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Fabrication of Hole-type Microcantilevers using FIB and its Evaluations

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Abstract. A high sensitive mass biosensor using a piezoresistive hole-type microcantilever has been developed. To optimize the cantilever shape for high sensitivity easily, we demonstrate frame-type cantilevers with holes on the surface fabricated by Focused Ion Beam (FIB) method and evaluate their sensitivity in both air and liquid. Resonance frequency of hole-type cantilevers increased about 20% and 40% in air and in water, respectively. On the other hand, while Q value decreased from 10% to 30% in air, it increased sharply from 0% to 50% in water with increasing of hole-size. Especially, when we increased the frame size from 0.5 μm to 2.5 μm, resonance frequency changing and Q value changing in water were 60% and 125%, respectively. However, the Q value of large hole (36x60μm^2) in water inversely decreased due to the decline in rigidity. The sensitivity of hole-type cantilever of $\Delta m/\Delta f$ (hole-size = 12x12μm^2; frame-size = 2.5μm) was 23.1 fg/Hz, 10 times smaller than the conventional type cantilever.

Introduction

In biomedical engineering or environmental analysis, biosensors are required to detect physical and chemical quantities such as weight, temperature, pressure, and electricity with high sensitivity and low cost. Cantilever-based biosensor technology, proposed by Berger, et al [1, 2] has played an important role in various applications, including mass sensor [1], force sensor [3], temperature sensor [4], calorimetric sensor [5], and thermogravimetric sensor [6]. For instance, biomolecular detection of bolvine serum albumin and deoxyribonucleic acid hybridization using nanomechanical responses of a cantilever bending has been used [7]. Cantilever harmonic vibration-type mass sensors with picogram mass sensitivity using a laser beam deflection detection system have been reported in experiments of water molecular adsorption on the cantilevers [8]. In addition, to create a self-sensing system, piezoresistive strain sensor embedded in cantilever has been used [9, 10]. In recent year, our research group have reported a self-sensing mass sensor using the harmonic vibration of a piezoresistive cantilever with mass sensitivity of 190 fg/Hz in air and water [11, 12].

In medical and biochemical applications, cantilever-based biosensors are being used in liquid. The characteristic of resonant cantilevers in viscous liquid were investigated by Vancura, et al [13]. However, when a harmonic vibration-type frequency detection system is used, the sensitivity in liquid could be reduced sharply or unstable due to the viscous resistance of liquid. Viscosity and density of liquids from excitation level response of piezoelectric-excited cantilever sensor were studied by Wilson, et al [14]. To reduce viscous resistance in liquid, our research group proposed several kinds of frame-type cantilever with mesh and hole on surface using semiconductor process [15]. In this study, we have used Focused Ion Beam (FIB) processing to fabricate hole-type cantilevers with frames and their frequency properties in both of air and water, mass sensitivity were discussed. The novelty of this method is the fabrication of various structures of hole-type cantilevers efficiently with low cost.

Mass detection theory

When a microcantilever is vibrated by exciting piezoactuator which implanted in cantilever's base, the cantilever's motion could be written as:

$$m\frac{d^2z}{dt^2}+a\frac{dz}{dt}+kz=F_0 sin\omega_0 t \tag{1}$$

Where m is the effective mass of the cantilever, given by $m=nm_0$, where n is a proportional constant and m_0 is the mass of the cantilever [16], z is the deflection of the cantilever top, a is the viscous resistance modulus, k is the spring constant, and $F_0\sin\omega_0 t$ is the external vibration force from the exciting piezoactuator. The frequency at which cantilever resonances depends on the effect of mass change and the resonance frequency can be expressed as:

$$f=\frac{1}{2\pi}\sqrt{\frac{k}{m}-\frac{a^2}{2m^2}} \tag{2}$$

The above equation suggests that resonance frequency change depends on the spring constant and the viscous resistance. Here, we could divide into two cases. First is in the air environment (or very low viscosity environment) where the viscous constant a could be negligible, and second is in the liquid (in this paper we used water) where viscous constant a must be discussed. In both of two cases, the spring constant will not be changed. Therefore, the resonance frequency change, Δf in air and in water could be expressed by:

$$\Delta f=\frac{1}{8\pi^2 mf}\left\{\frac{a^2}{m^2}-\frac{k}{m}\right\}\Delta m \tag{3}$$

When the material is attached on cantilever, m changes to $m+\Delta m$, and the resonance frequency, f, changes to $f+\Delta f$. From Eq. (2) and Eq. (3), the mass change in water and air, Δm, is written as:

In water $$\Delta m=-2\frac{m\Delta f}{f}\Big/\left\{1-\frac{a^2}{8\pi^2m^2f^2}\right\} \tag{4}$$

In air $$\Delta m=-2\frac{m\Delta f}{f} \tag{5}$$

As the microcantilever satisfies the requirement included small mass and high resonance frequency, we could detect small amount of Δm value.

