Paper

Development of inkjet head for DNA chip

Member Dongyoun Sim(Tohoku University)
Member Takeshi Ono(Tohoku University)
Member Masayoshi Esashi(Tohoku University)

Summary

Inkjet head with four micropumps for DNA chip is described. The micropump injects each nucleotides alternately on the substrate. Electrostatic microactuator which consists of a thin Si diaphragm and Si electrode with a narrow gap is used for driving the pump. The inkjet head doesn't have an increasing temperature not to heating the nozzles directly and then the nucleotides don't have damages. The typical driving voltage is 150 V and the head achieved an uniform drop ejection at a driving frequency of 1 kHz.

Keywords: spotting head, DNA chip, electrostatic microactuator, micropump, electrical discharge

1. Introduction

Hybridization-based DNA sequence analysis is carried out by immobilizing probe molecules on a solid surface in a two-dimensional array in such a manner that the probe's sequence at each array site is known. Generally, there are two methods to fabricate DNA chip. One is an on-chip method which fixes four kinds of nucleotides directly on the substrate as light-directed synthesis[1-2]. The other is a method which spots the synthesized oligonucleotides on the substrate[3].

As the former method, Forder reported synthesis of two dimensional DNA probe arrays using photolithography in 1991. Many kinds of probes can be made on a substrate by using light directed synthesis chemistries to construct the desired oligonucleotides directly on the substrate. However this method requires many optical and chemical steps, for examples, probes consisting of 10 nucleotides require 40 steps for the synthesis, and is inefficient from the view points of fabrication cost[4].

In order to solve these problems, using the inkjet method to deliver small drop of reagent to hybridize the DNA probe[5], we developed a new inkjet head which can precisely control four kinds of nucleotide. This method is different to the general spotting method using the synthesized oligonucleotides,as mensioned above. The spotting head has four nozzles in order to alternately inject each nucleotides solution on the substrate and CCD camera is utilized for the alignment, as shown in Fig1.

Each head is composed of micro-nozzle, pump, orifice and nucleotide solution storage[6-8]. The nucleotides including organic solution are utilized, so that outer solution storage can not be connected using resin as epoxy because it is damaged by the organic solution, for examples, acetonitryl, dichloromethane, etc. Therefore this micropump has the solution storage in it which contains enough nucleotide solution for fabricating DNA chip. The micropumps consist of a Si diaphragm, a narrow gap under the diaphragm and a Si electrode, and are driven by an electrostatic force[9].

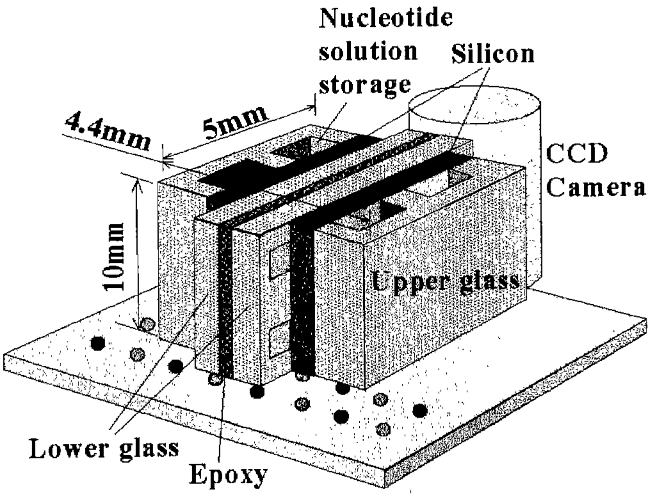


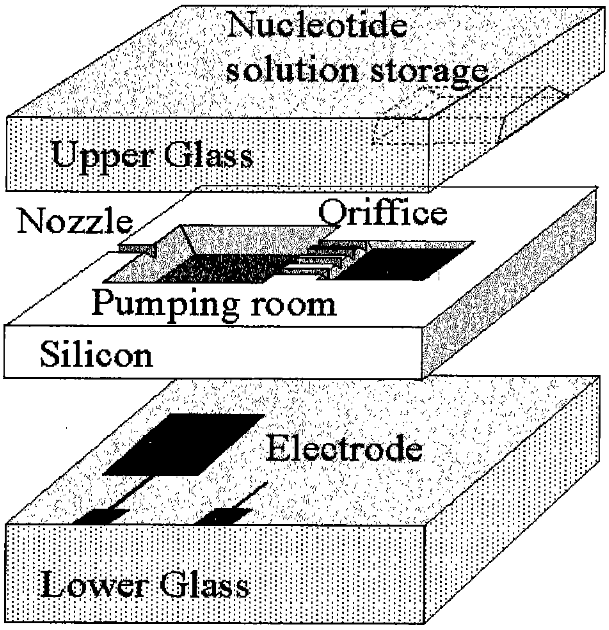
Fig.1 Spotting head for fabricating DNA chip

