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Breaking strength tests on silicon and sapphire bondings for Gravitational Wave detectors

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Abstract. The realization of third generation gravitational wave interferometric detectors is under way. An important improvement in detector sensitivity can be obtained with the reduction of thermal noise by reaching a cryogenic stage. Two materials have been identified to perform efficiently at low temperature: silicon and sapphire. In this work the breaking strength of the silicate bonding that glues cylinders of silicon (Si) and sapphire (Al_2O_3) has been investigated. All material combinations, flatness quality and orientations have been tested over time. Breaking strength between $Si-Si$ and $Si-Al_2O_3$ shows a value similar to that of fused silica, while $Al_2O_3-Al_2O_3$ does not seem to be a promising combination.

1. Introduction

Almost all the GW interferometric detectors (LIGO, TAMA and Virgo) have been built by suspending their mirrors by metallic wires. At present, the Virgo mirror suspensions are made of a specific kind of steel ($C85$). It is foreseen that in the very near future a monolithic fused silica suspension setup will replace the present one, as just realized in GEO600. In fact, thermal noise investigations for GW detectors in the low frequency range ($50 - 200Hz$) have underlined the need of a monolithic suspension stage [1]. At this stage, noise is composed of three different contributions: clamping losses, material internal loss angle and geometrical dilution factor. The best solution to the first contribution is to obtain a monolithic structure where frictional losses can be minimized [2]. Furthermore, it is well known that, using fused silica (FS) wires, it is possible to significantly reduce the thermal noise that affects suspensions and optics at room temperature [3, 4, 5, 6, 7, 8].

This work is in the way of recent studies that are in progress for 3rd generation GW detector sensitivity improvements [9]. In fact, there are fundamental limitations that occurs at low frequency: the seismic noise, the related gravitational gradient noise and the aforementioned thermal noise. Attention will be focalized on the last kind of noise. According to the fluctuation - dissipation theorem, the thermal noise intensity directly depends on the temperature [2]; thus a proposed solution for 3rd generation interferometers is to decrease thermal fluctuations by working at cryogenic temperature.

In these conditions, FS fibres are not a good candidate; in fact, the dissipation angle of FS shows a (Debye) peak at low temperature and the low thermal conductivity of FS does not allow an efficient extraction of the heat deposited by the laser on the mirror.

The present goal is to understand if it is possible to build a monolithic suspension system also in the cryogenic regime with a lower contribution to thermal noise and with low mechanical losses. Recent works [10, 11, 12] proposed crystalline materials as good candidate to realize high performance suspensions at low temperature; in particular silicon and sapphire have been taken into consideration. Crystalline silicon is a good candidate thanks to its high thermal conductivity ($k(300\text{ K}) = 1.48 \times 10^2 \text{ Wm}^{-1}\text{K}^{-1}$). Thanks to this property in a fibre, the thermoelastic peak is moved toward higher frequencies, where the pendulum thermal noise is not dominant [13]. In addition, the large thermal conductivity at low temperature permits the extraction of a large fraction of thermal energy from the interferometer mirror [14, 15]. In the same manner, sapphire has been chosen because it has shown a high quality factor, a high thermal conductivity and a small thermal expansion coefficient at cryogenic temperature. These characteristics drastically reduce the effects of thermal noise, thermal lensing and thermoelastic noise [16, 17].

In this work, the characterization of silicon and sapphire is performed to verify their silicate bonding strength. *Si* and Al_2O_3 bonding for different factors and conditions are of strong importance for all the scientific community involved in this field. In this paper first results on the bending stress measurements on the silicate bonding at room temperature are presented. In the near future, cryogenic measurements will be considered.

2. The hydroxide-catalysis bonding

Bonding may occur between almost flat surfaces of different materials if a silicate-like network can be created between the surfaces, as discussed by Gwo [18, 19]. It is important to use a bonding method which may easily and inexpensively perform either at room temperature and over a broad temperature range. The chemistry of hydroxide-catalysis bonding can be considered to take place in the following steps: hydration and etching, polymerization and dehydration [20]. In absence of any catalyst, the activation energy of the dehydration process ranges from few eV to several eV , depending on the material. Using a hydroxide catalyst, the energy can be lowered in some cases to less than few eV , according to the precise temperature at which the reaction occurs. Therefore, the hydration and dehydration processes can be favorable also at room temperature through hydroxide catalysis. In our particular case the catalyst is potassium hydroxide. A solution concentration of 1 : 250 (molecular ratio $\text{KOH} : \text{H}_2\text{O}$) has been used.

