A setup for measuring mechanical losses of silicon wafers has been fully characterized from room temperature to 4 K in the frequency range between 300 Hz and 4 kHz: it consists of silicon wafers with nodal suspension and capacitive and optical vibration sensors. Major contributions to mechanical losses are investigated and compared with experimental data scanning the full temperature range; in particular, losses due to the thermoelastic effect and to the wafer clamp are modeled via finite element method analysis; surface losses and gas damping are also estimated. The reproducibility of the measurements of total losses is also discussed and the setup capabilities for measuring additive losses contributed by thin films deposited on the wafers or bonding layers. For instance, assuming that additive losses are due to an 80-nm-thick wafer bond layer with Young modulus about ten times smaller than that of silicon, we achieve a sensitivity to bond losses at the level of $5 \times 10^{-7}$ at 4 K and at about 2 kHz. © 2008 American Institute of Physics.

I. INTRODUCTION

A number of silicon wafer bonding methods have been developed to obtain vacuum sealing and to realize complex, three-dimensional mechanical systems, overcoming the limits of photolithography. In basic research, bonding techniques are applied mainly to realize monolithic objects with high stability and mechanical strength at room temperature. For instance, in the field of gravitational wave experiments, stable platforms and quasimonolithic suspensions are constructed by means of the hydroxy-catalyzed bonding, also known as “silicate bonding.” Originally developed for joining fused-silica parts to realize the star-tracking telescope of the Gravity Probe-B space experiment, this technique is now applied with success to different materials such as sapphire and silicon. While the characterization of bonding techniques is being pursued since several years, little has been done at low temperatures.

In this paper we present an experimental apparatus designed for measuring the mechanical losses which are contributed at cryogenic temperatures and in the acoustic frequency range (100 Hz–10 kHz) by silicon wafer bonding techniques. Our motivation comes from the need of developing very large silicon masses (order of 10 ton) with extremely low mechanical losses for the purpose of constructing a cryogenic DUAL detector of gravitational waves. Silicon is already known to possess very low mechanical loss angles ($\phi \sim 10^{-8}–10^{-9}$) from room to cryogenic temperatures but no measurements of losses contributed by bonding methods are available at such low temperatures.

The loss of any elastic body is the sum of different contributions that can be generally divided into three main categories: fundamental losses $\phi_{\text{fund}}$ common to even perfect materials (i.e., materials with perfect crystal lattices), intrinsic losses $\phi_{\text{int}}$ due to material imperfections, and losses $\phi_{\text{ext}}$ due to coupling to the external environment. The total losses can thus be detailed as

$$\phi_{\text{tot}} = \phi_{\text{fund}} + \phi_{\text{int}} + \phi_{\text{ext}}.$$  

(1)

Low-loss measurements are often limited by $\phi_{\text{ext}}$. In the work we report here we set up a standard procedure for mechanical loss measurements: our main concern is to minimize $\phi_{\text{ext}}$ by isolating the sample under measurement from the lossy external environment and to understand the different contributions that sum up in the measured losses. We characterize our setup in the whole temperature range between 300 and 4 K stating the minimum losses contributed further by the bond layer and that we can measure on silicon wafers.

While our main concern regards mechanical losses due to bond layers, this setup is also well suited for characterizing additive losses contributed by other processes on silicon, such as surface coatings and other postproduction treatments.

II. THE EXPERIMENTAL APPARATUS

Our experimental apparatus consists of mechanical oscillators realized in the material under investigation, i.e., silicon for the purpose of the work reported here: each oscillator is supported by a holder which is fixed to the final part of the same mechanical suspension (see Fig. 1) and housed in the experimental chamber [internal vacuum chamber (IVC)] of a cryogenic facility; a system for exciting mechanically the
oscillators is available as well as nontouching sensors for measuring the displacements of the oscillators.

The cryogenic facility was developed for the purpose of testing the capacitive transducer and superconducting quantum interference device amplifier used in the detector of gravitational waves AURIGA (Ref. 9) and thus it is named Transducer Test Facility (TTF); it is located at the Legnaro National Laboratories of Istituto Nazionale di Fisica Nucleare, near Padova, Italy. While the work reported in this paper covers the temperature range from 300 to 4 K, the TTF is also equipped with a dilution refrigerator: extensions of the measurements we report here down to 50 mK will be performed in the near future. The TTF has an experimental chamber (IVC) of \(420 \times 700 \text{ mm}^2\): this hosts mechanical suspensions optimized for frequencies around 900 Hz, where they provide \(-170 \text{ dB}\) of attenuation from floor vibrations along the three orthogonal axes. The mechanical suspensions hung from the top: at their bottom they support the experimental apparatus. A view port on the center of the experimental chamber is available which is connected to the top of the TTF in air through a pipe line: it is used for the optical sensor (see Sec. II C 1). The TTF is described in more details in Ref. 10.

Figure 1 shows a view of the whole experimental apparatus: at the top of the TTF in air, a plate supports the optical components for the optical sensor described in Sec. II C 1. The lower part of the vacuum chamber is available for the experiment: at the bottom of the suspension, three rectangular-section rods support an aluminum disk (height 50 mm, diameter 300 mm) on top of which the oscillators and the capacitive sensor (see Sec. II C 2) are mounted. We refer to this disk as the “base.” A second, lower base was fixed to the first one by means of three copper rods: this lower level of the experimental setup was dedicated to measurements of mechanical dissipations on SiC samples but we do not report them in this paper. In the following we describe in more details the experimental apparatus which is specific to the research we report here.

One rectangular piezoelectric actuator is fixed to the base (see Fig. 1); it is mounted horizontally and a brass mass of 330 g is glued to its top so to increase its efficiency as force actuator. This actuator is used to excite the oscillators, as discussed in Sec. III.

In order to perform measurements at intermediate temperatures between room temperature and the liquid nitrogen temperature and between this and the liquid helium temperatures, we fixed three heaters to the curved surface of the base, about 120° apart (see Fig. 1): each heater consists of a Manganin wire wound around a copper core. The heaters are connected in parallel and the resulting resistance is 25 \(\Omega\). The heaters are the actuators of a feedback loop that vanishes the difference between a set temperature and the reading of a silicon thermometer (T1 in Fig. 1) mounted on the circular surface of the base: the loop is closed by a temperature controller (LakeShore 340) in air which regulates the current through the heaters. We never exceed a power of 25 W through the heaters to avoid damaging the heating device. A second silicon thermometer (T2 in Fig. 1) is placed on the lower base that supports the SiC samples.

