A Piezoelectric Micro-Cantilever Bio-Sensor Using the Mass-Micro-Balancing Technique with Self-Excitation

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ABSTRACT

A micro sensor is developed to detect bio-molecular mass, like protein or DNA, in a Lab-on-a-chip. The micro sensor uses the mass micro balancing technique, which is the foundation of the widely-used mass-detection sensor, QCM. Instead of using circular quartz crystal plate, a micro cantilever with PZT thin film deposited is used in the developed micro sensor. The micro sensor is operated by an oscillating circuit to activate the micro cantilever with self-excitation at its fundamental vibrant mode and detects the variation in the vibration frequency. Shape and size of the cantilever and PZT thin film are determined so as to maximize the sensitivity of the sensor. The micro-cantilever with PZT thin film deposed is fabricated using micromachining technology. The length, thickness and width of the micro cantilever are 100μm, 5μm and 30μm respectively, and the length and thickness of the PZT are 50μm and 2.5μm. The 1st resonant frequency of the system is designed to be about 1.2 ~ 1.3MHz in order to have sufficiently high sensitivity for small mass. With the fabricated sensor, the mass of mussel gluing protein(mgfp) and insulin-anti insulin protein attached at the ending tip of the micro cantilever is successfully detected.

Keywords: PZT-micro cantilever, mass micro balancing, self-excitation

1 INTRODUCTION

Recently biological researches such as detecting biochemical material [1, 2] and measuring adhesion force between antibody and antigen [3, 4] have been studied vigorously. Most of the researches are focused on detecting existence of specific bio/biochemical material or detecting quantitative quantity of bio material if it exists. The results of these researches can be used such as prevention of hereditary diseases by detecting specific substance of DNA and pre-diagnosis of some diseases caused by protein. For these purposes the most important thing is detection of bio/biochemical material, and one of the most widely used detection equipment is QCM(Quartz Crystal Mass micro balancing).

QCM uses piezoelectricity of a vibrating quartz crystal disk and principle of QCM is the mass micro balancing which measures change of mechanical resonant frequency of the quartz crystal due to attached target material on it [5].

Various researches using a micro cantilever and the mass micro balancing technique have been studied in order to apply to various detective sensors such as biosensor, chemical sensor, mass sensor and biochemical sensor [1-2, 6-10]. In these studies, shift of resonant frequency is measured optically using laser and PSD(Position Sensitive Detector). Optical measurement is that laser is irradiated to a micro cantilever and the reflected laser from the micro cantilever is injected to a PSD. At this time the micro cantilever is bent because of attached target material on end of the micro cantilever, and the bending of the micro cantilever causes change of position of the reflected laser in the PSD. Resultantly shift of resonant frequency can be measured by interpreting output signal from the PSD. But in the optical detection, the reflected laser must be in-lined with the PSD and an interpretation equipment which interprets the output signal from the PSD is needed. So a hole detection system is complex and enlarged though a cantilever can be micro scaled by MEMS fabrication, consequently the optical detection method can not be applied to a Lab-On-a-Chip(LOC).

Therefore in this article using a self-oscillating micro cantilever with fundamental mode, a micro biosensor which can detect biomaterial like protein or DNA in a LOC is studied. The biosensor adopts the mass micro balancing technique and the micro cantilever is self-oscillated at its first resonant frequency by an oscillating circuit. In order to achieve the biosensor, the micro cantilever actuated by a PZT thin film is designed and made by MEMS fabrication. In the design procedure, it is focused that the PZT-micro cantilever has optimal shape and size so as to maximize the sensitivity. And lastly using the fabricated PZT-micro cantilever, the mass of mussel gluing protein and insulin-anti insulin binding protein is detected.

