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The effect of crystal orientation on the cryogenic strength of hydroxide catalysis bonded sapphire

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Abstract

Hydroxide catalysis bonding has been used in gravitational wave detectors to precisely and securely join components of quasi-monolithic silica suspensions. Plans to operate future detectors at cryogenic temperatures has created the need for a change in the test mass and suspension material. Mono-crystalline sapphire is one candidate material for use at cryogenic temperatures and is being investigated for use in the KAGRA detector. The crystalline structure of sapphire may influence the properties of the hydroxide catalysis bond formed. Here, results are presented of studies of the potential influence of the crystal orientation of sapphire on the shear strength of the hydroxide catalysis bonds formed between sapphire samples. The strength was tested at approximately 8 K; this is the first measurement of the strength of such bonds between sapphire at such reduced temperatures. Our results suggest that all orientation combinations investigated produce bonds of sufficient strength for use in typical mirror suspension designs, with average strengths >23 MPa.

Keywords: hydroxide catalysis bonding, gravitational waves, sapphire

(Some figures may appear in colour only in the online journal)



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1. Introduction

A number of interferometric gravitational wave detectors exist around the world. These long-baseline interferometers are designed such that gravitational waves caused by astrophysical events such as the coalescence of binary neutron stars should induce measurable relative displacements of the interferometer mirrors. In order to be sensitive to such induced displacements (of order 10^{-18} m or less at audio frequencies [1]), it is essential that all sources of noise, including seismically induced noise and the thermal noise of the mirrors themselves, are minimized. There are a number of these detectors; the LIGO project [2], which operates long baseline (4 km arm length) interferometers at two sites in the US, GEO600 [3], which operates a detector with 600 m arms in Germany, and the Virgo project [4], which operates a detector with 3 km arms in Italy. Of these detectors, GEO600 is currently running while the LIGO and Virgo detectors are being upgraded to ‘advanced’ forms. A further detector, ‘KAGRA’ is currently under construction [5]. KAGRA is sited in Japan and will have 3 km arms.

GEO600 was the first interferometer in which the hydroxide catalysis bonding technique was implemented to create quasi-monolithic suspensions of fused silica to support the interferometer mirrors [6]. This bonding technique was first invented for use in the Gravity Probe B project [7] and developed further for gravitational wave detector applications [6, 8, 9]. In the mirror suspensions used in the GEO600 instrument, small interface pieces, or ‘ears’ were hydroxide catalysis bonded to the side of the 6 kg fused silica mirrors, providing welding points for fused silica suspension fibres [6, 9, 10]. The stability, vacuum compatibility, and strength of this bonding technique, when combined with the ability to form very thin joints made this an attractive method of forming low-thermal noise mirror suspensions [6]. In more detail, hydroxide catalysis bonding is a method for chemically joining materials at room temperature using a hydroxide solution, through hydration and etching, polymerization and dehydration of the surfaces as described for example by Gwo *et al* [10]. The bonds created are strong and can be precisely aligned [11]. Due to its demonstrated benefit to sensitivity, the technique was further developed and has been adopted by other gravitational wave detector projects [12, 13].

In order to further increase detector sensitivity, detector designs which have even lower levels of mirror and suspension thermal noise, are of interest. For KAGRA, for future upgrades to ‘advanced’ gravitational wave detectors and for future generations of detectors such as the ‘Einstein Telescope’ concept, operation at cryogenic temperatures is an option of considerable interest, where low temperature operation at 10–20 K of the key optical components may allow significant reductions in thermal noise [5, 14]. At low temperatures, fused silica has a broad dissipation peak so it becomes less attractive to use as a material for the suspensions and mirror substrates [15]. Silicon and sapphire however have thermomechanical and optical properties which make them interesting as candidate materials for such cryogenically cooled detectors [16–24].

