Report on Bonding Techniques for Suspension Designs

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on behalf of ELiTES WP2

Introduction:

Experiments have been carried out by the above institutes to investigate the bonding of sapphire test pieces by hydroxide catalysis bonding. During year 2 of the ELiTES project, Rebecca Douglas, a PhD student at the University of Glasgow and Karen Haughian, a post-doctoral researcher at the University of Glasgow visited the ICRR at the University of Tokyo. This trip was designed to focus on bonding experiments with sapphire with the possibility of also measuring the thermal conductivity of bonds. This was also a knowledge transfer trip with the objective of training a group of researchers at the ICRR in the hydroxide catalysis bonding technique.

University of Glasgow, Friedrich Schiller University, ICRR University of Tokyo

Experiments and Results:

Experiments carried out during the ELiTES bonding trip:

Crystal orientation

27 bonds were made in Glasgow for breaking with Suzuki-san's shear strength set-up in liquid helium at KEK. These were made with sapphire samples as shown in figure 1.

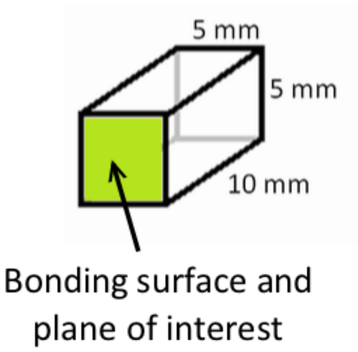

Figure 1. Sapphire samples from Moltech used in bonding experiments in which the shear strength of the bonds was measured.

Of these, different combinations of crystalline axes and cuts were bonded. 9 samples were bonded c axis-to-a axis ; 9 c axis -to-m axis and 9 c axis -to-c axis. These bonds were cured for one week at room temperature in Glasgow. They were then transported to Japan and allowed to cure at room temperature for a further four weeks. A schematic of the cryogenic shear strength test set-up is shown in figure 2 (a). The results from these bonds are shown in figure 3.

