Temperature Dependence of the Elastic Constants of Doped Silicon

Eldwin J. Ng, Vu A. Hong, Yushi Yang, Chae Hyuck Ahn, Camille L.M. Everhart, Thomas W. Kenny

Abstract—Resonators fabricated in heavily doped silicon have been noted to have a reduced frequency-temperature dependence compared to lightly doped silicon. The resonant frequency of silicon MEMS resonators is largely governed by the material’s elastic properties, which are known to depend on doping. In this work, a suite of different types and orientations of resonators were used to extract the first and second order temperature dependences of the elastic constants of p-doped silicon up to 1.7e20 cm\(^{-3}\), and n-doped up to 6.6e19 cm\(^{-3}\). It is shown that these temperature-dependent elastic constants may be used in finite element analysis to predict the frequency-temperature dependence of similarly doped silicon resonators.

Index Terms—Doping, Resonators, Silicon, Temperature Dependence.

I. INTRODUCTION

Silicon has been widely used as the base material for MEMS applications because of the availability of high quality wafers, established fabrication processes, and reliable techniques for integration and packaging. Excellent stability has been observed for well-packaged silicon devices, due to silicon’s near-ideal crystalline properties that include the absence of observable fatigue and temperature hysteresis [1-4]. Furthermore, the mechanical properties of monocrystalline silicon are well characterized [5, 6] and have been used successfully for the predictive modeling of numerous MEMS devices.

Resonant systems form an important group of MEMS devices. When used as time-keeping devices, the frequency stability of resonators is extremely important, and a crucial issue that needs to be addressed is the frequency-temperature sensitivity, which is often around -30 ppm/\(^\circ\)C for silicon. Many methods have been employed to compensate for this temperature dependence, such as using a composite silicon-silicon dioxide structure [7-9], or an active phase-locked loop combined with a micro-oven [10] or a temperature sensor [11, 12]. In recent times, another method for temperature compensation has emerged: a reduced temperature dependence has been observed for silicon resonators that have been heavily doped with either p-type [13] or n-type [14, 15] dopants. Typically, it is seen that a doping concentration of 1e19 to over 1e20 cm\(^3\) is necessary before frequency-temperature compensation effects start to become significant. A suggested mechanism is that heavy doping strains the crystal lattice and shifts the electronic energy bands, resulting in a flow of charge carriers to minimize the free energy, thereby changing the elastic properties [6, 13, 16, 17].

From previous work, it is observed that the frequency-temperature dependences are not tied solely to the doping concentration; resonators with different mode and orientations on the same wafer can exhibit markedly different temperature dependences [14], making it difficult for a MEMS designer to predict the frequency-temperature dependences for more complex geometries.

However, because the resonant frequency is largely governed by a material’s elastic properties, it is possible to use known elastic properties and their temperature dependences to predict the frequency-temperature dependence of arbitrary resonant modes. The temperature dependence of the elastic properties has previously been extracted for lightly doped silicon [6, 18], and more recent work has elucidated the heavily n-doped regime [19]. The heavy p-doped regime has yet to be characterized, and in this work, we characterize six different dopings of silicon (up to 1.7e20 cm\(^{-3}\) p-type and 6.6e19 cm\(^{-3}\) n-type). Based on the method outlined in [18], a suite of different types and orientations of resonators were fabricated with the various dopings, and the doping and the temperature dependences of the elastic constants were extracted. Further, it is demonstrated that the extracted material properties can be used to predict the frequency-temperature dependence of different types of resonators.

II. METHOD FOR EXTRACTING THE ELASTIC CONSTANTS

The elastic constants can be obtained by several methods, with the most commonly cited values measured using the transit times of pulse-echo techniques [6]. Resonant systems can also be used to characterize the elastic constants. The frequency of a resonator is dependent on its geometry and the material properties. By measuring the resonant frequencies of a variety of resonators and their temperature dependences, and linking this together with finite element models of the resonant geometries, the elastic constants and their temperature dependences can be extracted (Fig. 1).

Using a lumped element model, the resonant frequency can
be expressed as
\[ f = \frac{1}{2\pi} \sqrt{\frac{k}{m}} = \frac{1}{V_{\text{eff}}} \sqrt{\frac{s_{\text{eq}}}{\rho}} = \frac{1}{V_{\text{eff}}} \sqrt{s_{\text{eq}}} \]  
(1)
where \( k \) is the equivalent spring constant, \( m \) is the equivalent mass, \( V_{\text{eff}} \) is the effective length (or wavelength), \( \rho \) is the material density, \( c_{\text{eq}} \) is the equivalent material stiffness, and \( s_{\text{eq}} = 1/c_{\text{eq}} \) is the equivalent compliance. The frequency-temperature dependence can be categorized into two types: thermal expansion \((L, \rho)\), and elastic properties \((s_{\text{eq}})\).

A. Frequency Dependence on Thermal Expansion

\( L \) and \( \rho \) are affected by thermal expansion, which for silicon can be approximated with temperature coefficients of first and second order, \( \alpha^{(1)} = 2.84 \text{ ppm/C} \) and \( \alpha^{(2)} = 8.5 \text{ ppb/C}^2 \) respectively, as reported in [18]. The variation in frequency due to temperature can then be written in fractional form with a Taylor expansion, expanding up to the quadratic terms in \( \Delta T \):
\[ \frac{\Delta f}{f_0} \approx \sqrt{(1 + \alpha^{(1)} \Delta T + \alpha^{(2)} (\Delta T)^2)} - 1 - 1 \]  
(2)
where the subscript \( 0 \) is used to denote quantities at a reference temperature \( T_0 \) (here 25 °C), and \( \frac{\Delta f}{f_0\rvert_{T_0}} \) is the component due to the variation of elastic properties \( c_{\text{eq}} \) (or \( s_{\text{eq}} \)) with temperature, with the thermal expansion assumed to be zero. Consequently, the effect of thermal expansion can be decoupled from the effect of the elastic properties. For all dopings, the density at \( T_0 \) is assumed to be 2330 kg m\(^{-3}\) [6].

