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# YOUNG'S MODULUS MEASUREMENT METHOD FOR NANO-SCALE FILM MATERIALS BY USING MEMS RESONATOR ARRAY

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## ABSTRACT

This paper describes a quantitative measurement method for Young's modulus of nanometer-thick film materials by using a MEMS resonator array. The MEMS resonators fabricated by silicon micromachining techniques were designed to be driven at their resonant frequencies from 6.0 kHz to 35.5 kHz. From the difference in resonant frequencies between before and after deposition of these films onto the resonators, we successfully measured the Young's moduli of Al and plasma-polymerization films made from $CH_4$ and $CHF_3$ gases. The obtained Young's moduli of Al, $CH_4$ and $CHF_3$-derived polymer films were found to be 60.4, 35.7, and 30.0 GPa on average, respectively, and showed no film thickness dependency in the range from 50 to 150 nm. The obtained values for all the films by the resonance tests were comparable to those by tensile and nanoindentation tests. The proposed technique is effective for directly measuring Young's modulus of nanometer-thick film materials.

## KEYWORDS

Young's modulus, Material property, Thin film, Resonator, Resonant frequency, Structural design, NEMS

## INTRODUCTION

Micro- and nano-electromechanical systems (MEMS, NEMS) include nanometer-thick film materials having a variety of functions. For the reliable design and the improvement of performance and lifetime of those devices, understanding of their mechanical properties is significant. Especially Young's modulus is one of the most important material characteristics in order to carry out the structural design of devices. The basic material properties of materials are usually measured by tensile, bending, and nanoindentation tests. In the case of film specimens with a micrometer thickness, the tensile test is typical. To date lots of tensile test techniques have been developed for accurate material characteristics. In the case that the target material is a film having a nanometer thickness, those testing techniques are hard to be employed because there are lots of technical difficulties, such as specimen preparation, handling, and force-displacement relation measurement. Therefore, a new material test technique for simply characterizing nanoscale films should be developed.

The objective of this study is to propose a quantitative measurement method for Young's modulus of nanometer-thick films. This paper focuses on developing the test technique using a MEMS resonator array and measuring Young's moduli of Al and plasma-polymerization films made from $CH_4$ and $CHF_3$ gases.

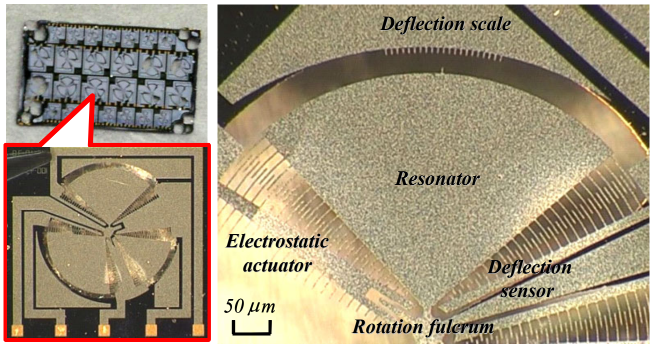

*Figure 1: Photograph of MEMS resonator array. 78 resonators were fabricated on one silicon chip of 13.0 x 22.5 mm*

## EXPERIMENTAL PROCEDURE

### MEMS Resonator Array

Figure 1 shows the photograph of the produced MEMS resonator array that was made from a SOI wafer. The resonator consists of a fan-shape resonator with rotation angle measurement gauge, a rotation fulcrum, an electrostatic comb-drive actuator, and a capacitive sensor. The resonant frequency, *f*, of the device is expressed as:

$$f = \frac{1}{2\pi}\sqrt{\frac{Ew^3}{6\ell\rho\theta R^4}} \tag{1}$$

where $E$, $w$, $l$, $\rho$, $\theta$, and $R$ are Young's modulus, width and length of fulcrum, density, rotation angle, and radius of resonator, respectively. The resonator was designed by finite element analysis to achieve proper vibration amplitude by resonance. The designed resonant frequency ranges from 6.0 kHz to 35.5 kHz.

