Hemispherical micro-resonators from atomic layer deposition

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Abstract

Hemispherical shell micro-resonators may be used as gyroscopes to potentially enable precision inertial navigation and guidance at low cost and size. Such devices require a high degree of symmetry and large quality factors ($Q$). Fabricating the devices from atomic layer deposition (ALD) facilitates symmetry through ALD’s high conformality and low surface roughness. To maximize $Q$, the shells’ geometry is optimized using finite element method (FEM) studies to reduce thermoelastic dissipation and anchor loss. The shells are fabricated by etching hemispherical molds in Si (1 1 1) substrates with a 2:7:1 volumetric ratio of hydrofluoric:nitric:acetic acids, and conformally coating and patterning the molds with ALD $\text{Al}_2\text{O}_3$. The $\text{Al}_2\text{O}_3$ shells are then released from the surrounding Si substrate with an $\text{SF}_6$ plasma. The resulting shells typically have radii around 50$\mu$m and thicknesses close to 50 nm. The shells are highly symmetric, with radial deviations between 0.22 and 0.49%, and robust enough to be driven on resonance at amplitudes 10 $\times$ their thickness, sufficient to visualize the resonance mode shapes in an SEM. Resonance frequencies are around 60 kHz, with $Q$ values between 1000 and 2000. This $Q$ is lower than the $10^6$ predicted by FEM, implying that $Q$ is being limited by unmodeled sources of energy loss, most likely from surface effects or material defects.

Keywords: hemispherical shell resonator, atomic layer deposition, MEMS, finite element modeling

(Some figures may appear in colour only in the online journal)
and sub-nanometer surface roughness [20, 21]. In contrast to previous HRG studies that have used ALD as a coating layer [11], this work utilizes ALD as the sole device material.

The key performance requirements for the HRG include low resonance frequency mismatch, $\Delta f$, between the degenerate resonance modes (indicating a need for highly symmetric shells) and long resonance decay times, given by $\tau = Q/\pi f_0$ [22] (indicating a need for low resonance frequencies, $f_0$, and high quality factors, $Q$). ALD offers benefits for both of these requirements: for the former, the films’ high conformity and low surface roughness minimize asymmetry due to film variation, while for the latter, the ultrathin thicknesses allow for lower $f_0$ and higher $Q$ (due to the reduced impact of thermoelastic damping (TED)).

The effects of thickness and other geometry variations on $f_0$, $Q$ due to TED, and $Q$ due to anchor loss were modeled with finite element method (FEM) software. Using those modeling results as a guide, hemispherical shell resonators were designed and fabricated with ALD aluminum oxide (Al$_2$O$_3$), and their mechanical resonance properties measured. The resulting shells have radii typically around 50 $\mu$m and thicknesses near 50 nm, as seen in figure 1. Both of these dimensions are one to two orders of magnitude smaller than similar devices [9–13], which have radii ranging from 370 $\mu$m to 2.5 mm and thicknesses from 1 to 10 $\mu$m.

2. Modeling

In order to design the geometry of the wineglass resonators to maximize $Q$ and minimize $f_0$, extensive FEM modeling of the lowest-order wineglass mode ($n = 2$, figure 2) was performed using COMSOL Multiphysics software. Two degenerate modes exist at this frequency, where the standing wave of mode 1 ($d$) is rotated 45° compared to the standing wave of mode 2 ($a$).

To model $Q$, the source of energy loss must be specified. Two of the most common sources of dissipation that wineglass resonators are likely to face are from anchor loss and TED [23]. Other sources of loss can include air damping, which is negligible when operating under vacuum, and surface effects, which are typically difficult to model and must often be measured empirically. To operate as precision gyroscopes, a $Q$ of $10^6$–$10^7$ is expected to be necessary.

