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Experimental Results on Cryogenic Strength Testing Silicon

Nicola Beveridge

on behalf of the Institute for Gravitational Research, University of Glasgow

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Silicon Bond Loss Experiment

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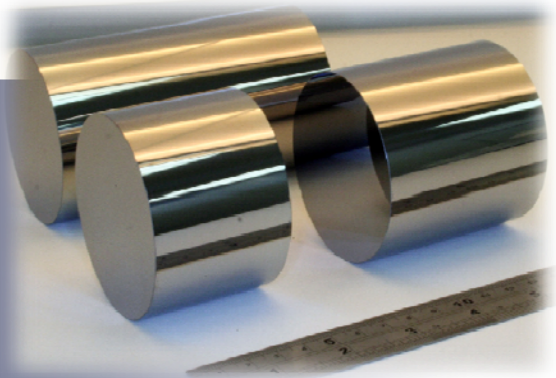
Silicon Bond Loss Experiment



- Preliminary measurements of the mechanical loss factors of hydroxide-catalysis bonds on silicon have been made
  - Two silicon (100) substrates were coaxially bonded [Ø65×50mm & Ø65×70mm]
- Comparisons made of the loss factors of the bonded sample with that of a 120 mm long witness sample
- In order to bond silicon components using hydroxide catalysis, bonding surfaces require SiO2 layer
  - Oxide layer formed by thermal oxidation in a quartz tube furnace at 1000°C in a wet N_2 environment for 45 mins

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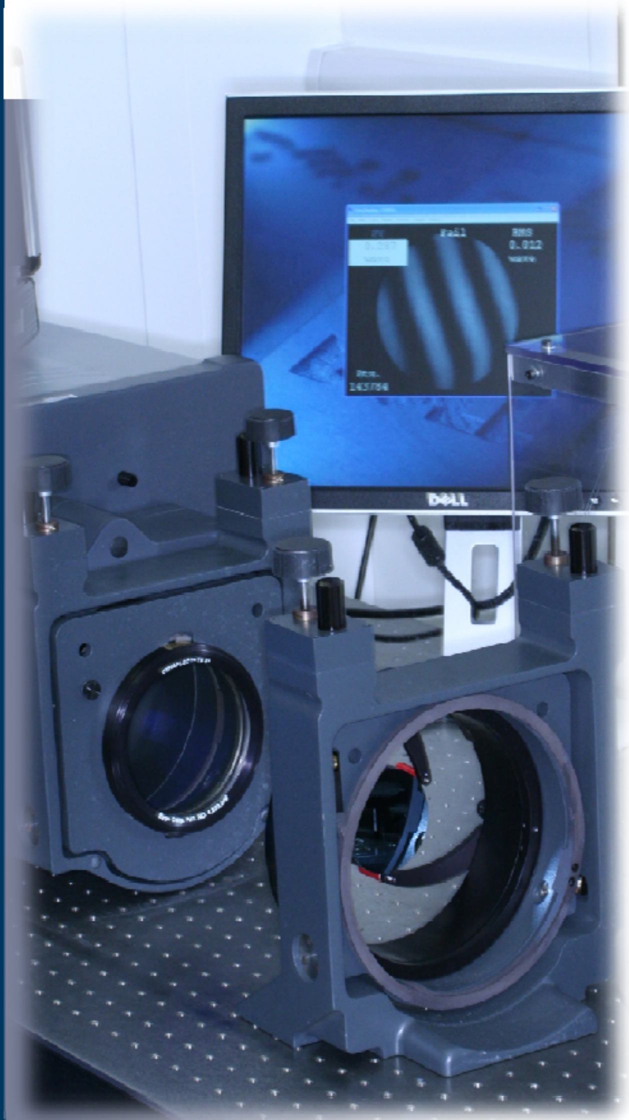
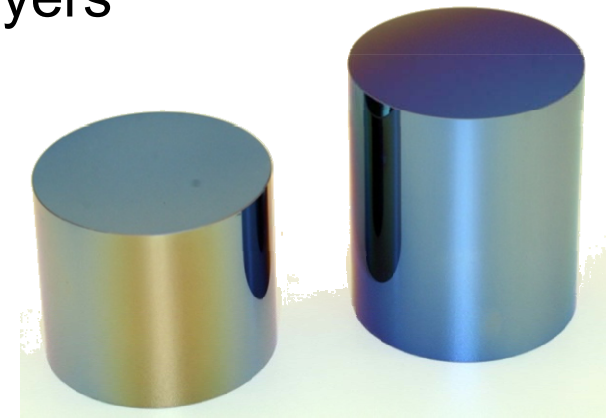
Silicon Bond Loss Experiment



- Ellipsometry was used to determine the thickness of the oxide layers
  - 65 × 50 mm → 171 nm
  - 65 × 70 mm → 317 nm



- Surface measurements determined the flatness of the bonding surfaces to be:
  - 65 × 70 mm → 118.57 nm
  - 65 × 50 mm → 118.39 nm
- i.e. Bonding faces are ~ λ/5 flat

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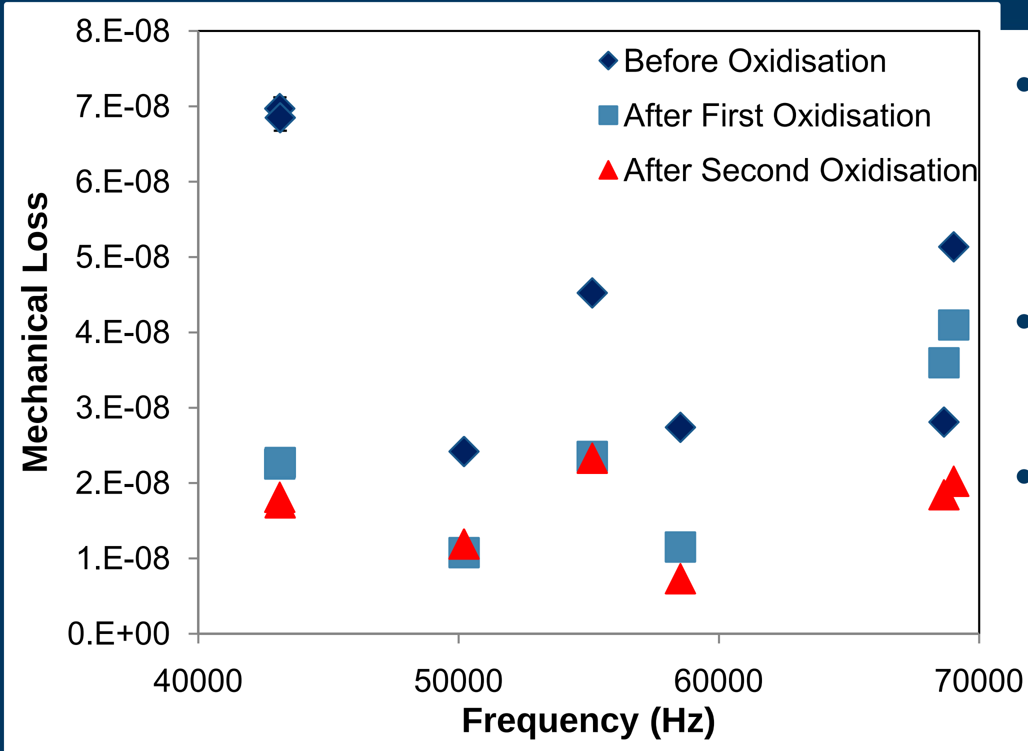
Silicon Bond Loss Experiment