We fabricated 78 resonators with different resonant frequencies on one Si chip so as to investigate the influence of resonant frequency on the measured values of Young's modulus of films. In this paper, we prepared sputtered Al and $CH_4$- and $CHF_3$-derived plasma-polymerized films with the nominal thicknesses of 50, 100, and 150 nm.

### Derivation of Young's Modulus

Firstly, the resonant frequency of the device was

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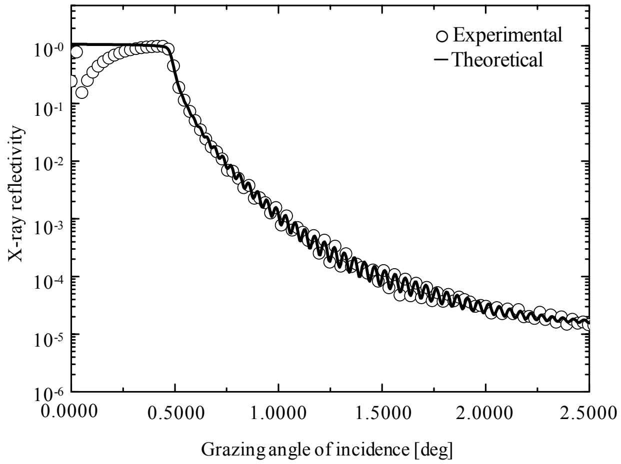


*Figure 2: X-ray reflectivity curve for Al film deposited on Si substrate. The solid line indicates the fitted curve for analysis.*

*Table 1: Measurement results of film density and thickness by XRR.*

| Film | Gas | Density [kg/m$^3$] | Thickness [nm] |
|---|---|---|---|
| Al | - | 2841 | 58.3, 99.5, 146.4 |
| Polymer | CH$_4$ | 1490 | 56.4, 85.1, 182.8 |
| | CHF$_3$ | 2155 | 45.7, 103.4, 159.4 |

measured before film deposition. Then the Young's modulus, $E_1$, of a Si substrate was calculated using the following equation:

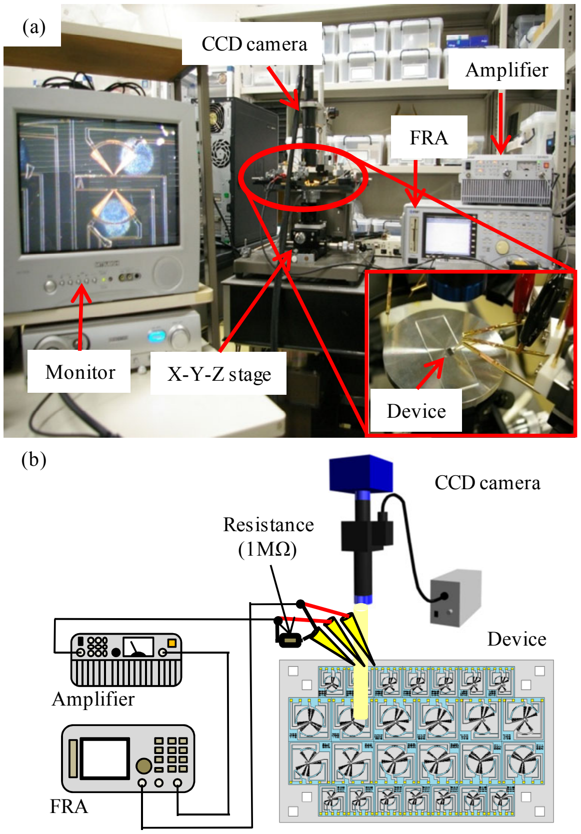
$$E_1 = (2\pi f_1)^2 \frac{6\ell\rho\theta R^4}{w^3} \tag{2}$$

After that, a nanometer-thick film that is a target material was deposited onto the substrate, and the resonant frequency of the sum of the film and substrate was measured. After these measurements, the Young's modulus, $E_2$, of the deposited film can be simply given by:

$$E_2 = E_1\left\{\left(\frac{f_{1+2}}{f_1}\right)^2\left(\frac{t_1}{t_2}+\frac{\rho_2}{\rho_1}\right)-\frac{t_1}{t_2}\right\} \tag{3}$$

where $t_1$ and $t_2$ are the thickness of substrate and film, and $\rho_1$ and $\rho_2$ are the density of substrate and film, respectively. Eq. (3) is obtained if Poisson's ratio of deposited film is the same as that of substrate. In terms of its simplicity, we utilized the equation for Young's modulus derivation of thin films.

