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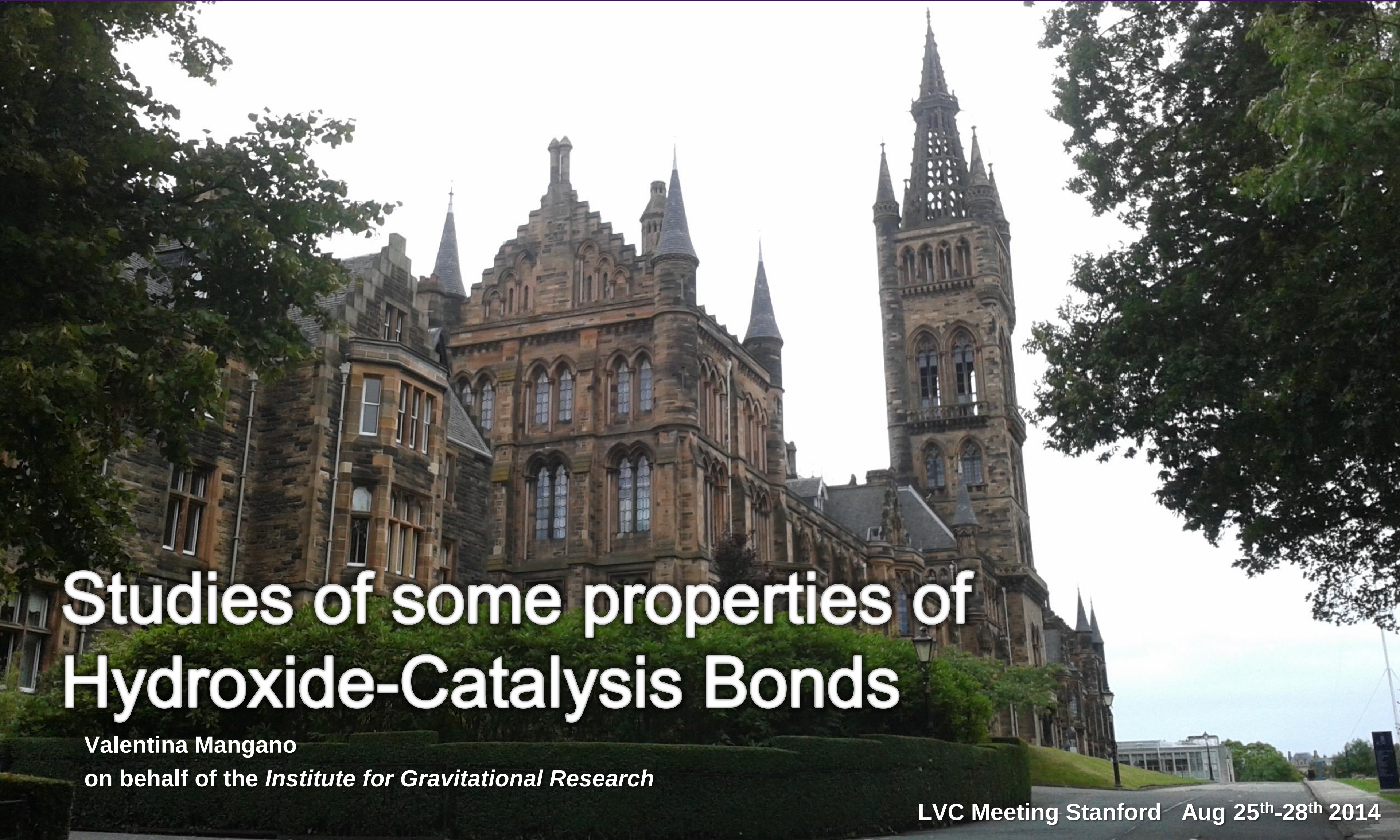

# Studies of some properties of Hydroxide-Catalysis Bonds

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**on behalf of the *Institute for Gravitational Research***

**LVC Meeting Stanford   Aug 25th-28th 2014**

# Outline

-Introduction

-Fabrication of bonds

-Other applications

-Experimental setup

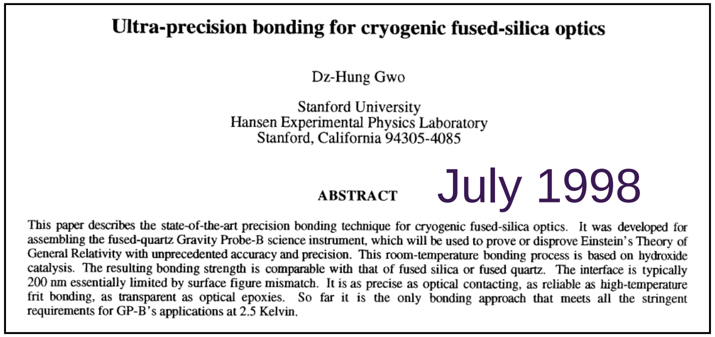
-Results and Analysis

-Conclusions and Future work

# Introduction

**Hydroxide-catalysis bonding** is a **jointing technique** created at **room temperature** and characterized by:

- **very thin bonds which only introduce low levels of mechanical loss**
- **high strength**
- **high precision alignment**



**Ultra-precision bonding for cryogenic fused-silica optics**

Dz-Hung Gwo

Stanford University
Hansen Experimental Physics Laboratory
Stanford, California 94305-4085

**ABSTRACT**

July 1998

This paper describes the state-of-the-art precision bonding technique for cryogenic fused-silica optics. It was developed for assembling the fused-quartz Gravity Probe-B science instrument, which will be used to prove or disprove Einstein's Theory of General Relativity with unprecedented accuracy and precision. This room-temperature bonding process is based on hydroxide catalysis. The resulting bonding strength is comparable with that of fused silica or fused quartz. The interface is typically 200 nm essentially limited by surface figure mismatch. It is as precise as optical contacting, as reliable as high-temperature frit bonding, as transparent as optical epoxies. So far it is the only bonding approach that meets all the stringent requirements for GP-B's applications at 2.5 Kelvin.

Hydroxide-catalysis bonding was used in GEO600, and it is being used in the quasi-monolithic fused silica mirror suspensions for the advanced gravitational wave detectors (aLIGO and advanced Virgo).

To reduce the thermal noise further, cryogenic temperatures and crystalline materials, such as silicon and sapphire, are proposed for the next generations of detectors.

