Advances in bonding for the construction of mirror suspensions of future generation gravitational wave detectors

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on behalf of the team at the Institute for Gravitational Research, Glasgow

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Overview of the presentation

- Setting the background – bonding in gravitational wave mirror suspensions
  - Room temperature quasi-monolithic suspensions in silica GEO600, aLIGO and Advanced Virgo
  - Cryogenic suspensions in silicon or sapphire
- Introduction to hydroxide catalysis bonding (HCB) and indium bonding
- Status of research in Glasgow
  - Cryogenic strength of sapphire and silicon
  - Thermal conductivity of sapphire and silicon
  - Mechanical loss
  - Indium bonding
- Conclusions

Setting the background

Considerable research across the field devoted to developing:

- Low thermal noise test masses (e.g. presentation Gerd Hofman)
- Low thermal noise suspension fibers (e.g. presentations Rahul Kumar, Eiichi Hirose, Alexander Khalaidovski)
- Low thermal noise coatings (e.g. presentations by Innocenzo Pinto, Iain Martin, Massimo Granata, Stefan Ballmer)

Essential not to introduce significant mechanical loss in mirror suspensions through assembly technology used.

Optimum technique has to take into account materials being jointed, any process experienced by the suspension (baking etc.), operating temperature of system etc.

GEO600

Quasi-monolithic suspensions in silica

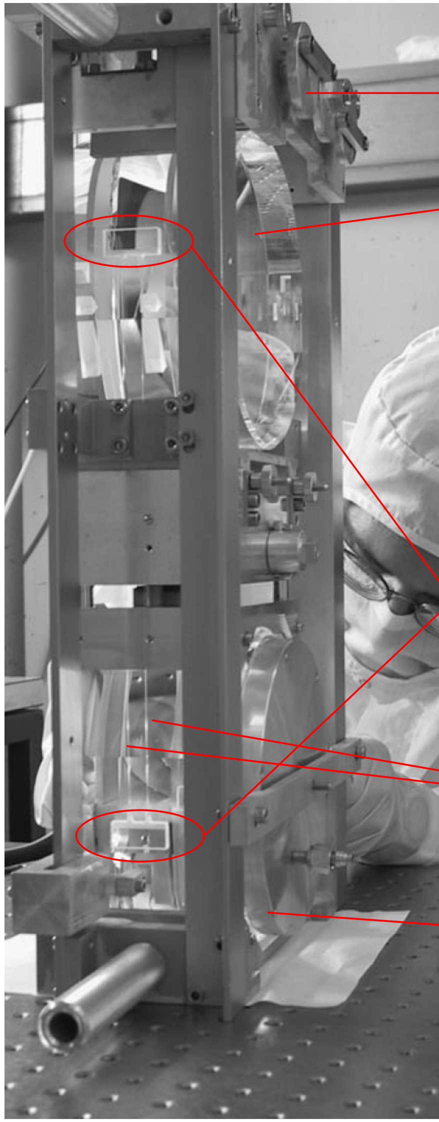
Design considerations made for GEO:

- Machined weld horns/studs into the sides of the masses is a high risk approach
  - Manufacturing
  - Thermal stresses transferring to mass
  - Contingency/repair scenarios
- **So some form of interface piece with joint was needed**
- Considered at the time:

(thesis S.M. Twyford, University of Glasgow, 1998)

  - Indium bonding – low loss @ R.T. *, however clean baking not possible
  - Optical contacting – risky
  - Hydroxide catalysis bonding – low loss* as thin, and clean bake possible

*(room T Q factors of 0.5kg mass supported by either Indium or HCB bonds broadly similar)



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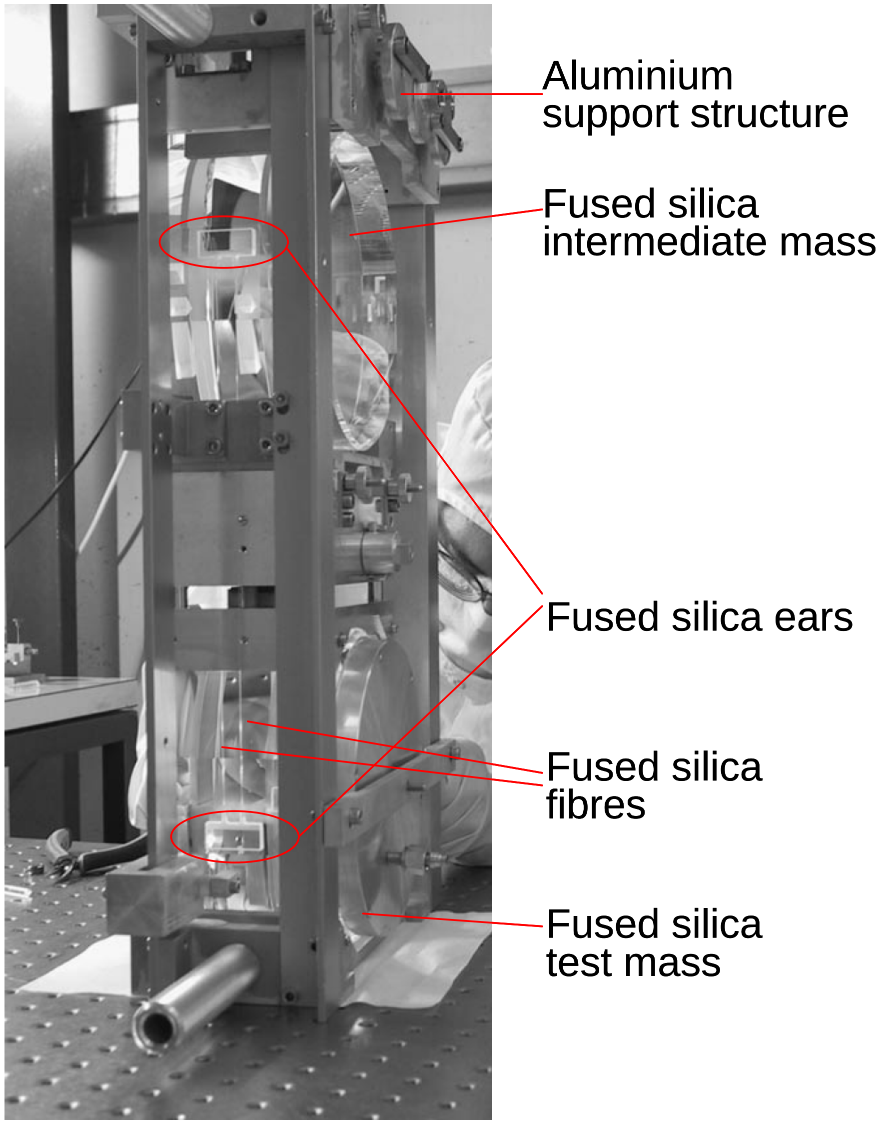
GEO600

Quasi-monolithic suspensions in silica

Hydroxide catalysis bonding

- The fused silica fibres are flame welded to fused silica ears which are hydroxide catalysis bonded (using sodium silicate solution) to the sides of the test mass and penultimate mass
- Bonds loaded in shear to ~0.16 MPa
- 4 suspension have been in operation since 2002



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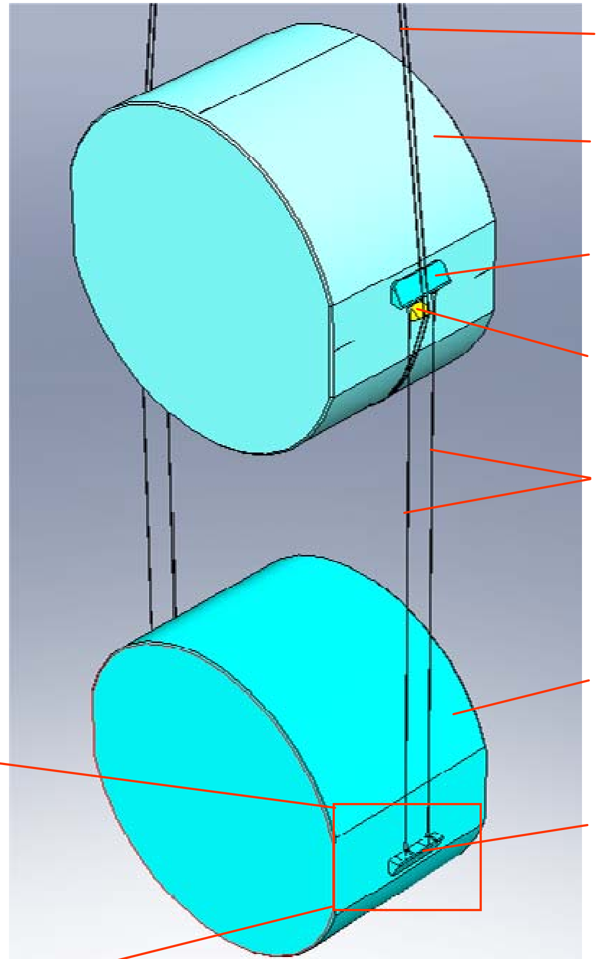
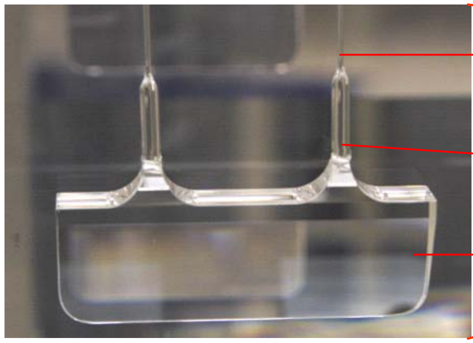
aLIGO

Quasi monolithic suspensions in fused silica

- Final stage of quadruple end and input mirrror suspensions
- Design very similar to GEO, but upscaled to take 40 kg instead of 10 kg.
- Fibres are laser pulled and laser welded to ears. Ears are bonded to masses using sodium silicate solution.
- Bonds loaded in shear ~0.16 MPa

