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Effects of annealing and temperature on acoustic dissipation in a micromechanical silicon oscillator

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The temperature dependence (15–320 K) of the acoustic dissipation was studied for some lower vibrational modes of a suspended silicon plate 1.5 μm thick. Our oscillator was exposed to the laboratory environment prior to measurement, laser annealed while in a cryogenic vacuum, and remeasured. We find a dissipation peak at 160 K, similar to results by others, and a second dissipation peak near 30 K. Annealing reduced the dissipation at 160 K by as much as a factor of 10, and gave quality factors as high as 1.4×10^6 at 470 kHz and our lowest temperature. Our data support the idea that the 160 K peak is related to adsorbates, and show this mechanism is important at room temperature. Post-anneal room-temperature dissipation appears to be limited by thermoelastic loss for certain modes. [DOI: 10.1063/1.1921354]

Stable silicon micromechanical structures with low dissipation are of considerable interest for both scientific and technological reasons. Acoustic (or mechanical) dissipation in these devices is not well understood. Dissipation in micro- and nanoscale oscillators has been found to be greater than in larger structures.[1,2] This has been ascribed to effects at or near the surface, including oxidation,[3,4] defects associated with surface roughness, and damage associated with plasma processing.[5] A further category of losses, addressed in this study, relate to material adsorbed onto the oscillator from the ambient environment.[6] We find that dissipation peaks in the temperature dependent losses of a silicon paddle are strongly affected by annealing.

Our sample was a perforated paddle 1.5 μm thick (Fig. 1) made from boron doped ⟨100⟩ silicon having a room temperature resistivity of 20 Ω cm. Standard techniques for silicon-on-insulator fabrication[7,8] were used: photolithography, reactive ion etching, HF release, and critical point drying. A rapid thermal anneal (in N_2 at 1070 K for 30 s) was used to repair process induced damage.[9] Oscillator mode shapes are shown in Fig. 2.

Measurements were carried out with the sample mounted in a He^4 optical cryostat. The acoustic loss and resonant frequencies were determined using a heterodyne Michelson interferometer[7] to measure normal velocity. The interferometer was fiber-coupled to a telescope focused (through windows) onto the sample. A beamsplitter and IR converter facilitated sample alignment. To reduce sample heating we chose a laser wavelength of 1.3 μm, below the Si band-gap energy, rather than visible light.[10] Adequate reflectivity was provided by the refractive index mismatch at the surface of the oscillator; no metal coating was used. We believe laser heating of this sample is primarily due to free carrier absorption, and cooling limited by the thermal resistance of the slender supports. Observations of the temperature dependence of the dissipation proved that optical power levels used for measurement caused negligible sample heating at temperatures above 15 K. Most measurements were made with the sample in a cryopumped LHe (4 K) vacuum. A single crystal piezoelectric[11] actuator was used to vibrate the entire sample chip. This gave adequate inertial drive down to the lowest temperatures tested. Efforts were made to measure dissipation in the small displacement limit to avoid nonlinear effects. The interferometer output was filtered using lock-in detection at the drive frequency, prior to sampling. Dissipation was measured using both ringdown (envelope decay) and resonance linewidth techniques. Discrepancies between techniques were typically <10%; ringdown measurements are shown below.

Annealing was performed using an argon ion laser operated at 488 nm and coupled to a telescope via single mode fiber, producing a near Gaussian spot about 200 μm in diameter. The oscillator was illuminated for 30 s (far longer than the thermal relaxation time determined by the paddle heat capacity and support conduction), after which the laser power was ramped down over several seconds to reduce possible quenching effects. The annealing process involved a sequence of such heating cycles. After each heating cycle the Q of the 1-1 mode was measured at a fixed temperature. Laser power was then slowly increased until a maximum in

