Haughian, K., van Veggel, A.A. , Cunningham, L., Hough, J., Murray, P.G., Reid, S. and Rowan, S. (2017) Effect of heat treatment and aging on the mechanical loss and strength of hydroxide catalysis bonds between fused silica samples. Physical Review D, 96(4), 042003.

There may be differences between this version and the published version. You are advised to consult the publisher's version if you wish to cite from it.

http://eprints.gla.ac.uk/145791/

Deposited on: 14 August 2017

Enlighten – Research publications by members of the University of Glasgow
http://eprints.gla.ac.uk

The effect of heat treatment and ageing on the mechanical loss and strength of hydroxide catalysis bonds between fused silica samples

K Haughian,[1,*] A A van Veggel,[1] L Cunningham,[1] J Hough,[1] P G Murray,[1] S Reid,[1,2] and S Rowan[1]

[1] *SUPA, School of Physics and Astronomy, University of Glasgow, Glasgow, G12 8QQ, UK*
[2] *SUPA, University of the West of Scotland, Paisley, PA1 2BE, UK*

Hydroxide catalysis bonds are used in the aLIGO gravitational wave detectors and are an essential technology within the mirror suspensions which allowed for detector sensitivities to be reached that enabled the first direct detections of gravitational waves. Methods aimed at further improving hydroxide catalysis bonds for future upgrades to these detectors, in order to increase detection rates and the number of detectable sources, are explored. Also, the effect on the bonds of an aLIGO suspension construction procedure involving heat, the fibre welding process, is investigated. Here we show that thermal treatments can be beneficial to improving some of the bond properties important to the mirror suspensions in interferometric gravitational wave detectors. It was found that heat treating bonds at 150 °C increases bond strength by a factor of approximately 1.5 and a combination of bond ageing and heat treatment of the optics at 150 °C reduces the mechanical loss of a bond from 0.10 to 0.05. It is also shown that current construction procedures do not reduce bond strength.

PACS numbers: 04.80.Nn

* Corresponding author: karen.haughian@glasgow.ac.uk

I. INTRODUCTION

Gravitational wave detectors, such as those operated by the LIGO, Virgo and GEO600 collaborations, are based on laser interferometer systems [1–3]. They are designed to detect a relative change in the separation of suspended mirrors caused by a passing gravitational wave. The change in length of the mirror separation expected to result from passing gravitational waves from large astrophysical sources, such as the black holes colliding from which gravitational waves were detected in September and December 2015 and January 2017, are extremely small (of the order 10^{-18} m or less) [4–7]. Thus all noise sources must be identified and reduced in order for the effect of the gravitational wave to be measurable. Now that gravitational waves have been directly detected it is essential that some of the techniques used for the construction of aLIGO are studied further and improvements made to enhance the sensitivities of future upgrades or detectors. This will increase both the detection rate and the number of detectable sources and facilitate the beginning of an extremely prosperous era of gravitational wave astronomy.

An important factor in determining the overall sensitivity of gravitational wave detectors is the thermal noise of the mirror suspensions. This can be a dominant noise source at the lower end of the operating frequency band (a few tens to a few hundred Hertz) [4]. In order to reduce the thermal noise of the suspensions, a low mechanical loss material, fused silica, was chosen for the mirrors and also the final stage of the suspensions of the previously mentioned detectors [8]. This quasi-monolithic design of the lower stage suspension was implemented in GEO600 and versions of it used in upgrades to the LIGO and Virgo detectors; aLIGO and Advanced Virgo respectively [9, 10]. In these detectors, silica suspension fibres are welded onto horns on small silica interface pieces, 'ears', which are hydroxide catalysis bonded to the mirrors. This bonding process is used to join surfaces through a hydroxide catalysed hydration and dehydration reaction [11, 12]. It was first developed for use in the Gravity Probe B mission [13, 14] and has been further developed for both ground and space based gravitational wave detectors [15–18]. Although the mechanical loss of a hydroxide catalysis bond is higher than that of bulk silica, it is at an acceptable level for advanced detector suspensions since the bond layer can be extremely thin (< 100 nm) [19, 20]. It is desirable to create very strong bonds in order to keep the bond area to a minimum, thus reducing the overall contribution to the mechanical loss of a mirror suspension and thermal noise level of the overall suspension.

Mirror suspensions in gravitational wave detectors are used for many years, thus, any evolution of the mechanical loss of the bond is of great interest. Investigations into how the age of a bond affects its mechanical loss are reported.

Improvement of the bond properties important for gravitational wave detectors, strength and mechanical loss, is of interest for proposed future upgrades to aLIGO [21]. During the curing process of a hydroxide catalysis bond, there is outward migration of water and formation of siloxane chains which increase the rigidity of the bond. The rate at which the water evaporates out of the bond or diffuses into the silica could be increased by a slight elevation of the temperature. Methods investigated in this paper, aimed towards improvements in bond strength and bond mechanical loss, include heat treating the bonds and quantifying the resultant effect.

There are specific processes that the bonds have gone through during the construction of aLIGO mirror suspensions which involve heat. Therefore they require investigation in order to quantify their influence on the strength of the bond. Here we investigate the effect of the fibre welding procedure. After the ears have been bonded onto the mirrors and the curing period has passed, fibres are welded to the area of the ears known as 'weld horns' using a CO_2 laser. This process heats the weld horns to approximately 1800 °C. The bond region is in close proximity to the weld horns and can reach temperatures of approximately 350 °C during this process, which lasts for a period of three to twelve minutes [22]. Experiments were carried out to quantify the effect of this thermal load on the strength of the hydroxide catalysis bonds.

II. MECHANICAL LOSS OF A HYDROXIDE CATALYSIS BOND

It is necessary to understand fully the mechanical loss of a bond in order to determine the level of thermal noise that it contributes to a mirror suspension in a gravitational wave detector and to find methods of reducing this contribution to increase potential detector sensitivity.