B. Frequency Dependence on Elastic Properties

Focusing now on the elastic properties, it is known that with the reference axis oriented along the <100> directions, the elastic stiffness properties of monocrystalline silicon (with cubic symmetry) can be represented as a 4th rank tensor that can be simplified (ignoring higher order terms) as [20]

\[ \begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3 \\
\sigma_4 \\
\sigma_5 \\
\sigma_6
\end{bmatrix} =
\begin{bmatrix}
c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\
0 & 0 & 0 & c_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & c_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & c_{44}
\end{bmatrix}
\begin{bmatrix}
\epsilon_1 \\
\epsilon_2 \\
\epsilon_3 \\
\epsilon_4 \\
\epsilon_5 \\
\epsilon_6
\end{bmatrix} \]

or in compliance form as
\[ \begin{bmatrix}
e_1 \\
e_2 \\
e_3 \\
e_4 \\
e_5 \\
e_6
\end{bmatrix} =
\begin{bmatrix}
s_{11} & s_{12} & s_{12} & 0 & 0 & 0 \\
s_{12} & s_{11} & s_{12} & 0 & 0 & 0 \\
s_{12} & s_{12} & s_{11} & 0 & 0 & 0 \\
0 & 0 & 0 & s_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & s_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & s_{44}
\end{bmatrix}
\begin{bmatrix}
e_1 \\
e_2 \\
e_3 \\
e_4 \\
e_5 \\
e_6
\end{bmatrix} \]

where there are three independent components \((s_{11}, s_{12}, s_{44})\), or \((c_{11}, c_{12}, c_{44})\) that define the material.

The equivalent stiffnesses and compliances for some ideal resonant modes are given in Table I. It is apparent that working in the compliance form compared to the stiffness form for these resonators is especially convenient due to the linear relations. From the table, it is seen that a choice of uniaxial and Lamé modes [21] in the <100> and <110> directions is particularly useful extracting the elastic constants because they span the vector space well: uniaxial modes along the <100> direction are dependent solely on \( s_{11} \); Lamé modes along the <110> direction are dependent solely on \( s_{44} \); the \( s_{12} \) term appears strongly in both the uniaxial mode in the <100> direction and the Lamé mode in the <100> direction. These equations could be directly used in the extraction of the elastic constants, however, because fabricated devices are expected to deviate slightly from these ideal modes due to geometrical differences (such as the presence of supporting anchors, and release-etch holes), finite element analysis (Section II.C) was used to account for the actual fabricated geometry.

It is first noted that the elastic constants do vary slightly with doping [6], resulting in a slight frequency shift. The elastic constants for doped silicon at the reference temperature \((s_{11,0}, s_{12,0}, s_{44,0})\) can be extracted by starting with values for undoped silicon \((s_{11,0} = 7.691\text{e-12 Pa}^{-1}; s_{12,0} = -2.142\text{e-12 Pa}^{-1}; s_{44,0} = 12.58\text{e-12 Pa}^{-1})\) and minimizing the least-squares fractional difference between the finite-element simulated frequencies and the measured frequencies using an iterative gradient descent. The extracted reference-temperature elastic constants for the various dopings can then be applied as the linearization point for subsequent temperature dependence analysis.

The temperature dependence of the elastic constants can then be extracted by first relating the fractional dependence of \( s_{\text{eq}} \) to that of the three independent elastic compliances, using a multivariable Taylor expansion to 2nd order about the reference-temperature compliance values \((s_{11,0}, s_{12,0}, s_{44,0})\):
\[ \frac{\Delta s_{\text{eq}}}{s_{\text{eq}}\rvert_{T_0}} \approx \sum_i \mu_i \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) + \frac{1}{2} \sum_i \sum_j \mu_{ij} \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) \left( \frac{\Delta s_j}{s_j\rvert_{T_0}} \right) \]  
(4)
where \( i \) and \( j \) each index the 11, 12 and 44 components, and
\[ \mu_i = \frac{\partial^2 \Delta s_{\text{eq}}}{\partial s_i^2\rvert_{T_0}} \text{ and } \mu_{ij} = \frac{\partial^2 \Delta s_{\text{eq}}}{\partial s_i \partial s_j\rvert_{T_0}} \]

The fractional frequency dependence on the equivalent compliance can also be expanded from (1) as
\[ \frac{\Delta f}{f_0\rvert_{T_0}} \approx -\frac{1}{2} \left( \frac{\Delta s_{\text{eq}}}{s_{\text{eq}}\rvert_{T_0}} \right)^2 \]  
(5)
and after substituting (4) in,
\[ \frac{\Delta f}{f_0\rvert_{T_0}} \approx -\frac{1}{2} \sum_i \mu_i \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) - \frac{1}{4} \sum_i \sum_j \mu_{ij} \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) \left( \frac{\Delta s_j}{s_j\rvert_{T_0}} \right) + \frac{3}{8} \sum_i \sum_j \mu_{ij} \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) \left( \frac{\Delta s_j}{s_j\rvert_{T_0}} \right) \]  
(6a)
\[ = \sum_i \gamma_i \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) + \sum_i \sum_j \gamma_{ij} \left( \frac{\Delta s_i}{s_i\rvert_{T_0}} \right) \left( \frac{\Delta s_j}{s_j\rvert_{T_0}} \right) \]  
(6b)
where the coefficients of the fractional compliances in (6a) have been grouped together as \( \gamma_i \) and \( \gamma_{ij} \) in (6b). Although (6b) can be derived directly from (1) by expanding with respect to the fractional compliances, the given relation...
Resonant frequencies are given by (1). The relation between the compliances and stiffnesses is given in [20]:

\[ \frac{c_{11} + c_{12}}{(c_{11} - c_{12})^2} \]

...directions. With the linear form of (7), (4) is reduced to

\[ \text{with the} \]

...between (6a) and (6b) is useful for analytically computing the coefficients in some cases, such as uniaxial, square extensional, and Lamé resonant modes.