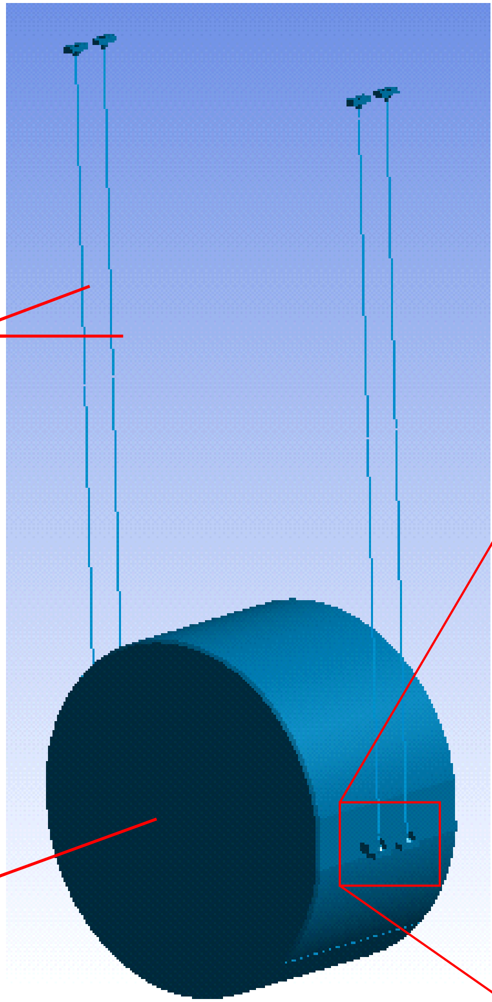


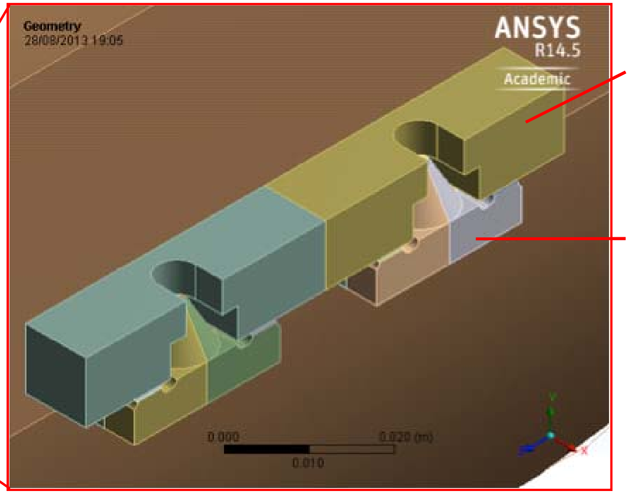


Advanced Virgo

Quasi monolithic suspensions in fused silica

- Final stage of quadruple end and input mirror suspensions
- An interface piece is bonded to the sides of the test mass. Bond in shear load.
- Laser pulled fibres with tapered nail heads are slotted into these interface pieces and bonded. Those bonds in compressive load.
- Ears are bonded to masses using potassium hydroxide solution.





Future generation detectors

Suspensions in silicon and/or sapphire?

- To further reduce thermal and suspension noise Sapphire is the baseline material for the mirror suspensions of KAGRA
- Silicon is currently considered for the Einstein Telescope and in some designs for upgrades to aLIGO.

Some questions to answer:

- What kind of design would theoretically give the lowest loss?
- Would a quasi-monolithic design with fibres/ribbons in the same material be feasible?
- What jointing techniques would be feasible and suitable?

Currently working on:

- Hydroxide catalysis bonding an indium bonding of silicon and sapphire

Introduction to
Hydroxide-catalysis bonding

First use: Gravity Probe B fused quartz telescope, ~40 bonds, operated in UHV at 2.5 K

- Materials that can be bonded:
  - Silica based materials
    - E.g. silica, Zerodur, fused silica, ULE glass and granite
  - Oxide/oxidisable materials
    - E.g. **sapphire,** PZT, silicon carbide, **silicon**

- Alkaline bonding solution
    - E.g. sodium hydroxide (NaOH), potassium hydroxide (KOH) or sodium silicate (Na_2SiO_3) dissolved in water

Introduction to Hydroxide-catalysis bonding

- To take into account when considering this as a jointing technique in cryogenic suspension
  - Bonds can be made at room temperature in air
  - Contribution to thermal noise low as it creates extremely thin bonds (<100 nm for surfaces with flatness < 100 nm peak-to-valley flatness)
  - Very strong bonds in tensile, shear and compressive load
  - Debonding (silica bonds) can be done in first few days with ability to rebond, debonding thereafter is risky (when using sodium silicate solution)
  - Clean baking possible and improves strength and loss performance

Introduction to Indium bonding

- Indium:
  - Rare, very soft, malleable and easily fusible heavy metal
  - Thermal conductivity reasonably good 82-600 W/m/K (between 273-10 K)*
  - Low melting point: 156.60 °C
  - Oxidises readily at room temperature in air
  - Joints in semiconductor industry made using indium bumps
  - Likes to ‘wet’ silica

- How to joint
  - Apply indium to both parts to be jointed
    - foil, coating or bump
  - Apply sufficient pressure to plastically deform the indium (make it flow) such that a chemical joint is made between both indium sides
  - Joints need to be made:
    - before oxidation of indium can take place,
    - in oxygen deprived environment, or
    - plastically deformed so much the native oxide layer is broken
  - Joints can be made at elevated temperature (up to 140 °C)
  - Joint thicknesses reported: 0.5 (G&H) – 50 μm (Strassle, 2011)

*Touloukian

Introduction to Indium bonding

- To take into account when considering as jointing technique in cryogenic suspension
  - Care required in fabricating indium bonds without oxidation
  - Very few strength test results reported in literature as not usually applied under load
    - Strassle et al however do report tensile strengths for rims up to 26 MPa
  - Tolerance to baking? Limited temperatures possible?
  - Mechanical dissipation of indium layers deposited by different methods?

Status of research in Glasgow

- Cryogenic strength of sapphire and silicon
- Thermal conductivity of sapphire and silicon
- Mechanical loss
- Indium bonding

Note: this work is being done in collaboration with Jena University and ICRR (Tokyo)

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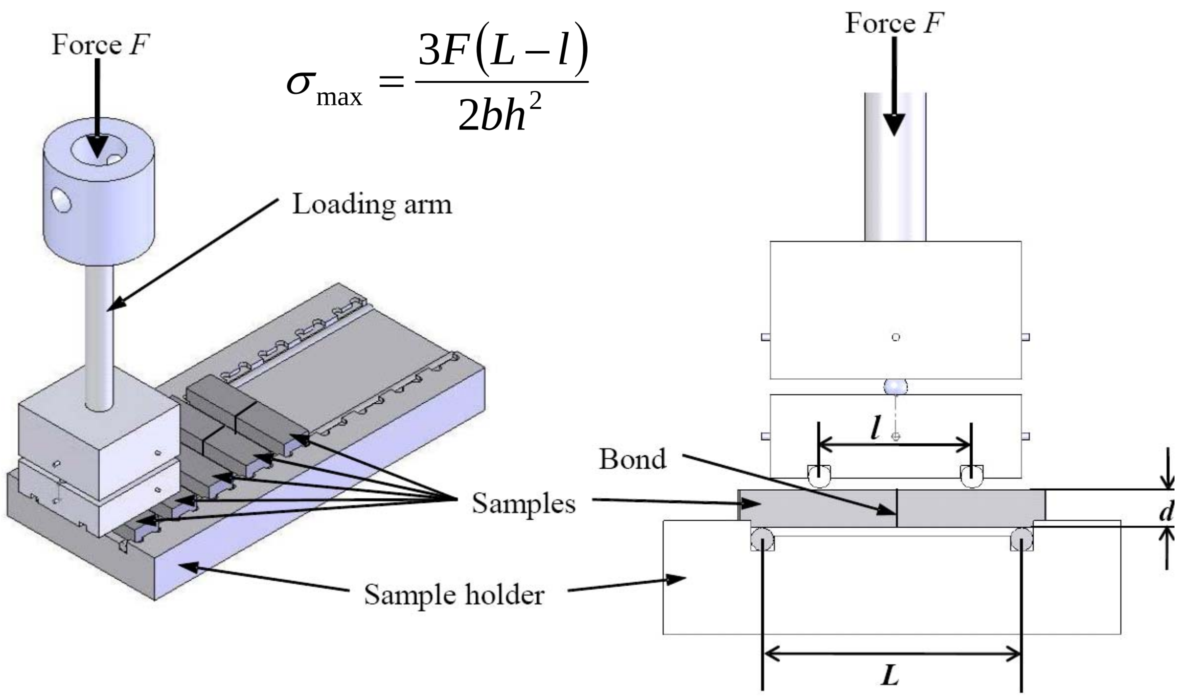
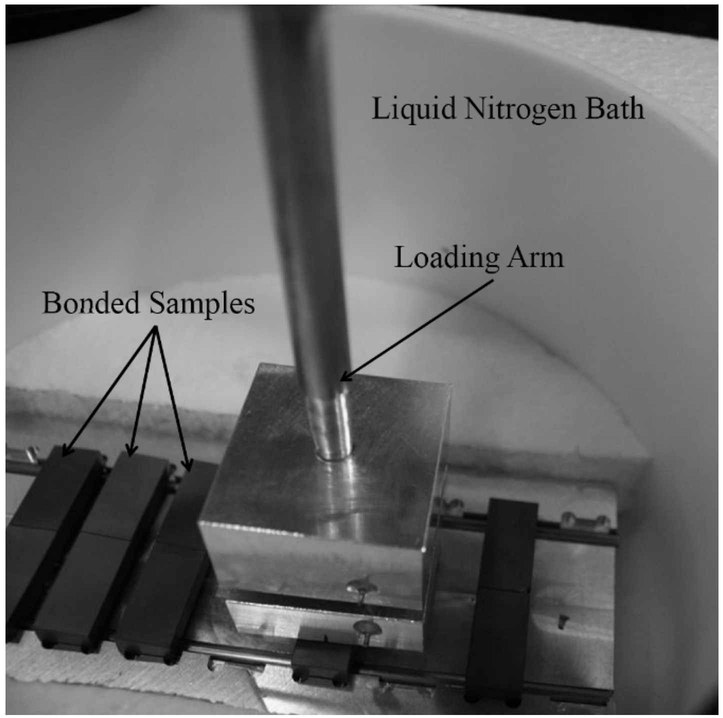
Cryogenic strength of HCBs between silicon