Gravitational wave detector suspensions have long lifetimes. Thus, how the mechanical loss of a bond changes with time is of great interest. Investigations aimed towards further improvements of the mechanical loss of hydroxide catalysis bonds for use in potential future upgrades to aLIGO were also carried out [21].

Hydroxide catalysis bonds have a curing period where water migrates out of the bond over time. It has been found that after four weeks of curing they have reached their maximum strength [15, 17, 23]. Investigations were carried out to determine whether heating bonded samples could aid the evaporation or diffusion of additional water and hence further improve bond mechanical loss.

A. Method

The experiments discussed here use some previously published results as the initial measurements [19]. Since the current results have been measured and analysed using the same method [19, 24–26], only a brief discussion of the experimental set-up is included here.

Two Suprasil 311 fused silica cylinders were joined using the hydroxide catalysis bonding technique. Both cylinders had a diameter of 65 mm, and lengths of 50 mm and 70 mm respectively. A schematic diagram of the samples is shown in Fig. 1. The samples were chamfered in such a way that the bonding surface

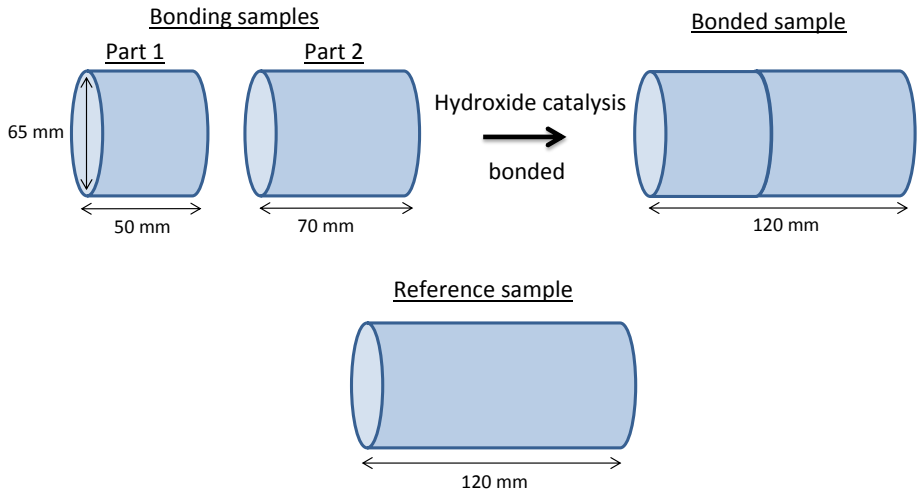

FIG. 1. A schematic diagram of fused silica samples used for hydroxide catalysis bond mechanical loss experiments. The diameter is 65 mm and the lengths are 50 mm, 70 mm and 120 mm respectively.

diameter was 64 mm. The bonded sample was suspended in a silk thread loop and excited at a number of its resonant mode frequencies in turn using an electrostatic drive plate. The motion of the front face of the sample was monitored using a table top interferometer. The silk thread suspension method was selected to ensure that the cylinder could resonate as freely as possible. Any clamping method or any method that uses attachments directly on the sample would effect the resonant frequencies and would inhibit the resonance movement. The silk thread provides minimal contact with the sample and thus minimal contact friction is produced. The contact friction is further reduced by adding a very small amount of grease to the thread. The decay of the motion of the front face of the sample was recorded over time, t, and from this data the mechanical loss was calculated using:

$$A(t) = A_o e^{-\phi(f)\pi f t}, \tag{1}$$

where A_o is the initial amplitude, f is the frequency of the resonant mode and $\phi(f)$ is the mechanical loss of the material at that resonant frequency [27].

The mechanical loss of the bonded cylinder was measured shortly after the curing period and the lowest loss value measured was found to be $(9.9 \pm 0.5) \times 10^{-8}$ [19, 28]. A reference sample of the same geometry as the bonded cylinder (65 mm diameter and 120 mm in length) was measured to provide the mechanical loss value for the bulk substrate material. These measurements aid in determining whether any changes which occur in the loss are due to a bulk material effect or due to changes in the bond layer. The lowest mechanical loss measured for the reference cylinder was $(3.2 \pm 0.1) \times 10^{-8}$ [19]. This measured mechanical loss value for the reference sample is slightly higher than the value which may be expected when using a semi-empirical

model for fused silica [29], possibly in part due to the surface polish of the sample. The barrel polish on the reference sample was visibly less smooth than that of the bonded sample and therefore additional friction with the suspension material may be introduced in the measurement. It was decided that the semi-empirical model would be used to provide values for the mechanical loss of the substrate material. Thus, an upper limit of the bond mechanical loss could be obtained.

The mechanical loss of the hydroxide catalysis bond was obtained using the measured loss of the bonded sample, $\phi_{\rm bonded}$; the loss predicted by the semi-empirical model for silica for a sample of this geometry [29], $\phi_{\rm substrate}$; the energy ratio indicating the amount of energy stored in the substrate divided by the total energy in the sample, $E_{\rm substrate}/E_{\rm total}$ and the energy ratio indicating the amount of energy stored in the bond divided by the total energy in the sample, $E_{\rm bond}/E_{\rm total}$ through use of Eq. (2):

$$\phi_{\rm bonded} \simeq \frac{E_{\rm substrate}}{E_{\rm total}}\phi_{\rm substrate} + \frac{E_{\rm bond}}{E_{\rm total}}\phi_{\rm bond}. \tag{2}$$

The substrate is considerably larger than the bond such that $E_{\rm substrate}/E_{\rm total}$ is ~ 1. As a result, Eq. (2) can be rearranged to give the mechanical loss of the hydroxide catalysis bond,

$$\phi_{\rm bond} \simeq \frac{\phi_{\rm bonded} - \phi_{\rm substrate}}{E_{\rm bond}/E_{\rm total}}. \tag{3}$$

The energy ratio for each resonant mode was obtained using the finite element analysis package ANSYS[1] [19]. Several finite element models were created using solid elements with a varying bond thickness value and an extrapolation technique was used to obtain the energy ratios for the actual sample (bond thickness of approximately 61 nm). A value for the mechanical loss of the bond was calculated for each resonant mode frequency.