For uniaxial stress along an arbitrary direction \( \mathbf{x} \), the equivalent compliance can be obtained from (3b) by means of a tensor rotation [20]:

\[ s_{eq} = \lambda_{11} s_{11} + \lambda_{12} s_{12} + \lambda_{44} s_{44} \]  

where

\[ \lambda_{11} = 1 - 2\beta \]  

\[ \lambda_{12} = 2\beta \]  

\[ \lambda_{44} = \beta \]

\[ \beta = a_x^2 a_y^2 + a_z^2 + a_z^2 a_x^2 + a_x^2 a_z^2 \]

...the with \( \mathbf{a} \)’s being the directional cosines between the stress direction \( \mathbf{x} \) and the reference axis (1,2,3) oriented along the <100> directions. With the linear form of (7), (4) is reduced to

\[ \frac{\Delta s_{eq}}{s_{eq,0}} = \sum \left( \lambda_{ij} \frac{s_{j0}}{s_{eq,0}} \right) \]

...and the coefficients \( y \) in (6b) then correspond to

\[ y_1 = -\frac{1}{2} \lambda_{44} \left( \frac{s_{40}}{s_{eq,0}} \right) \]

\[ y_{ij} = \frac{3}{8} \lambda_{ij} \left( \frac{s_{j0}}{s_{eq,0}} \right) \]

...where values for uniaxial stress orientations along the <110> and <100> directions have been calculated in Table I. Similarly, square extensional modes can be approximated to be in biaxial stress with two superimposed uniaxial modes perpendicular to each other [22], and from (3b), the equivalent compliance can be worked out assuming \( \sigma_1 = \sigma_2 \). Lamé modes are dependent on the shear modulus which is given for cubic crystals as a function of the perpendicular directions of propagation and displacement, \( \mathbf{x} \) and \( \mathbf{y} \) [23]

\[ \lambda_{11} = 4\psi \]

\[ \lambda_{12} = -4\psi \]

\[ \lambda_{44} = (1 - 2\psi) \]

\[ \psi = a_x^2 a_y^2 + a_x^2 a_z^2 + a_z^2 a_y^2 \]

The coefficients \( y \) for the Lamé and square extensional modes have similarly been analytically calculated (Table I). It is also useful to note that resonant orientations along the <100> and <110> directions typically represent the extremes for (100) silicon wafers according to (7) and (11), and are hence the most useful for characterizing the elastic properties.

C. Finite Element Analysis

For a general resonant mode, finite element analysis can be directly used to simulate the coefficients \( y \) in (6b). Each 2D resonant geometry (including support tethers and release-etch holes) as drawn in the mask layout was imported into COMSOL 4.3b. A lateral etch of 0.1 \( \mu \)m into the features (decreasing the device dimensions) was taken into account based on scanning electron microscope (SEM) cross-section images of a typical trench etch. This 2D geometry was extruded to the corresponding device layer thickness to form the 3D geometry. A planar mesh of triangular elements was first generated across the 2D geometry with a maximum element size of 5 \( \mu \)m, and a higher mesh density (1 \( \mu \)m) at the support tethers and release-etch holes. This planar mesh was then swep in the thickness direction with a maximum element size of 10 \( \mu \)m, from the top to the bottom faces of the geometry. Quarter- and half-symmetry conditions were exploited where possible to reduce the computation time.

Device support tethers were terminated in a fixed condition at the anchor. The initial elastic compliances were varied by a small amount \( \Delta s_1 \) (1% percent of \( s_{10} \), based on the expected temperature variation), and these elastic properties were substituted into COMSOL while retaining the same mesh. An eigenmode analysis was performed and the frequency change of the desired eigenmode was noted for the \( \Delta s_1 \) variation, thereby allowing for the coefficients \( y \) to be extracted. For the resonant geometries used in the extraction, a finer mesh with about 1.5 times the density was then used to ensure that values of coefficients \( y \) had converged to \( \pm 0.0002 \).

D. Extraction of the Temperature Coefficients of Compliance

Finally, the elastic compliances were approximated to have

<table>
<thead>
<tr>
<th>Resonant Mode</th>
<th>( s_{eq} )</th>
<th>( c_{eq} ) = ( \frac{1}{s_{eq}} )</th>
<th>Example Mode Shape</th>
<th>( \nu_{eff} ) of Mode</th>
<th>( L_{eff} ) (1st length extensional mode)</th>
<th>( \nu_{eff} ) (1st Lamé mode)</th>
<th>( \nu_{eff} ) (1st square extensional mode)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uniaxial longitudinal modes along &lt;100&gt;</td>
<td>( s_{11} )</td>
<td>( c_{11} + c_{12} )</td>
<td></td>
<td></td>
<td>( 2L )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uniaxial longitudinal modes along &lt;110&gt;</td>
<td>( \frac{1}{2} s_{11} + \frac{1}{2} s_{12} + \frac{1}{4} s_{44} )</td>
<td>( 4\nu_{eq} (c_{11} + c_{12} - 2c_{44}) )</td>
<td></td>
<td></td>
<td>( \sqrt{2L} )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shear modes along &lt;110&gt; (Lamé along &lt;100&gt;)</td>
<td>( 2(s_{11} - s_{12}) )</td>
<td>( \frac{1}{2} (c_{11} - c_{12}) )</td>
<td></td>
<td></td>
<td>Lamé</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shear modes along &lt;100&gt; (Lamé along &lt;100&gt;)</td>
<td>( s_{44} )</td>
<td>( c_{44} )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Square extensional longitudinal mode*</td>
<td>( s_{11} + s_{12} )</td>
<td>( c_{11} + c_{12} - 2\frac{c_{12}}{c_{11}} )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Resonant geometry as oriented on a (100) silicon wafer. Direction of wave propagation is diagonal to device edge [21].

