



Upper limits on the mechanical loss of silicate bonds in a silicon tuning fork oscillator



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ABSTRACT

Silicon optics suspended by silicon ribbons or silicon fibers and kept at low temperature are being considered, because of promising optical and mechanical properties, for the test masses of third generation interferometric gravitational-wave detectors. To interconnect the suspension elements the technique of hydroxide catalysis (or silicate) bonding can be used. In order to estimate the bond loss a tuning fork was fabricated from silicon ribbons, which were then silicate bonded. The temperature dependence of mechanical loss of this tuning fork was measured in the temperature range of 95–295 K. The ratio of the energy stored in the bond layer to the total energy of the tuning fork was calculated by numerical simulation. This provided an upper limit of the bond loss as a function of the temperature. It is $(5 \pm 2) \times 10^{-3}$ at 123 K, a proposed operating temperature of cryogenically operating ground based interferometric gravitational-wave detectors.

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1. Introduction

On 14th September and 26th December 2015 the detectors of the laser interferometer gravitational-wave observatory (LIGO) detected signals from separate pairs of binary black hole mergers [1,2]. The detectors continue observations and the next generation of detectors is under discussion. Single crystalline silicon test masses suspended by silicon ribbons or silicon fibers and kept at low temperature are considered for use in the third generation of interferometric gravitational-wave detectors [3,4]. Silicon has low mechanical loss in the temperature range of 4–300 K [5,6]. Operating such a detector at a temperature of about 123 K allows for significant reduction of the thermo-elastic noise of the test masses and their suspension due to the thermal expansion coefficient crossing zero at this temperature [7]. Hydroxide catalysis (or silicate) bonding is a room-temperature chemical bonding technique first invented by Gwo [8] and further developed for applications in the quasi-monolithic silica mirror suspensions of gravitational-wave detectors such as GEO600 [9] and the Advanced LIGO (aLIGO) detectors [10]. In these applications the surface flatness is typically

in the order of 60 nm peak-to-valley, which leads to bonds of similar thickness [11]. The hydroxide catalysis bonding technique can also similarly be used to form a silicon quasi-monolithic suspension [12–14].

It is essential to quantify the mechanical loss of the bond region created between the silicon substrates in order to calculate thermal noise associated with the bonds. The loss of the bonds can be estimated from the measurement of the Q-factor of a mechanical oscillator, fabricated from silicon elements using hydroxide catalysis bonding. It is important to have as large a ratio as possible between the strain energy of the bond and the strain energy of the entire system in order to increase the contribution of the bond loss to the total loss of the oscillator. A design based on a tuning fork allows us to provide such a ratio with clamping losses minimized as much as possible. In this article, we describe a tuning fork fabricated from silicon ribbons using the hydroxide catalysis bonding technique and present the results of measurements of loss in the tuning fork over the temperature range of 95–295 K.

2. Experimental procedure

2.1. Fabrication of the tuning fork

Silicon ribbons were fabricated at Moscow State University (MSU) from commercially available (University Wafer Inc.) 3-inch

