

### Silicate Bonding

**Peter Murray**<sup>1</sup>, Bostjan Besentzek<sup>2</sup>, Nicola Beverage<sup>1</sup>, Gianpietro Cagnoli<sup>4,1</sup>, Enrico Campagna<sup>4,6</sup>, Elisabetta Cesarini<sup>4,5</sup>, Liam Cunningham<sup>1</sup>, John Davidson<sup>2</sup>, Karen Haughian<sup>1</sup>, Jim Hough<sup>1</sup>, Matteo Lorenzini<sup>4</sup>, Giovanni Losurdo<sup>4</sup>, Filippo Martelli<sup>5</sup>, Donald Nicholson<sup>3</sup>, Francesco Piergiovanni<sup>4,6</sup>, Stuart Reid<sup>1</sup>, Sheila Rowan<sup>1</sup>, Jamie Scott<sup>1</sup>, David Skinner<sup>1, 2</sup>, Mariëlle van Veggel<sup>1</sup>, Flavio Vetrano<sup>4,6</sup>

<sup>1</sup>SUPA Institute for Gravitational Research, University of Glasgow

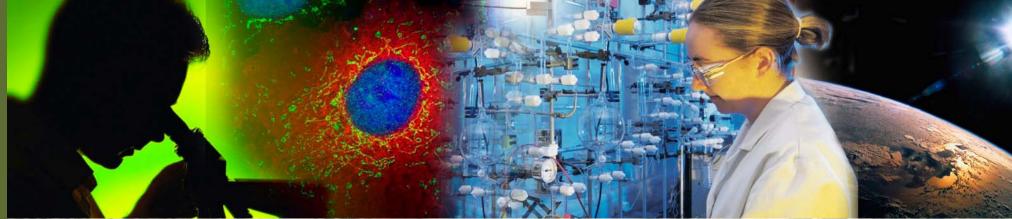
<sup>2</sup>Faculty of Mechanical Engineering, University of Glasgow

<sup>3</sup>Faculty of Electrical Engineering, University of Glasgow

<sup>4</sup>INFN Sez. Firenze

<sup>5</sup>Dip. di Astronomia e Scienza della Spazio

<sup>6</sup>Università di Urbino





- 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

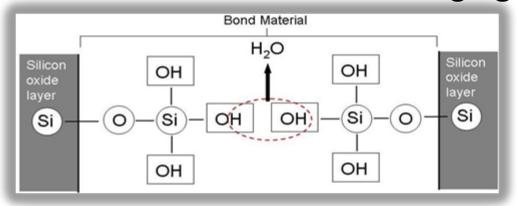




- Hydroxide-Catalysis bonding implemented in GEO600
- Was developed at Stanford University by D. H. Gwo for NASA's Gravity Probe B mission, launched April 2004, to meet the stringent strength requirements
- Enhancements to bonding technique made at University of Glasgow
- Advanced LIGO will incorporate 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



- Hydration and etching
- OH<sup>-</sup> ions in bonding solution etch the silica surfaces
- Polymerization
- Less OH<sup>-</sup> ions so pH decreases
- When pH < 11, silicate ions dissociate</li>
- 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**



The intrinsic mechanical loss factor of hydroxy-catalysis bonds for use in the mirror suspensions of gravitational wave detectors

- Investigations were made to quantify the bond loss of a hydroxide catalysis bond produced over a ~32 cm<sup>2</sup> 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

The intrinsic mechanical loss factor of hydroxy-catalysis bonds for use in the mirror suspensions of gravitational wave detectors

Pl Stadow', S Balf, G Cagady, D R M Couled, E J EBBly, J E Bale, M M Bight, J Blood and S Remail\*

\* Internal to factorize from his fraction of Street, and the couled are suppressed to the suppression of the suspension of t

Online at #16%s.

Abstract
This paper describes investigations into the machanical knots of brands ensured
by hydroxy-cardyink bradding. Evolution of the magnitude of each knots
particulated wave denotes. These samples were investigated with brands of
varying genometric and surface areas. In two cases, the brands were between
two person of leaded online, which in the first is fasted daily poer was attacked
to a suggestion relationate. In each case codium allocate ordates was used in the
variously applied to the code of the codium and the code of the code of the
variously applied to the code of the codium and the code of the
variously applied by when if the intrinsic conclusion is the clear of the brands or