** For all planar orientations on a (100) silicon wafer using a mode shape approximation [22].
a polynomial dependence on temperature:

\[
\frac{\Delta f}{f_0} = T_{S_{11}}^{(1)} \Delta T + T_{S_{12}}^{(2)} (\Delta T)^2 + \ldots
\]

(12)

where \(T_{S_{11}}^{(1)}\) and \(T_{S_{12}}^{(2)}\) are the temperature coefficients of the compliances of first and second order respectively and are the parameters to be extracted for each doping type/concentration. The fractional frequency change with temperature can thus be derived by substituting (12) into (6b) and then into (2). Expanding the result and keeping terms up to the second order in \(\Delta T\) gives

\[
\frac{\Delta f}{f_0} \approx (\Delta T) \left[ \frac{1}{2} \alpha^{(1)} + \sum \gamma_i T_{S_{1i}}^{(1)} \right] + (\Delta T)^2 \left[ \frac{1}{2} \alpha^{(2)} - \frac{1}{6} (\alpha^{(1)})^2 + \frac{1}{2} \alpha^{(1)} \sum \gamma_i T_{S_{2i}}^{(1)} + \sum \gamma_i T_{S_{2i}}^{(2)} + \sum \gamma_i T_{S_{1i}}^{(1)} T_{S_{1j}}^{(1)} \right]
\]

(13)

As there are three independent compliances \((s_{11}, s_{12}, s_{44})\), at least three measured frequency-temperature curves from different resonators are necessary to extract the \(T_{S_{1i}}\). The measured frequency-temperature curve for a resonator \(k\) can first be fitted in a least-squares sense with linear and quadratic temperature coefficients \(p_k\) and \(q_k\):

\[
\frac{\Delta f}{f_0} \approx p_k (\Delta T) + q_k (\Delta T)^2
\]

(14)

It is readily seen that by equating the first order temperature coefficients of (13) and (14),

\[
\begin{bmatrix}
\frac{p_{k_1}}{p_{k_2}}
\frac{p_{k_3}}{p_{k_4}}
\vdots
\end{bmatrix} = \frac{1}{2} \begin{bmatrix}
k_{1,11} & k_{1,12} & k_{1,44}
k_{2,11} & k_{2,12} & k_{2,44}
k_{3,11} & k_{3,12} & k_{3,44}
\end{bmatrix} \begin{bmatrix}
T_{S_{11}}^{(1)}
T_{S_{12}}^{(1)}
T_{S_{14}}^{(1)}
\end{bmatrix}
\]

(15)

where the subscripts \(k_1, k_2, k_3\) denote different resonator designs. If more than three resonators are available, the unknown \(T_{S_{1i}}^{(1)}\) are fitted in the least-squares sense. The second order coefficients \(T_{S_{ij}}^{(2)}\) can be fitted similarly.

III. MEASUREMENT OF FREQUENCY-TEMPERATURE DEPENDENCES

A. Fabrication

The resonators in this work were fabricated using an epitaxial polysilicon encapsulation process (epi-seal). The epi-seal process is a wafer-scale, hermetic encapsulation process that seals the MEMS within an ultra-clean, sub-Pa vacuum cavity that is free from native oxide [24, 25]. This results in high stability resonators with ppb-level frequency drift [4].

The process was originally proposed by researchers at the Robert Bosch Research and Technology Center in Palo Alto and then demonstrated in a close collaboration with Stanford University. This collaboration is continuing to develop improvements and extensions to this process for many applications, while the baseline process has been brought into commercial production by SiTime Inc.

In the baseline process, the device doping concentration and variation is determined by Czochralski-grown wafers. As heavily boron-doped wafers were not available, epitaxial deposition was used to fabricate the doped device layers on some starting silicon-on-insulator (SOI) wafers [26] prior to the epi-seal process. This also allows for large released areas without release-etch holes (unlike the baseline epi-seal process), and the doping concentration can be carefully controlled by gas flow rates. For the other dopings in this process, SOI wafers directly purchased from wafer vendors were used. A summary of the wafers used in this analysis is provided in Table II.

The epi-seal fabrication technique has been detailed before in [24], but in short, the process begins with deep reactive ion etching (DRIE) trenches (limited to 0.7 – 1.5 μm wide) to define the resonator and electrical isolation on a device layer. A 2 μm layer of oxide is deposited to bridge the DRIE trenches and then electrical contact vias are etched in the oxide. A 6 μm layer of epitaxial polysilicon is deposited over the oxide, and vent holes are then etched into the polysilicon. The oxide around the resonator is etched away through the vent holes with vapor HF, and the cavity is sealed by depositing a thick (~20 μm) layer of epitaxial polysilicon over the vents. It is this final seal in a clean, high temperature hydrogen environment that confers the ideal packaging properties mentioned above. Electrical isolation and contacts through the encapsulation are formed, completing the process. A cross-section SEM of the final device is shown in Fig. 2.