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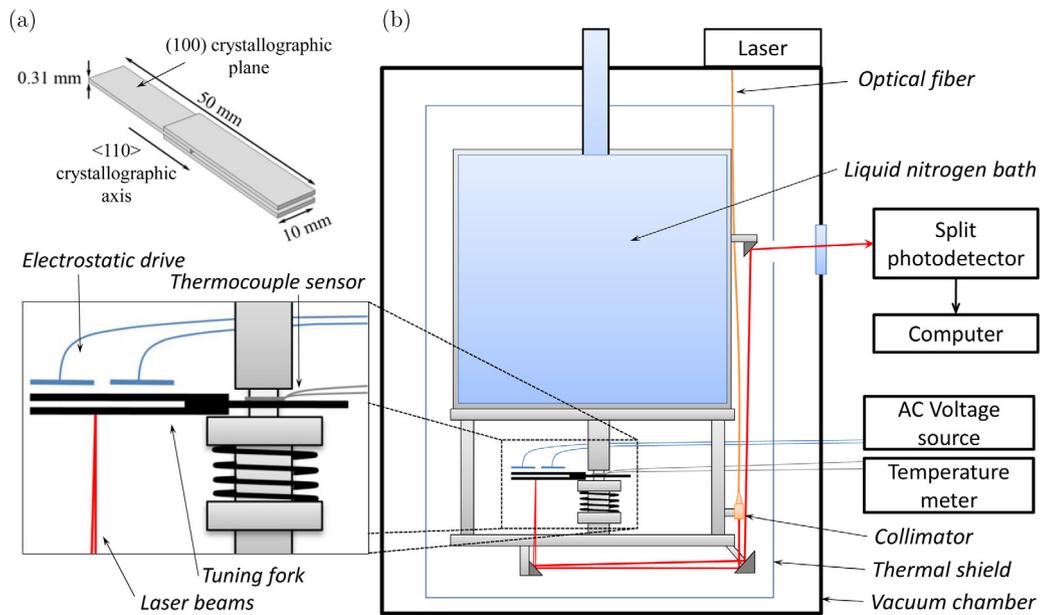


Fig. 1. Schematic of silicon tuning fork (a) and the experimental setup (b).

diameter, 310 μm thick double-side polished undoped (100) oriented single crystal Float Zone silicon wafers (room temperature resistivity $> 1000 \text{ Ohm}\cdot\text{cm}$). Two geometries of ribbon were fabricated with lengths of 26 mm and 29 mm, with both ribbons having a width of 10 mm. They were fabricated by making 1 mm wide slots in the wafer by means of anisotropic wet etching in KOH solution (30% concentration by weight in water) at a temperature of 80 $^{\circ}\text{C}$. A detailed description of this technique can be found in [15]. A protective mask was formed by depositing a 1.5 μm thick SiO_2 layer grown by thermal oxidation on each side of the wafer using a photolithographic technique. The slot direction was parallel to the crystal axis <110> which was denoted by the reference flat cut in the wafer. Sloping sidewalls, which have an angle of about 60 $^{\circ}$ to the surface of the wafer, are a characteristic feature of the etching process.

The tuning fork was comprised of three ribbons bonded together as shown in Fig. 1(a). The tuning fork handle ribbon was the shorter ribbon (26 mm). The prongs were formed by two of the longer 29 mm long ribbons. The length of bond overlap was 5 mm so the tuning fork full length was 50 mm, and the width was 10 mm.

The substrates were characterized and bonded at the University of Glasgow. To enable hydroxide catalysis bonding between silicon substrates a $\geq 50 \text{ nm}$ thick SiO_2 layer [13] has to be deposited on the silicon substrates. After cleaning the samples in a 1:7 solution of hydrogen peroxide and sulphuric acid a $163.1 \pm 1.8 \text{ nm}$ thick layer (measured using a SE850 spectroscopic ellipsometer¹) of SiO_2 was grown using dry thermal oxidation at 1000 $^{\circ}\text{C}$.

The surface roughness of the ribbons was measured using a WYKO NT1100 optical surface profiler² and the flatness was measured using a ZYGO GPI XP/D interferometer³ before and after oxidation. The average flatness for all ends of all three substrates was $639 \pm 158 \text{ nm}$ before oxidation and $616 \pm 137 \text{ nm}$ after oxidation, i.e. of the order of one optical wavelength. This is a factor of 10 less flat than required for typical gravitational-wave applications and it could be expected that the bonds are much thicker

than 61 nm [11]. They could be as much as 800 nm thick. The average surface roughness of the samples before oxidation was very low; averaging $2.7 \pm 0.9 \text{ nm}$. After oxidation, it increased slightly to $7.8 \pm 1.2 \text{ nm}$.

