Single Crystalline 4H-SiC Membrane Resonators

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Abstract—We report the first wafer-scale fabrication of semi-insulating, single-crystalline 4H-SiC membrane resonators by timed deep reactive ion etch (DRIE). Trenches were etched 184 μm deep with 84.7° sidewall angle to form 16 μm thick suspended membranes. Sidewall angle, DRIE footing, and surface roughness are characterized. Measured resonance frequencies match with COMSOL simulation within 4%. The modes have quality factors of 500 to 1000 at ambient condition.

Index Terms—Membrane resonator, Single-crystalline 4H-SiC, DRIE

I. INTRODUCTION

Single-crystalline 4H Silicon Carbide (SiC) is an intriguing material with exceptionally low acoustic loss [1]–[3], high stiffness, excellent thermal conductivity, and built-in optically active defects that can be used as spin qubits [4]. A free-standing structure of single crystalline SiC will open applications for harsh environment microelectromechanical systems (MEMS) [5], [6], high-performance filters [7], high-quality-factor MEMS gyroscopes [8-9], and hybrid quantum systems [10], [11].

Traditionally, the free-standing structure is defined by thin film deposition and subsequent removal of the underlying substrate. However, for single crystalline 4H-SiC, this planar approach is not feasible since epitaxial 4H SiC can only be grown on 4H SiC. Smart-cut technique has been demonstrated for 4H-SiC [12], but there are concerns of surface roughness as well as damaging the quantum coherence of the spin states of the defects. To maintain the pristine quality of the resonators top surface, we developed the first wafer-level fabrication of semi-insulating, single-crystalline 4H-SiC membrane micro-resonators by using backside Deep Reactive Ion Etch (DRIE). The etch chemistry allows us to selectively etch 184 μm SiC trenches with 84.7° sidewall angles to form 16 μm thick membranes. Resonance frequencies up to 5 MHz are measured. Quality factors of 500-1000 at room temperature and atmospheric pressure are found. We highlight the geometric features of the DRIE etched membrane that are important to get thinner and higher-Q 4H-SiC membrane resonators.

II. FABRICATION AND CHARACTERIZATION

A. Fabrication Process

Fig. 1(a)-(f) presents main process steps for the single crystalline 4H SiC membranes. We use a semi-insulating 4H-SiC wafer with 4-inch diameter and 500 μm thickness from Cree Inc. The wafer is (0001) oriented within 0.2° and has resistivity of $1 \times 10^{8} \Omega \cdot \text{cm}$. The fabrication starts with mechanical thinning of the wafer to 200 μm [Fig. 1(a)]. In Fig. 1(b)-(d), a lift-off process is employed to get a 2 μm Indium Tin Oxide (ITO) DRIE hard mask. 6 μm photoresist AZ9260 is applied and patterned on the wafer as the sacrificial stencil layer. 2 μm ITO and 20 nm Ti (adhesion layer) are deposited by an electron beam evaporator. The AZ9260 is removed by soaking in 70 °C PG remover for 15 mins. We utilize ITO because it does not react with fluorine ions and is stable under high RIE energies with minimal back-sputtering. In Fig. 1(e), the wafer is mounted on a 6 inch carrier wafer by Crystalbond™ and time etched 184 μm in Panasonic RIE/ICP etcher using SF₆. The following process parameters are used:
RF power 1150 W, platen power 100 W, pressure 0.8 Pa, and 80 sccm SF₆ flow rate. The etch chemistry is similar to [8] except for the DC bias voltage. We monitor the chamber temperature during the etching. Etching is carried out at intervals of 1 hour to avoid overheating. Etch depth and uniformity are monitored with Bruker GT-K Optical Profilometer after each interval. An etch rate of 12 μm/hour and an ITO:SiC selectivity of 1:146 are achieved. Crystalbond™ is chosen over photoresist as the adhesive because its thermal conductivity is better. The carrier wafer was also coated with ITO/Ti to prevent the loading effect and wafer thinning. Fig. 3 shows an example profile when the etch ends. After etching SiC of 186 μm, the SiC wafer and the carrier wafer are soaked in 70 °C hot DI water to dissolve Crystalbond and detach the two wafers. The carbide wafer is then transferred to 70 °C HCl to remove ITO and Ti. Two SEM images of the final devices are shown in Fig. 2.

B. Resonance Characterization

We characterize the membrane resonances with a Polytec MSA-400 scanning Laser Doppler Vibrometer (LDV). The membrane is excited by an axial PZT actuator with a bandwidth of 8 MHz. The PZT is bonded to the edge of the chip to prevent air trapping between the trenches and the PZT. A pseudo-random waveform is applied on the PZT actuator to generate broadband excitation. It is found that our PZT deliver sufficient energy to the membrane up to 6 MHz. The laser is raster scanned to measure the mode shapes. See Fig. 4(a) for the schematic of the experimental setup. Fig. 4(b) shows the average displacement spectrum from 0-5 MHz and the measured mode shape of the membrane shown in Figure 2(b). The measured resonance frequencies match well with COMSOL finite element simulations of the membrane modes, listed in Table 1. For the FEM simulation, we construct a circular membrane with a uniform thickness of 16 μm (Fig. 4(c)) and apply a 3D fixed boundary condition to the perimeter. The simulated thickness agrees well with the optical profilometer measurement of DRIE trench.

