Status of hydroxide catalysis and indium bonding research for use in cryogenic mirror suspensions in KAGRA, ET and upgrades to aLIGO

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Elites Meeting, 2-3 December 2015

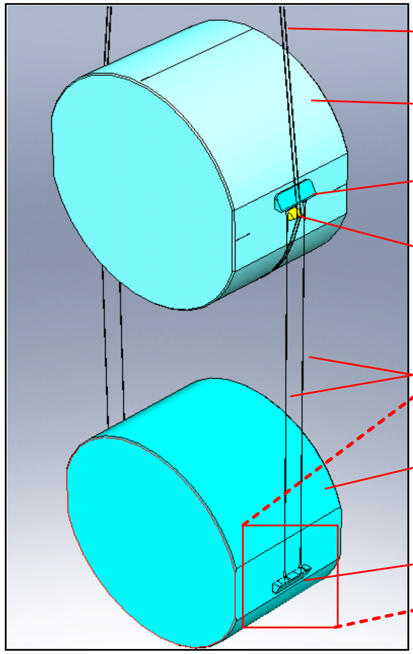
LIGO-G1501438

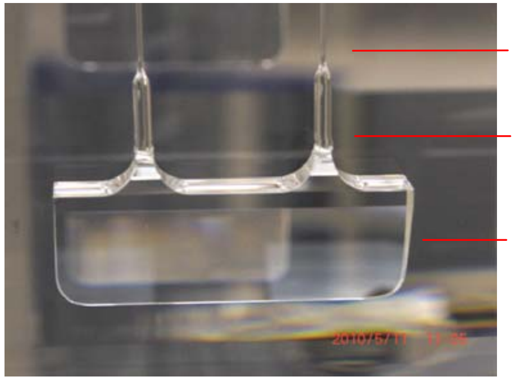
Introduction

- The mirror suspensions in ground based gravitational wave detectors need to have superior thermal noise performance.
- Apart from have sufficient strength, any joints used in these suspensions therefore also have to have extremely low contribution to the thermal noise.
- We – as gravitional wave community – are now working on/ talking about upgrades to detectors at room temperature (A+ and ET-HF) and various cryogenic temperatures (Voyager/Cosmic Explorer, ET-LF, KAGRA)
- In cryogenic suspensions joints also need to be able to maintain strength and noise performance down to cryogenic temperatures and have sufficiently high thermal conductivity to ensure efficient cooling of the mirrors.

Introduction to hydroxide catalysis bonding

- GEO600, aLIGO, and advanced VIRGO have quasi-monolithic test mass suspensions in fused silica which show superior thermal noise performance at room temperature
- Hydroxide catalysis bonding is used in all to attach some form of interface piece to the mirror to allow attachment of the fibres (which are welded)


Steel wires
Penultimate mass
Attachment or 'ear'
Steel wire break-off prism
Silica fibres
End/input test mass
Ear


Fibres
Weld horns
Ear

aLIGO monolithic mirror (ETM and ITM) suspension

Chemistry of hydroxide catalysis bonding

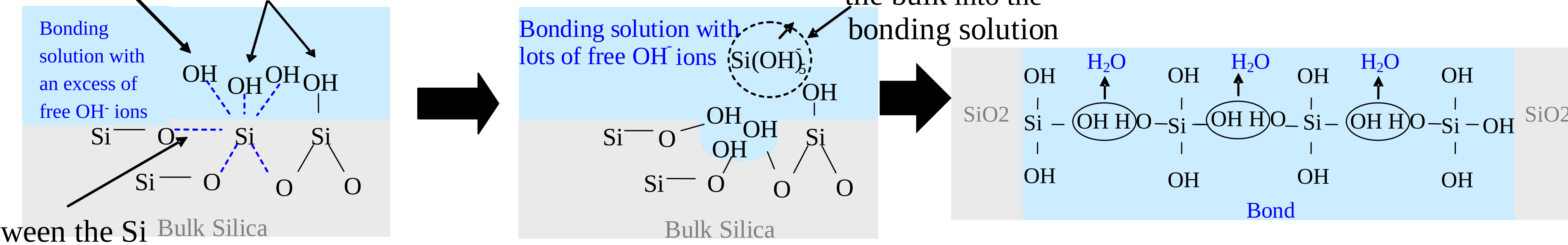
This method can create strong, durable bonds.

