G0900639-v1

Silicate Bonding

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Summary

- **Hydroxide-Catalysis Bonds for use in Advanced and future Gravitational Wave Detectors**
- **Silica-Silica Bond Loss**
- Thermal Noise Contribution for Advanced detectors
- **Silicon-Silicon Bonds**
- Room Temperature Strength
- Low Temperature Strength
- Thermal Conductivity
- **Conclusions**

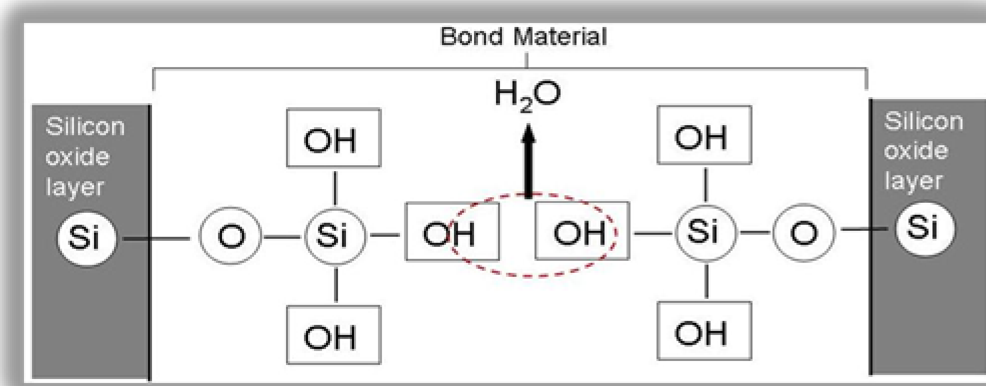
- Interferometric gravitational wave detectors operate by the **sensing of very small relative displacements** of their suspended mirrors
- **Friction** between the mirror substrates and their suspension fibres **can lead to increased levels of thermal noise**
- Therefore, **low mechanical loss jointing techniques are required** to attach the suspension fibres to the test masses
- **Hydroxide-catalysis bonding** has been studied for this use

Hydroxide-catalysis bonding

- Hydroxide-Catalysis bonding implemented in **GEO600**
- Was **developed at Stanford University** by D. H. Gwo for NASA's **Gravity Probe B** mission, launched April 2004, to meet the stringent strength requirements
- **Enhancements** to bonding technique **made at University of Glasgow**
- **Advanced LIGO** will incorporate this technique
- Used in construction of ultra-rigid, ultra-stable optical benches for **LISA Pathfinder mission**
- Hydroxide-Catalysis bonds **introduce** some **mechanical loss**
- Consequently, important to characterise the **mechanical loss** of the bonds and **its significance for thermal noise**

Hydroxide-catalysis bonding

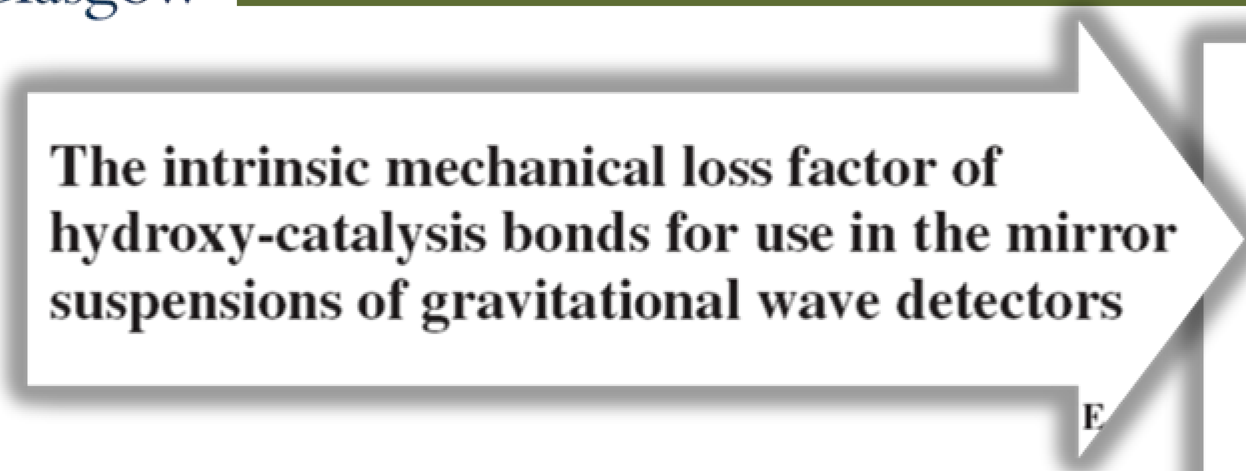
- **Hydration and etching**
  - OH^- ions in bonding solution etch the silica surfaces
- **Polymerization**
  - Less OH^- ions so pH decreases
  - When pH < 11, silicate ions dissociate
  - Siloxane chains and water are formed creating **rigid bond**



- **Dehydration**
  - Water migrates and evaporates
  - **After 4 weeks full strength** is achieved

