Influence of surface modification on the quality factor of microresonators

O. Ergincan, G. Palasantzas,* and B. J. Kooi

Zernike Institute for Advanced Materials, University of Groningen, 9747 AG Groningen, The Netherlands

(Received 28 February 2012; revised manuscript received 1 May 2012; published 11 May 2012)

Noise measurements were performed to determine the quality factor \( Q \) as a function of gas pressure \( P \) for microresonators in cantilever form with systematically modified surfaces. In the free-molecular regime, which is dominated by internal energy losses, \( Q \) was substantially decreased by more than an order of magnitude with increasing surface roughness. At higher pressures, within the molecular regime, \( Q \) showed the typical inverse linear dependence on pressure \( Q \sim P^{-1} \). However, in the molecular regime the \( Q \) factor also showed a strong dependence on surface morphology as indicated by surface area calculations using measured roughness data and compared to those obtained from \( Q \sim P^{-1} \) plots.

DOI: 10.1103/PhysRevB.85.205420 PACS number(s): 85.35.-t, 74.62.Fj

I. INTRODUCTION

Nowadays there is relentless effort for microelectronics technology to push deep into the submicron range, which inspires the extension of microelectromechanical systems (MEMS) into the nanometer range leading to nanoelectromechanical systems (NEMS).\(^1\)-\(^9\) In this way it is possible to attain extremely high fundamental frequencies in the microwave range,\(^4\),\(^10\) while preserving very high mechanical responsivity with mechanical quality factors \( Q \sim 10^3-10^5 \),\(^4\),\(^11\) active masses of femtograms (\( \sim 10^{-15} \) g),\(^4\) heat capacities far below a yoctocalorie (\( 10^{-18} \) cal),\(^12\) etc. This combination of properties translates into high force and mass sensitivity and the potential to operate at very low power levels.\(^13\)

Despite the enormous progress, a central theme of fundamental and applied research in MEMS/NEMS is the achievement of high \( Q \) factors. The latter is associated with damping and energy dissipation, which measures the ratio of the stored energy \( E_{\text{stor}} \) to the dissipated energy \( E_{\text{dis}} \) (within an oscillation cycle) and it is defined by the relation \( Q = \pi (E_{\text{stor}}/E_{\text{dis}}) \). The larger the value of \( Q \), the higher the sensitivity of the resonance system to external perturbations. The \( Q \) factor determines also the level of fluctuations that degrades the spectral purity of a resonance (linewidth broadening), and determines the minimum intrinsic power at which the device must operate.\(^4\) In any case, the \( Q \) factor of a resonator is determined by the various loss mechanisms and it can be approximated by the relation \( Q^{-1} = \sum_j Q_j^{-1} \). The index \( j \) includes for example attachment loss from gas molecules impinging the resonator, losses due to bulk and surface defects and impurities, thermoelastic losses (thermal currents generated by vibrational volume changes in elastic media with nonzero thermal-expansion coefficient), and losses due to phonon scattering (interaction between oscillatory sound waves and thermal phonons).\(^14\)

In addition a variety of studies have shown that surface roughness influences the quality factor for operation in vacuum.\(^15\)-\(^18\) In Si nanowires with 45 nm beamwidths and 380 MHz resonating frequencies, the quality factor was decreased from \( \sim 3000 \) to 500 by an increment of the surface area to volume ratio from \( \sim 0.02 \) to 0.07.\(^15\) Studies for SiC/Si resonators have shown that devices operational in the UHF/microwave regime had a low surface roughness (\( \sim 2.1 \) nm), while devices with rougher films (up to \( \sim 7.1 \) nm) were operational up to the VHF range.\(^16\) Recently it was shown theoretically that random surface roughness affects the quality factor, limit to mass sensitivity, Allan variance, and dynamic range of resonators.\(^7\),\(^18\)

However, so far a systematic experimental study of the influence of surface roughness on the quality factor of resonators is still missing. This will be the topic of the present paper, where we explore the dependence of the \( Q \) factor on the surface morphology of commercial microcantilevers (Table I) at various gas pressures covering the whole range from the free molecular up to the continuous regime (ambient regime \( \sim 1 \) atm). For our purposes the surfaces of the microcantilevers were systematically modified using ion etching in a focused ion beam (FIB), followed by surface morphology measurements using an atomic force microscope (AFM; Bruker Multimode).

