DIAMOND HEMISPHERICAL RESONATOR FABRICATION BY ISOTROPIC GLASS ETCH

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ABSTRACT

We have fabricated polycrystalline diamond hemispheres by Hot-Filament CVD (HFCVD) in spherical cavities wet-etched into a novel high temperature glass substrate. These resonators are 1.4 mm in diameter and have a Q of up to 143,000 in the fundamental wineglass mode, for a ringdown time of 2.4 seconds. As-fabricated resonators have the two degenerate modes frequency matched to as close as 7 Hz, or 0.04% of the resonant frequency (~16 kHz). We have demonstrated laser trimming on a segmented lip resulting in mode matching to better than 0.3 Hz. These resonators show great promise for use as Hemispherical Resonant Gyroscopes (HRGs) on a chip.

INTRODUCTION

There have been many types of gyroscopes successfully miniaturized by MEMS technology. The HRG is a robust, high accuracy vibratory gyroscope which can be operated either in rate or whole-angle mode [1,2]. The HRG requires closely matched frequencies of paired degenerate vibratory modes to operate properly, which in turn requires the resonator to be extremely symmetric (round). Previous axisymmetric ring gyros have been essentially two-dimensional, including the planar ring gyro [3], the disc resonator gyroscope [4], and the BAE-Sumitomo gyro [5].

HRGs have a vibrational node at the bottom where attachment can be made with low anchor losses. Previous approaches to forming round, 3D resonators include blowing glass bubbles [6, 7] and etching cavity molds into silicon [8]. A hemispherical diamond resonator fabricated on silicon substrates by combined rotary electron discharge machining and wet etching was reported in [9]. Our approach to etching a round cavity is to wet etch a hole into an isotropic (glass) substrate. In contrast, it is difficult to etch perfectly round holes in Si which has preferred crystal orientations.

Diamond was selected as the structural material because of its very high strength, low coefficient of thermal expansion (CTE) and high thermal conductivity. This combination of properties gives diamond low thermo-elastic damping (TED), making it an excellent material for high Q resonators. Recently diamond resonators have set records for $f-Q$ product [10]. Although single crystal diamond yields the highest Q ($>10^6$ at room temperature [11, 12]) due to a lack of grain boundaries, it would be difficult to create a single crystal diamond hemisphere. Microcrystalline, nanocrystalline and ultra-nanocrystalline diamond (UNCD) can be deposited by varying the deposition conditions in hot filament or plasma chemical vapor deposition (CVD) reactors. UNCD has very low thermal conductivity [13] and the high density of grain boundaries introduce bulk damping losses. Larger grain size and lower boron doping were found to give higher Q in a wide range of cantilevers and double-ended tuning fork structures [14]. Undoped single crystal diamond was also found to have higher Q than doped diamond [10].

To match the average CTE of diamond and polysilicon (the sacrificial layer) from room temperature to the deposition temperature (~715°C) we require a high temperature glass with a CTE of 3 to 3.5 ppm/°C [15]. Attempts to deposit on fused silica were unsuccessful due to CTE mismatch stress. We used wafers of Corning 1715 glass obtained from the Corning Research group for

this purpose [16]. Corning 1715 has a CTE of 3.6 ppm/°C (average from 0 to 800°C), and has an anneal point of 866 °C, which is 305 °C higher than Schott Borofloat.

Testing of the resonators was carried out using a Polytec MSA-400 Laser Doppler Vibrometer with a custom vacuum chamber. Resonators were actuated either electrostatically (using the integrated electrodes) or by shaking the whole chip with a piezoelectric stack. A custom trim station was constructed with a femtosecond laser used to ablate small Au pads from a segmented lip on the hemispheres. Mode frequencies and positions were recorded on the Polytec; this information was used to set up a trim recipe to bring the two degenerate modes to the same frequency.

FABRICATION

Figure 1 shows cross-sections at key steps in the process. A bi-layer of 2 μm UNCD (Ultra Nano-Crystalline Diamond [17]) on top of 2 μm of polysilicon was used to mask the wet etch. Some wafers used 1 μm of CVD SiC [19] instead of diamond. The diamond layer was coated with a 1.3 μm layer of PECVD SiO₂ which was used as an etch mask for the diamond. The diamond film was plasma etched in pure O₂ in an ULVAC model NE-550 etcher. Because the glass contains significant concentrations of Al and Ca, a mixture of 50% HCl, 46% H₂O and 4% HF was used at 40 °C to etch the glass. The high ratio of HCl to HF prevents Ca and Al fluorides from precipitating and interfering with the etch. Following the glass etch, the diamond and polysilicon films were stripped in an O₂ plasma and XeF₂ respectively.