- Bumps appeared after first oxidation
  - Samples etched and re-oxidised
  - Bumps remained
- Mechanical loss measurements made after each oxidation
- Mechanical loss improved after each oxidation
  - Shown for the 70 mm mass

Lowest mechanical loss before

50 mm

$\phi_{lowest} = (1.33 \pm 0.01) \times 10^{-8}$

70 mm

$\phi_{lowest} = (2.42 \pm 0.02) \times 10^{-8}$

120 mm

$\phi_{lowest} = (2.13 \pm 0.04) \times 10^{-8}$

~1.5×

~3.3×

~3×

Lowest mechanical loss after

50 mm

$\phi_{lowest} = (8.55 \pm 0.18) \times 10^{-9}$

70 mm

$\phi_{lowest} = (7.30 \pm 0.13) \times 10^{-9}$

120 mm

$\phi_{lowest} = (6.99 \pm 0.06) \times 10^{-9}$

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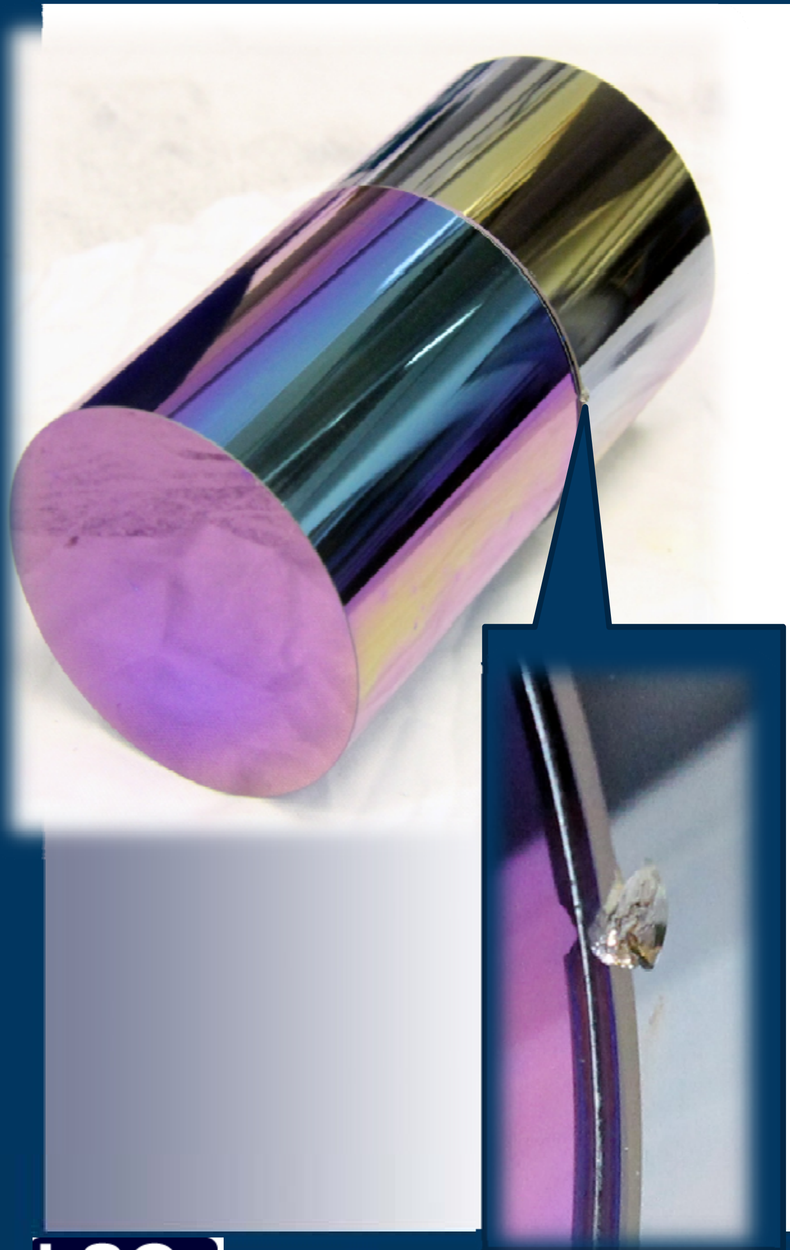
Silicon Bond Loss Experiment



- Bumps removed for bonding
  - ~300 nm remained on 50 mm sample
- Bond offset by ~2 mm to avoid wedging
- Same bonding solution as aLIGO
  - Commercially available
  - NaOH ~14%, SiO_2 ~27% by weight; 59% DI water
  - Diluted 1:6 in de-ionised water
- The samples were aligned and bonded using 12.244 μl of bonding solution
- Mechanical loss measurements taken after 4 week cure period

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Silicon Bond Loss Experiment

- For each resonant mode

$$\varphi_{bonded} = \varphi_{substrate} + \frac{E_{bond}}{E_{total}}\varphi_{bond} + \frac{E_{oxide}}{E_{total}}\varphi_{oxide}$$

  - From I. Martin's thesis $\phi_{oxide} = 1 \times 10^{-4}$

- Energy ratios calculated using ANSYS® FEA package

- The flatness of the two bonding surfaces was used as an estimate of the bond thickness
  - Thus for a bond thickness of ~118.48 nm a preliminary average bond loss of 0.6 ± 0.1 is estimated

- Comparable to previous results obtained by E. Chalkley where a bond loss of 0.4 was determined from bonded cantilevers

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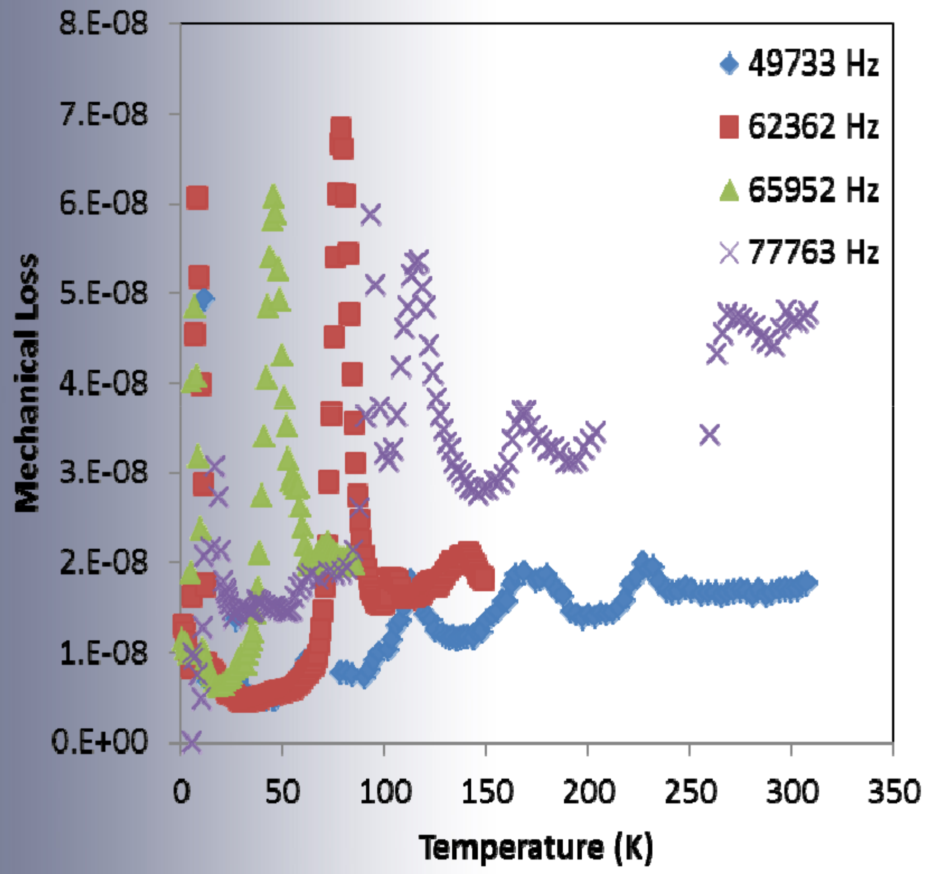
Silicon Bond Loss Experiment



