Current status of bonding research for sapphire and silicon suspensions

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1. Introduction

Interferometric gravitational wave detectors have used fused silica test masses suspended on fused silica fibres with hydroxide catalysis bonds (HCBs) at interfaces to minimise thermal noise[1]. Reducing the loss will reduce the thermal noise - some planned detectors aim to do this through cryogenic cooling. At ~40 K, silica has a well-defined loss-peak[2], hence crystalline materials such as silicon and sapphire are of interest for future aLIGO upgrades and for detectors such as ET and KAGRA. This poster reports progress with on-going experiments involving hydroxide catalysis bonding sapphire and silicon to investigate the suitability of HCBs for use in crystalline quasi-monolithic suspensions.

2. Recent results

Recent results are shown below for four experiments. This work follows from previous work on strengths for bonded silicon (van Veggel, CQG, 2009; Dari, CQG, 2010; Beveridge, CQG, 2011 and 2013) and sapphire (Dari, CQG, 2010; Douglas, CQG, 2014; Haughian, CQG, 2015) and the thermal conductivity of silicon-silicon bonds (Lorenzini, J. Phys. Conf. Ser., 2010; Lorenzini, ET meeting, Jena , 2010).

All samples studied below were bonded using aqueous sodium silicate solution. The bonding surfaces of all strength samples and thermal conductivity samples were polished flat to 60 nm peak-to-valley. All samples were cleaned using cerium oxide paste, bicarbonate of soda and wiped with methanol. The silicon samples were thermally oxidised with a layer thickness of 150 ± 20 nm.

3. Thermal cycling of silicon-silicon bonds

Silicon and sapphire suspensions are likely to go through multiple thermal cycles during their lifetime. To assess the effect of thermal cycling on the strength of HCBs, the tensile strength of silicon-silicon bonds after 0, 3, 10 and 20 thermal cycles between ~4 K and ~293 K (2 hours/cycle) was measured at room temperature. The thermal cycling was carried out in Jena.

Figure 3.1 Oxidised silicon sample 10x5x20 mm (material properties in Figure 5.1) ~150 nm dry thermal oxide Bonded sample 10x5x40 mm

The experiment was carried out twice (40 bonds in batch 1 and 28 bonds in batch 2). A visual fracture analysis was carried out. The average strength remains constant for increasing numbers of thermal cycles (Figure 3.2). Bonds showing diagonal breaks show the highest strengths (circled data points).

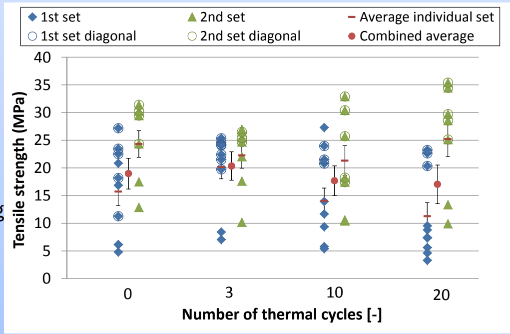

Figure 3.2 Tensile strength of silicon-silicon HCBs for different numbers of thermal cycles

4. Comparing bonds between C-C, A-A and M-M sapphire

Strength tests (Figure 4.1) have been carried out on sapphire samples of dimensions 5x5x40 mm where the C-planes of the samples (C-C bond) were jointed to investigate the curing time (blue diamonds in Figure 4.2) . Indeed at 8 and 12 weeks the strength appears to show a small drop. More tests are needed to confirm the statistical significance of these results. At 4 weeks curing also a direct comparison has been made between C-C, A-A (red squares in Figure 4.2) and M-M (green triangles) samples. The average strengths of all are comparable.

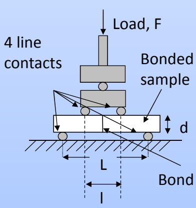

Figure 4.1 Schematic 4-point bend set-up to measure the tensile strength of the bond

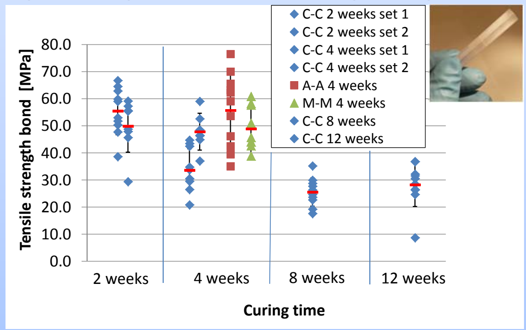

Figure 4.2 Tensile strength of sapphire-sapphire HCBs at room temperature for different curing times and C-C, A-A and M-M compared directly for 30 days curing.

5. Thermal conductivity of silicon-silicon bonds

The thermal conductivity of an HCBed and reference silicon sample (Figure 5.1) has been measured down to 8 K. The set-up is shown in Figure 5.2. The method used is a steady-state heat flow technique and multiple levels of heat flow have been tested.