**Is it possible to realize such a mirror suspension at cryogenic temperatures with these materials using the hydroxide-catalysis bonding?**

# Fabrication of bonds

It is possible to combine any material, provided that **a silicate-like network** can be formed between them.

Example materials that can be bonded:

○ **oxide materials**: silica, fused silica, Zerodur, ultra low expansion glass, granite and sapphire

○ **oxidisable materials**: silicon and silicon carbide

The materials are bonded by means of an alkaline bonding solution:

○ **potassium hydroxide** $KOH$

○ **sodium hydroxide** $NaOH$

○ **sodium silicate** $Na_2SiO_3$

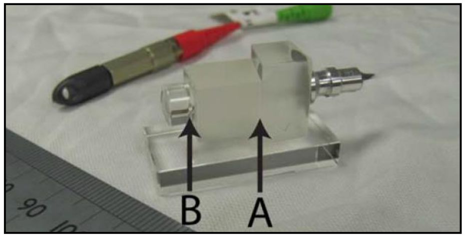
Want to **develop techniques to characterize bond properties for different substrates and solutions** to allow us to **tailor bond properties to the various utilizations of interest**.

# Other applications

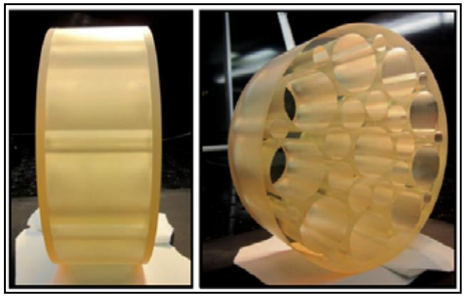
**Jointing optical components with low losses and high strength can also be employed in various applications,** outside gravitational wave field.

(van Veggel, Killow - Adv. Opt. Techn. 2014; 3(3): 293–307)

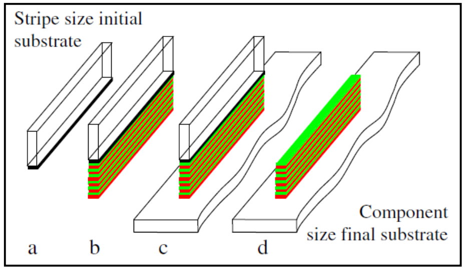
- ➢ **connection of a fiber injector with a collimated lens** through a fused silica spacer (Taylor et al. - ASP Conference Series, 467 (2012))



- ➢ **adaptive mirrors of the telescopes** to bond actuators to substrates (Strachan et al. - Proc. SPIE 7736, 773664 and 773661 (2010))



- ➢ **lightweight composite mirrors** (MacKay et al. - Proc. SPIE 8884, Optifab 2013, 88841N (2013))
- ➢ **construction of high power lasers** (Sinha et al. - Opt. Express 15, 13003 (2007))
- ➢ **transfer of a multilayer coating** from a substrate to another one (Duchêne et al. – Opt. Commun. 285, 128-132 (2011))



# Experimental setup

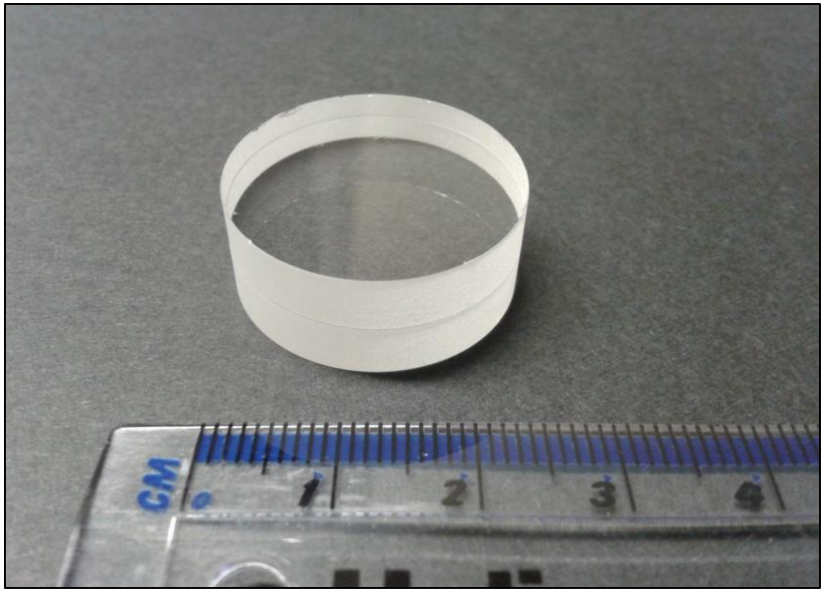
Aim:

to measure the **magnitude of the light reflected from bonded interfaces** and use as a tool to determine **bond thickness** in a non-destructive manner.

For this purpose:

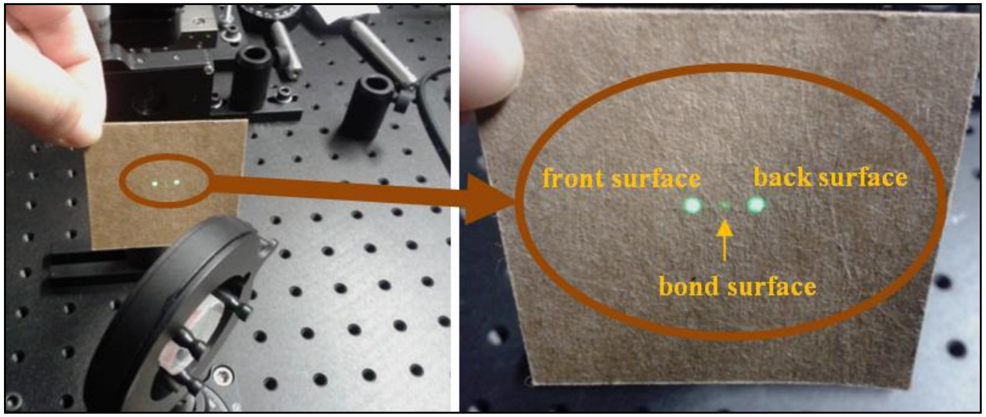
- a **pair of fused silica discs** was bonded by means of **sodium silicate solution**;

- a **setup to measure the reflectances** of both polarisation components of the incident light of a green laser diode module (λ=532nm) on the sample was realised.