- Experiment is currently being repeated using three silicon (111) substrates in collaboration with colleagues at Jena
  - At room temperature and at cryogenic temperatures down to 5K
- Essential to consider how the loss of the substrates change with temperature
  - Shown for the 70 mm mass
- Should provide information on the levels of the bond loss down to the temperatures at which a 3rd generation detector might operate

Hydroxide Catalysis Bond Strength Tests

N. Beveridge
On behalf of the Institute for Gravitational Research, University of Glasgow

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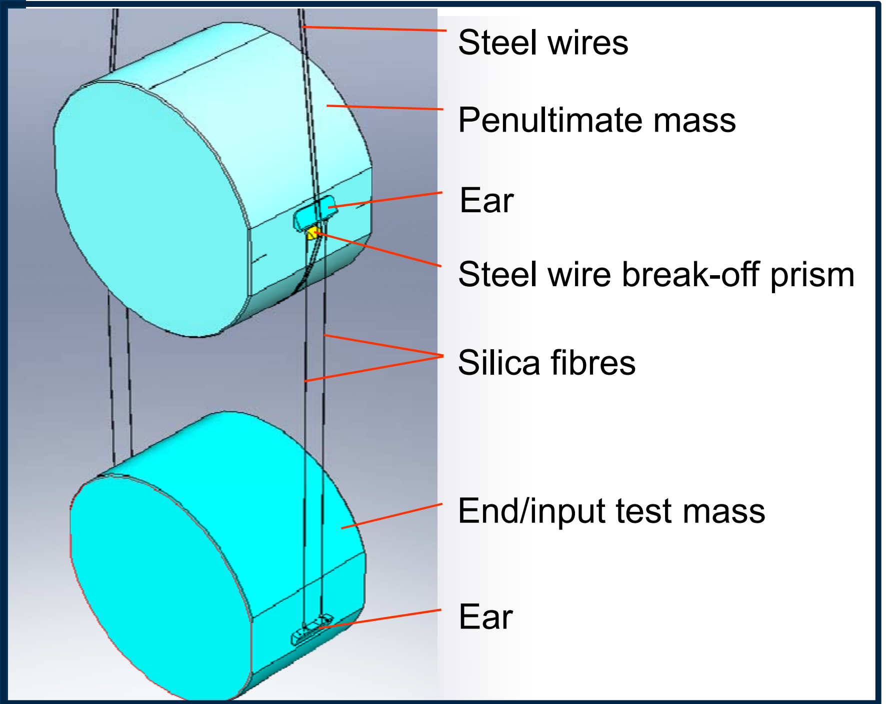
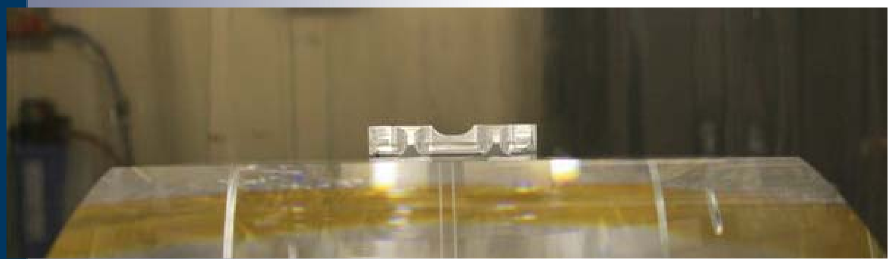
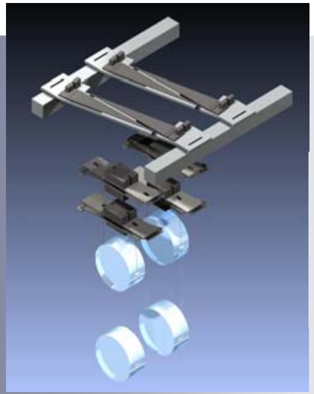
The Role of Hydroxide-Catalysis Bonding





- Developed by Gwo for Gravity Probe B
- Used to bond silica interface pieces, 'ears', on side of silica test masses
  - Provides welding point for suspension fibres
- Currently used in GEO600, and being installed in the silica suspensions for Advanced LIGO and Advanced Virgo

Future Generation Detectors



- Reduction of thermal noise
  - Cool test masses and suspensions
- Silicon and sapphire potential replacement materials due to low dissipation at cryogenic temperatures
  - Silica has high dissipation at low temperature
- Application of hydroxide-catalysis bonding in silicon suspension
  - Silicon – silicon bonds require an oxide layer
  - What strength would bonds have?
  - What influence does temperature have on bond strength?

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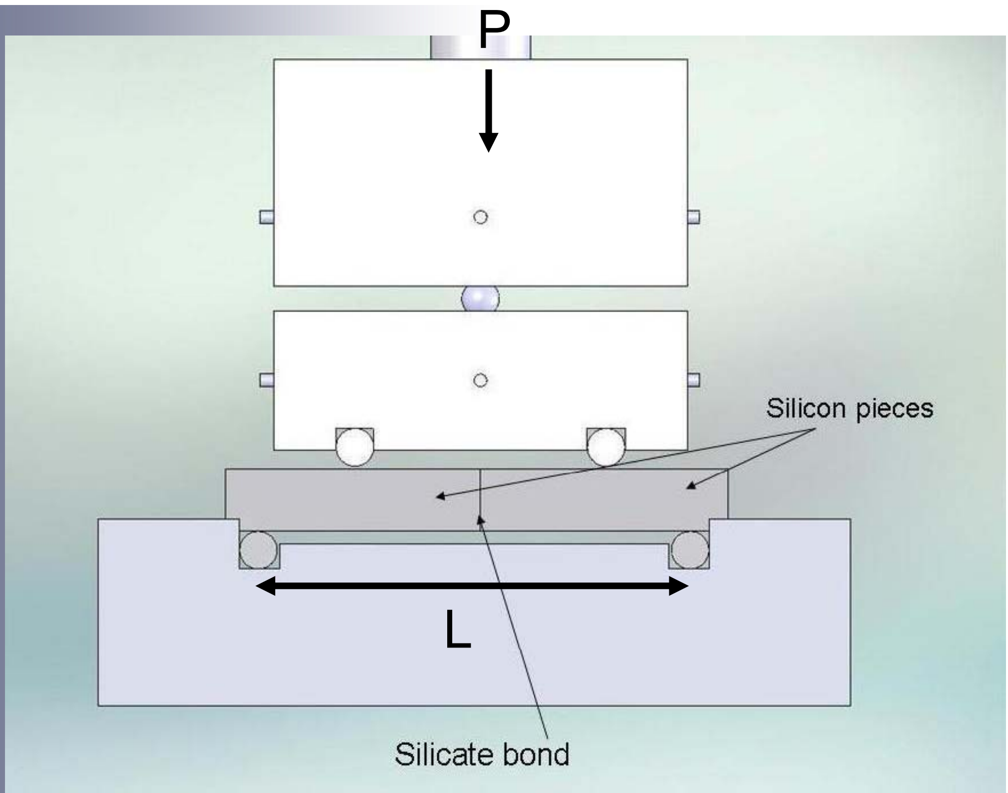
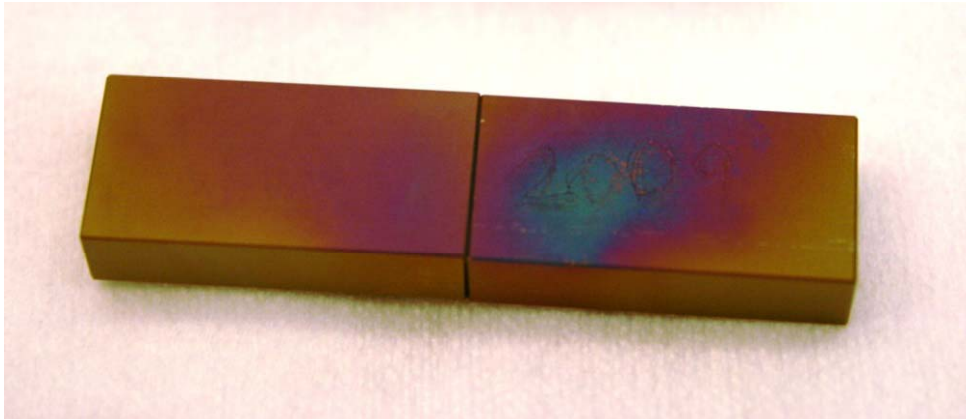
Strength Test Set-up