In particular, there have been a number of studies targeted at the use of large single crystals of sapphire as a mirror substrate material for advanced interferometric gravitational wave detectors [25–31]. In general, these conclude that to minimize the effects of birefringence in transmissive optics, it is desirable to use crystals cut such that the (0001) plane (i.e. the ‘c-plane’) of the crystal is in a plane perpendicular to the plane of incidence of the laser beam hitting the mirror. (Or equivalently that the *c*-axis of the crystal forms the cylindrical axis of the mirror.) Further, recent studies of the possibility of using commercially-grown sapphire fibres as suspension elements for the sapphire mirrors have focussed on fibres in which the cylindrical axis again corresponds to the *c*-axis of the material [32]. As in other

advanced gravitational wave detectors, it is likely that ears may be used to attach the ends of the fibres to the mirrors [33]. Thus in a crystalline material system typical bonds could be formed between dissimilar axis or planes; the c -plane of the ear may be bonded to the m -plane or the a -plane flats polished on the sides of the mirror and the c -plane of the fibre ends could be bonded to the m -plane or the a -plane of the ear. Thus, for quasi-monolithic suspension designs, the properties of bonds formed between dissimilar crystal axis and planes are of particular interest.

In gravitational wave detectors such as GEO600 and LIGO, the suspension designs are such that the hydroxide catalysis bonds are predominantly under shear stress. The final suspension design for KAGRA has not yet been determined and therefore it is of interest to understand the bonds' behaviour under a variety of forms of stress [33–35]. Initial investigations into the strength of hydroxide catalysis bonds created between sapphire substrates have been carried out where the bonded surfaces had nominally identical surface orientations. For example, average tensile strengths of approximately 65 MPa were found for m -plane to m -plane bonds formed using a sodium silicate bonding solution [36]. These studies also suggested that the strength was not degraded when the samples were cooled to liquid nitrogen temperatures of approximately 77 K [36]. Other authors (Suzuki *et al* [37]) found a torsional shear strength of 6.5 MPa for a c -plane to c -plane bond using a potassium hydroxide bonding solution, while Dari *et al* [38] have measured shear strengths of approximately 2 MPa for c -plane to c -plane bonds using a potassium hydroxide bonding solution. Here we present torsional shear strength results at approximately 8 K.

As the hydroxide catalysis bonding technique involves chemical etching of the surfaces to be jointed, it is of interest to quantify any effects of the crystalline structure and orientation of the material being jointed on bond formation and the strength of the resultant bond [39]. Thus the studies in this paper investigate how changing the crystal orientation of the bonded sapphire surfaces effects the torsional shear strength of the bonds formed. Before being strength tested, the optical clarity of each bond was recorded as the presence of visual defects can make the bonds unsuitable for some applications [12]. The strength of both the optically clear bonds and the bonds with visual defects are shown here for completeness. In order to investigate the strengths of hydroxide catalysis bonds at temperatures reached in a typical cryogenic gravitational wave detector, the bonds were cooled down with liquid helium to approximately 8 K for strength testing. These strength tests were carried out on a custom-built torsional shear strength tester at the High Energy Accelerator Research Organisation in Japan.

2. Hydroxide catalysis bonding

Hydroxide catalysis bonding is a technique that can be used to chemically join materials which can attach to or form a silicate or aluminate-like network. An aqueous bonding solution containing a hydroxide is placed on one bonding surface and the second surface is brought into contact. The bonding process then begins. It consists of three main steps; hydration and etching, polymerization and dehydration. An extensive description of the chemistry involved is given by Gwo [10]. The curing time for a hydroxide catalysis bond formed with sodium silicate solution between fused silica and glass substrates has been found to be approximately 4 weeks [6, 40, 41]. The surfaces to be joined are typically cleaned in a clean room environment to avoid contamination in the bond or obstruction to the chemical process [10] and ideally, the surfaces to be jointed should be 'optically flat' (global peak-to-valley $\leq \lambda/10$ where $\lambda = 633$ nm) [40]. This allows for very thin bonds to be created with a typical thickness of bond being measured to be (61 ± 4) nm [42] between silica substrates and (47 ± 15) nm [43]

between silicon substrates. Creating a thin bond is important for gravitational wave detector applications in order to keep the thermal noise contribution from the bond to the overall suspension noise low.