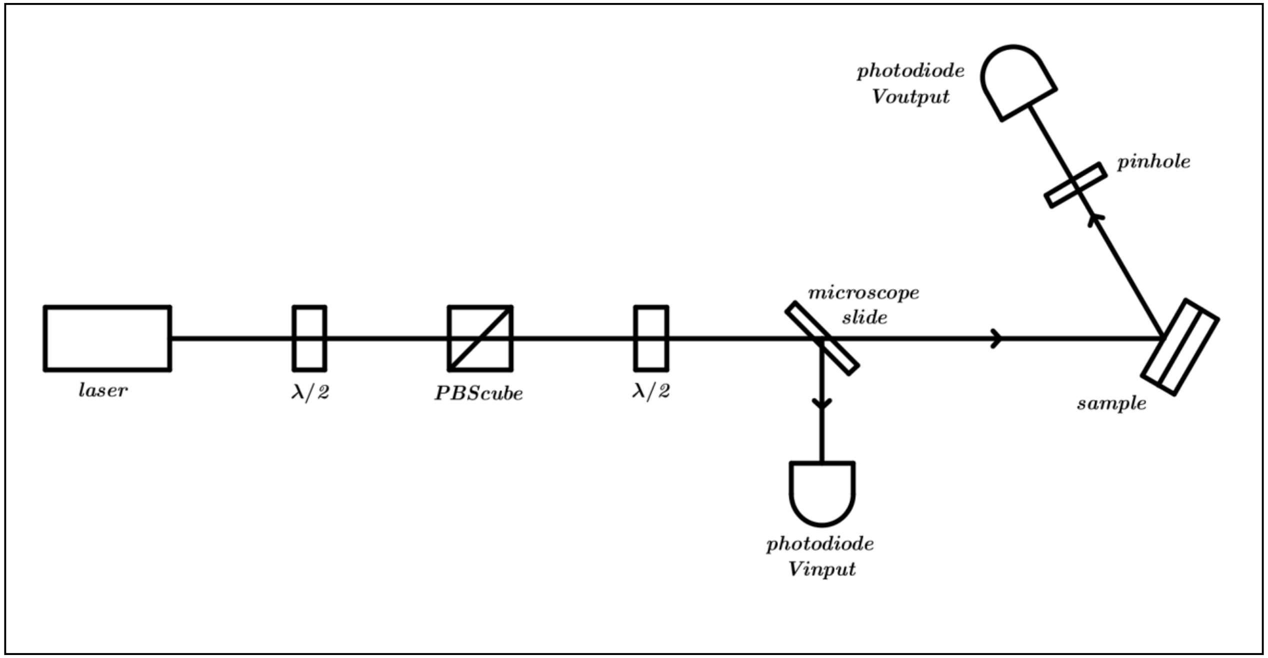


# Experimental setup

For both the polarisations, the **incident and reflected power** of laser beam are measured as a function of angle of incidence, and the corresponding **reflectances** determined.

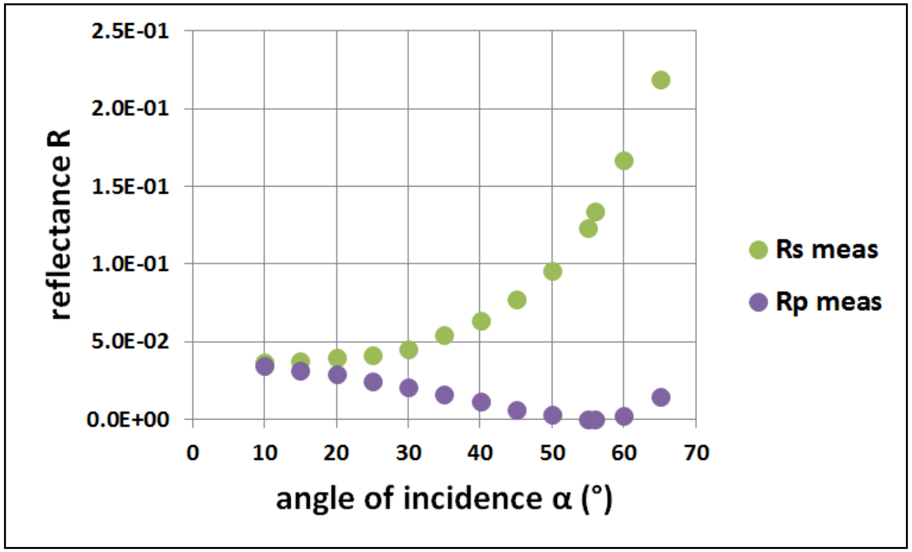


$$\begin{cases} R_{\perp} = R_S = \dfrac{V_{output,S}}{V_{input}} \\ R_{\parallel} = R_P = \dfrac{V_{output,P}}{V_{input}} \end{cases}$$