- Bend strength of silicon-silicon bonds @ R.T. and @ ~77 K
- Sample dimension 40x10x5 mm with bond 10x5 mm in middle
- Influence of thickness of thermal oxide layer on strength
- Influence of type of oxide layer on strength

$$\sigma_{\max} = \frac{3F(L-l)}{2bh^2}$$

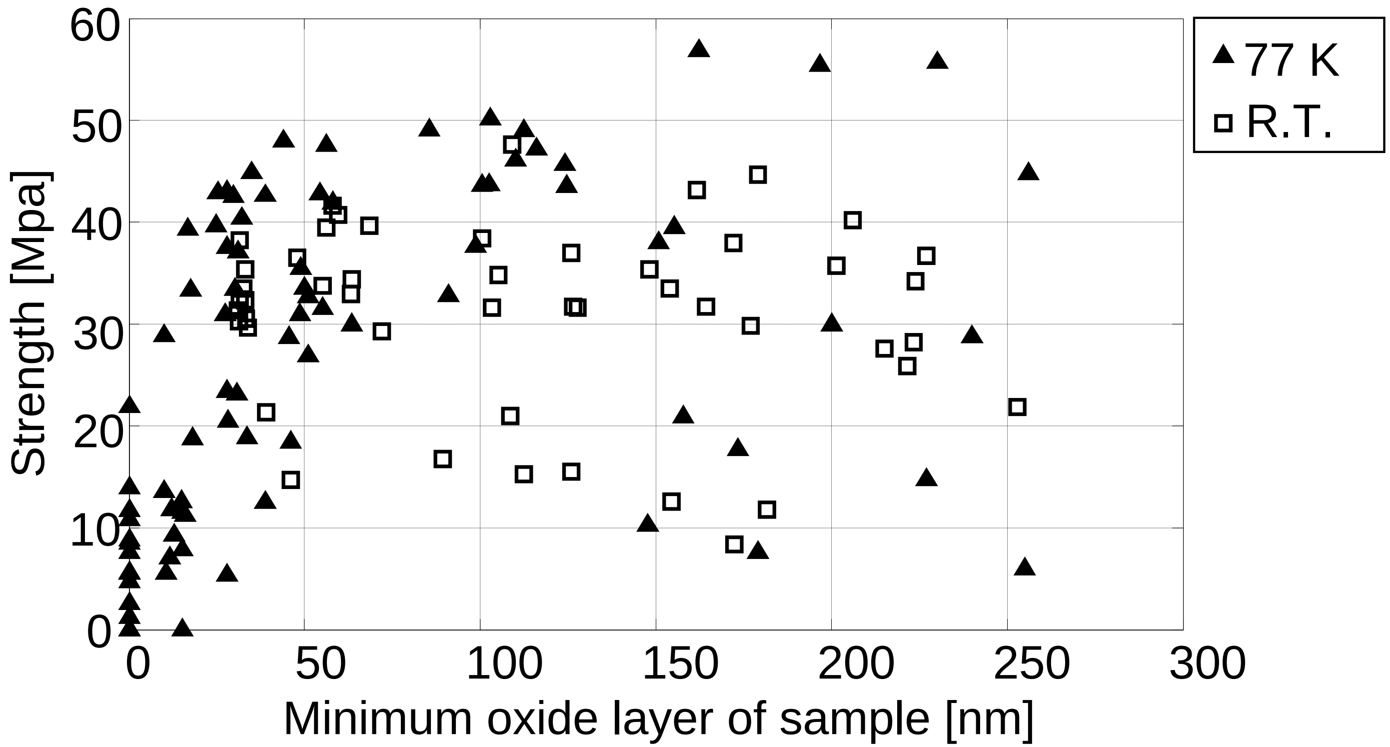




Bond strength silicon-silicon bonds

Influence of thickness of oxide layer on strength

- 49 samples @ R.T. 86 samples @ ~77 K
- Two ingot types
  - Prolog <100> (p-type Cz boron doped, unknown resistance)
  - Prolog <111> (p-type Cz boron doped, unknown resistance)
- Results published: Beveridge et al., CQG, 2011



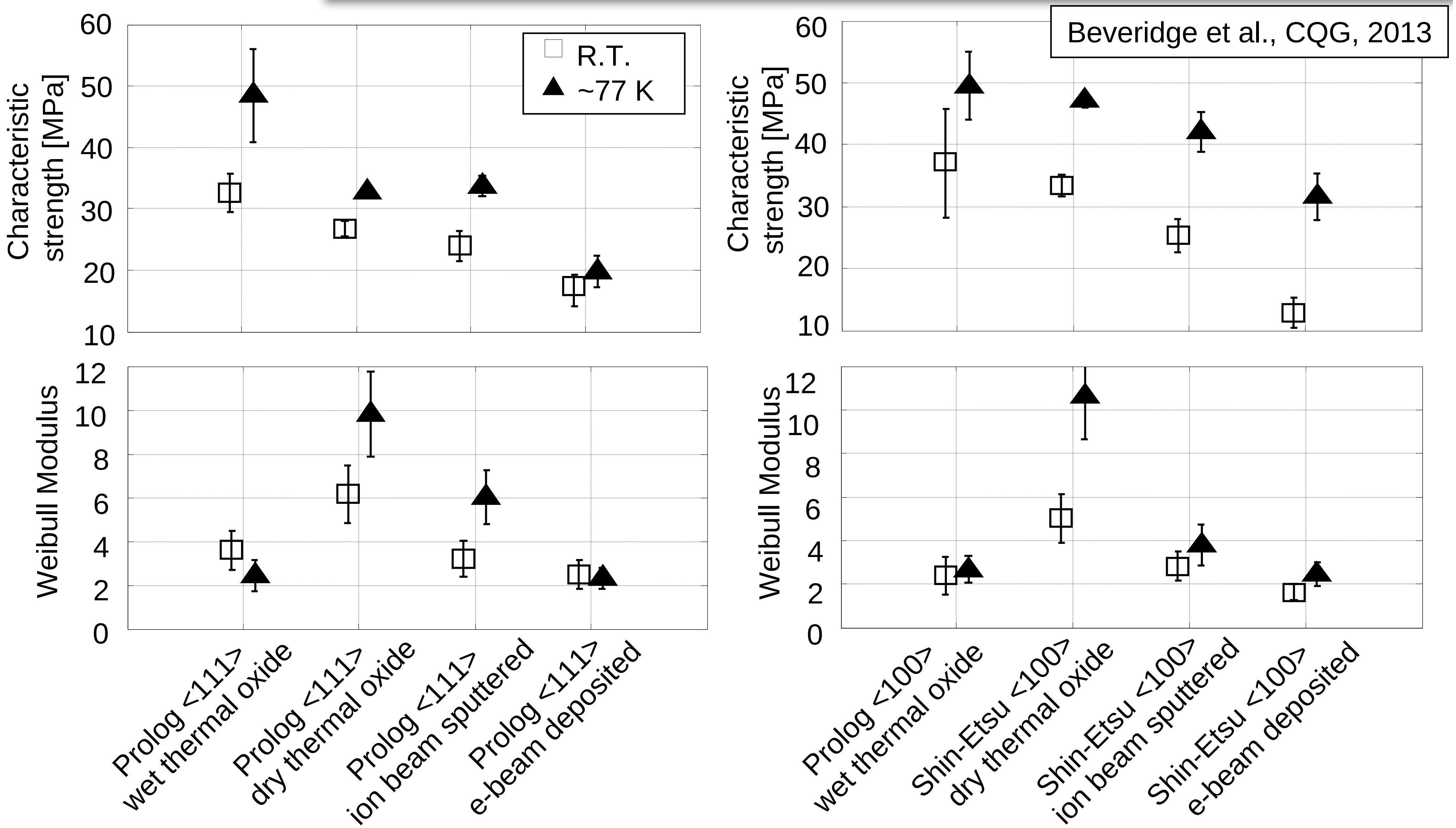
Results:

Drop in strength when minimum oxide thickness <50 nm

Characteristic strength
34 MPa @ R.T.
41 MPa @ 77 K

Bond strength silicon-silicon bonds

Influence of type of thermal oxide layer



Bond strength silicon-silicon bonds

Questions remaining

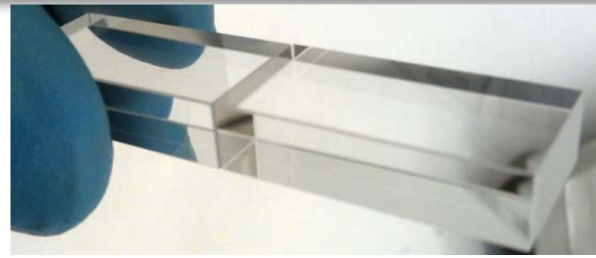
- How do silicon-silicon HCBs respond to thermal cycling?
- Why are CT results often stronger than RT?
- What is causing difference in strength: crystal orientation or purity? And why?
- Why do mixed orientations give stronger bonds?
- What is the minimum oxide layer thickness for ion-beam sputtered coatings?

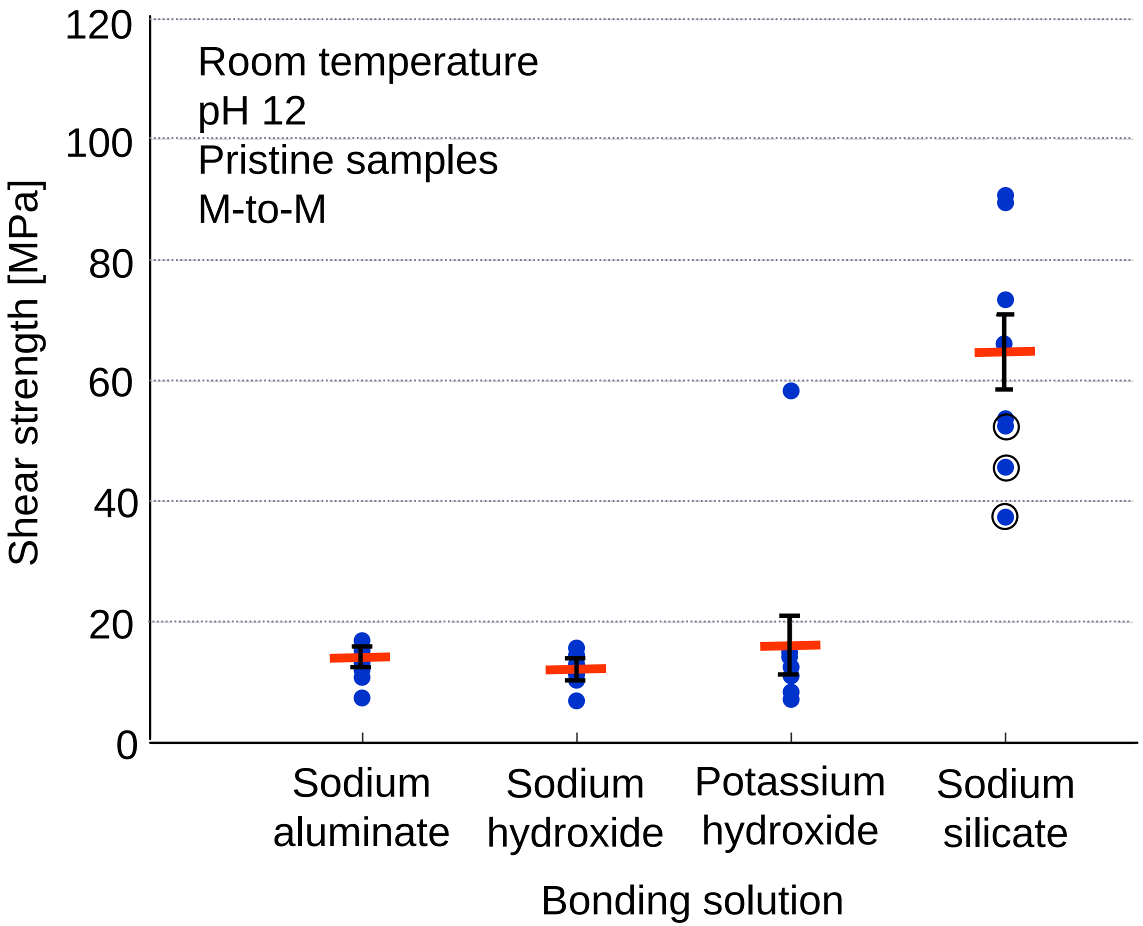
Cryogenic strength of HCBs between sapphire