After oxidation, the samples were cleaned in a UV ozone chamber for 15 minutes (operating with a maximum ozone level of $\approx 250 \text{ ppm}$) to remove any organic contaminants. This method was chosen over the cerium oxide cleaning method employed by Advanced LIGO [16] due to the fragile nature of these samples. The bonding solution was prepared by mixing 1 part of Sigma-Aldrich sodium silicate solution with 6 parts of reverse osmosis deionized water and thoroughly mixing it, followed by centrifuging the solution and filtering it through a 0.2 μm medical filter as per the aLIGO bonding procedure [16]. Prior to placing the samples in the bonding jig the samples were carefully wiped with 99.9% methanol and inspected for dust under a high intensity lighting. A 0.4 μl drop ($0.8 \mu\text{l}/\text{cm}^2$) of bonding solution was placed on the bonding area and the handle was placed on top of the tuning fork. This was quickly followed by bonding the second prong. Spacers were placed under the free end of the handle and between the two prongs to even the load through the bond. The bonds were then clamped with a small pressure (of the order 10 kPa) using a block and clamping plate attached to the jig. The sample was transferred to MSU a few days later for further curing. Hydroxide-catalysis bonds between silica substrates typically reach their maximum strength in 4 weeks of curing at room temperature [17].

2.2. Experimental setup and procedure of measurements

The temperature dependence of the fundamental frequency f and the mechanical loss ($\phi = 1/Q$) of the tuning fork were measured at MSU using the experimental setup described in detail in [18]. The schematic of the experimental setup is shown in Fig. 1(b). The tuning fork handle was clamped between two stems attached to an aluminum mounting block with a PTFE disk (8 mm diameter and 2 mm thickness). PTFE was used as a soft material in order to minimize the risk of breaking or scratching the sample. One of the PTFE gaskets was mounted on the aluminum stem; another one was on the spring-loaded disk. The spring stabilized the clamping force when the temperature changed. Vibration of the handle of the tuning fork is suppressed due to the antiphase vibration of

¹ http://www.sentech.de/en/Products/Thin-Film-Measurement/Spectroscopic-Ellipsometer/SENresearch_219/.

² <http://www.veeco.com/>.

³ <https://www.zygo.com/?/met/interferometers/>.

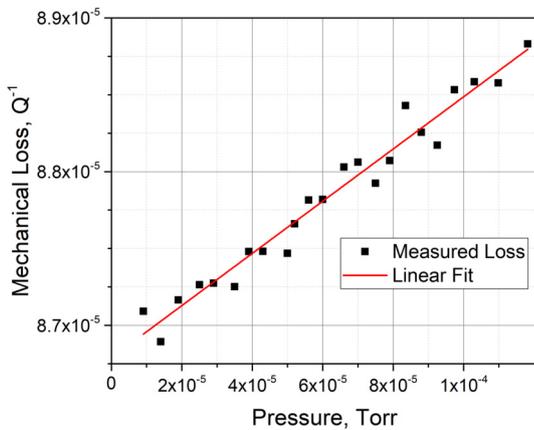


Fig. 2. Loss of the tuning fork as a function of the residual gas pressure in the vacuum chamber.

its prongs. The area of the handle with the minimal total displacement was determined from a finite element model of the tuning fork. The sample was therefore clamped at the same position as found in the FE model (about 15 mm from the end of the handle).

The clamping design allowed us to place a copper constantan thermocouple in direct contact with the tuning fork. It was attached to a thin copper foil disk, which is placed between the handle and the PTFE gasket in the clamp. The voltage produced by the thermocouple was measured using a microvolt meter B2-36 (a Russian product).

The mounted tuning fork assembly was attached to a vessel containing liquid nitrogen set inside a vacuum chamber. The entire structure was surrounded by a thermal shield. The tuning fork was cooled through radiation and thermal conductivity of the clamping structure. The cooling time from room temperature down to 100 K was found to be approximately 2 hours. This allowed us to measure the temperature dependence of the loss during the cooling of the tuning fork and during heating up to room temperature after evaporation of the liquid nitrogen. Both sets of measurements agreed to within experimental error. The uncertainty of the measured temperature is estimated to be ± 5 K. This error includes the effect of the thermal gradient within the tuning fork and the inaccuracy of the temperature sensor. The gradient is maximal in the high temperature range in which the cooling speed is highest. The inaccuracy of the temperature sensor is maximal in the low temperature range due to reduction of the thermoelectric coefficient.

