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Ultrathin single-crystalline-silicon cantilever resonators: Fabrication technology and significant specimen size effect on Young's modulus

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Ultrathin resonant cantilevers are promising for ultrasensitive detection. A technique is developed for high-yield fabrication of single-crystalline-silicon cantilevers as thin as 12 nm. The formed cantilever resonators are characterized by resonance testing in high vacuum. Significant specimen size effect on Young's modulus of ultrathin (12–170 nm) silicon is detected. The Young's modulus decreases monotonously as the cantilevers become thinner. The size effect is consistent with the published simulation results of direct-atomistic model, in which surface effects are taken into consideration. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1618369]

With the rapid development of nanoelectromechanical-system (NEMS) technologies, ultrathin cantilevers have been used as ultrasensitive sensors for ultrafine resolution applications.[1–3] Cantilever NEMS sensors can be classified into two kinds. The first kind borrows the sensing mode of scanning probe microscope by measuring the static deflection of the cantilevers, while the cantilevers in the second kind act as resonant sensors with frequency shift measured.[4–7] Compared with the first kind, the resonant cantilevers are easier to be integrated into miniaturized systems for portable applications. Single crystalline silicon (SCS) is suitable for NEMS resonant cantilevers due to its good mechanical properties and integration compatibility with microelectronic circuits.[8,9]

For high sensitivity, the resonant cantilevers should be mechanically compliant enough, while high resonant frequency is required for fine resolution. According to mechanics, the spring constant of a cantilever is $k = 0.25Ewt^3/l^3$, where l is the cantilever length, w is the width, t is the thickness and E is the Young's modulus.[10] Obviously, the spring constant decreases with either reduced t or increased l. As the fundamental resonant frequency of a cantilever is $f_0 \approx 0.162(E/\rho)^{1/2}t/l^2$ (where ρ indicates material density),[10] increasing cantilever length is not preferred as it lowers the resonant frequency more seriously than decreasing cantilever thickness. Taking mass-loading detection for example, frequency shift due to a mass (loaded at the end of a cantilever), Δm, can be expressed as $2\pi\Delta f_0 \approx (35Ewh^3)^{1/2}[(33m_c l^3)^{-1/2} - (33m_c l^3 + 140\Delta m l^3)^{-1/2}]$, where $m_c = \rho whl$ is the mass of the cantilever.[10] Assuming $\Delta m/m_c \ll 1$, the sensitivity of frequency shift is approximately $\Delta f_0/\Delta m \approx 2.12 f_0/m_c$. It is obvious that the thinner the cantilever, the higher the f_0 and the smaller the m_c, i.e., the higher the sensitivity. Therefore, thinning the cantilevers results in fine resolution of the resonant sensors.

Efforts have been made on the fabrication of ultrathin SCS cantilevers.[7,11–13] Using previous techniques we can fabricate cantilevers as thin as 60 nm.[9] However, the yield is low. For fabricating thinner cantilevers, a fabrication technique has been developed with following steps sketched in Fig. 1.

(1) The substrate is (100) silicon on insulator wafer with P^- top-layer silicon. The top-layer thickness, t, is 100 or 300 nm. With the first dry oxidation, the silicon layer is consumed by a thickness of t_1, while the thickness of grown SiO_2 is $2.27t_1$. Then $\langle 110 \rangle$-oriented cantilevers are patterned

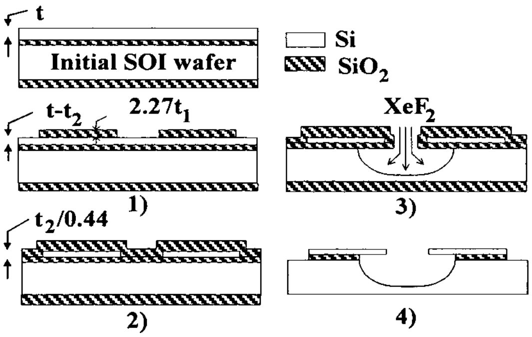

FIG. 1. Process flow of developed fabrication technique for ultrathin silicon cantilevers.

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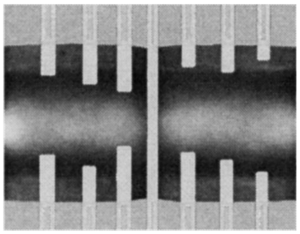


FIG. 2. Top-view photomicrograph of fabricated 12-nm-thick cantilevers.

by photolithography. The SiO_2 layer outside the cantilever regions is removed by HF.