$Q$ due to TED ($Q_{\text{TED}}$) was modeled as a function of thickness of the Al$_2$O$_3$ shell, using material properties from the literature (density = 3.0 g cm$^{-3}$, Young’s modulus = 175 GPa, Poisson’s ratio = 0.24 [24], coefficient of thermal expansion = 4.2 ppm °C$^{-1}$ [25], thermal conductivity = 1.5 W m$^{-1}$ K$^{-1}$ [26], and heat capacity at constant pressure (for bulk Al$_2$O$_3$) = 730 J kg$^{-1}$ K$^{-1}$). $Q_{\text{TED}}$ was seen to increase with decreasing thickness, as expected from previous TED studies [22, 27]. The reduced impact of TED demonstrates an advantage of ultrathin ALD materials over thicker conventionally deposited materials. At 150 nm Al$_2$O$_3$ thickness, the model predicts $Q_{\text{TED}} = 1.5 \times 10^6$. Hence, with typical thicknesses around 50 nm, TED will likely be negligible.

Modeling $Q$ due to anchor loss ($Q_{\text{anch}}$) was performed using the perfectly matched layer (PML) method, wherein acoustic energy lost through the anchor is absorbed by the PML, and the ratio of energy stored in the resonator over the energy lost in the PML is proportional to $Q_{\text{anch}}$ [28]. The modeled geometry is shown in figure 3, where the parameters that were varied include shell radius $r$, anchor radius $r_a$, and shell thickness $t$. Anchor height $h$ was also varied, but was found to have no impact on $f_0$ or $Q_{\text{anch}}$. The Al$_2$O$_3$ is meshed as a shell of 6000 mapped quadrilateral elements, the silicon mesh is ~150 000 solid tetrahedral elements, and the PML mesh is generated from sweeping the silicon mesh outwards radially.

The results of the FEM study are shown in figure 4. The shells were modeled as ALD Al$_2$O$_3$ with the same mechanical properties as given for the $Q_{\text{TED}}$ study. The substrate was modeled as isotropic silicon (density = 2.329 g cm$^{-3}$, Young’s modulus = 170 GPa, Poisson’s ratio = 0.28) surrounded by a PML. A standard geometry was chosen, with $r = 50$ $\mu$m, $r_a = 1$ $\mu$m, and $t = 50$ nm. From these initial values, one parameter
was varied at a time. The resulting influences on $f_0$ and $Q_{\text{anch}}$ can be summarized as follows:

- **Anchor radius**: has the strongest influence of any parameter on $f_0$ and $Q_{\text{anch}}$. Increasing $r_a$ from 1 to $>7 \, \mu m$ causes $Q_{\text{anch}}$ to drop from $10^6$ to 100. For $r_a > 5 \, \mu m$, the anchor begins contributing to the resonator’s effective stiffness, increasing $f_0$.

- **Shell radius**: the second leading contributor to $f_0$ and $Q_{\text{anch}}$, where increasing $r_s$ improves performance by decreasing $f_0$ and increasing $Q_{\text{anch}}$. For $r_s > ~50 \, \mu m$, $Q_{\text{anch}}$ rises above $10^6$.

- **Shell thickness**: $f_0$ is directly proportional to $t$, whereas $Q_{\text{anch}}$ displays a very small, nonlinear dependence with a peak at ~50 nm. This peak perhaps arises from the interaction between the thickness-dependent phonon mean free path and relaxation time, analogous to the thickness-dependent peak in $Q$ predicted by Kunal and Aluru [29] due to Akhiezer damping.

Because simplifications have been taken in the construction of this model, it is important to note that this computational analysis should not be used for quantitative $Q_{\text{anch}}$ predictions. Expecting that the qualitative trends are still valid, the computational analysis provides qualitative design rules regarding the variables discussed above. The general design rules for achieving the highest possible $Q$ are to minimize the anchor radius and maximize the shell radius. From these modeling results, the targeted geometric parameters for fabrication are chosen to be $t = 50 \, \text{nm}$, $r_s > 50 \, \mu m$, and $r_a < 1 \, \mu m$. For these parameters, FEM predicted both $Q_{\text{anch}}$ and $Q_{\text{TED}}$ to be $> 10^6$, with the caveats noted above.