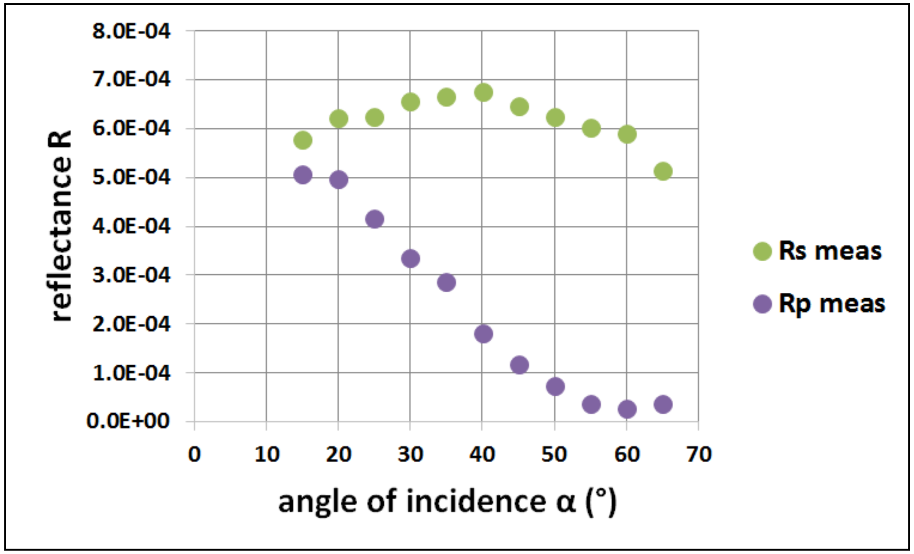


# Results

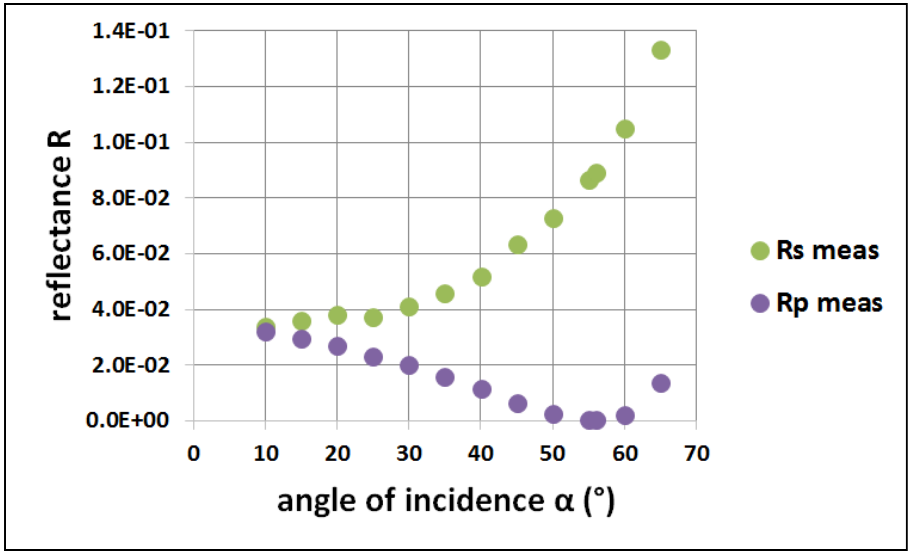
front surface



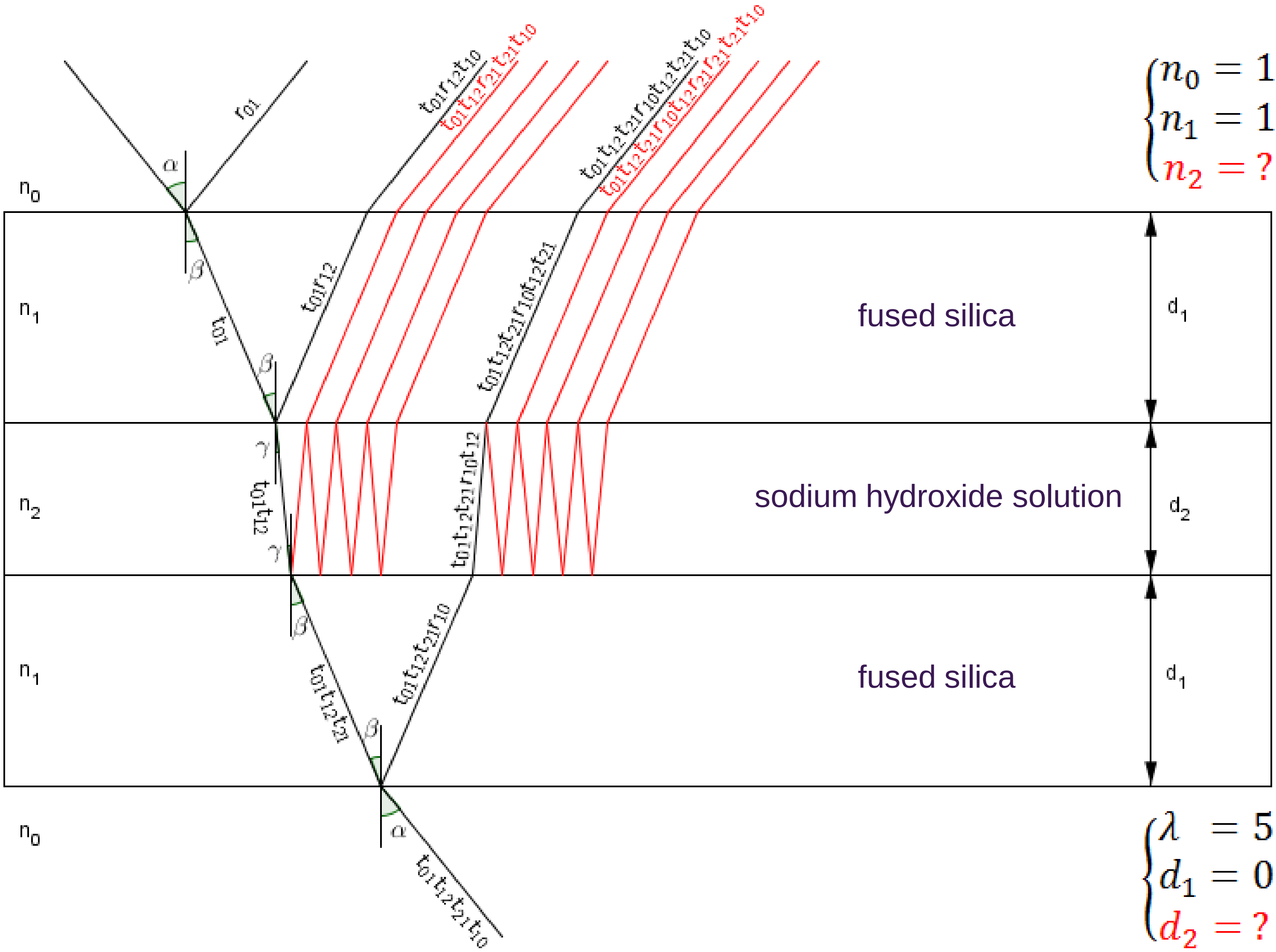
bond surface



back surface



# Analysis: sample



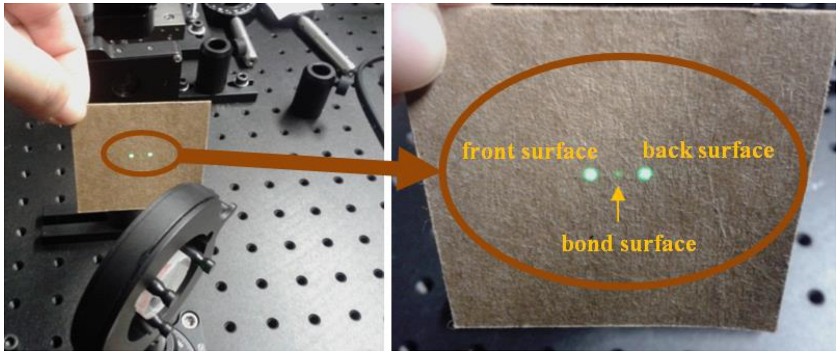
$$\begin{cases} n_0 = 1 \\ n_1 = 1.46 \\ n_2 = ? \end{cases}$$

$$\begin{cases} \lambda = 532nm \\ d_1 = 0.5cm = 5 \cdot 10^6 nm \\ d_2 = ? \end{cases}$$