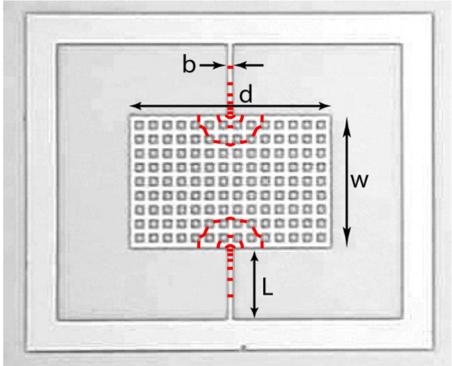

FIG. 1. Microscope picture of the single-crystal silicon paddle oscillator. The dimensions are: $d=147.0$ μm, $w=97.75$ μm, $b=4.15$ μm, and $L=50.9$ μm. Each perforation measures 5×5 μm^2. The paddle and supports are suspended from the rectangular frame. Superposed are simulated temperature contours during laser annealing. Parameters used are a net radiative flux of 2.7×10^6 W/m^2, equaling 80% of the estimated 130 mW annealing power, and a 204 μm diameter Gaussian spot, calculated from the optical geometry. Assuming ambient temperature (on the frame) fixed at 40 K, temperature at paddle center is 1422 K. The contour interval is 154 K. Material thickness is 1.5 μm for the paddle and supports, and 625 μm for the frame.

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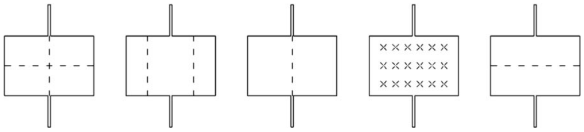

FIG. 2. Schematic of mode shapes for 1-1, 2-0, torsion, plunging, and rocking modes. Nodal lines are shown for the 1-1 and 2-0 "plate" modes, torsion mode, and rocking mode. In the plunging mode, motion is perpendicular to the plane of the paddle.

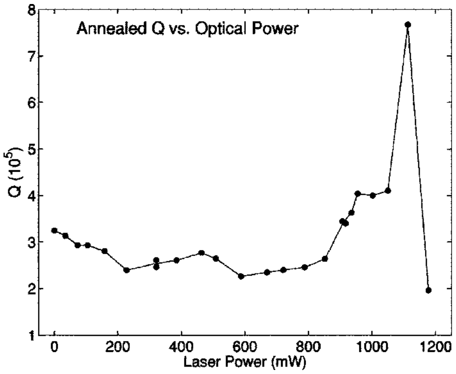

FIG. 3. Example of an annealing sequence showing Q of the 1-1 mode vs power in the optical fiber (which is proportional to the power incident on the sample). The line is a guide to the eye.

the measured Q was obtained. A typical plot of the dissipation varying with annealing power is shown in Fig. 3; similar results were obtained by Aubin *et al.*[12] Maximum Q was consistent across replicate oscillators on the sample. No direct thermometry was available to monitor the anneal. Testing on replicate oscillators demonstrated that power levels used for annealing were close to the threshold for melting the paddles. Based on this, as well as nonlinear FEM simulation,[13] we estimate that the paddles were likely raised to at least 1000 K when annealed into the low dissipation state. The ends of the supports farthest from the plate were at a temperature close to the sample holder (held at 42 K), whereas the paddle temperature is fairly uniform (see Fig. 1). Our simulation used bulk thermal conductivity values above room temperature,[14] and at low temperatures, boundary scattering limited values based on an experimental study.[15]

Prior to the measurements reported here the entire chip had undergone a rapid thermal anneal. Subsequently it was exposed to the atmosphere in the laboratory for several weeks; this is the preannealed state. The total dissipation in this preannealed state was measured from 15 to 320 K for each of the modes. The oscillator was then laser annealed[12,16] and the measurement repeated. The preanneal and postanneal data sets shown in Fig. 4 were each made without breaking the cryogenic vacuum, and 4 K cryopumping was maintained throughout the post-anneal data run.