### 3. Fabrication

The fabrication process flow for the wineglass resonators is provided in figure 5. A Cr/Au (20 nm/180 nm) mask is patterned with circular openings over a silicon substrate (a). Hemispherical molds are etched in the substrate, either with SF6 plasma or HNA (hydrofluoric, nitric, and acetic acids) (b). The wafer is coated with ALD Al2O3 (c). A thick photoresist is spun on and etched back, planarizing the molds. The Al2O3 is etched with buffered HF, leaving only the Al2O3 protected by photoresist in the molds (d). Finally, the silicon is etched back in an SF6 plasma, leaving the ALD shell supported by a silicon stem (e). This fabrication can be summarized in three principal steps: (1) mold fabrication, (2) ALD shell definition, and (3) shell release.

#### 3.1. Mold fabrication

The hemispherical mold is critical importance to device performance, as the conformal nature of ALD means the shell will reproduce the mold shape with high fidelity. Hence, the mold must be smooth and symmetric to avoid any anisotropy that would increase the resonance frequency mismatch between degenerate resonance modes. As seen in figure 6, the symmetry and roughness of the molds are strongly dependent on the composition of the etchant and the etch conditions.

Initially, the molds were etched with an SF6 plasma. While changing the etching parameters (including gas flow rate,
pressure, plasma power, and mask material) could change the isotropy and surface roughness of the mold, no set of parameters was found to provide adequate results. The resulting molds displayed crystal orientation dependence associated with the Si substrate’s crystal planes, with Si (100) wafers producing square molds and Si (111) wafers producing hexagonal molds, as seen in (a) and (b) of figure 6.

With SF₆ discarded as a suitable etchant, HNA was chosen as a potential replacement. Schwartz and Robbins [30] showed that the isotropy and surface roughness of the HNA etch is highly dependent on the volume ratio of the three acids. Following the recommendation of [30] and Hamzah et al [31], a volumetric ratio of 2:7:1 hydrofluoric:nitric:acetic acids was chosen. The solution temperature and sample agitation also impact the resulting etched profile. Figure 6(c) shows the results of an HNA etch with an uncontrolled temperature starting from room temperature and no agitation, causing rim discoloration and noticeable crystal orientation dependence. After experimenting with temperature and agitation, figure 6(d) shows the final optimized recipe, performed at 50°C with light, uniform agitation, displaying no obvious crystal orientation dependence and low surface roughness.

To quantify the symmetry of the molds, a circular fitting algorithm was developed. First, the overhead SEM image of the mold is run through Mathematica’s edge detection function. Then the resulting edge is fit to a circle and the radial deviation extracted (figure 7). Because the mold is likely tilted slightly from normal in the SEM image, either due to tilt of the sample itself or of the electron beam, the fit may be to an ellipse instead of a circle to remove the tilt-related distortion.

Figure 5. Fabrication process flow.

Figure 6. (a) SF₆-etched mold in Si (100). (b) SF₆-etched mold in Si (111). (c) HNA-etched mold in Si (111) at room temperature with no agitation. (d) HNA-etched mold in Si (111) at 50°C with light agitation.

Figure 7. (a) Overhead SEM micrograph of a hemispherical mold with an overlaid fit to the mold’s edge. (b) Radial deviation of the fit assuming a circular mold (black line) or elliptical mold (red line). The elliptical fit is intended to remove the error introduced by any potential tilt of the overhead image, and produces a lower root mean square (RMS) deviation.
An example of both circular and elliptical fits for a given mold can be seen in figure 7(b). The elliptical fit (red line) provides a lower limit to the radial error of 0.13 $\mu$m, while the circular fit (black line) provides an upper limit of 0.28 $\mu$m. Thus, given a radius of 57.4 $\mu$m, the radius is uniform to within 0.22–0.49%, indicating a highly symmetric mold. Additionally, the elliptical fit reveals the threefold symmetry associated with the Si (1 1 1) crystal planes [8], indicating that the HNA etch still has some slight crystal orientation dependence and could be optimized further.

