University of Glasgow

Studies of bond loss between silicon surfaces

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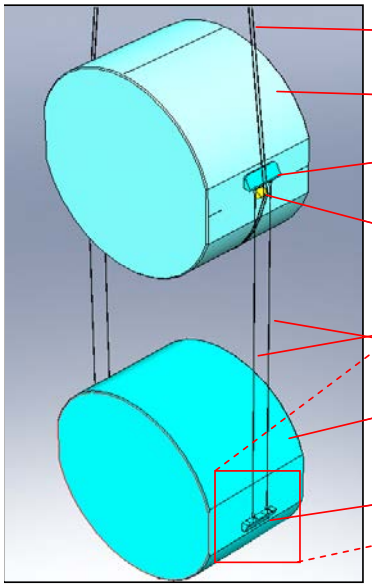
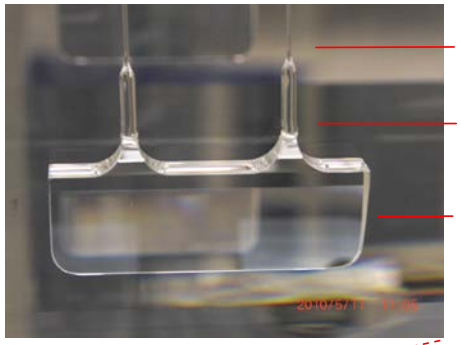
Introduction

- **Currently planned detectors will be limited in sensitivity by thermal noise.**

One possible way to reduce thermal noise is to cool test masses and suspensions (of appropriate materials). Silicon is one proposed material for future detectors

- **Hydroxy-catalysis bonding can be used (GEO, aLIGO) to joint silica to form quasi-monolithic suspension.**
- **Studies of its properties required to assess feasibility for use in cooled silicon suspensions**





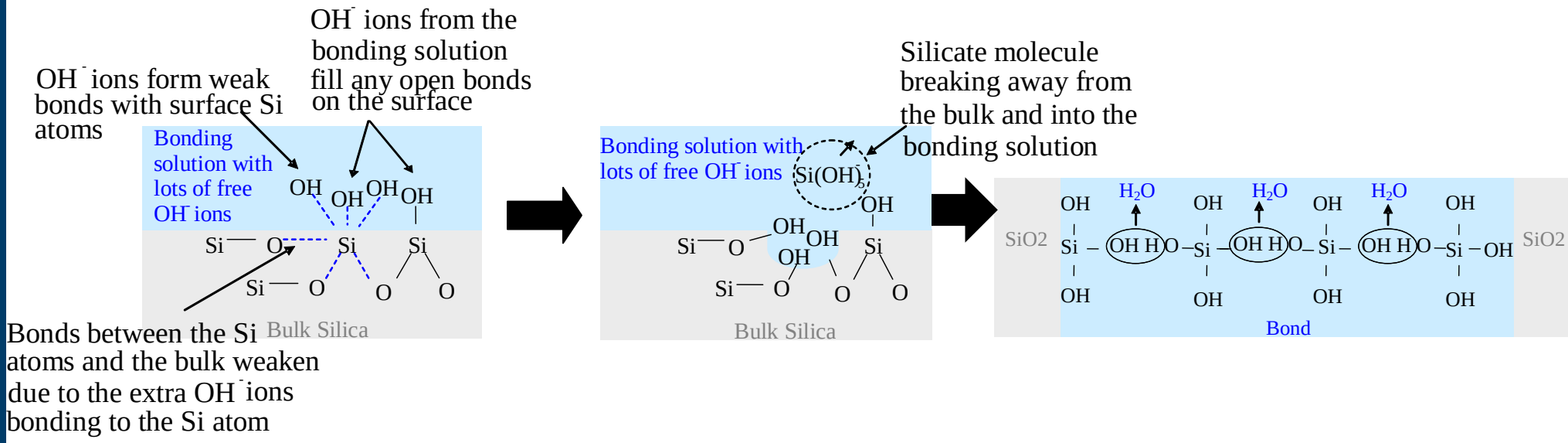
Hydroxy-catalysis bonding - chemistry

Hydroxy-catalysis bonding is used to create the quasi-monolithic fused silica suspensions for 2nd generation detectors.

This method can create strong, durable, bonds

Chemistry of bonding between silica surfaces:

- Hydration and etching
- Polymerisation
- Dehydration

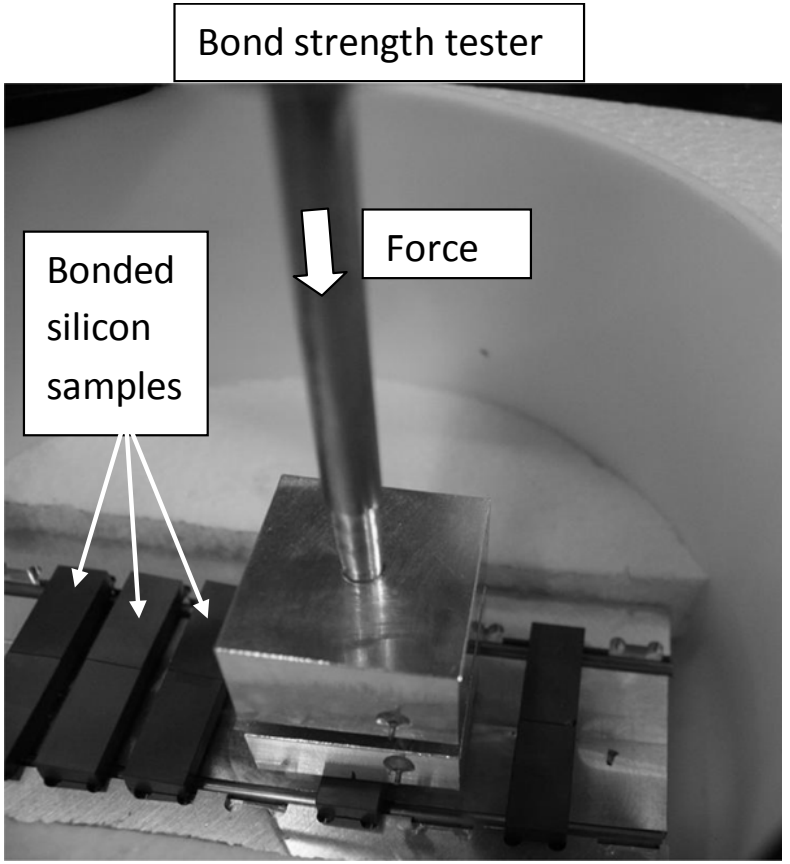


Introduction

Silicon surfaces must be oxidised to facilitate reliable bonding.