As seen in Table I, different modes had very different anneal induced fractional frequency shifts. The trends in frequency shifts reproduced across different replicate oscillators. Before annealing, the plunging and rocking mode frequencies show nonmonotone variation with temperature. We ascribe this to slight buckling of the support legs produced by differential thermal contraction; the legs are by inference under compressive stress. These anomalies disappear upon annealing. We speculate that annealing puts the legs under tensile stress, this stress also producing the observed large increase in the plunging mode frequency. In contrast, the 1-1 mode, which involves negligible strain in the supports, showed a monotone frequency variation with temperature before annealing and little anneal induced frequency shift.

Dissipation is shown as a function of temperature, prior to annealing in Fig. 4(a), and postannealing data in Fig. 4(b). There are two peaks readily apparent at temperatures of about 30 and 160 K. The preanneal 160 K peak has essentially the same dissipation for all five modes shown. Annealing reduces the dissipation by an order of magnitude for the 1-1 and 2-0 "plate" modes, a factor of 3 for the plunging mode, and has little effect on the torsional mode. In the case of the "plate" modes, the elastic energy is predominantly in the paddle, whereas the torsional and plunging modes have most of their energy in the support beams. Our thermal simulation (Fig. 1) indicates that large portions of the supports did not rise above room temperature during the anneal, making the anneal less effective for the torsion and plunging modes.

At low temperature, the low post-anneal dissipation values are stable over a period of two weeks. Stability under these conditions is further evidenced by the oscillator's 1-1 mode fractional frequency stability of 4×10^{-7} over 12 h, corresponding to a mass change of only <0.004 monolayer (if the material had negligible stiffness and density equaling Si). However, we were only able to maintain a cryogenic vacuum for a few hours with the sample held at room temperature, and were unable to verify the long term stability of the low dissipation state for sample temperatures above 100 K.

Temperature dependent dissipation peaks in single crystal silicon oscillators have been reported by others: in cantilevers at both 130 K and 4 kHz[17] and at 150 K and

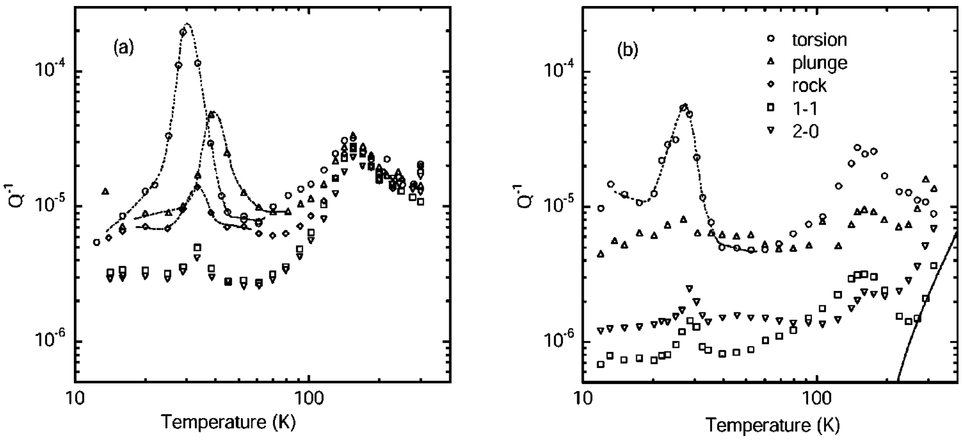

FIG. 4. Dissipation or Q^{-1} is plotted against temperature (a) before and (b) after laser annealing. Postanneal rocking mode data were of poor quality and omitted. For the 1-1 and 2-0 "plate" modes, dissipation near 160 K is reduced tenfold by the anneal. The solid curve in (b) shows the predicted thermoelastic loss for the 2-0 mode near room temperature. Dashed lines are a guide to the eye.

TABLE I. Experimentally measured resonance frequencies at 40 K before annealing f_0, after f_1, and the fractional change, for five resonant modes.