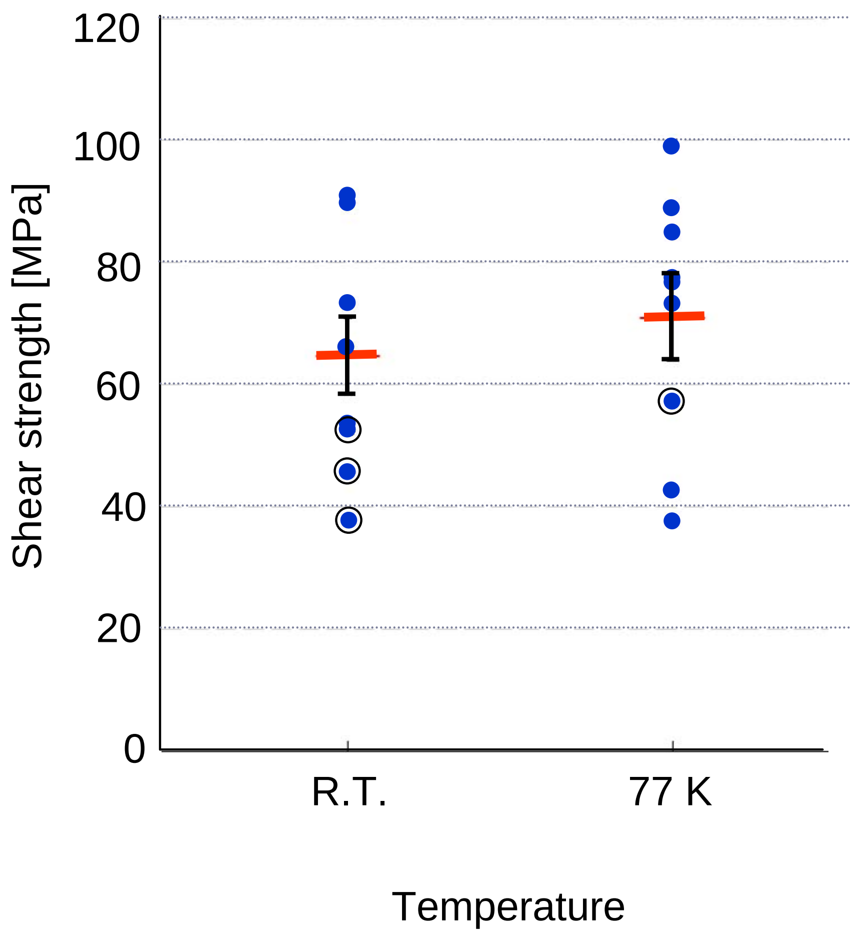
- Glasgow bend strength of sapphire-sapphire bonds at room temperature and at ~77 K (as silicon-silicon)
- Tokyo and Glasgow torsional shear strength of sapphire-sapphire bonds at ~7 K
- Influence of type of bonding solution, number of times (re-)bonded, crystal orientation
- See poster Rebecca Douglas

Cryogenic strength of HCBs between sapphire

- 40x10x5 mm samples, 10x5 mm bond in the middle
- M-to-M-axis perpendicular to bond
- Results published Douglas et al., CQG, 2014

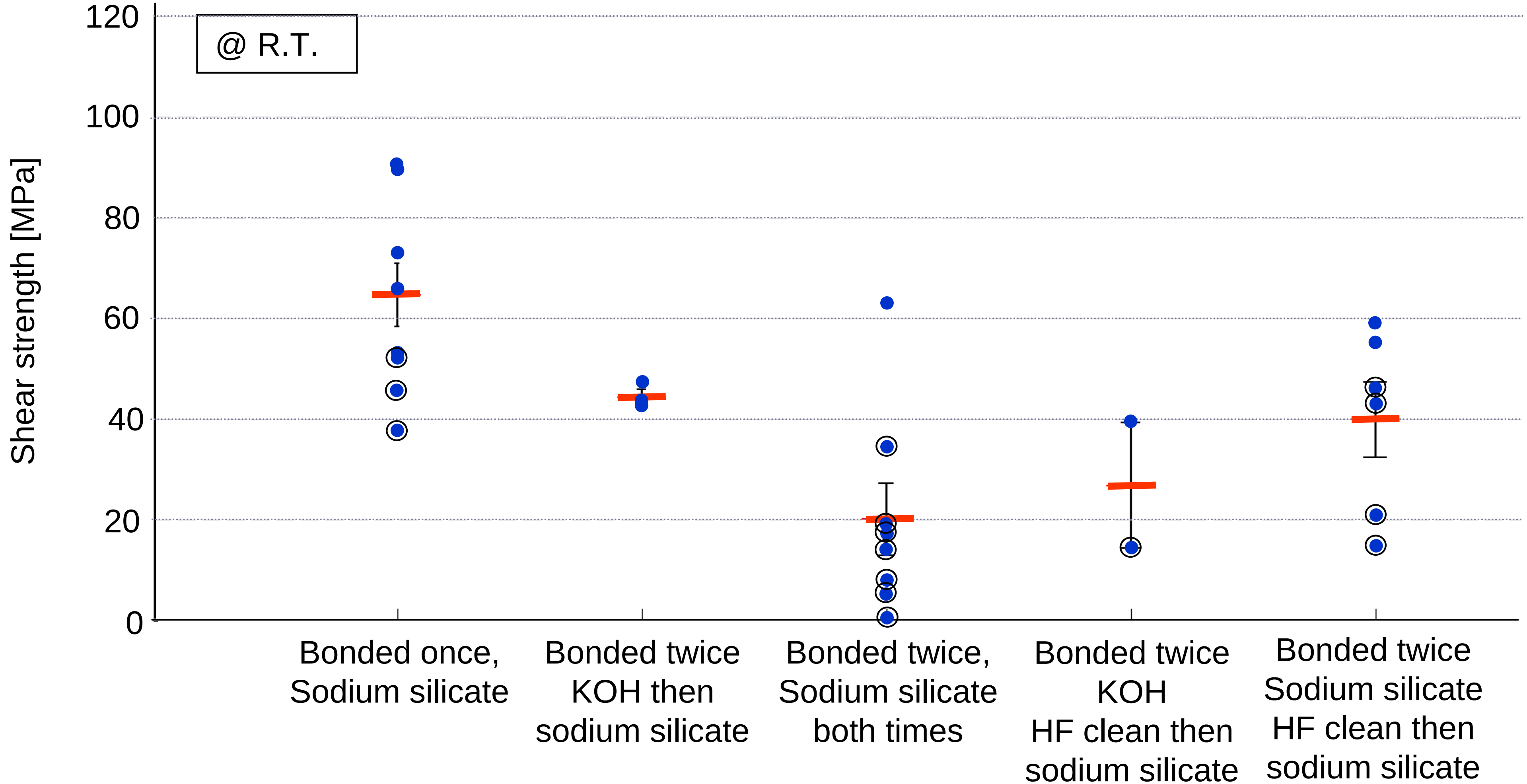






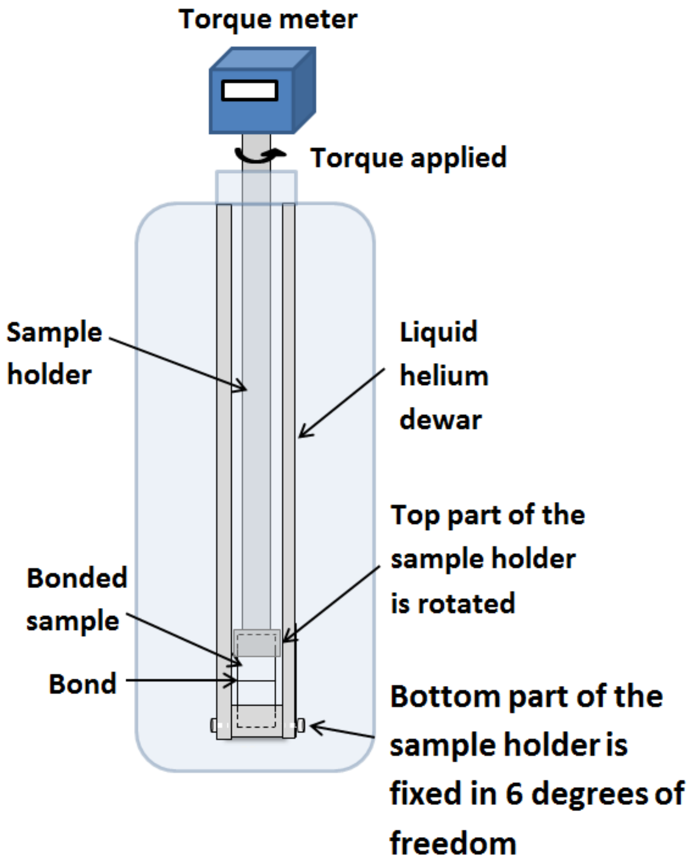
Cryogenic strength of HCBs between sapphire

