Quality Factor in Polycrystalline Diamond Micromechanical Flexural Resonators

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Abstract—This paper reports an investigation into the various dissipation mechanisms that can affect polycrystalline diamond micromechanical resonators. Double-ended tuning fork and cantilever resonators were fabricated from 1–5-μm thick microcrystalline diamond films. It is shown that the quality factor of the low frequency (<500 kHz) resonators is limited by surface loss, whereas the thermoelastic damping limits the quality factor of the higher frequency resonators. In resonators where surface loss is the dominant effect, the dependence of quality factor on resonator thickness is demonstrated. The addition of a lossy surface layer of Al2O3 deposited via atomic layer deposition is shown to degrade quality factor, and an experiment that further demonstrates the effect of surface dissipation and results in a reduction in quality factor that scales with the thickness of the Al2O3 layer. Heat treatment of cantilever resonators in N2 for various times up to 660 min is used to modify the resonator surface and is shown to result in a threefold increase in quality factor up to 365 000 at 26.6 kHz.

Index Terms—Quality factor, micromechanical resonators, diamond.

I. INTRODUCTION

ONE OF the key figures-of-merit in determining the performance of micro- and nano-electromechanical systems (MEMS and NEMS) resonators is their quality factor (Q). Quality factor is particularly important, and has been a subject of great interest, in applications such as oscillators for precision frequency standards [1], [2], filters for signal processing [3] and inertial sensors [4], where high Q-factors are desired.

For resonators operating in a low pressure environment, dissipation mechanisms mainly consist of thermoelastic loss, anchor loss, and surface loss. While extrinsic dissipation mechanisms, such as anchor loss [5], can be minimized by proper geometrical design of a MEMS resonator, intrinsic dissipation mechanisms such as thermoelastic damping (TED) [6]–[8] are strongly material dependent. Hence, in order to minimize dissipation in a micromechanical device, one must carefully consider not only different device geometries but also a proper choice of material. One material that has the potential to produce high Q micromechanical resonators is diamond. Diamond, in its single crystalline form, offers superior properties such as high thermal conductivity (>2000 Wm⁻¹K⁻¹), low thermal expansion coefficient (α ≈ 1.2 ppm°K⁻¹), high melting point, and high acoustic velocity (√E/ρ ≈ 17, 300 m s⁻¹) [9]–[12]. Thermal conductivity is particularly important for TED-limited MEMS resonators. For low frequency operating resonators, where mechanical frequency is smaller than thermal frequency, those composed of a material with higher thermal conductivity can have lower TED and therefore higher Q. Therefore, compared to commonly used materials for MEMS resonators, e.g. silicon, diamond’s high thermal conductivity results in very low TED at a given frequency. Single crystalline diamond (SCD) resonators [13], [14] have shown higher Q than polycrystalline diamond resonators; however, wafer-scale growth of SCD on silicon wafers is expensive and time consuming, making mass manufacturing of SCD resonators problematic. In contrast, polycrystalline diamond films can be deposited on silicon wafers via both microwave and hot filament chemical vapor deposition (HFCVD). Compared to microwave CVD, HFCVD systems are relatively simple, easy to operate, low cost, and produce reasonable diamond film quality at moderate deposition rates. In our case, the deposition rate is 1 nm/min but rates as high as 17-170 nm/min have been reported [15]–[17], as the deposition rate is a strong function of temperature, pressure and the concentration of the CH₄ precursor gas.

Resonator quality factor can suffer because the material properties of polycrystalline diamond are different from those of SCD. In previous work, we studied the connection between quality factor and the thermal conductivity of nanocrystalline diamond (NCD) [18] and microcrystalline diamond (MCD) [19] in resonators where TED is the dominant loss mechanism. The thermal conductivity of MCD and NCD is lower than that of SCD due to a variety of mechanisms including phonon scattering at grain boundaries [20], defect relaxation processes [21], and surface and bulk...
defects [21]–[25]. These dissipation mechanisms are greater in nanocrystalline diamond (NCD) [18] than in microcrystalline diamond (MCD) [9], [26] due to the fact that NCD possesses a higher density of grain boundaries and higher sp²:sp³ carbon bonding ratio, resulting in lower $Q$.