II. EXPERIMENTAL PROCEDURE

The noise measurements for the determination of the \( Q \) factor were performed at room temperature using the thermal or \( Q \)-tuning method\(^3\) and having the AFM head into a high vacuum bell-jar system with controlled pressure from ambient down to \( \sim 10^{-6} \) mbar. The \( Q \)-tuning method, which was developed for the determination of cantilever spring constants \( k \) with accuracy down to \( \sim 5\% \),\(^3\) involves measuring the cantilever’s mechanical response due to agitations of impinging molecules from the surrounding fluid (ambient air, gases at different pressures, or even liquids) and due to thermal dissipation via internal degrees of freedom.\(^3\) The AFM hardware measures the cantilever’s fluctuations as a function of time from which, by Fourier transformation, the frequency-dependent power spectral density \( |A(\omega)| \) (PSD) is obtained. Fitting the PSD spectrum \( |A(\omega)| \) (e.g., Fig. 1) with a Lorentzian form \( |A(\omega)|^2 = A_0/[|\omega^2 - \omega_0^2|^2 + (\omega_0/Q)^2] \),\(^3\) with \( \omega_0 \) the resonance frequency of the free cantilever, and after multiple data averaging of PSD spectra, the averaged \( Q \) factor (both due to intrinsic and fluidic or gas dissipation) is obtained. The noise measurements were repeated eleven times at each pressure to avoid the influence of instantaneous measurement drawbacks such as jitter effect and to confirm repeatability of the measurement. The acquired data were averaged allowing one to obtain the quality factor \( Q \) with accuracy of \( \sim 10\% \). Notably, extensive analysis of cantilever heating effects in both
TABLE I. List of investigated samples and summary of results.

<table>
<thead>
<tr>
<th>No.</th>
<th>( l \times t ) (( \mu \text{m} ))</th>
<th>( \omega_0/2\pi ) (kHz)</th>
<th>( k_b ) (N/m)</th>
<th>( k_a ) (N/m)</th>
<th>( S_{\text{int}}^b ) (10(^{-9}) m(^2))</th>
<th>( S_{\text{total}}^b ) (10(^{-9}) m(^2))</th>
<th>( Q_{\text{int}} )</th>
<th>( Q_{\text{clamp}} )</th>
<th>( Q_{\text{TED}} )</th>
<th>( Q_{\text{surface}} )</th>
<th>( Q_{\text{out}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>131 \times 3.9</td>
<td>343.7</td>
<td>37.3</td>
<td>31.4</td>
<td>2.18</td>
<td>2.29</td>
<td>221 00</td>
<td>338 00</td>
<td>126 646</td>
<td>128 860</td>
<td>118 100</td>
</tr>
<tr>
<td>S2</td>
<td>132 \times 4.0</td>
<td>342.7</td>
<td>38.2</td>
<td>32.3</td>
<td>2.92</td>
<td>2.35</td>
<td>223 00</td>
<td>323 40</td>
<td>123 886</td>
<td>174 600</td>
<td>119 500</td>
</tr>
<tr>
<td>S3</td>
<td>128 \times 4.1</td>
<td>337.1</td>
<td>36.1</td>
<td>37.2</td>
<td>2.02</td>
<td>2.28</td>
<td>149 20</td>
<td>315 00</td>
<td>119 862</td>
<td>372 80</td>
<td>122 340</td>
</tr>
<tr>
<td>S4</td>
<td>131 \times 3.9</td>
<td>343.8</td>
<td>37.3</td>
<td>31.4</td>
<td>2.20</td>
<td>2.34</td>
<td>188 32</td>
<td>338 00</td>
<td>126 631</td>
<td>640 20</td>
<td>118 100</td>
</tr>
<tr>
<td>S5</td>
<td>128 \times 4.1</td>
<td>330.7</td>
<td>34.7</td>
<td>37.2</td>
<td>2.30</td>
<td>2.52</td>
<td>909 00</td>
<td>315 00</td>
<td>122 203</td>
<td>142 70</td>
<td>122 340</td>
</tr>
<tr>
<td>S6</td>
<td>132 \times 3.9</td>
<td>333.7</td>
<td>34.1</td>
<td>28.5</td>
<td>2.42</td>
<td>1495</td>
<td>380 20</td>
<td>133 784</td>
<td>1575</td>
<td>116 540</td>
<td></td>
</tr>
</tbody>
</table>