Statistical analysis was carried out to determine whether the bond loss had any frequency dependence, as discussed in Cunningham et al. [19]. They found that the mechanical loss of a hydroxide catalysis bond is independent of frequency and the loss for each resonant mode was averaged to give a bond loss of 0.11 $\pm$ 0.02 [19]. It is possible to model the bonded sample directly using thin solid elements for the bond instead of using the extrapolation method, as input to calculate bond loss from the mechanical loss of the bonded sample. This increases the accuracy of the predicted strain energies. This method is discussed in Ref. [19, 30]. Using the energy ratios obtained with the direct modelling, a bond loss of 0.09 $\pm$ 0.02 was calculated. Results from the experiments reported here are analysed using this method and will therefore be compared to this latter bond loss number.

The sample, of which the mechanical loss was reported in Ref. [19], was remeasured again three years after the initial measurements. Then it was put through a heat treatment and measured again a third time. Only bonds which are free of any visual imperfections over $\geq 90\,\%$ of their bonding surface are accepted for use in a gravitational wave detector suspension. This is because the visual clarity is used as an indicator of quality. Thus, the visual clarity of the bond being investigated was noted before and after the heat treatment to observe if there was any degradation. The resultant bond loss values extracted from the experiments are reported here.

B. Results: Effect of time on the mechanical loss of a bond

The measured mechanical loss values for both the reference and bonded sample before and after a three year period are shown in Fig. 2. It can be seen that the mechanical loss of the reference mass remains at the same value within error after the three year period. The lowest loss of the bonded mass had decreased slightly from $9.9 \pm 0.3 \times 10^{-8}$ to $6.9 \pm 0.3 \times 10^{-8}$, which is an approximately 30% improvement, suggesting that the loss of the hydroxide catalysis bond does indeed decrease with time.

The mechanical loss of the bond for each resonant mode, calculated by the method shown in Sec. II A, is shown in Fig. 3. The bond loss was averaged over all the resonant modes using the same technique described in Ref. [19] and an average value of 0.06 $\pm$ 0.02 was obtained using the new finite element analysis method.

[1] www.ansys.com

The error shown indicates the spread of the bond loss values across the resonant modes. The variation of measured bond loss for different modes could be caused by a combination of suspension limitations for each resonant mode and inhomogeneities in the bond, such as bubbles or contaminants. Any resonant mode which deforms the area of the bond where an inhomogeneity is located could result in a higher measured mechanical loss value.

This observed change in the mechanical loss of the bond over time could be caused by the bond becoming thinner as water evaporates or diffuses into the substrate from the edges and migrates out of the bond. Using the modelling technique described in Ref. [19], the effect of this change in bond loss on the thermal noise of a single aLIGO test mass with two hydroxide catalysis bonded ears was calculated. The decrease from the previously calculated value (0.09 ± 0.02 to 0.06 ± 0.02) in bond loss reduces the thermal noise from 4.9×10^{-22} to 4.0×10^{-22} m/$\sqrt{\text{Hz}}$ at 100 Hz.

C. Results: Effect of a 150°C heat treatment for 48 hours on the mechanical loss of a bond

After the effect of time on the mechanical loss of a hydroxide catalysis bond had been investigated, the bonded and reference samples were heat treated to a temperature of 150°C for 48 hours in air. It was noted that this heat treatment did not visually degrade the bond area. Fig. 2 shows the value of mechanical loss obtained before and after the heat treatment for the resonant modes measured. The mechanical loss of the bond for each resonant mode, calculated by the method shown in Sec. II A, is shown in Fig. 3.

There was no systematic change in the loss of the modes of the reference sample, while the minimum loss of the bonded sample decreased slightly from $(6.9 \pm 0.3) \times 10^{-8}$ to a value of $(6.3 \pm 0.1) \times 10^{-8}$. This demonstrates again that the reduction in loss of the bonded sample is not a bulk material effect but is due to a reduction of the mechanical loss of the bond. This change can be seen more clearly for the lower frequency measurements because there is more energy stored in the bond region for these resonant modes. The heat treatment process reduced the average mechanical loss of the bond over all the measured resonant modes to 0.05 ± 0.02. The reduction could be the result of any remanent water being evaporated from the bond due to the heat or stresses being removed by an annealing effect. This change in bond loss reduces the thermal noise of a single aLIGO test mass with two hydroxide catalysis bonded ears from 4.0×10^{-22} to 3.7×10^{-22} m/$\sqrt{\text{Hz}}$ at 100 Hz.

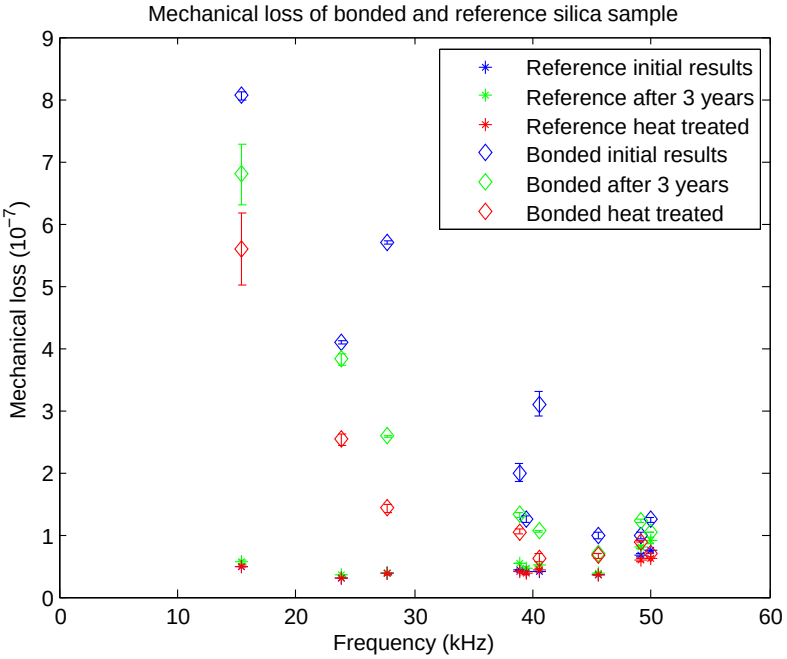

FIG. 2. The mechanical loss of a bonded and reference fused silica Suprasil 311 substrate measured after an initial cure, after a three year period and again after a heat treatment of 48 hours at 150°C.