(2) The second dry oxidation is processed to consume up the remaining silicon in the surrounding areas. The thickness of the consumed silicon is $t_2 = t - t_1$. During this step, the oxidization rate at the cantilever regions is slower than that at the surrounding areas due to existing SiO_2 layer on the cantilever surface prior to the second oxidation. Therefore, a layer of silicon with a definite thickness, i.e., the thickness of the cantilevers, remains after the second oxidation. The cantilever thickness can be controlled by adjusting the ratio of t_1/t_2. The experiment shows that the process tolerance on cantilever thickness is within $\pm 1-2$ nm.

(3) Windows on the field oxide are opened near the cantilevers and the SiO_2 in the windows is removed. XeF_2 isotropic dry etching is used to excavate into the silicon substrate. Along with the etching downwards, lateral underetch is simultaneously processed, removing the substrate silicon beneath the cantilevers. Etching end-point inspection under a microscope is used for required cantilever length.

(4) The SiO_2 layer covering the cantilevers is stripped with buffered HF. The cantilevers are released by CO_2 supercritical-point drying. The photomicrograph in Fig. 2 shows the fabricated 12-nm-thick cantilevers. A high yield ($>95\%$) is obtained.

Resonant properties of the cantilevers are characterized by using a similar setup described in Ref. 9. The naturally oxidized layer and absorbate on the cantilever surface are precleaned by heating the cantilever chip to 700 °C for 2 h and, then, pulse-heating to 1100 °C for three times with a heating period as 2 s. The resonance of the cantilever is excited by pulsed light beam from a laser diode and maintained via a feedback-signal controlled chopper. Another fiber-coupled laser is used for vibration signal picking-up.[9] Figure 3 shows the measured quality factor (Q) of a 12-nm-thick cantilever. The Q value is about 20 000, as expected for fine resolution of frequency shift. The 12-nm-thick cantilever merely consists of 22 layers of silicon lattice in its thickness. The high Q value indicates that so thin cantilevers still show satisfactory mechanical properties.

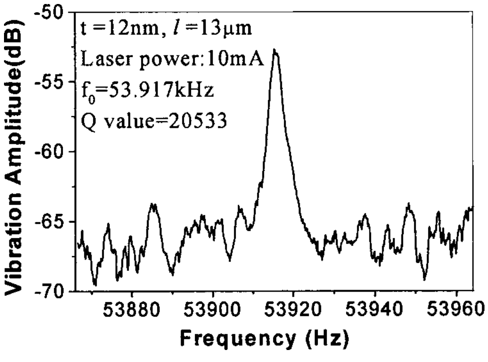
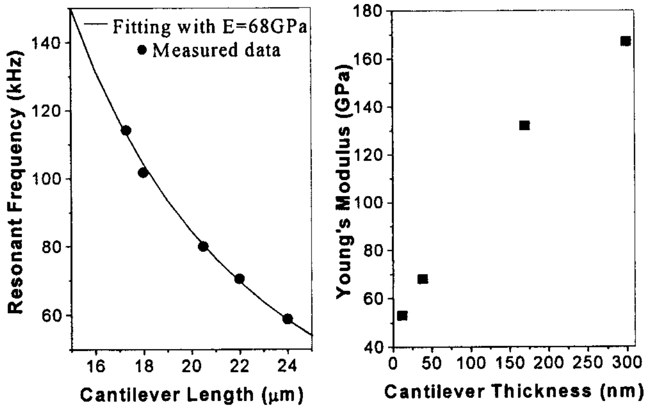


FIG. 3. Amplitude-frequency relation of a 12-nm-thick cantilever showing a quality factor value of about 20 000.