Finite element method simulation

The resonance frequency and strain distributions of cantilevers with several kinds of hole-types were studied by a computer simulation using the commercially available software COMSOL Multiphysics (COMSOL, Inc). Finite element modelling (FEM) method used in this software assumes piecewise continuous function, and obtains physical parameters of the function in the manner that reduce the errors in solution.

In this report, cantilever's 3D structure was designed by computer aided design (CAD) system which we could observe the cantilever in any angle. After the properties of cantilever including material, boundary were specified, the mesh of segment region were set in 3D structure. Accordingly, finite element analysis calculations were be executed. As a result of error control using a variety of numerical solvers, the original frequency analysis, strain energy and vibrational amplitude were obtained.

Holes processing were executed on the conventional piezoresistive cantilever (NPX1CT004, SII nanotechnology) so that the properties including resonance frequency change in both of simulations and experiments after fabrication would be compared with it. Figure 1 shows top-view structure of prototyped cantilevers in simulation processing of hole-fabrication. Figure 1(a) was the conventional cantilever before fabrication and Fig. 1(b) was an example of the cantilever after we designed several kinds of structure for hole-size (hole-height x hole-width) and frame-size. Figure 2 shows the comparison of resonance frequency changing between simulation and experiment. Changing amount is calculated as a proportional percentage of changing value after fabrication and the conventional cantilever. Firstly, to verify how cantilever's properties change according to the hole size, we fixed the hole areas (about 2160 μm^2), and increased hole size from 6x6 μm^2 to 36x60 μm^2. Secondly, to verify how frame size affects to properties, we stabilized hole-size (12 x12μm^2) and increased frame size from 0.5 µm to 3 µm. As shown in Fig. 2(a) and Fig. 2(b), the difference between simulation and experiment is less than 4 % due to processing accuracy. Therefore, we could conclude that simulation provided valuable data to estimate cantilever's properties before FIB processing.

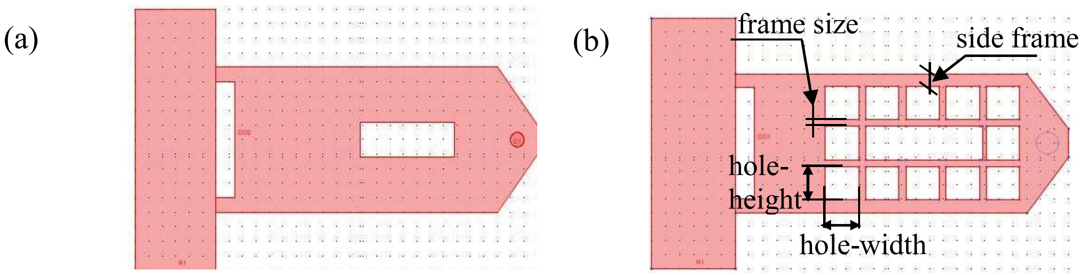

Fig.1. Top view structure of prototyped cantilevers in simulation step:
(a) before fabrication (conventional cantilever); (b) after fabrication

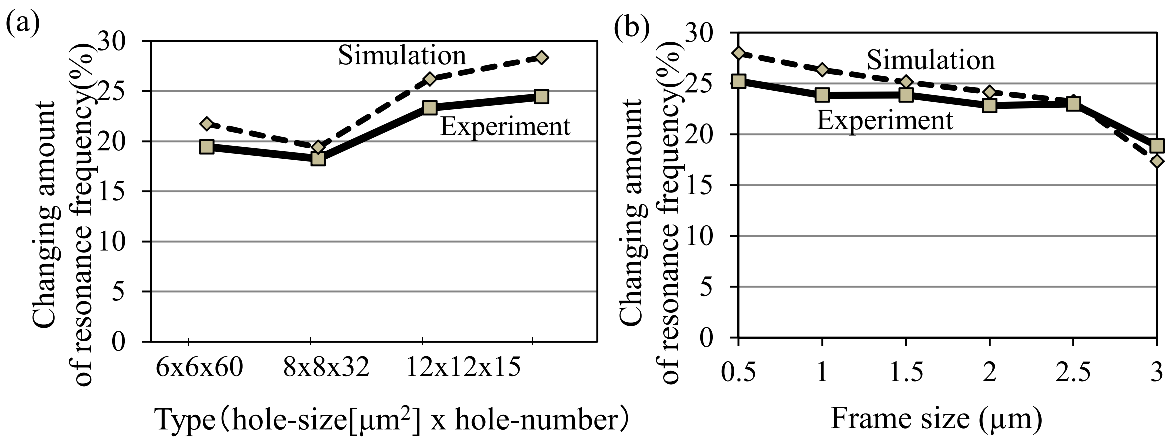

Fig. 2. Changing amount of resonance frequency in simulation and experiment. (a) according to hole- size and hole-number. (b) according to frame-size.

