Properties of hydroxy-catalysis bonds for use in advanced gravitational wave detectors

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Introduction

In order for advanced gravitational wave detectors to reach their design sensitivity, a variety of noise sources must be reduced to suitable levels. Thermal noise associated with the mirror substrates and suspension techniques employed, sets an important limit, particularly in the most sensitive frequency band (a few tens to a few hundred Hz) of planned second generation detectors, such as Advanced LIGO. Quasi-monolithic last-stage suspension systems are a key technical approach taken to reduce the levels of thermal noise in interferometric gravitational wave detectors to appropriate levels.

A method of interest for jointing the components in this type of suspension is hydroxy-catalysis (or "silicate") bonding. The technique was originally developed for the Gravity Probe B project [1], has been implemented for the construction of ultra-low loss suspensions in the GEO600 gravitational wave detector and is now being developed further for advanced detectors, see figure 1.

For Advanced LIGO, fused silica has been chosen as the substrate material for the mirrors and their suspension elements in part due to its low mechanical loss [2]. Silicon is being considered as the substrate and suspension material for third generation detectors, which may run at cryogenic temperatures. Understanding the properties of bonds between these materials, in particular bond strength, thickness and thermal conductivity, is crucial in optimizing suspension designs.

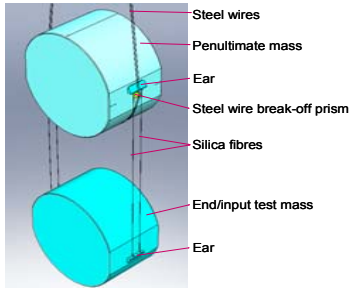

Figure 1. *Advanced LIGO suspension*

Hydroxide Catalysis Bonding

The method, placing sodium silicate solution between two clean substrates can be used to form strong, stable and precisely aligned bonds between any combination of materials that allow for silicate-like networks to be formed between their surfaces, and that have suitable flatness (typically 60nm peak-to-valley flatness or better).

The steps required to form silicate bonds are as follows, where the final dehydration step allows a rigid network of molecules to form between the two adjoining surfaces, as shown in Fig 2 [3]:

- Hydration and Etching
- Polymerization
- Dehydration

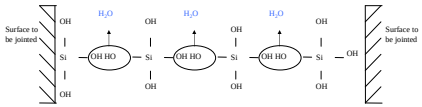

Figure 2. *Final stages of bonding (polymerization and dehydration)*

Bond Strength

(a) Silica-silica bonds

The strengths of bonds were tested using a 4 point bend testing machine. The set up used is shown in figures 3 and 4. The first set of samples were cured for the standard 4 weeks, then the bonds were heat treated differently. It was found that heating at 150°C for 48 hours increased the strength by a factor of ~1.6 and putting in a preheated oven for 3 minutes at 400°C increased the strength by a factor of ~1.1. 3 minutes in a pre-heated oven of higher temperatures 800°C and 1000°C decreased the strength by a factor of ~0.3 and ~0.7 respectively. There was evidence to suggest the sample geometry limited the experiment as the samples were breaking at the contact points. The samples were made longer (from 10 x 10 x 5mm to 20 x 10 x 5mm) and a higher strength was found. Longer samples were then strength tested for varying amounts of bonding solution. It was found that the strength appears to increase slightly as the amount of bonding solution is increased. This will be the subject of further investigations.

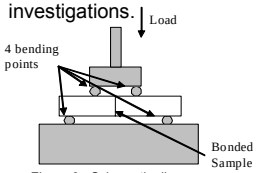

Figure 3. Schematic diagram of strength tester

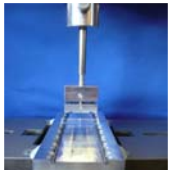

Figure 4. Strength tester

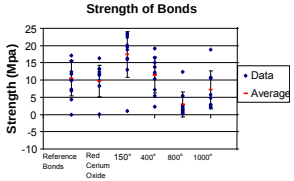

Figure 5. Strength of different heat treated bonds

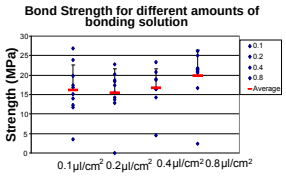

Figure 6. Strength of bonds with different amounts of bonding solution

(b) Silicon-silicon bonds

Silicon samples must be oxidised before bonding; oxidisation regimes using wet nitrogen and dry oxygen respectively were used. Investigations of the shear strength of silicon-silicon hydroxide catalysis bonds at room temperature were then carried out using these samples.