HYDROXIDE-CATALYSIS BOND LOSS

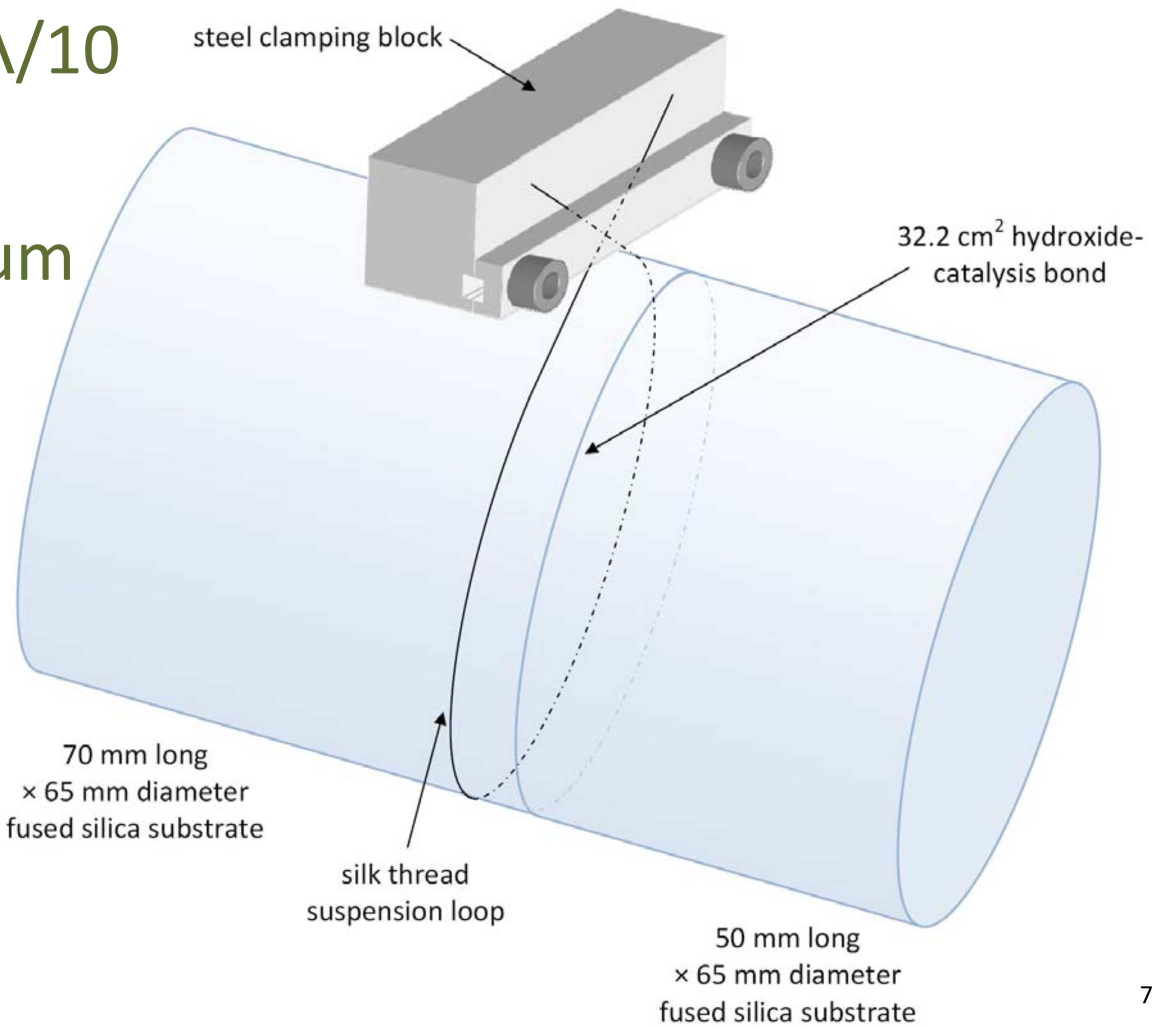
Hydroxide-Catalysis Bond Loss



- Investigations were made to **quantify** the **bond loss** of a hydroxide catalysis bond produced over a ~32 cm^2 bond
- **Two identical** 65 mm diameter by 70 mm long fused silica **cylinders were bonded**
- A **bond loss of 0.28 ± 0.04 was found** for the fundamental longitudinal mode
- However, the experiment had it's **limitations**

Hydroxide-Catalysis Bond Loss

- This experiment was **repeated** at the University of Glasgow **using** fused silica cylinders of **differing lengths**
- Flatness specified to be λ/10 (where λ = 633nm)
- Bonded using 12 µl sodium silicate solution (14% NaOH, 27% SiO_2 diluted 1:6 in de-ionised water)
- Measurements made five months after bonding



Hydroxide-Catalysis Bond Loss

- **Loss measurements** made by suspending mass under vacuum
- **Resonant modes excited** using an electrostatic actuator
- Michelson interferometer used to sense excited motion of the front face
- Mechanical loss of resonant mode **calculated from time** taken for the motion **to decay**

$$A = A_0 \exp\left(-\frac{1}{2}\phi(\omega_0)\omega_0 t\right)$$

Hydroxide-Catalysis Bond Loss

- Assuming all other losses in the system are negligible, the mechanical loss of the bonded sample may be expressed as

$$\phi_{\text{bonded}} \simeq \phi_{\text{substrate}} + \left(\frac{E_{\text{bond}}}{E_{\text{total}}}\right)\phi_{\text{bond}}.$$