Chemistry of bonding between silica surfaces:

Hydration and Etching

Polymerisation

Dehydration



OH^- ions form weak bonds with surface Si atoms

OH^- ions from the bonding solution fill any open bonds on the surface

Bonds between the Si atoms and the bulk weaken due to the extra OH^- ions bonding to the Si atom

Silicate molecule being released from the bulk into the bonding solution

In aLIGO sodium silicate solution is used as the bonding solution. In advanced Virgo potassium hydroxide is used [van Veggel & Killow, Adv. Opt. Appl., 2014].

Material properties of Indium

Useful properties of indium	
Physical characteristics	Soft, malleable, easily fusible
Temperature	Low melt point 157°C
Thermal conductivity	as low as 2×10^{-3} at 20 K and 2×10^{-2} at 300 K (Hofmann et al CQG 2015)
Mechanical loss	Comparably low (Murray CQG, 2015)
UHV compatible	Yes
Bonds to	Itself, fused silica, quartz, metallic oxides.
Opportunity to debond?	Yes. Bonds can be broken and re-bonded without damage to substrates.

Characteristics led to the use of indium for many industrial uses, from electronic components to malleable UHV vacuum seals.

GW Detectors - Past

- Indium, welding, and hydroxide catalysis bonds were considered for GEO600 suspension bonds in 1998 (Sharon Twyford, Glasgow thesis 1998)
- Hydroxide catalysis selected instead, for overall strength and suitability at room temperature.
  - For room temperature detectors indium’s 156 °C melting point meant bonds could creep during vacuum bake outs.
  - HCBs are stronger than indium bonds.
- Suggested for use in cryogenic systems in past, and is active area for KAGRA.

GW detectors - Future

Benefits and drawbacks of indium bonding

Drawback	Property	Benefit
Could creep at high temperatures. Not suitable for vacuum baking	Low melting temperature	Bond formation at relatively low temperatures
Not suitable for high tensile stress applications	Bond strength lower than HCBs	Repairable bonds without interface damage. Suitable in compression.

Proposed for use in future cryogenic GW detectors.

- KAGRA design: Hybrid sapphire suspensions.
  - HCB for strength, indium for ease of repair. LIGO-G1500951
- ET Design: Silicon suspensions with proposed indium bonds on some interfaces. ET-0106C-10