In order to successfully design a high $Q$ micromechanical resonator, it is crucial to know and understand the dissipation mechanisms that can affect the resonator’s operation. Although there has been many researchers investigating the dissipation mechanisms of silicon resonators [27]–[29], similar analysis for diamond films has been scant. Therefore, the innovation of this work is to present a thorough experimental analysis of various damping dissipations, namely anchor loss, TED, and surface loss, that can affect and determine the $Q$ of polycrystalline diamond micromechanical resonators. We further clarify the connection between diamond film properties and these dissipation mechanisms, which has not been focused on in the past. Furthermore, to reduce the surface loss in diamond films, some researchers have suggested annealing SCD resonators in air [14]. The proposed annealing treatment did not have much effect on our polycrystalline diamond films. Alternatively, we propose a step-by-step annealing process using a furnace, to improve the $Q$ of MCD resonators by three-fold to 365,000 exceeding the $Q$ of some of the reported SCD resonators [13]. To enable this study, resonators were fabricated via a simple two-mask surface micromachining process. Two types of resonators were fabricated and tested: cantilevers, having out-of-plane vibration modes, and double-ended tuning fork (DETF) resonators, having in-plane vibration modes. As explained later in detail, DETF resonators are known to have low anchor loss, facilitating the study of TED and surface loss without the influence of anchor loss. Cantilevers were also designed such that the anchor loss is not the limiting factor in these devices. The resonant frequency and quality factor of devices of both types were measured. Devices with resonant frequencies below 500 kHz exhibited $Q$ factors that were independent of the resonant frequency, a characteristic of devices with $Q$ limited by surface loss, while higher frequency resonators exhibited constant $fQ$ product, a characteristic of devices with $Q$ limited by TED. Surface loss is known to depend on the ratio of the resonator thickness to the thickness of a lossy surface layer. In devices where surface loss was the dominant effect, the dependence of $Q$ on the MCD thickness was studied and atomic layer deposition (ALD) was used to add a thin lossy surface layer of Al₂O₃ to demonstrate that $Q$ decreased as predicted by the standard model for surface loss. Finally, resonators were annealed in N₂ to modify the resonator surface, thereby reducing surface loss and increasing $Q$.

II. DEVICE FABRICATION AND TESTING

A. Diamond Film Deposition

Diamond films were deposited under two different process conditions on two different types of wafers. The starting substrates were 150 mm [100] silicon wafers, some of which were coated with a 1 $\mu$m SiO₂ layer as an insulation layer prior to diamond deposition. All the wafers were seeded with 5-50 nm diamond nanoparticles in an ultrasonic bath containing nano-diamond powder dispersed in solvent. The seeding process is a pre-treatment step to reduce the nucleation induction time and increase the nucleation site density in two ways: (a) by creating a growth template through introducing scratches onto the surface and (b) by embedding nano-sized diamond particles to act as seed crystals [15], [17], [30].

MCD deposition was conducted at a pressure of 25 Torr in a large area, multi-wafer HFCVD system (SP3 Model 655 Series) using H₂ and CH₄ as precursor gases. The diamond was in-situ boron doped using trimethylboron (TMB, (CH₃)₃B) at a TMB:CH₄ concentration of 444 ppm. Two different CH₄:H₂ ratios were used, 1% on bare Si wafers and 1.5% on SiO₂-coated wafers. Reducing the CH₄ concentration slows the deposition rate (from 1 nm/min to 2 nm/min) and was observed to produce films with higher thermal conductivity [31]. Fig. 1(a) illustrates a top view of a sample diamond film grown on Si showing the shape and size of the grains, and Fig. 1(b) shows the cross-section of the sample with columnar grains spanning the thickness of the MCD film.