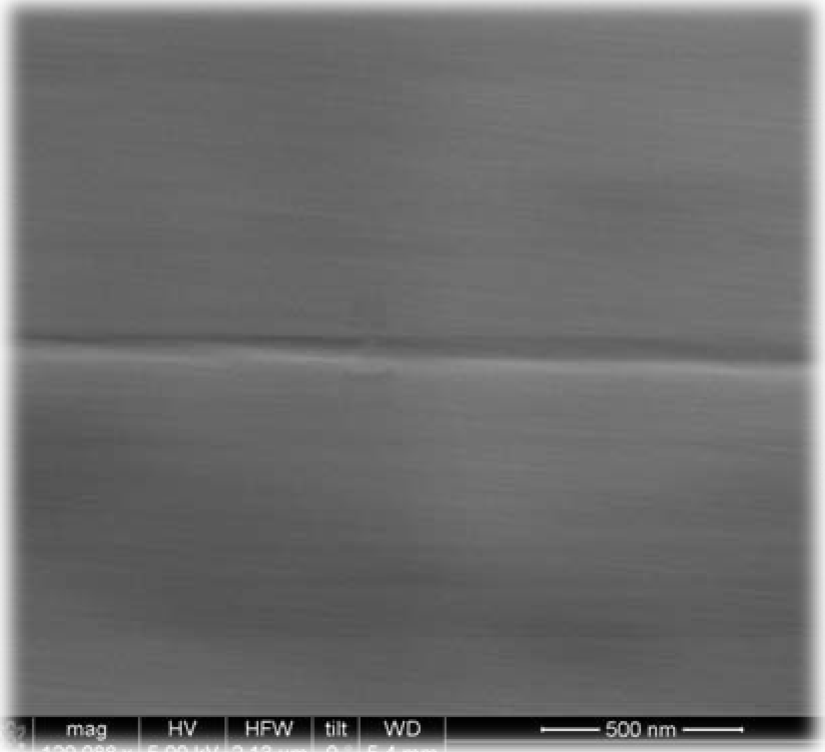
Measured

Wanted

- Therefore, **need to determine**
- **Substrate Loss**
- **Fraction of Strain Energy Stored in Bond**
- Thickness of bond

Hydroxide-Catalysis Bond Loss

- Measurement of **thickness** of hydroxide-catalysis bonds
- Two one inch samples bonded to create **witness sample**
- Thickness measurements made using
- Scanning Electron Microscope
  - 60 ± 2 nm
- Transmission Electron Microscope
  - 58 ± 4 nm
- Atomic Force Microscope
  - 65 ± 5 nm
- Average of bond thickness measurements is **61 ± 4 nm**



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Hydroxide-Catalysis Bond Loss

- The **mechanical loss** of several resonant modes of the cylinders were **measured before bonding**
- The **lowest losses** were then **fitted to** the **Semi-empirical model** of Penn for loss in silica

Surface loss term

Bulk loss term

Thermoelastic loss term

$$\phi\left(f, \frac{V}{S}\right) = C_1 \left(\frac{V}{S}\right)^{-1} + C_2 f^{C^3} + C_4 \phi_{\mathrm{th}}$$

- The mechanical losses here are measured at frequencies which lie well away from the peak of thermoelastic loss
- **Suprasil 312**

$$C_1 = (6.5 \pm 0.2) \times 10^{-9}$$

$C_2 =$ Reduction of 17%

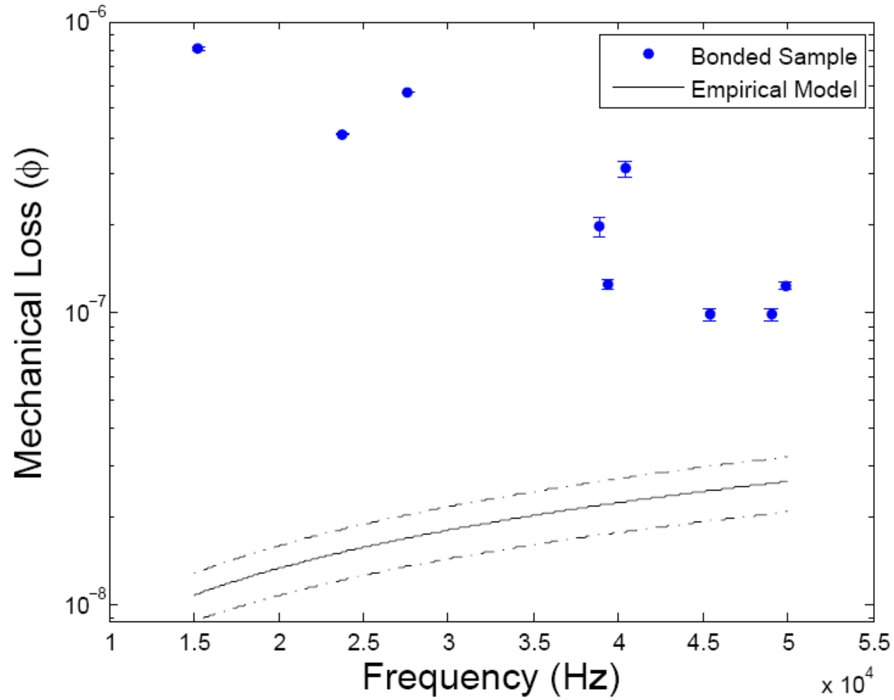
$$C_3 = (0.77 \pm 0.02).$$

- **Suprasil 311**

$$C_2 = (0.63 \pm 0.02) \times 10^{-11}$$

Hydroxide-Catalysis Bond Loss

- These numbers were then used to evaluate the substrate loss of a 120 mm long fused silica cylinder



$$\phi_{\text{bonded}} \simeq \phi_{\text{substrate}} + \left(\frac{E_{\text{bond}}}{E_{\text{total}}}\right) \phi_{\text{bond}}.$$

Calculate

Measured

Modelled

Modelled

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Hydroxide-Catalysis Bond Loss