2. Structure of a spotting head

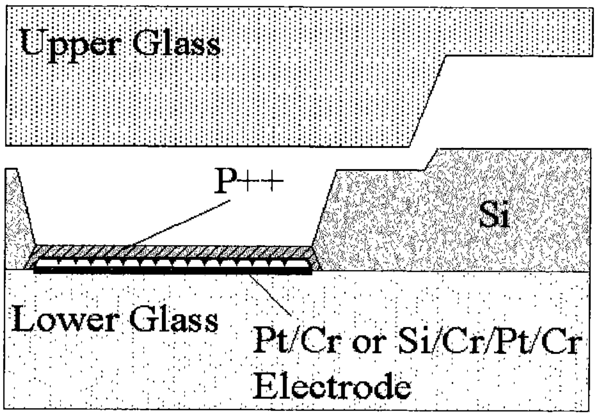
The inkjet head is composed of glass-silicon-glass structure as shown in Fig.2. Upper glass plate is a 1 mm thick Pyrex glass. Silicon plate is 200 μm thick and is etched anisotropically to construct the nozzle, diaphragm, narrow air gap and orifice. The nozzle has a V-groove shape, and the width and length are 60μm and 10μm respectively.

The diaphragm is 10μm thick and is made by P++ etch-stop. The orifice has four flow paths. Lower glass is 1 mm thick Pyrex glass which has electrodes to drive the diaphragm by the electrostatic force. Two kinds of the electrode's materials are tested to restrain micro-discharge in the narrow gap as explained later.

As shown in Fig.1, the inkjet head consists of two parts and each parts have two micropumps fabricated on the same substrate.



(a) Three dimensional structure



(b) Cross-sectional structure

Fig.2 Structure of the micropump

3. Design consideration of the flow paths

The main specifications of the micropump was decided as follows:

1) Drop resolution : 600[dpi]
2) Drop volume : 10[pl]
3) Drop velocity : 10[m/s]
4) Driving voltage : ≦ 100[V]

The flow paths of the nozzle and the orifice, and the actuator of the micropump are designed according to the flow and mechanical equations. The cross-sectional area of the nozzle A_n is determined by eq.(1) using the specified drop velocity V_n and the flow rate Q_n in the nozzle. The ejecting pressure ΔP_n is calculated by the drop velocity V_n, as shown in eq.(2)[10-11].

$$Q_n = A_n V_n = \frac{1}{C_R} \times \frac{A_n^{\,2}}{\mu\, l_n} \Delta P_n \qquad (1)$$

$$\Delta P_n = \frac{V_n C_R \mu\, l_n}{A_n} \qquad (2)$$

where C_R, μ, l_n are coefficient of the flow path, viscosity and nozzle length, respectively.

The electrostatic pressure ΔP_D must be larger than the restoring pressure ΔP_r of the diaphragm which is equal to the ejecting pressure ΔP_n, as shown in eq.(3). The electrostatic pressure ΔP_D is equal to the suction pressure of the nucleotide solution to the pumping room through the orifice.

$$\Delta P_D = \frac{1}{2}\varepsilon_0 \left(\frac{V_D}{d_D} \right)^2 > \Delta P_n \qquad (3)$$

where ε_0, V_D and d_D is the permittivity of vacuum, the applied voltage and the the narrow gap between the diaphragm and lower electrode, respectively.