B. Resonator Fabrication

Resonators were fabricated via a two-mask fabrication process. First, plasma-enhanced chemical vapor deposition (PECVD) was used to deposit 1 $\mu$m SiO₂ as a hard mask for MCD etching. The hard mask was lithographically patterned and plasma etched, after which the MCD film was etched using an inductively coupled plasma (ICP) etcher with O₂ and CF₄ at a 50:1 flow rate ratio and a pressure of 30 mTorr. The diamond etch rate was ~250 nm/min with RF power of 1500 W and DC power of 300 W. Ti/Au bond pads were created via a lift-off process with 30 nm Ti and 120 nm Au deposited by sputtering. Resonators having a sacrificial SiO₂ layer were released using a 49% HF solution to remove the SiO₂ layer beneath the resonator tines, after which a brief 2:7:1 HNA (HF:HNO₃:C₂H₄O₂) etch was used to remove a small amount of Si beneath the resonator to avoid stiction.

Some cantilever resonators were fabricated without the sacrificial SiO₂ layer. Here, the MCD was etched using an ICP etcher with O₂ and CHF₃ at 49:3 flow rate ratio with RF power of 600W and DC bias power of 300W at 85 mTorr. The MCD etch rate in this condition was ~130 nm/min. The chosen hard
mask was polysilicon with a thickness of approximately twice the diamond thickness. The cantilevers were then released using deep reactive ion etching (DRIE) on the backside of the wafer to form trenches nearly as deep as the wafer thickness, followed by wet etching in KOH solution (27%, 80 °C) to etch the underlying remaining silicon layer. Some devices were released via XeF$_2$ etching of Si, but this was found to result in ∼40% lower $Q$ than cantilevers released via either HF or KOH etching. This result may be due to fluorine termination of the XeF$_2$-released surface, a surface condition associated with reduced $Q$ in SCD resonators [14].

Fig. 2 shows the SEM image of a single-anchored DETF (SA-DETF) resonator and the resonator’s first in-plane symmetric vibration mode-shape. The DETF resonators were designed with 4.5 μm and 5.3 μm tine widths and tine lengths varying from 70 μm to 359 μm such that their in-plane resonant frequencies spanned a frequency range of ∼1-10 MHz. The out-of-plane resonant frequencies of DETFs and cantilevers spanned 62 kHz-780 kHz. The cantilever resonators fabricated on wafers with no sacrificial oxide, had lengths from 100-700 μm, resulting in natural frequencies spanning a frequency range of 18-140 kHz.

C. Measurement Setup

For measurement of in-plane vibration modes, the resonators were placed inside a vacuum probe station (MMR Technologies, Inc.) with a pressure below 100 μTorr to minimize air damping. The DETFs were excited electrostatically and the response was detected capacitively using an off-chip trans-impedance amplifier (Femto® model DHPCA-100). The $Q$ factor was extracted from the frequency response after subtracting the capacitive feedthrough. A sample frequency response of an in-plane mode SA-DETF is shown in Fig. 3. To measure the out of plane modes, the resonators were placed in a vacuum chamber (Janis Research Company) and were mechanically excited using a piezo-actuator and the responses were measured using a laser Doppler vibrometer (LDV).

The Young’s modulus ($E$) of MCD was extracted from the measured resonant frequency of 8 cantilevers of 8 different lengths. Undercut at the base of the cantilever is an inevitable part of the fabrication, and is generally a source of error that can result in inaccurate estimates for Young’s modulus. In order to obtain the correct modulus value, the undercut was measured from SEM images and was incorporated in an FEM model. The Young’s modulus in the FEM model was varied until the simulated resonant frequency matched the experimentally observed value. This method resulted in an estimated value of $E = 935 \pm 5$ GPa for the MCD film deposited on a 1 μm SiO$_2$ film.

A second approach was adopted from [32] by adding a constant length correction term to the length of each cantilever to account for the increase in length due to the undercut, $L_{\text{effective}} = L_{\text{cantilever}} + L_{\text{corrected}}$. $L_{\text{corrected}}$ was identified by minimizing the error of the least-squares fit to the data, as illustrated in Fig. 4. The length-correction method resulted in an estimated value of $E = 944 \pm 4$ GPa for the MCD film deposited on a 1 μm SiO$_2$ film, in good agreement with the value estimated from FEM. Due to its ease of use and close agreement with the FEM result, only the length-correction method was used to estimate the Young’s modulus for MCD deposited on bare Si wafers, resulting in $E = 987 \pm 7$ GPa. This value
was slightly higher than that of the MCD film deposited on SiO₂, possibly due to having a different deposition condition (CH₄:H₂ ratio of 1.0% for MCD deposited on Si versus 1.5% for MCD deposited on SiO₂). Nonetheless, both values are in very close agreement with previously reported Young’s modulus of polycrystalline diamond films using [26], [33].