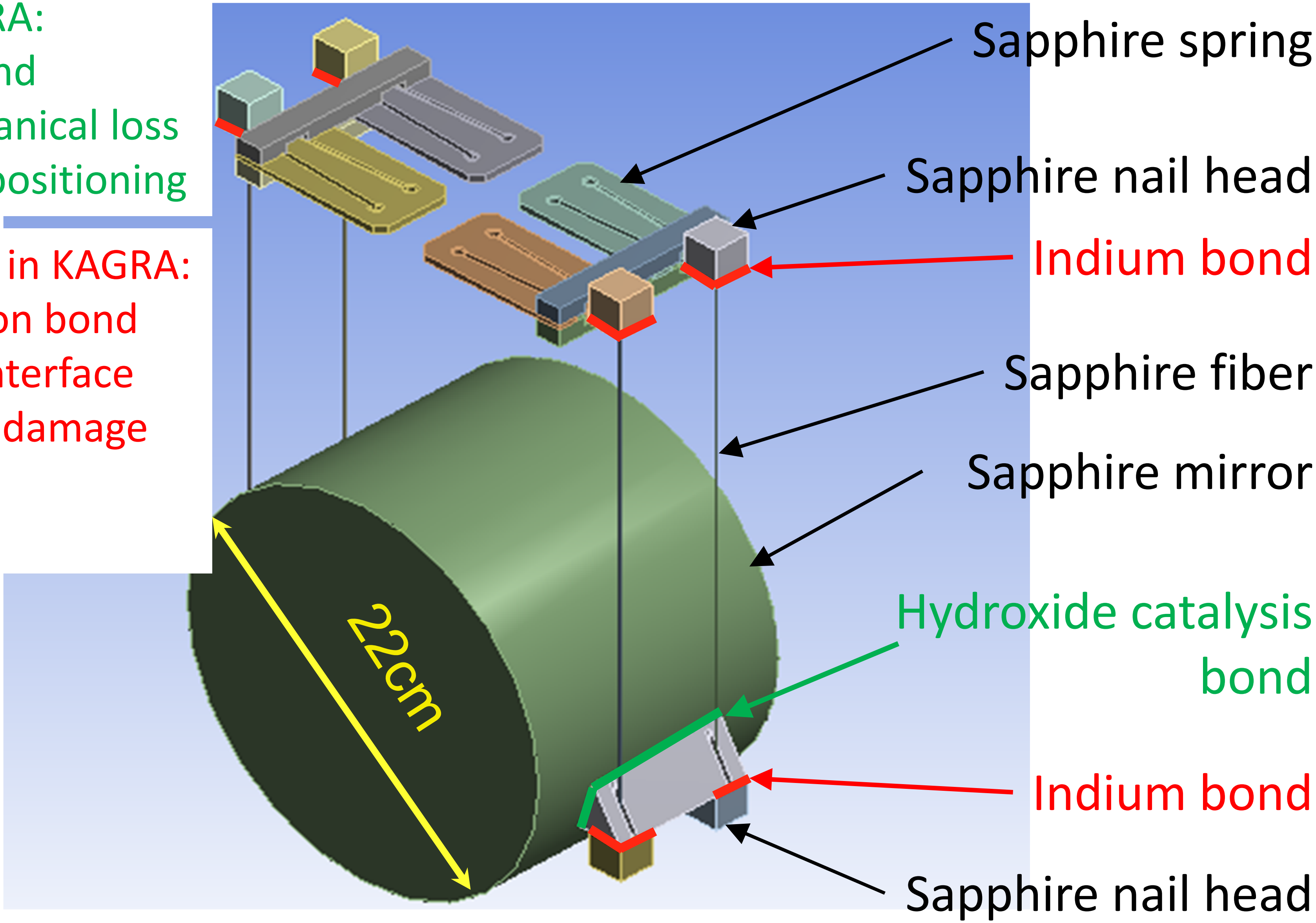
bKAGRA cryogenic suspension design

HCBs in KAGRA:

- Tensile bond
- Low mechanical loss
- Precision positioning

Indium Bonds in KAGRA:

- Compression bond
- Repair to interface
- Low risk of damage to optic or suspension



Sapphire spring

Sapphire nail head

Indium bond

Sapphire fiber

Sapphire mirror

Hydroxide catalysis bond

Indium bond

Sapphire nail head

From G1500951-Status of KAGRA

Cryogenic (crystalline) mirror suspensions

Name detector	ET-LF[1] (option 1)	ET-LF[1] (option 2)	Voyager[3] (option 1)	Voyager[3] (option 2)	KAGRA[4]
TM material	silicon	sapphire	silicon (with silicon ribbons)	silicon (with silica fibres)	sapphire
Mass TM [kg]	211	211	143	143	21
Diameter [mm]	500	500	560	560	220
Thickness [mm]	463	340	250	250	150
Beam radius [mm]	90	90	81	81	
Temperature TM [K]	10	10	123	123	20

[1] ET design document (ET-0106C-10)

[3] L1500077 (derived from blue team design)

[4] Yamamoto, ET meeting 2014

* Chosen to keep clipping losses the same as for aLIGO

Feasibility study: how?

- Can we bond sapphire and silicon using hydroxide catalysis bonding?
  - Yes, we can
  - Silicon surfaces must be oxidised to facilitate reliable bonding.
  - Chemistry is different for sapphire: aluminate chains.
- Are there other jointing techniques that may be suitable?
  - Indium bonding but only under compression and if suspension is not baked out.
- What is the strength of bonds in conditions that would occur in cryogenic suspension assembly and operation?

Feasibility study: how? Cont'd

- What stresses could occur in the bonds that would occur in cryogenic suspension assembly and operation?
  - Stress under the load of the suspension (mostly shear, some peel)
  - Stresses due to thermal expansion differences between the two parts bonded, if the thermal expansion of these parts doesn't match (Voyager (option 2), KAGRA, ET-LF (option 2), or a potential silicon/sapphire suspension)

- What is the thermal conductivity of these bonds?

- What is the thermal noise arising from bonds in cryogenic mirror suspensions?

HCB strength: what do we know?