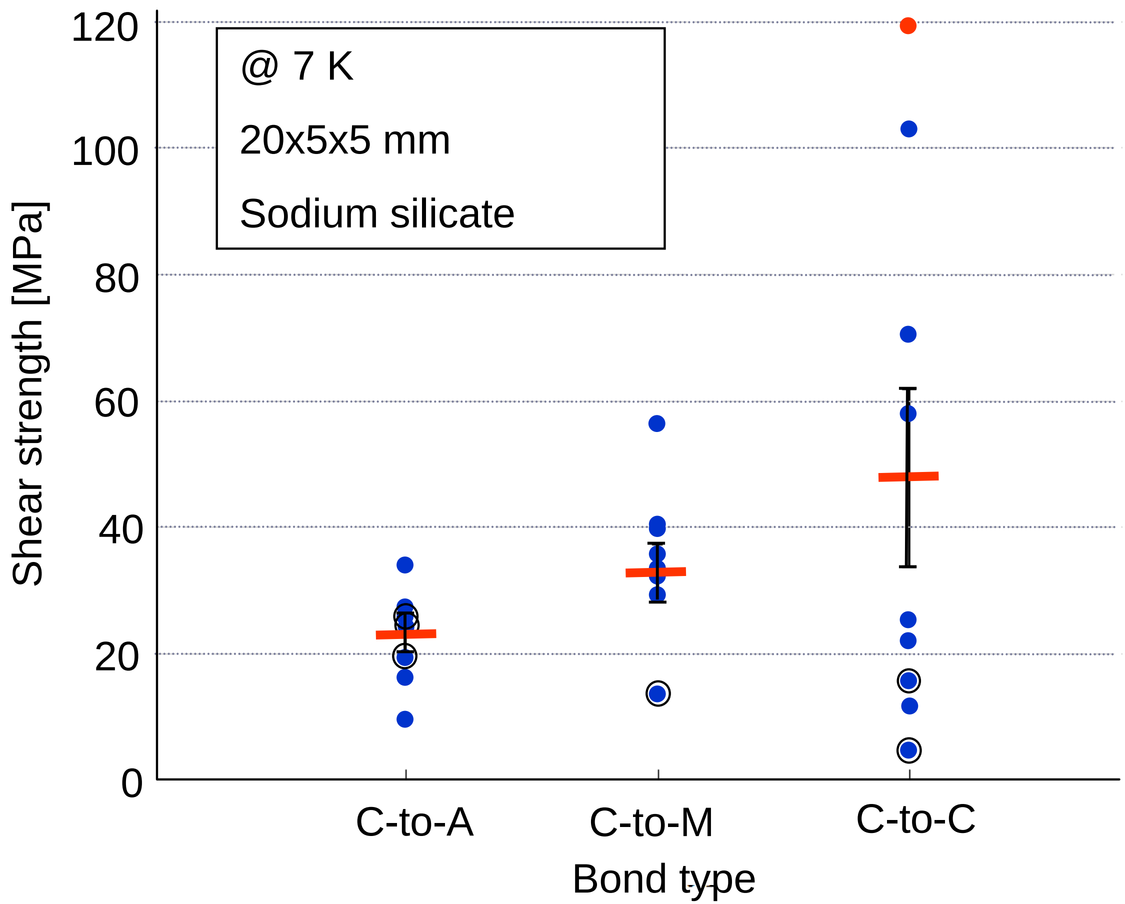
- Further experiments re-used samples: experiments ongoing, nearing completion



Cryogenic strength of HCBs between sapphire

- Samples bonded and tested collaboratively between Tokyo and Glasgow (Elites Exchange Programme), analysis ongoing, nearing completion





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Bond strength sapphire-sapphire bonds

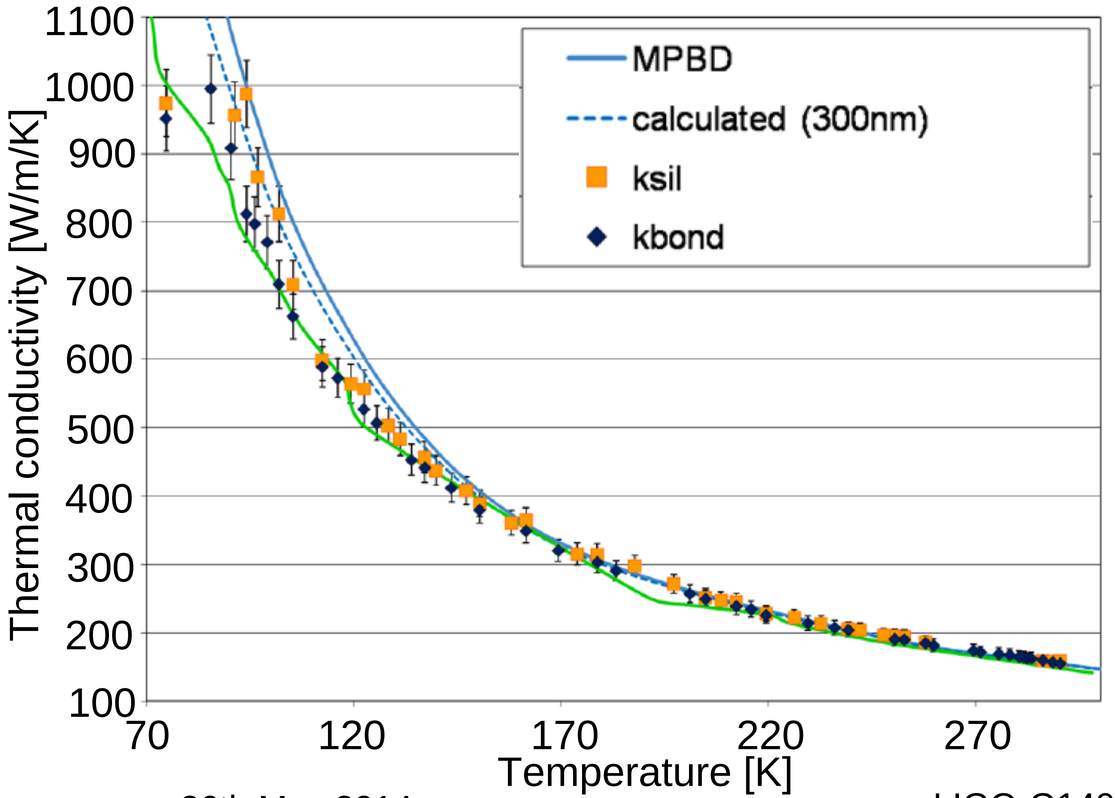
Questions remaining

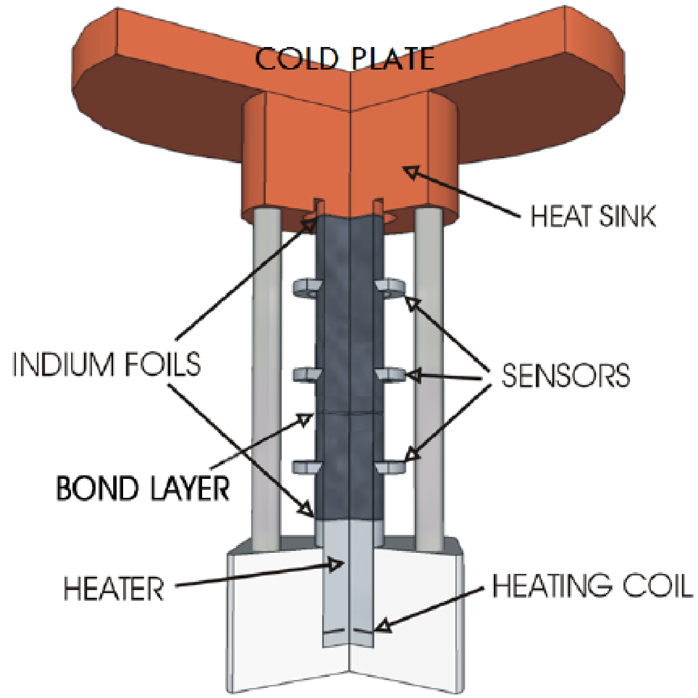
- How do sapphire-sapphire HCBs respond to thermal cycling?
- What is the curing time for sapphire bonds to reach maximum strength?
- Why does bonding different crystal orientations lead to different strengths?
- How can we improve re-bondability?

Thermal conductivity of HCBs between silicon and sapphire

Research done by Florence in collaboration with Glasgow

- Silicon sample 25 mm diameter cross section 80 mm long
- Down to liquid nitrogen temperatures





No significant reduction thermal conductivity from bond compared to bulk

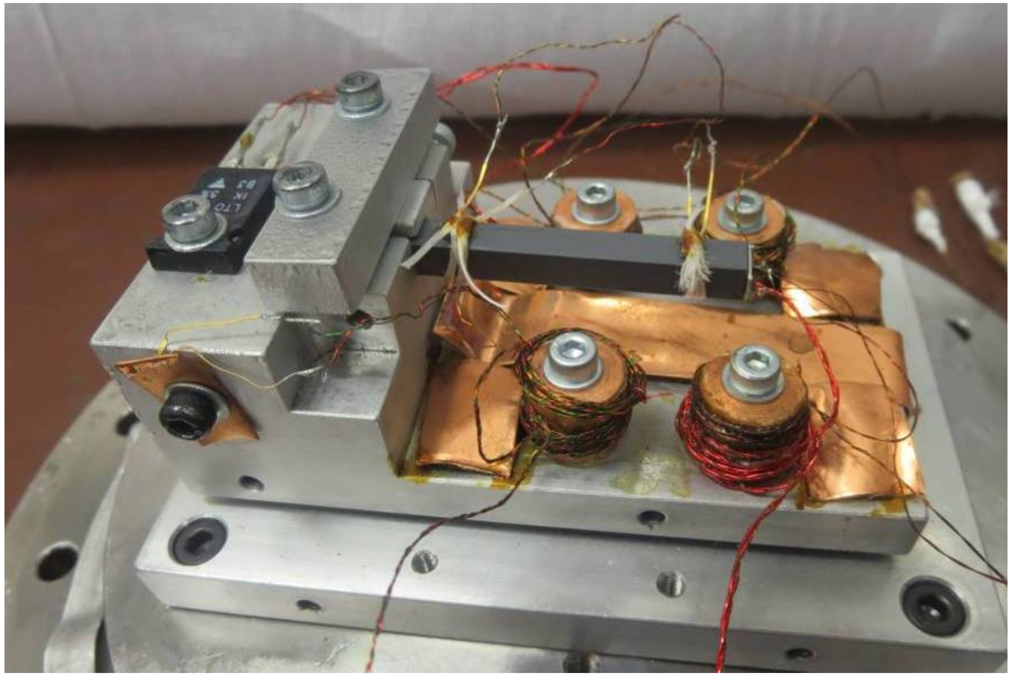
Lorenzini et al. Journal of Physics: Conference Series **228** (2010) 012019

Thermal conductivity of HCBs between silicon and sapphire

- Two experiments in refinement stage to measure thermal conductivity of bonds.
  - Both experiments measuring 5x5 mm cross-section samples
  - Minimising heating power loss

- ICRR focussing on measuring thermal conductivity of HCB and indium bonds between sapphire (see poster Dan Chen)

- Glasgow focussing on measuring thermal conductivity of HCB and indium bonds between silicon



Thermal conductivity set-up Glasgow

Mechanical loss of HCBs between silicon and sapphire

- Loss of bond between silicon-silicon bulk cylinder upper limit Karen Haughian thesis 0.26-0.52 (this experiment was limited due to an off-set between bonded cylinders)
- Sapphire bulk cylinders (from ICRR) currently being measured at R.T. before bonding to ensure no excess losses. Control sample already measured at Jena University. They will be bonded and then loss measured down to cryogenic.
- Modelling exercise in Glasgow to optimise sample geometry.