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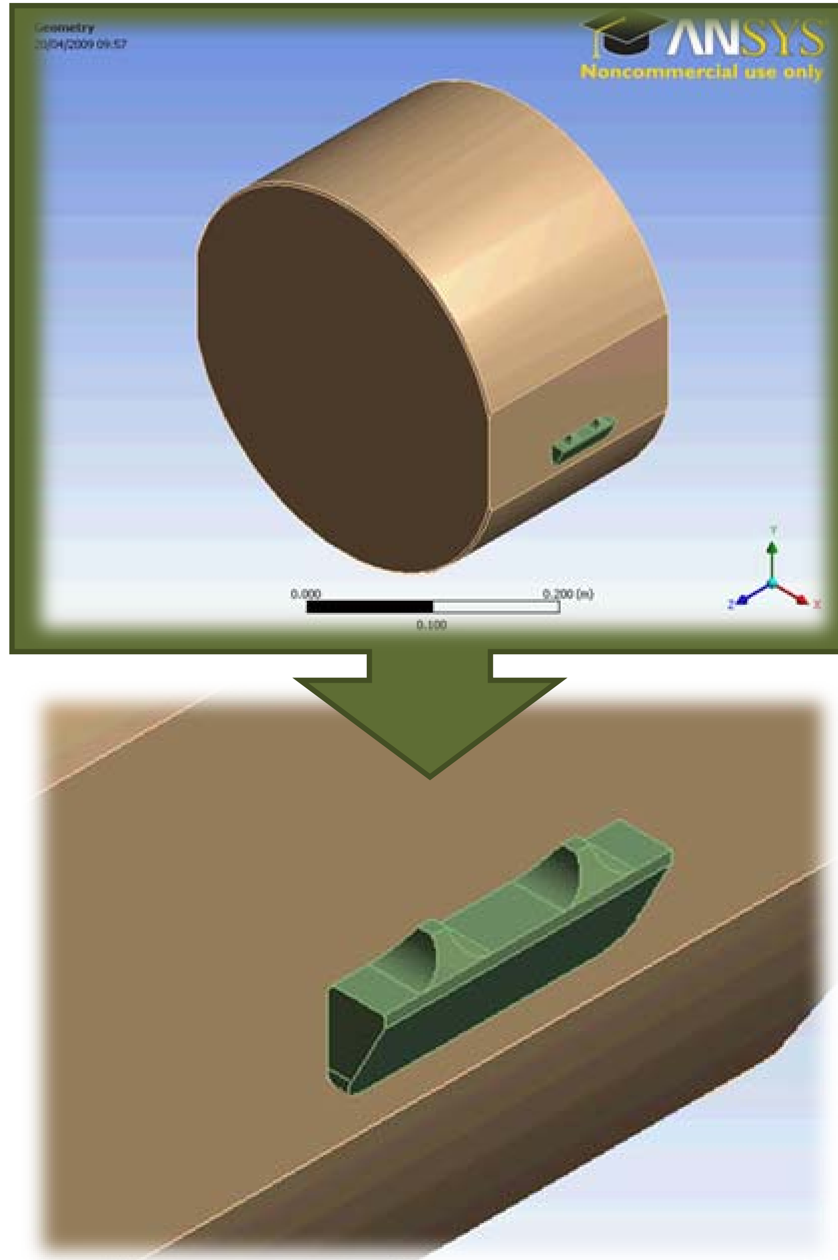
- It is now possible to calculate the bond loss for all nine resonant modes

Frequency (Hz)	$\phi_{\mathbf{bond}}$ $(\times 10^{-1})$
15351	1.05 ± 0.07
23822	0.51 ± 0.03
27674	0.63 ± 0.04
38889	1.44 ± 0.17
39412	1.16 ± 0.11
40464	0.78 ± 0.07
45463	0.48 ± 0.06
49043	1.28 ± 0.18
49896	2.03 ± 0.22

- **Average bond loss** for the 61 nm thick bond of **0.10 ± 0.01**
- Close to **three times lower than previous experiment suggests**

Hydroxide-Catalysis Bond Loss

- Main optical substrates for **Advanced LIGO**
- 40 kg fused silica cylinders
- 340 mm diameter by 200 mm thick
- 95 mm diametrically opposite flats
- **Ears** for attaching the silica suspension elements **are bonded on each of the flats**
- Total bond area 23.7 cm^2



Hydroxide-Catalysis Bond Loss

- **Using Levin's approach** and new **bond loss** value the overall **thermal noise contribution** of the bond layer on silica test masses was **calculated**

$$S_x(f) = \frac{2k_B T}{\pi^2 f^2} \frac{W_{diss}}{F_o^2}$$

- Using ANSYS® to calculate the energy density ε(x,y,z) of elastic deformation in a test mass to obtain W_{diss}
- This yielded a value of **5.2 × 10^{-22} m/√Hz**
- In comparison the allowed upper limit for Advanced LIGO is **7 × 10^{-22} m/√Hz (LIGO-T010075-01-I)**
- *"Level of thermal noise contributed to the test masses of advanced LIGO by the bonds used to attach suspension elements" – Liam Cunningham et al,* **In Preparation**

SILICON-SILICON BOND STRENGTH ROOM TEMPERATURE

Silicon-Silicon Hydroxide-Catalysis Bonds

- **Silicon under consideration** as a substrate material **for mirror masses and quasi-monolithic stages of possible future GW detectors eg E.T.**
- **Essential to joint** silicon suspension to optics **with high strength and low loss**
- Hydroxide-catalysis bonding good candidate for this
- However **oxide layer essential** for bond formation

Silicon-Silicon Hydroxide-Catalysis Bonds

- To make bonding of silicon possible, bonding **surfaces must** have a thin **coating of SiO_2**
- 50 – 200 nm
- This **layer formed through thermal oxidation** of pieces in a quartz tube furnace
- Temperature around 1000 °C
- Run with either a dry air or oxygen or a wet environment using air, nitrogen or oxygen as a carrier gas
- **Set of samples** with oxide layers of 50 – 200 nm **produced**
- Pairs were then taken and bonded together
- Shear strengths measured