and the codium and the code of the code of the code of the

conduction of the code of the code of the code of the

code of the code of the code of the code of the

code of the code of the code of the code of the

code of the code of the code of the

code of the code of the code of the

code of the code of the code of the

code of the code of the code of the

code of the code of the

code of the code of the

code of the code of the

code of the code of the

code of the code of the

code of the code of the

code of the code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code of the

code

PACS number: 64.80 Na

#### 1. Introduction

Interferenceivic gereinziatud were detecture speciel by sensing mey small relative deplicements of supposted elizares. Desirated noise from the minimi test manus and their suppraison forms one ficial to detective displacement associativity in the frequency range from a few was to banded of them. Continuousfalles can be an expensions miniging four affect aftern joined to found office or supplies not manus have been adopted [1], or are plasmed [2] for use in interferenceive few less than expensions are designed to have very low mechanical loss factors [2], and accordingly very low few he of themsel moise of temperature of absence. To present the low institute in the four-off or feet great on request to their temperature of the feet of the sension of the sension of the feet of the minimal moise at frequencies of the feet of the sension of the present of the sension.

CONTRACTOR AND ADDRESS OF THE PERSON ADDRESS OF THE PERSON AND ADDRESS OF THE PERSON ADDRE

often the justing of two mask complex or the addition of grands must be complex by allbonding. The fundamental models were the own to sail the analysis of the energy fundamental in the systems. These energy fundaments are enquired in order to obtain a value of the loss

the best married.

Assuming all other levers in the review to be negligible, and letting the measured in factors of the samples before and after the skilling of bonded attackment to written as a game.

$$\rho_{\text{table}} = \left(\frac{E_{\text{const}}}{E_{\text{const}}}\right) \rho_{\text{constant}} = \left(\frac{E_{\text{const}}}{E_{\text{const}}}\right) \rho_{\text{trace}}$$

$$= \rho_{\text{constant}} + \left(\frac{E_{\text{const}}}{E_{\text{const}}}\right) \rho_{\text{trace}}$$
(3)

By calculating the ratio of the energy stored in the actual bond exaction,  $E_{max}$ , to the energy stored in the substrate plus the bond,  $E_{max} \equiv E_{max_{max}}$ , it is then provide to obtain a value for the lors of the bond naturals,  $\phi_{max}$ . There complex were studied, and a value resistant of the lower of the bond naturals of  $\phi_{max}$ .

Measurements were made of the time factors of accions of authorized to stream supposed in various using suspensions of this fines of wire. Motion of the samples were existed introductionally and the remailing depletements of the forms factor of the sample server.

The specific generation of the bushed samples under the shows in Signer 2. Each sample was the no recognized on with firsted and the loss factors of the appropriate modes necessarial after the addition of the bushed stackments. The necessarial loss factors for the sample believe and after bushing, and the frequencies on which they were executed, are shown in table 1. For each case, reveal decays were measured and the results swraped. The errors queed state them this statistical means. For case it, is where two pieces of folice diffice of equal sizes were bushed superfice, the loss of each piece was measured argumently believe bushed. The best value channel for the pair was the same of an amounted or the believe Times.

In each case the addition of a bouled statement resulted in an increase of the measured has factors of the modes of the man studed. This is a result of come of the energy survivales with the reconstructed being dissipated by the this buys layers of silicate broading caseroid. In the case of the found sides samples the additional loss succious with the stacked pieces itself in negligible as the piece is of ensetting the same caseroid as the submitted.

\* Standard Understein bei a passer for the pressure of light-resident entities for the state of the pressure on the constant of application is as the changed from the field offs, offs out flushed type Lamentag. Standard University, 600 Wald Stand, Oato 2011, Park Alley & Committee University, 600 Wald Stand, Date 2011, Park Alley & Committee University.

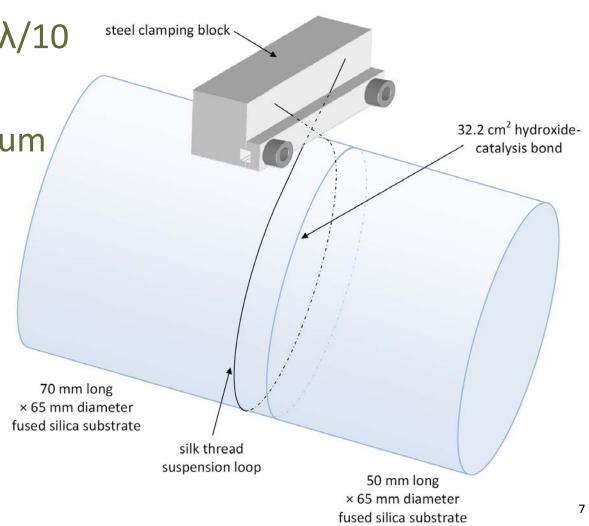


 This experiment was repeated at the University of Glasgow using fused silica cylinders of differing lengths