Mode	f_0	f_1	$(f_1-f_0)/f_0$
Torsion	43 810	44 133	0.74%
Plunging	94 619	130 945	38%
Rocking	265 705	265 438	−0.10%
1-1	472 069	471 775	−0.06%
2-0	520 857	535 874	2.9%

11 kHz,[18] and in a geometry similar to ours at 120–200 K and 10 MHz.[19] The dissipation in these experiments varies over about a factor of 30; very roughly, larger dissipations correlate with thinner oscillators. In all these cases, as in our preanneal data, near room temperature dissipation is *decreasing* with rising temperature. This indicates that the loss mechanism dominant at 160 K remains important at room temperature.

Our preanneal data show a similar dissipation for all modes from 160 K up to the highest temperature measured. Below 160 K the dissipation decreases with temperature more quickly for the higher frequency modes. The data are not consistent with the standard Debye model, as the peak temperature is not frequency dependent. One possibility is that there is a phase transition at 160 K associated with a lossy surface layer, while most of the strain energy resides in the bulk Si. Assuming a layer coating the oscillator, with a peak intrinsic dissipation of unity and modulus equal to that of Si, a beam bending approximation to the strain distribution[17,20] gives a thickness of ~ 0.008 nm. Thus a submonolayer sample alteration could produce the peak at 160 K; any corresponding frequency shift would be small in comparison to the observed anneal induced shifts.

The annealed plate modes show a sharp rise in attenuation near room temperature which may be due to thermoelastic loss.[21,22] The solid line in Fig. 4(b) shows predicted thermoelastic loss for the 2-0 mode; predicted loss for the 1-1 mode (off scale) has identical temperature dependence but is 0.08 times smaller. While agreement for the 2-0 mode is fair, we lack a good explanation for the large discrepancy in the 1-1 mode prediction.

Acoustic energy radiated by the oscillator into the substrate through the supports would produce losses showing little temperature dependence. Post-anneal data for the 1-1 and 2-0 is suggestive of a temperature independent floor. Attachment loss[23] calculated for the 2-0 mode is about half the lowest observed value, but the 1-1 mode has a predicted attachment loss orders of magnitude smaller than the measured values.

The preanneal 30 K peak is very pronounced for the torsional and plunging modes, and is small for the plate modes. In sharp contrast to the 160 K feature, the 30 K dissipation maxima vary greatly depending on mode. This peak also appears at different temperatures for different modes (compare the pre-anneal plunging and torsion peaks, or the pre- and post-anneal plate modes), showing no apparent pattern of variation with mode frequency. Peaks appear to shift slightly lower in temperature after annealing. All these facts indicate different mechanisms apply for the 30 K and 160 K peaks.

In conclusion, we have carried out a study of the the effect of laser annealing on the temperature dependent acoustic dissipation of a silicon micromechanical oscillator. Both dissipation peaks seen in Fig. 4 are reduced by the annealing process, suggesting both are related to adsorbates. Previous work[9] found slow increases in room temperature dissipation for samples in air or poor vacuum after rapid thermal anneal; in both that work and the present study, temperatures of at least 1000 K are required to return to the original low dissipation state. These facts are suggestive of SiO_2 formation. In previous work, minimum dissipation (equal to the post anneal dissipation observed in this study) was achieved by rapid thermal annealing 30 s at 1070 K (in N_2 at ambient pressure); lower temperatures were less effective. There is evidence[24,25] such annealing could remove a thin native oxide layer. Water, also a common contaminant on Si surfaces, could play a role; there are reports of phase transitions in water near 160 K.[26] Moreover, several other groups have observed dissipation peaks near 160 K suggesting this may be a generic source of mechanical loss for small Si oscillators exposed to the atmosphere. Further work is required to identify adsorbate species, the details of the surface chemistry, and the associated loss mechanisms.

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