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Effect of ion attachment on mechanical dissipation of a resonator

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The influence of adsorbates on the surface-related mechanical dissipation of a silicon resonator was investigated. Different ion species were attached on a silicon resonator with a native oxide, and the quality factor (Q factor) and resonant frequency changes were observed by *in situ* measurement. It was found that water ion attachment creates OH terminations and results in a similar Q factor to that after exposure to the atmosphere. Nitrogen ions created very active sites for surface mechanical dissipation. In contrast, exposure to hydrogen ions increased the Q factor by a factor of 2. Hydrogen ions caused the dissipation sites on the surface to become inactive or less active for surface-related mechanical dissipation. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1993771]

Silicon resonant sensors play a key role in certain applications, such as scanning probe microscopy,[1] chemical sensors,[2] and mechanical filters.[3] It has been known that the miniaturization of the sensor can reduce thermomechanical noise;[4] therefore, the detectable minimum value can be decreased by miniaturization.[5] This is the driving force for miniaturization of the sensor; however, as the volume of the sensor decreases, the quality factor (Q factor) decreases proportionally to the volume.[6–8] This degradation of the Q factor is an obstacle to advantages gained from scaling, limiting the sensitivity in nanomechanical sensors. In some interesting applications such as magnetic resonance force detective microscopy,[9] the lack of sensitivity related to the resonating sensor is a crucial problem. The vibration energy loss is inversely proportional to the corresponding Q factor, and the total energy losses are given by the sum of losses of various origins, including gas dumping loss, intrinsic mechanical loss in the bulk, support loss, thermoelastic loss, and surface loss.[8] At the submicron scale, surface loss is considered to be the dominant mechanism in the degradation of the Q factor. On the surface, there are many defects, such as adsorbed gas molecules and contaminants, which capture single or multiphonons in a state of mechanical motion and emit them as an acoustic dissipation. The defect density on the surface is much larger than that in the bulk, and this is the reason why the Q factor is proportional to the volume in the nano- to microscale. However, it is difficult to quantitatively evaluate the losses, so that very little has been understood regarding surface loss.

Yasumura *et al.* found that the annealing of single crystalline silicon cantilevers in a nitrogen atmosphere at 700 °C improves the Q factor by a factor of approximately 3, because the adsorbates or surface defects created during the cantilever fabrication are reduced with this thermal treatment.[6] An improvement of the Q factors, of over 20 times, has been demonstrated by flash annealing in UHV,[10] which removes the surface oxides and contaminants and reconstructs the silicon atoms. However, the clean silicon surface is unstable and easily oxidized in a gaseous environment, even in a vacuum, due to residual gases, and consequently the Q factor gradually decreases.[11,12] The surface defect density depends on the temperature and residual gases in UHV, and different annealing conditions lead to different Q factors.

The surface sensitivity has been observed in the mechanical behavior of the cantilever exposed to various gases.[12] In the case of atomic hydrogen adsorption on a clean silicon surface, a high Q factor is maintained, even after exposure to the atmosphere.[7] In contrast, exposure to oxygen molecules slightly decreases the Q factor, and exposure to hydrocarbons significantly decreases the Q factor.[11,12] Under normal atmospheric exposure, a long-term degradation of the Q factor over 6 months has also been observed.[13] Such degradation is probably caused by the gradual deposition of contaminants.[13] From a practical point of view, such degradation of the Q factor in micro- or nanomechanical sensors is a critical issue. In this letter, the surface loss due to ion attachment on a silicon cantilever with a native oxide has been reported. Different ion species will react with the silicon dioxide surface and form chemical bonds with the ions, which leads to changes in the mechanical Q factors. The effect of ion attachment on the Q factors is experimentally shown for several ion species.

Figure 1 shows a schematic figure of the experimental setup. A single crystalline silicon cantilever was placed in a vacuum chamber and self-oscillated at constant vibration amplitude. The rectangular single crystalline silicon cantile-

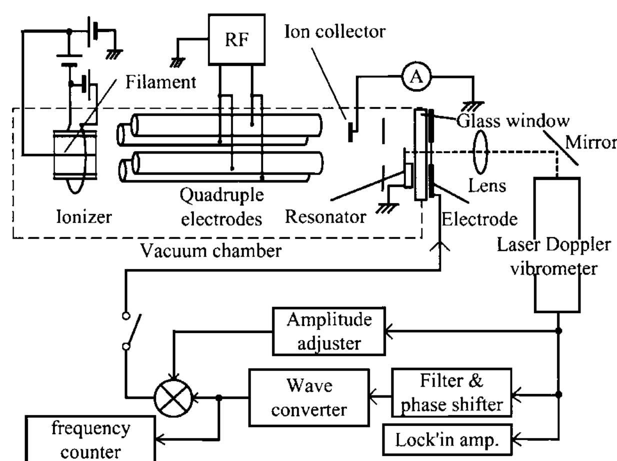

FIG. 1. Schematic diagram of the experimental setup.