Silicon

- In Glasgow we bond with sodium silicate solution
- Potassium hydroxide is also possible (Dari et al., CQG, 2010)
- Using a wet thermal oxide layer, we need 50 nm oxide layer to get reliably strong bonds (Beveridge et al., CQG, 2011)
- Strengths tested at R.T. and at 77 K
- Ion beam sputtered oxide layer is an excellent and probably more practical alternative (Beveridge et al., CQG, 2013)
- Average strength values in 20 – 40 MPa (depending on type of material, crystal orientation, temperature of test).
- Strength at 77 K higher than at R.T.
- Recent thermal cycling tests (3,10 and 20 times down to ~8 K) of <100>-<100> silicon (2 sets tested), suggest that the average stays constant (G1500837). We are developing techniques to assess the quality of bonds during the bonding procedure to ensure only high quality bonds are accepted.

HCB strength: what do we know?

Sapphire

- M-M sapphire can be bonded with sodium silicate solution (65 MPa), potassium hydroxide (>10 MPa), sodium hydroxide (>10 MPa) and sodium aluminate (>10 MPa). These were tensile tests. (Douglas et al., CQG, 2014)
- C-A (25 MPa), C-M (32 MPa) and C-C (50 MPa) bonds tested at 8 K. These were torsional shear tests. (Haughian et al., CQG, 2015)
- Sapphire doesn't break very often when a bond is broken and can be rebonded (be it with some loss of strength).
- Thermal cycling (M-M) suggests a drop in average strength (still > 5 MPa) experiments (K. Yamamoto, ET meeting 2014, Lyon). Not enough statistics though. Torsional shear tests.
- Curing time (C-C) at room temperature is not yet understood. Four weeks curing gives high strength (as with silica). However, 8 and 12 weeks curing seem to indicate a slight drop (See G1500837). Research ongoing to understand curing better. Samples for tensile tests expected before end 2015.
- Recent measurements have shown that when directly comparing C-C sapphire, to A-A and M-M doesn't show a significant difference in strength. Though fracture behaviour is different (see G1500837)

HCB strength: questions remaining?

Silicon

- What is the minimum oxide layer thickness for ion-beam sputtered coatings?
- Is chemical oxidation an option?
- What is causing difference in strength: crystal orientation or purity? Why?
- Can strength/mechanical loss be improved through heat/vacuum treatment?
- What is the curing time?
- How can we best to assess bond quality?

Sapphire

- What is the curing time?
- Influence of thermal expansion differences between different crystal orientations on bond strength when bonding e.g. c-a plane or c-m plane?
- Influence of polishing process/surface damage on strength.
- Literature study suggests surface reactivity higher for mechanically polished surfaces a.o.t. pristine surfaces
- Can strength/mechanical loss be improved through heat/vacuum treatment?
- Bond strength of sapphire not polished or cut on specific crystal plane?

Indium bonding in Glasgow

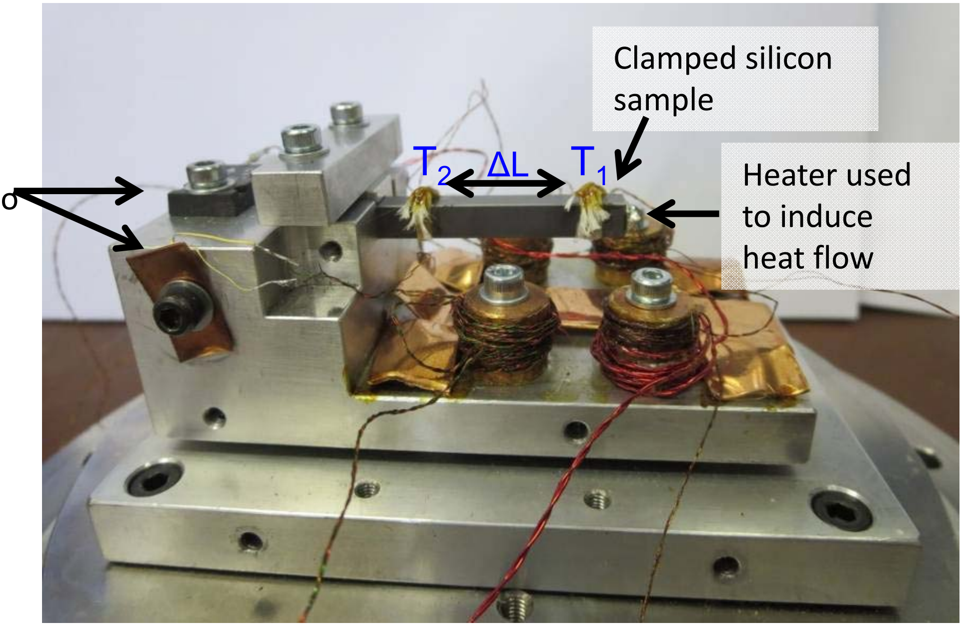
Creating a bond between silicon substrates using thermally evaporated indium (thickness ~ 500 nm):