Electrode layer: to electrically contact the diamond layer at the anchor, a metal compatible with high temperature diamond deposition is needed [18]. We have used Zr and Nb for this layer. Nb was etched in an SF₆/Ar plasma, Zr was etched using a BCl₃/Ar etch. Some wafers were fabricated without these metal layers.

Figure 1: Fabrication cross-sectional drawings: a. isotropic glass etch using UNCD diamond or SiC mask. b. metal sputtering and patterning c. PECVD oxide and polysilicon sacrificial layer deposition and patterning d. HFCVD of micro-crystalline diamond film and patterning e. Liftoff of Cr/Au metal pads f. XeF₂ release (more details in the text).

Cavity Formation: An etched cavity is shown in Figure 2 before stripping the etch mask. Automated image-based measurements are made of cavity size, roundness and centricity with respect to the diamond mask hole. 90 diameters are drawn through the center of the hole and a histogram is made (Fig. 3). Due to the isotropic nature of glass, very round holes are possible.

Corning 1715 glass is a high temperature glass made in small batches at the Corning Research facility, and the quality of samples received so far is highly variable. Some wafers yield cavities with diameter, shape and centricity of 80%, some wafers yield near zero. We believe this is due to variations in the concentration of Ca and Al, which cause fluoride precipitates and smaller, off-center or misshapen holes. Commercialization of these resonators will require a supply of high quality, striation-free glass substrates.

Figure 2. Etched cavity before stripping diamond/polysilicon mask viewed in transmitted light. Central circle is the edge of the diamond mask, black circle is the extent of wet etch (1.38mm).

Figure 3. Histogram of hole radii (spaced every 4 degrees) for hole shown in figure 2. Radius variation is $\pm 1 \mu\text{m}$, close to measurement error.

The electrode metal (Zr or Nb) needs to be protected from the sacrificial Si layer, which would react with the metal and form a silicide. We used a combination of 100 nm of Atomic Layer Deposited (ALD) ZrO_2 or Al_2O_3 , followed by 400 nm of PECVD SiO_2 as a barrier between the metal layer and the sacrificial poly-Si. A 5 μm thick layer of polysilicon was then deposited at 650 $^\circ\text{C}$, and patterned in an SF_6 plasma etch. Figure 4 shows a device after polysilicon deposition on top of patterned Nb electrodes.

Lithography: Once the deep cavities are etched into the wafer, subsequent resist must be applied by spraying a mixture of AZP-4620 photoresist, MEK and PGMEA solvents. Most exposures were carried out using a Suss MA6 contact aligner, however, neither contact lithography nor a stepper is capable of forming an image at the bottom of a 0.7 mm deep cavity.

Lithography in the cavities was accomplished using a JPSA IX-1000 excimer laser system at 248 nm to ablate 50 μm diameter anchor regions into the photoresist. Once the polysilicon is patterned, the oxide and ALD layers are plasma etched to allow ohmic contact from diamond to metal.

The wafers were then sent out for Hot-Filament CVD (HFCVD) at SP3 diamond technologies. Microcrystalline diamond was deposited at 715 $^\circ\text{C}$ in 1.5 % CH_4 in H_2 and 444 ppm tri-methyl boron for doping. The diamond film is coated with 1.5 μm of PECVD oxide, which is used to pattern the diamond in pure O_2 plasma. After stripping the mask SiO_2 , Cr/Au is deposited and lifted off to leave bond pads for electrical contacts and trim-pads on diamond tabs at the edge of the hemispheres. The wafer is then diced and a final release is carried out in XeF_2 to remove the polysilicon layer. This release can be carried out before or after packaging.

Figure 4. Nb metal pattern around etched cavity, covered with 1 μm of SiO_2 and 5 μm of polysilicon. Central bright spot is an image of the tungsten filament from the microscope illuminator.