• Flatness specified to be  $\lambda/10$  (where  $\lambda = 633$ nm)

Bonded using 12 µl sodium silicate solution
 (14% NaOH, 27% SiO<sub>2</sub>
 diluted 1:6 in de-ionised water)

 Measurements made five months after bonding





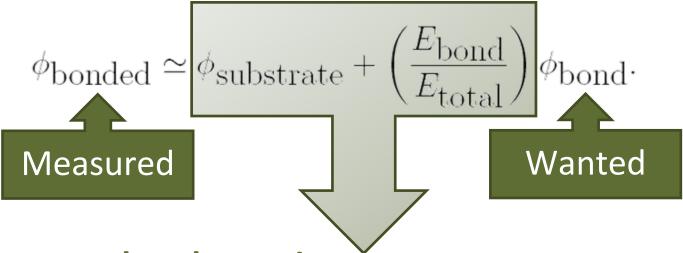


- Loss measurements made by suspending mass under vacuum
- Resonant modes excited using an electrostatic actuator
- Michelson interferometer used to sense excited motion of the front face
- Mechanical loss of resonant mode calculated from time taken for the motion to decay

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



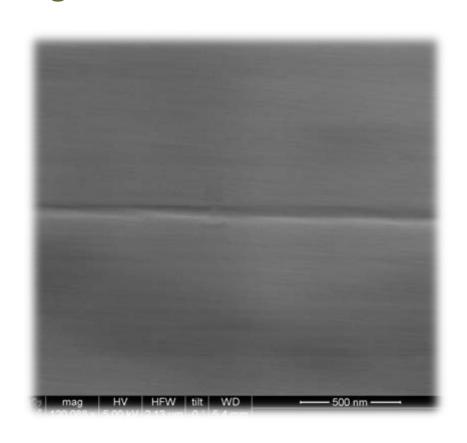
 Assuming all other losses in the system are negligible, the mechanical loss of the bonded sample may be expressed as



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



- 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



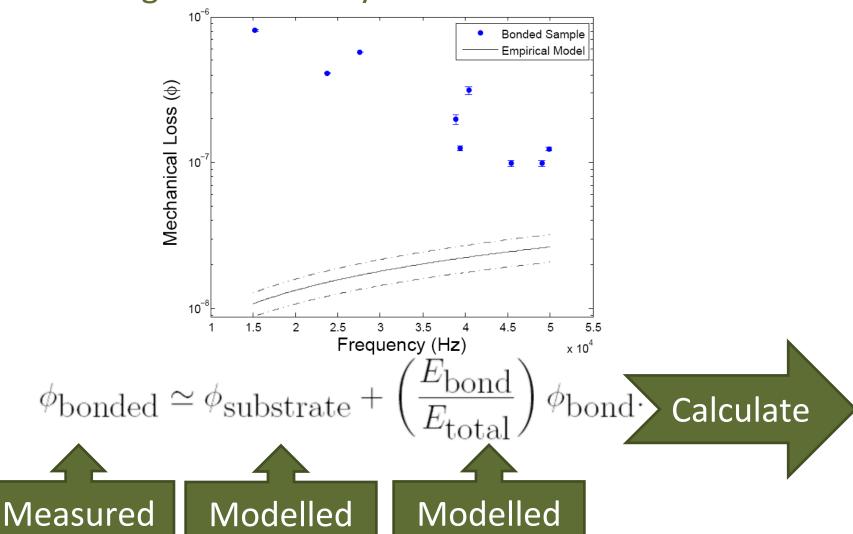


- The mechanical loss of several resonant modes of the cylinders were measured before bonding
- The lowest losses were then fitted to the Semi-empirical model of

Penn for loss in silica Surface loss term 
$$\phi\left(f,\frac{V}{S}\right) = C_1\left(\frac{V}{S}\right)^{-1} + C_2f^{C^3} + C_4\phi_{\text{th}}$$
 Thermoelastic loss term

- The mechanical losses here are measured at frequencies which lie well away from the peak of thermoelastic loss
- Suprasil 312  $C_1 = (6.5 \pm 0.2) \times 10^{-9}$   $C_2 = \text{Reduction of } 17\%$   $C_3 = (0.77 \pm 0.02).$
- Suprasil 311  $C_2 = (0.63 \pm 0.02) \times 10^{-11}$