After collecting the measurements of mechanical losses we performed a cryogenic run to calibrate the sample temperature against the base temperature. For this purpose we fixed a thermometer (LakeShore, model CX-1050) close to the outer diameter of the wafer with hole H2 (see Sec. II B) by means of silver paint; we also screwed a similar thermometer on the base close to the wafer holder. Then we performed a temperature scan and recorded the readings of the two thermometers, along with the reading of the temperature feedback thermometer T1. The temperatures relative to all measurements reported in the following are that of the sample, inferred from the reading of the base temperature and this temperature calibration.

A. The oscillators

To perform an intensive experimental campaign we decided to adopt the geometry of thin circular disks (wafers) for the silicon samples: this simple geometry is the standard in the fabrication of semiconductor devices such as integrated circuits and it is thus largely available. In particular, we focused on single crystals of \(n\)-type silicon in the form of wafers, polished on both sides, with a radius of 100.2 \(\pm\) 0.1 mm and a thickness of 500 \(\pm\) 15 \(\mu\)m. They were doped with phosphor to an impurity concentration of \(10^{14} - 10^{15} \text{ cm}^{-3}\) (resistivity \(\sim 9 - 24 \Omega\) cm). The crystal orientation of the wafer, defined by the plane of its top surface, is \{100\}. According to production standards, each wafer was shaped with a flat cut perpendicular to the \{110\} crystallographic plane. In order to symmetrize the geometry and make the center of the wafer circumference to coincide with the oscillator center of mass, we refined the original cut and applied a second, identical cut, parallel to the first one: we used a diamond saw blade in this process. We measured the distance between the flats to be 94.8 \(\pm\) 0.1 mm. As discussed in Sec. II B, the symmetry of the oscillator is an important detail for realizing the central, nodal holder. The wafer geometry is shown in Fig. 2. The wafers exhibit a number of resonant modes in the acoustic frequency range of our interest (see Table I).
Almost all thermomechanical properties of silicon, used as the input values for the numerical analysis discussed in the following, are taken from Ref. 11. The thermal conductivity values of n-type silicon below 100 K was found in Ref. 12. We also note that silicon is an anisotropic crystal, with cubic symmetry; so its Young’s modulus varies along different directions in the material relative to the crystal orientation. In the crystal axis coordinate system, its elastic properties can be described by a 6 x 6 symmetric stiffness matrix $C_{ij}$ with only three independent constants: $C_{11} = C_{22} = C_{33} = 167.5$ GPa, $C_{12} = C_{13} = 65.0$ GPa, and $C_{44} = C_{55} = C_{66} = 80.1$ GPa. These values determine the following values for the Young modulus in the plane of our (100) wafer: parallel to flat, $Y^p = 169$ GPa and 45° diagonal to flat, $Y^d = 130$ GPa.

B. Oscillator holder

In order to minimize mechanical losses that might be contributed by the sample holder, we decided to minimize the contact area between sample and holder and to realize a nodal contact between the two. Nodal contact means that the sample is supported at a point which remains at rest when the sample oscillates; so the holder does not exert any reaction force. In fact, the holder is an elastic body, which can apply the reaction force only by changing its dimensions, according to its stress-strain relations. If a lossy, not nodal holder participates in the motion, it dissipates part of the sample energy, thus increasing the total losses. The experimental implementation of a nodal contact between sample and holder is obviously approximated and hence we expect the latter to contribute to the total mechanical losses: this issue is discussed in detail in Sec. V C.

To realize a central nodal holder we sandwiched the wafer between two aluminum plates, connected by two screws (see top of Fig. 3): the wafer is kept in the horizontal position. Similar to Ref. 14, the contact between the plates and the wafer is realized by two balls (1 mm in diameter) made of sapphire, a very low-loss material, the bottom ball is simply placed on top of a small dip at the center of the lower plate while a steel spring (elastic constant $=0.75$ N/mm) connects the top ball to the top plate (see top of Fig. 3). Acting on the two screws the position of the top plate is lowered by $\sim 2$ mm: the spring is thus compressed and it exerts a vertical force of 1.5 N on the top ball, thus holding the wafer in place. We took great care to guarantee that the imaginary line passing through the center of the top and bottom balls is perpendicular to the wafer and passes through its center: this is critical in order to avoid damaging the wafer when uncontrolled tilt is applied and to realize a central suspension. To this end we adopted two solutions (see Fig. 3): (H1) we drilled a through hole (0.5 mm in diameter) at the center of the wafer using a dental tool (this is done after the flat cuts are applied as discussed in Sec. II A) and (H2) we excavated two cuts at the center of both sides of the wafer via chemical wet etching (this is done before the flat cuts are applied as discussed in Sec. II A). Since it follows the $\{111\}$ crystal plane, the latter solution ends up in pyramidal cuts, the contact area between sample and holder and to realize a nodal contact between the two.

TABLE I. Resonant frequency in hertz of the first acoustic modes of the wafer shown in Fig. 5: FEM estimation (second column, see Sec. 4) and room-temperature experimental measurement for the sample with H1 hole (third column) and H2 hole (fourth column). The fifth column describes the acoustic modes as labeled in Fig. 5. The sixth column lists the loss factor measured at about 7 K on the sample with hole H1.

<table>
<thead>
<tr>
<th>Mode No.</th>
<th>$\nu_{\text{FEM}}$</th>
<th>$\nu_{H1}$</th>
<th>$\nu_{H2}$</th>
<th>$(a, n, s)$</th>
<th>$\phi_{\text{cop}}^2$ K</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>275.4</td>
<td>275.3</td>
<td>275.3</td>
<td>(0, 0, 0)</td>
<td>$1.1 \times 10^{-7}$</td>
</tr>
<tr>
<td>2</td>
<td>386</td>
<td>380.1</td>
<td>383.0</td>
<td>(2, 0, x)</td>
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<tr>
<td>3</td>
<td>483</td>
<td>489.3</td>
<td>484.6</td>
<td>(2, 0, +)</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>984</td>
<td>972.9</td>
<td>975.7</td>
<td>(3, 0, x)</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>997</td>
<td>985</td>
<td>989</td>
<td>(3, 0, +)</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1518</td>
<td>1478</td>
<td>(0, 1, 0)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1543</td>
<td>(1, 0, x)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1607</td>
<td>(1, 0, +)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
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<td>1721</td>
<td>1720</td>
<td>(4, 0, x)</td>
<td>$1.3 \times 10^{-8}$</td>
</tr>
<tr>
<td>10</td>
<td>1766</td>
<td>1784</td>
<td>1772</td>
<td>(4, 0, +)</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>2539</td>
<td>2500</td>
<td>2515</td>
<td>(2, 1, x)</td>
<td>$1.1 \times 10^{-7}$</td>
</tr>
<tr>
<td>12</td>
<td>2649</td>
<td>2633</td>
<td>2629</td>
<td>(5, 0, x)</td>
<td></td>
</tr>
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<td>13</td>
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<td>2805</td>
<td>2846</td>
<td>2814</td>
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<td></td>
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<td>15</td>
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<td>3701</td>
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</tr>
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<td>3744</td>
<td>3703</td>
<td>3712</td>
<td>(6, 1, +)</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>3950</td>
<td>3924</td>
<td>3923</td>
<td>(3, 1, x)</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>3995</td>
<td>(3, 1, +)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
175 µm deep and with square section having a side of 0.5 mm at the surface.