### III. RESONATOR LOSS MECHANISMS

For resonators operating in vacuum, the dominant dissipation mechanisms are support loss, thermoelastic damping, and surface loss [34], and the resultant \( Q \)-factor is the parallel combination of individual \( Q \)-s:

\[
Q_{\text{total}}^{-1} = Q_{\text{support}}^{-1} + Q_{\text{TED}}^{-1} + Q_{\text{surface}}^{-1}
\]

In this section, we examine the contribution of each loss mechanism to the experimentally-observed quality factor. The material properties of MCD used in analyzing these loss mechanisms are listed in Table I.

### A. Support Loss

Support loss, also known as anchor loss, is the elastic vibration energy dissipated by transmission through the resonator’s supports. Support loss is strongly geometry dependent and for simple clamped-free or clamped-clamped beams, the support-loss-limited \( Q \) is usually expressed as [28], [35]:

\[
Q_{\text{support}} = D \frac{L^n}{t^n}
\]

where \( L \) is the length, \( t \) is the thickness, \( n \) is an integer (usually \( n = 3 \)), and \( D \) is an experimental participation factor which is reported as \( \sim 2.17 \) for silicon cantilevers [36]. Hao et al. [28] developed an analytical model for beams using 2D elastic wave theory with the assumption that the beams have the same thickness as the semi-infinite support plane. This model accounts for material properties such as Poisson’s ratio and Young’s modulus. Applying this model to the fundamental frequency of MCD beams with material properties listed in Table I, the participation factors are \( D_{\text{C-F}} = 2.353 \) for a cantilever and \( D_{\text{C-C}} = 0.732 \) for a clamped-clamped beam. For cantilevers with lengths of 150 \( \mu \)m to 700 \( \mu \)m, Equation (1) predicts \( Q \) to range from \( 3 \times 10^5 \) to \( 3 \times 10^7 \). These values are higher than the experimentally observed values reported below, indicating that anchor loss is not an important component of the resonator’s \( Q \) factor. Note that the anchor loss in DETF devices is expected to be even lower since the symmetric vibration mode results in energy transfer between the tines rather than into the anchor.

### B. Thermoelastic Damping

Fundamentally, TED is a dissipation mechanism caused by the conversion of elastic strain energy into thermal energy conducted through the resonator material. The simplified \( Q_{\text{TED}} \) model proposed by Zener for flexural resonators is expressed as [6]:

\[
Q_{\text{TED}}(f) = \frac{Q_{\text{min}}}{2} \left( 1 + \frac{(f/f_{\text{min}})}{(f/f_{\text{min}})} \right)^{-2}
\]

where \( Q_{\text{min}} \) is the minimum \( Q \) and \( f_{\text{min}} \) is the frequency at which this occurs,

\[
Q_{\text{min}} = \frac{2C\rho}{E\alpha^2T_0}
\]

\[
f_{\text{min}} = \frac{\kappa}{C\rho} \pi \frac{w}{2w^2}.
\]

In these equations, \( f \) is the mechanical resonant frequency of the resonator, \( C \) is the specific heat capacity, \( \rho \) is density, \( E \) is the Young’s modulus, \( \alpha \) is the thermal expansion coefficient, \( T_0 \) is the resonator’s absolute temperature, \( \kappa \) is the thermal conductivity and \( w \) is the geometrical dimension along which the resonator vibrates (i.e., width in DETF and thickness in cantilever resonators).