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







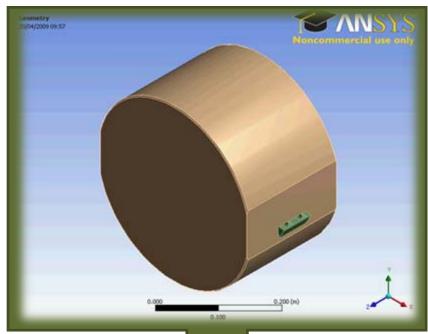
 It is now possible to calculate the bond loss for all nine resonant modes

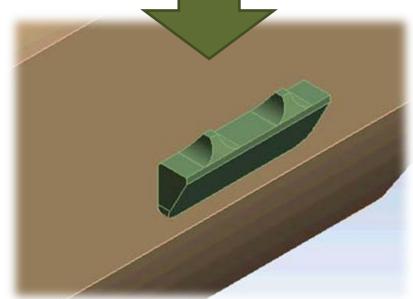
Frequency (Hz)	$\phi_{\mathbf{bond}} \ (\times 10^{-1})$
15351	$1.05 \pm 0.07$
23822	$0.51 \pm 0.03$
27674	$0.63 \pm 0.04$
38889	$1.44 \pm 0.17$
39412	$1.16 \pm 0.11$
40464	$0.78 \pm 0.07$
45463	$0.48 \pm 0.06$
49043	$1.28 \pm 0.18$
49896	$2.03 \pm 0.22$

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



- 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<sup>2</sup>









 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  $\epsilon(x,y,z)$  of elastic deformation in a test mass to obtain  $W_{diss}$
- This yielded a value of 5.2 × 10<sup>-22</sup> m/ $\sqrt{\text{Hz}}$
- In comparison the allowed upper limit for Advanced LIGO is  $7 \times 10^{-22}$  m/ $\sqrt{\text{Hz}}$  (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

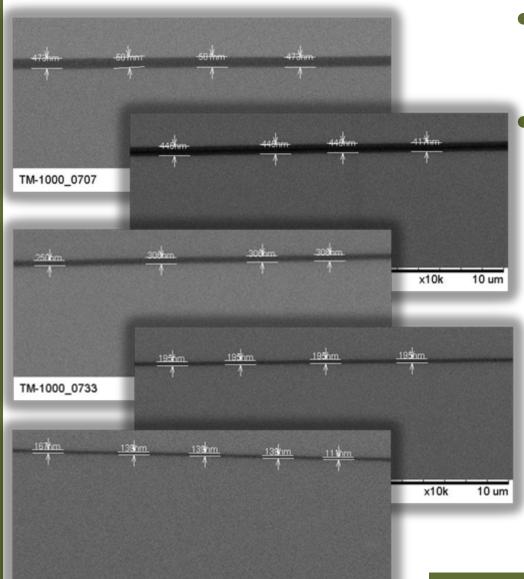
- To make bonding of silicon possible, bonding surfaces must have a thin coating of SiO<sub>2</sub>
- 50 200 nm
- This layer formed through thermal oxidation of pieces in a quartz tube furnace
- Temperature around 1000 °C
- Run with either a dry air or oxygen or a wet environment using air, nitrogen 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



TM-1000 0755

2009/02/23

#### Silicon-Silicon Hydroxide-Catalysis Bonds

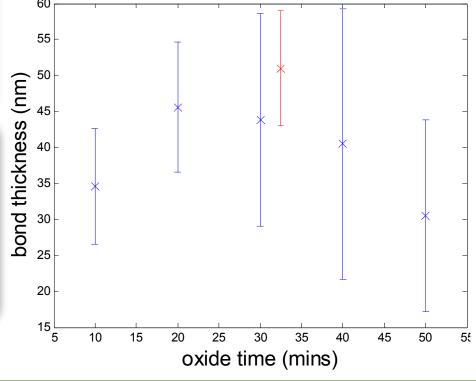


10 um

x10k

 Thicknesses of wet N<sub>2</sub> bonds measured with table-top SEM

Subtract **oxide thicknesses** measured using **ellipsometry** 



Average: 41 ± 13 nm



#### Silicon-Silicon Hydroxide-Catalysis Bonds

- Shear strength testing
- Roel-Zwick 250 kN tensile testing machine
- Maximum force applied 3500 N (equivalent to  $6.8 \times 10^6$  Nm<sup>-2</sup>)