The tuning fork resonant frequency f was excited by an electrostatic drive which consisted of the plate with two electrodes placed 2 mm above the top prong. An AC voltage of approximately 40 V, with a frequency $f/2$, was applied to the electrodes. The peak vibration amplitude was about 10^{-8} m. This vibration was monitored by using an optical sensor (like described in e.g. [19]) which measured the amplitude of the bottom prong. Local bending of the bottom prong caused a deflection of the laser beam reflected from the prong surface. The reflected beam passed through a system of mirrors that steer the light and was detected by a split photodiode placed outside the vacuum chamber. The tuning fork quality factor Q was determined from the ring-down time τ according to the relation $Q = \pi f \tau$.

The main source of external losses that needed to be minimized besides the clamping loss was gas damping. The characteristic feature of the fabricated tuning fork is the small distance between its prongs which is 0.31 mm. At such a narrow gap the squeeze film air damping effect plays a central role [20,21]. The effect of gas damping was observed at room temperature. The mechanical loss of the tuning fork as a function of the residual gas pressure p in the vacuum chamber is shown in Fig. 2.

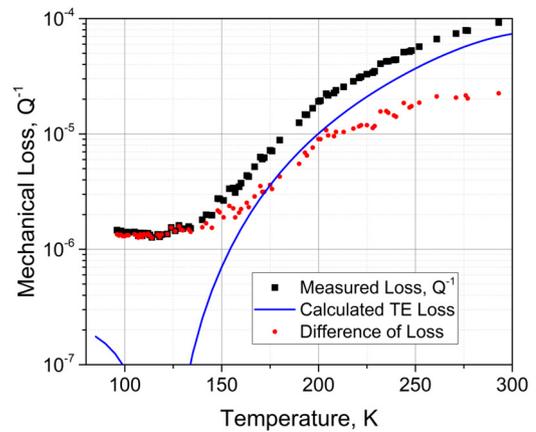


Fig. 3. Temperature dependences of the mechanical loss of the tuning fork: measured loss (black squares), calculated thermo-elastic loss (solid curve) and a difference between them (red circles). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

A linear function $Q^{-1} = (0.019 \pm 0.002)p$ (Torr) gives a good approximation of the dependence of the loss on the residual gas pressure and can be extended to lower pressures. After filling the vessel with liquid nitrogen the residual pressure in the chamber is about 10^{-6} Torr. Measurements of the temperature dependence of the tuning fork loss were carried out at this pressure. Taking into account that the temperature dependence of the gas damping in the free molecular regime is $Q^{-1} \sim T^{-1/2}$ [20], we can conclude that the residual gas damping is $Q^{-1} \leq 3.5 \times 10^{-8}$ over the entire temperature range of 95–295 K.

3. Results of measurements and calculation of the bond loss

The measured temperature dependence of the mechanical loss factor Q^{-1} of the tuning fork with the resonant frequency of 742 Hz is shown in Fig. 3. This measurement was carried out 20 days after its fabrication. The loss is found to have a minimal value at a temperature of about 123 K. This indicates that the thermo-elastic loss is suppressed. In Fig. 3, the temperature dependence of the calculated thermo-elastic (TE) loss factor is shown by the solid line. We use the approximate expression for the thermo-elastic loss factor obtained by Zener for flexural vibrations of a thin rectangular beam [22].

The thermo-elastic loss is the main mechanical loss of the material at room temperature for the tuning fork's fundamental mode with bending deformation of the prong. When the temperature approaches the zero-crossing temperature of silicon's coefficient of thermal expansion, near 123 K, the thermo-elastic loss decreases significantly. The difference between the measured loss of the tuning fork and the calculated thermo-elastic loss is shown by red circles in Fig. 3. It is likely that the main contributions are from the bond, clamping, oxide layer and surface loss. Note that these losses can also be temperature dependent. The substrate material loss in silicon is less than 2×10^{-8} at 123 K [23].