Hole H2 is obtained using the microelectromechanical system technologies available at the Fondazione Bruno Kessler-IRST in Trento, Italy: initially, a 500-nm-thick thermal silicon oxide layer and a 100-nm-thick low pressure chemical vapor silicon nitride layer are, respectively, grown and deposited on both wafer sides. Then a central hole is opened in the oxide/nitride layers via photolithography followed by a plasma dry etching; this etching stops selectively at the silicon surface. The silicon is then etched with wet tetramethylammonium hydroxide (TMAH) 25% 90 °C solution for several hours on each side: TMAH is highly selective with respect to the oxide nitride mask. Once the central holes (one per wafer side) have the desired size, the mask layers are removed.

C. Readouts

We employed two displacement sensors that do not touch the wafers to minimize sensor contribution to mechanical losses: one is based on the beam deflection technique and one on capacitive strips. The sensors are vacuum compatible and work in the full temperature range from 300 to 4 K. The position of the sensors relative to the wafers was not optimized with respect to the acoustic modes under measurement. In the following the two sensors are described.

1. Beam deflection sensor

The beam deflection sensor\textsuperscript{16} consists in monitoring the deflection of a laser beam by a displaced reflecting surface with a split photodetector. This technique works for any material but it is better suited for highly reflective ones so that the sample under measurement is not heated by absorption of the incident laser beam or of scattered light. A 370×250 mm\textsuperscript{2} aluminum plate is placed horizontally on top of the TTF, in air (see Fig. 1): it supports a 100 mW cw neodymium-doped yttrium aluminum garnet laser, focusing optics and a split photodiode (partially shown in Fig. 1). The light path is folded twice on the plate before entering a beam splitter with 25% reflectivity: the transmitted beam is directed downward and, after passing through a 1 in. optical window that realizes the vacuum sealing, it reaches the oscillator with hole H2 about 2 m below. The upward beam reflected by the oscillator escapes from the same optical window and the part transmitted by the beam splitter is directed to a quadrant photodiode (EG&G 30845). The resulting four photocurrents are combined so to measure the change in the electrical signals of top/bottom or left/right parts of the photodiode. The laser spot size at the sample is about 0.2 mm. At 1 kHz the sensor is sensitive to about 1 nm of vertical movement of the wafer reflecting surface. During the temperature calibration run we confirmed that the sample heating due to the impinging laser beam is negligible at 4.2 K, as expected from the high reflectivity of silicon.

The laser beam deflection method is widely employed because of the simple experimental setup and the easy optical beam alignment; in our case its usage is complicated by the configuration of the experimental chamber and of the TTF (see above). In particular, the beam must propagate on the same 40-mm-diameter tube in both downward and upward directions and pass through the same 1-in.-diameter window; the minimum distance between the oscillator and the nearest optics is about 2 m and for most part of it there is no access to the beam for monitoring its propagation. Moreover it is not possible to perform measurements on different oscillators during the same run since only one laser beam is available with fixed path. This is the main reason why we decided to develop the capacitive strips.

2. Capacitive strips

In alternative to the optical sensor, we developed a capacitive displacement sensor for measurements on dielectric materials. To this purpose we adopted the idea of the capacitive strips reported in Ref. 17, using them as sensor. This consists in an array of 84 coplanar, rectangular strips, resembling a bar code (see Fig. 4); it is obtained by machining with a circuit board plotter a 10-µm thick copper film on top of a commercial fiberglass substrate of thickness h=1.6 mm. The strips have width a=0.4 mm, length L=10 mm and are displaced by a period b=1.2 mm: the total area is 1×5.2 cm\textsuperscript{2}. In the experiment only half of the strips were faced to the silicon samples. The strip board is glued to an aluminum block which is fixed with screws to the same platform supporting the oscillators; we estimate the gap between the strip plane and the wafer with hole H1 at its bottom to be 80±20 µm.

Figure 4 also shows the electrical scheme of the sensor: half of the strips are connected to ground while the other half are connected to one side of a decoupling low-loss, high-voltage capacitor C\textsubscript{d}=150 nF, which is placed on the lowest part of the mechanical suspension of the TTF (see Fig. 1): the voltage between this and ground constitutes the signal. A charging line is also present (see Fig. 4): at the beginning the sensor output is short-circuited and the charge line is connected (i.e., both switches in Fig. 4 are closed) to a high-voltage generator providing a bias voltage V\textsubscript{0}=80 V. Then the short across the sensor is removed and the high-voltage generator is disconnected, leaving a constant charge on the strips.

A relative movement of the sample with respect to the sensor plane generates a change in the charge distribution on
the strip electrodes and in the capacity of the strip array, since the total charge is constant; the sensitivity is about $2 \times 10^4 \text{ V/m}$.

### III. EXPERIMENTAL PROCEDURE

When measuring the elastic properties of solids through mechanical oscillators, it is desirable to study more than a single frequency. The first 18 modes of our silicon wafer resonate at frequencies below 4 kHz, but only a few of them turn out to have small external losses $\phi_{\text{ext}}$ (see Sec. V C) and can be actually used to evaluate the intrinsic properties of the material.

For each temperature point, we waited several hours to allow for thermalization of the samples, at a pressure of $10^{-6} \text{ mbar}$. Then for each mode we excited the resonant vibration by feeding a monochromatic signal to the piezoelectric actuator fixed to the base (see Fig. 1); amplitude of the excitation wave ranges between 40 and 600 V p.p. The output of the employed displacement sensor is demodulated by an SR830 lock-in amplifier which mixes it with a reference signal phase locked to the excitation signal; for the excitation and reference signals we use HP3325B synthesizer with high stability frequency reference. Before feeding it to the actuators, the excitation signal is amplified by a custom high-voltage amplifier. The demodulated signal is acquired via GPIB (IEEE 488) with sampling rate ranging from 0.125 Hz (for lower-loss measurements) up to 4 Hz (for higher-loss measurements). We actually have three such signal filtering systems so that we can monitor up to three modes at the same time. The fact that an acoustic mode can be excited by shaking the wafer holder indicates that the contact between wafer and holder is not perfectly nodal for that mode (see discussion in Sec. V C 2).