Experimental procedure

We fabricated hole-type cantilevers based on SII cantilevers which were initial models by using Focused Ion Beam (FIB; SMI 500, SII nanotechnology) instrument. To fabricate several kinds of hole-structure, bitmap files of hole-designs were prepared and inputted into computer before the fabrication. The beam current played an important role in controlling beam-size, and could affect directly to hole-size. As the beam current would control beam-diameter which made hole-size larger than design, we reduced the designed hole-size by beam-diameter.

The frequency properties of the prototyped cantilevers were measured by the frequency sweep method using piezoelectric oscillator and Weatstone bridge circuits. The resonance frequency and quality (Q) factor were calculated from these data. At last, in order to evaluate the mass sensitivity of prototyped cantilevers, we deposited thin gold films with different thickness on the surface of cantilevers by RF sputtering (JFC-2300HR, JEOL) and measured frequency changing after each deposition.

Results and discussion

We fabricated hole-type microcantilevers using FIB instrument in the conditions including acceleration voltage = 30 kV; beam current = 10 nA (beam diameter = 1 µm). Figure 3(a) shows hole-design for the hole-type microcantilever which designed hole-size was 11x11 µm^2, frame-size was 3.5 µm. Figure 3(b) shows scanning ion microscopy (SIM) images of hole-type cantilever (hole numbers = 15; hole-size = 12x12 µm^2; frame-size = 2.5 µm) after fabrication. As this figure shown, hole-size was 11x11 µm^2 in design and has become 12x12 µm^2 after fabrication. It indicated that hole-width and hole-height increased 1µm which was equal to beam diameter after fabrication. We also show the SIM images of prototyped cantilevers when hole-size was changed from small (6x6 µm^2) to large (36x60 µm^2) in Fig. 4 below. These figures indicated that we succeeded in fabricating hole-type microcantilevers using FIB.

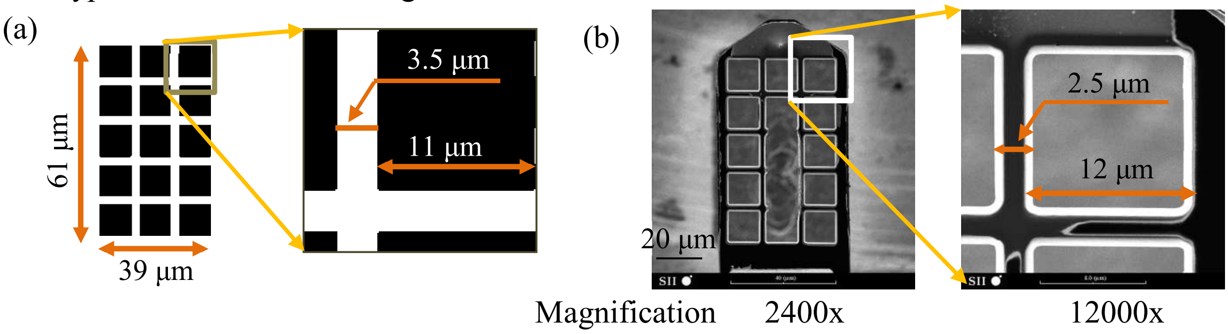

Fig. 3. (a) Bitmap images of hole-design; (b) SIM images of cantilever after hole processing using FIB instrument (hole number = 15, hole size =12x12 µm^2, frame size = 2.5µm)