The loss of the tuning fork ϕ_{bonded} can be divided into the loss associated with the bonding layer ϕ_{bond} and the loss of the material $\phi_{\text{substrate}}$. They are connected by the following relationship [24]:

$$\phi_{\text{bonded}} = \left(\frac{E_{\text{substrate}}}{E_{\text{total}}} \right) \cdot \phi_{\text{substrate}} + \left(\frac{E_{\text{bond}}}{E_{\text{total}}} \right) \cdot \phi_{\text{bond}} \quad (1)$$

where E_{bond} is the energy stored in the bond layer, $E_{\text{substrate}}$ is the energy stored in the silicon material of the tuning fork $E_{\text{total}} = E_{\text{substrate}} + E_{\text{bond}} \approx E_{\text{substrate}}$, if $E_{\text{bond}} \ll E_{\text{substrate}}$. The ratio of energies is calculated numerically using a finite element model (FEM).

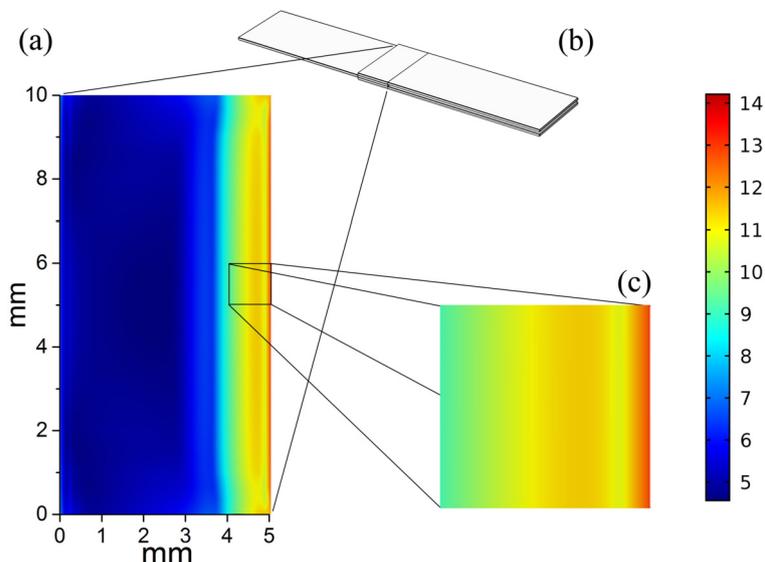


Fig. 4. The strain energy density distribution in the middle plane of the bond layer (a), (c). Illustration of the layer position in the tuning fork (b). “0” of the horizontal axis corresponds to the end of the prong nearest to the handle, the vertical axis corresponds to the tuning fork width, logarithmic color scale.

In doing this modeling there is uncertainty in the values of the Young’s modulus of the bond (which potentially varies between 20 and 60 GPa) [25] and the bond thickness could be in the range from 60 to 800 nm (excluding oxide layer). In order to understand the effect of these uncertainties the FE modeling was done across the full range of Young’s moduli and bond thickness.

The numerical model of the vibrating tuning fork comprises the fork handle, the two prongs and the two bonding areas, modeled as two thin rectangular blocks between the handle and the prongs. Parameters of the silicon are as follows: Young’s modulus $Y_s = 156$ GPa, Poisson’s ratio $\nu_s = 0.27$, density $\rho_s = 2300$ kg/m³. The silicon is considered lossless in the model. Parameters of the bond layer: Young’s modulus $Y_b = 20$ –60 GPa [25], Poisson’s ratio $\nu_b = 0.17$, density $\rho_b = 2000$ kg/m³. We can estimate the thickness of the bonding layer in the fabricated tuning fork as the total thickness of the oxide layers on the two bonded ribbons plus 61 nm at least (this number can be used for $\lambda/10$ flatness substrates) for the bond itself [25]. So taking into account the measured ribbon flatness the thickness of the bonding layer was determined to be 390–1120 nm. This value was used for the FEM calculations. The calculations are performed for room temperature (295 K). The effect of the temperature dependence of these parameters is insignificant. This can be seen from the temperature change of the tuning fork frequency, which is determined by the same parameters. The measured relative change of the tuning fork frequency is 4.6×10^{-3} when the temperature changes from 295 K to 95 K.