ASTM C1161-02c four point ¼ point flexural strength test





$$S = \frac{3PL}{4bd^2}$$

- Silicon piece size: 5 x 10 x 20 mm
  (*b* x *d* x *l*)
- Bonded sample: 5 x 10 x 40 mm
  (*b* x *d* x *l*)
- Bonding surface has PV flatness < 60 nm

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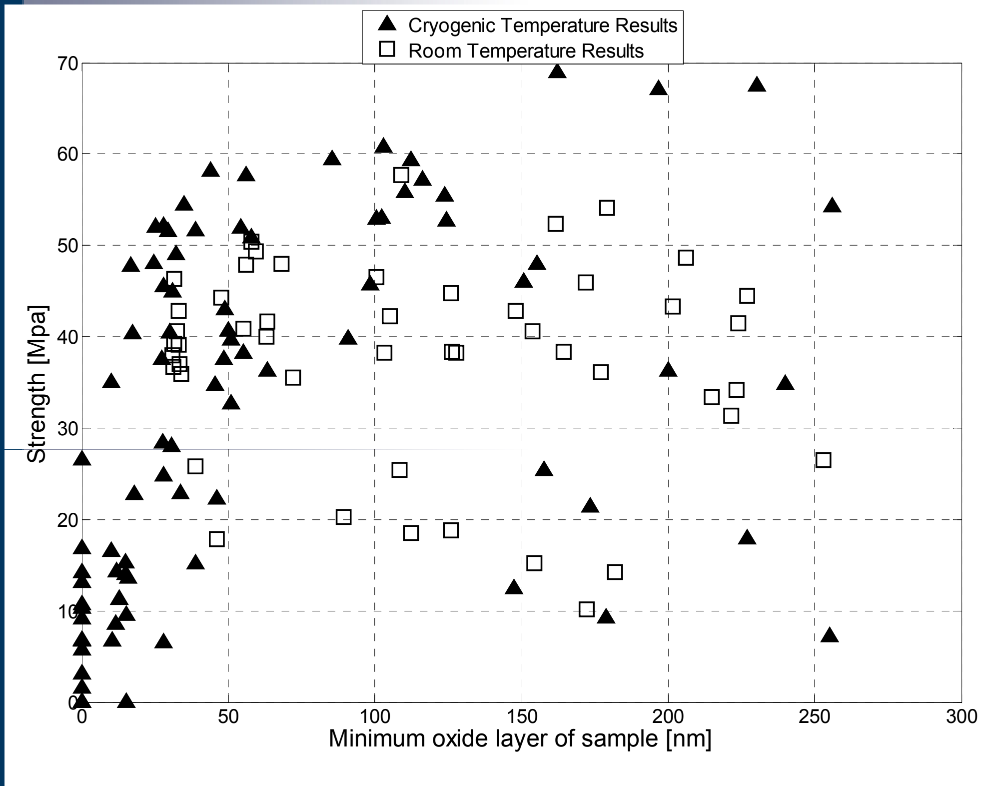
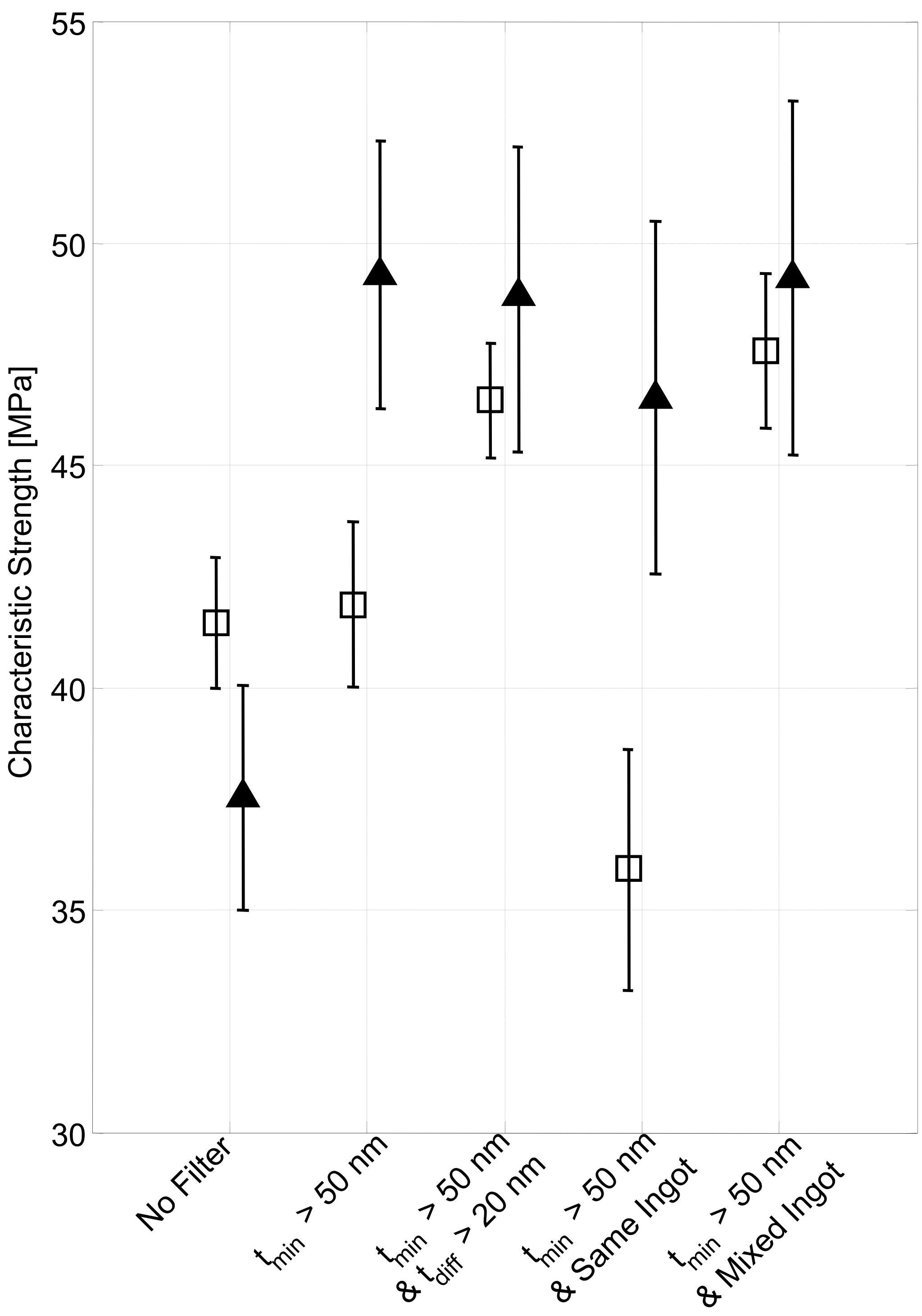
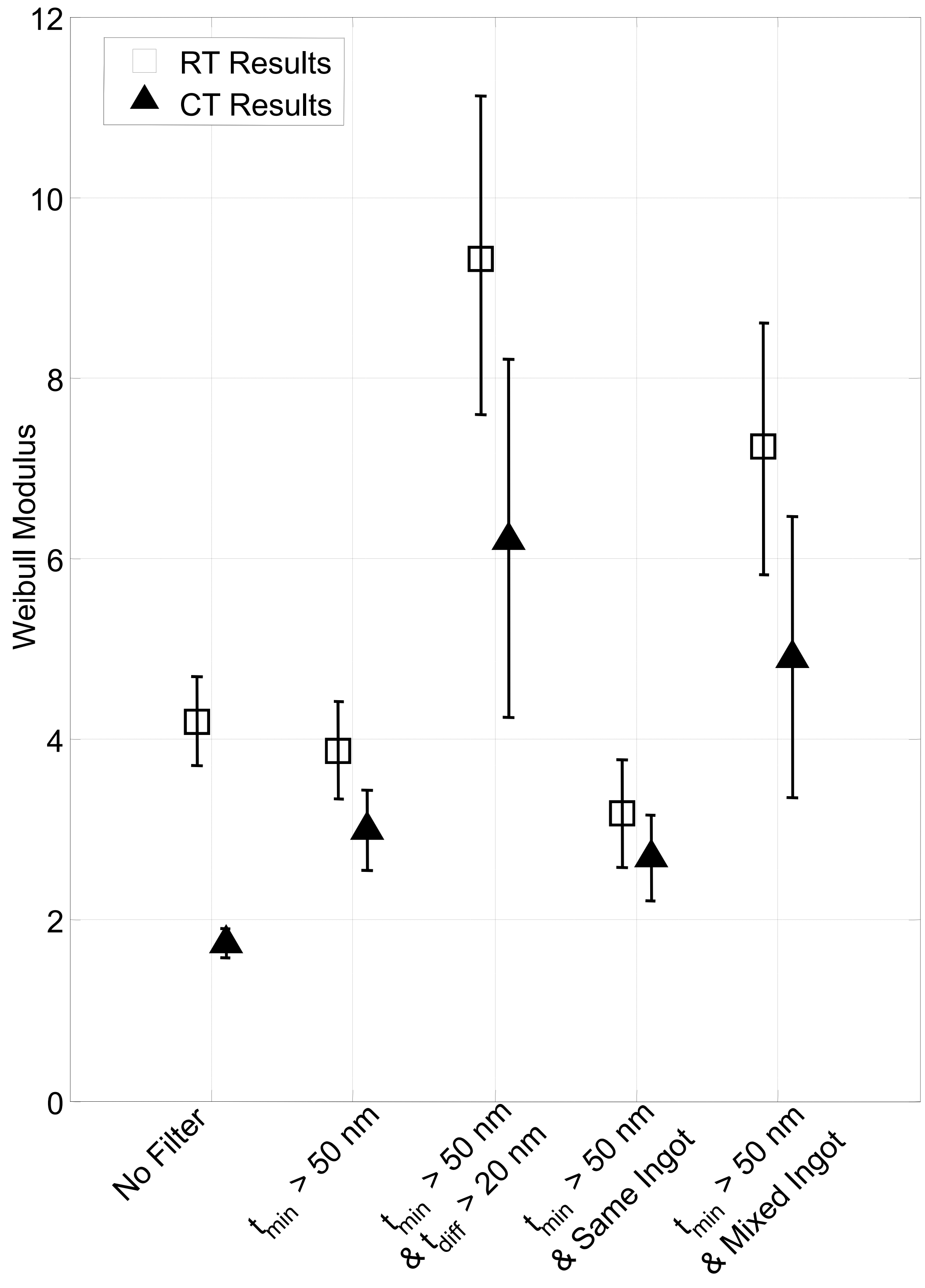
Previous Experiments and Results