For each acoustic mode of the sample we obtain a first estimation of the resonant frequency from the excitation frequency that minimizes the decay of the phase of the demodulated signal. Let $\nu_m$ be the resonant frequency of a mode; if this is excited by a monochromatic signal of frequency $\nu_s$ and then at time $t=0$ suddenly the excitation signal is removed, the phase $\phi$ of the demodulated signal evolves in time $t$ as

$$\phi(t) = \phi_s + 2\pi(\nu_s - \nu_m)t,$$

where $\phi_s$ is a constant. Correspondingly the mechanical vibration follows an exponentially damped decay whose envelope amplitude varies according to

$$u(t) = u_0 \exp\left(-\frac{t}{\tau}\right).$$

Once the first estimation of the resonant frequency is obtained for a given mode, we remove the excitation signal to the piezoelectric actuator and acquire both the phase and amplitude of the demodulated signal. The final measurement of the mode resonant frequency is obtained from a fit of the time evolution of the phase once the excitation signal is switched off, according to Eq. (2). The time constant of the lock-in is of the order of 1 s; since the linewidths that we measure are $\lesssim 0.05 \text{ Hz}$, the bandwidth of the lock-in amplifier is large enough to cover the whole mode linewidth with large margins. Therefore our sensitivity to frequency drifts of the reference signals are much reduced in the amplitude signal: still they would appear as a contribution to the time evolution of the phase in addition to what expected from Eq. (2). The experimental data are listed in Table I, along with their estimation via a finite element method (FEM) analysis (see Sec. IV): the agreement of the numerical predictions with experimental data is good. Table I also shows the difference in the measured mode resonant frequencies obtained between the two samples: one with hole H1 and the other with hole H2 (see Sec. II B). By exchanging the samples with respect to the sensors, we made sure that the reported differences are reproducible and do not depend on the sensor technology. The relative difference varies with the mode shape and never exceeds about 1%.

An exponential fit of the time evolution of the demodulated amplitude gives the decay time $\tau_s$ [see Eq. (3)] and hence the mechanical loss angle at the frequency $\nu_s$ via the simple formula

$$\phi(\nu_s) = \frac{1}{\pi \nu_s \tau_s}.$$  

For all measurements the time span of the fitted data covers at least two decay times: for instance, this means that acquisition time for a single decay at a typical frequency of 1 kHz and with loss $\phi=10^{-7}$ is $\approx 2 \text{ h}$. Therefore to speed up the measurement campaign we measure the decay time of three different modes of the same oscillator at the same time, having three lock-in amplifiers, as explained above: while a mode is decaying we can excite a second mode, find its resonant frequency, and follow its decay, without disturbing the measurements of the first mode, provided the two modes do not resonate at similar frequencies.

### IV. ELASTIC MODEL OF THE OSCILLATOR

The equations describing oscillations of a thin circular disk are usually written under the Kirchhoff hypotheses.\(^8\)

(a) The straight lines, initially normal to the middle plane before bending, remain straight and normal to the middle surface during the deformation, and the length of such elements is not altered. This means that the vertical shear strains $\varepsilon_{xz}$ and $\varepsilon_{yz}$ are negligible. This assumption is referred to as the “hypothesis of straight normals.”

(b) The stress normal to the middle plane, $\sigma_z$, is small compared with the other stress components and may be neglected in the stress-strain relations.

Here $z$ is the axis normal to the disk while $x$ and $y$ are orthogonal axes parallel to the disk, the origin of the coordinate system is chosen at the center of mass of the disk hence its middle plane is at $z=0$. We also note that because the disk is thin, the in-plane (tangential and extensional) vibrations are decoupled from the out-of-plane (transverse) vibrations.

Let $w(x,y)$ represent the displacement of the middle
plane along the $z$ axis and $h$ the disk thickness. The engineering strain components in the disk due to this transverse displacement are given as\textsuperscript{18}

\[
\begin{align*}
\varepsilon_x &= \gamma_z \frac{h}{2}, \\
\varepsilon_y &= \gamma_z \frac{h}{2}, \\
\gamma_{xy} &= -2(\partial^2 W/\partial y^2).
\end{align*}
\]  

(5)

where $-h/2 \leq z \leq h/2$ and the functions $\{k\}$ represent the middle surface curvature of the plate:

\[
\begin{align*}
\begin{bmatrix}
\kappa_x \\
\kappa_y \\
\kappa_{xy}
\end{bmatrix} &= \begin{bmatrix}
-\partial^2 W/\partial x^2 \\
-\partial^2 W/\partial y^2 \\
-2(\partial^2 W/\partial x \partial y)
\end{bmatrix}.
\end{align*}
\]  

(6)

The strain variations [Eq. (5)] determine the stress components through the relation

\[
\begin{bmatrix}
Q_{11} \\
Q_{12} \\
Q_{16}
\end{bmatrix} = Y \begin{bmatrix}
1/(1 - \sigma_p^2) & \sigma_p/(1 - \sigma_p^2) & 0 \\
\sigma_p/(1 - \sigma_p^2) & 1/(1 - \sigma_p^2) & 0 \\
0 & 0 & 1/2(1 + \sigma_p)
\end{bmatrix}
\begin{bmatrix}
\sigma_x \\
\sigma_y \\
\gamma_{xy}
\end{bmatrix},
\]

(7)

where $Q_{ij}$ ($i,j = 1, 2, 6$) are the reduced stiffnesses for a plane stress state in the $x$-$y$ plane, obtained by the full stiffness matrix $C_{ij}$ of the material under hypotheses (a) and (b):

\[
Q_{ij} = C_{ij} - \frac{C_{13}C_{33}}{C_{33}}.
\]

(8)

The constitutive equations [Eq. (7)], together with the differential equations of equilibrium and the proper boundary conditions, represent the governing equations of a generic disk. In the case of an isotropic material these equations admit an analytical solution\textsuperscript{19} which we review here to obtain a better insight into the normal modes of our disk. In this case the $Q_{ij}$ can be simplified considerably and written in terms of the Young modulus $Y$ and of the Poisson modulus $\sigma_p$:

\[
\begin{bmatrix}
\sigma_x \\
\sigma_y \\
\gamma_{xy}
\end{bmatrix} = \begin{bmatrix}
Q_{11} & Q_{12} & Q_{16} \\
Q_{12} & Q_{22} & Q_{26} \\
Q_{16} & Q_{26} & Q_{66}
\end{bmatrix}
\begin{bmatrix}
\varepsilon_x \\
\varepsilon_y \\
\gamma_{xy}
\end{bmatrix}.
\]