2 DESIGN OF PZT-MICRO CANTILEVER

To design the micro cantilever actuated by the PZT thin film, sensitivity is defined first. The sensitivity is defined as a ratio of the first resonant frequency shift to increased biomaterial mass on end of the micro cantilever.

$$\frac{\Delta f}{\Delta m} \tag{1}$$

The first resonant frequencies of the micro cantilever by the biomaterial mass attached before and after are [11]

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{0.07m}}\ ,\ f = \frac{1}{2\pi}\sqrt{\frac{k}{\Delta m + 0.07m}} \tag{2}$$

And the fundamental frequency shift $\Delta f (\Delta f = f_0 - f)$ due to the increased mass Δm is

$$\Delta f = \frac{1}{2\pi}\sqrt{\frac{k}{0.07m}}\left\{1 - \frac{1}{\sqrt{1+\Delta m/0.07m}}\right\} \tag{3}$$

In Eq. (3) it is assumed that $\Delta m/m \ll 1$ since the mass of the micro cantilever m is large enough compared to the Δm and taking the Taylor's expansion the Δf. And the stiffness k and the mass m expressed by length(L), width(b), thickness(t), elastic modulus(E) and density(ρ) of the micro cantilever are applied to the Taylor's expanded equation of Δf. Then the sensitivity is expressed as follow.

$$\frac{\Delta f}{\Delta m} \approx \frac{30}{2\pi}\left(\frac{E^{1/2}}{\rho^{3/2}}\right)\left(\frac{1}{bL^3}\right) \tag{4}$$

From Eq. (4) if the Δf is measured when biomaterial mass is attached then the increased mass Δm can be calculated.

Now the shape of the micro cantilever is determined, and the separation factor is defined first. The separation factor is defined as the value that difference of the first and the second resonant frequency is divides by the second resonant frequency. The separation factor represents how much the first and the second mode are close. The reason of defining the separation factor is to measure only the first resonant frequency exact by separating the first and the second resonant frequency enough, and it is expressed as Eq. (5).

$$\frac{f_2 - f_1}{f_1} \tag{5}$$

Fig. 1 is the separation factor against the normalized width by the length, b/L, in which t/L is calculated in the cases of 1/20, and it is known that the separation factor is constant with maximum value in some section. In Fig. 1 the normalized width is maximum value within the section of 3/10<b/L<9/10. And inserting the maximum section of b/L to Eq. (4), b/L is chosen as 3/10 which maximizes the sensitivity under the fixed length L. Therefore from the above results, t/L is 1/20 and b/L is 3/10, the geometric ratio of the triangular micro cantilever is determined as L : b : t = 20 : 6 : 1. From this geometric ratio the micro cantilever having 100μm of length, 30μm of width and 5μm of thickness will be made by MEMS fabrication. To detect infinitesimal mass such as protein/DNA using the mass micro balancing, it is necessary that a detector must have very high resonant frequency like QCM that uses a few mega hertz as detection vibrant frequency. Therefore the size of the micro cantilever is chosen as L×b×t = 100μm ×30μm×5μm and the first resonant frequency is about 1.2MHz calculated by ABAQUS.

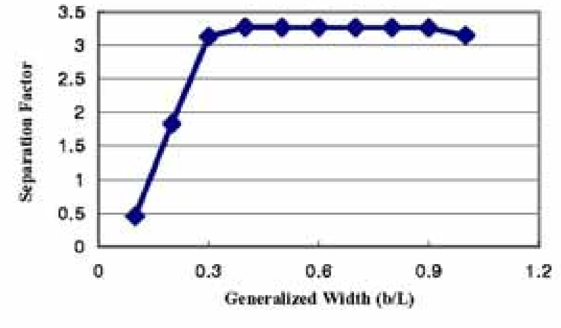


Figure 1: Separation factor

Since the micro cantilever is actuated by the PZT thin film deposited on it, the size of the PZT film must be determined so as to have maximum piezoelectricity. Piezoelectric thin film has the best piezoelectricity when the output current from the piezoelectric film has the largest value. And magnitude of the output current varies against size of the piezoelectric thin film. So the shape of the PZT film is decided as trapezoid so as to cover the topside of the micro cantilever because as the area of the PZT film becomes larger the output current increases. To determine the thickness of the PZT film, the output current with respect to its thickness variation is investigated under the fixed length, 30μm, along the longitudinal direction of the micro cantilever. The output current is maximal when the thickness is 2.5μm. Lastly, the length of the PZT film is decided by calculating the output current with length variation under the fixed thickness, 2.5μm, and the output current has maximum value when the length is 50μm. From the above results the thickness and length of the PZT thin film are 2.5μm and 50μm.