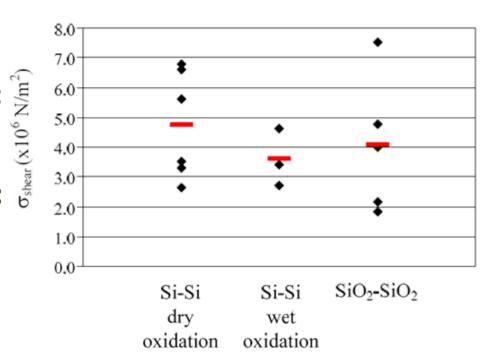




#### Silicon-Silicon Hydroxide-Catalysis Bonds

- **Average Strengths:**
- Silicon bonds, oxidised via Dry  $O_2$ :  $\sqrt[2]{\mu_N}$   $\sqrt[3]{01}$   $\sqrt[3]{01}$   $\sqrt[3]{100}$  Silicon bonds oxidised via Wet  $N_2$ :  $\sqrt[3]{01}$
- - $3.6 \times 10^6 \, \text{Nm}^{-2}$
- Silica bonds:

 $4.1 \times 10^6 \, \text{Nm}^{-2}$ 



- However, variations at level of approx  $\pm 3.0 \times 10^6 \, \text{Nm}^{-2}$
- 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

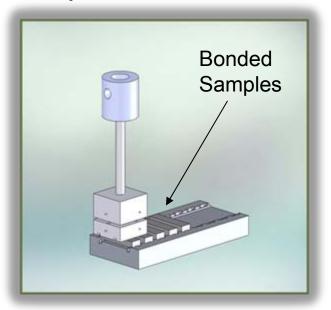


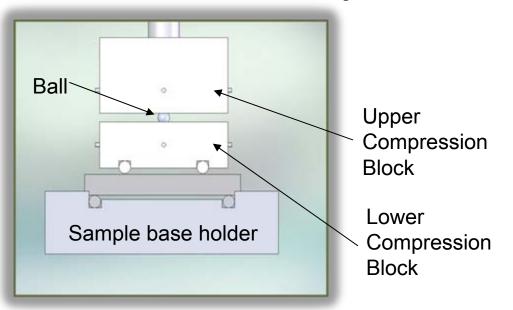
# SILICON-SILICON BOND STRENGTH LOW TEMPERATURE



#### 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 submerged 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.



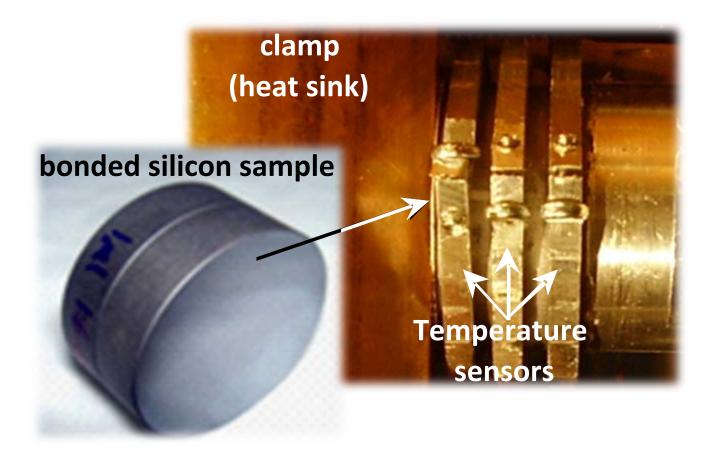
Samples After Testing



# SILICON-SILICON BOND THERMAL CONDUCTIVITY

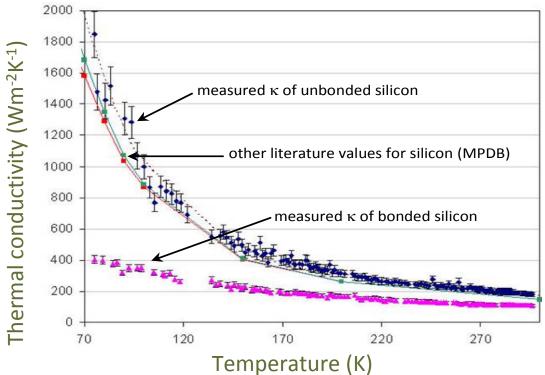


- Pairs of silicon discs of 1" diameter, 0.25" thick were bonded
- Studies of thermal conductivity of the samples are underway using a custom made facility at the University of Florence









Thermal conductivity 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



- 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)