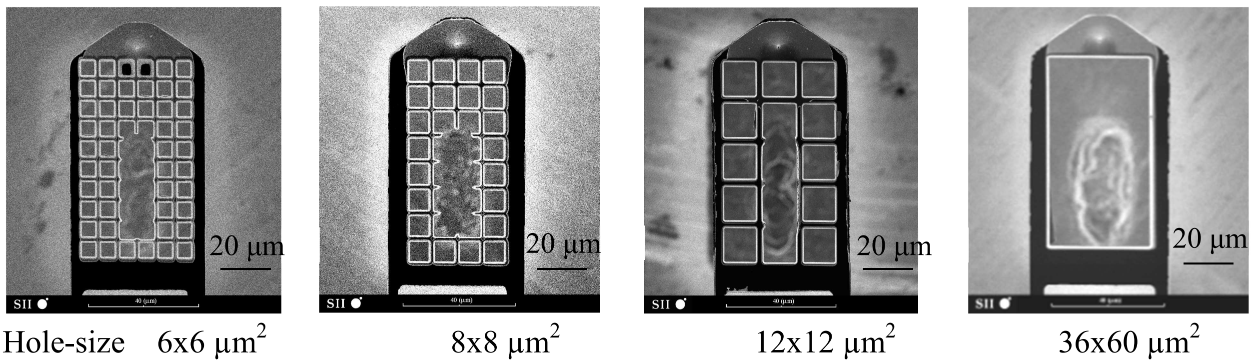

Fig. 4. SIM images of prototyped cantilevers according to hole-size (magnification = 2400 times)

Moreover, experimental results for the changing amount of frequency properties including resonance frequency, Q factor in air and in water according to the change of hole-size and frame-size were obtained, as shown in Fig. 5(a) and Fig. 5(b), respectively. The changing amount is calculated as the percentage proportion of the change after fabrication and the conventional type.
As shown in Fig. 5(a), the hole-sizes were changed from 6x6 µm^2 to 12x12 µm^2 and 36x60 µm^2, which the total hole-areas were about 2160 µm^2 for each cantilever. Resonance frequency of hole-type cantilevers increased about 20 % and 40 % in air and in water, respectively. It indicates that changing amount of resonance frequency caused by a same mass reduction due to hole-fabrication. On the other hand, while Q value decreased from -10% to -30% in air, it increased sharply from 0% to 50% in water with increasing of hole-size from 6x6 µm^2 to 12x12 µm^2. These results indicate that hole-type had an advantage of reducing viscosity of water, assisted microcantilever in moving easily in water. However, the Q value of large hole-size type (36x60 µm^2) in water inversely decreased due to the decrease in torsional rigidity. This decrease can be explained by the fact that the inner frames of cantilever were removed.
Figure 5(b) shows the changing amount of frequency properties when the hole-size and hole-number were fixed at 12x12 µm^2 and 15, respectively, and the frame size were increased from 0.5 µm to 3µm. Resonance frequency changing increases about 25% in air and from 40% to 60% in

water. Q value changing increases according to frame-size from 0.5 µm to 2.5 µm. Resonance frequency changing of 60%, Q value changing of 125% were investigated when frame-size becomes 2.5 µm. However, when the frame size was 3µm, Q values have decreased both in air and water. It could be explained that the width of side frame which became thinner after hole-fabrication has caused torsional rigidity decreased. This result indicate that hole-type cantilever with frames has an advantage in reducing viscosity resistance and optimised structure is the-size of 12x12µm^2, the hole-number of 15, the frame-size of 2.5 µm.

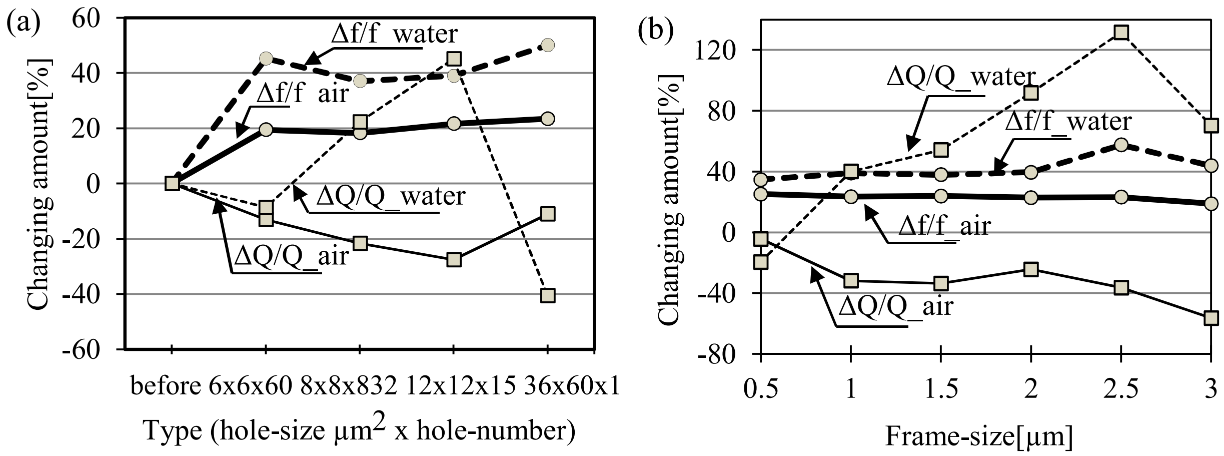

Fig. 5. The changing amount of resonance frequency and Q value in air and water (a) according to hole-size; (b) according to frame-size