Results here show that silicon-silicon bonds are at least as strong as their silica-silica counterparts (Figure 7). No correlation was found between oxide layer thickness and strength but evidence suggests there is a minimal oxide thickness required for bonding [4]. As silicon is being investigated for use in third generation gravitational wave detectors the bond strength was compared at room temperature and on cooling to 77K. It was found that the decrease in temperature did not affect the average strength of the bonds but did appear to result in an increased variation in individual bond strengths.

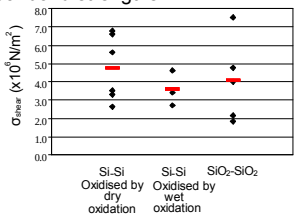

Figure 7. Measured individual (•) and mean (-) shear strength test results

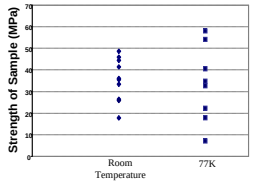

Figure 8. Strength at room and cryogenic temperature

Bond Thickness

Silicon-silicon

As silicon samples have to be oxidised before bonding investigations were made of any effect this might have on the bond thickness. Oxide layers between 50 and 200nm were produced and tested. The bonds were imaged using a scanning electron microscope. No correlation was found between the oxidising time, which is directly related to the oxidisation thickness level and the bond thickness.

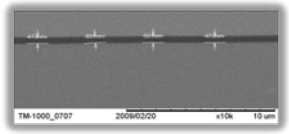

Figure 9. SEM image of a bond

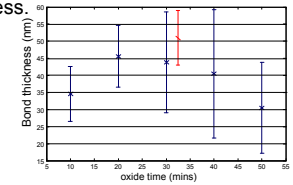

Figure 10. Bond thickness for different oxide times

Bond Thermal Conductivity

Pairs of silicon disks were bonded and extensive preliminary studies are underway using a custom made facility for investigating the thermal conductivity (Figure 11). Results shown in Figure 12 show the thermal conductivity of a bonded sample was observed to be similar to pure silicon at room temperature. When the temperature was decreased, the observed conductivity of the bonded sample increased but at a lower rate as compared to the pure silicon measurements. Further studies to investigate the thermal conductance of these bonds are underway and are detailed in the poster by M. Lorenzini.

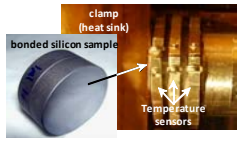

Figure 11. Experimental setup for measuring thermal conductivity of 1" silicon samples

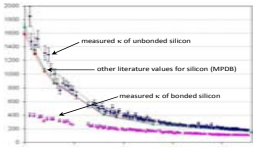

Figure 12. Measured and literature values for the thermal conductivity of silicon compared to the measured values of bonded silicon

Conclusions and Future Research

For the cryogenic strength testing, more bonded samples will be tested to improve the statistics we have at each temperature. Similar samples will be prepared to test the dependence of the thermal conductivity of silicon-silicon bonds on the *e.g.* volume and concentration of the bonding solution used.

Our results, in combination with the observed robustness during repeated temperature cycles, suggest silicate bonding is a reliable technique for jointing parts of the silica suspensions and may be suitable for use in the quasi-monolithic silicon suspensions central to the construction of third generation gravitational wave observatories from both a reliability and thermal loading point-of-view.

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References

[1] E. J. Elliffe, J. Bogenstahl, A. Deshpande, J. Hough, et al., Hydroxide catalysis bonding for stable optical systems for space, Class. Quantum Grav. 22 S257–S267
[2] Test Mass Material Downselect Plan, 2002, LIGO-T020103
[3] R. Iler. The Chemistry of Silica, ch.2&6, London press, (1979)
[4] A. A. van Veggel, J. Scott, D. A. Skinner, B. Bezensek, et al., Strength testing and SEM imaging of silicate bonds of silicon (In preparation)