- Benefit is that bonds are very thin; minimising thermal noise and maximising thermal conductance
- Efforts are focussed on assuring we deposit a highly pure indium layer.
- Experimenting with two approaches:
    1. Prevent oxide from forming with a vacuum and/or inert gas environment.
    2. Etch off oxide that has been formed using dilute hydrochloric acid.
  - ➢ Approach 2 is showing the most promising initial results; high tensile strengths and surface analysis after breaking showing good bond coverage
- Aim to statistically test strength and measure thermal conductivity

Thermal conductivity

- Thermal conductivity has now been measured of:

1. Silicon-silicon HCB bonded cylinders (∅25 mm x 76 mm) down to 30 K (Lorenzini, ET meeting Jena, 2010)
2. Silicon-silicon HCB bonded samples (5x5x40 mm) down to 8K
3. Sapphire-sapphire HCB bonded samples (Poster C. Schwarz, D. Chen, ET meeting 2014, Lyon).



Heater and temperature sensor used to thermally stabilise the clamp

Clamped silicon sample

Heater used to induce heat flow

- Results suggest drop in thermal conductivity is very small.

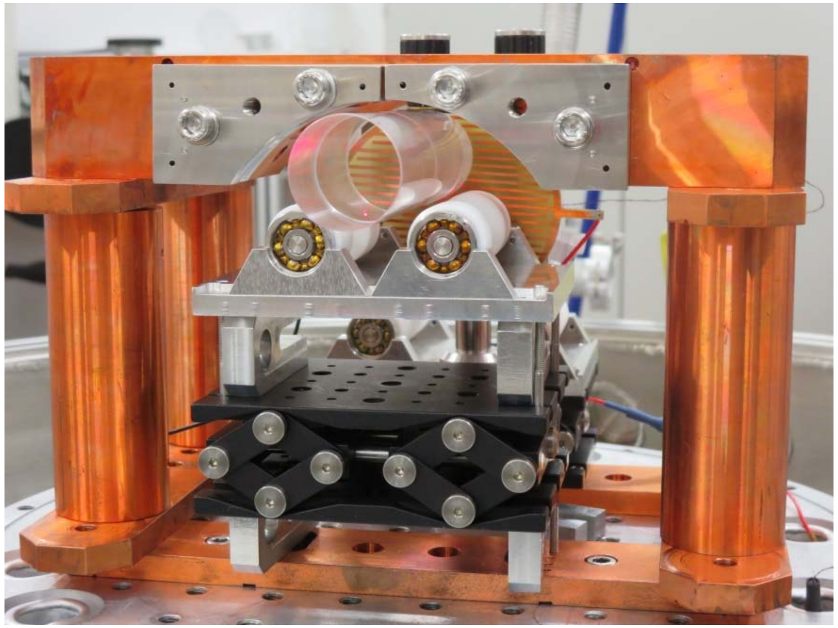
Heat extraction experiment has been carried out on a sapphire-sapphire indium bonded (using indium foil) sample (Hofmann, CQG, in press). Bond thickness of ~77 ±2 µm.