The electrical resistivities of the device layers were calculated by measuring electrical test structures in the device layer located near the resonators to give a good estimate for the doping concentration. The device layer thicknesses were measured using a cross-section SEM of a location on the wafer in proximity to the tested devices.
<table>
<thead>
<tr>
<th>Modeled Resonator Mode</th>
<th>Mode Shape</th>
<th>Compliance Coefficients*</th>
<th>Modeled Frequency f&lt;sub&gt;0&lt;/sub&gt;</th>
<th>Typical Measured Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>LE eEH &lt;100&gt;</td>
<td>Length Extensional</td>
<td>-0.5016 0.0003 0.3765</td>
<td>5.34 MHz 1.1 M</td>
<td>15.44 -15.32 -25.81 -29.14</td>
</tr>
<tr>
<td>No Etch Holes</td>
<td>-0.0001 0.0003 0.0000</td>
<td>5.254 5.269</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700 x 50 x 17 µm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LE wEH &lt;100&gt;</td>
<td>Length Extensional</td>
<td>-0.4795 0.0316 0.3426</td>
<td>6.055 MHz 200 k</td>
<td>-14.52 -27.31 9.596 1.24 +11.98</td>
</tr>
<tr>
<td>With Etch Holes 600 x 300 x 40 µm</td>
<td>-0.0178 0.0004 -0.0033</td>
<td>-26.58 -35.13 6.056 -83.91 -76.36</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.0027 0.021 0.0008</td>
<td>-26.20 40.71 5.981 5.933</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LE eEH &lt;110&gt;</td>
<td>Length Extensional</td>
<td>-0.3196 -0.0867 0.1565</td>
<td>6.10 MHz 1.1 M</td>
<td>-8.95 -23.28 -28.96</td>
</tr>
<tr>
<td>No Etch Holes</td>
<td>-0.0895 -0.0711 0.0119</td>
<td>5.969 5.983</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700 x 50 x 17 µm</td>
<td>-0.2695 0.2357 0.1115</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LE wEH &lt;110&gt;</td>
<td>Length Extensional</td>
<td>-0.3137 -0.0583 0.0886</td>
<td>6.961 MHz 220 k</td>
<td>-8.20 24.63 6.845</td>
</tr>
<tr>
<td>With Etch Holes 600 x 300 x 40 µm</td>
<td>-0.2743 0.3516 0.0774</td>
<td>-25.56 -34.11 -6.978</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>-29.36 -27.39 -6.936</td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td>-22.53 -40.47 -36.77</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>-19.48 -19.48 -6.914</td>
<td></td>
<td></td>
</tr>
<tr>
<td>WE eEH &lt;100&gt;</td>
<td>Width Extensional</td>
<td>-0.4843 0.0329 0.3436</td>
<td>46.1 MHz 60 k</td>
<td>-15.56 -28.22 46.02 46.15</td>
</tr>
<tr>
<td>With Etch Holes 296 x 80 x 40 µm</td>
<td>-0.0138 -0.0025 -0.0055</td>
<td>-26.58 -32.18 46.15</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.0018 0.0059 -0.0003</td>
<td>-36.15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>WE wEH &lt;110&gt;</td>
<td>Width Extensional</td>
<td>-0.3089 -0.0314 0.1574</td>
<td>51.9 MHz 150 k</td>
<td>1.11 26.10 51.91</td>
</tr>
<tr>
<td>With Etch Holes 296 x 80 x 40 µm</td>
<td>0.0479 -0.0016 -0.197</td>
<td>-35.66 51.08 51.74 51.58</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.2390 0.1799 0.0901</td>
<td>-49.96 51.58 51.36</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lamé eEH &lt;100&gt;</td>
<td>Lamé mode</td>
<td>-0.3910 0.1329 0.2274</td>
<td>8.27 MHz 1.7 M</td>
<td>-11.64 -25.30 8.097 8.125</td>
</tr>
<tr>
<td>No Etch Holes</td>
<td>-0.1095 0.0000 0.0166</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>400 x 400 x 17 µm</td>
<td>-0.0000 0.0000 0.0000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lamé wEH &lt;100&gt;</td>
<td>Lamé mode</td>
<td>-0.4005 0.1021 0.2486</td>
<td>8.078 MHz 130 k</td>
<td>-11.99 -26.04 7.895 7.919</td>
</tr>
<tr>
<td>With Etch Holes 400 x 400 x 40 µm</td>
<td>-0.0964 0.0023 0.0194</td>
<td>-31.17 8.088 8.087 7.956</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.0031 0.0008 0.0008</td>
<td>-41.76 7.956 7.838</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lamé eEH &lt;110&gt;</td>
<td>Lamé mode</td>
<td>-0.0000 0.0000 0.0000</td>
<td>10.4 MHz 1.7 M</td>
<td>-2.27 -21.34 10.04 10.08</td>
</tr>
<tr>
<td>No Etch Holes</td>
<td>-0.4993 0.0000 0.0000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>400 x 400 x 17 µm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lamé wEH &lt;110&gt;</td>
<td>Lamé mode</td>
<td>-0.0345 -0.0166 0.0108</td>
<td>9.991 MHz 90 k</td>
<td>0.066 +1.048 6.975 9.715</td>
</tr>
<tr>
<td>With Etch Holes 400 x 400 x 40 µm</td>
<td>0.0141 -0.0194 0.0074</td>
<td>+23.03 -29.08 10.02 9.953</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.4796 0.0469 0.3459</td>
<td>-29.85 -19.62 -17.90</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SqExt eEH &lt;100&gt;</td>
<td>Square Extensional</td>
<td>-0.6502 -0.2999 0.6326</td>
<td>14.2 MHz 30 k</td>
<td>20.62 23.79 13.97 13.99</td>
</tr>
<tr>
<td>No Etch Holes 300 x 300 x 17 µm</td>
<td>0.1527 -0.0073 0.0390</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.0025 0.0112 -0.0000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SqExt eEH &lt;110&gt;</td>
<td>Square Extensional</td>
<td>-0.6090 -0.3205 0.5827</td>
<td>14.6 MHz 250 k</td>
<td>-19.10 -23.96 14.36 14.36</td>
</tr>
<tr>
<td>No Etch Holes 300 x 300 x 17 µm</td>
<td>0.1699 -0.0209 0.0436</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.0608 0.0699 0.0212</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ring &lt;110&gt;</td>
<td>Double Breathe-</td>
<td>-0.4158 -0.0525 0.2577</td>
<td>19.8 MHz 200 k</td>
<td>-12.26 -27.21 19.47 19.49</td>
</tr>
<tr>
<td>mode Ring</td>
<td>R&lt;sub&gt;a&lt;/sub&gt;: 59; R&lt;sub&gt;a&lt;/sub&gt;: 69.5;</td>
<td>-0.0427 -0.0185 0.0030</td>
<td>-12.27 -32.17 19.76 19.77</td>
<td></td>
</tr>
<tr>
<td>t&lt;sub&gt;a&lt;/sub&gt;: 40 µm</td>
<td>-0.1269 0.1605 0.0241</td>
<td>-34.56 19.59 19.46</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DEF&lt;110&gt;</td>
<td>Double-ended</td>
<td>-0.3743 -0.1924 0.2309</td>
<td>1.25 MHz 10 k</td>
<td>-10.28 -38.62 1.184 1.198</td>
</tr>
<tr>
<td>Tuning Fork 200 x 6 x 40 µm</td>
<td>0.1340 -0.0116 0.0535</td>
<td>-40.49 -27.62 1.276 1.276</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-0.2597 0.2920 0.1067</td>
<td>-44.06 1.244 1.247</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DRG &lt;100&gt;</td>
<td>Disk Resonating</td>
<td>-0.4421 -0.2464 0.3429</td>
<td>262 kHz 100 k</td>
<td>-25.54 -36.03 0.2627 0.2546</td>
</tr>
<tr>
<td>Gyroscope</td>
<td>R&lt;sub&gt;g&lt;/sub&gt;: 143; R&lt;sub&gt;a&lt;/sub&gt;: 299;</td>
<td>-0.1258 -0.0767 0.0696</td>
<td>-29.92 -28.03 0.2546</td>
<td></td>
</tr>
<tr>
<td>t&lt;sub&gt;a&lt;/sub&gt;: 40 µm</td>
<td>-0.1837 0.2236 0.0644</td>
<td>0.2546</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DRG &lt;110&gt;</td>
<td>Disk Resonating</td>
<td>-0.4993 -0.2010 0.4184</td>
<td>247 kHz 100 k</td>
<td>-26.16 -28.06 0.2488 0.2422</td>
</tr>
<tr>
<td>Gyroscope</td>
<td>R&lt;sub&gt;g&lt;/sub&gt;: 143; R&lt;sub&gt;a&lt;/sub&gt;: 299;</td>
<td>-0.0919 0.1220 0.0249</td>
<td>-26.20 39.03 0.2422</td>
<td></td>
</tr>
<tr>
<td>t&lt;sub&gt;a&lt;/sub&gt;: 40 µm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*These columns give typical values for the given mode. These values depend on factors such as doping and thickness. Mode shapes are shown with the colors indicating the vector sum of the displacement. Blue: minimum, Red: Maximum. Data from shaded cells were used for the extraction of the elastic constants. The other data were used for verification of the extracted values (Section V).
B. Measurement Setup