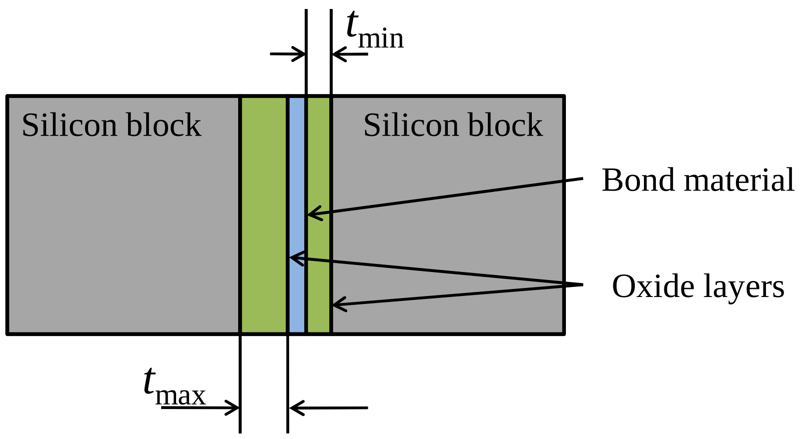
N L Beveridge, A A van Veggel, M Hendry et al,
Low-temperature strength tests and SEM imaging of hydroxide-catalysis bonds in silicon
Classical and Quantum Gravity, Volume 28, Issue 8, pp. 085014 (2011).

- Test parameters:
  - 49 samples tested at 293 K
  - 86 samples tested at ~77 K
  - Two ingot types
    - Prolog <111> (Ingot 1)
    - Prolog <100> (Ingot 2)
  - Bonding surfaces oxidised in a wet thermal environment
  - Oxide layer thickness varied
- Results
  - No reduction of strength at ~77 K
  - Minimum oxide layer of 50 nm required for a reliable bond at cryogenic temperature
  - Weibull analysis performed on data

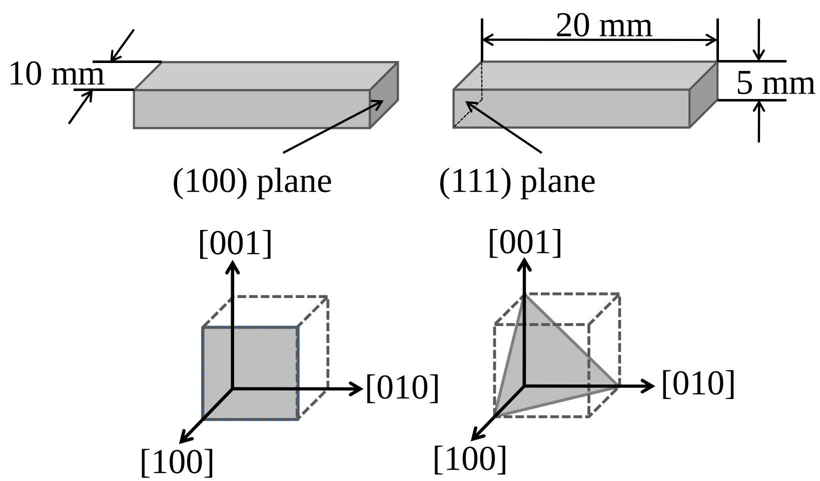

Characteristic Strength [MPa]
55
50
45
40
35
30
No Filter
t_{min} > 50 nm
t_{min} > 50 nm & t_{diff} > 20 nm
t_{min} > 50 nm & Same Ingot
t_{min} > 50 nm & Mixed Ingot