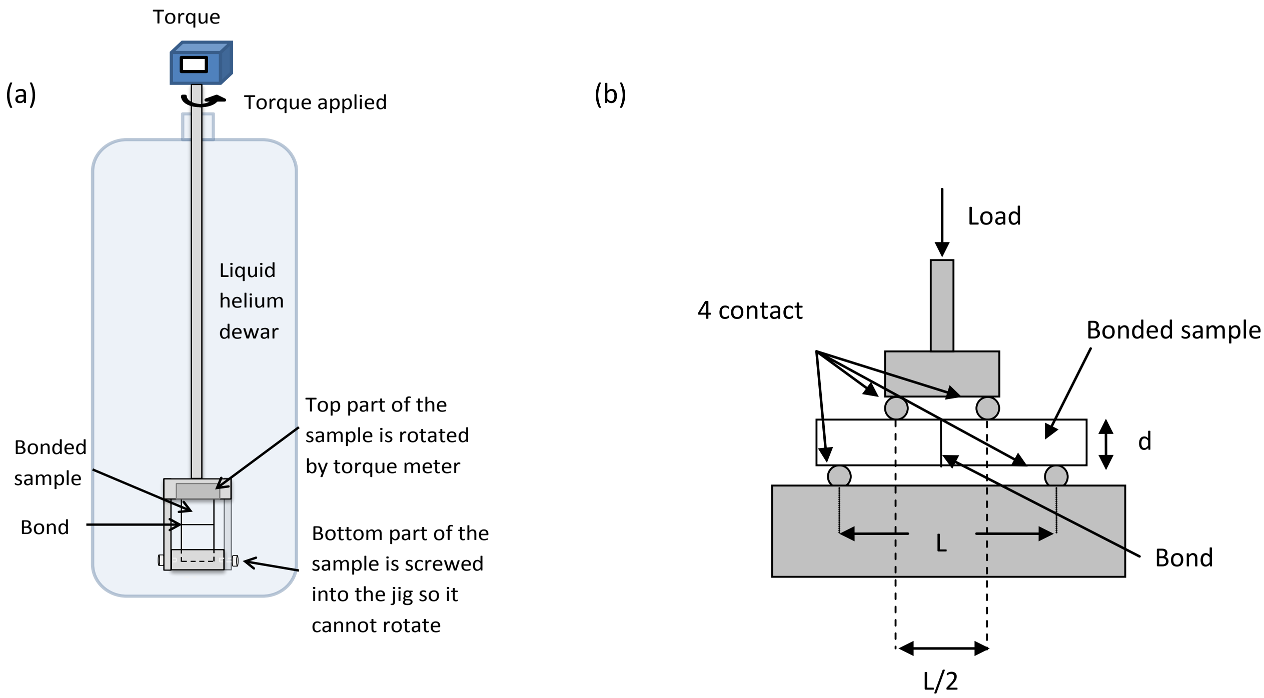

Figure 2: Strength testing equipment. (a) Schematic of the shear strength test set-up at KEK. This set-up can be immersed in liquid helium while a torque is applied from above. (b) Schematic of four-point bending test. This allows an even force to be applied from above whilst the sample is supported from below such that a constant bending moment is applied between the top supports causing a compressive stress at the top and a tensile stress at the bottom. The force is steadily increased by moving the upper stage downwards at a rate of 2 mm/min until the sample breaks, and the total force required to do so is recorded.

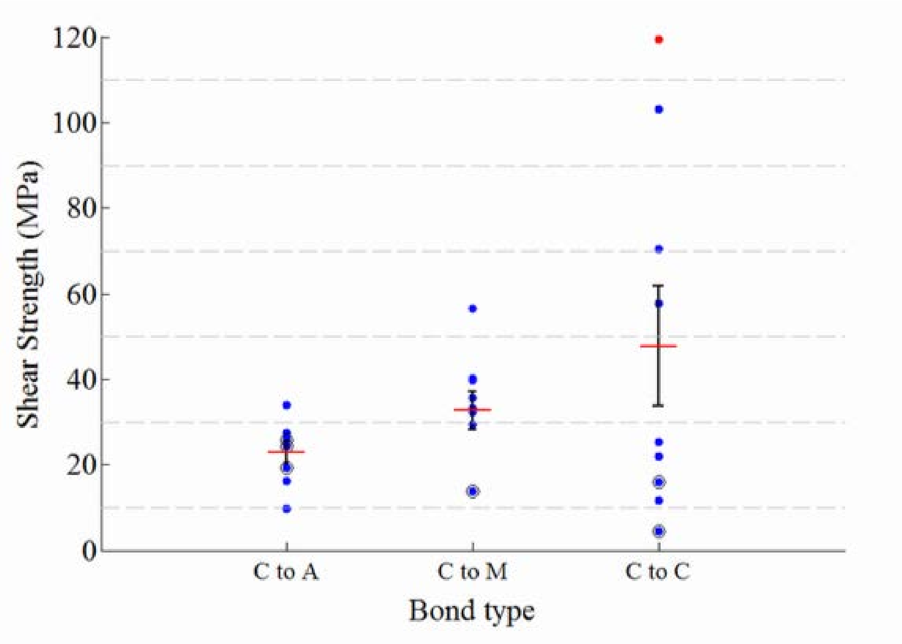

Figure 3. Cryogenic shear strength results from bonds made with samples bonded with different axes parallel to the bonding surface. Blue points are individual strengths, red bars are the mean and the error bars show the standard errors on the mean. The red point shows a bond that could not be broken with the shear strength test – this bond has in principal a strength even greater than 119 MPa. This first set of tests suggests that c axis -to-m axis type bonds may be most promising for KAGRA

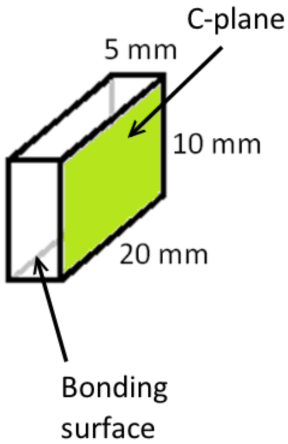

Figure 4. Crystran sapphire samples used in previous bonding tests in which the tensile strength of bonds were measured at Glasgow. Used here to provide a comparison of the two breaking strength tests