To demonstrate the dependence of \( Q \) on frequency, the observed \( Q \) of each cantilever and DETF resonator is plotted against its resonant frequency in Fig. 5. Results are presented for both in-plane and out-of-plane vibration modes. To be able to show the results for resonators with different dimensions on the same graph, the frequencies were normalized by \( f_{\text{min}} \), the frequency at which the minimum \( Q \) occurs. The corresponding values of \( f_{\text{min}} \) for dimensions of \( t = 3.1 \) \( \mu \)m (out-of-plane modes), and \( w_1 = 4.5 \) \( \mu \)m, and \( w_2 = 5.3 \) \( \mu \)m (in-plane modes)

### Table I

**Summary of MCD Film Properties**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific heat capacity</td>
<td>( C )</td>
<td>510</td>
<td>J kg⁻¹ K⁻¹</td>
</tr>
<tr>
<td>Density</td>
<td>( \rho )</td>
<td>3515</td>
<td>kg m⁻³</td>
</tr>
<tr>
<td>Thermal expansion coefficient</td>
<td>( \alpha )</td>
<td>1.2</td>
<td>ppm K⁻¹</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>( E )</td>
<td>944/987</td>
<td>GPa</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>( \nu )</td>
<td>0.057[22]</td>
<td>-</td>
</tr>
</tbody>
</table>

*Value used for single crystalline diamond

**Values for MCD film on SiO₂ and Si, respectively.
are 15.50 MHz, 7.36 MHz, 5.22 MHz, respectively. The measured $Q$ is well-modeled by (2) for $f/f_{\text{min}}$ within the interval of $2 \times 10^{-2}$ to $\sim 1$, while at lower frequencies (indicated by a black box in Fig. 5), $Q$ is frequency-independent and is dominated by surface loss, as described below. The measured $Q$-factors are lower than the theoretical $Q_{\text{TED}}$ above the relative frequency of 1 in Fig. 5, indicating that support loss is higher in these devices, which have relatively short tines.

The thermal conductivity of the diamond film was estimated to be $\sim 185 \text{ Wm}^{-1}\text{K}^{-1}$ from the least-squares fit of (2) to the data shown in Fig. 5. This value is close to the value measured using time-domain thermo-reflectance (TDTR) [37], [38] for MCD deposited using a similar recipe [19]. Both values, however, are about an order of magnitude lower than that of SCD [39].

C. Surface Loss

Surface loss describes the energy loss due to the presence of lattice defects, impurities, adsorbates, and undesired materials on the surface of a resonator [29], [34], [40]. Studies have shown that surface loss can significantly affect the resonator quality factor [24], [40] as the surface-to-volume ratio increases [41]. In spite of its significance, surface loss is not entirely understood and modeling of it has been scant. Following the ideas of Zener and Siegel [7] and Nowick [42], some studies developed models for surface dissipation based on “anelastic” behavior [40], [43], [44]. One attempt to obtain an analytical model of surface loss has resulted in the following equation [40]:

$$Q_{\text{surface}} = \frac{wt}{3w + t} \frac{E^2}{\delta E_S'}$$

(5)

where $E$ is the Young’s modulus of the cantilever material, $\delta$ denotes the thickness of the undesired surface layer, and $E_S'$ is the imaginary part of the surface layer’s Young’s modulus, otherwise known as the dissipative Young’s modulus. Therefore, assuming a fixed resonator geometry and equivalent environmental conditions, one would expect diamond to have lower surface loss than other materials (e.g., silicon) due to diamond’s very high Young’s modulus.

If $t \ll w$, (5) simplifies to

$$Q_{\text{surface}} = \frac{t}{2\delta} \frac{E}{E_S'}$$

(6)

and it becomes apparent that the $Q_{\text{surface}}$ is proportional to the thickness ratio, $t/\delta$. Note that for resonators with fixed thickness and assuming $t \gg \delta$, Equation (6) is frequency independent, and our experimental data in the boxed area in Fig. 5 also exhibits frequency independent behavior.

To quantify this behavior better, we demonstrated the effect of thin surface layers on surface loss by depositing two 12 nm layers of Alumina ($\text{Al}_2\text{O}_3$) on cantilever resonators using atomic layer deposition (ALD). As depicted in Fig. 6, the average and standard deviation of $Q$ for the as-released cantilevers was measured to be 161,000 ± 19,000 over 9 samples. The average $Q$ dropped to 56,000 ± 8,000 after the first 12 nm layer and again down to 26,000 ± 4,000 after the second. The observed twofold reduction in $Q$-factor when the surface Alumina layer was doubled in thickness is in good agreement with (6). The experimental results were used to estimate the loss modulus for Alumina to be $2.44 \pm 0.45 \text{ GPa}$, and the ratio of this loss modulus to the Young’s modulus of MCD was found to be 0.25%. It should be noted that the TED-limited $Q$ of the composite cantilever (i.e., $\text{Al}_2\text{O}_3$ and MCD) is in excess of $10^6$, roughly an orders of magnitude higher than the observed $Q$, confirming that the dominant effect of this layer is to increase surface loss.