RT Results
CT Results
Weibull Modulus
12
10
8
6
4
2
0
No Filter
t_{min} > 50 nm
t_{min} > 50 nm & t_{diff} > 20 nm
t_{min} > 50 nm & Same Ingot
t_{min} > 50 nm & Mixed Ingot

Previous Analysis



$$t_{diff} = t_{max} - t_{min}$$



- At cryogenic temperature, large increase in strength after t_{min} set to 50 nm
- Improvement in reliability of F_0 value when t_{diff} > 20 nm
- Improvement seen in both F_0 and m when blocks of different orientations bonded together

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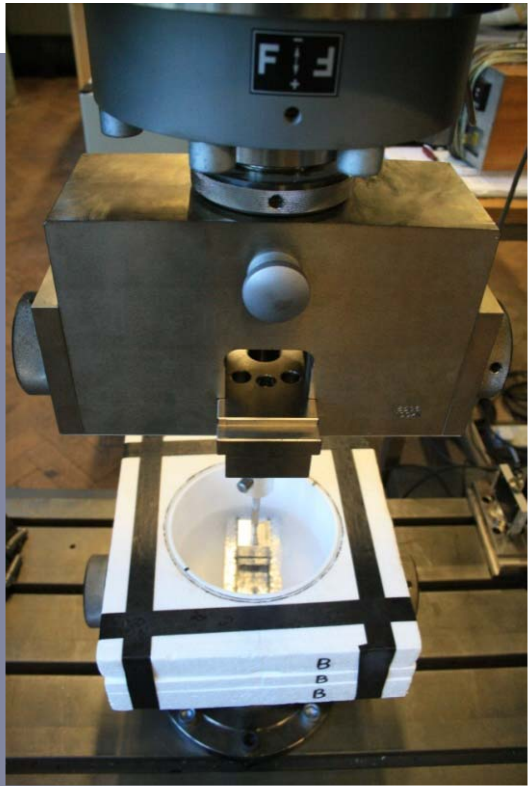
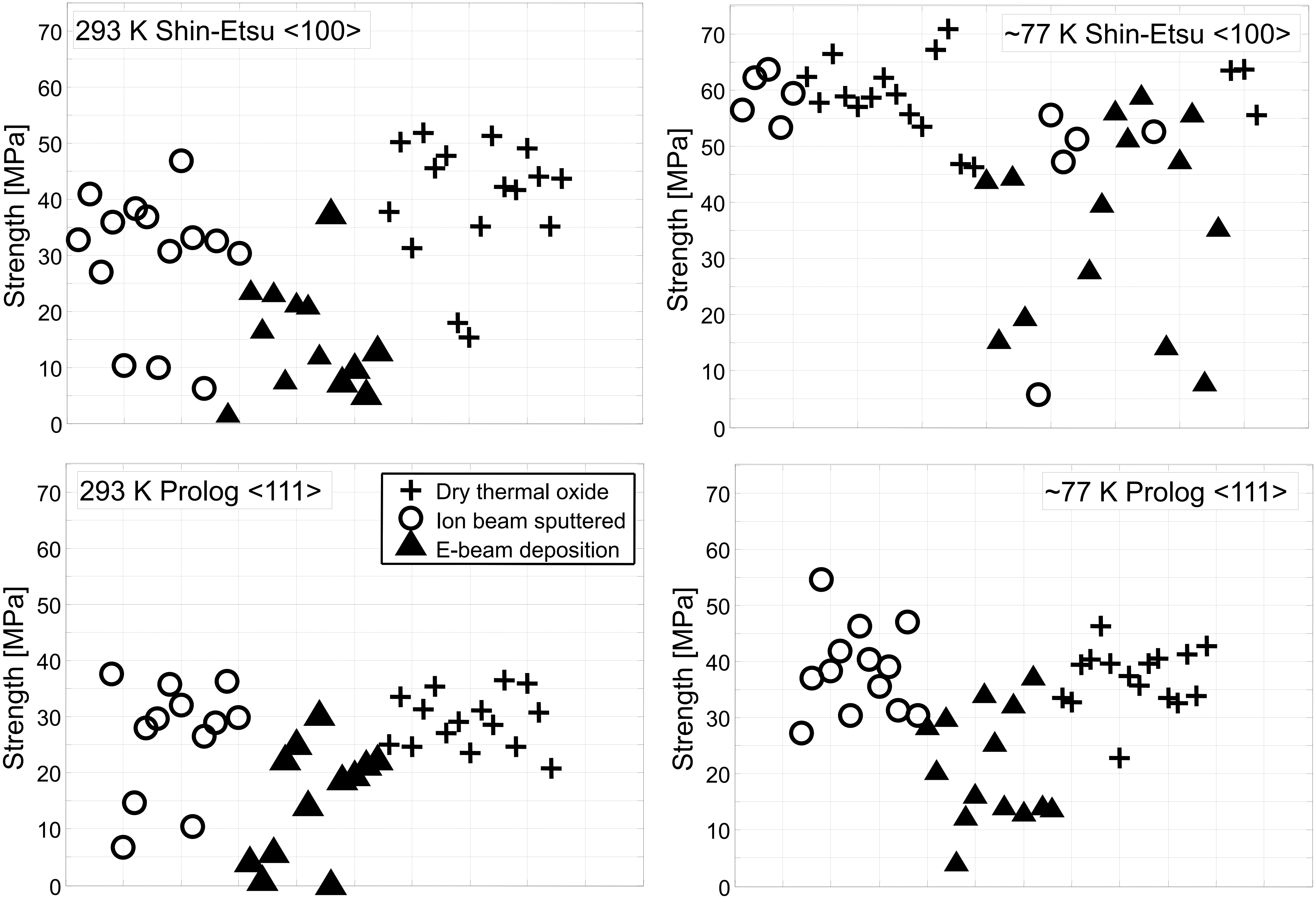
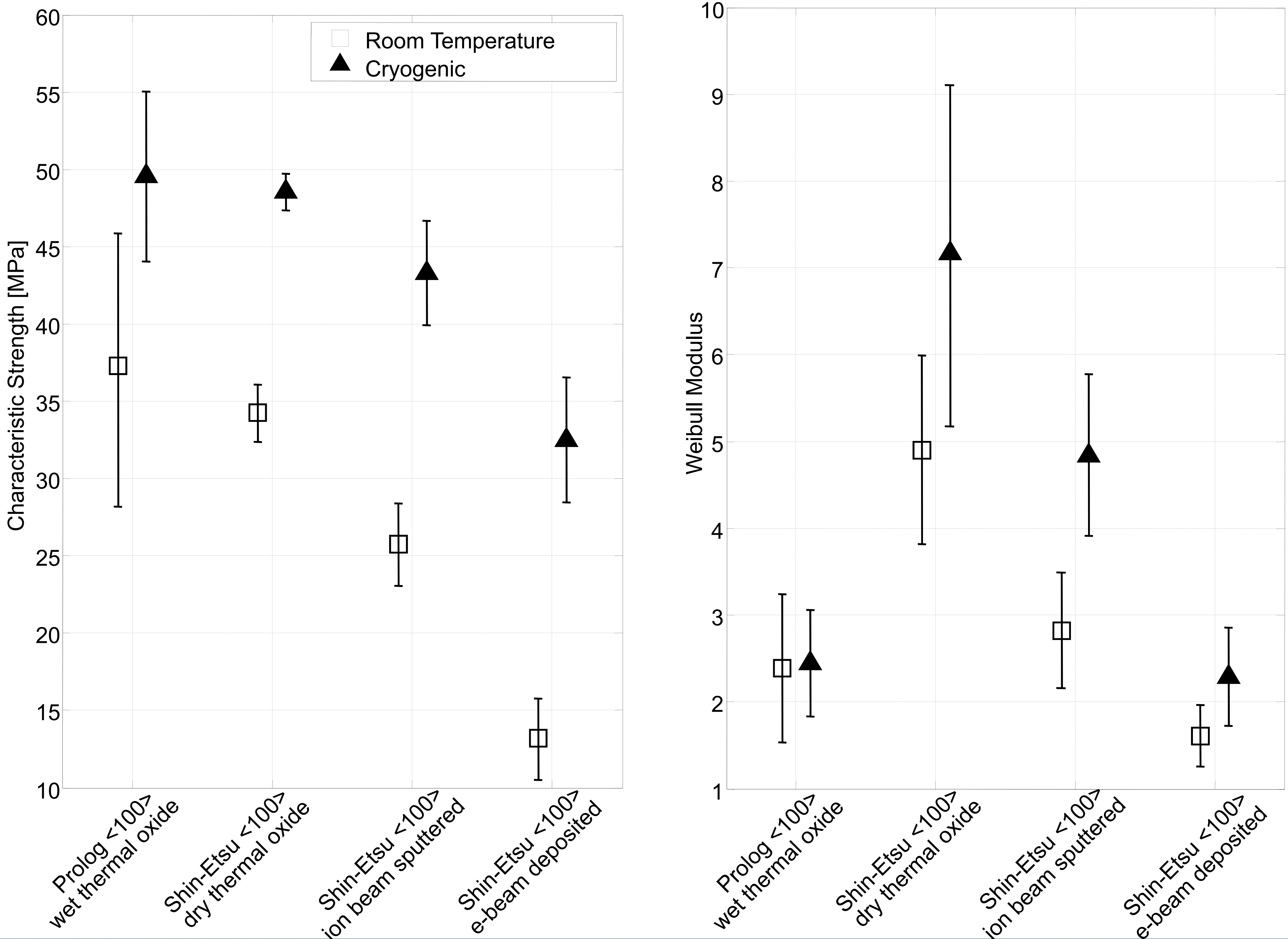
Recent Experiments and Results