\(^a\)w is the same for all the samples (\(=38 \mu\text{m}\)).

\(^b\)\(S_{\text{int}}^b\) is calculated from the fit factor \(b\) (with a standard error \(\sim5\%-10\%\)) shown in Fig. 2.

\(^c\)\(S_{\text{total}}^b\) is calculated theoretically using the \(\rho_{\text{air}}\) (S4, \(\sim0.03\); S5, \(\sim0.35\); S6, \(\sim0.11\)) obtained from AFM images. For Si we have \(E = 1.69 \times 10^{11} \text{N/m}^2\), \(\alpha_T = 2.6 \times 10^{-6} \text{m/(m} \cdot\text{K)}\), \(C_p = 1.64 \times 10^4 \text{J/(m}^3 \cdot\text{K)}\), \(\rho = 2330 \text{kg/m}^3\), \(\kappa = 148 \text{W/(m} \cdot\text{K)}\) (Ref. 22).

\(^d\)Calculated subtracting \(\delta E_{\text{sl}}\) and \(\delta E_{\text{clamp}}\) from \(\delta E_{\text{int}}\).

\(^e\)Averaged \(Q_{\text{out}} = (\delta E_{\text{int}})\) calculated via Eq. (4) using the average value \(\langle\delta E_{\text{int}}\rangle\) \(\approx 0.9\).

Air and vacuum resulted in negligible frequency shifts \(\Delta\omega_0 \approx 10^{-10}\text{Hz} \approx 5\%-10\%\) of \(\omega_0\).

The insets of Fig. 1 show the AFM images of nonmodified [Fig. 1(a)] and FIB modified [Fig. 1(b)] cantilever surfaces. The write capability of FIB allows nanofabrication of grids on a specific region of the cantilever without requiring an etch mask. Choosing the area of interest, without changing on a specific region of the cantilever without requiring an etch mask. Choosing the area of interest, without changing the cantilever width and length, the surfaces of the cantilevers are exposed to impinging \(\text{Ga}^+\) ions in the FIB altering the surface morphology in different directions (Fig. 2, over the whole surface area of the resonator with dimensions given in Table I). Moreover, this FIB treatment was performed in such a way that the contamination and defect formation was minimized. Varying the morphology with wavelike modulation parallel or perpendicular to the length of the cantilever affects the reduction of the \(Q\) factor.

III. ENERGY DISSIPATION MECHANISMS

For the noise measurements cantilevers from the same batch were used to gauge and minimize variations of the \(Q_{\text{int}}\) factor due to internal losses (Table I). The latter is given by

\[
\frac{1}{Q_{\text{int}}} = \frac{1}{Q_{\text{TED}}} + \frac{1}{Q_{\text{clamp}}} + \frac{1}{Q_{\text{surface}}},
\]

where \(Q_{\text{TED}}, Q_{\text{clamp}},\) and \(Q_{\text{surface}}\) denote respectively the quality factors due to thermoelastic damping, clamping losses, and surface losses. Energy losses due to clamping \((Q_{\text{clamp}})\) occur because of the strain at the connection with the support base. For cantilevers with semi-infinite base or \(t_b > \lambda_b\),