Cantilevers fabricated with MCD thicknesses from 0.73 $\mu\text{m}$ to 4.5 $\mu\text{m}$ demonstrate the linear dependence of $Q$ on thickness predicted by Equation (6), as shown in Fig. 7.
Fig. 8. Plot of the maximum measured $Q$ versus thickness along the direction of the resonator’s vibration for the MCD studied in this work. The dashed blue line is to show the trend of increasing $Q$ with increasing thickness for surface loss limited resonators. Inset shows the frequency response of the highest achieved $Q$.

Fig. 9. Experimental data showing a trend of increasing mechanical $Q$-factor with increasing thickness along the direction of the resonator movement. Single crystalline Si resonators are shown in blue diamonds from references [34], [40], [45]–[50], the SCD in red hexagons from references [13], [14], the NCD in cyan triangles from references [22], [25], [51], [52]. The MCD data in green circles include data from this study and references [21], [53]. The dashed red lines are trend lines. The data shown here are the highest achieved $Q$ for flexural resonators at room temperature prior to any kind of material treatment, and are not limited by TED, air damping or anchor loss. To be able to observe this trend more clearly, the maximum $Q$ measured for each thickness is plotted in Fig. 8. The highest $Q$ achieved prior to any material treatment is 292,000 at $f_n = 24$ kHz (inset of Fig. 8). This is the highest reported $Q$ factor to date for flexural resonators made from a polycrystalline material at room temperature.

To compare MCD with other materials, we have plotted the published $Q$-factor versus thickness for resonators fabricated from single crystalline Si (SCS) [34], [40], [45]–[50], SCD [13], [14], MCD (this work and [21], [53]) and NCD [22], [25], [51], [52] in Fig. 9. In this figure, the thickness is the dimension along which the resonator flexes. The figure does not include results for resonators having post-fabrication heat-treatment or surface treatment, such as the ∼100 nm thick SCD cantilever resonators reported in [14], where $Q$ greater than 100,000 was achieved. Resonators whose $Q$ was limited by air damping, anchor loss or TED are also not included in this figure. While all four materials demonstrate similar trends, diamond is observed to achieve a higher $Q$ than Si for the same film thickness. This is particularly noticeable for thicknesses greater than 0.6 μm where the data converge to their corresponding trend lines. The $Q$ factors for SCD resonators are mostly above the MCD trend line while NCD and UNCD resonators are generally below the trend line.

IV. MATERIAL TREATMENT

Treatments such as annealing can reduce the effect of surface loss and increase $Q$ [29], [54]. Thermal treatment experiments were conducted in a N$_2$ furnace under various conditions. The temperature profile was a 30 min ramp-up to 800 °C after which the temperature was held constant for 1 hr. Initial experiments conducted at 100 Torr and 10 Torr showed similar results, with the average $Q$ increasing from 100,000 before heat treatment to 140,000 following the treatment. Heat treating the same sample a second time did not result in further increases in $Q$, and the measured resonant frequencies were changed by less than 1% by the heat treatment process.

A second set of experiments was conducted at a pressure of 1 Torr with no gas flow into the furnace during the temperature ramp-up stage. N$_2$ was introduced after the temperature reached 800 °C and the rest of the 1 hr anneal was performed in N$_2$ at 1 Torr. The $Q$ following thermal treatment was increased by 35%, higher than the results under the previous treatment conditions. Significantly, the measured frequency following thermal treatment was observed to be 3.2% lower than the initial value. Experiments were conducted with the same samples subjected to 11 cycles of treatment under these same conditions and the $Q$ factors of at least twelve cantilevers were measured after each cycle. SEM images taken before and after heat treatment demonstrate that the reduction in frequency is due to erosion of the diamond film. Fig. 10 shows cross-section SEM images of a cantilever after the 7th and 9th heat treatment steps. The thickness is reduced from 2.0 μm to 1.6 μm, in good agreement with the thickness inferred from the frequency shift. One can observe from the
SEM images that the bottom surface of the diamond, which was in contact with the silicon wafer and not exposed during the heat treatment step, has not been etched. We attribute this result to the presence of residual O2 in the furnace during the ramp-up stage. It has been shown that sp2 and sp3 carbon components of polycrystalline diamond can oxidize in presence of oxygen above 400 °C [55].