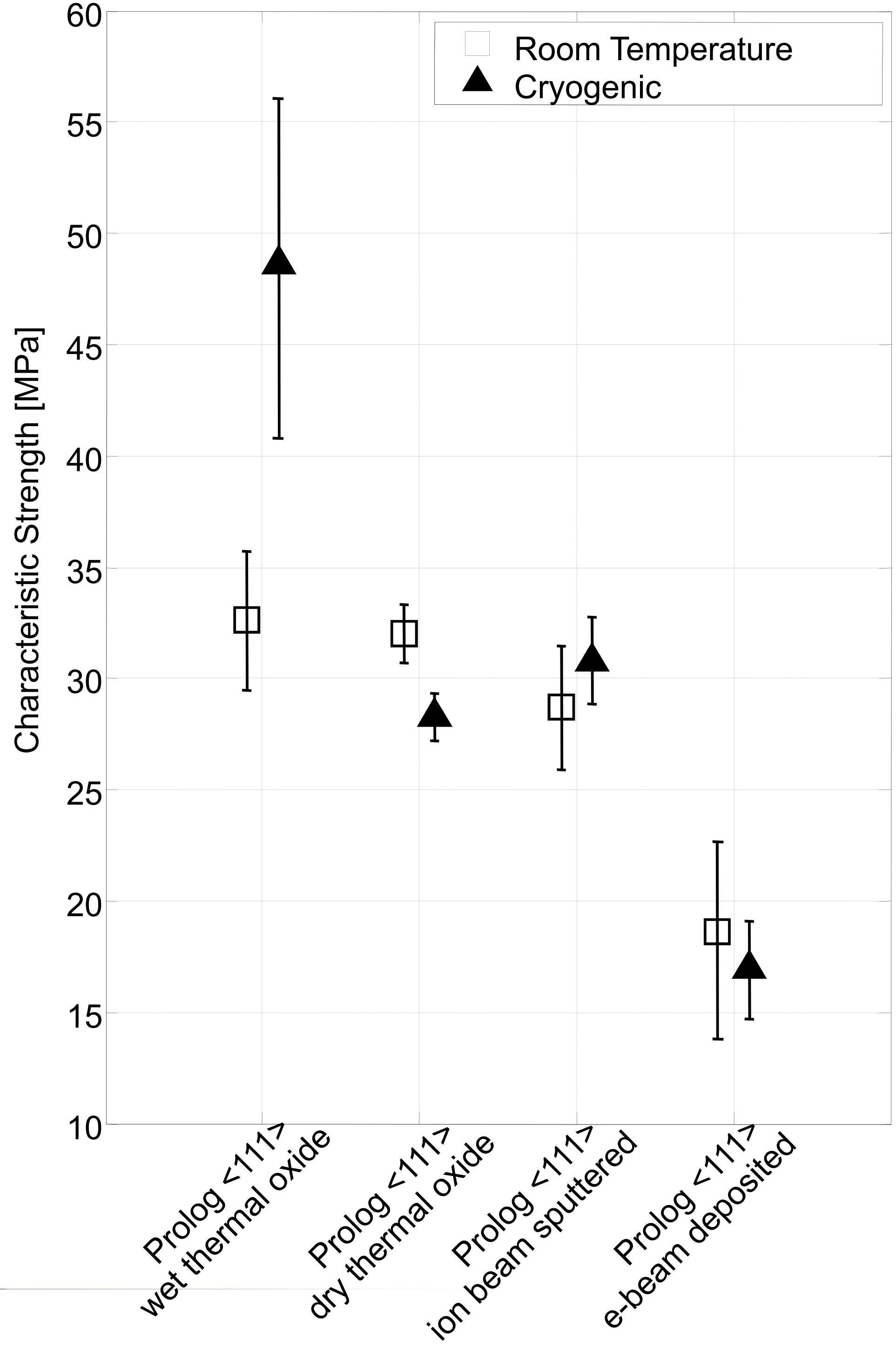


- 82 room temperature tests (RT)
- 88 cryogenic tests (CT)
- Three types of oxide layer
  - Dry thermal oxide (165 ± 14 nm)
    - Oxidised at Uni. Glasgow
  - E-beam (144 ± 1 nm)
    - Gooch and Housego
  - Ion beam (154 ± 1 nm)
    - Advanced Thin Films
- Two ingot types
  - Shin-Etsu <100> (high purity) (Ingot 3)
  - Prolog <111> (Ingot 4)