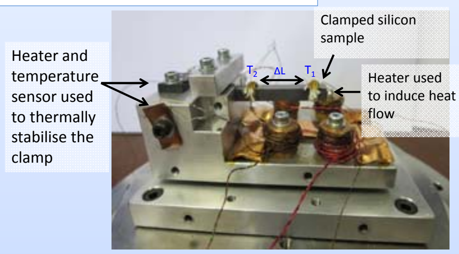

Figure 5.2 Silicon sample mounted in thermal conductivity set-up

The drop in thermal conductivity due to the bond is confirmed to be minimal (Figure 5.3) (Lorenzini, J. Phys. Conf. Ser., 2010; Lorenzini, ET meeting, Jena , 2010). This has also been seen in sapphire bonds (C. Schwarz, D. Chen, ET meeting 2014, Lyon).

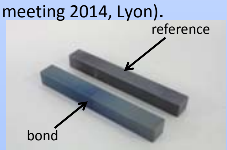

Figure 5.1 Silicon samples (5x5x40 mm Prolog Boron doped P-type <100>, O-concentration $5.1 \cdot 10^{17}$ /cm³, resistivity 2.2 Ω·cm)

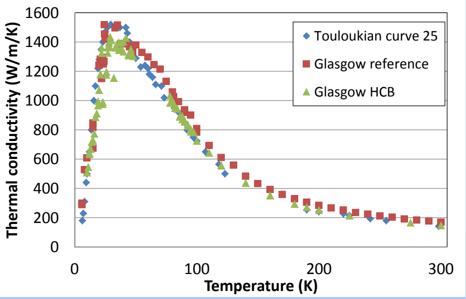

Figure 5.3 Thermal conductivity of both the reference sample and the bonded sample as a function of temperature compared to Curve 25 (Touloukian, 1977, Boron doped P-type silicon 6.2x 6.2mm silicon O-concentration $7 \cdot 10^{17}$ /cm³, resistivity 3 Ω·cm),

6. Mechanical loss of a C-C sapphire bond

The mechanical loss of an HCB formed between the C-planes of sapphire samples (∅ 30 mm x 120 mm) has been measured down to ~7K (Figure 6.1). Finite element analysis (FEA) was carried out using ANSYS, to find the energy distribution within the sample. The loss of the bond material was then extracted (Figure 6.2) from the measured data and FEA.

The possible bond thickness is limited by the flatness of the bonded surfaces. The bond thickness is assumed to be 74.5 to 149 nm.

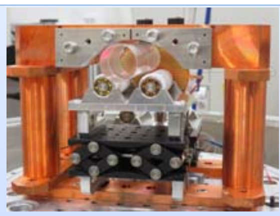

Figure 6.1 C-C sapphire sample (∅30 mm x 120 mm) suspended in wire loop

The substrates were provided by ICRR, bonded in Glasgow, and measured and analysed in Glasgow and Jena.

The sapphire bond loss is between 0.03 ± 0.01 at room temperature and $(3 - 7) \cdot 10^{-4} \pm 1 \cdot 10^{-4}$ at 20 K.

This is comparable to upper limits found for the mechanical loss at room temperature of a bond between fused silica, 0.06 (Haughian, thesis, 2012).

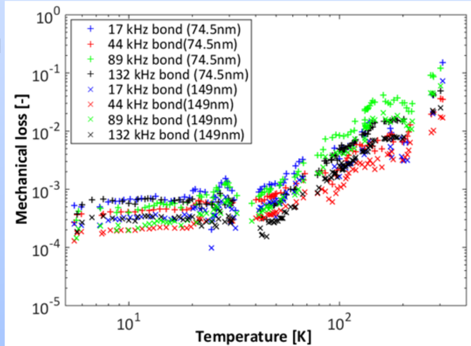

Figure 6.2 The range of bond loss extracted from measurements and FEA analysis

7. Conclusions

Thermal cycling of hydroxide catalysis bonds between silicon samples does not appear to have a negative impact on bond strength. Methods are being investigated to assess bond quality of silicon-silicon bonds (as silicon is opaque) during the bonding procedure so inferior quality bonds can be rejected.

Silicon-silicon HCBs have a high thermal conductivity down to 8 K. Thermal conductivity measurements of an silicon-silicon indium bond will be carried out.

The strength of hydroxide catalysis bonds with C-C sapphire does not increase after 2 weeks of curing. However, it may be the case that the chemical process forming the bonds is not complete at that point. This needs further investigation.

Direct comparison of strengths between C-C, A-A and M-M sapphire doesn't show a significant difference.

The mechanical loss of a sapphire-sapphire HCB is $(3 - 7) \cdot 10^{-4} \pm 1 \cdot 10^{-4}$ at 20K.

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