The volume variation ΔW_D in the pumping room is identical to the supplied drop volume through the orifice, as shown in eq.(4)[12-13].

$$\Delta W_D = \frac{8}{15} ab\delta_D \qquad (4)$$

$$\delta_D = k\frac{12\left(1-\nu^2\right)}{Eh^3}\Delta P_D a^4 \qquad (5)$$

where a and b are the dimension of shorter and longer side of the diaphragm. δ_D shows the deflection by the electrostatic pressure ΔP_D. k, ν, E and h are the shape constant, poisson's ratio, Young's moduls and thickness of the diaphragm.

The flow rate Q_o in the orifice is calculated by the volume variation ΔW_D and the driving frequency f_D of the diaphragm as eq.(6). The shape of the orifices is designed in terms of eq.(6). A_o is the cross-sectional area and V_o is the velocity in the orifice. The area and length of orifices are decided by Eq.(6).

$$\begin{aligned} Q_o &= \Delta W_D \times f_D \\ &= A_o \times V_o \end{aligned} \tag{6}$$

At supplying the electric field in the micropump, the diaphragm is gone down by the electrostatic force and then the solution flows into the pump room from the orifices, and the nozzle doesn't have an inflow of air because of surface tension. On the other hand, at turning off, the diaphragm is restored to an original position and the solution is ejected from the nozzle because the flow pass of the nozzle is very shorter than them of orifices therefore the nozzle has a small flow resistance.

4. Fabrication process

The fabrication process is shown in Fig.3.

(a) A double-side polished silicon wafer is used. The wafer is oxidized in a wet atmosphere, and the front-side is patterned and etched using 25% TMAH to form the nozzle and the orifice. The oxide on the back-side is patterned for the recess to make the air gap under the diaphragm. TMAH etchant is utilized for minimizing the surface roughness of the diaphragm.

(b) The oxide is all removed. The wafer is oxidized again and patterned on the backside in order to dope high concentration boron for the P++ diaphragm.

(c) The oxide is all removed and the thick oxide is deposited using TEOS-CVD. The oxide is patterned for making stoppers to protect the sticking on the electrode of lower glass.

(d) The wafer is oxidized again and the oxide on the front side is patterned. The wafer is etched using the EPW etchant to form the diaphragm.

(e) The silicon dioxide is all removed. Upper glass is etched using 50% HF with Cr as a mask for making the nucleotide solution storage. The Cr mask is removed and then the upper glass is anodically bonded to the front-side of the silicon wafer.

(f) Lower glass is patterned. Pt/Cr is deposited and the electrode pattern is made by lift off. The Pt surface is covered by Cr and amorphous Si using sputter deposition. This is required to increase the minimun breakdown voltage as explained later. The lower glass is also anodically bonded to the backside of the silicon wafer.

The fabricated inkjet head is shown in Fig.4.