Moreover it is preferable to choose those materials that can form and/or be chemically linked to a silicate-like network. Silicon has been tested among those materials capable of forming silicate-like network by themselves, while sapphire has

been chosen among those materials that can be linked to a silicate-like network.

It is widely accepted [21] that silicon surface hydrophilicity, due to hydroxyl groups and adsorbed water, is the principal element in bonding processes between silicon surfaces. The hydrophilic nature of the silicon surface can be obtained by wet oxidizing agents (aqueous solution, cleaning solution). At room temperature and relative humidity of 50 %, hydrophilic surface is undoubtedly covered by a few layers of water molecules attached to the OH groups via hydrogen bonds. Brought into contact, the surfaces adhere. At this point dehydration of the $Si - OH$ groups, caused by KOH , starts and siloxane bridges ($Si - O - Si$) are formed. These bridges, which connect the silicate chains in solution, are responsible for the construction of the solid interface between the two bonding surfaces. The result is the rigid bonding, shown in Fig. 1.

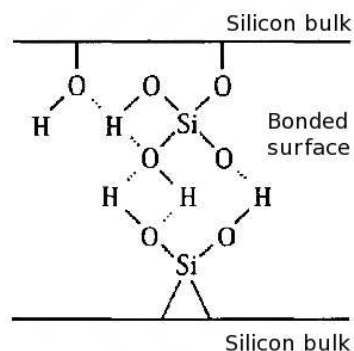


Figure 1. Model of room temperature bonding between two hydrophilic silicon surfaces.

In these kind of samples that form a silicate-like network, such as silicon, the bonding is a relatively thick three-dimensional one, harder than those materials that cannot form the silicate-like bond themselves [19]. The maximum bond strength should be achieved after some weeks: the exact time it takes depends on temperature, on the concentration of the aqueous solution used, on the relative humidity of the environment and on the degree of mismatch of the surfaces of the bounded pieces. It is not important that material substrates are soluble in the bonding solution, but they must have or can generate hydroxyl groups at the interface.

In Fig. 2, two SEM/EDX (scanning electron microscope/energy dispersive x-ray) spectra of respectively silicon-to-silicon and silicon-to-sapphire bonds are reported. In the first case, elements that characterise the silicon surface are Si , but also oxygen and potassium. The last two come from the bonding solution. The same conclusion holds for the silicon-to-sapphire bonding, where O , K and Al exist. The presence of oxygen confirms the hydrophilic nature of silicon and the possibility to form siloxane groups. However, after only a few hours the bond has sufficient strength to allow the pieces to be handled: initially a hydrogen bond is formed, subsequently surfaces are connected

by siloxane bridges.