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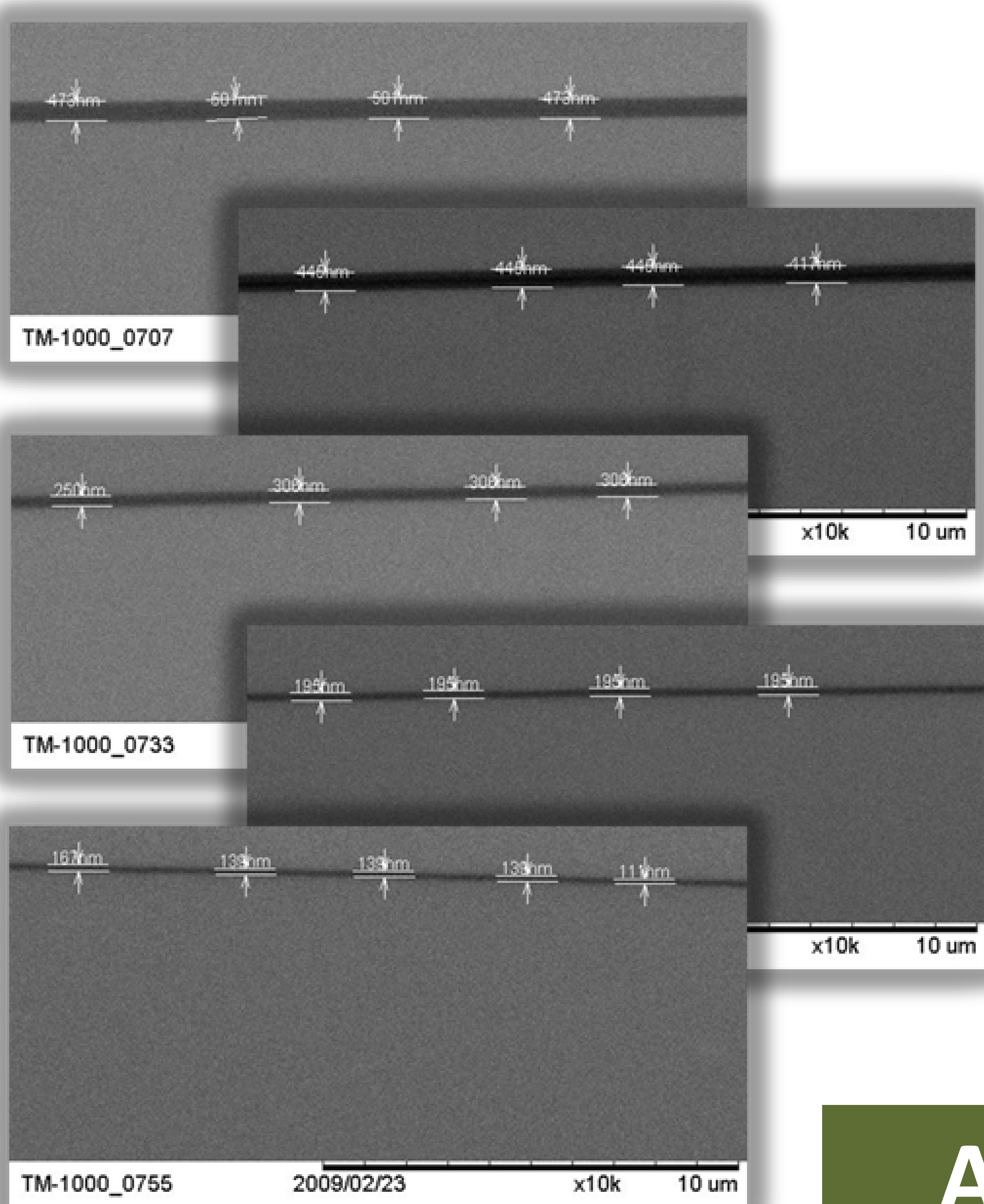
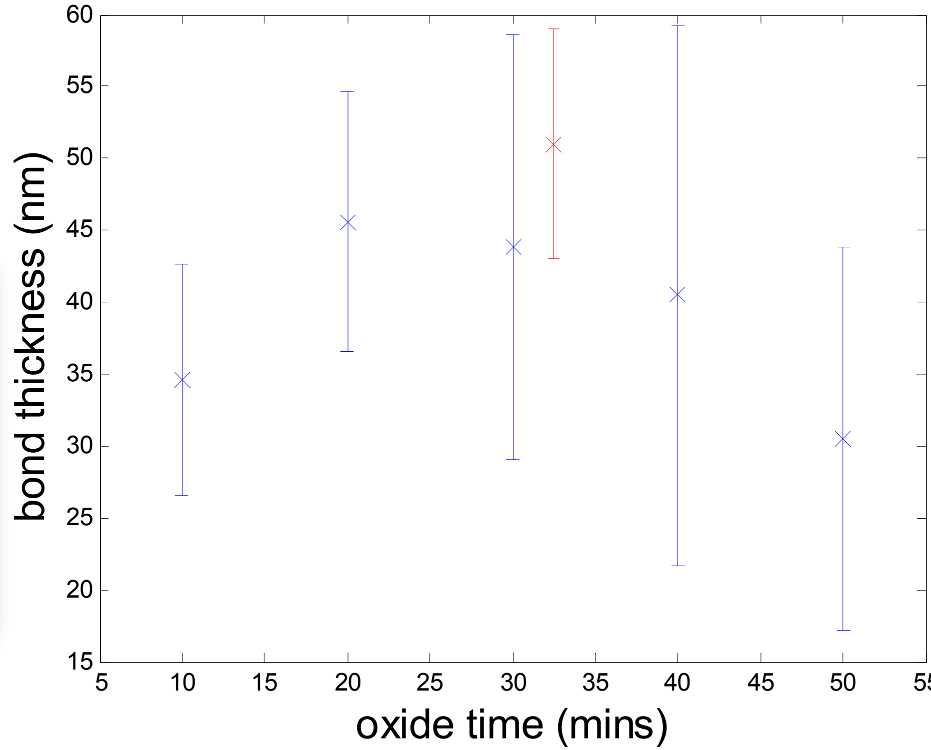
Silicon-Silicon Hydroxide-Catalysis Bonds