2.1. Bonding procedure

Three sets of samples were obtained from Moltech gmbh⁴ where in each set a different crystal axis was perpendicular to the bonding surface, the crystal axis being c , a or m . A diagram showing the geometry and crystal orientation of the samples is shown in figure 1. The samples were cuboids, $5 \times 5 \times 10$ mm and the bonding surface in each case was the 5×5 mm face. Due to the hardness of sapphire it can prove difficult to polish, thus each sample was characterized to ensure suitability for bonding [31, 44]. These measurements were carried out using a ZYGO GPI XP/D interferometer⁵. The flatness of each sample was recorded and any samples with a flatness value less than $\lambda/4$ were deemed acceptable for the bonding tests. From these, three different combinations of bonded samples were made. One set of samples was bonded such that the c -plane of the first sample was bonded to the a -plane of the second sample, the second set was bonded such that the c -plane of the first sample was bonded to the m -plane of the second sample and the third set was produced where the c -plane of the first sample was bonded to the c -plane of the second sample. These combinations were selected to investigate the best option for bonding the current KAGRA optics as discussed in section 1. A comprehensive cleaning regimen involving application to the surfaces of a paste made from cerium oxide and deionized water, followed by cleaning with bicarbonate of soda paste, and then methanol was used for all the samples. This ensures the cleanliness of the surface and increases surface hydrophilicity allowing the solution to flow smoothly over the whole surface and aiding the hydration step of the bonding process [45]. A commercially available sodium silicate bonding solution (14% NaOH and 27% SiO₂) was diluted to a volumetric ratio of 1:6 with deionized water. A volume of $0.4 \mu\text{l}$ of solution per cm^2 of bonding area was used to join the surfaces as this is deemed to be the smallest amount required to cover the surface and produce strong bonds [46]. After bonding, the optical clarity of the bond was recorded as an indicator of the success of the bond. Bonds that are optically clear and without visible defects over 95% of the surface area are acceptable for use in gravitational wave detector suspensions [47]. Table 1 shows the number of samples of each bond type tested and indicates the number of samples that are without optical defect over 90–95% of the surface area. All bonds were left to cure for one week at room temperature in Glasgow before being transported to Japan and left to cure for a further three weeks. A photograph of a bonded sample is shown in figure 2.

3. Strength testing

The shear strength measurements of the bonded samples were carried out at the High Energy Accelerator Research Organisation, Japan, in a custom-built system which had been used previously to measure the shear strength of a hydroxide catalysis bond at room temperature [37]. A diagram of the experimental apparatus and a photograph of a loaded sample are shown in figure 3.

The samples were secured in the sample holder which was submerged directly into a liquid helium dewar, allowing the liquid helium to wet the sample. The sample was lowered

⁴ www.mt-berlin.com

⁵ www.zygo.com

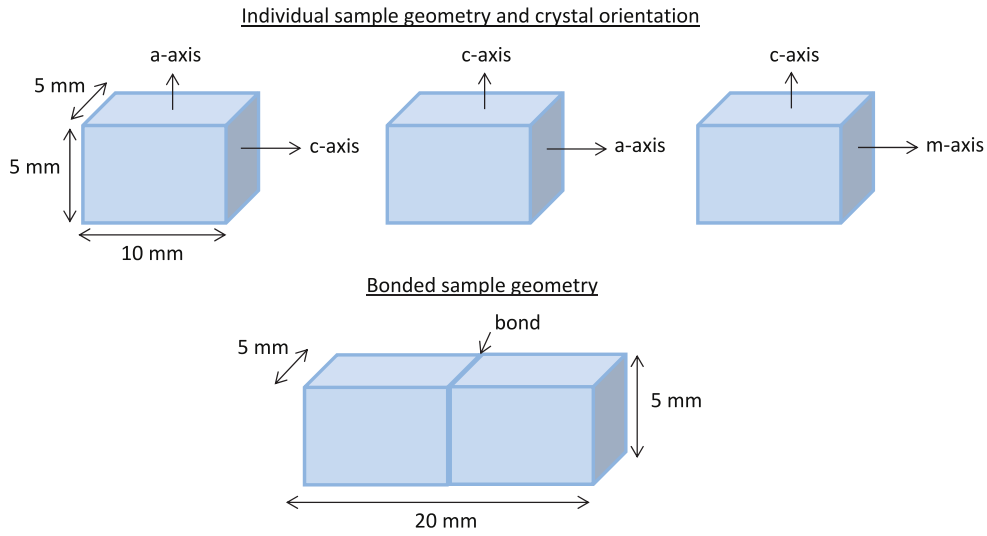


Figure 1. Diagram showing the geometry and crystal orientation of the unbonded samples and the geometry of a bonded sample.

