PDMS is very thin; (b) PDMS is very thick. The diffusion equation only proves effective for 1-D case here; 2-D diffusion equation is not the governing equation for the pressure field in this case and cannot predict the leakage trend depicted in TABLE I.

TABLE I.
DIFFUSION TIME OF VARIOUS PDMS IN DIFFERENT THICKNESS

Sensor No.	PDMS thickness (μm)	Measured diffusion time (s)	Equivalent diffusion length (μm)
1	2000	455	1000*
2	500	464	1010
3	45	1535	1838