When $E_2$ is calculated using Eq. (3), the film density, $\rho_2$, and film thickness, $t_2$, have to be known. We measured both the values for Al, CH$_4$- and CHF$_3$-derived polymer films with X-ray reflectometry (XRR). Figure 2 shows a typical XRR curve of Al film deposited on a Si substrate. The circle plots and the solid line are indicative of the measured and fitted data, respectively. The X-ray reflectivity drastically decreased at around the grazing angle of incidence of 0.5 deg. The reflectivity at the drop point provides information of density for the measured film. The X-ray reflectivity decreases with an increase of the angle, showing a waveform. The cycle of the waveform provides information of film thickness. The obtained density of sputtered Al film was 2841 kg/m$^3$, comparable to bulk value, 2700 kg/m$^3$. The thickness was also calculated to be 58.3 nm.

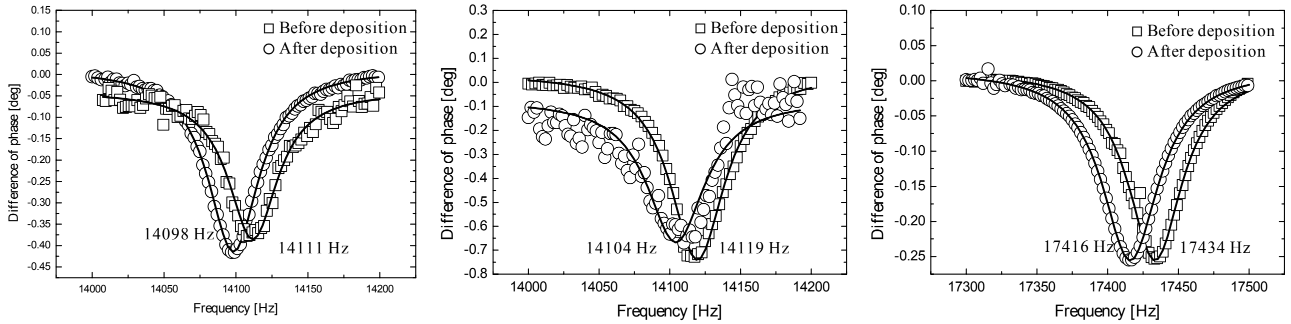


*Figures 3: (a) Photograph and (b) schematic of the resonance testing setup.*

Table 1 lists the measured density and thickness of Al, CH$_4$- and CHF$_3$-derived polymer films. All the data listed were used for calculating Young's modulus, $E_2$, of those films using Eq. (3).

## Resonant Frequency Measurement

Figure 3 shows the experimental setup for resonant frequency measurement of the MEMS resonator array. The test system is composed of frequency response analyzer (FRA) for applying a voltage and analyzing a resonance, high voltage amplifier for amplifying a driving voltage, micro-probes made of Au with X-Y-Z positioning stages for applying a voltage, a charge coupled device (CCD) camera for positioning micro-probes onto electrode pads. A driving voltage ranging from 20 to 40 V$_{pp}$ with sinusoidal waveform was applied to the electrodes. During operation, the phase and amplitude signals from a capacitive sensor



*(a) Al* *(b) $CH_4$* *(c) $CHF_3$*

*Figures 4: Phase curves before and after depositions of (a) Al film, and (b) $CH_4$-, (c) $CHF_3$-derived polymer films. Resonant frequency, which was the frequency at the peak position, was obtained by Lorenz function fitting.*