3.2. ALD shell definition

After mold fabrication, the next task is to deposit and pattern the ALD Al$_2$O$_3$, as shown in figures 5(c) and (d). The Al$_2$O$_3$ is deposited at 130 °C using trimethylaluminum and water as precursors, in a procedure described elsewhere [20]. To pattern the ALD film, a thick photoresist is spun on the wafers and etched back in an O$_2$ plasma until the molds are planarized. The Al$_2$O$_3$ is then etched from the substrate while the molds are protected by the resist. After the Al$_2$O$_3$ etch, the resist is removed to leave suspended ALD shells.

The resist must be well-planarized to prevent exposing any part of the Al$_2$O$_3$ shell to the Al$_2$O$_3$ etch. The type of resist, as well as its spin speed conditions, heavily influences the quality of planarization. The best planarization was provided by 3–5 repetitions of the spin and etch-back process, using AZ-9260 resist nominally spun at a thickness of ~10 $\mu$m. Fewer coatings of a thicker resist resulted in problems with resist bubbling, while more coatings of a thinner resist caused extensive cracking due to the repeated thermal cycling. Larger molds also experience more problems with resist bubbling, limiting the current maximum mold radius to 50–60 $\mu$m. Larger shells will require either a further-optimized planarization process or an alternate etch technique (such as chemical mechanical polishing).

The resist etch-back conditions also play a large role in the quality of the planarization. When etching the photoresist with an O$_2$ plasma at pressures ranging from 10 mTorr to 1 Torr, the photoresist does not etch uniformly or cleanly, resulting in hardened resist debris and an uneven thickness, as seen in figure 8(a). By switching to a barrel asher operating at 10 Torr and an elevated temperature (up to 200 °C), no residue remained and the height of the resist stayed constant, as in figure 8(b). With the molds properly protected, the uncovered ALD material is etched using a buffered HF solution.

3.3. Shell release

The final step of the wineglass fabrication is to release the shell from the surrounding substrate, defining the wineglass anchor. The anchor radius $r_a$ largely determines $Q_{\text{anch}}$ and, if too large, can limit $Q$ to ~100. Fortunately, this fabrication scheme provides accurate control over $r_a$.

The anchor control is provided by a timed Si etch, using an SF$_6$ inductively coupled plasma (ICP) with the platen power set to 0W to reduce physical ablation of the Al$_2$O$_3$. Initially, the Si etch is timed for an approximate target anchor radius. Because the Al$_2$O$_3$ is transparent, imaging the shells from overhead with an optical microscope clearly reveals the width of the anchor under the shell. If the anchor is not yet to the desired size, the wafer can be re-etched with shorter and shorter time intervals. Using this technique, anchors with radii between 0.5 and 1 $\mu$m can consistently be fabricated, as seen in figure 9. The anchors exhibit triangular or hexagonal...
cross-sections indicative of the Si (1 1 1) crystal planes, due to the crystal-orientation-dependent SF$_6$ etch.

4. Measurement

After the shells were fabricated, their resonance characteristics were measured via an optical reflection technique. A schematic of the measurement setup is provided in figure 10. Briefly, the shells are driven into resonance by mechanical coupling to a lead–zirconate–titanate (PZT) piezoelectric shaker driven by broadband white noise. A laser diode (670 nm) reflects off of the shell at grazing incidence (~80° from normal) and is measured by a photodetector. The resulting signal is amplified and processed with a Fourier transform by a spectrum analyzer. All data is taken under vacuum at 10$^{-5}$ Torr and at room temperature.