Bond mechanical loss, overview

- Silica-silica HCB bond made with sodium silicate solution @ R.T.
  Bond loss: 0.11 ± 0.02 (Cunningham, CQG, 2010)
- Silica-silica HCB bond (same as above) after 3 years and after heat treatment 150 °C for 48 hours. Bond loss: 0.06 ± 0.01 (Haughian, thesis, 2012)
- Silicon-silicon HCB bond made with sodium silicate solution @ R.T. Not an ideal bond (offset). Bond loss: 0.27-0.52 (Haughian, thesis, 2012)
- Sapphire-sapphire HCB made with sodium silicate solution a.f.o. temperature
  - Bond loss @ R.T. 0.03± 0.01
  - Bond loss @ 20 K $(3 - 7)\cdot 10^{-4} \pm 1\cdot 10^{-4}$

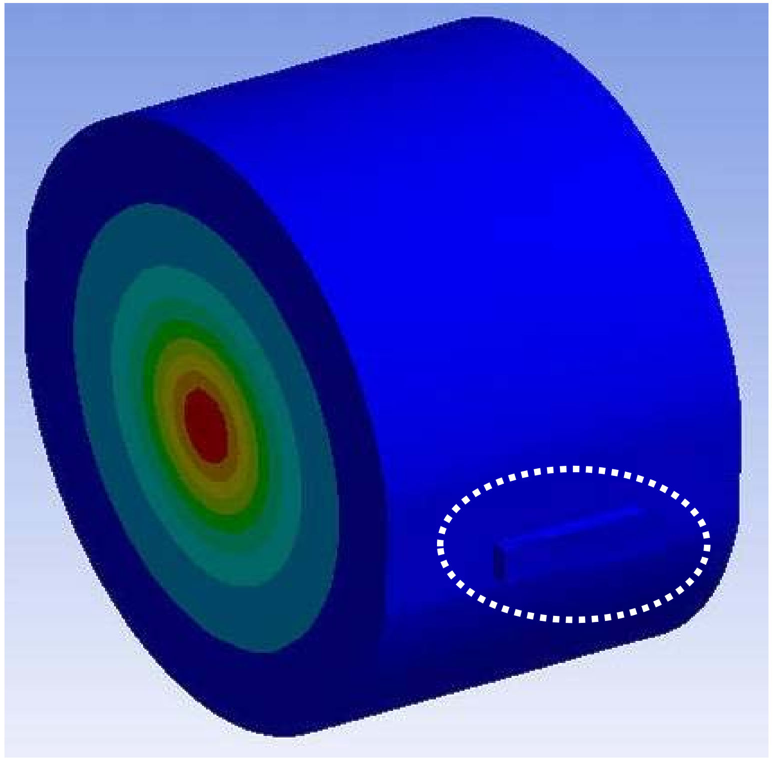
  (see G1500637)
- Sapphire-sapphire indium bond made with indium foil (~14 ±2 μm)
  - Bond loss @ 20 K $(2 – 3)\cdot 10^{-3}$

  (Hofmann et al., CQG, in press).