3 FABRICATION OF PZT-MICRO CANTILEVER

The PZT-micro cantilever was made by MEMS fabrication depicted in Fig. 2. (1) SiO_2 was deposited by thermal oxidation on upside of a SOI wafer to insulate electrically between bottom electrode and silicon of the wafer. A bottom electrode layer, Pt/Ti, was deposited by sputtering and a 2.5μm thick PZT film was constructed by sol-gel method. And a top electrode layer, Pt, was piled up by sputtering on the PZT layer. (2) The top electrode was etched by plasma of reaction gas of Ar and Cl_2, (3) by plasma of reaction gas of Ar, Cl_2 and CF_4 the PZT was dry-etched. (4) The bottom electrode was made the same way to the top electrode. (5) To insulate the top and bottom electrode, an isolating layer, SiO_2, was constructed by IAD(Ion Assisted Deposition) and its thickness was 5000Å considered step-coverage. And the isolator was dry-etched using reaction gas of Ar and CHF_3. (6) A contact pad, Au/Cr, was constructed by lift-off. (7) The SiO_2 which was deposited by thermally to insulate the bottom electrode and silicon of the wafer was removed the same method to etching of isolator, and shape of the cantilever was formed by ICP-RIE(Inductively Coupled Plasma-Reaction Ion Etching). (8) In order to release the micro cantilever, backside of the wafer was etched by ICP-RIE, (9) the SiO_2 between the upper and lower silicon of the SOI wafer was removed by BOE(Buffered Oxide Etchant). In Fig. 3 the fabricated PZT-micro cantilever is represented by SEM.

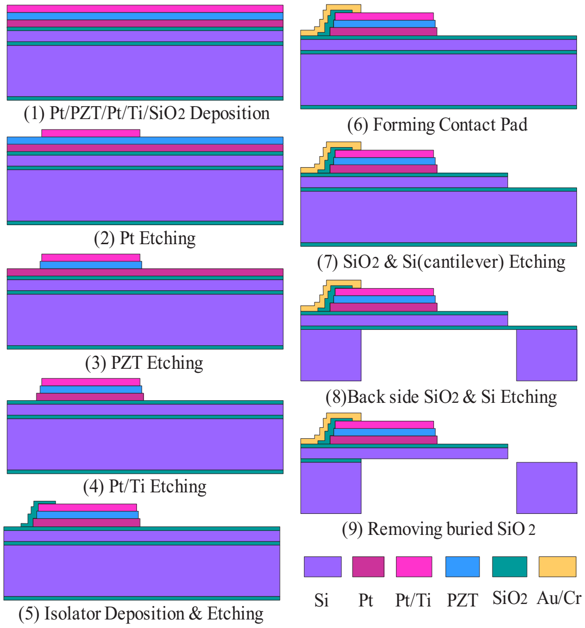


Figure 2: Fabrication process of the PZT-micro cantilever

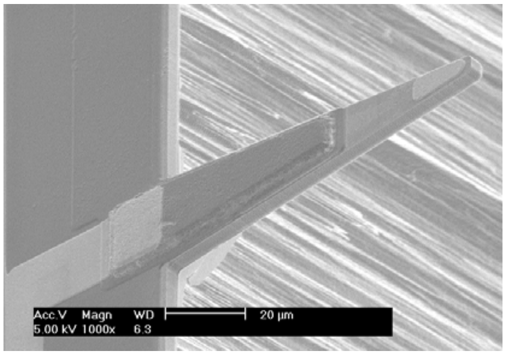


Figure 3: Fabricated PZT-micro cantilever

4 ELECTRO-MECHANICAL CHARACTERISTIS OF PZT-MICRO CANTILEVER

The first resonant frequency of the fabricated PZT-micro cantilever is about 1.2~1.3MHz and it is similar to the value of FEM analysis, about 1.2MHz, by ABAQUS. In Fig. 4, the micro cantilever is vibrated with its fundamental mode by rectangular impulse input, and Fig. 5 represents the impedance characteristic of the PZT-micro cantilever.