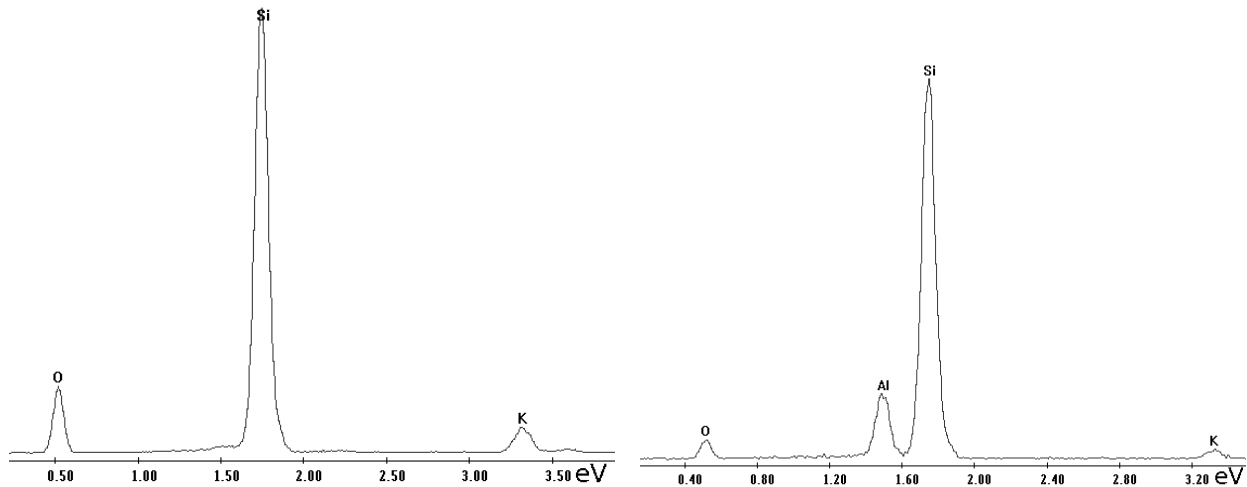


Figure 2. SEM/EDX spectra of $Si-Si$ (random - random) sample with $\lambda/10$ flatness and $Si-AL_2O_3$ (random - random) sample with $\lambda/4$ flatness.

3. Experimental bonding procedures

In order to do the programmed studies, a number of silicon and sapphire cylinders have been used with different crystalline orientations and flatness as listed in Table 1. The bonding strength is expected to depend on the interface mismatch.

Samples are commercially available from the Impex High-Tech GmbH company, with both sides polished to three different optical flatness of approximately $\lambda/10$, $\lambda/7$ and $\lambda/4$, where $\lambda = 632.8 \text{ nm}$. In order to successfully bond cylinders, fundamental steps have to be followed:

- surface preparation, because a high level of cleaned area is demanded
- bonding procedure, both at room temperature and in the air

3.1. Surface preparation

To allow the growth of a good bond it is essential that surfaces are rigorously cleaned to eliminate contamination as much as possible; this operation has an impact on the silicon surface chemistry and topography and, consequently, on the mismatch at the interface [22]. Firstly, the sample is cleaned with isopropanol, then it is dipped in a cleaning solution (whose mixture is 5:1, H_2SO_4 (96 %): $K_2Cr_2O_7$), rinsed with water and dried with gaseous nitrogen. The surface preparation is, hence, completed. The bonding process is carried out under a laminar flow in a Class 100 clean environment at room temperature ($T = 23 \pm 5^\circ C$).

Bond combination	Flatness	Orientation	Number of successfully bonded samples		
			1st week	2nd week	3rd week
Si-Si	$\lambda/10$	rand-rand	5/7	5/7	3/8
Si-Si	$\lambda/7$	rand-rand	0/0	4/5	5/5
Si-Si	$\lambda/4$	rand-rand	0/0	2/5	3/5
Si-Si	$\lambda/10$	100 -100	5/5	5/5	5/5
Si-Si	$\lambda/7$	100 -100	5/5	5/5	5/5
Si-Si	$\lambda/4$	100 -100	4/5	4/5	3/5
Si-Si	$\lambda/10$	111-111	5/5	5/5	5/5
Si-Si	$\lambda/7$	111-111	5/5	5/5	5/5
Si-Si	$\lambda/4$	111-111	5/5	5/5	2/5
Si- Al_2O_3	$\lambda/10$	rand-rand	3/5	4/5	4/5
Si- Al_2O_3	$\lambda/7$	rand-rand	5/5	5/5	5/5
Si- Al_2O_3	$\lambda/4$	rand-rand	5/5	5/5	4/5
Si- Al_2O_3	$\lambda/10$	100 - C	5/5	5/5	5/5
Si- Al_2O_3	$\lambda/7$	100 - C	5/5	5/5	5/5
Si- Al_2O_3	$\lambda/4$	100 - C	5/5	5/5	5/5
Al_2O_3 - Al_2O_3	$\lambda/10$	rand-rand	4/5	2/5	5/5
Al_2O_3 - Al_2O_3	$\lambda/7$	rand-rand	4/5	4/5	2/5
Al_2O_3 - Al_2O_3	$\lambda/4$	rand-rand	0/5	5/5	5/5
Al_2O_3 - Al_2O_3	$\lambda/10$	C - C	4/5	4/5	4/5
Al_2O_3 - Al_2O_3	$\lambda/7$	C - C	0/5	2/5	3/5
Al_2O_3 - Al_2O_3	$\lambda/4$	C - C	3/5	3/5	4/5

Table 1. Samples considered for the breaking strength analysis. For each combination, orientation and flatness, the number of successful bonded samples over the number of total available samples has been reported, respectively waiting 1, 2 or 3 weeks before the breaking strength test. The diameter of all samples is 5 mm.