\[
Q_{\text{clamp}} = 0.95 \frac{l}{w} \frac{i_b^2}{l^2},
\]

with \(w\) and \(l\) the width and length of the cantilever, respectively. Moreover, the \(Q_{\text{TED}}\) due to thermoelastic dissipation (associated with thermal currents generated by vibratory volume changes in elastic media with nonzero thermal-expansion coefficient) is given by

\[
\frac{1}{Q_{\text{TED}}} = \frac{E \alpha_T^2 T}{C_p \rho} \left( \frac{6}{\xi^2} - \frac{6}{\xi^2 \cosh \xi + \cos \xi} \right),
\]

\(E\) is the Young’s modulus, \(\alpha_T\) the thermal expansion coefficient, \(C_p\) the specific heat at constant pressure, \(T\) the system temperature, \(\rho\) the material density of the cantilever.
INFLUENCE OF SURFACE MODIFICATION ON THE . . .

PHYSICAL REVIEW B 85, 205420 (2012)

and \( \zeta = \sqrt{(\text{op} C_p)/2k} \) with \( k \) the thermal conductivity of the cantilever. 21 Finally, we consider the surface losses to originate from a thin surface layer of complex modulus \( E_s \) due to the disruption of the atomic lattice at the surface and due to a thin layer of surface contamination. 5,21 Although this layer does not influence drastically the stored energy in the cantilever, it can enhance energy dissipation, \( Q_{\text{surface}} \), which can be described by

\[
\frac{1}{Q_{\text{surface}}} = \frac{2\delta(3w + t) E_{sl}}{w \tau E}.
\]

with \( \delta \) the thickness of the surface layer and \( E_{sl} = \text{Im}[E_s] \). 5,21 The saturated or free molecular regime in Fig. 2 yields \( Q_{\text{int}} \) allowing calculation of \( Q_{\text{surface}} \) [after subtraction of \( Q_{\text{TED}} \) and \( Q_{\text{clamp}} \) obtained via Eqs. (2) and (3)]. Subsequently Eq. (4) yields the product \( \delta E_{sl} \) having an average value \( \delta E_{sl} \sim 0.9 \) for the unmodified cantilevers (S1→S3; Table I). Although the cantilevers come from the same batch, \( \delta E_{sl} \) varies as \( \sim 0.7–1.1 \) indicating variation of surface dissipation, with \( Q_{\text{surface}} \) deviating significantly from its average value \( Q_{\text{surf}} \).

The calculations are shown in Table I.

In the intrinsic region, it is the subtraction of the thermoe-Lastic and clamping losses from the measured \( Q \) factor of the resonator which actually gives us the surface-related losses of the resonators, shown in Table I as \( Q_{\text{surface}} \). Moreover, once the internal loss is known the molecular parameter in the molecular regime is the surface area variation and not the surface contamination or surface defects (which are of secondary importance). Indeed, the first nonmodified resonator shows excellent agreement between the calculated and measured \( Q \) factors, showing that this procedure is appropriate for comparison with the modified resonators, i.e., to properly distinguish the effect of the surface modification.

Furthermore when operating in a fluid (gas or liquid), mechanical resonators generate a rapidly oscillating flow in the surrounding environment. 23 A transition from Newtonian \((\omega \tau \ll 1)\) to non-Newtonian \((\omega \tau \gg 1)\) flow occurs at \( \omega \tau \approx 1 \) with \( \tau \) a fluid relaxation time \((\sim 1/P)^{24} \) The Newtonian approximation, the basis for the Navier-Stokes equations, breaks down also when the particulate nature of the fluid becomes significant to the flow. It is common to consider the validity of the Newtonian approximation by comparing the mean-free path \( \lambda \) in the medium to an ill-defined characteristic length (the width of the cantilever \( w \)) or using the Knudsen number \( \text{Kn} = \lambda/w \gg 1 \) (Fig. 3). For fluidic dissipation \( Q_{\text{gas}} \) is given by

\[
Q_{\text{gas}} = \frac{M_{\text{eff}} \omega v}{P S_{\text{total}}} \int f(\omega \tau) \frac{1}{f(\omega \tau)} d\omega \tau.
\]

with \( f(\omega \tau) = (1 + \omega^2 \tau^2)^{-3/4} \), \( S_{\text{total}} \) is the smooth surface area of the cantilever, \( v = \sqrt{k_B T/m} \) is the thermal velocity of impinging molecules with mass \( m \), and \( M_{\text{eff}} \) is effective mass of the cantilever that vibrates.