![Fig. 3. Optical profilometer measurements of DRIE trench. ContourGT-K 3D Optical Microscope is used to characterize the etch depth. The top graph shows the landscape viewed from the etched side. The color represent the height. The two plots below are the 1D profiles defined by the X and Y direction cut lines in (a), respectively.](image)

The quality factor (Q) of each mode is obtained by fitting the power spectrum (proportional to the square of the displacement) to a Lorentzian function, as shown in Fig. 4(b).

<table>
<thead>
<tr>
<th>Measured Frequency (MHz)</th>
<th>Simulated Frequency (MHz)</th>
<th>Relative Deviation (%)</th>
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<tr>
<td>0.366</td>
<td>0.376</td>
<td>2.57</td>
</tr>
<tr>
<td>0.782</td>
<td>0.763</td>
<td>-2.42</td>
</tr>
<tr>
<td>1.259</td>
<td>1.251</td>
<td>-0.60</td>
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<tr>
<td>1.476</td>
<td>1.426</td>
<td>-3.38</td>
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<tr>
<td>1.854</td>
<td>1.829</td>
<td>-1.32</td>
</tr>
<tr>
<td>2.251</td>
<td>2.181</td>
<td>-3.12</td>
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<tr>
<td>2.477</td>
<td>2.495</td>
<td>0.73</td>
</tr>
<tr>
<td>3.100</td>
<td>3.030</td>
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<tr>
<td>3.318</td>
<td>3.194</td>
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<tr>
<td>4.321</td>
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<td>4.835</td>
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<td>3.40</td>
</tr>
<tr>
<td>4.996</td>
<td>5.002</td>
<td>0.11</td>
</tr>
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</table>

The quality factors are found to be in the range of 500-1000. The quality factors are the same in both atmosphere pressure and vacuum, indicating that air loss is not the dominant mechanism. Since these membranes are relatively thick and the anchor loss is inversely proportional to the cube of the thickness [13], we attribute the low Q to anchor loss.

III. DISCUSSION

Profimeter and resonance frequency measurements show that membranes with different diameters have different thickness. Although the etch rates only change by 7% from 1000 μm opening to 300 μm opening, the membrane thickness can differ by almost a factor of two, as shown in Fig. 5. Fig. 5(a) plots the etch rate as a function of etch opening.
Fig. 4. Resonance mode characterization with Laser Doppler Vibrometer. (a) Schematic of the experimental setup. The membrane device is shown in blue. The PZT actuator is shown in green. (b) Displacement spectrum of the membrane in Fig. 2 measured by the LDV in PPT mode. The mode shapes are mapped by raster-scanning the laser. (c) Zoom-in of the spectrum in the dashed box in (a). Q is extracted by fitting the power spectrum (proportional to displacement square) to a Lorentzian function. A quality factor of 630 is obtained for the (1,1) mode at 0.781 MHz at room temperature and atmosphere pressure.

Fig. 5(b) plots the etch depth and the membrane thickness as a function of membrane diameter. The membrane thickness is calculated by subtracting the etch depth from the 200 μm substrate thickness.

Three DRIE features are crucial to achieve high quality factors: vertical walls, footing, and roughness of the etched surfaces.

The etch recipe realizes 84.7° sidewalls with a continuous single mode operation, without the need to alternate between two modes as in the Bosch process [See Fig. 6(a) and Fig. 7 for examples of the etch sidewall]. To understand the etch mechanism, we perform element analysis with Energy dispersion X-ray Spectroscopy (EDX) on the etched sidewall. We detect Carbon (C), Fluorine (F), and Oxygen (O) [Fig. 6(b)]. These elements indicate a passivated layer of materials such as CxFy and SiFxOy is formed during the DRIE [8], [15].

Fig. 6. Sidewall angles and element analysis: (a) SEM images of the cross-section of through DRIE. A sidewall angle of 84.7° is achieved. (b) Element analysis of the sidewall surface by performing Energy dispersion X-ray Spectroscopy (EDX).

Micro-masking is the re-sputtering of the mask material on the etched area during DRIE. These micro-masks results in SiC residues and pillars on the etched surface. Fig. 8(a) shows an optical micrograph of a membrane with micro-masked SiC.
residues. Fig. 8(b) shows two micro-pillars that occur due to micro-masking.

The etched surface quality is analyzed by using Bruker GTK Optical Profilometer. A roughness average $R_a$ (arithmetical mean deviation) of 4.31 nm (Fig. 9) is measured at the membrane with negligible micro-masking effect. This level of surface roughness is better than that from mechanical machining [14]. Although the measurement precision is limited by the resolution of profilometer, it confirms that our membrane is of high surface quality and the quality factor is not limited by surface loss.

In conclusion, we fabricated the first single crystalline 4H SiC membrane resonators fabrication by DRIE at wafer scale. The resulting membrane surfaces are characterized by SEM and optical profilometer, showing a smooth surface roughness of 4.31 nm. A single-mode DRIE with an etched sidewall angle of 84.7° is demonstrated. The mechanical resonances are measured by LDV from 0-5 MHz. Quality factors are 500-1000 at ambient condition. The measured mechanical modes and FEM simulation agrees well, suggesting well-confined resonances and a predictive FEM model. This work paves the way for realizing nanomechanical and nanophotonics devices to interface with coherent spin states in 4H SiC.

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REFERENCES