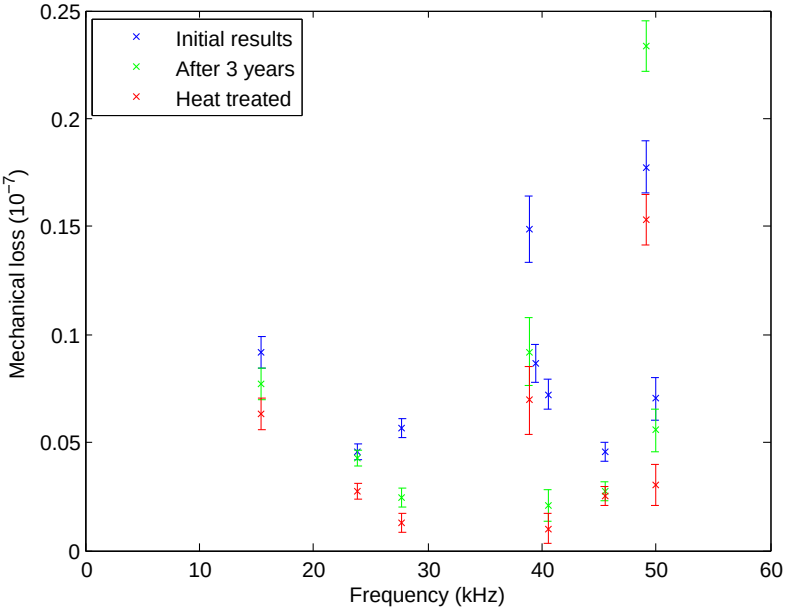

FIG. 3. Bond loss measured for each resonant mode after initial cure, after a 3 year period and after heat treatment.

As stated in Sec. II A, the value presented in this paper is an upper limit for the mechanical loss of a bond. This is because a calculated substrate loss from a semi-empirical model for the mechanical loss of fused silica was used rather than that measured from the reference sample as the sample measured a higher value than expected. If the measured mechanical loss values of the reference sample were used as the substrate loss, a slightly lower value for the bond loss would be obtained 0.03 ± 0.01. Using this value, the thermal noise of a single test mass with two hydroxide catalysis bonded ears is further reduced to 2.8×10^{-22} m/$\sqrt{\text{Hz}}$ at 100 Hz.

III. STRENGTH TESTING

The strength of the hydroxide catalysis bonds used in a gravitational wave detector suspension is important as they support the weight of the mirrors: one 40 kg mirror is supported by two hydroxide catalysis bonds in aLIGO. If the bonds could be made stronger, a bond of a smaller surface area could be used to support the same weight. By decreasing the area of the bond, the volume of bond material in the mirror suspension would be reduced, thereby decreasing the bond thermal noise contribution within the detectors.

The strength of hydroxide catalysis bonds between different materials such as fused silica, silicon, sapphire, ULE and zerodur have been studied previously [17, 20, 31–37]. A summary of most of the results can be found in Ref. [38]. However in many cases different types of strength tests were carried out and therefore the results are not directly comparable. In the case of Beveridge et al. and Douglas et al. the same type of strength test was reported but different substrate materials were used and most measurements were taken at different temperatures. Beveridge et al. found bond strengths of 42 MPa at 77 K between silicon substrates [20]. Douglas et al. took measurements at room temperature and found bond strengths of 65 MPa between sapphire substrates [36]. As the substrate material and temperature used by Beveridge et al. and the substrate material used by Douglas et al. were different to the bonds reported in this article, the results cannot be directly compared. For room temperature gravitational wave detector design it is important to investigate the tensile strength of bonds between fused silica substrates.

A. Samples

Hydroxide catalysis bonds were created between two different geometries of fused silica samples; a schematic diagram of these is shown in Fig. 4. All samples were made from polished Corning 7980[2] fused silica. The bonding surfaces were polished to ensure a peak-to-valley global flatness of $\lambda/10$ where λ =633 nm.

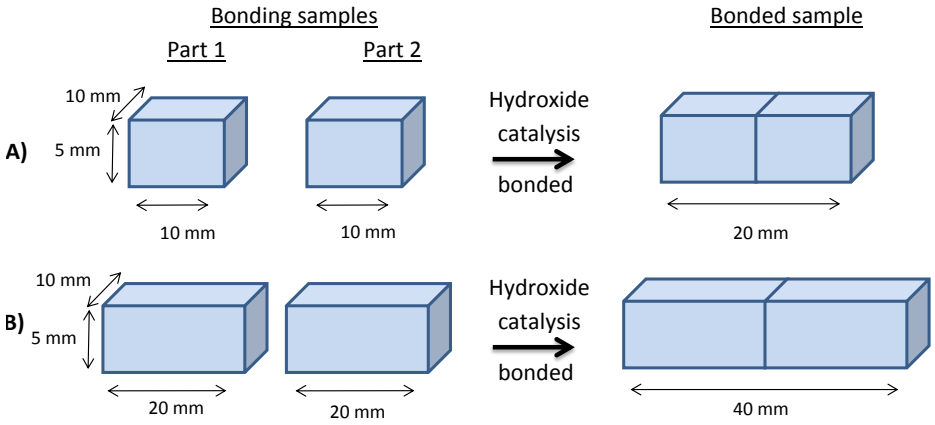

FIG. 4. A schematic diagram of fused silica samples used for hydroxide catalysis bond strength testing. A) Samples have a chamfered bonding surface area of 4.7 mm × 9.7 mm and are 10 mm in length. B) Samples have a chamfered bonding surface area of 4.7 mm × 9.7 mm and are 20 mm in length.