Table 1. Number of samples tested for each bond type and the visual quality of the bonds achieved.

Bond type	Total bonded samples	>90–95% optically clear
<i>c</i> -plane to <i>a</i> -plane	8	5
<i>c</i> -plane to <i>m</i> -plane	9	8
<i>c</i> -plane to <i>c</i> -plane	10	8

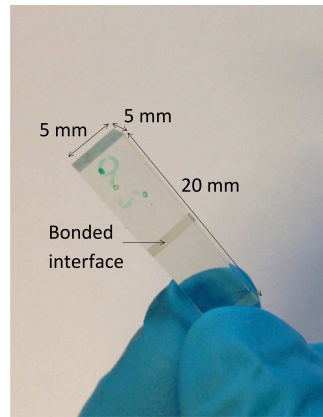


Figure 2. Two sapphire cuboids jointed using hydroxide catalysis bonding. The bonding solution used was 1 part sodium silicate to 6 parts deionized water. A volume of $0.4 \mu\text{l}$ per cm^2 of bonding area was used.

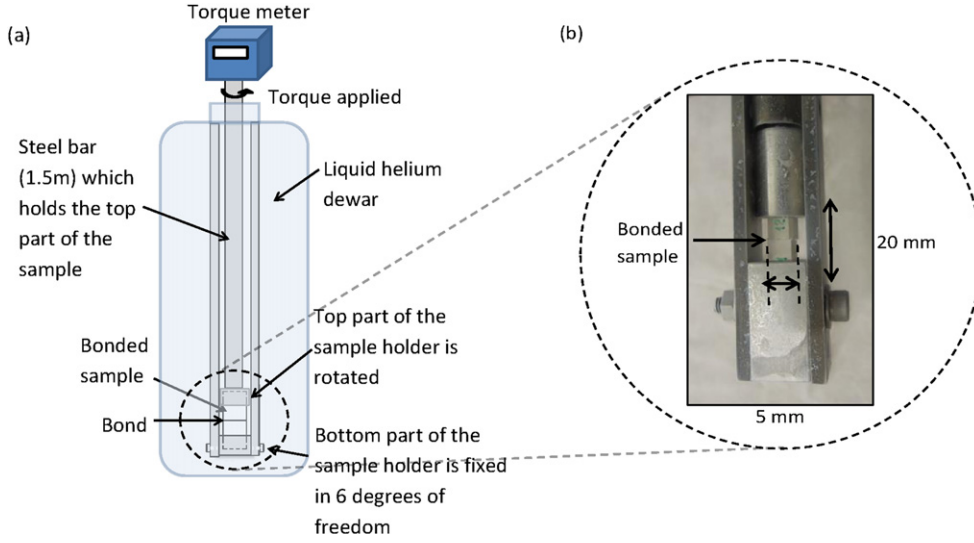


Figure 3. Experimental apparatus used to measure the torsional shear strength of the sapphire bonded samples at liquid helium temperatures. (a) A schematic diagram of the whole set-up, (b) a photograph of a sample loaded into the holder.

slowly into the helium in order to avoid thermally shocking the bond or material. It took approximately 2–3 min to fully submerge the sample. Once the sample was fully submerged and thermal equilibrium had been achieved, (indicated by the settling of the helium after it had been boiling due to the starting temperature of the sample and holder) a torque was applied to the top of the sample holder which rotated a long metal bar and the top of the sample. The maximum torque applied, T_{applied} , before the sample broke was recorded with a Sugisaki-keiki co., DI-9 F IP5RG torque meter. The set-up was designed to have a close fit between the metal bar applying the force to the sample and the outer tube into which it fit to prevent any misalignment of the sample. Thus some level of frictional contact could occur between the rotating and rigid parts of the sample holder. The same process was thus carried out several times with no sample in the holder in order to evaluate the torque required to overcome this friction, $T_0 = 0.058 \text{ Nm}$, and therefore allow the torque, T , required to break the samples to be calculated, using $T = T_{\text{applied}} - T_0$.

As the resultant of the stress distribution is equal to the torque, T , we can obtain an average value of the torsional shear stress at the breaking point, τ , using equation (1) [48]

$$T = \int \rho (\tau \, dA) = \tau \int \rho \, dA, \quad (1)$$

where A is the cross-sectional area and ρ is the distance from the axial centre. By using this method we assume that there is a uniform distribution of stress through the bond layer.