To obtain frequency-temperature curves, the encapsulated resonators were wire bonded to a package and mounted on a printed circuit board. These were then placed in a Thermotron S-1.2 environmental chamber for temperature control and allowed to stabilize at the initial temperature step (-40°C) for 60 min and subsequent temperature steps (5°C increments to 85°C) for 30 min each. The temperature was measured using a platinum resistance temperature detector (RTD) placed near the device, and it was observed that the temperature reached stabilization within 30 min for the initial step and 10 min for subsequent steps. Since these were encapsulated resonators, no purge gas was used (air ambient). Frequency sweeps were performed with an Agilent 8753ES Network Analyzer while operating the devices in the linear regime, and the RTD temperature was recorded at the time of the frequency sweep. The resonant frequency was determined by the maximum point of a fitted curve around the peak of the frequency sweep, and the quality factor (Q) was computed using the -3 dB bandwidth method. Frequency-temperature curves were thus mapped out for the various doping concentrations and least-squares quadratic curves were fitted with coefficients $p$ and $q$ around $T_0 = 25\degree C$.

IV. RESULTS

A. Comparison of Analytical and Finite Element Results

Several resonators were designed around the resonant modes in the above analysis and are presented in Table III. Uniaxial resonant modes include the length extensional (LE) and width extensional (WE) modes, as well as double-ended tuning forks (DETF) [7]. Lamé, square extensional (SqExt), double breathe-mode rings (Ring) [27] and disk resonating gyroscopes (DRG) [28] represent typical resonant designs of interest, and all these devices together span resonant frequencies of over two orders of magnitude. Devices without release-etch holes were fabricated only for the heavy boron-doped wafers and not the other due to limited release areas of the baseline epi-seal process (Section III.A).

The devices were simulated for the corresponding device thickness in Table II, with the coefficients $\gamma$ for a representative thickness listed in Table III. The values of the coefficients $\gamma$ obtained by analysis (Table I) and finite element simulation (Table III) are seen to be in good agreement, especially for the LE and Lamé modes without release-etch holes. Release-etch holes, support tethers, and mode shape differences are seen to cause the coefficients of the actual devices to deviate slightly from the analytical model.

B. Quality Factors

High quality factor (Q) resonators are necessary for accurate frequency measurements. Typical measured Qs (Table III) for this work are in the range of several hundreds of thousands, indicating that the peak frequency can typically be determined with frequency sweeps accurately to well under a ppm. The Q for most of these resonators is known to be limited by thermoelastic dissipation [29-31], but could also be limited by the Akhieser effect [32] for the Lamé modes without release-etch holes. Anchor losses could also be a limiting factor for the WE resonators.
C. Elastic Properties of Doped Silicon

The frequency-temperature dependences of the various resonators were fitted with quadratic curves centered around 25°C – the coefficients are given in Table III and the plots in Fig. 3. Not all devices were fabricated on all wafers: a couple of lower stiffness designs experienced stiction issues, leading to gaps in the table.