B. Hydroxide catalysis bonding

The hydroxide catalysis bonds were created using the same bonding solution and procedure as used in aLIGO suspensions [39]. Therefore, the fused silica substrates to be bonded were cleaned thoroughly using a paste formed from cerium oxide and deionised water, followed by a second paste formed from bicarbonate of soda and deionised water before being rinsed with methanol [20]. This procedure ensures that the bonding surfaces are hydrophilic and free from contaminants which could prevent a good bond from being formed. The bonding solution used was a commercial sodium silicate solution[3] diluted to one part sodium silicate (14% NaOH, 27% SiO_2 and 59% H_2O) to six parts deionised water. A volume of 0.4 μl of the solution per cm^2 of bonding surface was used as this volume has been shown to be sufficient to cover the bonding area [13]. After bonding, the samples were left to cure for four weeks at room temperature.

As discussed in Sec. II A, only bonds which are observed to have a bond area which is free of visual defects over $\geq$ 90 % of the area are considered acceptable for use in practical suspensions [39]. Therefore the quality of each bond is recorded and the strength results averaged for all samples and averaged only for the samples of gravitational wave detector quality will be discussed.

C. Strength Tester

The strength of the bonds were measured at room temperature using a 4-point strength testing arrangement inserted into a Zwick-Roell static 2 kN machine [40]. This experimental arrangement is discussed in Refs. [18, 20, 31, 34, 36] and a schematic representation is shown in Fig. 5. The applied force at which the sample fractures is recorded and the tensile strength of the bond can be calculated using,

$$\sigma_{\text{tensile}} = \frac{3}{2}\frac{F(L-l)}{bd^2}, \tag{4}$$

[2] www.corning.com
[3] www.sigmaaldrich.com

where F is the breaking force, L is the separation between the two lower contact points, l is distance between the two upper contact points, b is the width and d is the thickness of the sample.

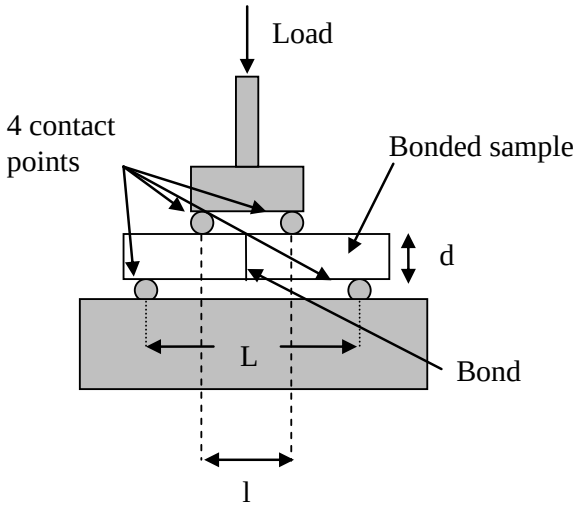

FIG. 5. A schematic diagram of a bonded sample in the 4-point strength testing arrangement. b is the width of the sample directly into the page.

D. Effect of a 150°C heat treatment for 48 hours

As shown in Sec. II C, heat treating bonds could potentially be of interest when implementing upgrades to aLIGO due to it benefits to bond mechanical loss. It is clearly important to understand fully any effect this would have on other bond parameters, such as the bond strength.

Two sets of 10 bonds were created using forty fused silica blocks of type (A) shown in Fig. 4. After a four week curing time, one set was heated in air to 150°C for 48 hours, the second set received no heat treatment to allow it to act as a reference. The temperature and length of time were selected to allow the evaporation of water without affecting the structure of the material or thermally shocking the bonds. The tensile strength of the samples were then measured in the experimental set-up described in Sec. III C.

Results

Fig. 6 and Tab. I show the resultant bond strengths measured for reference bonds (no heat treatment) and bonds which were heat treated at 150°C for 48 hours when including samples of all visual qualities. The heat treatment appeared to increase both the average bond strength and standard deviation. If only bonds which are $\geq 90\,\%$ bonded are considered, then this heat treatment was found to increase the bond strength by a factor of 2, from 13 ± 2 MPa to 26 ± 1 MPa, whilst the standard deviation decreased from 6 MPa to 4 MPa. This decrease in the standard deviation indicates that the heat treatment also increases the likelihood of having strong bonds. It was further noted that this slow heat treatment did not degrade the bonds visually.

E. Effect of the fibre welding procedure on strength of hydroxide catalysis bonds

During the construction of the suspension in a gravitational wave detector, the hydroxide catalysis bonds which attach the fused silica ears to the mirrors experience a heat load. This is due to the fibre welding procedure in which a CO_2 laser is used to weld the ends of the silica fibres to the weld horns on the fused