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ver with a length, width and thickness of 545, 163, and 0.50 μm, respectively, was made from a (100)-oriented silicon wafer. The calculated spring constant of the cantilever is 0.0053 N/m. The cantilever was electrostatically oscillated by applying an ac voltage between an electrode and the cantilever, and the vibration was measured using a laser Doppler vibrometer via an optical window of the chamber.[10,11] The output signal of the laser Doppler vibrometer was sent to the metal electrode via a phase shifter, filter, and wave converter, which self-oscillates the cantilever. In order to oscillate the cantilever at constant amplitude, an amplitude adjuster consisting of a lock-in amplifier and differential amplifier was employed in the feedback loop. The Q factor was measured from the decay curve of the vibration amplitude when the feedback loop was turned off and the amplitude was ringing down.[11] The oscillation frequency was measured using a frequency counter, and the vibration amplitude and ring-down curve were measured using a lock-in amplifier.

The cantilever was exposed to low energy ions that were generated using an ionizer, in which electrons from a hot filament are accelerated by a grid electrode and ionized residual gases resulting from electron bombardment in a vacuum chamber. The vacuum chamber was pumped to a pressure of approximately 2×10^{-2} Pa using a turbomolecular pump. The generated ions are accelerated to the quadrupole electrodes with an acceleration voltage of 12 V, which acts as a mass filter. Depending on the frequency of the rf applied to the quadrupole electrodes, specific ions are filtered and attached to the cantilever. The ion flux density was monitored using an ion collector, and the value was calibrated to flux density at the cantilever. In front of the cantilever, an electrically shielded mesh electrode was placed, in order to confirm that there was no electric field influence on the cantilever. Altering the current provided to the hot filament varied the ion density flux.

The ion attachment effect on the Q factors was investigated with regard to H_2^+, O_2^+, H_2O^+, and N_2^+ ions. The cantilever was self-oscillated at a fundamental resonant frequency of approximately 2280 Hz. The initial Q factor was approximately 13 200 without any ion attachment after introduction into the chamber. The cantilever surface was then exposed to O_2^+ ions with an ion current of approximately 1 pA/cm^2 for 30 s, and consequently the Q factor was increased up to approximately 19 000; a factor of 1.44. The cantilever was then exposed to H_2O^+, with an ion density of approximately 0.3 pA/cm^2 for about 7.5 min, as shown in Fig. 2(c). It was found that exposure to H_2O^+ ions quickly decreases the Q factor to 13 500, close to the initial value. Figure 2(b) shows the typical change of the Q factor when H_2O^+ is attached after O_2^+ exposure. Figure 2(a) shows the frequency response when exposed to H_2O^+ ions. In a similar way, the effects of exposure to H_2^+ and N_2^+ ions were also investigated.

It was discovered that when the cantilever was exposed to H_2^+ ions, the Q factor was gradually increased up to 25 000; the typical behavior of the Q factor and the ion flux density are shown in Figs. 3(a) and 3(b), respectively. In contrast, the Q factor was decreased to approximately 10 000 in the case of exposure to N_2^+ ions. These modified surfaces were stable, and in all cases, the Q factor reaches a constant value if the exposure dose is sufficient. After leaving the cantilever in a vacuum for at least 1 week, the Q factor was measured again, and the same value resulted. Therefore, it is considered that built-in charges, due to ion attachment, do not affect to the mechanical characteristics. The measured Q factors are summarized in Fig. 4(a) and Fig. 4(b), which show the corresponding frequency changes after attachment of the ions.

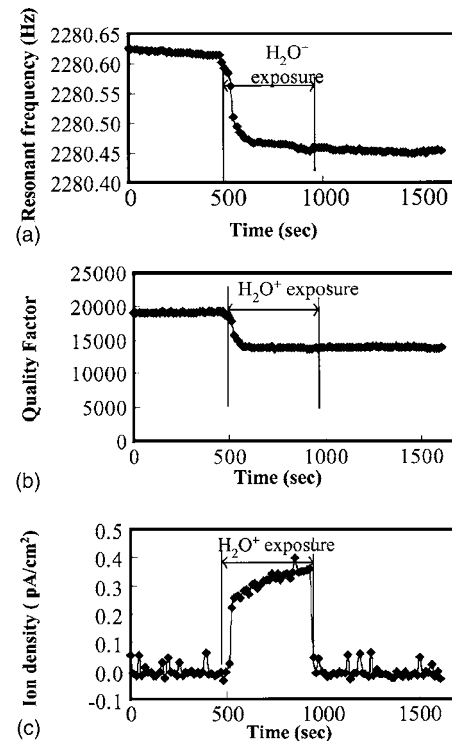

FIG. 2. Effect of H_2O ion irradiation: (a) change of the frequency, (b) change of the Q factor, and (c) corresponding ion flux density.