# Analysis: formulae

## front surface

$$\begin{cases} R_{1,\perp} = \left( \dfrac{\cos\alpha - \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}}{\cos\alpha + \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}} \right)^2 \\ R_{1,\parallel} = \left( \dfrac{\left(\dfrac{n_1}{n_0}\right)^2 \cos\alpha - \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}}{\left(\dfrac{n_1}{n_0}\right)^2 \cos\alpha + \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}} \right)^2 \end{cases}$$



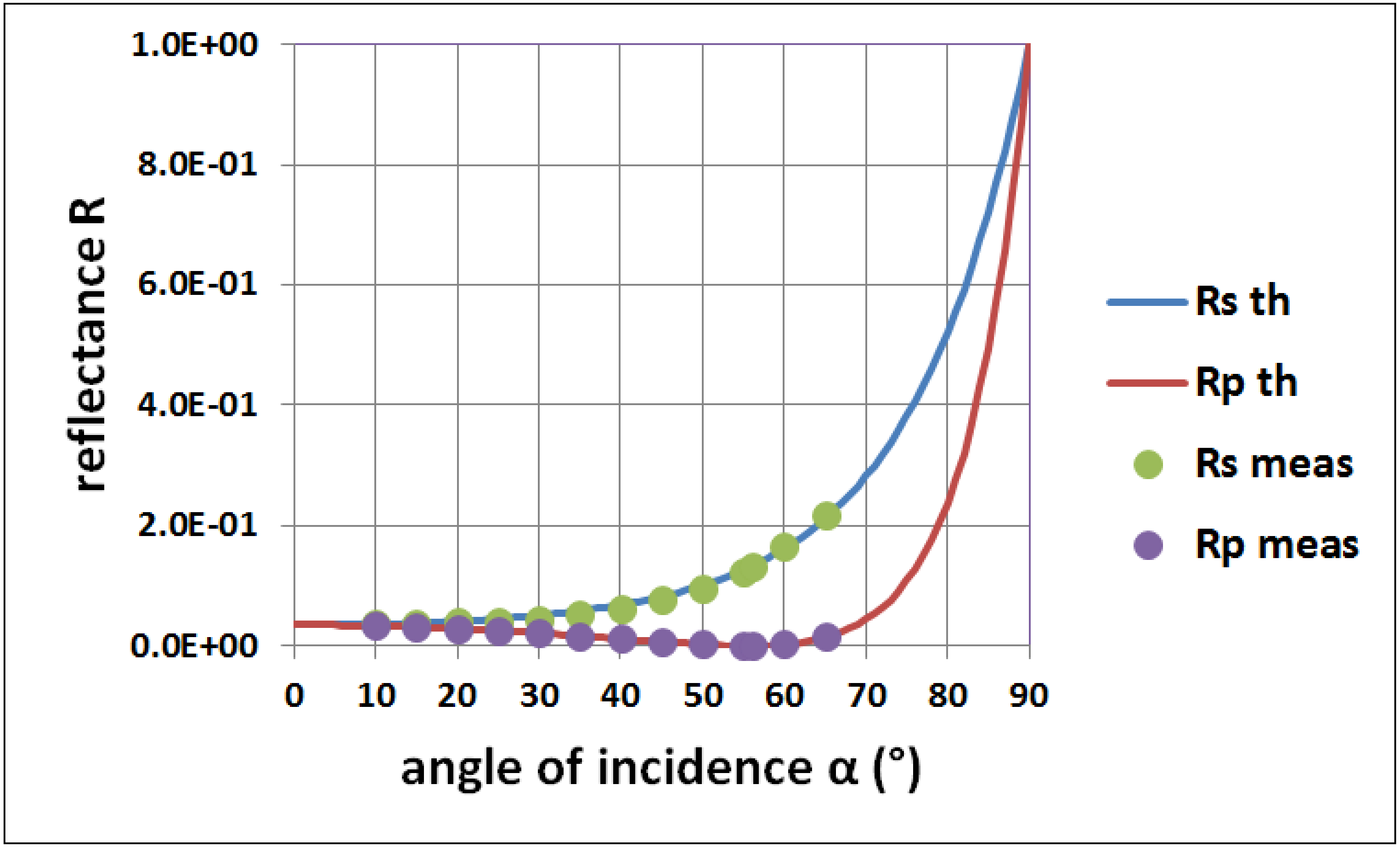
## bond surface

$$\begin{cases} R_{2,\perp} = \dfrac{2\left(1 - r_{01,\perp}^2\right)^2 r_{12,\perp}^2 (1 - \cos 2\delta_2)}{1 - 2r_{12,\perp}^2 \cos 2\delta_2 + r_{12,\perp}^4} \\ R_{2,\parallel} = \dfrac{2\left(1 - r_{01,\parallel}^2\right)^2 r_{12,\parallel}^2 (1 - \cos 2\delta_2)}{1 - 2r_{12,\parallel}^2 \cos 2\delta_2 + r_{12,\parallel}^4} \end{cases}$$

## back surface

$$\begin{cases} R_{3,\perp} = \dfrac{\left(1 - r_{01,\perp}^2\right)^2 \left(1 - r_{12,\perp}^2\right)^4 r_{01,\perp}^2}{1 - 2r_{12,\perp}^2 \cos 2\delta_2 + r_{12,\perp}^4} \\ R_{3,\parallel} = \dfrac{\left(1 - r_{01,\parallel}^2\right)^2 \left(1 - r_{12,\parallel}^2\right)^4 r_{01,\parallel}^2}{1 - 2r_{12,\parallel}^2 \cos 2\delta_2 + r_{12,\parallel}^4} \end{cases}$$