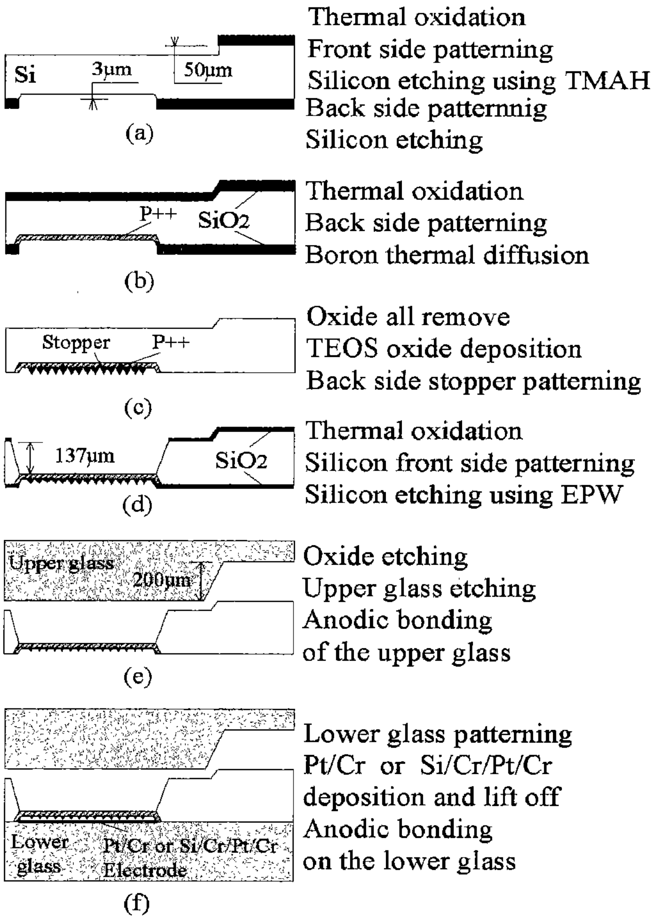


Fig.3 Fabrication process

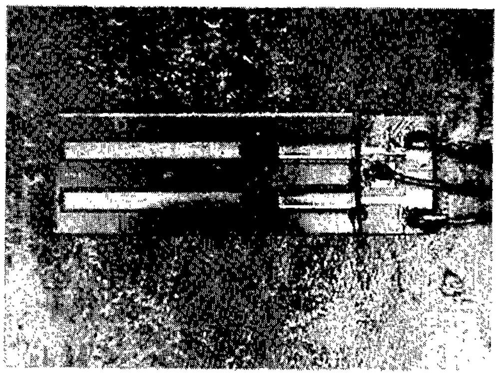


Fig.4 Spotting head having two micropump

5. Experimental results

To investigate the mechanical property of the diaphragm, the deflection of the diaphragm to the applied electrostatic pressure was measured using optical displacement system as shown in Fig.5. LT-810 of Keyence Co. and MLD-102 of NEO ARK Co. was used for the static measurement and dynamic measurement, separately. The static characteristics without water in the pumping room is shown in Fig.6. The air gap under the diaphragm is 3μm, but the maximum deflection is 1.5μm because the thickness of the stoppers is about 1.5μm. The dynamic characteristics in two cases of no water and water in the pumping room are shown in Fig.7.

The driving frequency is 0.0001~10 kHz and the applied voltage is 100 V. The deflection with the water in the pumping room is less than that of no-water. This is attributed to the viscosity and time constant of the fluidic part including the mechanical part.