(9)

Then we search for solution where the displacement in the $z$ direction can be written as

\[
W(r, \theta, t) = W(r, \theta)e^{i\omega t},
\]

(10)

where $r - \theta$ are the polar coordinates, $t$ is the time coordinate, and $\omega$ is the angular frequency. In this case the governing equation of the free transverse vibration writes as

\[
\nabla^4 W = \frac{\rho h \omega^2}{D} W.
\]

(11)

Here $\rho$ is the density and $D$ is the flexural rigidity of the disk

\[
D = \frac{Y h^3}{12(1 - \sigma_p^2)}.
\]

(12)

The solutions of Eq. (11) can be written as

\[
\begin{align*}
W^r(r, \theta) &= \cos(a \theta)[B_n^{(a)} I_n(\beta_n^{(a)} r) + C_n^{(a)} I_n(\beta_n^{(a)} r)], \\
W^\theta(r, \theta) &= \sin(a \theta)[B_n^{(a)} I_n(\beta_n^{(a)} r) + C_n^{(a)} I_n(\beta_n^{(a)} r)],
\end{align*}
\]

(13)

\[
\begin{align*}
\beta_n^{(a)} &= \left. \frac{d}{dr} \right|_{r=1} I_n(\beta_n^{(a)} r), \\
a &= 0, 1, 2, \ldots, k,
\end{align*}
\]

(14)

where $J_n$ is the $n$th order Bessel function of the first kind and $I_n$ is the $n$th order modified Bessel function of the first kind. For each integer $a$, the constants $B_n^{(a)}$, $C_n^{(a)}$ and the eigenvalue $\beta_n^{(a)}$ are obtained when the boundary condition on the motion is applied to the solution. In our experiment the disk center is at rest due to the reaction from the support, while the disk circumference is free. The eigenfrequency of the mode is then obtained by the relation $\beta_n^{(a)} = \sqrt{\rho h (a \beta_n^{(a)})^2}/D$. The value of $a$ in Eqs. (13) and (14) indicates the number of nodal diameters, $n$ the number of nodal circles, and $s = +, \times$ is the symmetry index of the displacement fields; a solution is fully identified by the set $(a, n, s)$. The orthogonal displacement fields $W^r(r, \theta)$ and $W^\theta(r, \theta)$ represent mode doublet: they have the same radial distribution of the deformation, mutually rotated by $\pi/2a$, and share the same eigenvalue $\beta_n^{(a)}$. When $a = 0$ the doublet merges in a single solution with rotational symmetry around the $z$ axis.

In the case of silicon the solutions shown in Eqs. (13) and (14) are approximated, because the crystal is anisotropic and its Young’s modulus along a given direction depends on the orientation with respect to the crystal axes. To take into account the material’s anisotropy and the real geometry, we developed a FEM model of the wafer using a commercial software, using the stiffness matrix described in Sec. II A. Similar to the experimental apparatus, in the model the wafer is constrained on both sides, on two small circular areas with diameter 0.5 mm, along its $z$ symmetry axis.

The stiffness anisotropy and the flat cuts induce a splitting of the frequency of the mode doublets. This splitting is quite large for $a = 1$, when the modes are mutually rotated by $\pi/2$ and the effect of the flat cuts is maximized, and for $a = 2$, when the modes are mutually rotated by $\pi/4$ and the effect of the stiffness anisotropy is maximized. We show in Fig. 5 the model plots of the first 13 eigenmodes with solution of the type described by Eq. (13) and (14). The $(a, n, s)$ indices of the corresponding mode in the isotropic case are also shown. We note that the FEM analysis predicts the ex-
istence of “cantilever” modes of the disk pivoting about its suspension point, which cannot be described by periodic functions in $H_9^{25}$; these modes resonate at frequencies that depend critically on the suspension details and they were not experimentally investigated.

V. ESTIMATION OF LOSS CONTRIBUTIONS

A. Fundamental losses

In insulating solids such as silicon there are two fundamental mechanisms that set the lower limit to the total mechanical losses: the thermoelastic effect and the phonon-phonon interaction. Since the latter contributes a negligible loss in our case, we do not investigate it further.

The thermoelastic effect was investigated first by Zener; in the presence of a nonzero coefficient of thermal expansion, when a solid undergoes a vibration other than pure torsion, the strain field generates a thermal gradient and thus a heat flow which dissipates elastic energy. The expected losses depend on the geometry of the elastic structure; it can be demonstrated that for pure flexure the thermoelastic loss is

$$\phi_{\text{th}}(\omega) = \frac{Y\alpha^2 T}{\rho C_V} \frac{\omega \tau_{\text{th}}}{1 + \omega^2 \tau_{\text{th}}^2},$$

(15)

where $\alpha$ is the thermal expansion coefficient, $C_V$ the specific heat per unit volume of the material, and $T$ is the temperature. The loss factor due to the thermoelastic effect depends on the oscillator dimensions through the relaxation time $\tau_{\text{th}}$:

$$\tau_{\text{th}} = \frac{h^2}{\kappa C_V},$$

(16)

where $h$ is the oscillator thickness and $\kappa$ the thermal conductivity. Given our geometry the formula of Eq. (15) cannot be applied. To estimate the thermoelastic damping of all modes under measurement, we performed a FEM analysis using a commercial software: thus we could take into account of both the material anisotropy and the mixture of flexural and torsional strains. We performed a structural-thermal harmonic analysis at room temperature, forcing one mode at a time. The model is built using the coupled-field thermoelastic analysis options of a three-dimensional 20-node solid element, and the convergence of the analysis was checked against the mesh density.

The results are shown in Fig. 6, where the measured losses at room temperature are compared to that evaluated by the FEM analysis. The agreement with experimental data is within 10% for the majority of the modes, confirming that thermoelastic damping is the main dissipative phenomenon at room temperature in our oscillator.
This FEM model allows us to compute the thermoelastic damping at a given temperature simply by using the appropriate thermomechanical properties of silicon: we get these from Refs. 11 and 12 for the temperature range between 300 and 4 K. In Fig. 7 we show as a gray line the losses expected for mode No. 9 as effect of the thermoelastic damping and computed via the FEM analysis: due to the large number of computations involved here, a coarser mesh was employed with respect to what was shown in Fig. 6. This accounts for the discrepancy between the predictions for mode No. 9 at room temperature between Figs. 6 and 7. The thickness of the gray line in Fig. 7 accounts for the uncertainty in our estimates due to variation in thermal conductivity between different silicon samples. In Fig. 7 we also show the losses measured for modes Nos. 2, 9, and 11 (as labeled in Table I), obtained by scanning the sample temperature between 300 and 4 K: overall the agreement of the experimental data for mode No. 9 and our FEM prediction is very good above 40 K, indicating that the thermoelastic damping originates the dominant loss contribution above 40 K. In this temperature range the only deviation is observed at about 120 K where the coefficient of linear expansion of silicon vanishes and so does the thermoelastic mechanism [see, for instance, Eq. (15)]: the observed local minimum is set by a different mechanism (see, for instance, Sec. V C 2). Similarly below 40 K the measured losses of mode No. 9 are larger than those expected from the thermoelastic damping, indicating a different dominant loss contribution. The losses of mode No. 11 show a dependence with temperature similar to that of mode No. 9, indicating a similar explanation for the dominant loss mechanism. Mode No. 2 has a smoother dependence with temperature, suggesting that it is dominated by different damping mechanisms.