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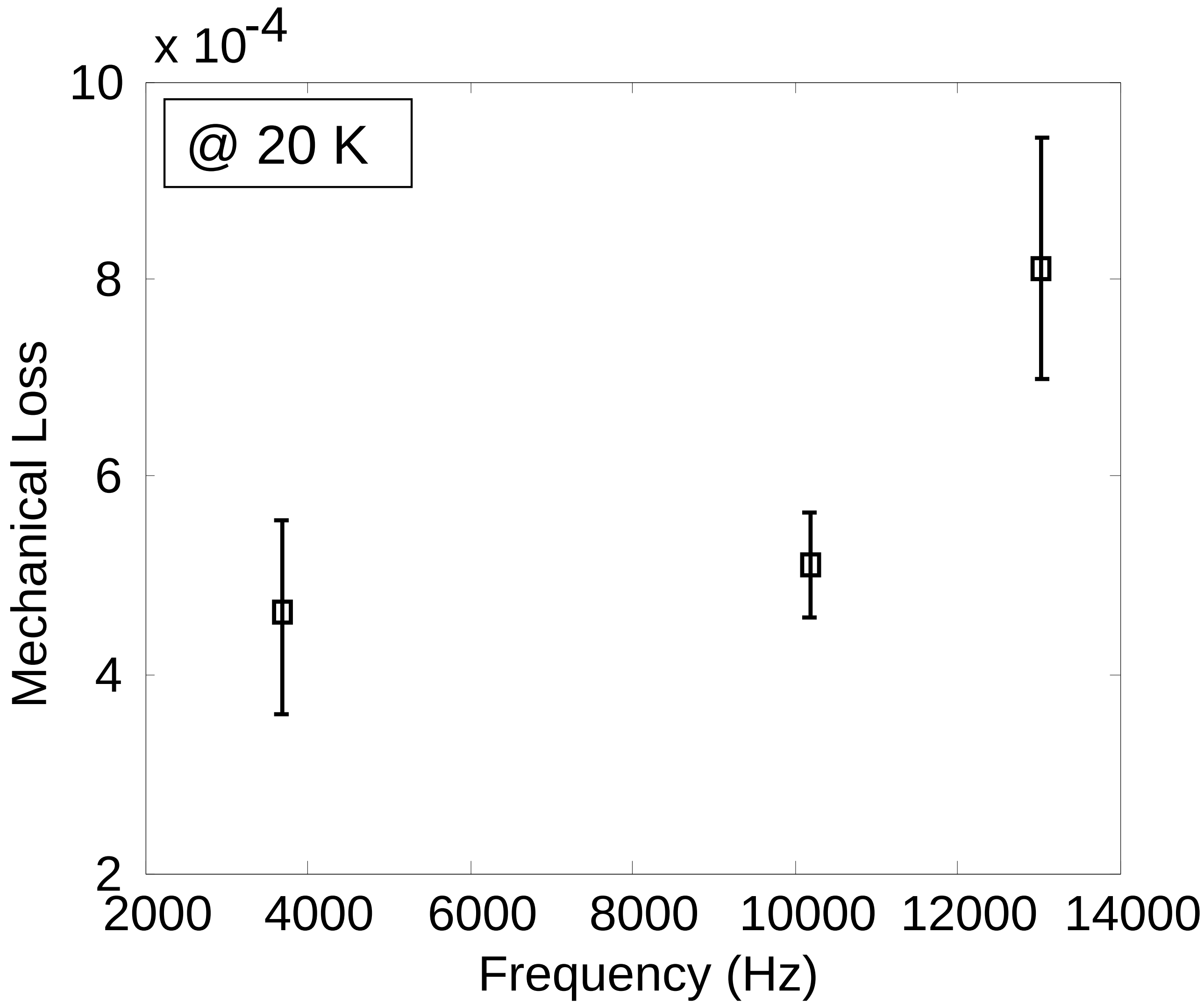
Indium bonding

- Jena and Glasgow: loss measurements of 530 nm sputtered indium thin film down to 10 K (paper in preparation)
- Glasgow: working on producing indium bonds using evaporated coatings
- Glasgow: have ordered indium bonds made using sputtered indium coatings between silicon and sapphire samples
- ICRR: have produced an indium bond with an indium foil between sapphire samples for thermal conductivity measurements (see talk Alexander Khalaidovski and poster Dan Chen)

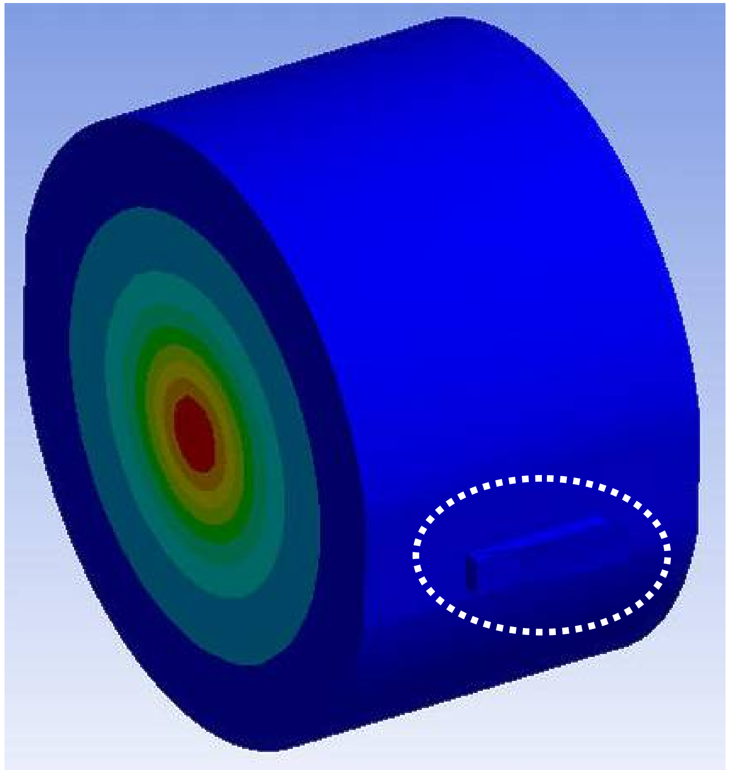
Loss results for indium thin film

- Silicon cantilever with 530 nm +/- 30 nm sputtered indium film
- Result shown for 3 modes at 20 K (loss ~constant between 10 – 80 K)
- Loss at R.T. is 0.02; same order of magnitude as bond loss



Thermal noise associated with HCBs or indium bonds between silicon and sapphire

Aim: some initial idea of levels of thermal noise



FE models of aLIGO size test mass with aLIGO size ears

A Gaussian pressure wave is applied to front surface which causes deformation of the bond to calculate strain energy in the bond

Using Levin's method thermal noise associated with the strain energy at 100 Hz is calculated.

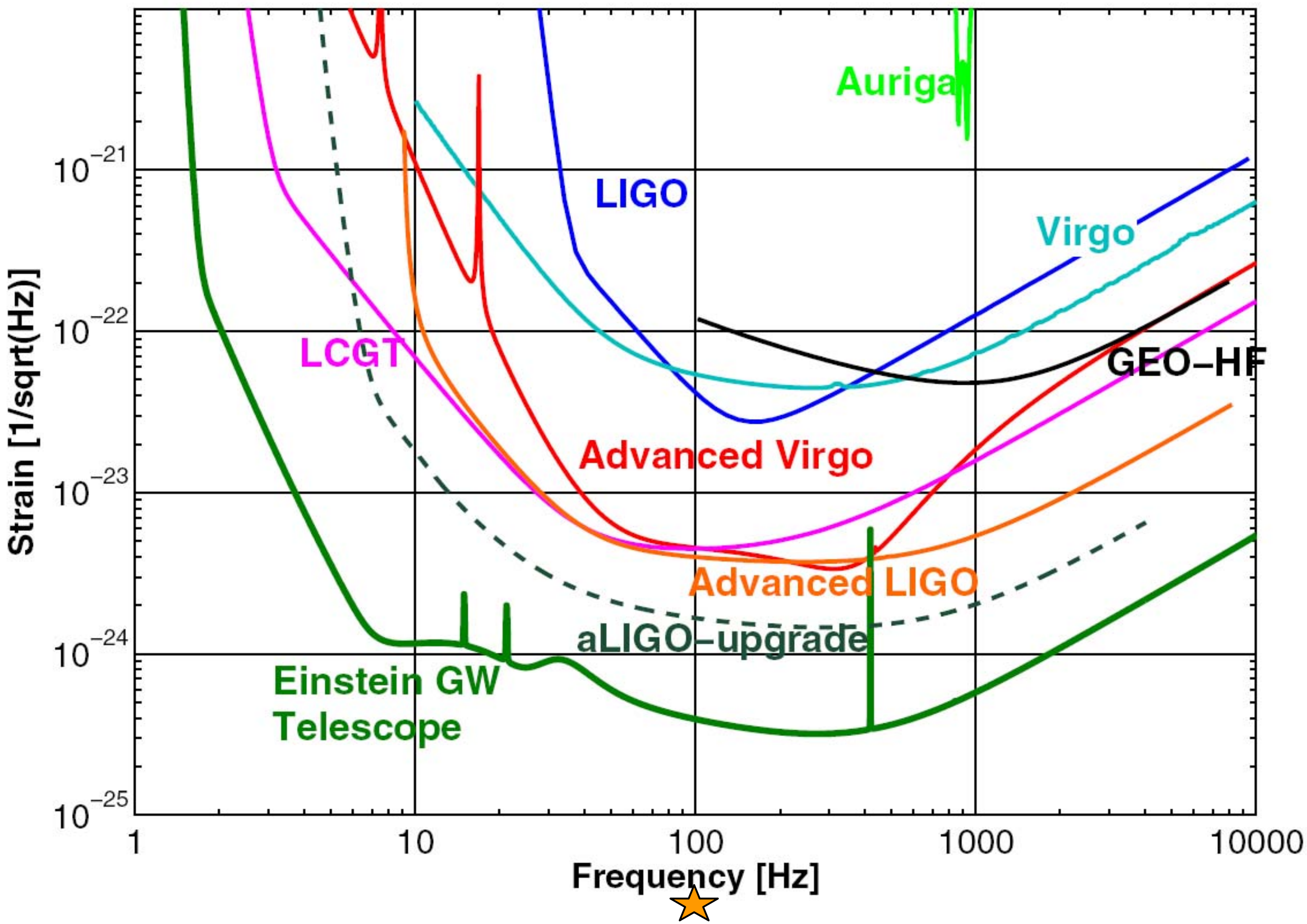
$$S_x(f) = \frac{2k_B T W_{diss}}{\pi^2 f^2 F_0^2}$$

$$W_{diss} = 2\pi f \int_{vol} \varepsilon(x,y,z)\phi(x,y,z)\, dV$$

Thermal noise associated with HCBs between silicon and sapphire

system	40K	125K	Assumptions:
sapphire mass indium bond	$4.73 \cdot 10^{-23}$	$1.11 \cdot 10^{-22}$	Indium loss values used from recent loss measurements 530 nm sputtered indium coating Indium loss @ 40 K 5.8e-4 @ 125 K 1.0e-3
sapphire mass HCB bond	$2.30 \cdot 10^{-22}$	$4.06 \cdot 10^{-22}$	
silicon HCB bond and oxide	$2.90 \cdot 10^{-22}$	$5.01 \cdot 10^{-22}$	Conservative values for oxide loss @ 40K 7.0e-4 @ 125K 3.5e-4 Bond mechanical loss measured at R.T. = 0.06 Assumed to be constant for lower T Bond thickness 61 nm

Thermal noise associated with HCBs between silicon and sapphire



S. Hild, CQG, 2012

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Conclusions and next steps

- HCB bond strength silicon and sapphire promising
  - Questions to answer: response to thermal cycling, curing time, repair scenarios
- Thermal conductivity of sapphire and silicon
  - Experiments running and being perfected to allow for first results of indium and HCB bonds between silicon and sapphire bonds
- Mechanical loss
  - Measurements of mechanical loss of HCBs or indium bonds at cryogenic temperature

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Thank you!