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

Aim: some initial idea of levels of thermal noise

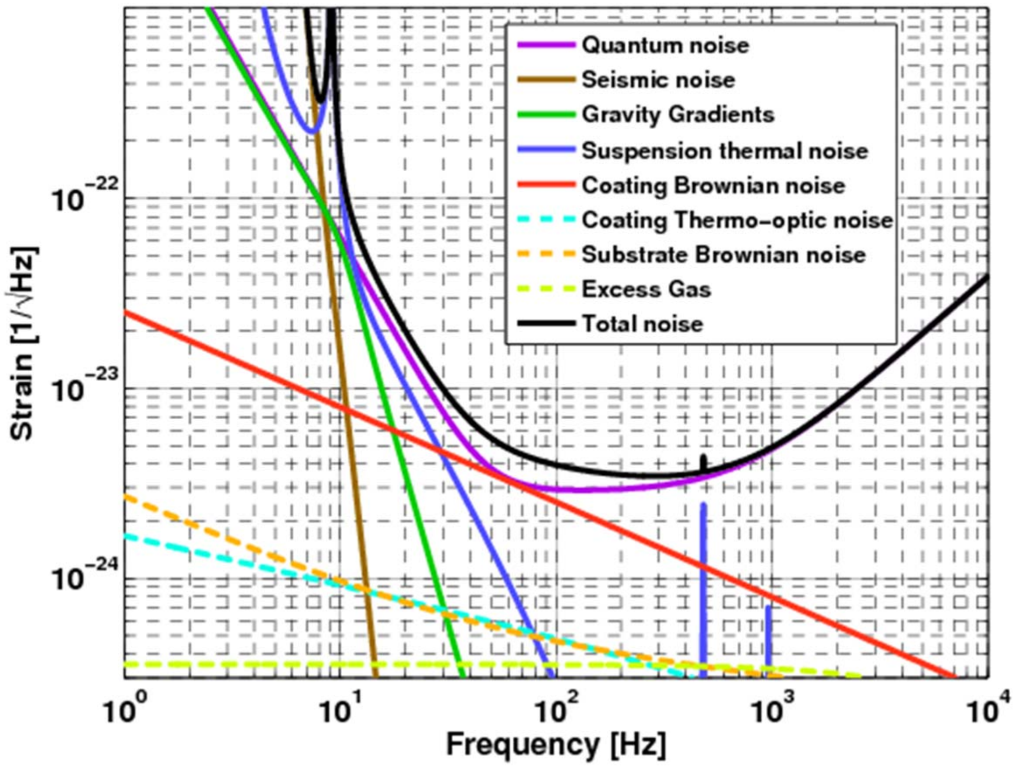


- FE models of aLIGO size test mass with aLIGO size ears
- A Gaussian pressure wave is applied to front surface which causes deformation of the bond to calculate strain energy in the bond
- Using Levin's method thermal noise associated with the strain energy at 100 Hz is calculated.

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

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

Thermal noise associated with HCBs in aLIGO



Hild, S., Class.Quant.Grav. 29 (2012) 124006

In aLIGO thermal noise arising from bonds was to be 10% of the total thermal noise budget @ 100 Hz

Thermal noise budget @ 100 Hz is $\sim 2\cdot 10^{-24}$ /√Hz ⇒

Bond thermal noise budget per test mass $\sim 2\cdot 10^{-25}$ /√Hz ($7\cdot 10^{-22}$ m/√Hz, (T010075-01,2009))

Calculated: $1.4\cdot 10^{-25}$ /√Hz ($5.4\cdot 10^{-22}$ m/√Hz)

assuming bond loss of 0.11 at 293 K

Assumptions for calculations following:

- bond thermal noise budget < 10% of overall thermal noise budget @ 100 Hz
- Ear size (where unknown): same ratios and average shear stress (0.17 MPa) as aLIGO.

Thermal noise from bonds for cryogenic

Name detector	ET-LF[1] (option 1)	ET-LF[1] (option 2)	Voyager[3] (option 1)	Voyager[3] (option 2)
TM material	silicon	sapphire	silicon (with silicon ribbons)	silicon (with silica fibres)
Temperature TM [K]	10	10	123	123
Bond area 1 ear or nail head [mm x mm]	45x136	45x136	52x157	52x157
Assumed bond thermal noise requirement [1/√Hz]	$4\cdot10^{-26}$	$4\cdot10^{-26}$	$3\cdot10^{-26}$	$3\cdot10^{-26}$
Bond loss [-]	**$7\cdot10^{-4}$	*$7\cdot10^{-4}$	**$7\cdot10^{-4}$	**$7\cdot10^{-4}$
Calculated expected thermal noise (±15%) [1/√Hz]	$2.2\cdot10^{-27}$	$1.1\cdot10^{-27}$	$1.4\cdot10^{-26}$	$5.8\cdot10^{-27}$

* Measured value HCB between sapphire

** Assumed value (bond loss sapphire-sapphire bond @ 10K)

Calculated expected thermal noise KAGRA from indium bonds $5\cdot10^{-25}$ /√Hz (total thermal noise requirement $5\cdot10^{-24}$ / √Hz)
[Hofmann, CQG, 2015, in press]

Summary of HCB work

- HCB between silicon and sapphire have been extensively demonstrated to be strong enough for typical gravitational wave detector suspensions.
  - ⇒ Work on curing time, thermal cycling, cross-axis bonding, etc. is ongoing to refine knowledge of chemistry and strength.
- Thermal conductivity measurements of HCB and indium bonds appear to be high enough for suspension attachments.
- The mechanical loss of a HCB between sapphire samples is ~0.03 at room temperature and is ~(3 - 7) x 10^{-4} at 20 K. An upper limit of the mechanical loss of a HCB between silicon has been measured at RT.
- Bonding mixed materials or cross-axis sapphire may cause very high shear stresses in the bonds. This could cause damage to bonds and substrate material. Further investigation needed.
- Thermal noise calculations of sapphire and silicon suspensions, assuming a similar suspension design as aLIGO, show that expected bond thermal noise at cryogenic temperature is below the expected requirement.

Thank you for your attention!