- **Thicknesses of wet N_2 bonds measured** with *table-top* SEM
- Subtract **oxide thicknesses** measured using **ellipsometry**



Average: 41 ± 13 nm

- Shear strength testing
- Roel-Zwick 250 kN tensile testing machine
- Maximum force applied 3500 N (equivalent to 6.8×10^6 Nm^{-2})

Silicon-Silicon Hydroxide-Catalysis Bonds

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- **Average Strengths:**
- **Silicon bonds, oxidised via Dry O_2 :**

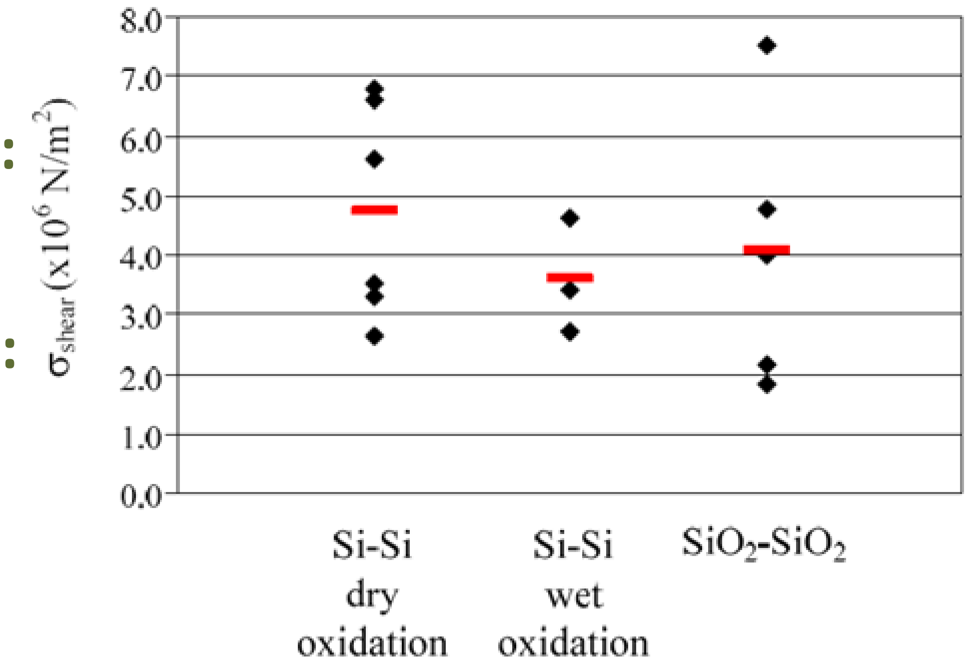
  4.8×10^6 Nm^{-2}

- **Silicon bonds oxidised via Wet N_2 :**

  3.6×10^6 Nm^{-2}

- **Silica bonds**:

  4.1×10^6 Nm^{-2}

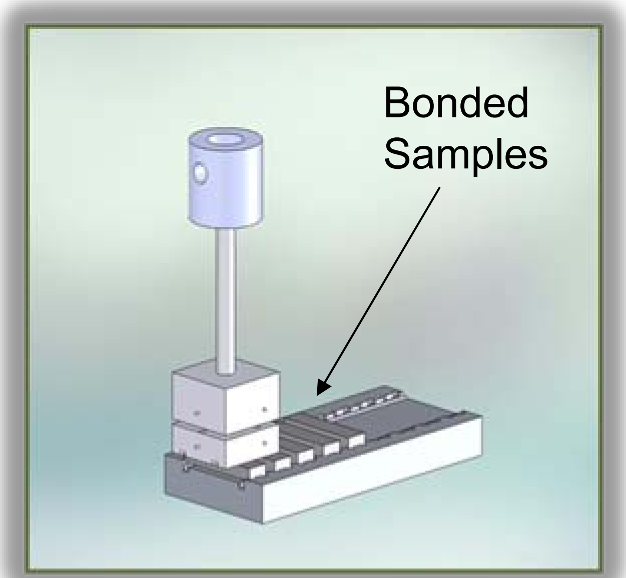


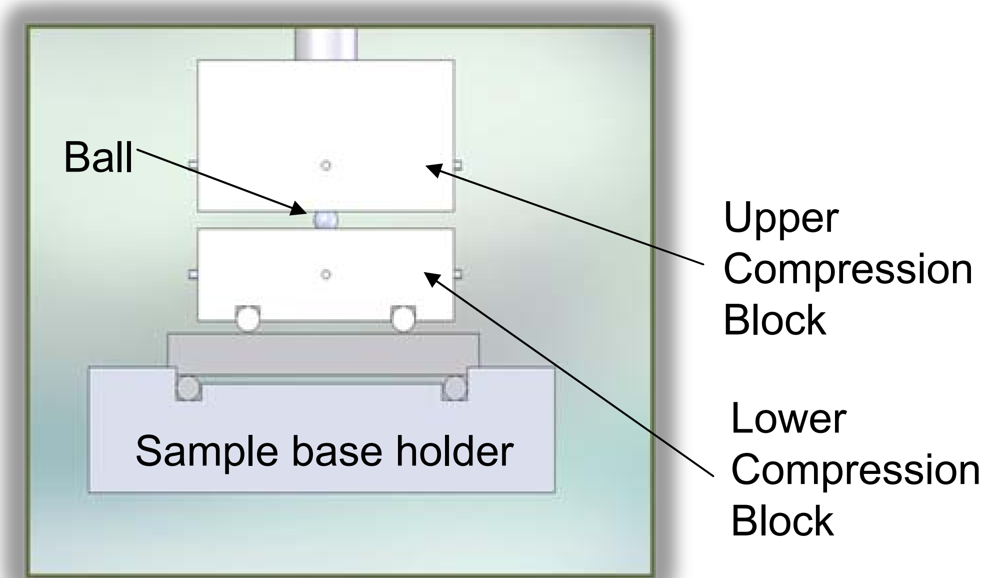
- However, variations at level of approx ± 3.0×10^6 Nm^{-2}
- No statistically significant difference in strength
- No obvious correlation between **flatness and strength**
- No obvious correlation between **bond thickness and strength**
- *"Strength testing and SEM imaging of hydroxide-catalysis bonds between silicon" – Mariëlle van Veggel et al,* **Submitted to CQG**