3.2. Bonding procedure

This step follows immediately after surface preparation. The first cylinder is placed into a teflon fixture to insure immobilization of the sample. Typically a 0.5 to 0.8 $\mu\text{l}/\text{cm}^2$ drop of potassium hydroxide solution is applied to the upper surface using a micropipette. Then the second sample is lowered onto the first one where the bonding solution has been applied. This small quantity of solution spreads out eventually across the entire contact region. Pieces are carefully aligned and minor adjustments could be made within the first minutes. This solution will react with the surfaces extracting the silicates necessary to obtain the bond. A strong, rigid, very thin bond thus forms between the pieces in contact. This procedure has been tested for FS samples and seems to work well using crystalline silicon. It has been noticed that the settling time for such bonds can be within of hours or days [23]. The samples are held in position until dehydration starts

and, using the micropipette, some drops of potassium hydroxide solution are added to decrease imperfections in the bonding growth for the first five hours.

4. Experimental set up and results

After the sample preparation and after waiting few weeks for the bonding formation, the breaking strength has been measured. A test bench has been realized to measure the strength of the bonding in operative conditions. The force is applied at 0.05 cm from the sample interface. This configuration allows the only bending stress measurement. The facility consists of two parallelepipeds vertically translated. The bonded cylindric block (characterized by different parameters, see Table 1) is inserted between the two sample holders and then is stressed with the mechanical apparatus, which applies a progressive force close to the bonding surface. The sample holder is illustrated in Fig. 3.

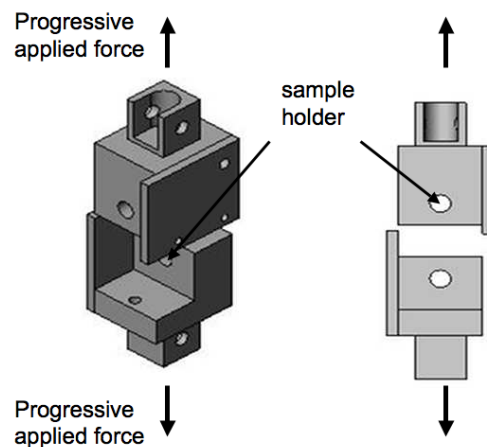


Figure 3. Breaking strength test facility (three-dimensional and front views, not to scale) The sample diameter is 5 mm .

A load cell is used to measure the stress required to break the bonded samples. Measurements show that fractures frequently occur in the bonded interface because the bonding strength is weaker than the material strength. Fig. 4 represents a $Si - Si$ sample after the breaking strength test. In this case the fracture started from the sample interface (*starting point* in figure) and ended at the bulk (see *first cylinder bulk* in figure).

For these measurements, fifteen bonded samples (that means five for each selected time interval) for each orientation and flatness quality (λ/k with $k = 10; 7; 4$) have been studied (see Table 1). The procedure has been repeated for three different surface combinations: silicon-silicon, silicon-sapphire and sapphire-sapphire.

Results of the bonding strength are reported in figures 5, 6, 7 and 8, where the stress is obtained dividing the force applied by the bonding area. Tests have shown that the bond reaches its best strength value just after a week. In Figs. 5, 6 and 7,

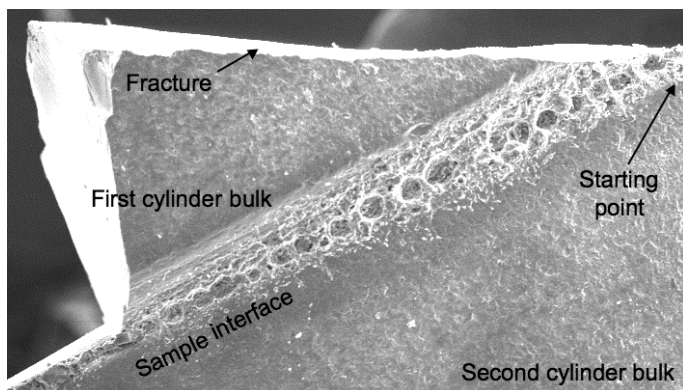


Figure 4. Broken bond between two silicon cylinders with a (111-111) orientation and a $\lambda/4$ flatness after a curing time of a week: scanning electron microscope image.

each value has been obtained by taking a statistics from five samples. In the analysis, only successfully bonded samples were considered (see Table 1). Sometimes the waiting time before the breaking strength tests is not sufficient for the bond formation and a poor statistics occurs: this mainly depends on the surface preparation and the bonding procedure. This is responsible for the large error bars that occur in our plots.