The model is computed using a standard equation system for the linear elastic material, assuming the only lossy regions are the bonding layers. The loss is described by the loss angle ϕ_{bond} . The most sensitive part of the model is the large ratio of the bonding layer thickness to the overall thickness of the fork. When developing such a model one should take care building the mesh of this part. A process called “sweeping” is used. One of the planes between the bonding layer and the prong is meshed with rectangular elements. The FEM simulation shows a highly non-uniform distribution of the elastic strain energy density inside the bonding layer. The strain energy density distribution in the middle plane of the bond layer is shown in Fig. 4. The energy is mainly concentrated near the bases of the prongs of the tuning fork at a distance of less than 1 mm from the end of the handle and therefore the main bond loss is also concentrated here. Therefore, the mesh ele-

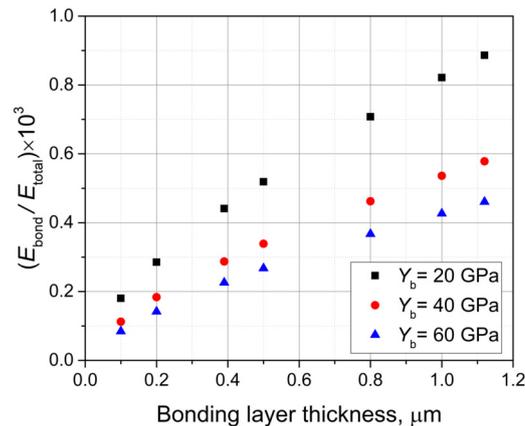


Fig. 5. Ratio of the $E_{\text{bond}}/E_{\text{total}}$ vs thickness of the bonding layer (calculated with FEM).

ments in this region are made smaller. Then the mesh is translated to the other planes parallel to the first one. Thus, the bonding region is meshed with very fine elements of appropriate distribution. Then the handle and the rest of the prongs that are away from the regions of high strain energy density are meshed using coarser elements.

The calculated dependence of the ratio $E_{\text{bond}}/E_{\text{total}}$ as a function of the bonding layer thickness for the values of $Y_b = 20$, 40 and 60 GPa is shown in Fig. 5.

The bond loss (upper limit) can be determined by subtracting the calculated thermo-elastic loss from the measured loss (see Fig. 3). The temperature dependence of the upper limit of the bond loss is shown in Fig. 6 for values covering the range of bond Young’s modulus, this upper limit being for the minimum bond thickness (390 nm). As can be deduced from Fig. 6 the bond loss could be approximately 2 times lower if the bond thickness was 1120 nm. The values obtained for the lower bond thickness are taken as the upper limit of the bond loss and its uncertainties.

In the analysis above it is assumed that all loss comes from the bonded layer. Of course, the measured loss of the tuning fork also in principal includes other losses (such as surface loss, clamping loss and oxide coating loss). The surface loss in the temperature range around 123 K is likely less than 2×10^{-7} which is significantly below the loss of the bond. This value was obtained for