# Analysis: front surface

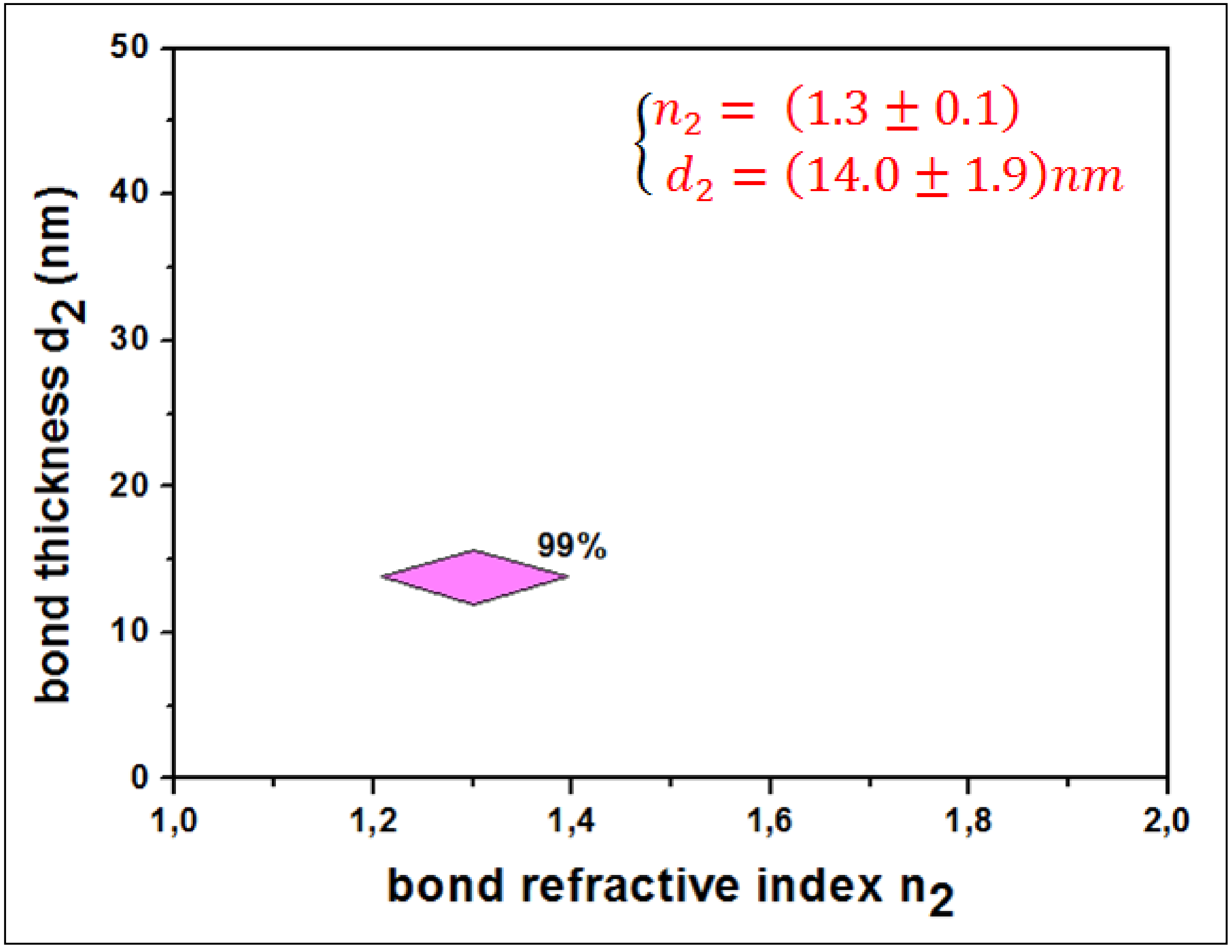


# Analysis:
# bond and back surface

$$R_2(\perp,\parallel)\ and\ R_3(\perp,\parallel) = f(n_2, d_2)$$

$$P_n = exp\left[-\frac{1}{2}(\chi^2 - \chi^2_{min})\right]$$

$$\chi^2 = \sum_{i=1}^{n} \frac{\left(R_i^{th} - R_i^{sper}\right)^2}{\sigma^2_{sper,i}}$$