**B. Intrinsic losses**

Bulk silicon is demonstrated to possess extremely low mechanical losses both at room temperature$^7$ and 4 K.$^8$ Thus we do not expect that crystal defects contribute significantly to the total losses of our samples. On the contrary surface losses might be a limiting factor: since the source of this damping is still not clear, in order to estimate it, we compare our oscillator to other silicon ones of different volume-to-surface ratios.

In Ref. 22 authors collect from the literature data of room-temperature losses for single-crystal silicon oscillators of different dimensions: in Fig. 5 of Ref. 22 they summarize such information and evidence a dependence of $1/\phi$ versus oscillator size for oscillators such as ours which have not been annealed or for which reactive-ion etching has been involved. From this collection we infer that with a thickness of 500 $\mu$m, surface losses at room temperature should be $\phi_s \approx 10^{-7}$. For the modes that show the lowest losses at room temperature this figure is not far from the experimental values (see Fig. 6): thus for such modes, surface losses might give a not negligible contribution at room temperature, even though not the dominant one (see Fig. 6 and related discussion).

In Fig. 8 we plot the loss angle against the smallest oscillator dimension for a number of silicon oscillators of different shapes found in the literature:$^8,23–28$ all measurements are taken at temperatures in the range 1–15 K. For each group of researchers (i.e., for each oscillator design) we quote the smallest reported loss angle. The dependence of the measured losses on the size (or, equivalently, the volume-to-surface ratio) suggests a relevant damping mechanism from the surface. Our measurement of the losses of mode No. 9, also shown in Fig. 8, does not depart significantly from this trend: thus we conclude that surface losses explain at least part of the total losses for mode No. 9 at liquid helium temperatures.

**C. External losses**

1. Gas damping

The residual gas present in the vacuum chamber that hosts the oscillators constitutes an additional source of mechanical losses: in the free-molecule regime, where we performed all our measurements, they are due to the momentum which is transferred from the vibrating body to the gas molecules during the collisions. According to the model by Christian$^{29}$ which assumes a Maxwell–Boltzmann distribution for the gas velocity, if this mechanism were the limiting one then we would measure loss angles given by
\[
\phi_{\text{gas}} = \left( \frac{2}{\pi} \right)^{3/2} \frac{1}{\rho \nu_m} \sqrt{\frac{M_e}{RT}} \frac{P}{P_{\text{bar}}},
\]

where \( R \) is the gas constant, \( M_e \) is the molar mass of the residual gas, and \( P \) is the pressure. In the last equality we consider helium to be the largest component of the residual gas since we use it to thermalize the cryogenic liquid bath of the TTF; \( P_{\text{bar}} \), \( \nu_{\text{Hz}} \), and \( T_{(K)} \) are, respectively, the values of pressure in mbars, of frequency in hertz, and of temperature, in kelvins. Thus at \( T=4.2 \text{ K} \), \( P=10^{-6} \text{ mbar} \) we get \( \phi_{\text{gas}}=1 \times 10^{-9} \) for mode No. 2 and \( \phi_{\text{gas}}=3 \times 10^{-10} \) for mode No. 9.

The model of Ref. 29 assumes that the sample is placed in an infinitely large volume; if the distance \( d \) between the walls and the sample becomes smaller than the typical dimension \( L \) of the vibrating body, then a larger number of collisions should be expected and hence the damping should be larger. In Ref. 30 for a rectangular plate the damping in this so-called squeezed-film regime is expressed as

\[
\phi_{sg} = \phi_{\text{gas}} \frac{L}{16 \pi d}. \tag{18}
\]

This damping mechanism is present in the sample measured by the strip sensor: using \( d=0.08 \text{ mm} \) and \( L \sim 25 \text{ mm} \), i.e., respectively, the gap between the strips and the sample and the length of the strip section which is faced to it, we calculate \( \phi_{sg}=6\phi_{\text{gas}} \). At \( T=4.2 \text{ K} \), \( P=10^{-6} \text{ mbar} \) we get \( \phi_{sg}=8 \times 10^{-9} \) for mode No. 2 and \( \phi_{sg}=2 \times 10^{-9} \) for mode No. 9.

Our estimates of the damping from the residual gas at \( 4 \text{ K} \) for mode Nos. 2 and 9 are about a factor of 10 below the experimental measurements (see, for instance, Fig. 7), thus suggesting a negligible contribution. On the other hand, it should be noted that the above estimates should be refined taking into account the real geometry, the different modal shapes, and the surface outgassing; moreover, given the position of our vacuum gauge which is on top of the TTF at room temperature, we underestimate the residual gas pressure in Sec. IV C and thus our estimates of the gas damping are affected by a systematic error.

Therefore to measure the size of the combined gas damping effects and check the above estimates, we performed measurements of the loss angle at \( 77 \text{ K} \) of the sample with hole H1, i.e., that read by the capacitive strips, as function of the pressure in Sec. IV C chamber for mode Nos. 2, 9, and 15 of Table I; the data are shown in Fig. 9. We infer that at \( 77 \text{ K} \) the gas damping is negligible below \( 10^{-4} \text{ mbar} \) for frequencies \( \gg 1.7 \text{ kHz} \); resonating at a lower frequency, mode No. 2 shows a dependence of the loss angle on the residual pressure even in the \( 10^{-6} \text{ mbar} \) range where we performed most of the measurements reported in this paper. For this mode the data above \( 10^{-3} \text{ mbar} \) are proportional to the pressure, as indicated by Eqs. (18) and (17); at lower pressures the proportionality is lost, but a power-law trend (with exponent \(<1\)) is still present.

We also repeated the measurements at \( 77 \text{ K} \) of the loss angle of mode No. 2 as function of pressure (in the range \( 0.006-0.03 \text{ mbar} \) and at \( 10^{-6} \text{ mbar} \)) but on the sample read by the beam deflection sensor (sample with hole H2): this is not expected to suffer from the squeezed-film damping.