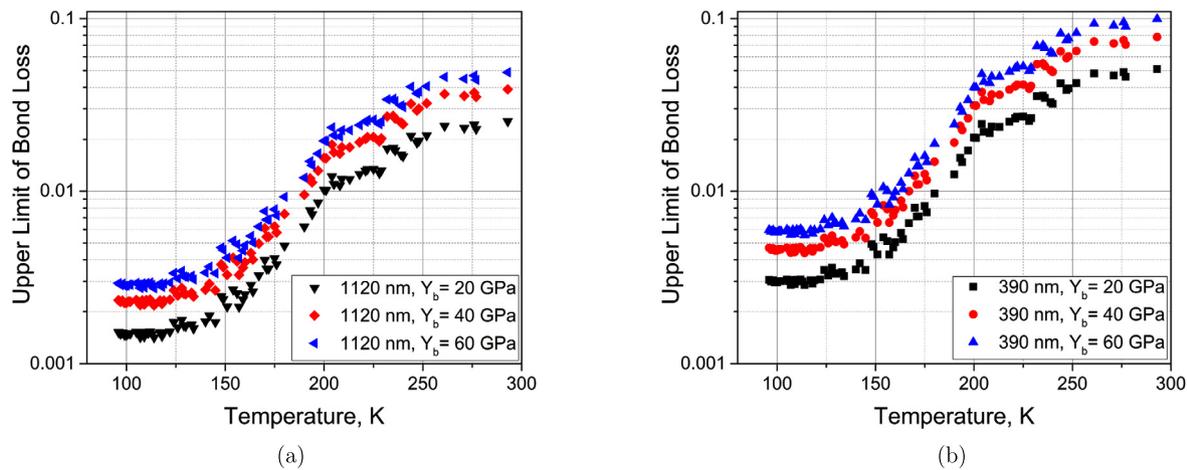


Fig. 6. Upper limit of the bond loss ϕ_{bond} of the tuning fork marked with circles. The triangles and squares show boundaries of errors associated with an uncertainty of Young's modulus of the bond layer. The plots are presented for the bond thickness of 390 nm (a) and for the bond thickness of 1120 nm (b).

a ribbon type oscillator, fabricated by the same chemical etching technique without bonding [26]. Using FEM simulation we have calculated the loss of the tuning fork associated with the SiO_2 layer having a thickness of $163.1 \text{ nm} \pm 1.8 \text{ nm}$ deposited on the silicon ribbon of the tuning fork using dry thermal oxidation. Assuming the loss angle of the thermal oxide film $\phi_{\text{oxide}} = (1-2) \times 10^{-4}$ [27, 28] we obtain the oxide coating loss $= (3-6) \times 10^{-7}$. This loss is also well below the bond loss levels found, though it should be noted that this value is an estimation. For a more accurate assessment one needs to use the value of the loss angle measured at the tuning fork frequency for the oxide layer fabricated using the exact same technology. The clamping loss level is more difficult to assess and could be dominant. Variation of the tuning fork loss measured after its reclamping did not exceed 10%.

4. Conclusion

Silicon test masses suspended by silicon fibers or ribbons and kept at temperature of about 123 K are considered as promising candidates for the third generation of interferometric gravitational-wave detectors. To interconnect the suspension elements the technique of hydroxide catalysis (or silicate) bonding can be used. The key requirement is to minimize loss in the bond. We fabricated a tuning fork from silicon ribbons, which were then silicate bonded. The ribbons are fabricated from commercially available silicon wafers using the anisotropic wet etching. The temperature dependence of the mechanical loss of the tuning fork has been measured in the temperature range of 95–295 K. The calculated thermo-elastic loss was subtracted from the measured loss. The ratio of the energy stored in the bond layer to the total energy of the tuning fork was calculated by numerical simulation. This provides the bond loss as a function of the temperature. We have found an upper limit of the bond loss because the measured losses include not only the bond loss, but also other losses, the magnitude of which is not known except the thermo-elastic loss. The upper limit of the bond loss at about 123 K was found to be $(5 \pm 2) \times 10^{-3}$. This is in the same order of magnitude as the loss found for a bond between sapphire substrates [29]. An uncertainty of the upper limit is determined by measurement errors, unknown elastic parameters of the bond layer and numerical modeling errors. The greatest contribution is made by the uncertainty of the Young's modulus. We assume that other errors give a much smaller contribution. The upper limits of the bond loss were calculated for upper and lower boundaries of the bond layer Young's modulus. These values determine errors of the upper limit of the bond loss found.

Further work is needed to improve the tuning fork fabrication technology, which will allow a decrease of the bond loss. Refinement of elastic and geometrical parameters of the bond layer will allow reducing the bond loss uncertainty. It is also necessary to investigate the effect of cooling – warming up cycles on the bond loss because the difference between the thermal expansion coefficients of silicon and SiO_2 can create defects in the bond layer.

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