Experiments have shown:

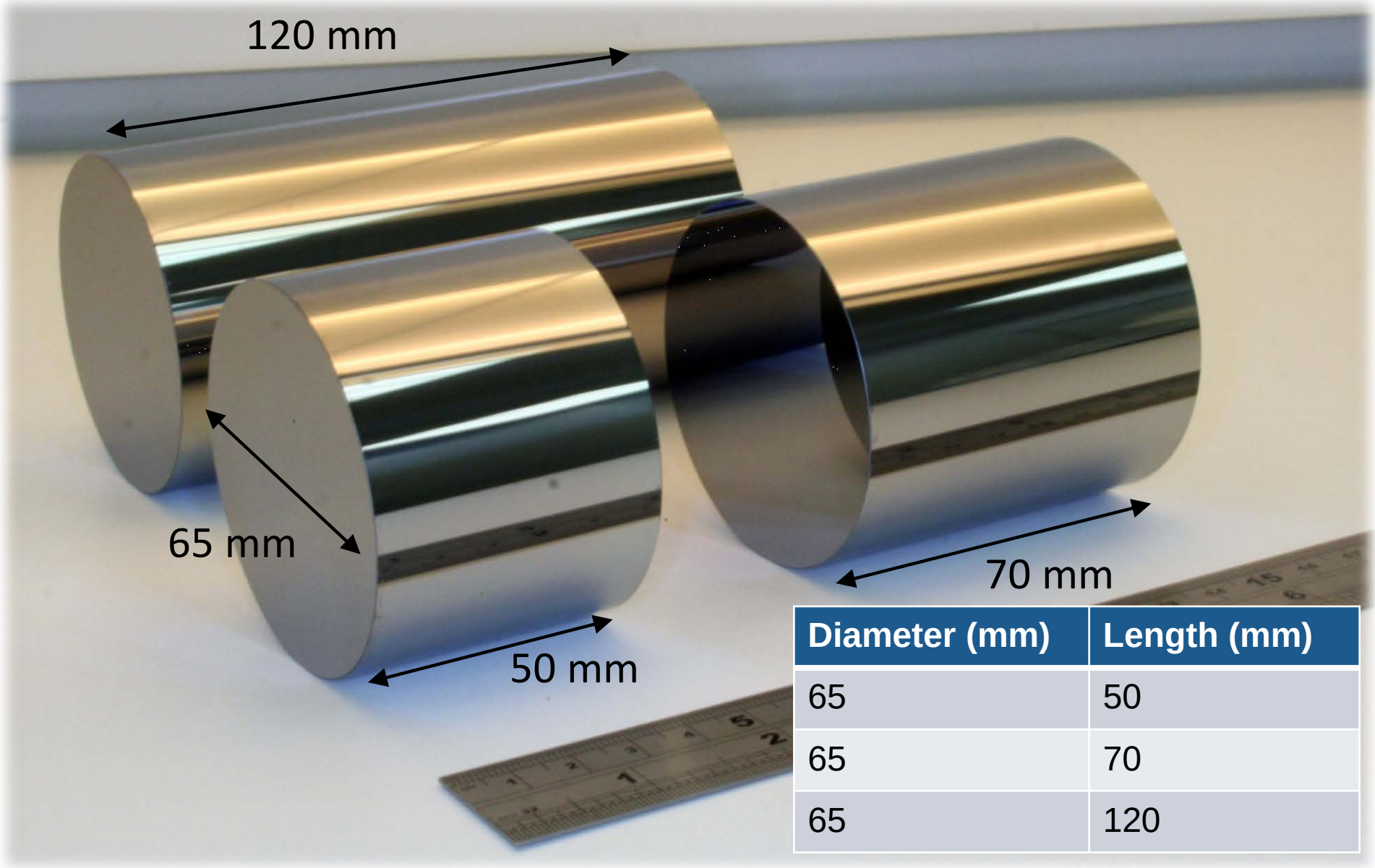
- **The minimum oxide thickness which allows reliably strong bonding found to be ~50 nm[1].**
- **The average strength of bonded silicon is ~36 Mpa[1]**
- **The average strength of bonded silica is ~15 Mpa.**
- **Previous measurements of silicon bond loss have been dominated by effects of thermoelastic loss at room temperature[2].**



Accuracy of the bond loss value may be improved by studies of bulk bonded silicon.

1. N. Beveridge, Low-temperature strength tests and SEM imaging of hydroxide catalysis bonds in silicon, Class. Quantum Grav. **28 (2011)**
2. E. Chalkley. Investigations of the Properties of Materials for the Optics and Suspensions of Future Gravitational Wave Detectors. PhD thesis, University of Glasgow, 2010.

Silicon (100) samples



Diameter (mm)	Length (mm)
65	50
65	70
65	120

Mechanical losses in the bonded system

$$\phi_{bonded} \cong \frac{E_{substrate}}{E_{total}}\phi_{substrate} + \frac{E_{bond}}{E_{total}}\phi_{bond} + \frac{E_{oxide}}{E_{total}}\phi_{oxide}$$