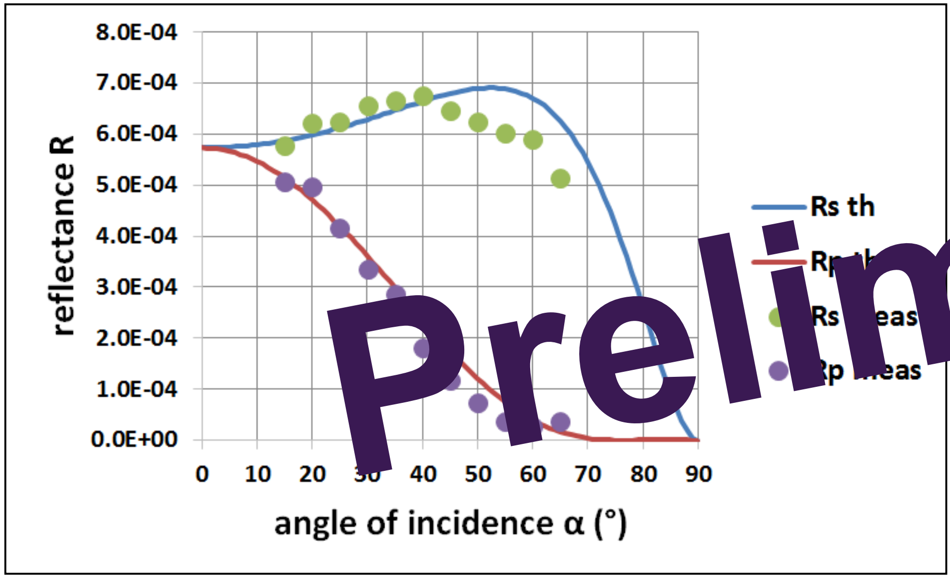


$$\begin{cases} n_2 = (1.3 \pm 0.1) \\ d_2 = (14.0 \pm 1.9)nm \end{cases}$$

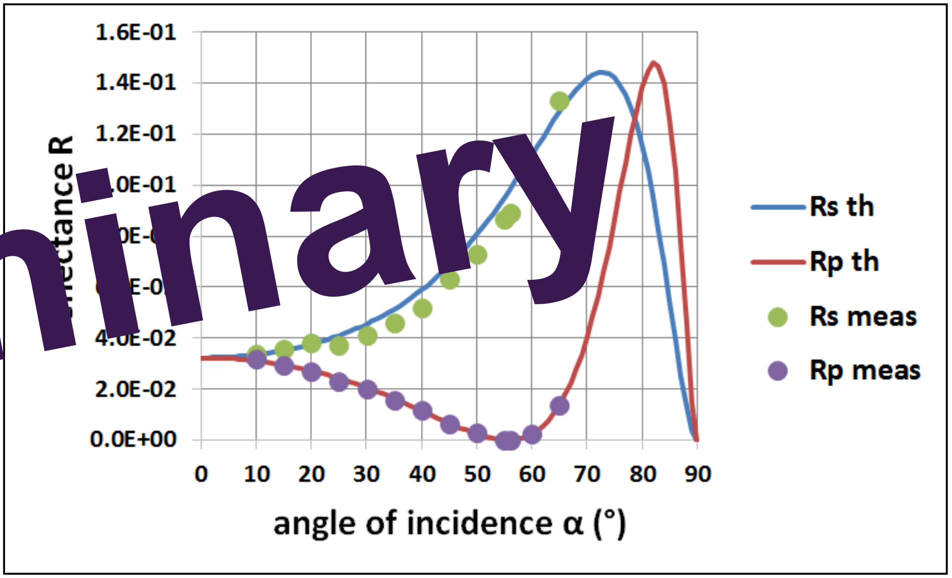
The thickness of bond can be made **less than tens of nm** for precision applications.
(Gwo – US Patent 6284085B1 and 6548176B1).

# Analysis: bond and back surface

bond surface



back surface



The reflectivity of bond is **< 7.00·10$^{-4}$**.
(Sinha et al. - Opt. Exp. 15 (2007): **< 7.08·10$^{-4}$**)

$$\begin{cases} n_2 = (1.3 \pm 0.1) \\ d_2 = (14.0 \pm 1.9) nm \end{cases}$$

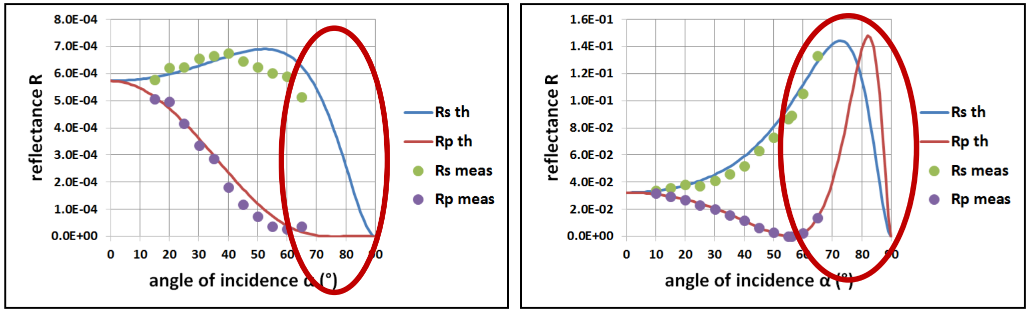
# Conclusions

- There is a reasonable **agreement** between measurements and model **up to 65 degrees** angle of incidence.

- If the model is confirmed, it will be a **powerful tool** to:
  - measure the **bond thickness in a non-destructive manner**;
  - predict and measure **how the reflectivity of a bond can be altered by the chemistry of a different bonding solution**.

- In this silica sample:
  - reflectivity of bond is **< 0.1%**;
  - bond refractive index is **(1.3±0.1)**;
  - bond thickness is **(14.0±1.9)nm**.

# Future work

- 

  → **new sample**

  (ø50.0 × 5.0)mm

- **For cross-check:**

  **refractive index of the bond** → refractive index matching liquids sold by Cargille Laboratories

  **thickness of the bond** → using SEM or AFM microscopy techniques

- **Homogeneity** across a bond and **reproducibility**

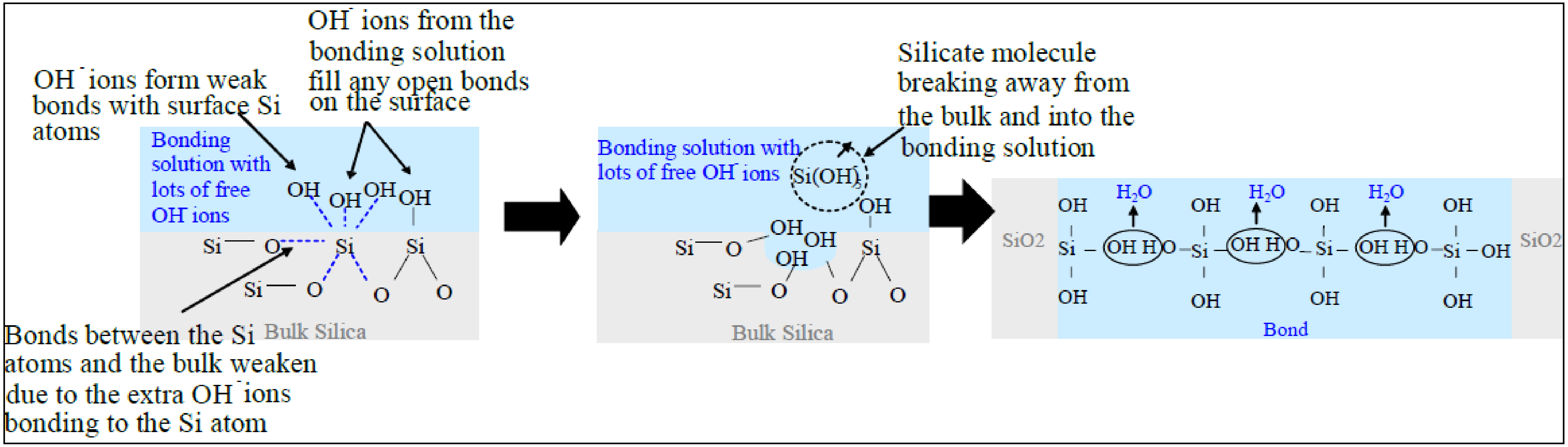
- **Three sapphire samples bonded with sodium aluminate, sodium hydroxide and sodium silicate solution** → knowledge of how the reflectivity of the bond varies as a function of the chemistry of the bonding solution

# Thank you for your attention!

# Fabrication of bonds

The process of hydroxide-catalysis bonding can be summarized in the following steps:

- **hydration**
- **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 breaking away from the bulk and into the bonding solution