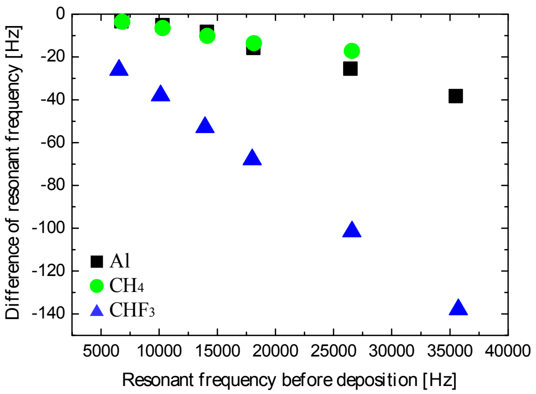


*Figure 5: Relation between resonant frequency before film deposition and the change in the frequency by film deposition.*

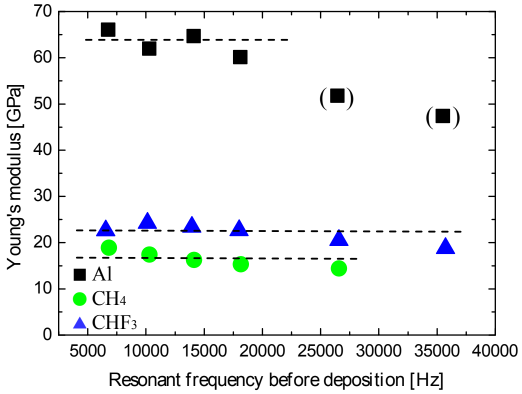


*Figure 6: Relation between calculated Young's modulus and resonant frequency of a resonator before deposition.*

*Table 2: Summary of resonance test results by using a MEMS resonator with the resonant frequency of 10 kHz.*

| Film | Gas | Thickness [nm] | Resonant frequency Before deposition [Hz] | Resonant frequency After deposition [Hz] | Difference of resonant frequency [Hz] | Calculated young's modulus [GPa] |
|---|---|---|---|---|---|---|
| Al | - | 58.3 | 10312.0 | 10309.0 | -3.0 | 64.68 |
| | | 99.5 | 10286.1 | 10281.0 | -5.1 | 61.98 |
| | | 146.4 | 10138.0 | 10123.0 | -15.0 | 49.12 |
| Polymer | $CH_4$ | 56.4 | 10015.0 | 10013.0 | -2.0 | 38.45 |
| | | 85.1 | 10297.7 | 10291.2 | -6.5 | 37.95 |
| | | 182.8 | 10343.8 | 10331.0 | -12.8 | 38.73 |
| | $CHF_3$ | 45.7 | 10328.0 | 10305.0 | -23.0 | 29.25 |
| | | 103.4 | 10124.5 | 10086.7 | -37.8 | 36.17 |
| | | 159.4 | 9758.3 | 9701.0 | -57.3 | 32.97 |

were recorded. All the tests were carried out at ambient temperature in laboratory air.

## RESULTS AND DISCUSSIONS

### Resonance Test Results

Figures 4 show the phase curves of (a) Al, (b) $CH_4$-, and (c) $CHF_3$-derived polymer films obtained by FRA measurements during the resonance tests. In all the graphs, the square and circle plots indicate the phase curves before and after film deposition, respectively. The solid lines are indicative of the fitted curves with Lorenz function. In Fig. 4(a), before deposition of Al film, the phase curve has a downward sharp peak. The frequency at the peak position was calculated to be 14.111 kHz, which indicates the resonant frequency of the resonator used. By deposition of the film with 99.5 nm-thick, the resonant frequency shifted to 14.098 kHz because the mass of the resonator increased by the deposition. In Figs. 4(b) and (c), the resonant frequencies dropped by several Hz by depositions of $CH_4$- and $CHF_3$-derived polymer films with 85.1 and 159.4 nm-thick as with Al film. This indicates that the resonant frequency is sensitive to the change in the mass of resonator by film deposition.