Measured loss of the bonded sample

Measured loss of the reference sample

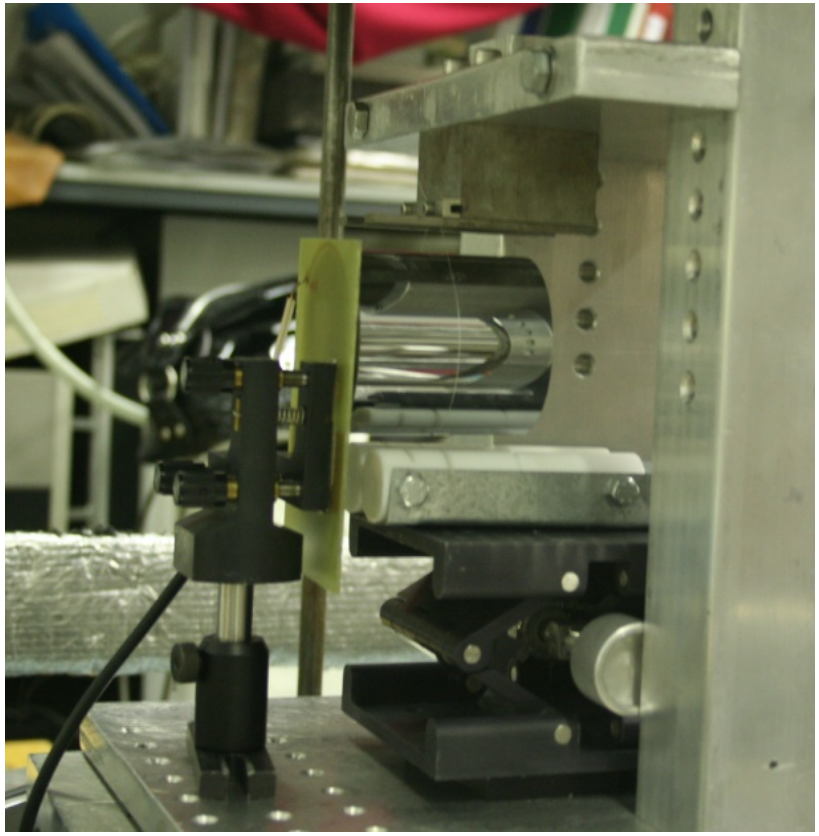
Calculated from a semi-empirical model[3]

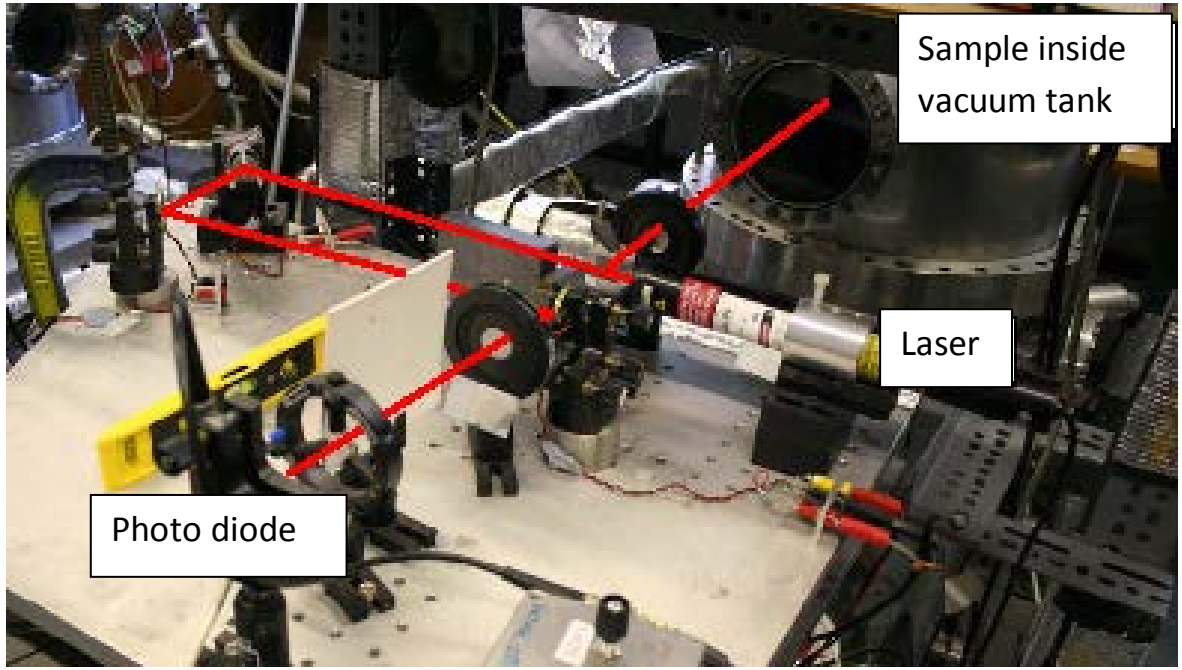
$$\phi_{bond} \approx \frac{\phi_{bonded} - \phi_{substrate} - \frac{E_{oxide}}{E_{total}}\phi_{oxide}}{\frac{E_{bond}}{E_{total}}}$$