Effect of flatness and volume of bonding solution

Six other sets of bonds were produced during the ELiTES bonding trip at ICRR. These sets allowed for strength tests to be carried out on bonds with different amounts of bonding solution and also to strength test bonds created between a surface which has ~λ/4 flatness and a surface which is a lot less flat. These sets were:

1. A set of ten bonds were made using pre-used Crystran samples as shown in figure 4. See the section on recycling bond surfaces for more information.
2. Moltech samples bonded C-to-A with the C samples have flatnesses of ~λ/4 and the A samples having flatnesses of > λ/4. Ten bonds made. These will be ready for breaking after 6th January 2014. They should also be broken with the shear strength set-up in liquid nitrogen for comparison with the similar bonds whose strengths are shown in Figure 3. This is hoped to show the effect of bonding a flat surface to a less flat surface
3. Moltech samples bonded A-to-A with 0.4µl/cm^2 of solution All samples have a flatness of > λ/4 but samples were selected so that each pair had a combined flatness consistent with the rest in the set. Seven bonds made. These will be ready for breaking after 6th January 2014.
4. Moltech samples bonded A-to-A with 0.8µl/cm^2 of solution All samples have a flatness of > λ/4 but samples were selected so that each pair had a combined flatness consistent with the rest in the set. Seven bonds made. These will be ready for breaking after 6th January 2014. For comparison with set 3
5. Moltech samples bonded M-to-M with 0.4µl/cm^2 of solution All samples have a flatness of > λ/4 but samples were selected so that each pair had a combined

flatness consistent with the rest in the set. Seven bonds made. These will be ready for breaking after 6th January 2014.

6. Moltech samples bonded M-to-M with $0.8 \mu l/cm^2$ of solution All samples have a flatness of > λ/4 but samples were selected so that each pair had a combined flatness consistent with the rest in the set. Seven bonds made. These will be ready for breaking after 6th January 2014. For comparison with set 5

Sets 3-6 were also used as training sets to allow researchers at the ICRR to learn the hydroxide catalysis bonding technique.

One C-to-C bond made with Moltech samples was reserved and this will be used in attempts to measure the thickness of the bond layer.

Thermal Conductivity

Attempts at measuring the thermal conductivity of a bond layer between sapphire substrates were made. The results were inconclusive due to problems with thermal contacts. This is ongoing research.

Experiments carried out at University of Glasgow:

Investigation of bonding solutions and the effect of temperature on bond strength for hydroxide catalysis bonded sapphire [1]

100 sapphire samples were procured from Crystran. These had dimensions 5 x 10 x 20 mm as shown in the diagram in figure 3, above. With these, 9 bonds were made using sodium silicate solution, 10 using sodium aluminate, 10 using potassium hydroxide and 10 using sodium hydroxide.

All of these were cured for four weeks at room temperature in Glasgow. They were then broken at room temperature using a four-point bending test (also at Glasgow) to measure the tensile strength of the bonds (see diagram in figure 2(b)).

Sodium silicate solution was used to produce the strongest bonds so this was then used to create a further 9 bonds. These were also cured at room temperature in Glasgow for four weeks before being broken using the same four-point bending test, but this time whilst submerged in a liquid nitrogen bath. The graph in figure 5 shows the results of these strength tests.