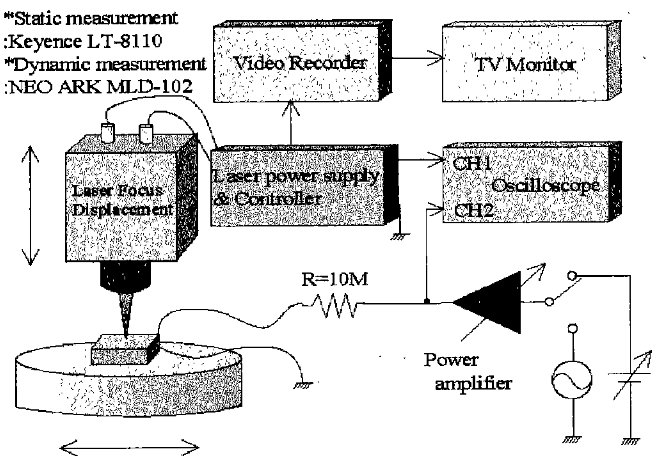


Fig.5 Optical measurement setup for the static and dynamic characterization

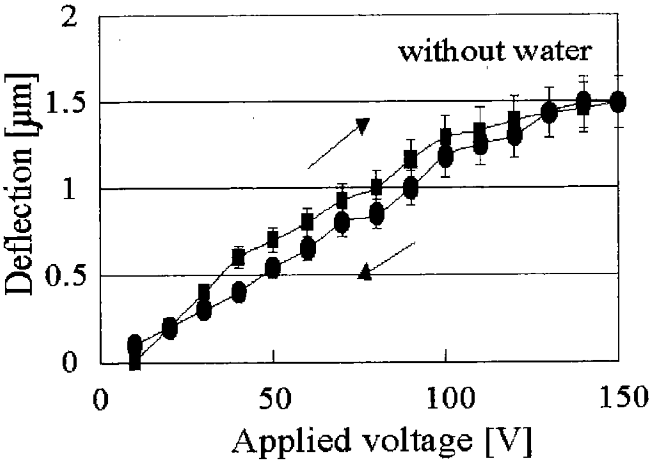


Fig.6 Deflection of the diaphragm versus the applied voltage

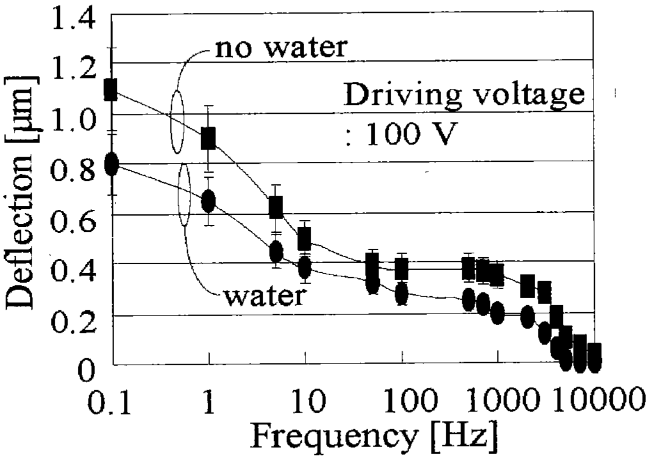


Fig.7 Deflection of the diaphragm versus driving frequency

The flow rate of the micropump was measured at different driving frequency, as shown in Fig.8. The drop ejection rates depended on the driving frequency, surface roughness and surface tension of the outlet in the nozzle. In order to decrease the surface tension, the outlet of the nozzle is coated with Au/Cr to build up the hydrophobic property. These results were measured by using the micropump which has Pt/Cr electrode.

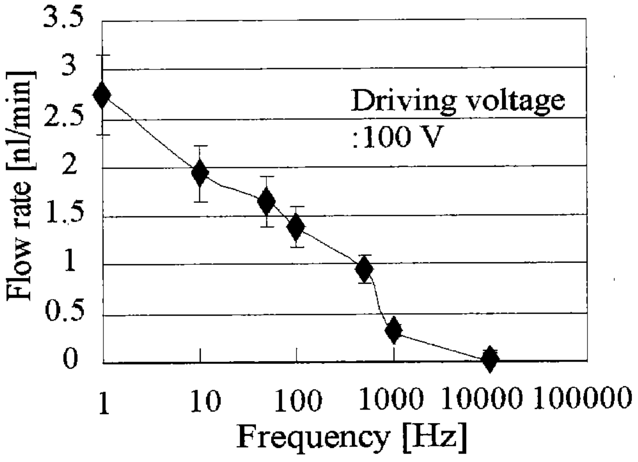


Fig.8 Flow rate versus driving frequency

However this micropump has one problem which gives rise to an electrical discharge between the diaphragm and electrode as shown in Fig.9. The electrical discharge is generally due to the cooperation of two processes. One is the ionization of gas molecules by electron collision in the narrow gap, and the other is the increment of secondary electron emission which attributes to the collision of ions on the surface of the electrode. Specially, metal electrode occurs the electrical discharge in the narrow gap because it is easy to emit the secondary electron by the collision of the ion in the low electric field[9].

To solve the problem, we investigated the electrode materials to suppress the discharge under the applied high voltage. The experimental results are shown in Fig.10. The electrical discharge occurs easily on the metal electrodes in the narrow air gap and the breakdown voltage is much smaller than that of the expected Paschen's curve.

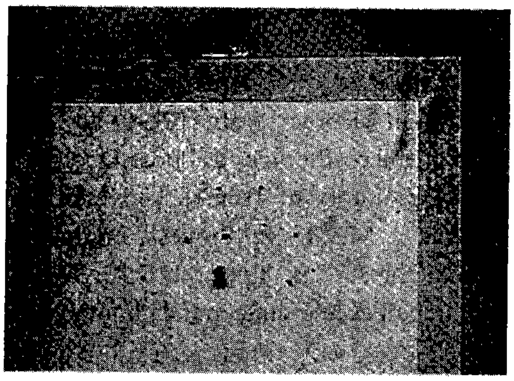


Fig.9 Electrode broken by the electrical discharge

On the other hand, the breakdown voltage of silicon to silicon electrode is similar to that of the Paschen's curve. This is considered that the sputtered amorphous silicon has less secondary emission than the metal to the collisions of the ions and then the electrical discharge in the narrow gap is difficult to occur.

Therefore we modified the fabrication process of the lower glass. The thin film silicon is deposited on the Pt/Cr electrode.