Calculated using ANSYS® finite element modelling

3. Penn et al. Frequency and surface dependence of the mechanical loss in fused silica, Physics Letters A, 352:3{6, 2006

Mechanical Loss Measurements





$$A(t) = A_0 e^{-\frac{\omega_0 t}{2/\phi(\omega_0)}}$$

Resonant frequency

Amplitude after time, t

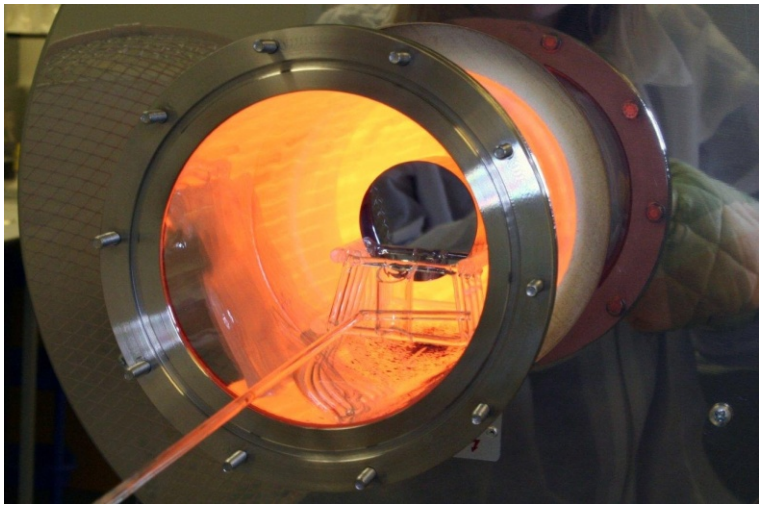
Initial amplitude

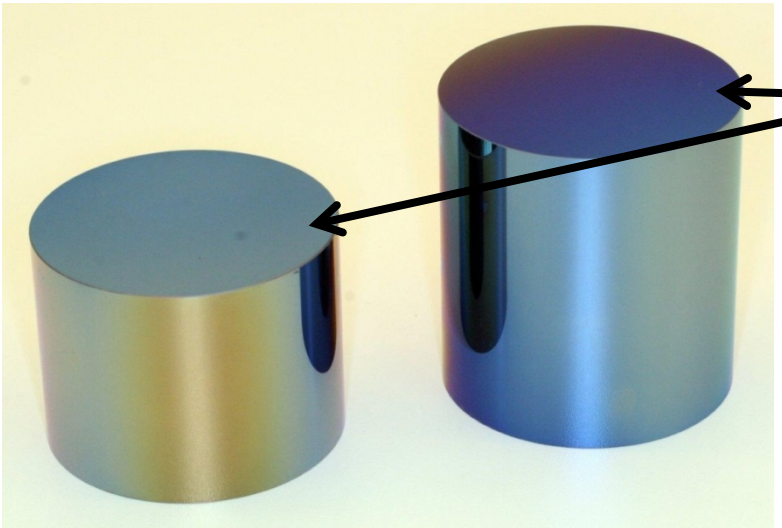
Loss at resonant frequency

Oxidisation process

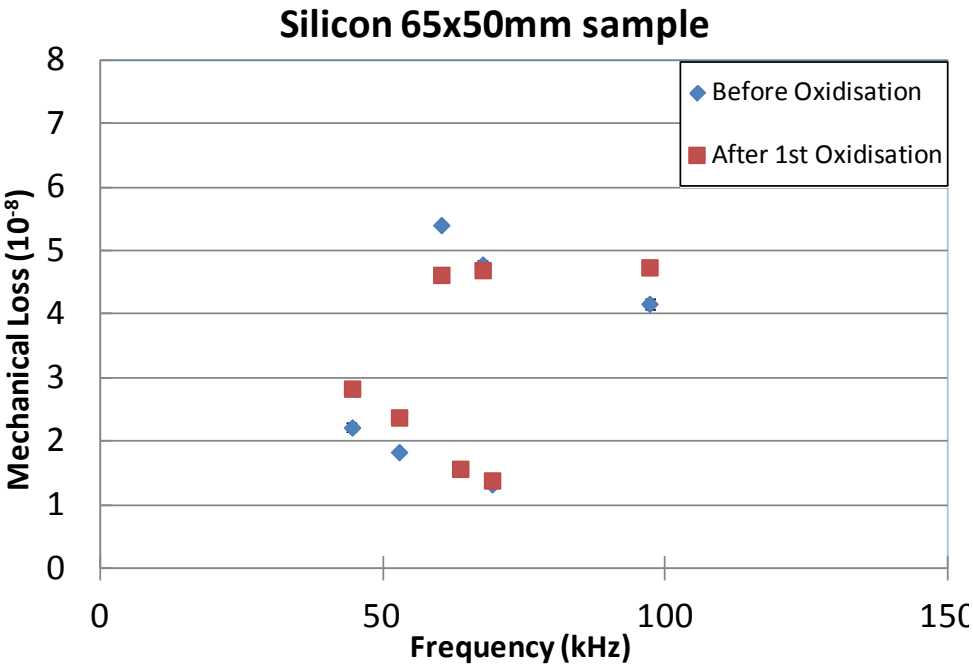
Wet thermal oxidisation:

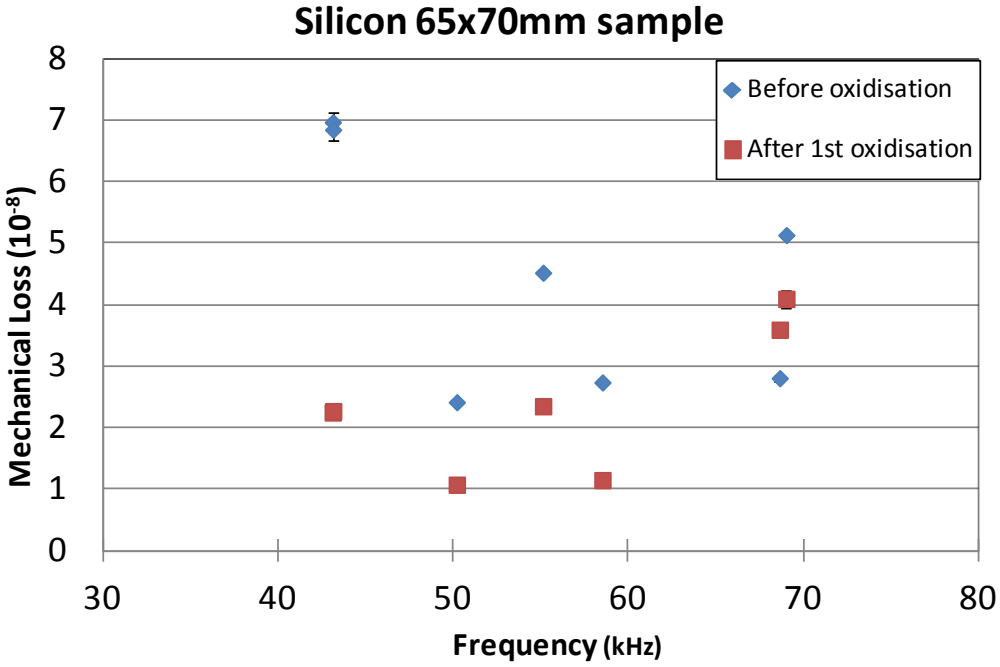
N_2 environment for 45 minutes at 1000ºC





Measured losses before and after oxidisation





For 70mm long sample most losses decreased after the oxidisation process.
Possible reasons:

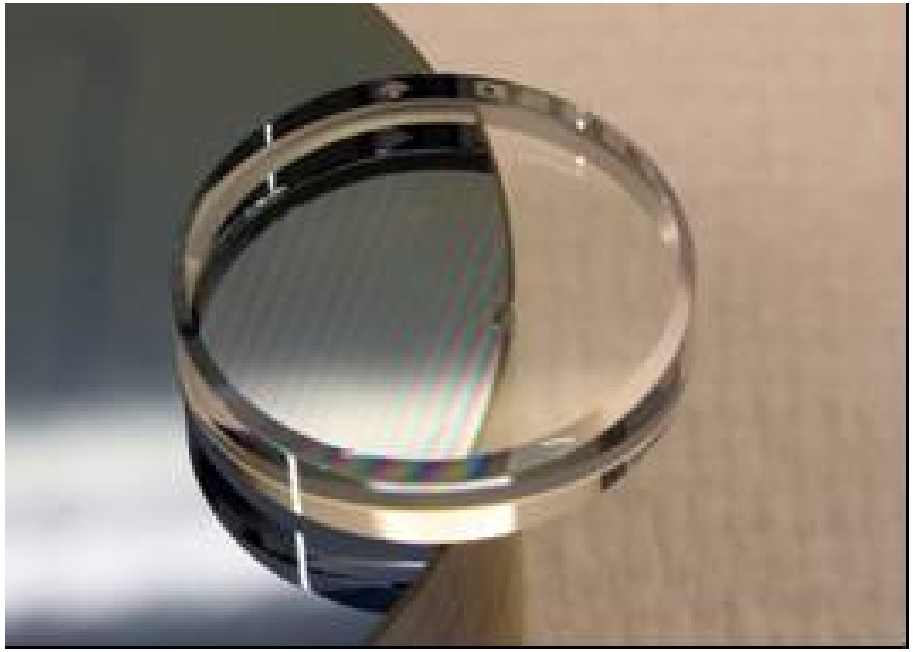
- **Heat treatment has reduced bulk loss**
- **Decreased suspension loss**

Lowest loss of the 70 mm sample reached the same level as the lowest loss of the 50 mm sample, ~1 × 10^{-8}.

Slight damage observed at edge of sample

Small raised area appeared on the bonding surface ~1000 nm in height.

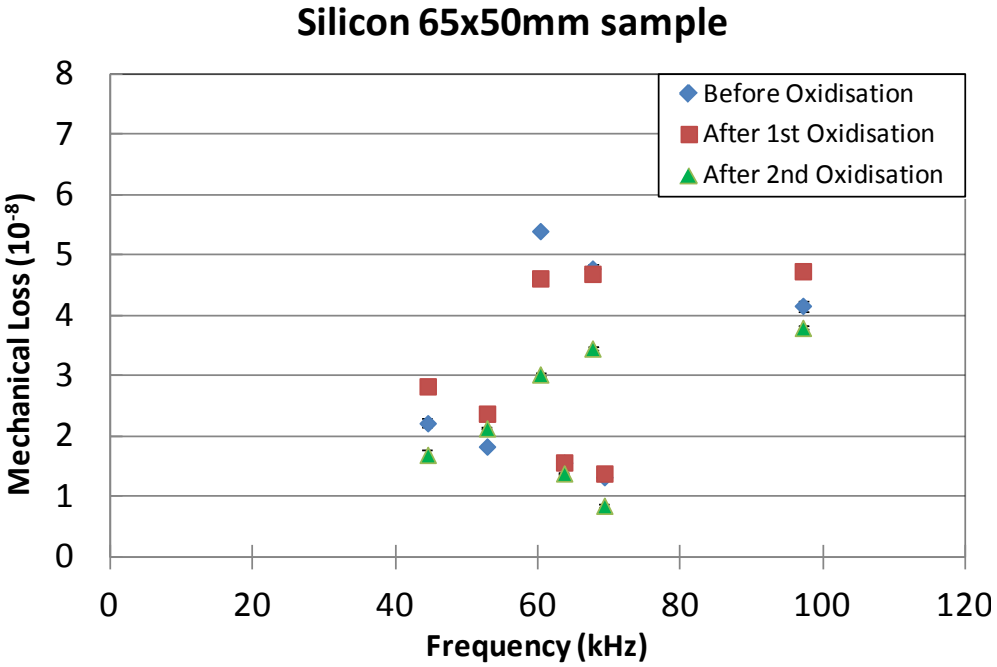
Etched the sample in hydrofluoric acid to remove oxide layer but the raised area remained, indicating that it was silicon.

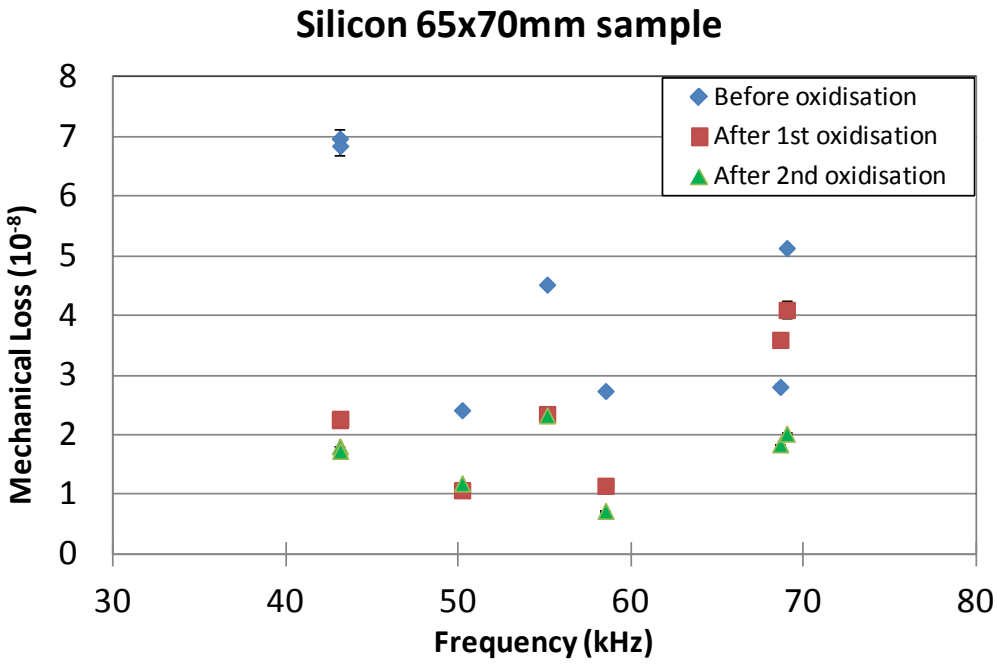


Placed a flat on the sample which chipped most of the raised area off, ~300 nm remained.

<u>Propose:</u>
Re-oxidise the samples and then bond with an offset of ~ 1-2 mm.

Measured losses after 2nd oxidisation step





After 2nd oxidisation step the losses in general decreased further for –both- samples.
Lowest loss ~ 9 × 10^{-9}

The reference sample was then used to investigate whether this is a bulk effect or is due to decreased suspension loss.

