WINEGLASS-ON-A-CHIP

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ABSTRACT

This paper introduces free-standing, stem-supported silicon dioxide hemispherical shells (μ -wineglasses) that are thermally-grown and have high-quality-factor resonances due to unrestricted support stem diameter and low internal thermoelastic damping. The fabrication process offers a direct path to batch fabrication of hemispherical wineglass microstructures with ultra-thin conformal conductive coatings using atomic layer deposition (ALD). A novel assembly method forms capacitive electrodes for rapid electrical characterization of the silicon dioxide μ -wineglass resonators. A quality factor of 5,600 is measured for the $m=4$ resonance of a 740 μ m diameter, 2 μ m thick thermal oxide shell with 30 nm ALD TiN coating and 77 μ m diameter silicon support stem.

INTRODUCTION

Three-dimensional microstructures hold great potential for a multitude of applications. An example of a desirable and truly 3D structure is a hemispherical wineglass (Figure 1). Such μ -wineglasses can yield very high mechanical quality factors at low frequencies (low kHz) due to their thin flexural shell and highly-balanced symmetry, all within a small die area. However, difficulty in fabricating free-standing, stem-supported wineglass hemispherical shells with capacitive transduction electrodes has prevented thorough mechanical characterization of these structures. This paper introduces thermally-grown silicon dioxide hemispherical shells released from microfabricated single-crystal silicon molds, which are subsequently blanketed with an ultra-thin atomic layer deposition (ALD) conductive coating for electrical actuation and sensing. The nearly ideal symmetry of the shells confines the vibration energy near the shell rim, minimizing acoustic radiation through the support into the substrate. Furthermore, the low coefficient of thermal expansion of thermally-grown oxide results in reduced internal thermoelastic damping [1]. The oxide wineglass-on-a-chip resonators share these advantageous features with ultra-high-performance macroscale hemispherical resonator gyroscopes (HRGs) [2].

Several reports have been made in literature on different approaches for fabricating macroscale hemispherical shells. PMMA and boron-doped silicon shells were first fabricated in 1979 for thermonuclear fusion research [3]. Two years later, shells made from gold or oxide were fabricated by a similar technique [4]. However, these shells were fully detached from the substrate, and the resonance characteristics of these shells were not measured. A blow-molding method based on thermoplastic forming of bulk metallic glass has been used to fabricate 3D micro shells [5]. UCI has recently reported ‘inverted’ Pyrex wineglass structures fabricated by wafer-level glassblowing. The inverted Pyrex hemispherical structures are mechanically supported at the rim, limiting their use as high- Q resonators [6]. The same group has demonstrated stem-supported hemispherical shells through laser-cutting of individual glass spheres [7]. However, laser-cutting is a serial process that is difficult to implement and control at wafer-level. Furthermore, the stem support diameter is determined by the opening size in the silicon stencil wafer, which must be large to create large diameter shells.

Our group recently reported polysilicon hemispherical shell resonators with integrated capacitive transducers for electrical operation [1]. The isolated electrodes are created by boron-doping of n-type silicon wafers, forming PN junctions for isolation. The

Figure 1: Perspective view of a fully-released 2 μ m thick, 740 μ m diameter, TiN-coated thermally-grown silicon dioxide μ -wineglass with small support (77 μ m diameter).

capacitive gap is defined by the sacrificial oxide layer, whose thickness is limited to a few microns. In this work, a novel assembly method forms electrodes with large capacitive gaps surrounding the shell for rapid characterization of the oxide shell resonances, enabling full electrical characterization of macroscale stem-supported hemispherical shells.

MATERIAL SELECTION

Various energy dissipation mechanisms limit resonator quality factor. Low frequency resonators operate in the Akhiezer regime, so the limit of Q is inversely proportional to frequency [8]. However, factors such as surface roughness, support loss, and thermoelastic damping (TED) may dominate the energy dissipation, leading to lower Q than the Akhiezer limit predicts [1]. Surface roughness is ultimately determined by the smoothness of the mold and support loss can be suppressed by anchor design. TED can be alleviated through material selection.