GEO600

Quasi-monolithic suspensions in silica

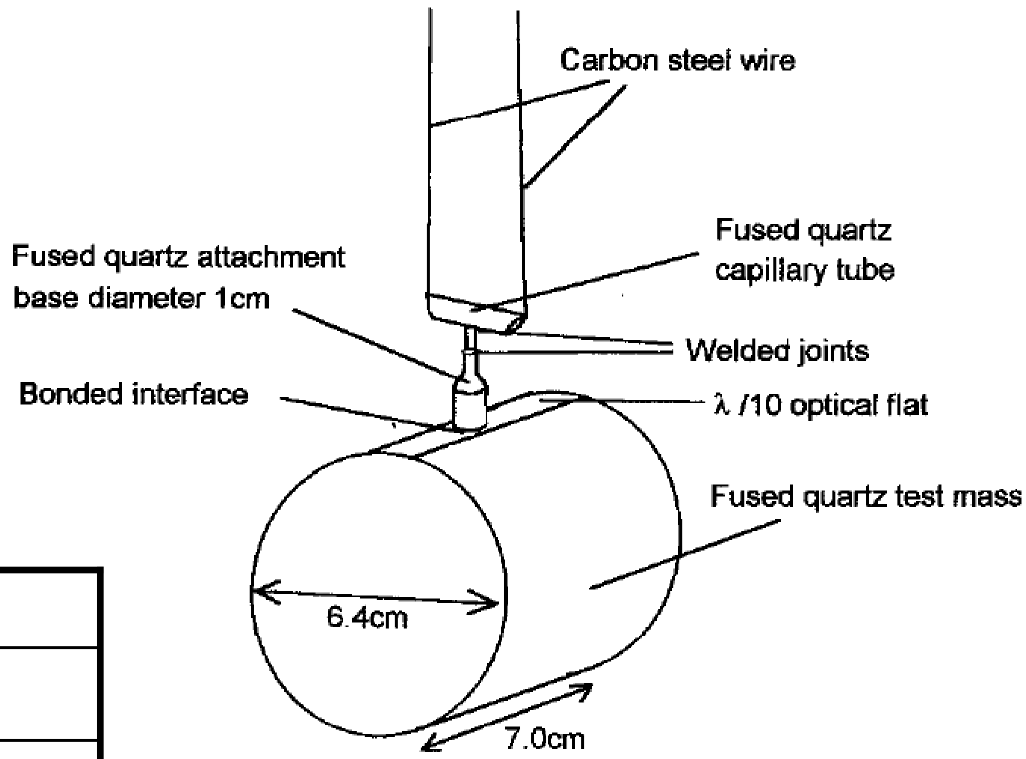
Design considerations made for GEO:

- Jointing techniques considered at the time:

  (thesis S.M. Twyford, University of Glasgow, 1998)

  - Indium bonding – did show low loss @ R.T., however clean baking not possible
  - Hydroxide catalysis bonding – low loss as thin and clean bake possible
  - Optical contacting – risky

Excess loss scaled to GEO mass from	
Indium bond	$1.3 \cdot 10^{-7}$
HCB (KOH)	$6 \cdot 10^{-9}$



(thesis S.M. Twyford, University of Glasgow, 1998
Rowan et al., Phys. Lett. A, 1998)

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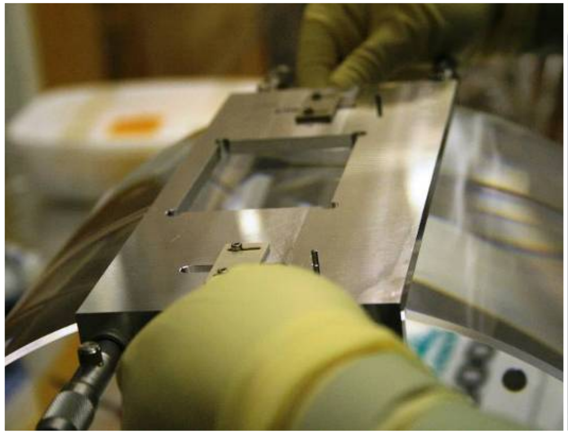
aLIGO
Monolithic suspension procedure

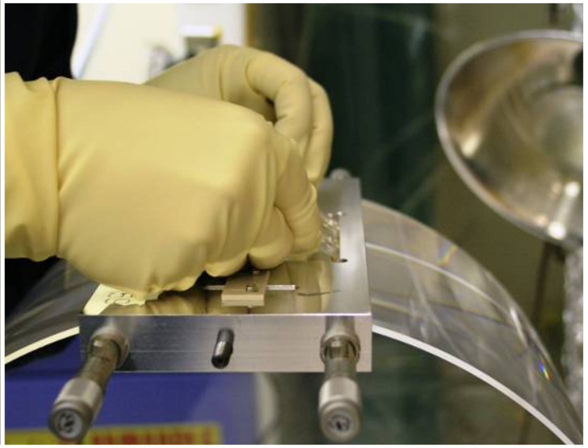
3 main stages

- Preparing masses by hydroxide catalysis bonding of the ears to:
  - the test mass and
  - the penultimate mass
- Manufacturing and testing of the fibres
- Installation of fibres using laser welding



Placing bonding jig

Applying bonding solution

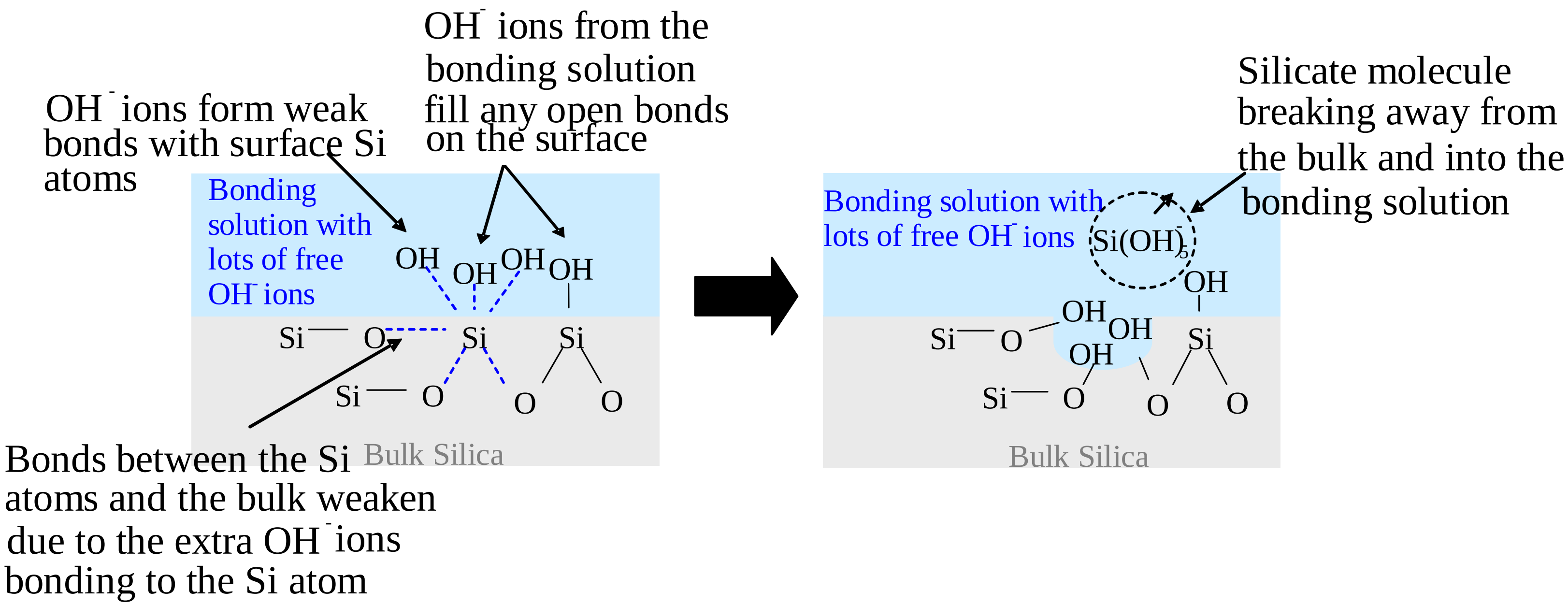


Putting down ear

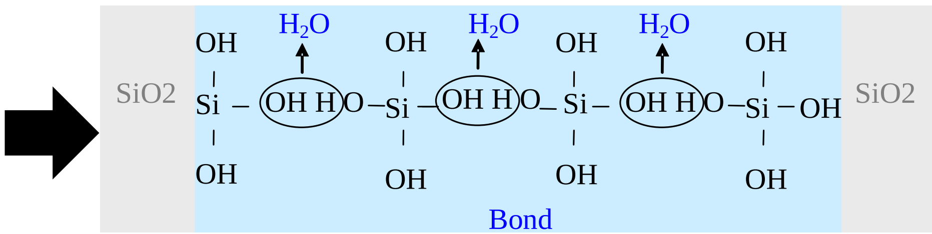
Introduction to Hydroxide-catalysis bonding

- Chemistry in three stages:

1. Hydration and etching **2. Polymerisation**



3. Dehydration



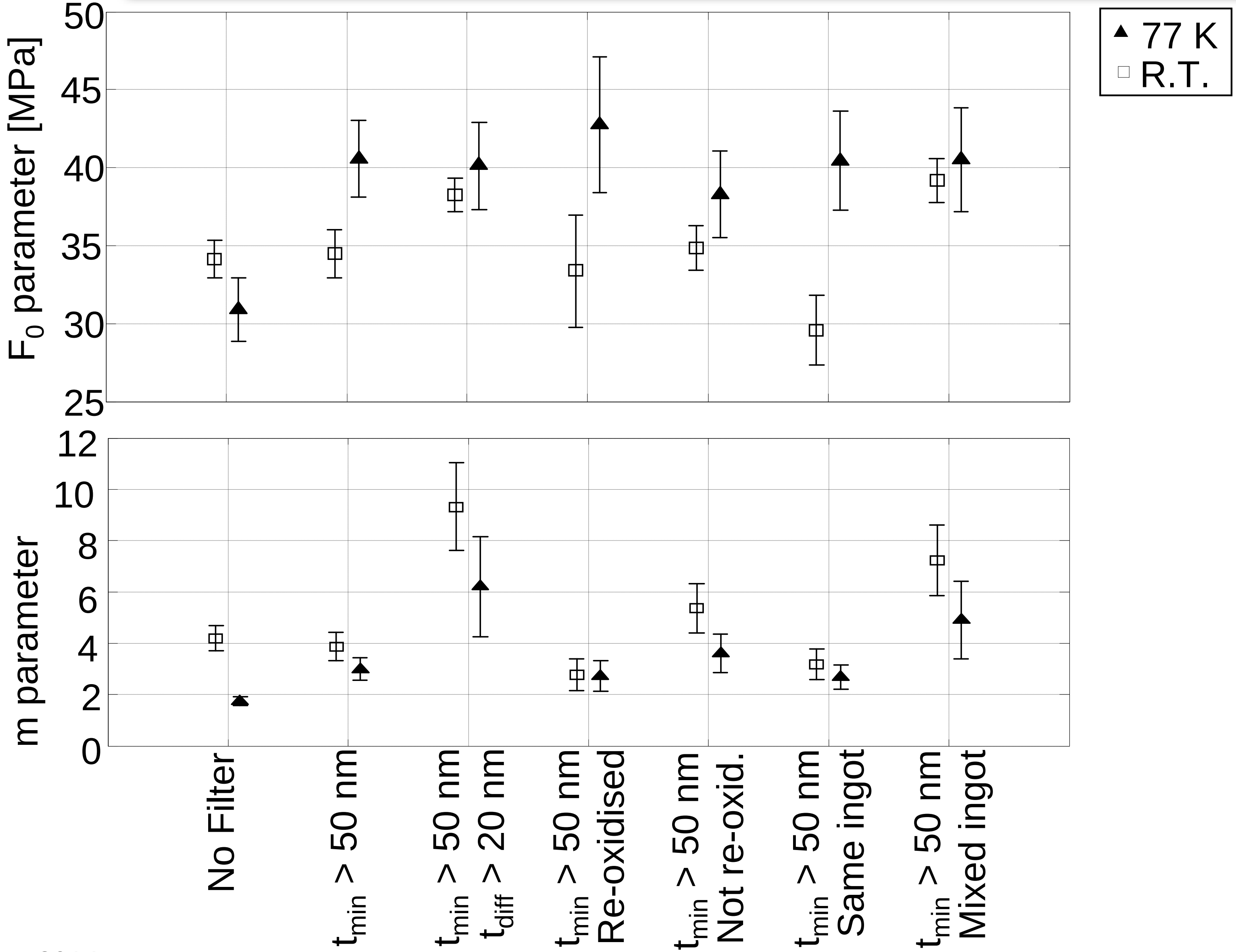
Bond strength silicon-silicon bonds

Influence of type of oxide layer

- 82 samples @ R.T.   88 samples @ ~77 K
- Three types of oxide layer
  - Dry thermal oxide (165 ± 14 nm) - University of Glasgow
  - E-beam deposition (144 ± 1 nm) - Gooch and Housego
  - Ion beam sputtered (154 ± 1 nm) - Advanced Thin Films
- Two ingot types
  - Shin-Etsu <100> (n-type F-Z phosphorous doped, 56.0 - 76.0 Ohm-cm)
  - Prolog <111> (p-type Cz boron doped, unknown resistance)
- Results published: Beveridge et al., CQG, 2013

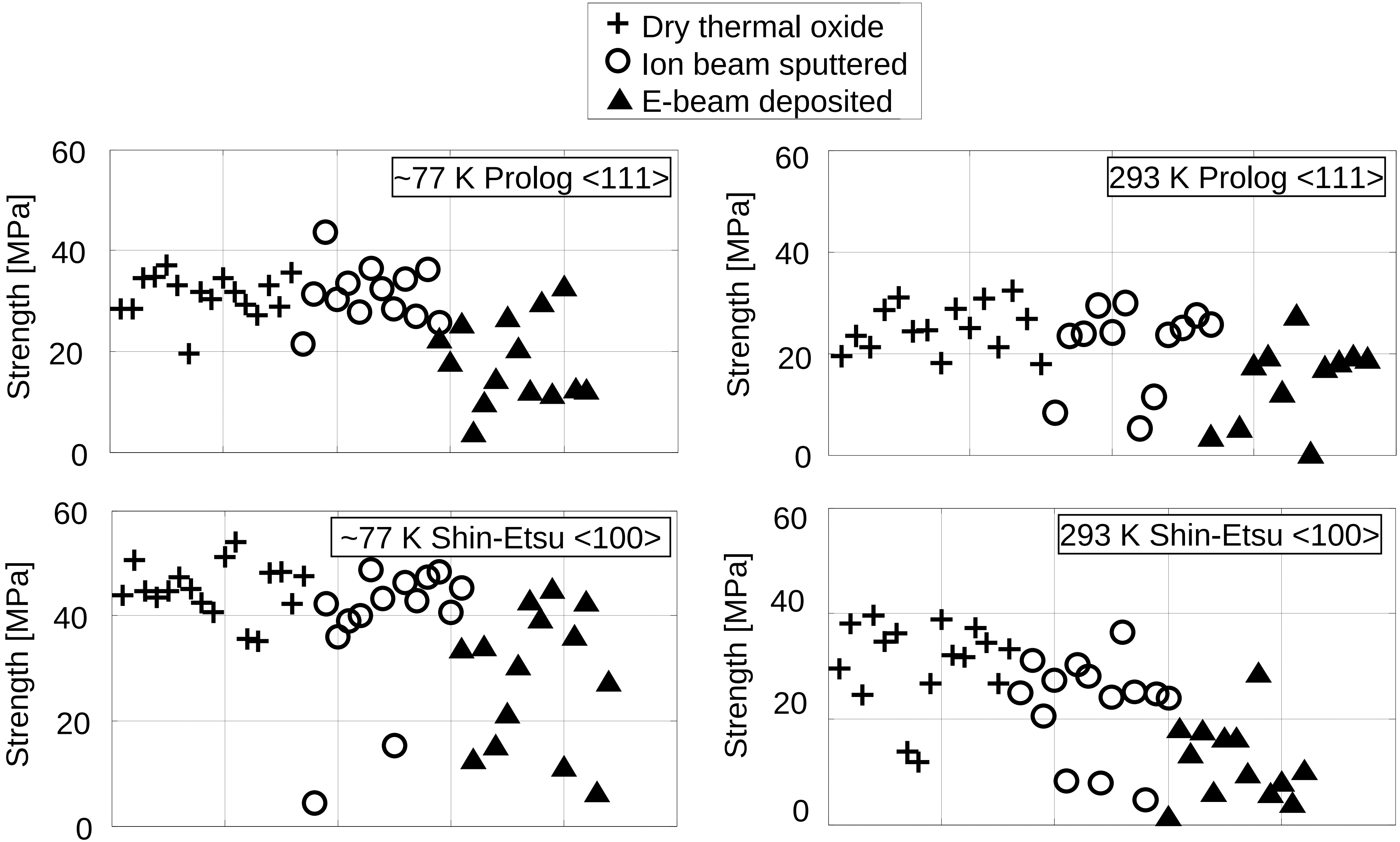
Bond strength silicon-silicon bonds

Influence of thickness of oxide layer on strength



Bond strength silicon-silicon bonds

Influence of type of thermal oxide layer

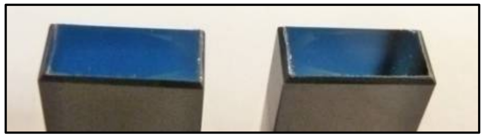


Bond strength silicon-silicon bonds

Influence of type of thermal oxide layer

- Temperature - CT results often stronger than RT
- Oxide layer type
  - E-beam results weakest
  - Thermal oxides strongest
  - Dry thermal oxide weaker but more reliable
- Ingot orientation - CT results always stronger for <100>

		% Bond Breaks CT	% Bond Breaks RT
Dry Ox	Shin-Etsu <100>	24%	19%
	Prolog <111>	88%	33%
Ion Beam	Shin-Etsu <100>	36%	36%
	Prolog <111>	77%	92%
E-Beam	Shin-Etsu <100>	57%	54%
	Prolog <111>	93%	72%



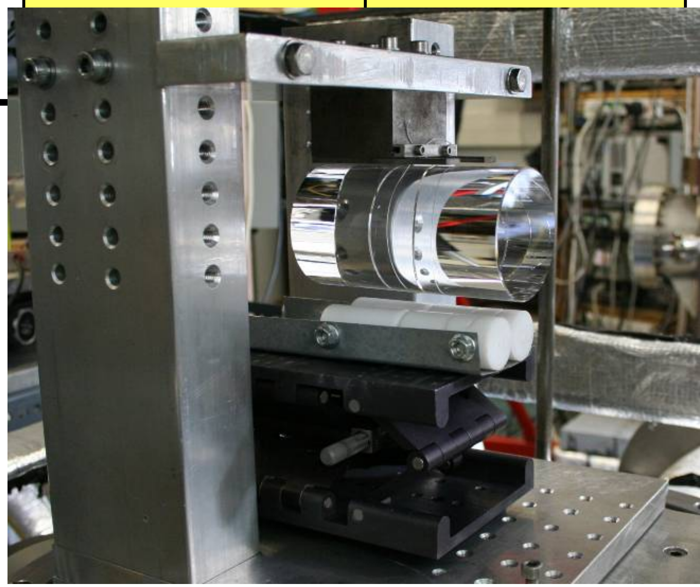
Hydroxide catalysis bonding for eaLIGO

Improving bond loss

Age bond	Temperature treatment	Average loss over 8 modes	Thermal noise aLIGO TM [m/√Hz]	Comments
5 months	Room temperature only	0.11 ± 0.02	$5.4 \cdot 10^{-22}$	(Cunningham et al., Physics Letters A 374 (2010) 3993–3998)
3 years	Room temperature only	0.08 ± 0.02	$4.6 \cdot 10^{-22}$	Thesis Karen Haughian, University of Glasgow, 2011
3 years	Room temperature for 3 years then 48 hrs at 150 ºC	0.06 ± 0.02	$4.0 \cdot 10^{-22}$	Thesis Karen Haughian, University of Glasgow, 2011

∅76 mm x 120 mm

Suprasil 311 cylinder



Loss results for indium thin film

Modes 2-9 measured

This is mode 9

Typical curve