IV. RESULTS AND DISCUSSION

In order to show that the surface area increase governs the development of the \( Q \) factor in the molecular regime we used exactly the same FIB treatment (accelerating voltage, probe current, milling time) to produce the different types of patterns. Therefore, we are able to compare the results without the need to exactly know the individual effects of surface contamination and surface defects. Figure 3 shows that in the molecular regime \( Q_{\text{gas}} \) scales with pressure \( P \) as \( Q_{\text{gas}} \sim 1/P \), while in the continuous regime the scaling changes to \( Q_{\text{gas}} \sim 1/\sqrt{P} \). 24 The transition regime is also shown qualitatively in Fig. 4. In more detail, the total factor \( Q_{\text{total}} \) is given by

\[
\frac{1}{Q_{\text{total}}} = \frac{1}{Q_{\text{int}}} + b P
\]

with \( b = M_{\text{eff}} S_{\text{total}}/v \). The latter yields the total effective surface area \( S_{\text{total}} \) of the cantilever. For consistency an analytical estimation of \( S_{\text{total}} \) was performed. For this we used the rough area calculation

\[
S_{\text{rough}}/S_{\text{smooth}} = \int_0^{+\infty} du \sqrt{1 + \rho_{\text{rms}}^2 u e^{-u}}
\]

with \( \rho_{\text{rms}} \) the average local slope. 27 In all cases \( \rho_{\text{rms}} \) was estimated from AFM profile analysis of cantilever surfaces. Therefore we can define the effective total surface area of the cantilever as \( S_{\text{total}} = \sum_{\text{rough}} + \sum_{\text{smooth}} \). Moreover, since the cantilever’s cross section is trapezoidal the area of side walls is also taken into account for the calculation of \( S_{\text{total}} \).

A crucial parameter for the calculation of the effective area \( S_{\text{total}}^* \) (Table I) via the fitting factor \( b \) is \( M_{\text{eff}} \).
order to cross-check the result, values for $M_{\text{eff}}$ have been obtained both via theoretically and experimentally determined cantilever spring constants. Indeed, the theoretical spring constant for rectangular cantilevers with trapezoidal cross section is given by $k_{\text{eff}} = 3EI/1^3$ (Table I) with $I$ the second moment of inertia.\textsuperscript{28} Alternatively, from $Q$ tuning one obtains the experimental cantilever spring constant $k_{\text{eff}}^b$ (Table I).\textsuperscript{3} Hence from the first resonance frequency of the cantilever (obtained from the maximum of the PSDs as in Fig. 1) $\omega_0 = (k_{\text{eff}}^b/M_{\text{eff}})^{1/2}$, one obtains an $M_{\text{eff}} \approx 23.5\% M_T$ with $M_T$ the total mass of the cantilever.

Alternatively, the total effective surface area, denoted as $S_{\text{total}}$, is also calculated using the average local slope $\rho_{\text{rms}}$ from AFM topography images (e.g., Figs. 1 and 2) and the expression $S_{\text{total}} = \sum S_{\text{smooth}} + \int_0^{\infty} du \sqrt{1 + \rho_{\text{rms}}^2} e^{-u}$ (Table I).\textsuperscript{20} Both results for $S_{\text{total}}$ agree rather well, $S_{\text{total}} \approx S_{\text{total}}$, except for sample S6 for which both top and bottom surfaces were modified. In this case only $S_{\text{total}}$ can be calculated; the estimation of $S_{\text{total}}$ is not feasible, because in this specific case the molecular regime, $Q_{\text{gas}} \sim 1/P$, is absent. The reason for this absence is the large energy dissipation due to intrinsic losses making the cantilever sensitive only to drastic energy losses in the continuum regime. In any case, it can be concluded that the reduction in $Q$ within the molecular regime is associated with an increase in surface area (Table I).