Figure 5 shows a diamond hemisphere pulled from its cavity with tape for SEM inspection. The small hole at the top was the anchor.

Figure 5. A diamond hemisphere pulled out with tape for examination.

Figure 6 shows a completed device with 16 radial diamond electrodes after dicing. These electrodes were used to drive and sense the resonant motion by edge capacitance.

FREQUENCY TRIMMING

To perform as an HRG, the two degenerate modes used for the gyroscope must be extremely well matched in frequency. Minor imperfections in fabrication cause some mode splitting, which is corrected by laser trimming. Diamond tabs at the lip of the resonator are coated with Cr/Au (Figure 7), which is laser ablated. Mass is removed at the four amplitude peaks of the lower frequency mode, corresponding to the nodes of the upper mode (Figure 8), raising the lower mode with little affect on the upper.

Figure 6. Completed device with 16 radial diamond electrodes.

The laser used for ablation was a Spectra Physics Ti: Sapphire Hurricane producing 100 fs pulses with 1 mJ/pulse power. Apertures were used to reduce the power to avoid damaging the diamond tabs under the Au layer (Figure 8).

Figure 7. Trim tabs at edge of hemisphere are coated with Cr/Au.

Figure 8. Laser trim tabs after low power laser ablation of the Au layer. Diamond electrodes in proximity to the tabs are used to drive and sense device motion.

TEST RESULTS

A vacuum chamber with electrical feedthroughs and a glass window was constructed for use with a Polytec MSA- 400 scanning laser vibrometer system. Inside the vacuum chamber a pair of Physik Instrument piezoelectric stacks were attached for in-plane and out-of-plane actuation. Chips were mounted temporarily on the piezo stacks using a low melting point wax and a heat lamp. Devices with integrated on-chip actuation electrodes were actuated electrostatically after wirebonding in ceramic chip carriers. Drive signals from the Polytec were used to drive the piezo stacks or the device electrodes, while the resonant modes were measured using a ring of laser spots on the rim of the hemispherical resonators. Drive signals used were 5V of pseudo-random noise offset by 5VDC, with opposite sign applied to an opposing pair of electrodes. The laser vibrometer computes mode shapes which can be viewed as static images or animations. Mode shape and frequency are compared with FEA predictions.

Figure 9 shows data for as-built frequency separation of the $n=2$ modes of 7 Hz. From the Polytec mode image, the peaks of the lower mode (nodes of the higher node) are noted for laser trim. Figure 10 shows mode data of a chip with as-built separation of 35.5 Hz which was laser trimmed to 0.35 Hz.

Figure 9. Resonator with $n = 2$ mode separation of 7 Hz as-built.

Laser trimmed Δf of 0.35 Hz

Figure 10. Two $n = 2$ wineglass modes laser-trimmed from 35.5 Hz (as built) to 0.35 Hz final mode separation.

Q and Ringdown: Q was measured using the laser vibrometer either in the frequency domain ($Q = f_0 / \Delta f_{-3\text{dB}}$) or time domain by the ringdown method. The ringdown time constant τ is related to the quality factor by $\tau = Q/(\pi f_0)$. Time series data was exported to Matlab for curve fitting to extract τ . Typical Q for these hemispherical resonators ranged from 60,000 to 150,000. A ringdown time of 2.4 seconds is shown in figure 11.

Figure 11. Ringdown measurement for $n = 2$ mode at 18.399 kHz. Data collected with laser vibrometer. $Q = 143,000$. $\tau = 2.4$ s at $P < 10^{-5}$ Torr.

CONCLUSIONS

A process for fabrication of high-Q diamond hemispherical resonators has been developed based on isotropic wet etching of a novel high-temperature glass. Diamond-compatible metals (Nb and Zr) survive the high temperature diamond deposition with protective ALD and PECVD oxide layers.

Trimming the $n = 2$ modes to 0.35 Hz Δf was accomplished by laser ablation of Cr/Au pads on diamond trim-tabs around the periphery of the resonator.

Diamond is a high Q resonator material of extreme strength and chemical resistance. Q as high as 143,000 was observed. Energy loss mechanisms are currently being studied to extend the Q even higher. These hemispherical resonators should form the foundation technology for a new generation of small, accurate MEMS hemispherical resonant gyroscopes.

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