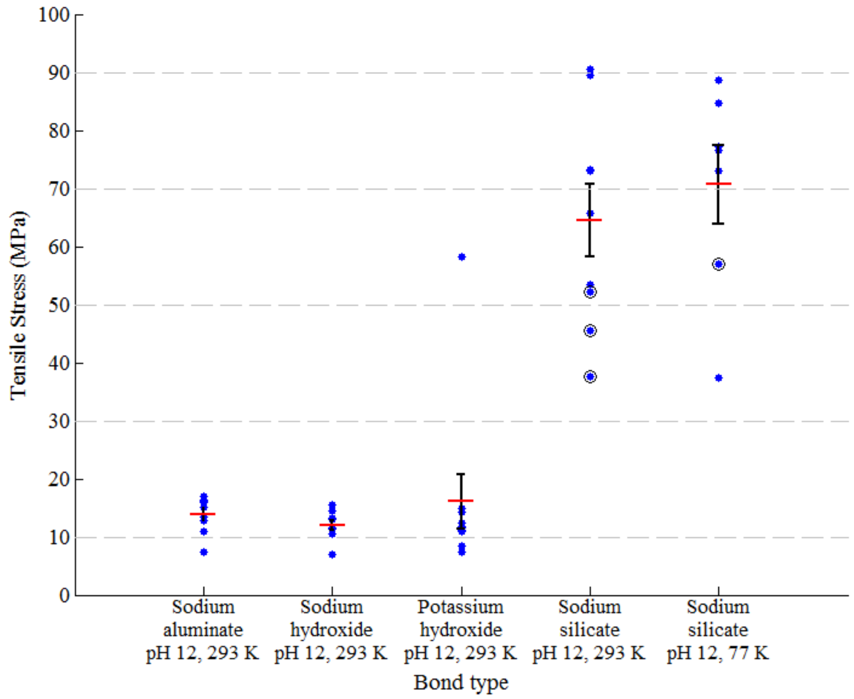

Figure 5: Graph showing the strengths of bonds formed with various bonding solutions. Blue points are the strengths of individual bonds, the red bars give the mean strength and the error bars are the standard error on the mean. Where a point is circled this indicates an incomplete bond with less than 90% of the intended bonding surface in covered with bonding material.

It is clear from these results that sodium silicate solution produces the strongest bonds and that the strength of those bonds are not diminished at cryogenic temperatures[1].

Comparison with silicon bonds:

The average strength of a hydroxide catalysis bond between sapphire substrates was found to be ~60MPa at room temperature [1]. This is stronger than the average strength found for a bond between silicon substrates, ~36MPa, which is already extremely robust [2]. For bonds between both materials, sapphire and silicon, the strength was not diminished by cryogenic temperatures showing that these bonds would have more than sufficient strength to join components of cryogenically run gravitational wave detectors [1,2].

Orientation of the crystal axes with respect to the bonding surface was investigated for both materials and was shown to have an effect. It was seen that bonding the C-axis to the A-axis or the C-axis to the M-axis is more reliably strong than bonding the C-axis to the C-axis. However, bonding the C-axis to the C-axis could produce stronger bonds but further investigations would have to be carried out in order to fully understand what is causing the extreme spread in results. When bonding silicon, it was found there was no significant change in strength when bonding the same axis to itself ((100) to (100) and (111) to (111)). However, the bonds created between different axes ((100) to (111)) tended to be slightly stronger [3].

The hydroxide catalysis bonds created between silicon or sapphire substrates have proven to be strong enough and durable enough to support the mirror suspensions in cryogenic gravitational wave detectors.

Other bonding methods:

In addition to the ongoing experiments into the hydroxide catalysis bonding of sapphire, investigations there have also been investigations into indium bonding. Following on from initial studies carried out in Glasgow by Twyford et al [4] on the suitability of indium bonding for use in jointing quasi-monolithic suspensions for gravitational wave detectors, investigations have been resumed. Promising initial measurements of the mechanical loss associated with thin indium layers on silicon have been carried out and samples for strength testing of indium bonds on sapphire and silicon are in the early stages of production.

A literature review on this technique was carried out by 2 ELiTES PhD students, Gerald Bergmann and Manuela Hanke, from the AEI Hannover.

References:

[1] *Cryogenic and room temperature strength of hydroxide catalysis bonded sapphire for various bonding solutions,* R. Douglas et.al, Class. Quantum. Grav. 31 (2014)

[2] *Dependence of cryogenic strength of hydroxide catalysis bonded silicon on type of surface oxide*, Class. Quantum. Grav. 30 (2013)

[3] *Low temperature strength tests and SEM imaging of hydroxide catalysis bonds in silicon,* N. Beveridge et al, Class. Quantum Grav. 28, 2011

[4] *Developments Towards Low Loss Suspensions for Laser Interferometric Gravitational Wave Detectors*, S. Twyford, PhD thesis, University of Glasgow, 1998