In Fig. 5, increasing time interval, samples with a $\lambda/4$ flatness appear worse than $\lambda/10$ and $\lambda/7$, as supposed in [24]. In figures 5, 6 and 7, the breaking strength values seem constant within the statistical fluctuation of the measured quantities. It shows that results are independent of the settling time before breaking the samples and of the flatness quality of the cylinder surface. From this evidence, all measurements have been summarized in Fig. 8.

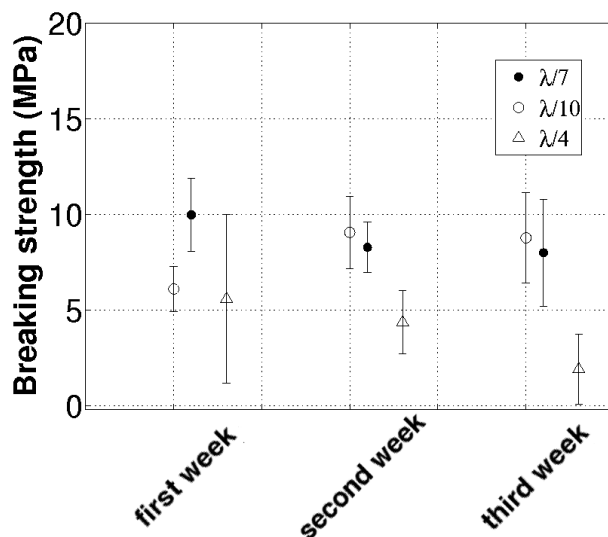


Figure 5. Bonding strength for $Si-Si$ (111-111) combination: three different flatness qualities are reported for each selected time (first, second and third week).

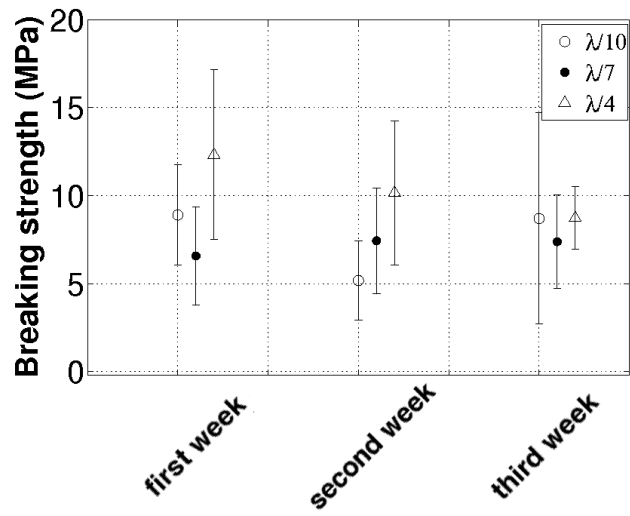


Figure 6. Bonding strength for $Si - Al_2O_3$ (random - random) combination: three different flatness qualities are reported for each selected time (first, second and third week).

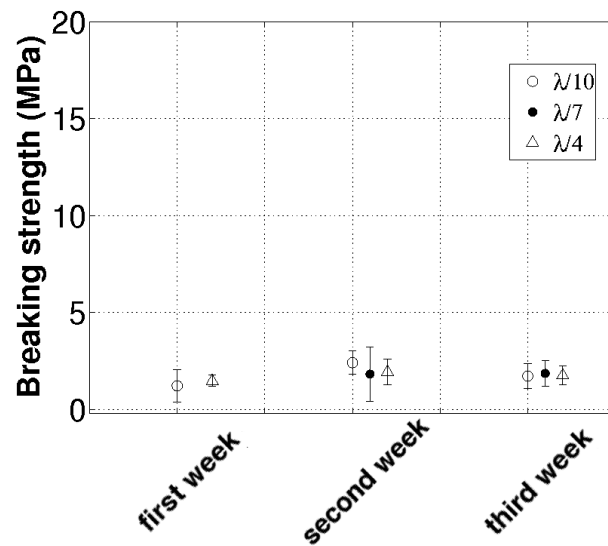


Figure 7. Bonding strength for $Al_2O_3 - Al_2O_3$ (C-C) combination: three different flatness qualities are reported for each selected time (first, second and third week).