Finite element analysis was used to model the stress distribution through a sample as the torque is applied. The resulting stress in the bond location is shown in figure 4. It can be seen that the stress is not totally uniform but instead is at a minimum at the centre and increases to a maximum at the centre of the flat edges. Both the average shear stress and the maximum shear stress will be reported here. The maximum torsional shear stress was calculated using Prandtl's membrane analogy and was confirmed by Roark's formulas for stress and strain [48, 49]. Prandtl's equation [49] for the shear stress is shown in equation (2)

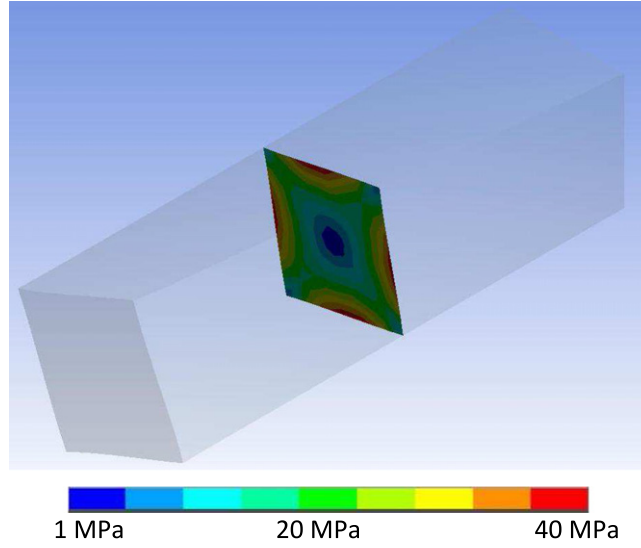


Figure 4. A finite element model showing the shear stress distribution in a cross-sectional surface at the centre of a monolithic sapphire bar caused by an applied torque of 1 Nm. The colour represents the magnitude of the stress, with red being the maximum and blue the minimum stress.

$$\tau_{\max} = \frac{T}{\alpha ab^2}, \quad (2)$$

where α is a constant related to the ratio of the length, a , to the width, b , of the cross-section of the sample under study. For the geometry used here, a square cross-sectional area, $a = b$ and $\alpha = 0.208$ [49].

3.1. Results

For all samples, both the calculated average assuming a uniform stress distribution and the maximum shear strengths of bonds obtained at liquid helium temperatures are shown in figure 5 and in table 2. In figure 5 an average value for each set is shown with error bars showing the standard error in the spread of results.

Bonds created between sapphire surfaces parallel to the c -plane and a -planes of the crystal had an average strength throughout the bond layer of 23 ± 3 MPa at liquid helium temperature and a maximum strength of 42 ± 5 MPa. This orientation combination gave the smallest spread of results with a standard deviation of 8 MPa on the averaged strength and 14 MPa on the maximum strength. The c -plane to m -plane bond set had an averaged strength of 33 ± 4 MPa and maximum strength of 60 ± 8 MPa. The standard deviations of the strength found here were 13 and 24 MPa respectively. As can be seen in figure 5, the average and spread of the strength results for this set were affected by a bond which was not optically clear over more than 90% of the bond area (reducing the average of both sets by between 6 and 10% and increasing the spread in results by approximately 8%). This could be caused by contamination or air bubbles trapped in the bond layer. The set of bonds with c -plane bonded to c -plane had an averaged strength of 48 ± 14 MPa and a standard deviation of 42 MPa and a maximum strength of 82 ± 27 MPa and a standard deviation of 80 MPa. This large standard