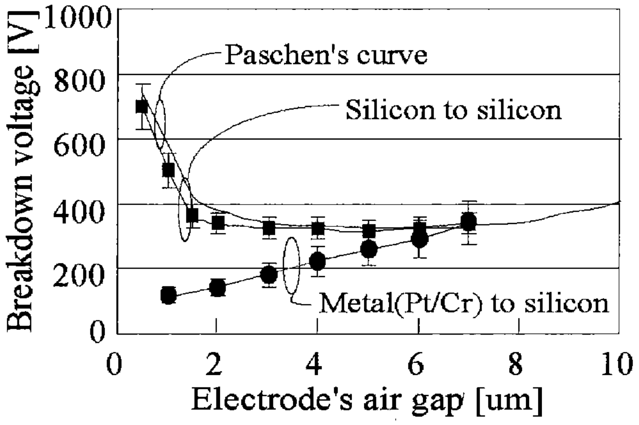
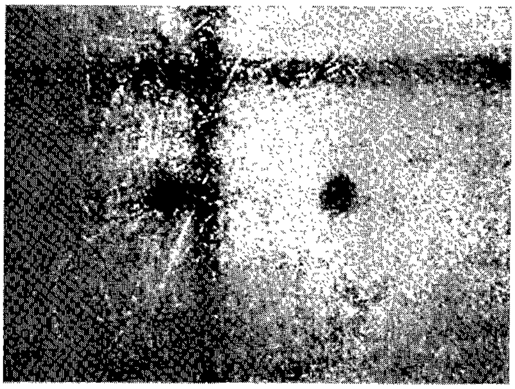


Fig.10 Breakdown voltage of the electrode materials to the air gap[14]

(a) Injecting motion



(b) Ejected drop mark

Fig.11 Testing of the drop ejection

Using the micropump with the silicon electrode the drop ejection is tested as shown in Fig.11. Fig.11(b) shows the drop mark ejected on the paper with impulse voltage of 150 V, 10% duty cycle of 0.1 Hz. The drop size to the one pulse driving shows the diameter of 80μm on the paper.

The continuous drop ejection is difficult in the wide frequency range because of the surface tension in the outlet of the nozzle. However the micropump can inject droplet continuously at the driving frequency, 1 kHz. It is supposed that the system of the micropump including mechanical and fluidic parts has a resonance in this driving frequency.

6. Conclusion

Inkjet head was developed in order to apply for the DNA chip. This paper describes the modeling of the micropump and fabrication process of the inkjet head. The flow rate of the micropump is about 0.3 nl/min at driving voltage 150 V, 1 kHz.

We will consider both mechanical and fluidic properties of the micropump and optimize the parameters in order to stabilize the drop ejection in the wide frequency range.

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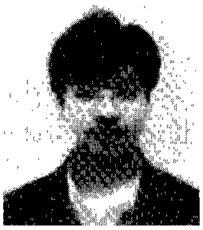
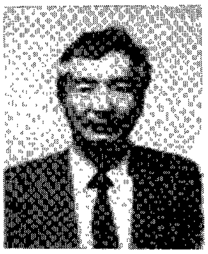
Dong Youn Sim [member] received the M.E. degree in electric and communication engineering from Pusan Fish University in 1991. He worked as a researcher at Eagle Co. of NOK group in Japan from 1991 to 1993 and received the Doctor of engineer degree in 1996 from Tohoku University. He was lecturer at Pukyong University in Korea from 1996 to 1997 and researched micro-fluidics as Post-Doc at venture Business Laboratory of Tohoku University in Japan. Now, he is a MEMS & Process Engineer at Ball Semiconductor Inc. in Texas, U.S.A.. He has been developing ball accelerometer and clinometer.



Takahito Ono [member] received D.E. degree in 1996 in Mechatronics and Precision Engineering, Tohoku University. During 1996-1999, he was a research associate in Department of Mechatronics and Precision Engineering, Tohoku University. From 2001, he has been an Associate Professor. He has been studying nano-fabrication and nano-mechatronics.



Masayoshi Esashi [member] received B.S. and Ph. D. degrees in electronic engineering from Tohoku University in 1971 and 1976, respectively. From 1976 to 1981, he served as a research associate at the Department of Electronic Engineering, Tohoku University and he served as an associate professor from 1981 to 1990. Since 1990, he has been a professor in the Department of Mechatronics and Precision Engineering, Tohoku University. He has been studying microsensors and microsysrems fabricated by macromachining.