Figure 11 shows two example measurements from the same shell, before and after its anchor is thinned from $r_a = 7$ to 3 $\mu$m. The Lorentzian fit to the thick-anchor data reveals $f_0 = 68.71$ kHz and $Q = 230$, while the thin-anchor data shows $f_0 = 58.54$ kHz and $Q = 1270$. The improvement in $Q$ implies that as the anchor is thinned, the impact of anchor loss is reduced, in agreement with the trend from the FEM model in figure 4. Subsequently, $r_a$ was thinned again, from 3 to 0.6 $\mu$m, with no corresponding improvement in $Q$. The lack of improvement implies that anchor loss is no longer the dominant contributing factor to $Q$. Some possible alternate dissipation mechanisms are proposed in section 5, below.

Resonance frequency is also a function of anchor width, and there is a discrepancy in the modeled versus measured $f_0$. For $r_a = 7$ $\mu$m, the FEM model predicts $f_0 = 62$ kHz, 10% lower than the measured 68.71 kHz, while for $r_a = 3$ $\mu$m, the error increases to 25% (44 versus 58.54 kHz). The most likely explanation for this discrepancy is residual stress: ALD Al$_2$O$_3$ is known to have a relatively high residual stress, around 400 MPa when grown at 130°C [24], which can shift $f_0$ substantially from the unstressed case. For the case of the shell in figure 11, the closest match to the resonances of both anchor widths was provided by including 120 MPa residual stress in the FEM model, for which the predicted frequencies are 73 kHz and 55 kHz for $r_a = 7$ $\mu$m and 3 $\mu$m, respectively, improving the error to 6% in both cases (down from 10 and 25%). The 120 MPa stress is lower than the expected 400 MPa, and may be an indication of stress relaxation in the suspended shell.

The preceding FEM analysis assumes the measured mode is the $n = 2$ wineglass mode. To verify whether the measured resonance modes are the expected lowest-order wineglass modes (and not, for example, the $n = 3$ mode or the $n = 1$ rocking mode), the resonance mode shapes were imaged inside an SEM. For this measurement, the shells are again driven with a PZT shaker, as in the optical technique, but instead of driving...
with white noise, the PZT is driven at a coherent frequency. This frequency is scanned through $f_0$; upon resonance, the shell’s rim blurs and reveals the corresponding mode shape. Figure 12 shows an example of the visible mode shape of the same shell as seen in figure 11(b). The first mode occurs at 58.3 kHz, very near the 58.5 kHz measured optically, and the $n = 2$ mode shape (figure 2) is plainly visible, with nodes and antinodes evenly spaced every 45°. Further, the second (nominally degenerate) mode appears at 59.5 kHz—a mode not seen in the optical data, likely obscured by noise—with its standing wave rotated 45° compared to the first mode. The periodic distortion is likely due to aliasing between the shell’s vibration and the scan rate of the SEM. This SEM technique confirms that the measured resonances are the two $n = 2$ wine-glass modes.

It is worth noting that while the 1.2 kHz split between the two degenerate modes is relatively large, it is not necessarily accurate. As a consequence of measuring inside an SEM, the shell is undergoing competing effects of mass-loading-induced softening (from electron beam induced deposition [32]) and electrostatic stiffening, and as such, its resonance frequencies are asymmetrically drifting. Thus, the optical technique is required to measure accurate $\Delta f$ values. Figure 13 shows an example of the mode splitting of a different device measured optically, showing $\Delta f = 100$ Hz, corresponding to a relative split of 0.3%. This split is likely due to a combination of the measured asymmetry in the mold geometry, fabrication-induced defects in the Al$_2$O$_3$ (causing, for example, local variations in Young’s modulus), and surface contamination (causing local variations in density).