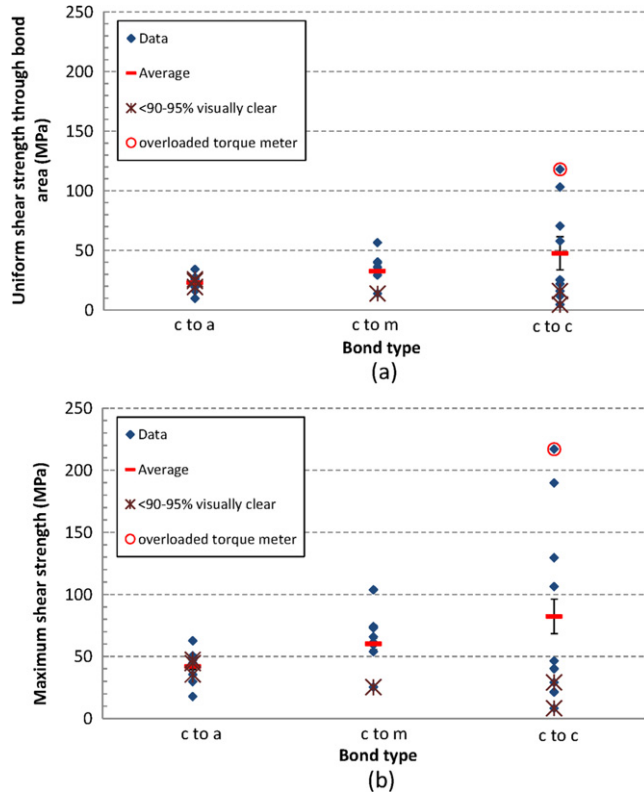


Figure 5. Shear strength results obtained for bonded sapphire samples at liquid helium temperature. (a) Uniform shear strength through the bond area. (b) Maximum shear strength. The results from three sets of samples are shown. The first set shows results for samples where a surface formed parallel to the c -plane of the crystal was bonded to one parallel to the a -plane, the second set shows strengths of c -plane bonded to m -plane and the third shows the strength of c -plane bonded to c -plane. The optical clarity of the bond is indicated on the plots. The average strength of all bonds and standard error on this value are given. A red circle indicates a bond which overloaded the torque meter and could not be broken.

deviation is caused by the fact that this set contained both the strongest and weakest bond created in this study. The strongest point in figure 5, denoted by a red circle, shows the maximum capability of the experimental set-up as it was not possible to break this particular bond. It is worth noting that even with the large spread in these results, the weakest bond which was optically clear over 90% of the bond area, 11.6 MPa for the stress being averaged through the bond created between two c -plane orientated bonding surfaces, is still very strong, approximately 70 times the minimum shear strength required for a hydroxide-catalysis bond created between fused silica substrates for an Advanced LIGO mirror suspension design [50]. The bond strength required for the KAGRA detector is at the same level to that of Advanced LIGO due to the similarity of the geometry of the mirror suspensions. In the majority of cases the sample broke at the bond. The bulk sapphire was only damaged in 4 of the 54 samples used.

Table 2. Average and maximum torsional shear strength for bonds created between two *c*-plane surfaces, between a *c*-plane and an *a*-plane surface and between a *c*-plane and a *m*-plane surface.

	Stress averaged through bond		Maximum stress in bond	
	Strength (MPa)	Standard deviation (MPa)	Strength (MPa)	Standard deviation (MPa)
<i>c</i> -plane to <i>c</i> -plane	48 ± 14	42	82 ± 27	80
<i>c</i> -plane to <i>a</i> -plane	23 ± 3	8	42 ± 5	14
<i>c</i> -plane to <i>m</i> -plane	33 ± 4	13	60 ± 8	24

4. Discussion and summary

Hydroxide catalysis bonds were created between surfaces with three different combinations of crystal orientation; *c*-plane to *a*-plane, *c*-plane to *m*-plane and *c*-plane to *c*-plane. These samples were then shear strength tested at approximately 8 K. Results suggest that the crystal orientation of the bonding surfaces does indeed have an influence on the strength of the hydroxide catalysis bond formed, but more notably it appears to have a strong influence on the spread in strength of the bonds formed and therefore on their reliability. In particular, the set of *c*-plane to *c*-plane bonds had an extremely large spread of strength values. A strength of 48 ± 14 MPa with a standard deviation of 42 MPa was obtained when assuming a uniform stress distribution through the bond. The *c*-plane to *a*-plane and the *c*-plane to *m*-plane bonding sample sets gave an average strength which was slightly lower but much more consistent than *c*-plane to *c*-plane: 23 ± 3 MPa with a standard deviation of 8 and 33 ± 4 MPa with a standard deviation of 13 MPa, respectively. In reality, the distribution of stress is not uniform through the bond layer and therefore the maximum strength for each bond set was also calculated. Strong bonds were demonstrated for all of these crystal orientation combinations, stronger than that required for a typical gravitational wave detector suspension design, for example, in a GEO600 suspension, a bond is under 0.16 MPa of shear stress caused by the weight of the mirror [50].