SILICON-SILICON BOND STRENGTH LOW TEMPERATURE

Low Temperature Bond Strength

- Strength at low temperature measured again using a **4-point bend testing setup**
- The sample **base holder is able** to house **nine samples**

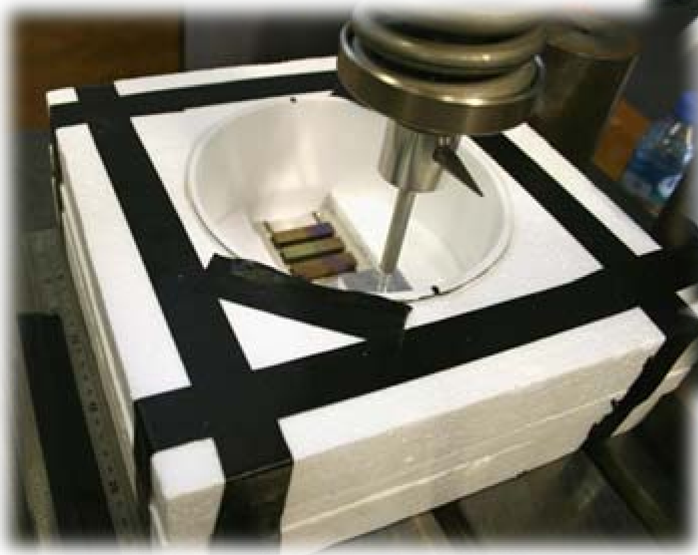




- The sample base holder is s**ubmerged in** a **liquid nitrogen** bath to maintain samples at temperature of ~77 K
- Twenty bonded samples have been oxidised, bonded and cured

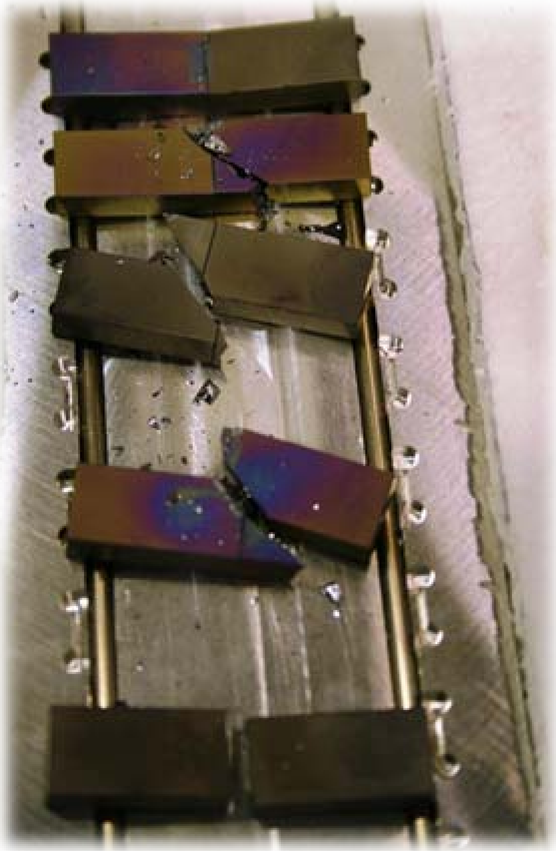
Low Temperature Bond Strength

- **Preliminary results suggest** that **bond strengths** measured for samples at Nitrogen temperature are **approximately the same** as those found at room temperature
- Nicola Beveridge and Mariëlle Van Veggel
- A slightly larger spread in strengths is found
- Work ongoing