Figure 5 depicts relationship between resonant frequency before film deposition and the change in the frequency by film deposition. In the case of a resonator having the resonant frequency of 7 kHz, the change of the frequency is found to be only -3.4 Hz by depositing Al film with 99.5 nm-thick. In a resonator having the resonance frequency of 35.5 kHz, the change of the frequency by the same film deposition is approximately -38 Hz, which is 11 times larger than that in the resonator having a 7 kHz-resonant frequency. Other two films exhibit the same trend as Al film. This suggests that the use of a resonator with higher resonant frequency provides nanoscale film's Young's modulus calculation with higher accuracy.

Table 2 lists the resonance test results by using a resonator with the resonant frequency of 10 kHz. The

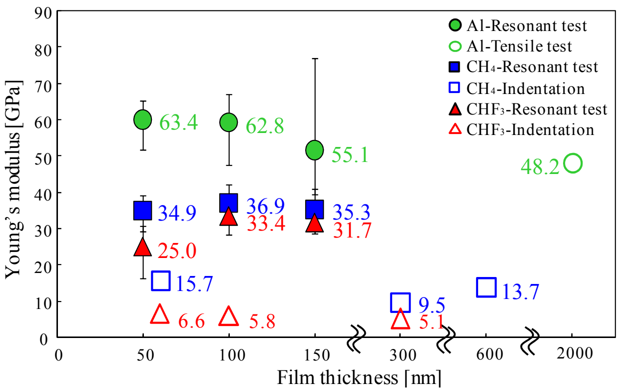


*Figure 7: Measured Young's modulus as a function of film thickness.*

amount of the change in resonant frequency also depended on the film thickness. Thicker film provided larger change in resonant frequency.

### Young's Modulus Measurement

Figure 6 shows relation between the calculated Young's modulus and resonant frequency of a resonator before deposition. The mean Young's modulus of Al film measured by a 18 kHz resonator is found to be 62 GPa, which is smaller by approximately 8 GPa than that of the bulk. The measured value stays constant throughout the resonant frequencies of resonators though the data derived from resonators with their resonant frequencies over 25 kHz deviate from the constant value. This would be caused by an experimental error in measuring film thickness. In the cases of $CH_4$- and $CHF_3$-derived polymer films, the measured Young's moduli are found to be 35.7 and 30.3 GPa on average, respectively, and shows no significant influence of resonant frequency of a resonator used.

Figure 7 represents the measured Young's modulus as a function of film thickness. The closed plots of all the styles indicate the moduli derived from the resonance tests, and the open plots are indicative of that from the tensile and nanoindentation tests. The mean Young's modulus of 50 nm-thick Al film is found to be 63.4 GPa, which is approximately 10 % smaller than the bulk value. The modulus gradually decreases with increasing film thickness. We considered the change of Young's modulus as not specimen size dependency but an experimental error. The mean value of the modulus obtained from the resonance tests is 60.4 GPa, 25 % larger than that from the tensile tests of 2 μm-thick Al films. The tensile test setup may have a technical problem regarding an accuracy of load cell when the minute specimens are subjected to tensile loading. If the issue is overcome, the tensile test would provide a similar value to the resonance test. On the other hand, the Young's moduli of $CH_4$- and $CHF_3$-derived polymer films of 50 nm-thick are 34.9 and 25.0 GPa on average, respectively. Those values are kept almost constant in spite of a film thickness change ranging from 50 to 150 nm. The mean values of Young's modulus are larger by about 19.2 and 18.4 GPa than those by nanoindentation tests. The smaller values by nanoindentation tests would be caused by the tip-shape effect originating from very small penetration depth.

All the films tested did not show the effect of film thickness on the Young's moduli. In future, we will improve the resonance test setup in order to investigate Young's modulus of several-nm-thick films.

## CONCLUSION

We proposed novel Young's modulus measurement method for nanometer-thick film materials. The Young's moduli of Al, $CH_4$- and $CHF_3$-derived polymer films were determined by measuring the resonant frequencies of a resonator before and after film deposition. The mean values of Young's modulus for those films were 60.4, 35.7, and 30.0 GPa. All the films were not dependent on the thickness. By virtue of its simplicity and adequacy, the proposed measurement method would be useful when information about the Young's modulus of nanometer-thick film materials has to be obtained.

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