The bulk material of the sapphire sample was only damaged in a limited number of the tests; 4 out of 54 sapphire cuboids. This was observed to occur for samples which demonstrated some of the highest strengths. In all cases, the visible damage which occurred was a small chip on the side of the sample. As shown in figure 4, the stress was at a maximum around the circumference of the sample during a strength test. For these stronger bonds, it is possible that the stress on the edges became too high and caused the damage. In general, this type of damage is especially likely to occur if there is a weak point in the material at one of the edges due to a micro-crack, contamination or dislocation of the crystal structure.

The difference in strength of hydroxide catalysis bonds created between surfaces of sapphire samples formed from different crystal planes could be caused by a number of factors. The first of these is that the surface structure of the bonded planes could be different for differently orientated sapphire. Vardiman, Hockey and Williams *et al* [51–53] suggest that although a pristine sapphire surface is not readily etched by a hydroxide (for example potassium hydroxide, KOH), if the surface is damaged through mechanical polishing, dislocations or imperfections in the crystal, the KOH will etch/react with the surface which could possibly aid in forming aluminates or hydroxylating the surface. Vardiman *et al* also suggest that KOH more readily etches *c*-plane sapphire surfaces, albeit at elevated temperatures of ~ 300 °C.

A second possible explanation could be that *c*-plane sapphire surfaces more readily hydroxylate than sapphire surfaces of other orientations. Eng *et al* [54] state that the reactivity of metal oxides is strongly influenced by the degree of surface hydroxylation. They also find that *c*-plane sapphire in water at room temperature has a surface layer which has a semi-ordered crystal structure between fully hydroxylated alumina and crystalline alumina and is oxygen-terminated. It is possible that in the presence of a hydroxide the surface becomes fully hydrated due to a lowering of the activation energy. By measuring the zeta potential as a function of pH, Kreshner *et al* [55] reported that *c*-plane sapphire is more readily hydroxylated than *a*, *m* or *r*-plane sapphire.

Further, the possibility exists that surfaces of certain crystal planes may prove harder to clean than others [56]. This could be a direct consequence of the different surface structure or

level of hydroxylation discussed above. The cleanliness of a bonding surface is very important in creating good, strong bonds as discussed in section 2.

The bonded samples investigated here were cooled down from room temperature to liquid helium temperature for strength testing. Therefore, any difference in the thermal expansion of the materials must be considered. Indeed, the thermal expansion coefficient of sapphire varies depending on how the surface is orientated with respect to the crystal structure [57]. For example, at room temperature, the thermal expansion of the sapphire material parallel to the three fold axis of symmetry at $\sim 5.80 \times 10^{-6} \text{ K}^{-1}$, is larger than the thermal expansion perpendicular to the symmetry axis, $\sim 5.06 \times 10^{-6} \text{ K}^{-1}$, by a factor of ~ 1.15 [58]. This difference in thermal expansion between the two bonded surfaces could cause a variation in the strength of the bonds formed between differently orientated planes if it were causing the bond to be stressed differently. Also, the thermal expansion of the bond material is not yet known. Further investigations are therefore required in order to fully understand and quantify the magnitude of this effect.

Another possible reason for the difference in strength could be that the strength of the bulk material between which the bonds are formed could have a dependence on its crystal cut. Evidence for this has been reported by Fischer *et al* [59]. They reported that the *c*-plane orientation was stronger than the *m*-plane or *a*-plane (by a factor of approximately 35–50%), agreeing with the results presented here. However, as a result of the fact that we do not achieve strengths close to the bulk strength and that the bulk material was only damaged in a limited number of samples, it seems unlikely that the difference in bulk strength is the major factor in causing the difference in bond strength found here.

In conclusion, we find that while the reasons for the differing bond strengths at cryogenic temperature between sapphire samples of different cut require further study, in all cases the strength is sufficient to allow the use of hydroxide catalysis bonded sapphire in the suspensions of a cryogenic gravitational wave detector such as KAGRA [31].

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