The Q-factor for each cantilever was extracted from 10 consecutive ring-down time-responses measured using a lock-in amplifier (HF2LI, Zurich Instruments) and the average Q was recorded. A sample ring-down that resulted in the highest Q is shown in Fig. 11. The Q versus frequency shift (Δf/f) and inferred film thickness (t) after each treatment step is plotted in Fig. 12. In this figure, the thickness was estimated based on the observed frequency shift.

The behavior of Q over the heat treatment steps shown in Fig. 12 exhibits three different phases. In the first phase, steps 1 to 4, the Q increases, and the thickness is reduced by 600 nm. In the second phase, the Q is approximately constant. The third phase begins when 1.27 μm of diamond has been removed, and Q begins to decrease. The maximum Q, 365,000, occurs at the end of phase I, and is approximately 3 times the initial, as-fabricated value. The thickness reduction at this point, 600 nm, corresponds well with the observed thickness of the diamond nucleation layer (~300 nm) at the bottom of the diamond film. Similar nucleation thicknesses have been reported in literature for diamond films [56] showing that full coalescence of diamond grains occur after 300 nm of growth. Since this nucleation layer is known to have poor quality, including reduced thermal conductivity [56], smaller grain size, and higher sp2:sp3 ratio, we believe that the increase in Q is primarily due to the removal of this layer. The reduction in Q during phase III is due to an increase in the surface-to-volume ratio, leading to increased surface loss, as was explained in Section III-C.

Surface roughness is often believed to be a potential contributor to surface loss. However, in our heat treatment experiments we observed a very large increase in Q despite an increase in surface roughness. Analysis performed by Shiar and Najafi [43] shows that while surface roughness can increase surface loss, this increase is minimal in materials with low Poisson’s ratio. In materials like gold with ν = 0.44, roughness has a very large effect, while in materials like Silica with ν = 0.17, roughness has almost no effect on Q. Since diamond has a very low Poisson’s ratio with ν = 0.057 [22], we believe this is the reason that Q increased in our samples despite an increase in surface roughness. Draper labs researchers have experimentally shown that changing the surface roughness of MCD films through ion bombardment in hemispherical and cantilever resonators is not effective in increasing Q [57].

V. Conclusion

Dissipation mechanisms in micromechanical resonators fabricated from MCD films were investigated. To enable this study, DETF and cantilever resonators were fabricated from films that were deposited using HFCVD. It was shown that when air damping and anchor loss were minimized, the Q of higher frequency resonators was limited by TED while lower frequency resonators (~500 kHz) had Q-factors that were limited by surface loss. To illustrate the effect of thin coatings on surface loss, 12 nm layers of Alumina were deposited on the resonators’ surface which produced a reduction in Q that was in proportion to the deposited thickness, consistent with the model for surface loss. It was further demonstrated that, in the surface-loss-limited resonators, Q increased as the thickness of the diamond film increased. When compared to published data on low-frequency Si resonators, Q exhibited
a similar dependence on thickness but MCD resonators exhibited higher $Q$ than Si resonators of the same thickness. A step-by-step heat treatment process produced a gradual increase in $Q$-factor, resulting in a maximum increase of approximately $3\times$. The maximum $Q$ achieved after thermal treatment was $365,000$ at $26.60 \text{ kHz}$. Based on the observed reduction in resonance frequency and reduced thickness observed in cross-section SEM images, we attribute the increase in $Q$ to removal of lossy material on the resonator surface. In particular, the diamond nucleation layer is removed, suggesting that this layer may contribute to reduced $Q$.

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