Above \( 0.006 \text{ mbar} \) the losses measured with the optical read-out are about a factor of 0.2 smaller than the isobar measurements taken with the capacitive readout: this suggests in the latter a significative contribution from the squeezed-film mechanism. On the other hand at \( 77 \text{ K} \) the loss angles of mode No. 2 measured at \( 10^{-6} \text{ mbar} \) with the two sensors differ by less than 30% and amount to about \( 2.5 \times 10^{-7} \). Thus at such pressure we can rule out the (squeezed-film) damping from residual gas as dominant contribution even for mode No. 2. To establish firmly the size of the gas damping effect at \( 4 \text{ K} \), a similar experimental campaign should be repeated at that temperature; on the basis of the above estimates we expect not a large effect.

2. Clamping losses

A rough estimation of the interaction between each acoustic mode and the holder can be quantified by considering the elastic energy dissipated by the holder when this moves. A displacement in the holder can be induced by a not perfectly nodal suspension or by a change in the thickness \( h \) of the wafer where it is in contact with the sapphire balls. A reliable FE model of the complete system cannot be developed, due to the presence of pressed contacts and springs. Thus for each mode we estimate the goodness of the nodal suspension by evaluating the maximum thickness change during one period induced when only one face of the disk is constrained in the central area. The surface of the wafer is constrained on one side over a circular area of diameter \( 0.3 \text{ mm} \). The displacement of the points along the symmetry axis of the wafer is constrained along the \( z \) axis, to avoid rocking movements. The model, with the constraints and the measured area, is shown in Fig. 11. To catch reliably second order effects we took care in meshing the central area of the disk where we used about 2000 elements. The model consists of 16 500 elements in all, and we checked the convergence of the results against the mesh density.

With this model we performed a number of harmonic analysis, forcing one mode at a time, measuring the rms value \( H_e \) of the displacement of a circular area of diameter \( 0.3 \text{ mm} \) on the free side of the wafer (where we placed the pressed sapphire ball during the experiment). In Fig. 10 we plot for each mode the losses measured at \( 121 \pm 1 \text{ K} \) versus the square of the computed thickness change normalized to the mode energy \( E_{\text{mode}} \); this quantity is an estimation of the
interaction between the wafer and the holder. The experimental data were taken for both H1 and H2 samples and the chosen temperature is such that the coefficient of linear expansion of silicon vanishes: thus the thermoelastic damping is negligible and we estimate clamping losses to arise the dominant loss contribution. As expected lower losses are measured for the modes that induce little thickness variation through the thickness is not necessarily linear, and stresses must be evaluated in each layer by Eq. (5) adopting the contracted notation for stress, strain, and curvature:

\[ \begin{align*}
\epsilon_i &= \{ \epsilon_x, \epsilon_y, \gamma_{xy} \}, \\
\kappa_i &= \{ k_x, k_y, k_{xy} \}, \\
\sigma_i &= \{ \sigma_x, \sigma_y, \tau_{xy} \}.
\end{align*} \]

The matrix \( \hat{Q} \) is given in terms of the reduced stiffnesses \( Q_{ij} \) [see Eq. (8)] as

\[ \begin{bmatrix}
\hat{Q}_{11} & \hat{Q}_{12} & \hat{Q}_{13} \\
\hat{Q}_{12} & \hat{Q}_{22} & \hat{Q}_{23} \\
\hat{Q}_{13} & \hat{Q}_{23} & \hat{Q}_{33}
\end{bmatrix} =
\begin{bmatrix}
Q_{11} & Q_{12} & Q_{16} \\
Q_{12} & Q_{22} & Q_{26} \\
Q_{16} & Q_{26} & Q_{66}
\end{bmatrix}. \]

FIG. 10. Loss angle at the temperature of 121 ± 1 K as a function of the normalized squared thickness change at the center of the wafer, \( H_{E_{\text{mode}}}^2 \). The experimental data refer to the sample with hole H2 (top plot) and H1 (bottom plot). As thermoelastic damping is negligible at this temperature, the effect of the holder dissipation is seen as a monotone trend with respect to \( H_{E_{\text{mode}}}^2 \). The numbers close to the points refer to the mode labels of Table I. For a few modes two different measurements are shown, taken on the same oscillator with both sensors in two different runs and after the oscillator is removed and replaced back in the apparatus.

VI. EXPERIMENTAL RESULTS AND LOSS BUDGET

As explained in Sec. I, the goal of experimental apparatus characterized in the above is to measure the loss contributed by post-treatments of the silicon samples, in particular, by layers of silicon-onto-silicon bonds. The minimum bond losses that add to those discussed in Sec. V and that the setup is sensitive to depend on the ratio between the energy \( E_{\text{bond}} \) stored in the bond layer and the total energy \( E_{\text{tot}} \) in the body. If \( \phi_0 \) indicates the losses of the untreated sample and considering only intrinsic mechanisms, the total losses \( \phi_1 \) of the bonded sample, with overall dimensions identical to the untreated sample, can be written as

\[ \phi_1 = \phi_0 + \frac{E_{\text{bond}}}{E_{\text{tot}}} \phi_{\text{bond}}. \]
If $h$ is the total thickness and $h_b$ and $z_b$ are, respectively, the thickness of the bond layer and its mean distance from the middle plane (see Fig. 12), the total energy is obtained as the sum of the energy stored in each layer:

$$E_{\text{tot}} = \frac{1}{2} \int_{-h/2}^{h/2} \int_{-h/2}^{h/2} k_{m} Q_{lm}^{(\text{bulk})} dxdy + \frac{1}{2} \int_{-z_b}^{z_b} \int_{-h/2}^{h/2} k_{m} Q_{lm}^{(\text{bond})} dxdy + \frac{1}{2} \int_{-h/2}^{h/2} \int_{z_b}^{h+z_b/2} k_{m} Q_{lm}^{(\text{bulk})} dxdy,$$

where the second term is the energy ($E_{\text{bond}}$) stored in the bonding layer. If the bonding layer is thin, we approximate the energy ratio as

$$\frac{E_{\text{bond}}}{E_{\text{tot}}} \approx \frac{\int_{-z_b}^{z_b} \int_{-h/2}^{h/2} k_{m} Q_{lm}^{(\text{bond})} dxdy}{\int_{-h/2}^{h/2} \int_{-h/2}^{h/2} k_{m} Q_{lm}^{(\text{bulk})} dxdy}.$$

If the bulk material and the bond are both isotropic and have the same Poisson ratio, from Eqs. (9) and (22) we see that the two surface integrals differ only by the Young moduli (we use the apexes bulk and bond to distinguish between the two), and we can write

$$\frac{E_{\text{bond}}}{E_{\text{tot}}} \approx \frac{12 Y_{\text{bond}}}{h^3} \frac{z_b^2 h_b + h_b^3}{12} \approx \frac{12 Y_{\text{bulk}}}{h^3} \frac{z_b^2 h_b}{h^3}.$$

Then the energy coupling is minimum when the bonded sample is symmetric with respect to the bond layer (i.e., $z_b=0$). This result is a good approximation for isotropic materials in the Kirchhoff hypothesis.