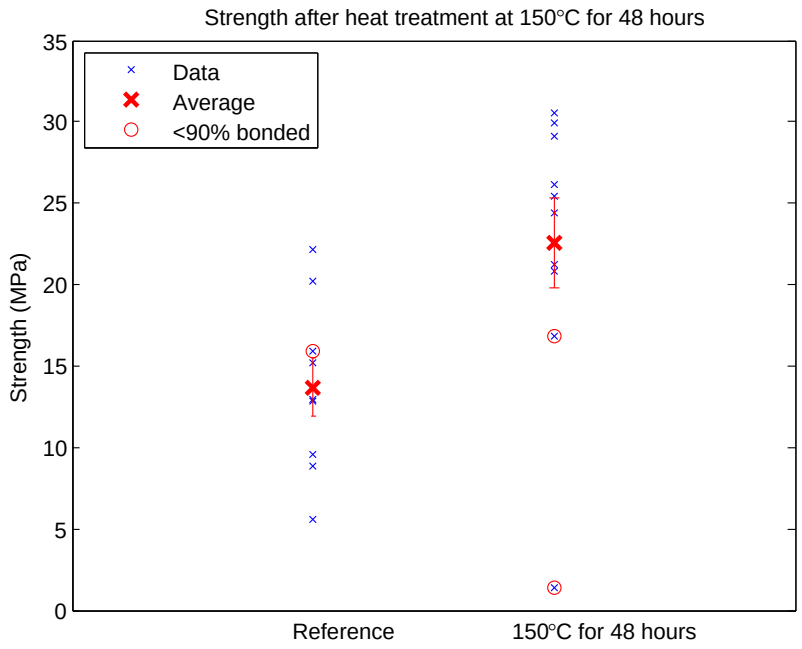

FIG. 6. Tensile strength of reference bonds and bonds which have been heated to 150 °C for 48 hours. (Geometry type (A), $L = 18\,\text{mm}$, $l = 8\,\text{mm}$, $b = 9.7\,\text{mm}$, $d = 4.7\,\text{mm}$)

	All Samples (MPa)			No defects over >90% (MPa)		
	Strength	Standard Deviation	Number of samples	Strength	Standard Deviation	Number of samples
Reference	14 ± 2	5	10	13 ± 2	6	9
150 °C for 48 hours	23 ± 3	9	10	26 ± 1	4	8

TABLE I. Average tensile strength of reference samples and samples which were heated at 150°C for 48 hours in air (Geometry type (A), $L = 18\,\text{mm}$, $l = 8\,\text{mm}$, $b = 9.7\,\text{mm}$, $d = 4.7\,\text{mm}$). The standard error on the mean is shown with the mean strength and the standard deviation and number of samples are also shown.

silica ears. The strength of hydroxide catalysis bonds which had experienced a heat treatment regime that simulated this thermal load was studied. Due to the location of the weld horns, and therefore the location at which the heat is applied, with respect to the bond area, a thermal gradient is present [41]. The area of the bond closest to the weld horns never exceeds temperatures of 340°C during the welding process which has a maximum duration of 12 minutes [41, 42]. Twenty bonds were created using forty fused silica samples of type (B) (see Fig. 4) following the procedure described in Sec. III B. After curing, ten of the bonds were placed in an oven, which had been preheated to 350°C, for 12 minutes. The temperature and time were chosen to assess the worst case scenario for the welding procedure. After the 12 minute time period, the bonds were removed from the oven and allowed to cool naturally at room temperature. The remaining bonds were not heat treated and function as the reference.

Results

The strengths of both the set of reference samples (which were not heat treated) and the strengths of the samples after heat treatment are shown in Fig. 7 and Tab. II. For the bonds which originally had no visual defects over 90 % of the bond area and which underwent simulated welding conditions, the average strength measured is within error of that obtained for reference bonds which had not undergone any heat treatment. However, the spread of strengths measured for the heat treated results was greater than the reference results with the standard deviation doubling from 5 to 10 MPa. It was observed that bonds experienced a slight

decrease in their visual clarity due to the heat treatment. However, the bonds which were originally of high visual quality were not observed to degrade as much as bonds which had visual defects prior to undergoing the treatment. If the samples with visual defects are removed from the analysis, the average strength for both sets are still within error of each other but are slightly higher values and the standard deviation drops to 3 MPa for the reference set and 6 MPa for the heat treated samples.

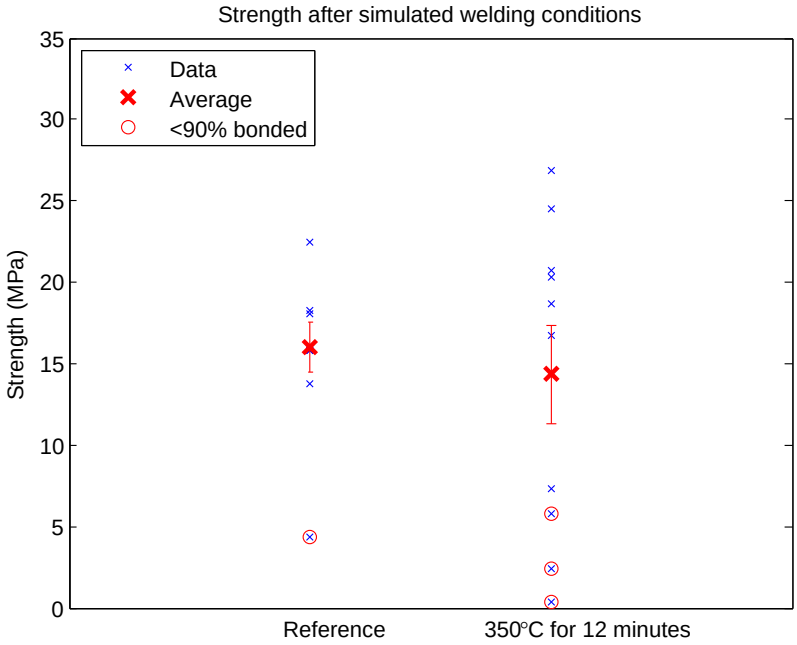

FIG. 7. Tensile strengths of bonds which have been heat treated (350°C for 12 minutes) to simulate the thermal conditions which would be caused by the aLIGO welding procedure (Geometry type (B), $L = 34$ mm, $l = 20$ mm, $b = 9.7$ mm, $d = 4.7$ mm).

	All Samples (MPa)			No defects over >90% (MPa)		
	Strength	Standard Deviation	Number of samples	Strength	Standard Deviation	Number of samples
Reference	16 ± 2	5	10	17 ± 1	3	9
Welding conditions	14 ± 3	10	10	19 ± 2	6	7

TABLE II. Average tensile strength of samples (Geometry type (B), $L = 34$ mm, $l = 20$ mm, $b = 9.7$ mm, $d = 4.7$ mm) which have undergone simulated welding conditions (350°C for 12 minutes). The standard error on the mean is shown with the mean strength and the standard deviation and number of samples are also shown.