The extracted doping dependences of the elastic constants as well as their first and second order temperature dependences are listed in Table IV. Data from the shaded cells in Table III were used for the elastic constant extraction, as these were believed to best span the vector space of the elastic constant variations. The other data were used for verification of the fits allowing for frequency predictions at 25°C – the coefficients are given in Table III and the plots in Fig. 3. N-type doping achieves similar significant temperature compensation effects by means of $s_{11}$ and $s_{12}$.

The results of the heavily p-doped devices are particularly interesting for temperature compensation. Comparing devices with first-order compensation (turnover points) within the temperature range, the frequency-temperature curvatures for p-doped Lamé modes oriented in the $<110>$ direction are much smaller than those achieved by n-doped devices. The curvature coefficients $q$ are seen to be around $-25$ ppb°C$^2$ for the p-types, compared to about $-80$ ppb°C$^2$ for n-type devices. Furthermore, even though the two heavy p-doped concentrations are close to each other at 1.4 and 1.7e20 cm$^{-3}$, a significantly lower curvature of the heavier p-doped device is observed ($-21$ ppb°C$^2$ vs $-31$ ppb°C$^2$). It is expected that even heavier doping could yield lower curvatures. It is also noted that the quality factors of these Lamé mode resonators are also largely unaffected by the heavy doping – heavily p-doped temperature-compensated resonators with high f-Q products of 2e13 Hz have been demonstrated [26].

D. Uncertainty Analysis

The uncertainties of the elastic constants and their temperature dependences are listed in Table V. Uncertainties are believed to arise from several sources:

1) Process Variation

Fabrication process variations around the wafer such as in doping concentration and etch profiles are known to cause a spread in the measured resonant frequency, resulting in an uncertainty in the extracted values. The measured resonant frequency at $T_0$ for the resonant modes used in the elastic constant extraction was observed to fall within ±1000 ppm

### TABLE IV

<table>
<thead>
<tr>
<th>Doping Type and Concentration (cm$^{-3}$)</th>
<th>$c_{11}$ (GPa)</th>
<th>$c_{12}$ (GPa)</th>
<th>$c_{44}$ (GPa)</th>
<th>$T_{c11}^{(1)}$ (ppm/°C)</th>
<th>$T_{c12}^{(1)}$ (ppm/°C)</th>
<th>$T_{c44}^{(1)}$ (ppm/°C)</th>
<th>$T_{c11}^{(2)}$ (ppb/°C)</th>
<th>$T_{c12}^{(2)}$ (ppb/°C)</th>
<th>$T_{c44}^{(2)}$ (ppb/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron 1.7e20</td>
<td>163.2</td>
<td>63.5</td>
<td>75.3</td>
<td>-56.7</td>
<td>-104.8</td>
<td>1.9</td>
<td>-54</td>
<td>-44</td>
<td>-52</td>
</tr>
<tr>
<td>Boron 1.4e20</td>
<td>163.3</td>
<td>63.8</td>
<td>75.5</td>
<td>-56.9</td>
<td>-105.5</td>
<td>2.2</td>
<td>-57</td>
<td>-37</td>
<td>-71</td>
</tr>
<tr>
<td>Boron 4.1e18</td>
<td>165.7</td>
<td>64.5</td>
<td>79.2</td>
<td>-63.4</td>
<td>-77.5</td>
<td>-48.3</td>
<td>-50</td>
<td>-7</td>
<td>-88</td>
</tr>
<tr>
<td>Antimony 1.3e18</td>
<td>165.6</td>
<td>64.4</td>
<td>79.3</td>
<td>-65.5</td>
<td>-85.1</td>
<td>-60.9</td>
<td>-68</td>
<td>-28</td>
<td>-53</td>
</tr>
<tr>
<td>Arsenic 1.2e19</td>
<td>164.2</td>
<td>65.6</td>
<td>78.6</td>
<td>-46.9</td>
<td>-134.6</td>
<td>-63.1</td>
<td>-105</td>
<td>32</td>
<td>-45</td>
</tr>
<tr>
<td>Phosphorus 6.6e19</td>
<td>164.0</td>
<td>66.7</td>
<td>78.2</td>
<td>-34.2</td>
<td>-135.2</td>
<td>-67.8</td>
<td>-103</td>
<td>-1</td>
<td>-40</td>
</tr>
</tbody>
</table>

Ranges are used to represent values across the different wafers and devices. The larger values of the range correspond to wafers on which release-etch holes were required.
(95% confidence interval). This was propagated to the elastic constants by rewriting (1) as
\[ S_{eq} = \frac{1}{f^2 \rho_{eff} T_{eq}} \]
and using the relations in Table I to estimate the uncertainty in \( s_i \) and then \( c_i \).

The uncertainties of the temperature coefficients were estimated as the 95% confidence intervals of the fitted parameters \( T_{S_i} \) in the least-squares linear regression performed in (15), and these were converted to the corresponding uncertainties for \( T_{C_i} \).

2) Dimensional Offsets

While the above section on process variations addresses the spread of the data, offset errors could also be incurred during fabrication. The DRIE process is known etch laterally, causing the resonant geometry dimensions to be smaller than designed, and hence \( L_{eff} \) in (16). While a lateral etch of 0.1 \( \mu \)m was accounted for to capture this offset (Section II.C), there could be some error in this value, and the lateral dimensions of the finite element model were hence varied \( \pm 50 \) nm in addition to the 0.1 \( \mu \)m already accounted for. Re-running the finite element model with these dimensional variations resulted in changes to the resonant frequencies as well as the coefficients \( \gamma \). The resonant frequency changes were propagated as uncertainties in the elastic constants \( c_i \), while changes in \( \gamma \) resulted in uncertainties of the temperature coefficients \( T_{C_i} \) using the relation (15). It is observed that the uncertainties due to lateral etch offsets were larger for devices with release-etch holes compared to devices without release-etch holes, where the higher values of the specified ranges in Table V correspond to the devices with etch holes. For devices with etch holes, the lateral dimension offset is seen to be one of the largest causes of uncertainty in \( c_i \).