Even if FEM analysis should be done to obtain precise results for (100) silicon, the estimate [Eq. (24)] can be used to evaluate the minimum $\phi_{\text{bond}}$ that our apparatus is sensitive to, computed as the minimum difference in the total losses that we can observe between the unbonded and bonded samples, both having identical overall dimensions. If $\sigma_T$ represents the standard deviation of loss measurements at a temperature $T$ and for a given frequency, with a confidence level of 99.73% we can write the minimum detectable losses as

$$\phi_{\text{bond},T} \geq \frac{E_{\text{tot}}}{E_{\text{bond}}} 3\sigma_T.$$

If the bonded sample is composed by three identical slices in stack (thus $z_b \sim h/6$) and $h_b \ll h$ (see Fig. 12), we get

$$\phi_{\text{bond},T} \geq 4.5 \frac{h_{\text{bulk}}}{h_b} \frac{h}{h_b} \sigma_T.$$

Estimation of the standard deviation of the loss measurements is a delicate issue. For mode No. 9, at room temperature and at 77 K, where losses are dominated by the thermoelastic mechanism, measurements are well reproducible (within 10%): we checked this to be the case even after the oscillator is removed and then replaced back in the apparatus and even by interleaving the measurements by a full thermal cycle to 4 K. So we estimate the standard deviation of the loss angle of mode No. 9 to be $\sigma_{300 K}=1 \times 10^{-6}$ at 300 K and $\sigma_{77 K}=5 \times 10^{-8}$ at 77 K. For the same mode, measurements at 4 K are less reproducible: the standard deviation of measurements on mode No. 9 is $\sigma_{4 K}=1 \times 10^{-8}$; obtained after removing and replacing back the sample in the apparatus and by interleaving a full thermal cycle. In the literature we found that loose reproducibility is associated with clamping losses; this is also one of the dominant loss mechanisms for mode No. 9 at 4 K. We note that at all temperatures the above quoted scatter is much larger than the errors from the exponential decay fit of the data, in the literature usually reported as the error in the loss factor; moreover at all temperatures the measured losses are better reproducible within a sequence of consecutive measurements with respect to the above reported scatters. Finally we note that the above reported scatters refer to a thermalized oscillator: in fact, the scatter between the measurements is larger when not enough time is allowed for the system to thermalize.

To give a number of our sensitivity we consider the case of a sample of total thickness $h=500 \mu m$ obtained by stacking three identical wafers (see Fig. 12) via hydroxy-catalysis bonding, a technique for which data are already available: in this case $h_b=80 \text{ nm}$ and $h_{\text{bond}}=7.9 \text{ GPa}$ and $\phi_{\text{bond}}$ is of the order of $10^{-1}$ at room temperature. Thus for the mode (4,0,0) at about 1.7 KHz (mode No. 9) we are sensitive to $\phi_{\text{bond},4 K} \approx 5 \times 10^{-3}$ at room temperature, $\phi_{\text{bond},77 K} \approx 3 \times 10^{-2}$ at 77 K, and $\phi_{\text{bond},4 K} \approx 5 \times 10^{-3}$ at 4 K.

The maximum bond losses that would allow the construction of a sensitive DUAL detector in silicon depend critically on the detector configuration, presently still under study, and construction procedure. However, we can give a rough approximation of the maximum tolerable losses by considering the following case: it is pessimistic since it enhances the effect of bond losses, assuming a uniform distribution of the elastic energy. Let us consider to build a silicon body of volume $V_{\text{tot}}=1 \text{ m}^3$ by bonding together 10^3 cubes with edge length of 0.1 m; let us also consider the facing faces of adjacent cubes bonded over their whole surfaces and let $h_{\text{bond}}=100 \text{ nm}$ be the bond thickness. The total volume of
the bond layers is thus $V_{\text{bond}} \approx 3 \times 10^{-7}$ m$^3$. Then, if such a mass must show losses at the level of $\phi_M \approx 10^{-8}$ at 4 K, the bond losses $\phi_{\text{bond}}$ should be

$$\phi_{\text{bond}} \leq \frac{V_{\text{tot}}}{V_{\text{bond}}} \phi_M \approx 3 \times 10^{-3}. \quad (27)$$

Our sensitivity is thus enough for the purpose of constructing a DUAL gravitational wave detector, if the hydroxy-catalyzed bond is a good approximation. By the way, we note that the sensitivity could be optimized by choosing a clever geometry for the bonded sample. For instance, at room temperature a thicker sample would suffer less from the thermoelastic damping and thus lead to a better sensitivity, provided the relative error in the loss measurement is constant.

VII. CONCLUSIONS

We have performed a through characterization of a setup capable of measuring mechanical losses contributed by post-processing treatments on silicon wafers, in the temperature range between room temperature and 4 K and in the frequency range between 300 Hz and 4 kHz. To get a full understanding of the measured total losses we compared the experimental data to estimations of the losses expected from major contributing mechanisms: the thermoelastic damping, computed for our anisotropic wafers by a detailed FEM model, was shown to dominate our lowest-loss mode in almost the entire temperature range from ambient down to 40 K. Below such temperature surface losses and residual clamping losses are estimated to give the largest damping component. Overall, we were able to measure losses as low as $10^{-8}$ at 4 K for our 100-mm-diameter, 500-μm-thick wafers. We also investigated the reproducibility of our loss measurements at 300, 77, and 4 K: thus we were able to estimate our sensitivity to further losses contributed by post-production treatments on silicon wafers. With a confidence level of 99.7%, in the case of a 100-mm-thick lossy layer with Young modulus a factor of 10 smaller than that of silicon, we are able to measure additive loss contributions at the level of a few $10^{-3}$ at 4 K; this figure is sufficient for characterizing losses at cryogenic temperatures due to silicon wafer bonding methods to be employed in the construction of a DUAL detector of gravitational waves. This same apparatus can be used to investigate the losses due to other types of thin films on silicon, such as optical coatings.

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3. B. Wilke et al., Class. Quantum Grav. 19, 1377 (2002).
18. We consider acoustic waves of angular frequency $\omega$ such that $\tau_\omega \ll 1$, where $\tau_\omega$ is given in Eq. (16).