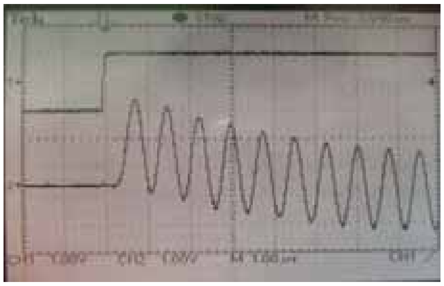


Figure 4: Rectangular impulse test

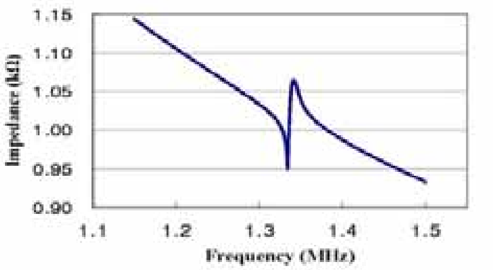
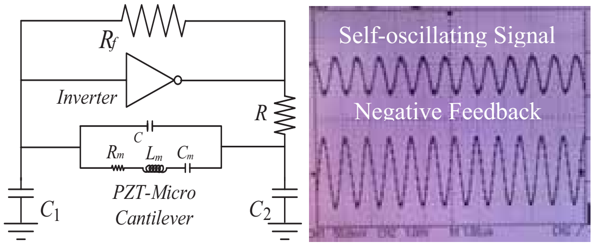


Figure 5: Impedance curve of the PZT-micro cantilever

Since the micro biosensor uses the self-excited micro cantilever by an oscillating circuit, the oscillating circuit was made and it is depicted in Fig. 6(a). The oscillating circuit, composed with a part of amplifying and feedback, can generate an AC signal using feedback loop without an external input signal just if a minimum electric energy to operate the electric components like an O.P.-Amp is supplied to the circuit [12]. The upper signal in Fig. 6(b) is the self-excitation signal of the PZT-micro cantilever and the lower one is feedback signal from the oscillating circuit.



(a) (b)

Figure 6: (a) Oscillating circuit (b) Self-excitation of the PZT-micro cantilever

5 DETECTION OF BIOMATERIALS

Using the self-oscillated micro cantilever mussel gluing protein(mgfp) and insulin-anti insulin binding protein were detected by measuring the first resonant frequency change, which was measured by a frequency counter. And the mass of the biomaterials is calculated by substituting the changed resonant frequency to the sensitivity equation(Eq.(4)). The changed resonant frequencies of the mgfp and insulin-anti insulin were 85Hz and 217Hz respectively. And their calculated masses were 0.1798×10^{-15}g and 0.4581×10^{-15}g respectively. Fig. 8 represents attaching the biomaterials to end of the micro cantilever, and Fig. 9 is measurement of the first resonant frequency change of the micro cantilever using the frequency counter.

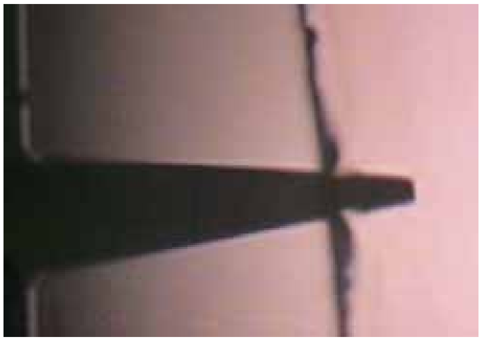


Figure 8: Attaching biomaterial to the micro cantilever

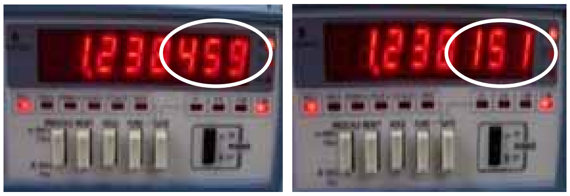


Figure 9: Measuring the resonant frequency change

6 CONCLUSION

We designed the PZT-micro cantilever and made it using MEMS fabrication. The fabricated PZT-micro cantilever performed very similar to the analytical results, and it was self-excited by the oscillating circuit. Using the self-oscillated micro cantilever, the mussel gluing protein and insulin-anti insulin binding protein were detected and their masses were calculated. Therefore, if the PZT-micro cantilever and the oscillating circuit are integrated then it can be a biosensor to be applied to a LOC.

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