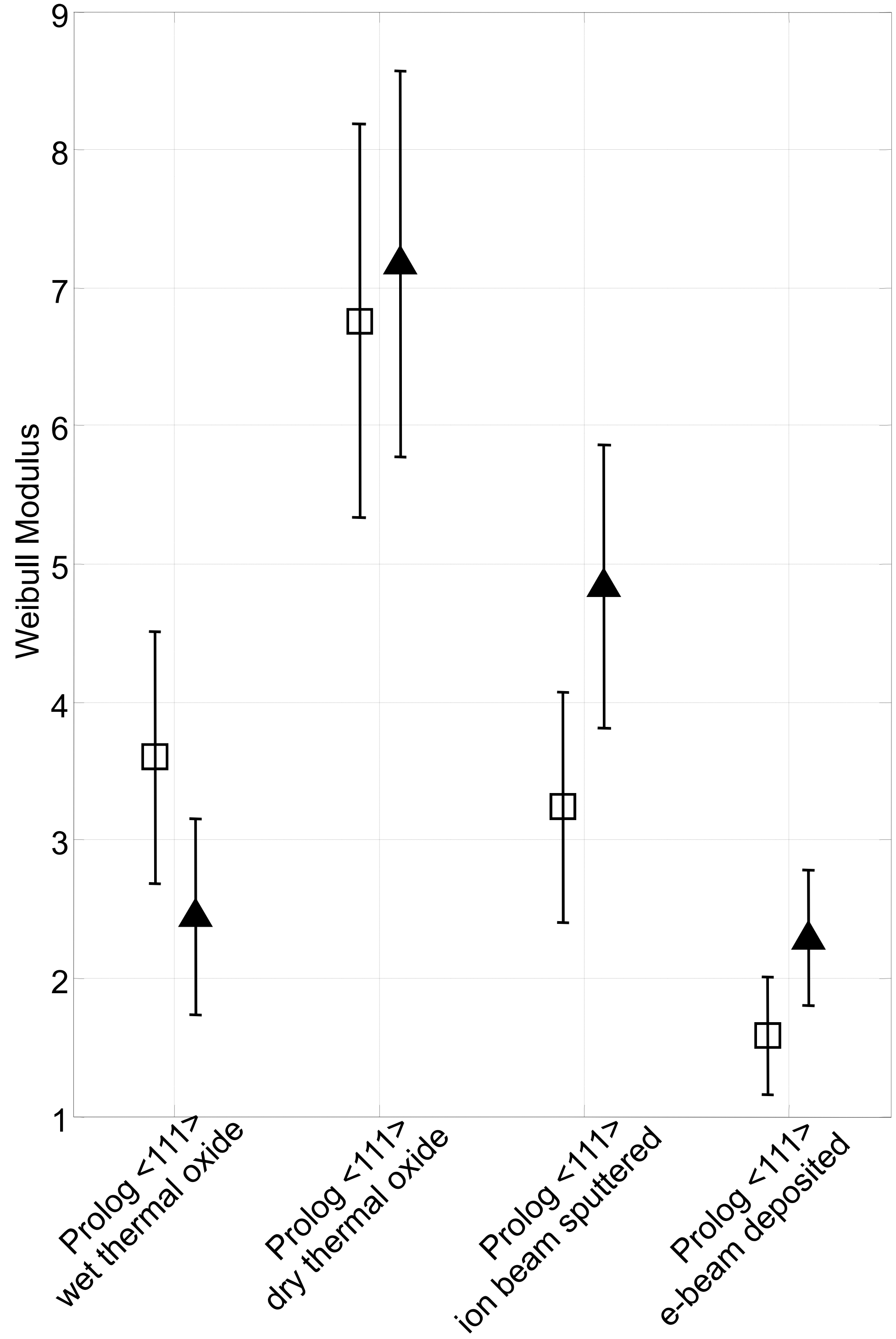

293 K Shin-Etsu <100>
~77 K Shin-Etsu <100>
293 K Prolog <111>
~77 K Prolog <111>
Strength [MPa]
0
10
20
30
40
50
60
70
Dry thermal oxide
Ion beam sputtered
E-beam deposition



Room Temperature
Cryogenic
Characteristic Strength [MPa]
Weibull Modulus
Prolog <100> wet thermal oxide
Shin-Etsu <100> dry thermal oxide
Shin-Etsu <100> ion beam sputtered
Shin-Etsu <100> e-beam deposited



Room Temperature
Cryogenic
Characteristic Strength [MPa]
60
55
50
45
40
35
30
25
20
15
10
Prolog <111> wet thermal oxide
Prolog <111> dry thermal oxide
Prolog <111> ion beam sputtered
Prolog <111> e-beam deposited



Weibull Modulus
9
8
7
6
5
4
3
2
1
Prolog <111> wet thermal oxide
Prolog <111> dry thermal oxide
Prolog <111> ion beam sputtered
Prolog <111> e-beam deposited

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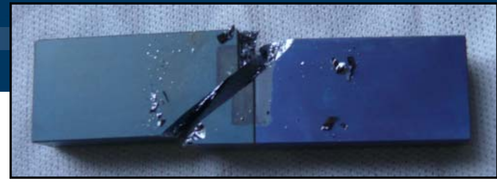
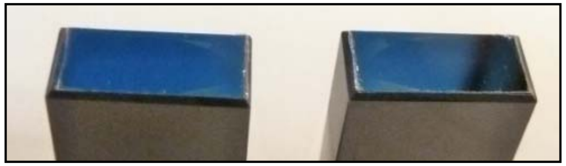
Analysis



		%age Bond Breaks CT	%age Bond Breaks RT
Dry Ox	Ingot 3	24%	19%
	Ingot 4	88%	33%
Ion Beam	Ingot 3	36%	36%
	Ingot 4	77%	92%
E-Beam	Ingot 3	57%	54%
	Ingot 4	93%	72%



- Bond breaks generally weaker than 'diagonal' breaks
- In all cases, increased number of bond breaks for ingot 4 (Prolog <111>)
- Higher percentage of bond breaks for the e-beam coating
  - Matches the lower strengths of e-beam samples

Summary

Liquid may supress surface crack propagation

Possible geometric effect

Possibly due to density

- Temperature
  - Cryogenic temperatures do not weaken bond
  - Cryogenic results often stronger than room temperature
- Oxide layer thickness
  - Minimum oxide required: 50 nm
  - t_{diff} > 20 nm improves reliability
- Ingot orientation
  - CT results always stronger for <100>
  - Mixed ingot samples produce better results
- Oxide layer type
  - E-beam results weakest
  - Thermal oxides strongest
  - Dry thermal oxide weaker but more reliable
  - E-beam more likely to break across bond

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Future Work

- Strength of bond
  - Strength test bulk samples
    - Influence of cryogenic temperature
    - Influence of purity level
  - Density measurements of oxide layers

- Other properties of bond
  - Bond thickness
  - Elastic modulus
  - Thermal conductivity
  - Mechanical loss

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Thank you for your attention

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Weibull Analysis

- Distribution of data not Gaussian
- Variation in strengths of brittle samples typically modelled by a Weibull probability density function (pdf):

$$P(F) = \frac{m}{F_0}\left(\frac{F}{F_0}\right)^{m-1} \exp\left[-\left(\frac{F}{F_0}\right)^{m}\right]$$

  - F_0 represents a characteristic strength
  - Weibull modulus, *m*, provides a measure of variability of strength of material
- Bayesian analysis used to estimate parameters from the Weibull pdf
- MCMC method used to obtain confidence regions for *m* and F_0

Hydroxide – Catalysis Bonding