The thicknesses of the device layers were measured using SEM imaging at locations close to the tested devices. However, there could also be offset errors due to within-wafer thickness variations: \( \pm 1 \mu \)m variations in thickness were simulated due to wafer specifications. The effect of thickness was simulated by varying the finite element model and performing a similar procedure to the lateral dimension offset analysis above. The uncertainties due to thickness variation were found to be relatively small compared to the other error sources as Lamé modes are in-plane modes that are theoretically insensitive to thickness variations, while length extensional modes have a low sensitivity to thickness variations for a device length much greater than the device thickness.

Fig. 4. The elastic constants of doped silicon and their temperature dependences, with p-type doping represented on the left with increasing doping and n-type on the right. The extracted values in this work are compared with values reported in literature.
3) Rotational Misalignment

The device-to-crystal alignment from wafer specifications could be off by as much as 1° in plane, and 0.5° out of plane. The effect on the elastic constants and their temperature dependences can be estimated using (7), (8), and (11) to analytically obtain the uncertainties due to the crystal misalignment. It is seen that the misalignment does not significantly affect the results at these small angles.

4) Temperature Inaccuracies

The chamber temperature varied as much as ± 1°C at -40°C. However, the temperature used in this calculation was measured with an RTD (accuracy of ± 0.15°C), taken at the time of the frequency measurement. To investigate the temperature variation in the chamber, two RTDs on circuit boards similar to the resonator setup were placed at different locations in the chamber and measured simultaneously. It was found that the two RTDs tracked each other to within ± 0.4°C at -40°C and within ± 0.2°C from -30°C to 85°C. An uncertainty of ± 0.4°C at the temperature extremes was propagated to the temperature coefficients using (14) and (15).

5) Linearization Point

The linearization points \( \mathbf{c}_{11,0}, \mathbf{c}_{12,0}, \mathbf{c}_{44,0} \) that were used in the temperature dependence analysis derives from the extracted values for the corresponding doping concentration. The uncertainty of the linearization point was propagated to the temperature coefficients by repeating the finite element analysis using the error bounds of the extracted elastic constants as the linearization points.

6) Thermal Expansion and Density

The thermal expansion coefficients and density of silicon have been assumed to be constant across all dopings in this analysis. Measurements in literature have shown minimal effects due to doping, resulting in uncertainties on the order of a few hundred ppm [6, 19] – small compared to the other uncertainties. Though the thermal expansion coefficient was assumed to be constant, its effect on the temperature coefficients has been accounted for (Section II.A).

7) Electrostatic Spring Softening

The electrostatic actuation force is known to cause a reduction of the spring stiffness proportional to the square of the applied bias voltage, resulting in a lowering of the resonant frequency. This effect was characterized by measuring the resonant frequency at several bias voltages. For the bulk-mode resonators used in this extraction, the frequency shift due to the spring softening effect was under 20 ppm, negligible compared to the other uncertainties detailed above.

V. Predicted Frequency-Temperature Dependence and Verification of Extracted Values

The importance of the extracted temperature-dependent elastic constants is that they allow for the frequency-temperature dependence of doped silicon resonators to be predicted. The temperature dependence of the elastic constants can be accounted for in the material properties of finite element analysis software by substituting in the relevant elastic properties for each temperature step and performing an eigenmode analysis. The fractional frequency change is then
noted for each step, giving the frequency-temperature dependence due to the elastic properties, i.e. the $\frac{df}{f_0}$ component in (2). Finally, the thermal expansion effect is then added back as per (2), resulting in the total frequency-temperature dependence. The code for performing this simulation in COMSOL with MATLAB LiveLink is available on our group’s website [33].

Resonant modes not used in the elastic constant extraction are then used for verification. Since only a few modes (as shaded in Table III) were used for the extraction, predictions were performed for the other available modes, and these predictions are compared to experimental data (Fig. 5). The discrepancies between the predicted and measured values are listed in Table VI, for the resonant frequencies at 25°C as well as the fractional frequency variations across the -40°C to 85°C temperature range. For bulk-mode resonators, the mean resonant frequency discrepancy is about 0.5%, while the maximum discrepancy for the fractional frequency variation across the temperature range is on average around 20 ppm. Larger discrepancies of about 2% and 80 ppm respectively are noted for the flexural mode resonators (DETF and DRG). Since the DETF beams are 6 µm in width, and the DRG consists of numerous thin concentric rings of 3 µm width, fabrication process variation is believed to have a more pronounced effect, leading to larger discrepancies. Another cause could be the dopant out-diffusion that occurs from the oxide-free silicon surface [34] during the high temperature sealing process. The experimental frequency-temperature plots for the DRG correspond to a slightly lower doping concentration than modeled, indicating some dopant loss may have occurred.

VI. CONCLUSION

Silicon resonators have differing frequency-temperature characteristics that are dependent on doping and the resonant mode. Using a suite of different types of resonators, the first and second order temperature dependences of the elastic constants were extracted for p-doped silicon up to 1.7e20 cm$^{-3}$, and n-doped silicon up to 6.6e19 cm$^{-3}$. Encapsulated Lamé resonators at 10 MHz with quality factors of 1.7 million have been temperature compensated to first order using p-type doping, and are additionally seen to have a much reduced second order temperature dependence, as low as -21 ppb/°C$^2$,

compared to n-doped silicon. With the extracted elastic constants, it is seen that the fractional frequency-temperature dependence of arbitrary linear bulk resonant modes with similar doping concentrations can be predicted to about 20 ppm.

REFERENCES


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