Reference sample

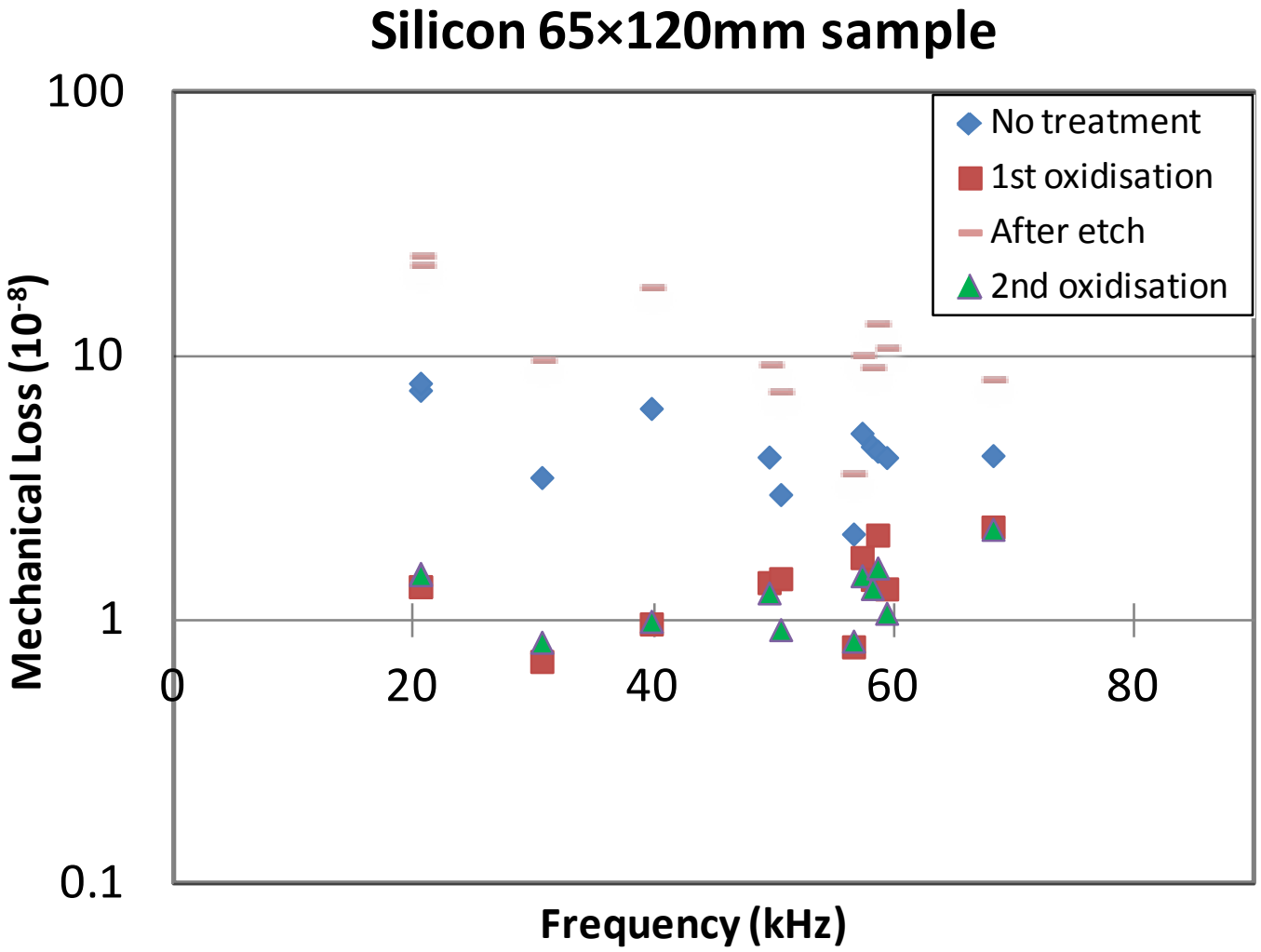
The reference sample was oxidised and etched to remove the top, oxidised surface layer.

After etching the loss increased to a level higher than it was before oxidisation.

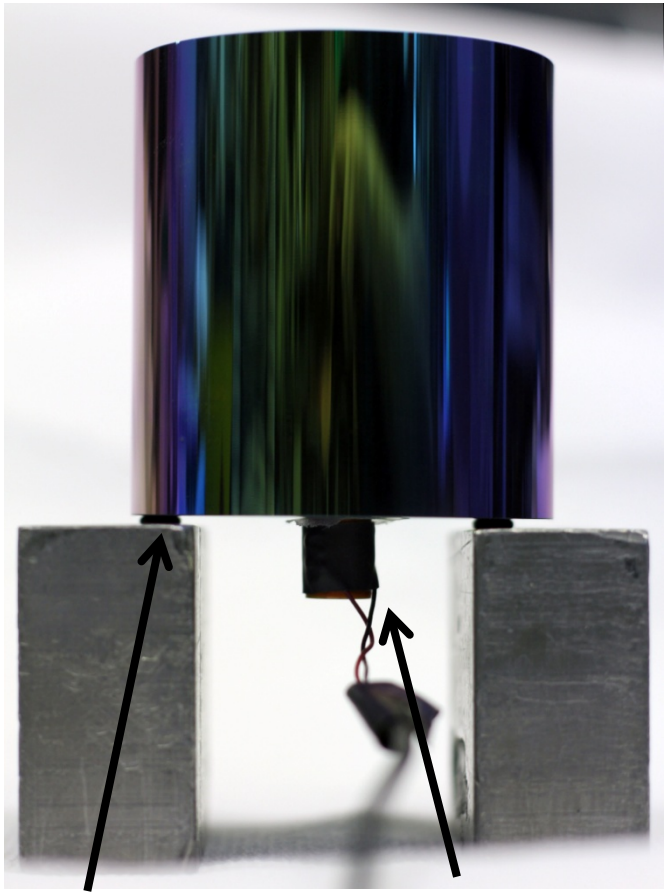
Possible reason:

- Increased surface roughness of the sample causing increased suspension losses?

Further investigations will be carried out.