To minimize TED, which originates from the coupling of the stress and thermal fields, a material with a low coefficient of thermal expansion (CTE) over the operating temperature range is desired. For example, quality factors greater than 25 million have been achieved, at the macroscale, using fused quartz as the structural material for the HRG [2]. Table 1 compares fused quartz, thermally-grown oxide, and titania silicate (ultra-low expansion—ULE) glass. Thermal oxide has a similar value of CTE to fused quartz and is very stable over a large temperature range. It can be noted that titania silicate glass show a very low CTE (~ 60 ppb/ $^{\circ}$ C) near room temperature. However, this ramps up rather quickly at 40 $^{\circ}$ C and will reach 7 ppm/ $^{\circ}$ C at 95 $^{\circ}$ C [9]. Therefore, thermal oxide was selected as the μ -wineglass structural material for low TED.

Table 1. CTEs of different materials.

Material	CTE @ 25 $^{\circ}$ C (ppm/ $^{\circ}$ C)	CTE @ 95 $^{\circ}$ C (ppm/ $^{\circ}$ C)
Fused quartz [10]	0.45	0.58
Silicon dioxide (thermal) [11]	0.5	0.8
Titania silicate glass [9] (Ultra low expansion glass)	0.06	7

Figure 2 (color online): Fabrication and assembly process flow of μ -wineglass: (a) etch back side blind holes for electrode assembly; (b) pattern front side PECVD oxide mask; (c) isotropically etch silicon mold in SF_6 plasma and remove oxide mask; (d) grow thermal oxide; (e) remove oxide on top side and back side while photoresist is protecting oxide in mold; (f) release μ -wineglass by XeF_2 ; (g) atomic layer deposition (ALD) conformal coating of titanium nitride (TiN) layer; (h) etch electrode pillars on silicon-on-insulator (SOI) wafer; (i) assembly of two dies with adhesive in between (not shown); (j) three-dimensional view of assembled μ -wineglass.

FABRICATION AND ASSEMBLY PROCESS

The silicon hemispherical molding technique introduced in [1] has been tailored for thermal growth of silicon dioxide shells. Figure 2 shows the wafer-level batch-fabrication process for oxide shells and the electrode assembly. Blind holes around the shell are first etched from back side of the wafer using the Bosch process. Later in the process, these blind holes will become through holes for electrode assembly. A $7\ \mu\text{m}$ thick PECVD oxide layer is deposited on the front side at 300°C . A circular opening is then etched into the PECVD oxide mask by a front side to back side alignment. Silicon is isotropically etched by SF_6 plasma to create a symmetric hemispherical mold. After the PECVD oxide mask is removed in hydrofluoric acid (HF), oxide is thermally grown at 1100°C using a wet oxidation method and reaches a final thickness of $3.3\ \mu\text{m}$. Due to the diffusion limited nature of thermal oxidation, oxide is very conformal to the shape of the silicon hemispherical mold. The thermally-grown oxide on the wafer surface is removed in C_4F_8 plasma while the hemispherical mold is protected by photoresist. The remaining photoresist is removed in Piranha, leaving only oxide in the hemispherical mold. The oxide shell is released by etching the surrounding silicon in XeF_2 . Figure 3 shows an optical image of an array of released oxide shells.

To enable electrical testing, the shells must be coated with a conformal conductive layer that electrically connects the shell and the supporting substrate, providing a path for polarization voltage.

Compared to evaporation and sputtering, atomic layer deposition (ALD) provides a higher quality film and a more uniform conformal coating. $30\ \text{nm}$ of titanium nitride (TiN) is deposited at 250°C , conformally coating the sharp transition between the oxide shell and the silicon stem. A resistance of $800\ \Omega$ was measured between the TiN-coated shell and substrate, confirming that this layer provides sufficient electrical connection.

Electrode pillars are etched by high aspect ratio silicon etcher on silicon-on-insulator (SOI) wafers using the Bosch process to ensure electrical isolation between electrodes. Finally, the shells and electrodes are assembled under microscope. Adhesive is dispensed and hard-baked between the two wafers to secure them together. Figure 4 shows a bird's eye view of an assembled device with electrodes for testing.

Figure 3: Wafer-level fabrication of μ -wineglass (field of view: $2\ \text{cm}$ by $2\ \text{cm}$).

Figure 4: Optical image of assembled oxide μ -wineglass with electrode pillars for electrical testing. The μ -wineglass is coated with $30\ \text{nm}$ TiN by atomic layer deposition (ALD).