This SEM technique reveals that the Al$_2$O$_3$ shells are robust enough that they may be driven to an amplitude of >500 nm, or more than 10 × the thickness of the device itself, without tearing or dislodging from their anchors. An approximate $Q$ can be extracted from the frequencies at which the amplitude reaches half its maximum. This shell displayed an approximate full width at half maximum of ~50 Hz, corresponding to a $Q$ of ~1200, which is in good agreement with the 1270 measured optically in figure 11(b).

This agreement in $Q$ implies that, even with such a large displacement amplitude, the resonator is not being over-driven to the point of causing nonlinear broadening. To ensure the optical measurement is also not broadened nonlinearly, $Q$ was measured at different drive voltages and was consistent. Furthermore, the drive for the optical measurement is two to three orders of magnitude lower than the drive in the SEM. This lower drive implies that the displacement amplitudes for the optical data should be between 0.5 and 5 nm, presumably well within the linear regime for a 50 nm thick device. In total, five different shells were measured optically, each with $Q$ values between 1000 and 2000.

5. Discussion
The measured $Q$s of 1000–2000 are well below the modeled $Q_{\text{anch}}$ and $Q_{\text{TED}}$ predictions of $10^6$. This discrepancy implies that anchor loss and TED are not the principal energy dissipation mechanisms in these shells, although further refinement of the FEM accuracy is needed to more definitively discount these loss mechanisms. The next most likely sources of energy dissipation are from defects and surface effects. Defects cause loss either through defect motion [33] or acoustic scattering [34]. The fabrication process might be
inducing defects in the shells that reduce $Q$, an effect that has previously been seen on torsional silicon micro-resonators [35]. The damage could occur either during the Al$_2$O$_3$ buffered HF etch or the Si SF$_6$ release etch. For example, HF is known to diffuse through photoresist and so might attack the masked shell, particularly through cracks in the resist, while the Si SF$_6$ release etch might cause some amount of physical ablation despite the absence of platen power. These defects could potentially be reduced or eliminated by modifying the fabrication process, for example, by changing the Al$_2$O$_3$ etch from buffered HF to ion milling or chemical mechanical polishing, and changing the Si release etch from an SF$_6$ plasma to a XeF$_2$ gas.

The other likely source of energy dissipation is from surface effects. As the thickness of a resonator decreases, its surface-to-volume ratio increases, increasing the contribution of surface effects on total energy loss relative to volume effects. For example, surfaces contribute additional phonon scattering mechanisms, resulting in a decrease in $Q$ [29]. This effect could be investigated by fabricating shells of different thicknesses: as thickness increases surface effects become less important, meaning $Q$ should increase if the surface is dominating energy loss.

6. Conclusions

ALD wineglass micro-resonators were successfully fabricated by first etching hemispherical molds in Si (1 1 1) with HNA, then conformally coating and patterning the molds with ALD Al$_2$O$_3$, and finally releasing the Al$_2$O$_3$ shells from the surrounding substrate. Radii typically were ~50 µm with thicknesses of ~50 nm. The shells are supported by Si stems that were consistently fabricated with radii around 0.5 µm. Because of the high conformality of ALD, this fabrication process is extendable to applications with arbitrarily shaped 3D molds.

The shells’ resonance properties were measured through an optical reflection technique, revealing typical resonance frequencies around 60 kHz, with quality factors between 1000 and 2000. An SEM imaging technique revealed the shells’ resonance mode shapes by driving the resonances at an amplitude 10 times their thickness, without causing any structural damage. Finite element modeling predicted that anchor loss and thermoelastic dissipation would lead to quality factors over $10^8$. The measured $Q$ of only $10^5$ suggests that alternative energy dissipation mechanisms are dominating. The most likely sources of dissipation are from defects and surface effects, which could be investigated further by modifying the fabrication process to reduce defects and increasing ALD thickness to reduce surface effects. Such studies will be important to achieve the high $Q$ ($10^6–10^7$) necessary for utilizing hemispherical micro-resonators as high-precision gyroscopes.

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