F. Discussion

The heat treatment procedure of 48 hours at 150°C does not appear to visually degrade bonds and could potentially be beneficial in increasing bond strength. This supports results from Green et al. [33] in which the authors have shown that a similar heat treatment (200°C for 24 hours) of hydroxide catalysis bonded Zerodur and ULE causes an increase in bond strength. The increase in strength could be caused by the evaporation or diffusion of water from the bond due to the elevated temperature or possibly the removal of stresses from the sample due to an annealing effect.

The simulated laser welding procedure of aLIGO does not reduce the strength of the bonds, but it does increase the standard deviation of the strength results obtained. There could be two reasons for the increase in standard deviation. Firstly, an increase in strength could be caused by the heat aiding the evaporation or

diffusion of water from the bonds or removing stresses via an annealing effect. However, a decrease could also be expected in imperfect bonds that had bubbles or other imperfections in the bond area. The thermal shock from the heat treatment could expand the air inside these bubbles rapidly which could push the bonded substrates apart. The visual quality of the bonds were seen to be slightly degraded by the thermal load in this simulation and this could be an indication of this rapid thermal expansion of the air. Further evidence is that the level of degradation appeared to be larger if the bonds were imperfect prior to the heating. For the bonds in which this happened we would expect weaker strengths. Therefore the initial quality of the bond prior to the heat treatment is of great importance.

The appearance of visual degradation to bonds after the welding heat treatment simulation does not agree with what has previously been seen in test suspensions and the detector suspensions. In these suspensions there was no visual change to the bond area related to the fibre welding procedure [41]. However, the bonds used in the test suspensions and detectors were of very high visual clarity. The observed difference may also be caused by the slightly different heating geometry in the test to the actual fibre welding procedure. For example, in the tests the whole sample is heated while in the welding procedure the heat source is localized and other areas are probably cooler than in the test.

IV. CONCLUSION

Investigations into methods of improving the mechanical loss and strength of hydroxide catalysis bonds for future upgrades to aLIGO and other gravitational wave detector suspensions were carried out. It is demonstrated that the mechanical loss of a bond decreases as the age of the bond increases (from after the initial 4 week cure to a three year period in air). This is an important factor since suspension systems can be used for many years after construction. Indeed, the mirrors in the GEO600 detector have been suspended for more than 13 years. However, it is noted that bonds in gravitational wave detectors will be suspended under vacuum and not at air pressure as the bond reported in this paper. It is also shown that a heat treatment of 48 hours at 150°C could be used to decrease further the mechanical loss of a bond (here the treatment was carried out on a 3 year old bond and we postulate that the same effect would be seen if it were carried out after the four week cure). This same heat treatment has also been shown to improve bond strength. The observed change could be caused by the heat treatment acting as a method of removing stresses and evaporating and diffusing water from within the hydroxide catalysis bonds thereby allowing for continued polymerisation.

This heat treatment is similar to a potential suspension cleaning regime which had been considered for the optics and optical components of aLIGO, 120°C for 48 hours down to pressures of order 10^{-9} mbar [43]. After initial consideration, it was decided not to carry out this procedure because of concerns over the effect of the heat on the highly reflective coatings on the mirror [44]. However, due to the benefit of this regime on the bond strength and bond loss presented here, it may be beneficial to reintroduce this procedure (e.g. after the ears are bonded but before the coatings are deposited) to potential future upgrades to gravitational wave detectors. The increase in bond strength could potentially allow for smaller area bonds to be created in gravitational wave detectors, thereby decreasing the volume of bond material required. Coupled with the decrease in the bond mechanical loss would therefore reduce the thermal noise contribution of the bonds to a gravitational wave detector mirror. Indeed, even before considering the further benefit of creating smaller area bonds, this paper has shown that bond thermal noise can be as low as 3.7×10^{-22} m/$\sqrt{\text{Hz}}$ at 100 Hz. This is approximately half of the allowed noise limit for the two bonds per test mass, 7×10^{-22} m/$\sqrt{\text{Hz}}$ at 100Hz, which is 10% of the noise budget for the whole test mass [45]. Therefore the mechanical loss results presented here have shown that the thermal noise of the test mass could be lower than the upper limit expected and could be reduced by approximately 5%.

The hydroxide catalysis bonds currently in the aLIGO suspension are subject to some procedures which involve heat during the suspension construction. In particular, the fibre welding procedure that potentially heats the bond area to a temperature of 350°C for a maximum of 12 minutes. The effect of this heat treatment on the strength of the bond was investigated and, although it was found to cause a slight increase in bond strength variability for all the samples, it was found to have no adverse effect on the strength of an aLIGO quality hydroxide catalysis bond.

ACKNOWLEDGMENTS

The authors would like to thank the UK Science and Technology Facilities Council (ST/I001085/1, ST/J000361/1 and ST/L000946/1), the Royal Society (DH120021 and RG1200367) and the University of Glasgow for the financial support which allowed us to carry out this research. We would also like to thank our colleagues in SUPA, the GEO600 and LSC-Virgo collaborations for their interest in this work. A. A. van Veggel is the holder of a Royal Society Dorothy Hodgkin Fellowship and research grant. Thanks also go to Mr John Davidson from the University of Glasgow School of Engineering for his help with the strength testing apparatus. Although at the time of the work Stuart Reid was based at the University of Glasgow, he has since moved institutions and is now a professor at the University of the West of Scotland.