Fig. 8 presents the breaking strength behavior with a $\lambda/10$ flatness versus all the material pairing combinations. It is clearly visible that the silicate bonding between $Al_2O_3 - Al_2O_3$ has the minimum breaking strength value. This is explained with the difficulty in creating a silicate bonding among such surfaces, as expected by Gwo [18, 19]: the mean value is $1.5 \pm 0.8 MPa$. Instead a mean value of about $8.7 \pm 3.7 MPa$ is obtained for samples with a flatness of $\lambda/10$ and silicon material in at least one of the two bonded cylinders. This behaviour is probably due to the formation of networks between surfaces, as explained in the previous paragraphs. Moreover, the obtained value is comparable to the FS one [6].

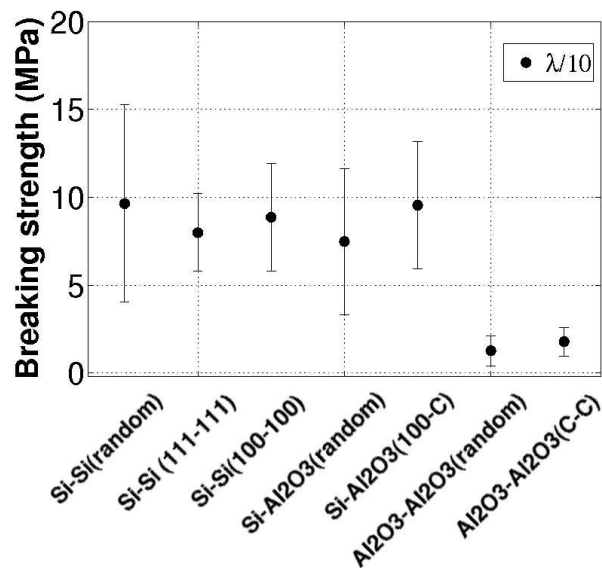


Figure 8. Breaking strength for all combinations and orientations of the $\lambda/10$ flatness. Each point is obtained by averaging over all the samples of three selected times (from between 10 to 15 depending on the unbroken samples at the moment of test).

5. Conclusions

The silicate bonding breaking strength of Si and Al_2O_3 samples with various flatness quality and crystal orientation has been investigated. Our results are compared to the previously published by van Veggel et al. in [25] and by Suzuki et al. in [26]. No significant dependence of the two material combinations on the bonding time or on the crystal orientation appears, while the $Si - Si$ combination shows a slight dependence of the breaking strength on the flatness quality (at least between $\lambda/10$ or $\lambda/7$ and $\lambda/4$).

As measured in previous papers for fused silica samples [6], also for $Si - Si$ and $Si - Al_2O_3$ bondings, the breaking strength shows a behaviour that can be considered promising for the realization of monolithic suspensions in future cryogenic GW detectors. In Fig. 8, the breaking strength, obtained for the $Si - Si$ and $Si - Al_2O_3$ combinations with a $\lambda/10$ flatness quality, provides a mean value of $8.7 \pm 3.7 MPa$, as just presented

in [27]. Comparing this measure with the *Si-Si* bonding value in [25], it is visible a small improvement even if the error is large. This difference is probably due to the distinct silicate bonding procedure used and to the sample geometry. For the $Al_2O_3 - Al_2O_3$ bondings the performance is not good. As supposed by Gwo [19], in this case the probability of generating through hydroxyl groups is very low and the breaking strength mean value is 1.5 ± 0.8 MPa. Although our tests seems not too promising, previous results open a new possibility to sapphire in future GW detectors, suggesting a waiting time of several years for the formation of the bonding [26]. These preliminary room temperature measurements seem promising enough to continue the study with other tests at cryogenic temperatures on silicon-to-silicon or on silicon-to-sapphire bondings.

In conclusion these results and the possibility of *Si* and Al_2O_3 to reduce thermal noise seem to indicate a positive perspective for the future of cryogenic detectors. However, several studies have to be considered in order to completely propose a realistic solution for 3rd GW detectors.

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