Finally, we have investigated the mass sensitivity of prototyped cantilever. The deposited mass was estimated by the thickness of the deposited gold film, the surface area of the cantilever and the gold density. We measured the resonance frequency after each gold deposition and calculated the changing of resonance frequency -Δ*f*. Figure 6(a) shows sensitivity characteristic of hole-type which hole-size was 12x12 µm^2, hole-number was 15 and frame-size was 2.5 µm. The changing of resonance frequencies -Δ*f* was proportionally increased with increasing the changing of deposited gold masses Δ*m*. Mass sensitivity Δ*m*/Δ*f* was calculated as the gradient of linear graph. Based on these experimental results, the sensitivities of prototyped hole-type microcantilevers were obtained. Figure 6(b) also shows sensitivity results of prototyped cantilevers. The sensitivity of hole-type cantilever of Δ*m* /Δ*f* (hole-size = 12x12 µm^2; frame-size = 2.5 µm) was 23.1 fg/Hz. This value is 10 times smaller than the conventional type cantilever (231.9 fg/Hz) and about 1000 times smaller than that for the conventional quartz crystal microbalance (QCM) sensor. These results indicated that hole-type microcantilevers with frames have an advantage in improving mass sensitivity of conventional type microcantilever.

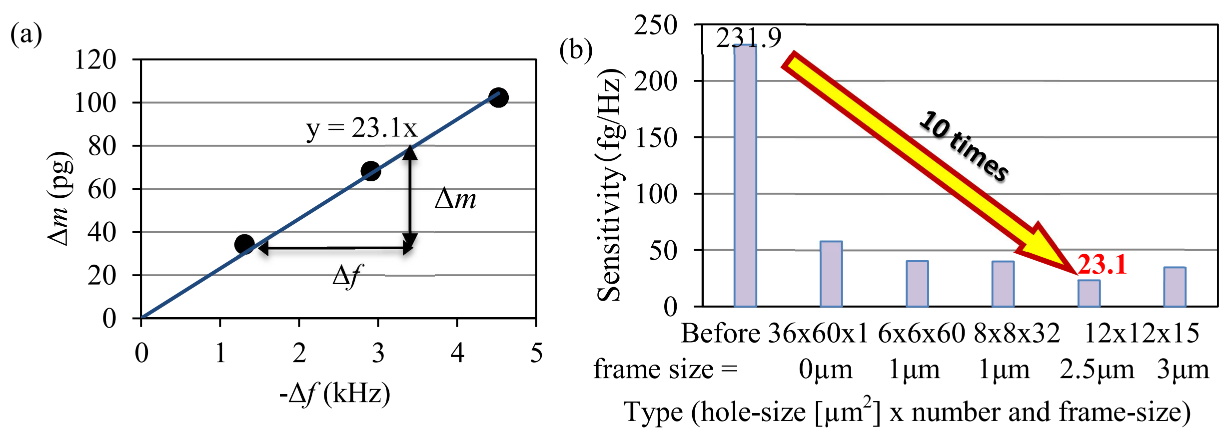

Fig. 6. (a) Sensitivity characteristic of prototyped cantilever by gold film deposition (hole-size= 12x12 µm^2; hole number = 15; frame-size = 2.5 µm). (b) Mass sensitivities of prototyped cantilevers

Conclusions

We fabricated the hole-type microcantilevers using FIB processing for high sensitivity biosensor. These following results were obtained

(1) The resonance frequency in the air and in water increased more than the conventional type.

(2) Q value of hole type cantilever (hole-size =12x12 μm^2; frame-size = 2.5 μm) in water increased 200% than the conventional cantilever. Q value of large hole-size (36x60 μm^2) cantilever in water inversely decreased due to the decline in rigidity.

(3) The sensitivity of hole-type cantilever (hole-size = 12x12 μm^2; frame-size = 2.5μm) is 23.1 fg/Hz. This value is 10 times smaller than the conventional type cantilever and 1000 times smaller than QCM cantilever.

In summary, we could conclude that hole-type microcantilever with frames has an advantage in reducing viscosity resistance as a result of the increase of resonance frequency and Q factor in water, and produce high mass sensitivity.

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