REFERENCES

[1] B. Abbott *et al.*, Reports on Progress in Physics **72**, 076901 (2009).
[2] T. Accadia *et al.* (VIRGO), JINST **7**, P03012 (2012).
[3] H. Grote (LIGO Scientific), *Gravitational waves. Proceedings, 8th Edoardo Amaldi Conference, Amaldi 8, New York, USA, June 22-26, 2009*, Class. Quant. Grav. **27**, 084003 (2010).
[4] M. Pitkin, S. Reid, S. Rowan, and J. Hough, Living Reviews Relativity **253**, 13 (2011).
[5] LIGO Scientific Collaboration and Virgo Collaboration, Phys. Rev. Lett. **116**, 061102 (2016).
[6] B. Abbott, R. Abbott, T. Abbott, M. Abernathy, F. Acernese, K. Ackley, C. Adams, T. Adams, P. Addesso, R. Adhikari, *et al.*, Physical Review Letters **116**, 241103 (2016).
[7] LIGO Scientific Collaboration and Virgo Collaboration (LIGO Scientific and Virgo Collaboration), Phys. Rev. Lett. **118**, 221101 (2017).
[8] H. Lück *et al.*, AIP Conf. Proc. **14** (2000).
[9] G. Harry and LIGO Scientific Collaboration, Classical and Quantum Gravity **27**, 084006 (2010).
[10] F. Acernese, M. Agathos, K. Agatsuma, D. Aisa, N. Allemandou, A. Allocca, J. Amarni, P. Astone, G. Balestri, G. Ballardin, *et al.*, Classical and Quantum Gravity **32**, 024001 (2015).
[11] D. H. Gwo, US Patent 6,284,085 (2001).
[12] D. H. Gwo, US Patent 6,548,176 B1 (2003).
[13] D. H. Gwo, SPIE's International Symposium on Optical Science, Engineering, and Instrumentation, International Society for Optics and Photonics , 136 (1998).
[14] D. H. Gwo, S. Wang, K. Bower, D. Davidson, P. Ehrensberger, L. Huff, E. Romero, M. Sullivan, K. Triebes, and J. Lipa, Advances in space research **32**, 1401 (2003).
[15] S. Rowan, S. Twyford, J. Hough, D. Gwo, and R. Route, Physics Letters A **246**, 471 (1998).
[16] S. Reid, G. Cagnoli, E. Elliffe, J. Faller, J. Hough, I. Martin, and S. Rowan, Physics Letters A **363**, 341 (2007).
[17] E. Elliffe, J. Bogenstahl, A. Deshpande, J. Hough, C. Killow, S. Reid, D. Robertson, S. Rowan, H. Ward, and G. Cagnoli, Classical and Quantum Gravity **22**, S257 (2005).
[18] A. Van Veggel, D. Van Den Ende, J. Bogenstahl, S. Rowan, W. Cunningham, G. Gubbels, and H. Nijmeijer, Journal of the European Ceramic Society **28**, 303 (2008).
[19] L. Cunningham *et al.*, Physics Letters A **374**, 3993 (2010).
[20] N. Beveridge *et al.*, Classical and Quantum Gravity **28**, 085014 (2011).
[21] LIGO scientific collaboration, LIGO documnet number T1200199-v2 (2012).
[22] A. Cumming *et al.*, Classical and Quantum Gravity **29**, 035003 (2012).
[23] H. Kim and T. Schmitz, Precision Engineering **37**, 23 (2013).
[24] P. Sneddon *et al.*, Classical and Quantum Gravity **20**, 5027 (2003).
[25] A. Cumming, *Aspects of mirrors and suspensions for advanced gravitational wave detectors*, Ph.D. thesis, University of Glasgow (2008).
[26] K. Haughian, *Aspects of material research for advanced and future generations of Gravitational wave detectors*, Ph.D. thesis, University of Glasgow (2012).
[27] H. J. Pain, *The physics of vibrations and waves* (John Wiley and sons, 2005).
[28] P. G. Murray, *Measurement of the mechanical loss of test mass materials for advanced gravitational wave detectors*, Ph.D. thesis, University of Glasgow (2008).
[29] S. Penn, A. Ageev, D. Busby, G. Harry, A. Gretarsson, K. Numata, and P. Willems, Physics Letters A **352**, 3 (2006).
[30] W. Cunningham, LIGO document number T080163-06 (2009).

[31] A. Van Veggel, J. Scott, D. Skinner, B. Bezensek, W. Cunningham, J. Hough, I. Martin, P. Murray, S. Reid, and S. Rowan, Classical and Quantum Gravity **26**, 175007 (2009).
[32] A. Dari, F. Travasso, H. Vocca, and L. Gammaitoni, Classical and Quantum Gravity **27**, 045010 (2010).
[33] K. Green, J. Burke, and B. Oreb, Optical Engineering **50**, 023401 (2011).
[34] N. Beveridge, A. Van Veggel, L. Cunningham, J. Hough, I. Martin, R. Nawrodt, S. Reid, and S. Rowan, Classical and Quantum Gravity **30**, 025003 (2013).
[35] H. S. Kim and T. L. Schmitz, Precision Engineering **37**, 23 (2013).
[36] R. Douglas, A. van Veggel, L. Cunningham, K. Haughian, J. Hough, and S. Rowan, Classical and Quantum Gravity **31**, 045001 (2014).
[37] K. Haughian, R. Douglas, A. van Veggel, J. Hough, A. Khalaidovski, S. Rowan, T. Suzuki, and K. Yamamoto, Classical and Quantum Gravity **32**, 075013 (2015).
[38] A. A. van Veggel and C. J. Killow, Advanced Optical Technologies **3**, 293 (2014).
[39] A. A. van Veggel and H. Armandula, LIGO document number E050228-v2 (2010).
[40] ASTM International C1161-02c (2008).
[41] A. Cumming, A. Bell, L. Barsotti, M. Barton, G. Cagnoli, D. Cook, L. Cunningham, M. Evans, G. Hammond, G. Harry, and K. Tokmakov, Classical and Quantum Gravity **29** (2012), 10.1088/0264-9381/29/3/035003.
[42] Private communication between A V Cumming and K Haughian (2009).
[43] Private communication between A A van Veggel and B Weaver (2014).
[44] M. Phelps, LIGO document number E1100439-v6 (2013).
[45] A. A. van Veggel, A. Bell, A. Cumming, L. Cunningham, G. Hammond, K. Haughian, J. Hough, R. Jones, R. Kumar, K. Strain, and S. Rowan, LIGO document number T0900447-v2 (2009).