PROCESS CHARACTERIZATION

Axial symmetry is an important geometric requirement of μ -wineglass structures. A software tool was developed for SEM image analysis to characterize the circular symmetry of the fabricated shells [1]. For the example shell with equatorial radius of $552.5\ \mu\text{m}$ shown in Figure 5a, a radial standard deviation smaller than 0.61% is reported (Fig. 5b), demonstrating that the shells are highly balanced. One uncertainty in this analysis is tilting of SEM stage and the mounting of the sample. If the sample is not mounted perpendicular to the electron gun, the rim of the shell will appear elliptical, resulting in systematic measurement error.

(a)

(b)

Figure 5: (a) least squares fit to the top view SEM image of μ -wineglass; (b) radial error of μ -wineglass results with a standard deviation of 0.61% ($3.37\ \mu\text{m}$ in diameter of $1105\ \mu\text{m}$).

Figure 6 presents additional details of the assembled μ -wineglass with electrodes. Although $3.3\ \mu\text{m}$ oxide was initially grown in the silicon mold, only $1.6\ \mu\text{m}$ is left at the rim after the XeF_2 release (Fig. 6a). The etching of oxide in XeF_2 , which is known as a high selectivity silicon etchant, is not negligible during silicon etching to this depth. The shell thickness is also found to vary along the depth of shell. During XeF_2 release, the top part of the shell is released first and exposed to XeF_2 from two sides. Thus, more oxide is consumed at the rim than at the unexposed

base. By cleaving the released shell, thickness variation along the depth of shell could be measured under SEM (Fig. 6b). The thickness smoothly tapers from $1.6\ \mu\text{m}$ at the rim to $2.2\ \mu\text{m}$ close to the base. Thickness variation in the depth direction is tolerable since it has no impact on the axial symmetry of the shell. The shell rim is also found to have a reentrant extension with projected dimension of $23\ \mu\text{m}$ near its rim (Fig. 6a). This is due to the nature of the isotropic mold formation, since SF_6 cannot etch laterally to the oxide mask opening.

(a)

(b)

Figure 6: Detailed geometric characterization of assembled μ -wineglass: (a) (top view) shell thickness of $1.6\ \mu\text{m}$ and reentrant projection of $23\ \mu\text{m}$; (b) thickness variation along the depth of the shell (measured by SEM), showing measured shell thickness at different locations schematically.

Figure 7: Top-view optical microscope image of octagonal-shaped support ($77\ \mu\text{m}$ support diameter). Substrate roughness created by XeF_2 release is visible through the transparent shell.

Figure 8: Capacitive gap of 29.4 μm between μ -wineglass and excitation electrode, giving efficient electrostatic transduction.

Figure 9: Electrical testing result of μ -wineglass (a) resonance peak of wineglass mode ($m=4$) at 113.9 kHz with quality factor of $\sim 5,600$; (b) COMSOL FEA modal analysis showing simulated resonance frequency of 110.1 kHz.

The support diameter is an important geometric parameter of the μ -wineglass. It has a strong effect on the resonance frequency and may allow support loss to limit the quality factor. Figure 7 shows an optical microscope image of the support structure. Interestingly, although XeF_2 is well-known for etching silicon isotropically, the etching does show some crystalline preference evidenced by an octagonal-shaped support with 77 μm diameter. Due to the transparency of the oxide shell, the substrate surface roughness that is formed during XeF_2 release is visible in Figure 7.

Figure 8 shows the 29.4 μm capacitive gap between the μ -wineglass structure and electrode pillar. A smaller gap will give better electrical transduction, thus lowering the motional impedance, while a larger gap can allow large drive amplitude.

TESTING RESULTS

The 740 μm device was tested in vacuum as a one-port-resonator. An Agilent 4395A network analyzer supplies AC drive voltage to one electrode pillar and a sense current is generated by the change in capacitance across the polarized gaps due to vibration of the shell. A trans-impedance amplifier (TIA) is used to amplify the signal from μ -wineglass. Using the electrical testing platform, we were able to excite the $m=4$ resonance mode at 113.9 kHz with a quality factor of 5,600 (Fig. 9a). Eigenfrequency analysis by COMSOL FEA software predicts a resonance frequency of 110.1 kHz (Fig. 9b), which is in good agreement with the measured frequency.

CONCLUSION

A wafer-level fabrication method was developed for highly-symmetric thermally-grown oxide μ -wineglasses. An ALD TiN conductive layer was able to establish sufficient electrical connection between the μ -wineglass and the substrate. A novel assembly process was developed for the electrical testing platform. The $m=4$ resonance of assembled μ -wineglass was measured in vacuum, with a Q of 5,600 at 113.9 kHz, demonstrating the strong potential of these devices for resonator applications.

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