Two influences on the frequency change are considered. One is the mass loading effect and the other is the surface stress induced frequency change due to ion attachment.[11] If the surface stress affects the frequency change, the loaded mass, Δm, is given by the frequency change before and after mass loading according to $\Delta m=(k/4\pi^2)(1/f'^2-1/f_0^2)$, where k is the spring constant, and f_0 and f' are the frequencies before and after mass loading, respectively. According to this assumption, mass loading in the case of H_2O^+ ions can

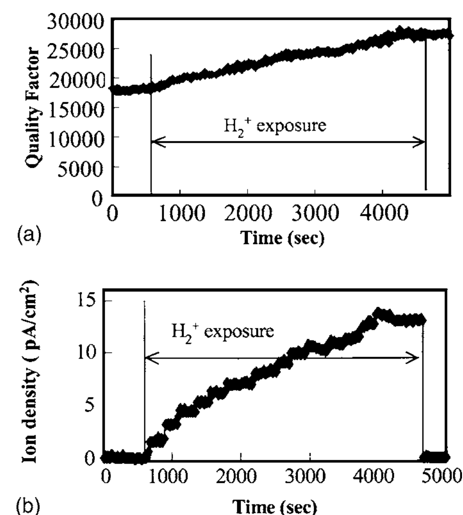

FIG. 3. Effect of H_2 ion irradiation: (a) change of the Q factor, (b) corresponding ion flux density.

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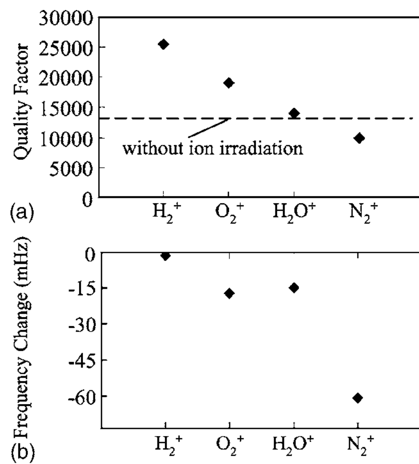

FIG. 4. (a) Q factors after irradiation with various ions. (b) Resonant frequency change due to ion attachment.

be calculated as 20 pg, which corresponds to the adsorption density of 6.8 water ions/nm^2. On the other hand, the resonant frequency, f_{stress}, due to surface stress, σ, can be expressed as $f_{\text{stress}}=f_0\sqrt{1-\sigma(60l^2/7Et^3)}$, where l and t are the length and width of the cantilever, and E is Young's modulus. Adsorbed ions cause compressive surface stress $(\sigma<0)$ from the charge interaction, and therefore, the resonant frequency must increase in this case. If the adsorbate induces tensile surface stress, the resonant frequency decreases, which is consistent with the measured result. However, it was difficult to estimate both contributions of mass loading and adsorbate stress, and from the earlier consideration it is possible that mass loading is the main contribution to the frequency response.

Adsorption of atoms and molecules create adsorbates or surface terminations,[14] which are active in the surface dissipation processes. Different surface states will exhibit different surface losses, depending on the activity for mechanical dissipation. In the atmosphere, an oxidized silicon surface is terminated by OH, and a thin water layer exists on the surface. Attachment of H_2O^+ ions creates a similar surface to that after exposure to the atmosphere; therefore, the Q factor is almost same. Adsorption of N_2^+ ions will cause strong dissipation sites on the surface. In contrast, the attachment of O_2^+ ions will change the surface state from an OH termination to an oxygen termination, which causes an increase in the mechanical Q factor. A large improvement of the Q factor was achieved by attachment of H_2^+ ions. Hydrogen ions will terminate the active sites on the surface and make them inactive or less active to mechanical dissipation. A very slow Q factor change during H_2^+ attachment suggests that the reaction rate of this inactivation process is low. Hydrogen or oxygen ion attachment may involve removal of hydrocarbons; however, with only this cleaning effect, the reversible change of the Q factor could not be explained. A reversible Q factor change is evidence that the chemical reactions involved occur at the surface, and that changes to the surface are related to mechanical dissipation.

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