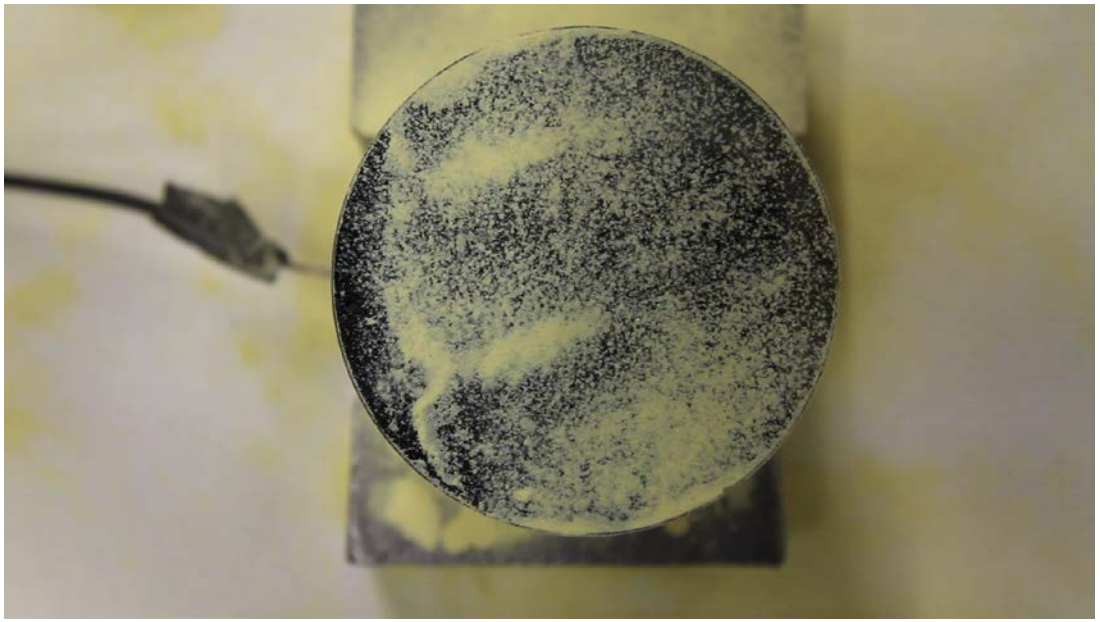


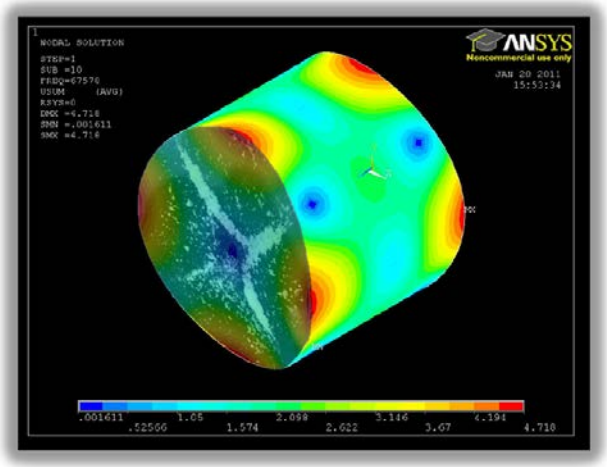
Mode shape identification



Small rubber washers

Piezo connected to a signal generator

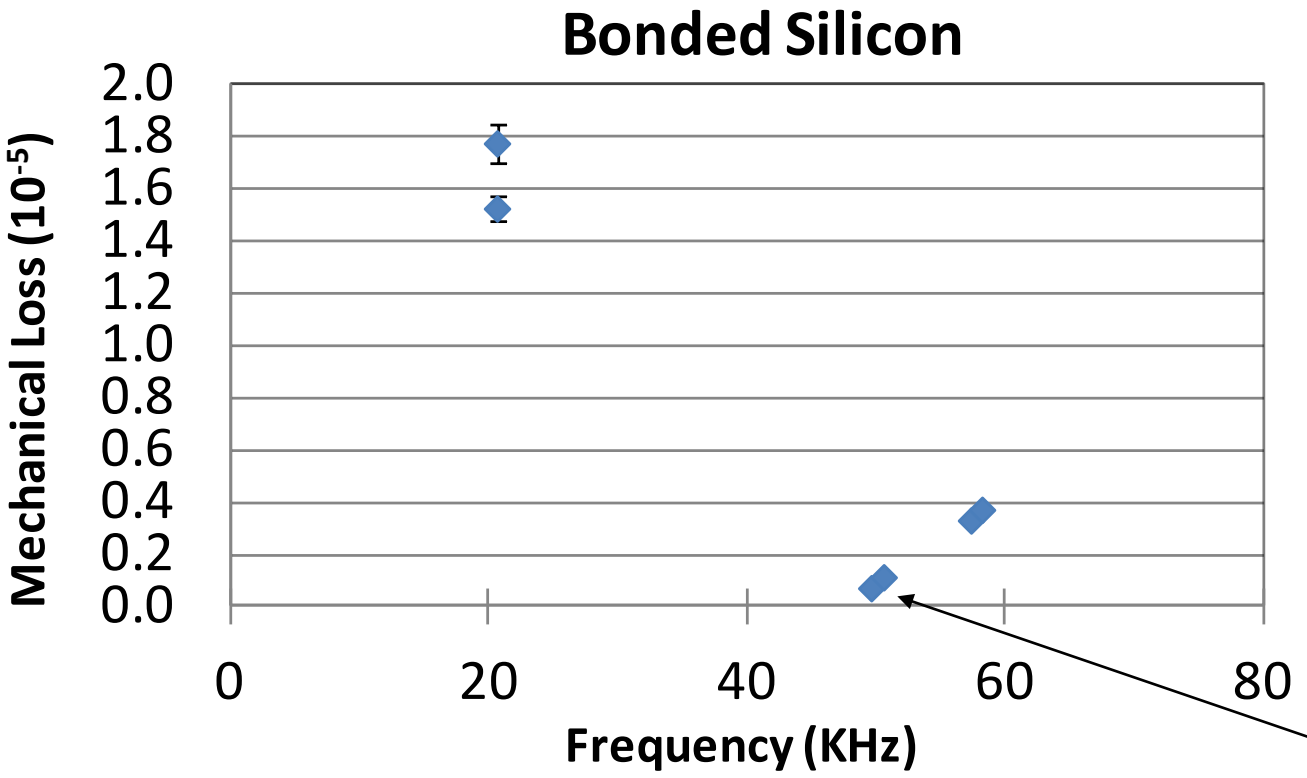




Bonded Samples

Hydroxy-Catalysis Bond:

- **Volume of solution used: 0.4 µl/cm^2**
- **Sodium silicate bond - Bonding solution: 14% sodium silicate, 27% silicate and 59% De-ionised water**
- **1:6 ratio of volume of bonding solution to DI water**



Lowest loss ~ 7 × 10^{-7}

Mechanical losses in the bonded system

$$\phi_{bonded} \cong \frac{E_{substrate}}{E_{total}}\phi_{substrate} + \frac{E_{bond}}{E_{total}}\phi_{bond} + \frac{E_{oxide}}{E_{total}}\phi_{oxide}$$

Measured loss of the bonded sample

Measured loss of the reference sample

Calculated from a semi-empirical model[3]

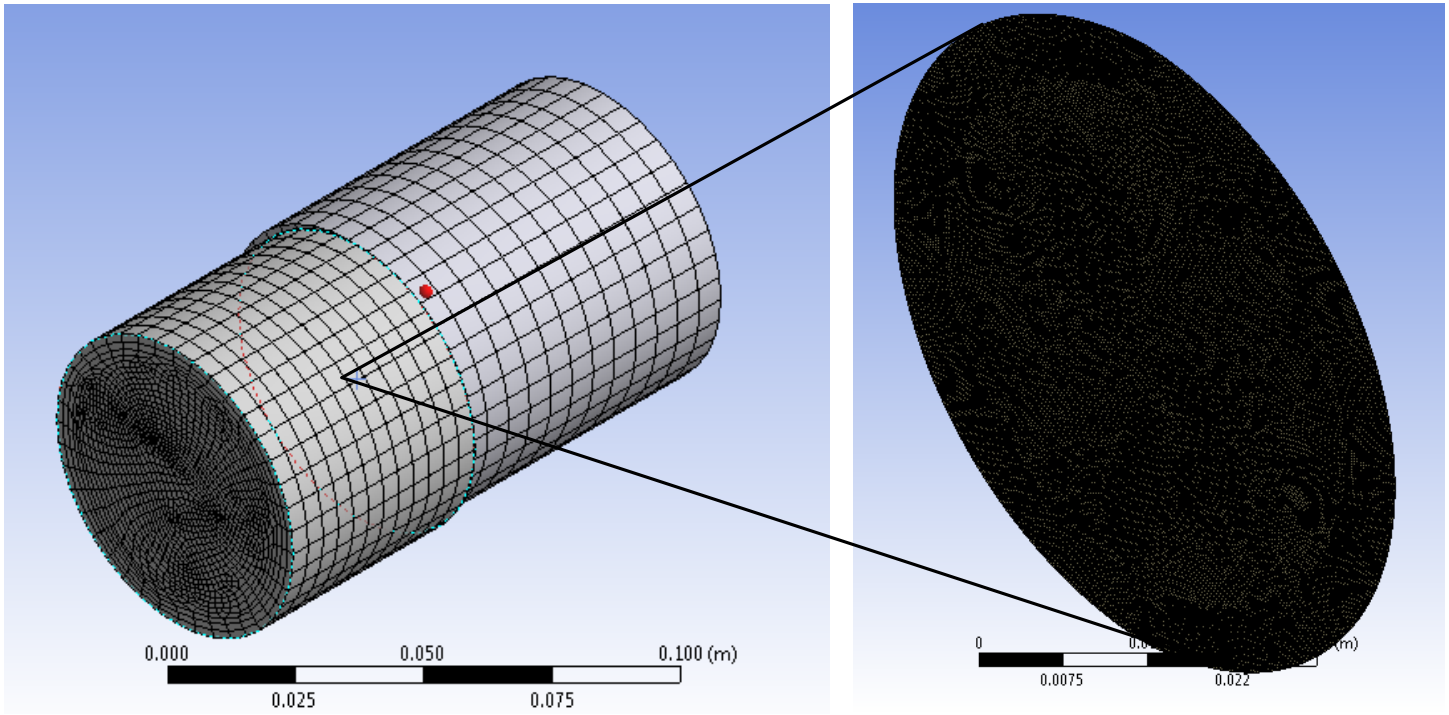
(need thickness of oxide layers and of bond)

$$\phi_{bond} \approx \frac{\phi_{bonded} - \phi_{substrate} - \frac{E_{oxide}}{E_{total}}\phi_{oxide}}{\frac{E_{bond}}{E_{total}}}$$

Calculated using ANSYS® finite element modelling

3. Penn et al. Frequency and surface dependence of the mechanical loss in fused silica, Physics Letters A, 352:3{6, 2006

Finite Element Modelling



Very dense mesh used for the bond and oxide layers

1) Oxide layer thickness was measured using an ellipsometer:

- **50 mm sample = 171 ± 0.5 nm; 70 mm sample = 317 ± 0.5 nm**

•2) Flatness of the two bonding surfaces was measured using a ZYGO interferometer.

A range of possible bond thickness values was estimated from this to be ~118-237 nm.

Bond loss result

The upper limit of the mechanical loss of a hydroxy-catalysis bond created between silicon substrates is within the range 0.21 - 0.64.

The lowest bond loss compares with limits for bonds between fused silica of 0.06.

Resonant Frequency (Hz)	Bond loss assuming 118nm bond thickness	Bond loss assuming 237nm bond thickness
20,512	0.44	0.22
20,536	0.43	0.21
49,530	0.17	0.09
50,486	0.37	0.18
57,229	0.85	0.45
58,084	0.89	0.47
Average	**0.52 ± 0.12**	**0.27 ± 0.06**

Our experiments have shown that the mechanical loss of bonds between silica substrates can be reduced through heat treatment:

Heat treatment: 48 hours at 150ºC

- **Reduces bond loss from 0.11 ± 0.02 to 0.06 ± 0.01.**
- **Increase bond strength from 12 ± 2 MPa to 22 ± 1 MPa.**

Investigation of the effect of this treatment on silicon bond loss will be carried out.

Next Steps

Aim: Bond loss measurements of silicon at room and cryogenic temperatures (with (111) crystal orientation)

Status: The mechanical loss of samples with the same geometry but with the crystal orientation (111) have been measured at room and cryogenic temperatures.

These loss measurements will be discussed in the next talk

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Thank you for your attention!

Heat treatment of bonded silica