# Analysis: formulae

$$\begin{cases} r_{01,\perp} = \dfrac{\cos\alpha - \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}}{\cos\alpha + \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}} \\ r_{01,\parallel} = \dfrac{\left(\dfrac{n_1}{n_0}\right)^2 \cos\alpha - \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}}{\left(\dfrac{n_1}{n_0}\right)^2 \cos\alpha + \sqrt{\left(\dfrac{n_1}{n_0}\right)^2 - (\sin\alpha)^2}} \end{cases}$$

$$\begin{cases} r_{12,\perp} = \dfrac{\cos\beta - \sqrt{\left(\dfrac{n_2}{n_1}\right)^2 - (\sin\beta)^2}}{\cos\beta + \sqrt{\left(\dfrac{n_2}{n_1}\right)^2 - (\sin\beta)^2}} \\ r_{12,\parallel} = \dfrac{\left(\dfrac{n_2}{n_1}\right)^2 \cos\beta - \sqrt{\left(\dfrac{n_2}{n_1}\right)^2 - (\sin\beta)^2}}{\left(\dfrac{n_2}{n_1}\right)^2 \cos\beta + \sqrt{\left(\dfrac{n_2}{n_1}\right)^2 - (\sin\beta)^2}} \end{cases}$$

$$\delta_2 = \frac{2\pi}{\lambda} n_2 d_2 \cos\gamma = \frac{2\pi}{\lambda} n_2 d_2 \cos\arcsin\left(\frac{n_0}{n_2}\sin\alpha\right) = \frac{2\pi}{\lambda} n_2 d_2 \sqrt{1 - \left(\frac{n_0}{n_2}\sin\alpha\right)^2}$$

$$\beta = \arcsin\left(\frac{n_0}{n_1}\sin\alpha\right) \qquad \gamma = \arcsin\left(\frac{n_0}{n_2}\sin\alpha\right)$$

# How $n_2$ can be estimated

## Refractive index of mixed solutions

**sodium silicate solution**:

$Na_2O$ ~ 10.6% + $SiO_2$ ~ 26.5% + $H_2O$ ~ 62.9%

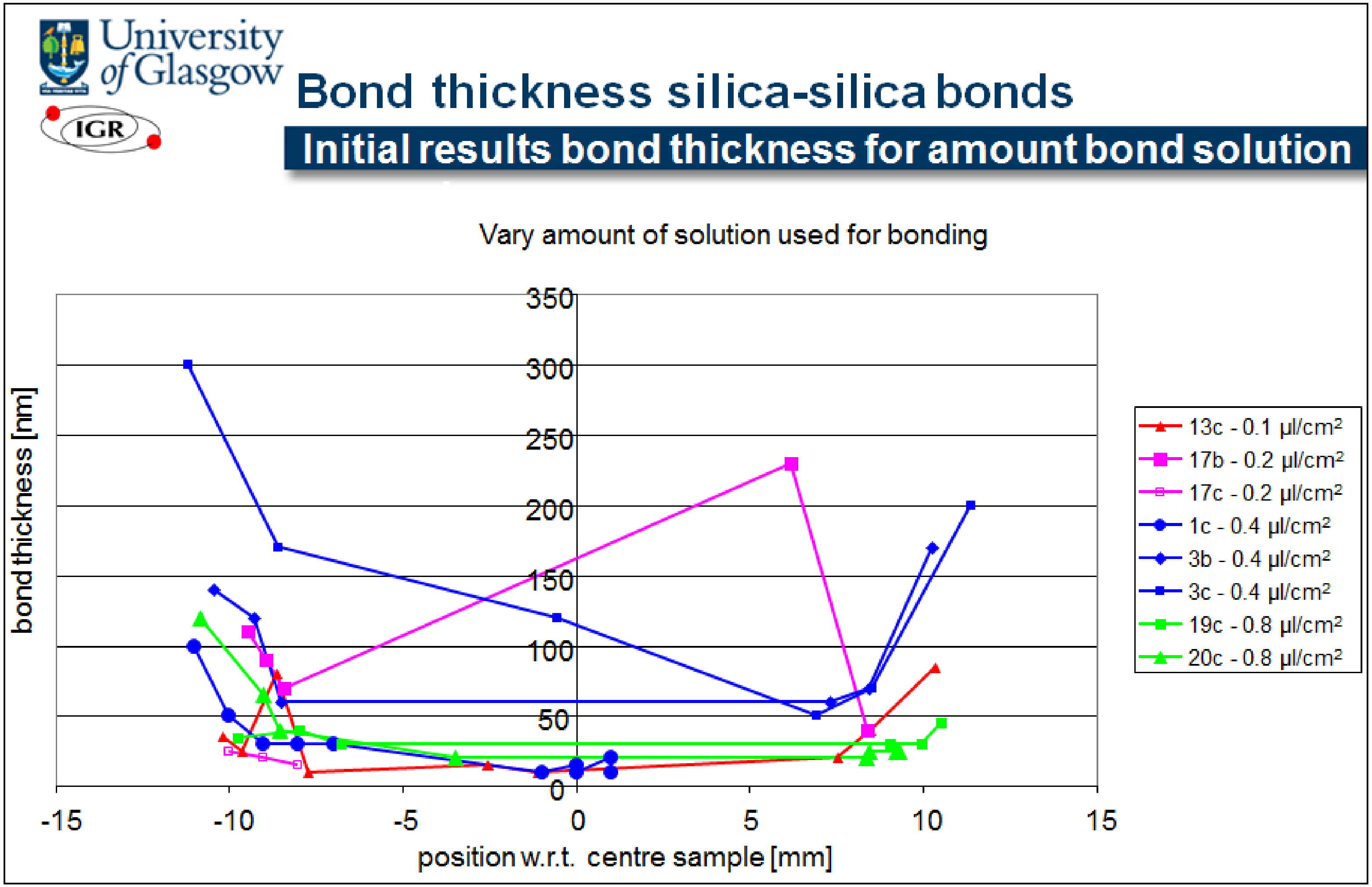
$$\left| \begin{aligned} & 26.5 \cdot 1.55 + 62.9 \cdot 1.33 = 89.4 \cdot x \\ & x = \frac{26.5 \cdot 1.55 + 62.9 \cdot 1.33}{89.4} = 1.39 \end{aligned} \right.$$

**bonding solution**:

2 ml of sodium silicate solution + 12 ml of DI water

$$\left| \begin{aligned} & 2 \cdot 1.39 + 12 \cdot 1.33 = 14 \cdot x \\ & x = \frac{2 \cdot 1.39 + 12 \cdot 1.33}{14} = 1.34 \end{aligned} \right.$$

# Measurements of $d_2$



## Bond thickness silica-silica bonds

**Initial results bond thickness for amount bond solution**

Vary amount of solution used for bonding

M. van Veggel (IGR group meeting - 2012)