Room Temperature Setup

Cryogenic Temperature Setup



Samples After Testing

SILICON-SILICON BOND THERMAL CONDUCTIVITY

Thermal Conductivity

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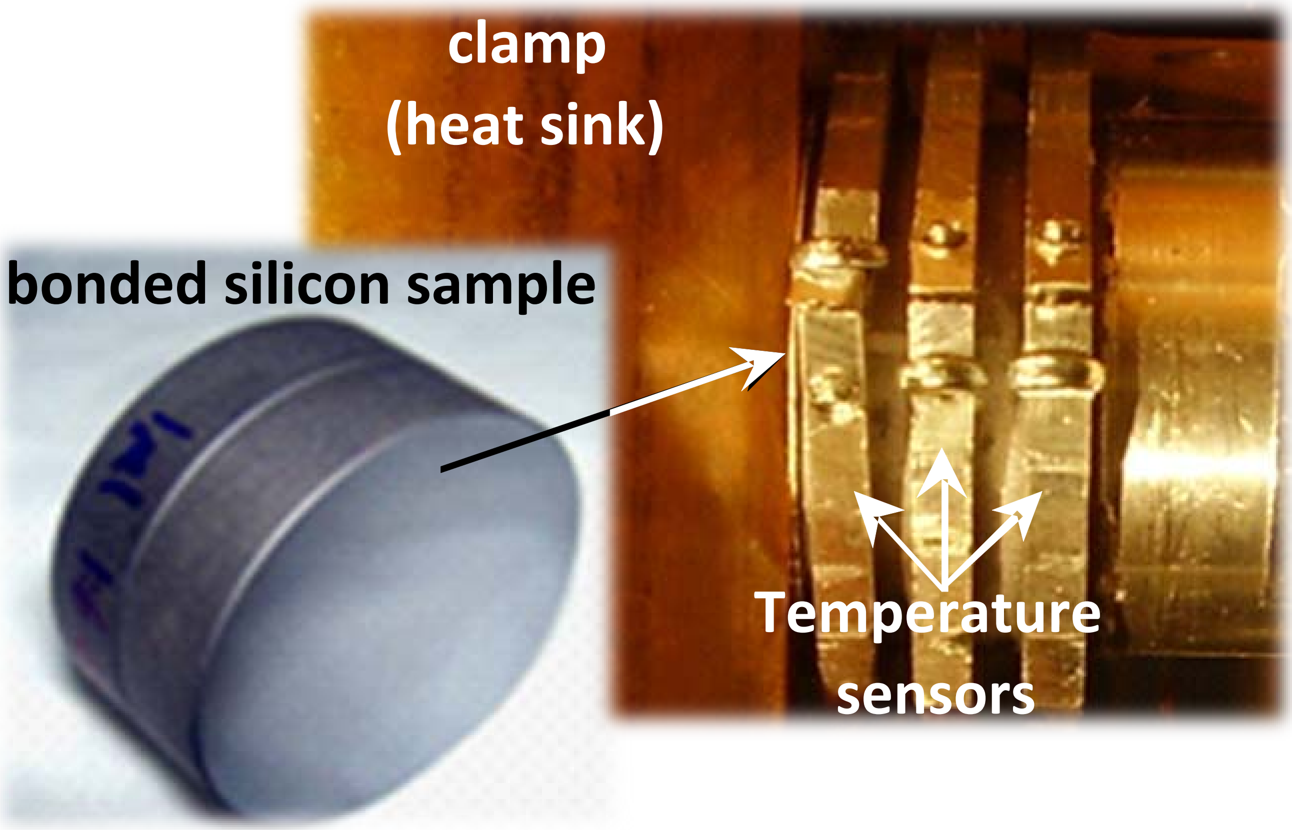
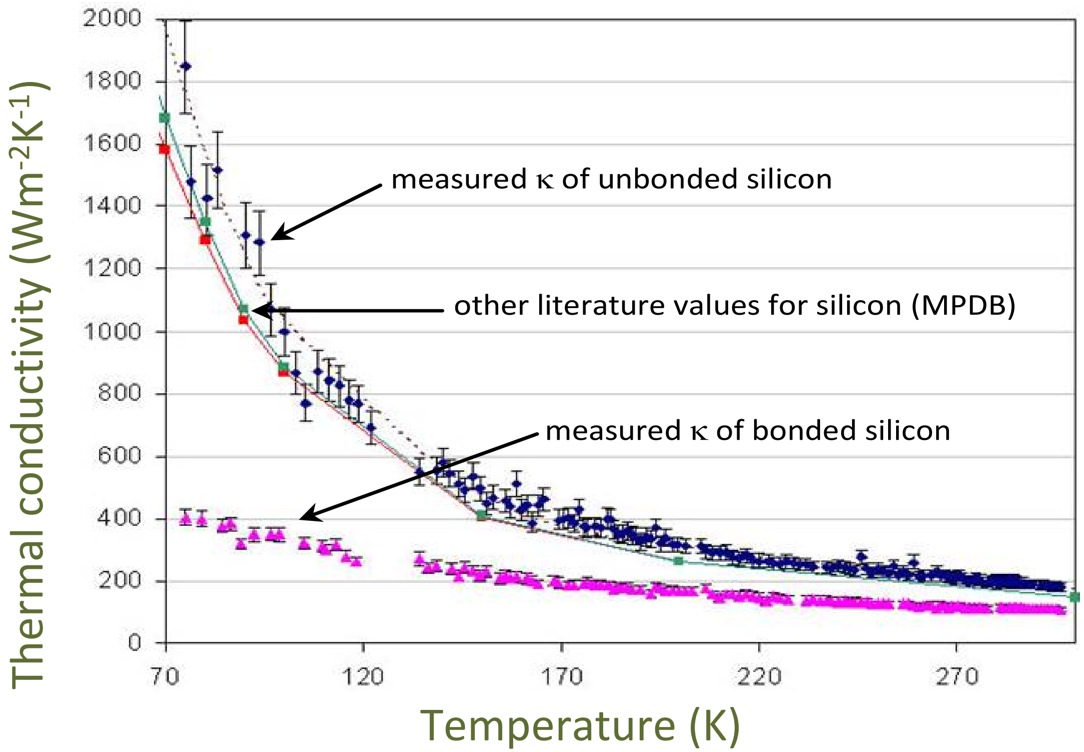
- Pairs of silicon discs of 1” diameter, 0.25” thick were bonded
- Studies of thermal conductivity of the samples are underway using a custom made facility at the University of Florence



Thermal Conductivity



Thermal conductivity of the bonded sample **similar to pure silicon at room temperature**

When the temperature was decreased, the **conductivity** of the **bonded sample increased at a lower** rate

Further studies (e.g. volume and concentration) of these bonds are underway

Conductivity levels make this a plausible technique for use in future low T detectors

Further studies on effect of thermal conductivity on heat extraction needed

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Conclusions

- **Bond loss** between silica cylinders is close to **three times lower** than previous experiments suggested
- **Thermal noise contribution** of bond layer is **below the required Advanced LIGO specification**
- **Strength of silicon-silicon** bonds is comparable to **silica-silica**
- **No correlation** between found between flatness / bond thickness / strength
- Preliminary results suggest:
- Strength at 77 K similar as at room temperature (work ongoing)
- **Thermal conductivity of bonded silicon** is approximately a **factor of four lower** than pure silicon at cryogenic temperatures (work ongoing)