Young's modulus, E, has strong effect on the resonant cantilevers, as it is proportional to the square of resonant frequency. The Young's modulus of single crystal material reflects physics of interatomic bond energy and lattice structure. For bulk material or millimeter to micron scale specimen, E value of ⟨110⟩-oriented SCS has been measured as about 170 GPa.[14] For ultrathin SCS, however, surface effects may play important roles in addition to bulk effects, as the number of the atoms at the double surfaces are comparable with that in the body. Continuum theory, like finite element (FE), cannot correctly simulate the NEMS surface effects.[15] Direct atomistic simulation has to be used for modeling, even though the simulation is very time consuming.[16] For improving the computation efficiency, a tight binding-molecular dynamics (MD)–FE seamless coupling model of length scales and a coarse-grained MD model are developed to simulate quartz crystal and SCS NEMS resonators.[15,17,18] The simulation results show significant size effect on Young's modulus. Based on the models, the Young's modulus should decrease monotonously from the value of bulk material when the thickness of the cantilevers is reduced into nanometric scale.[15,16] However, there has been no experimental work to verify the size-effect model. Though a 255-nm-thick SCS specimen was measured, there was no significant size effect of E detected.[19] The size effect is expected to be detected with even thinner specimens.

Resonant method is suitable for measuring Young's modulus of NEMS structures like carbon nano tubes.[20] As shown earlier, the measured high Q value of the ultrathin cantilevers indicates low mechanical energy loss. Under this condition, resonant measurement is reasonably used here for determination of Young's modulus, as the expression of resonant frequency in terms of Young's modulus and the cantilever geometries is based on mechanical energy conservation.[10] The frequency data for a set of cantilevers with the same thickness and different lengths are fitted into the frequency expression to extract the value of E. The results show significant specimen size effect on E value. Figure 4(a) shows a result of $E = 68$ GPa for 38.5-nm-thick cantilevers. With the extracted E value for varied cantilever thickness, the dependence of Young's modulus on cantilever thickness is obtained and shown in Fig. 4(b). E of ⟨110⟩-oriented SCS cantilevers decreases monotonously from 167 GPa for 300-nm-thick cantilevers to 53 GPa for 12-nm-thick cantilevers.

To rule out the possibility of laser heating effect, temperature coefficient of E is evaluated. Figure 5 shows the measured resonant frequency shift of a 38.5-nm-thick cantilever versus temperature. The cantilever is electrically heated from 20 to 753 °C. The shift rate of resonant frequency is found to be -0.45 ppm/°C. Temperature coefficient of E can be calculated as -82 ppm/°C with Eq. (7) of Ref. 21 and the corresponding material parameters in Ref. 22. Considering that the decrease of E value for 733 °C temperature range is only 6%, it is clear that the significant decrease of E



(a) (b)

FIG. 4. (a) Resonant frequency data for a set of 38.5-nm-thick cantilevers (with varied length) are fitted into theoretical curve based on resonant frequency expression. Young's modulus of 68 GPa is obtained from the fitted result. (b) The monotonous decrease in Young's modulus value of the ultrathin SCS cantilevers is obtained. The thickness of the cantilevers is in the range of 300–12 nm.

for ultrathin silicon is mostly induced by specimen size effect instead of the laser-light influence.

The monotonous decrease in Young's modulus of ultrathin SCS is consistent with the modeling, in which surface effects are considered.[15,16] A skin Young's modulus can be defined for surface region (skin depth can be in nanometric scale). The overall Young's modulus value is the weighted average of the bulk modulus and the skin modulus with the respective thickness as weight factor. The models indicate that even a nanoscale specimen with perfect lattice structure bears lower Young's modulus value than bulk specimens. Surface effects such as dangling bonds and surface relaxation account for the deviations from bulk behavior.[15,16] Besides, the models reveal that a structure with defects causes further decrease in E value.[15–17] Though plasma damage is avoided and surface precleaning is performed in our experiment, surface defects might still exist to further decrease the E value. It may explain why Young's modulus of our cantilevers decreases somewhat earlier than the simulated results for a perfect NEMS specimen.[16]

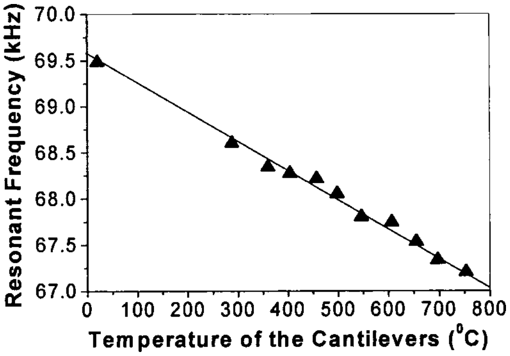


FIG. 5. Resonant frequency shift of a 38.5-nm-thick cantilever vs temperature change in the range of 20–753 °C.

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