The decrease in $Q_{\text{int}}$ after altering the surface of cantilevers shows a clear change in the dominating energy dissipation mechanism in the free molecular regime which can be due to defect dissipation associated with an effective surface layer\textsuperscript{5,21} and morphology changes. This is also depicted by the change of the PSD noise amplitude in Fig. 1 which is decreased and as we move from the molecular, to transition, and to the continuum regime where it is saturated. In addition, around the same pressure as we cross from the molecular to the continuum regime ($\omega \tau = 1$, Fig. 4), a negative frequency shift $\Delta \omega_{\text{int}}/\omega_0$ occurs indicating mass loading (Fig. 4). Although $\Delta \omega_{\text{int}}/\omega_0$ is small for relatively high frequency cantilevers the actual frequency change $\Delta \omega_0 \sim 1 \text{ kHz}$ is significant. Using the relation $\Delta m \approx 2(m/\omega)\Delta \omega$ one obtains a mass change $\Delta m \sim 200 \text{ pg}$, which is large enough to be taken into account for the calculation of the $Q$ factor in the continuum regime ($\omega \tau \gtrsim 1$ with $\tau = 1850/P^{1/3}$).

Finally, an important result of the present analysis is that the reduction of $Q_{\text{int}}$ due to FIB modification is clearly affected by the type of surface morphology that is created (Fig. 2). Indeed, since both resonators in Fig. 3 have been modified in FIB using the same ion probe current and exposure time (600 pA for 5 min, respectively), one would expect a similar amount of surface contamination and ion implantation. However, when the increased surface roughness corresponds to a wavelike modulation running parallel to the length of the cantilever, the reduction of $Q_{\text{int}}$ is clearly more pronounced than that when the modulation runs perpendicular to the cantilever length (Fig. 2). This difference is clearly a surface modification effect and it is related to the direction of wave propagation and surface scattering during resonation.\textsuperscript{29,30} In fact the quality factor associated with dissipation due to scattering of surface acoustic waves into bulk elastic waves is strongly influenced by the presence of surface roughness.\textsuperscript{29,30}

V. CONCLUSIONS

In conclusion, surface dissipation in the molecular regime is taken into account as a geometrical correction via the surface area calculation where our data provide a clear correlation between increasing surface area and decreasing $Q$ factor. In the intrinsic regime the results indicate that not only surface roughness but also the direction of the surface pattern affects $Q_{\text{int}}$. Etching in an in-plane direction perpendicular to the wave propagation (along the width of the resonator) decreases $Q_{\text{int}}$ less than that in the direction of wave propagation (along the length of the resonator) during resonation. Finally, extensive surface area modification of both faces of a cantilever leads to drastic reduction of $Q_{\text{int}}$ by more than an order of magnitude and thus to high energy dissipation.

Although it is more interesting to improve $Q$ rather than degrade it, improved understanding is one of the key factors enabling progress in this field and ultimately leading to proper $Q$ engineering and improved $Q$ factors. Indeed, our study shows that how you pattern resonator surfaces plays an important role in the $Q$ factor and this has to be taken into account in sensor technologies if surface patterning is necessary.

ACKNOWLEDGMENTS

The authors would like to acknowledge financial support by STW Grant No. 10082, and useful discussions with V. B. Svetovoy.
INFLUENCE OF SURFACE MODIFICATION ON THE...

PHYSICAL REVIEW B 85, 205420 (2012)

16X. M. H. Huang, Ph.D. thesis, California Institute of Technology, 2004, Fig 2.9, p. 36.