Metrological open issues in GeNS measurements

Francesco Piergiovanni*
on behalf of the VCR&D Metrology RL

*Università di Urbino - INFN Firenze

GWADW Online 2021

The Gentle Nodal Suspension

GeNS is currently **the most used technique** in **coating loss angle** measurements

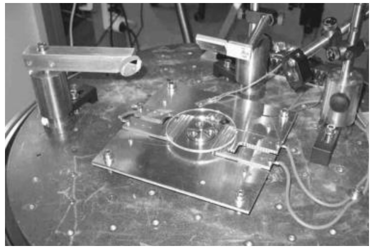
E. Cesarini et al., **A gentle nodal suspension for measurements of the acoustic attenuation in materials**, REVIEW OF SCI. INSTRUM. (2009) , vol.80, 5

"…relevant parameters of the suspension can be measured and eventually varied, giving the experimentalist the possibility to identify whether the measured loss is limited by the suspension system in use or it is an intrinsic property of the sample under investigation."

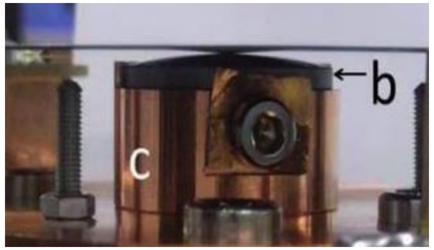
Typical repeatability:

$\approx 10\%$ for $\phi \approx 10^{-6} - 10^{-8}$ (substrates)

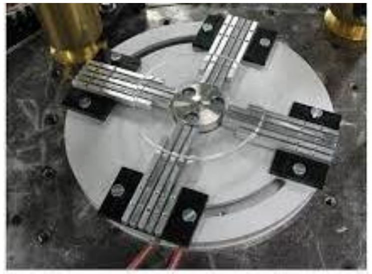
$\approx 5\%$ or better for $\phi \approx 10^{-4} - 10^{-5}$ (coated samples)



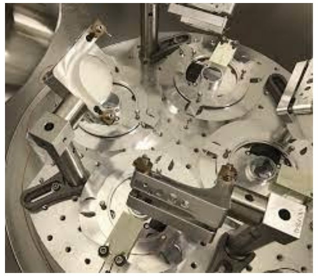
First GeNS in Florence



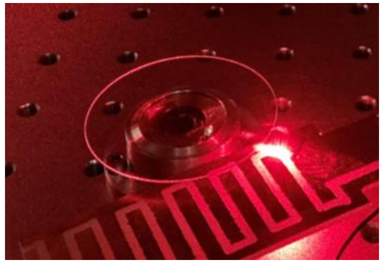
Cryo-GeNS at LMA



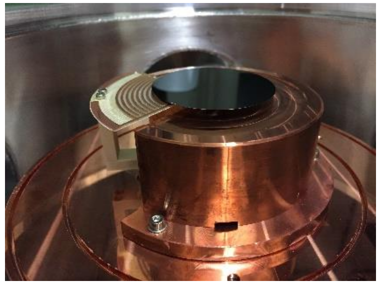
Univ. of Roma-ToV



Quad-GeNS at Caltech



GeNS at Urbino Univ.



Cryo-GeNS at ILM

…and…

- cryogenic GeNS at Caltech
- GeNS at Syracuse University
- at IGR in Glasgow
- in Maastricht
- in Perugia

…and maybe others

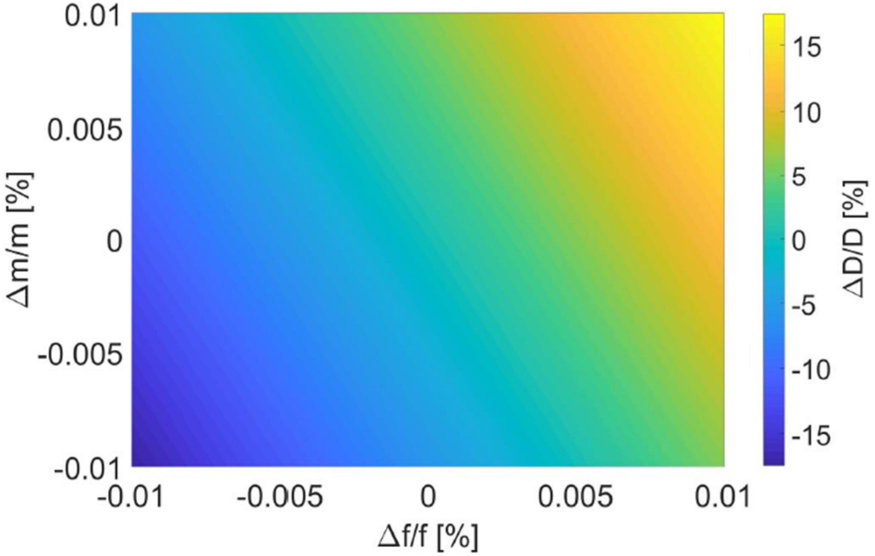
Metrological issue: Frequency shift vs temperature

Issue:

- Dilution measurement accuracy: **the role of temperature**

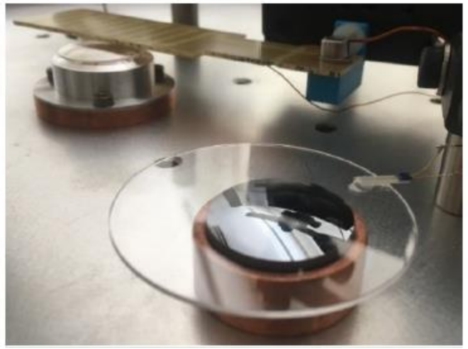
$$D = 1 - \left(\frac{f_0}{f}\right)^2 \left(\frac{m_0}{m}\right)$$

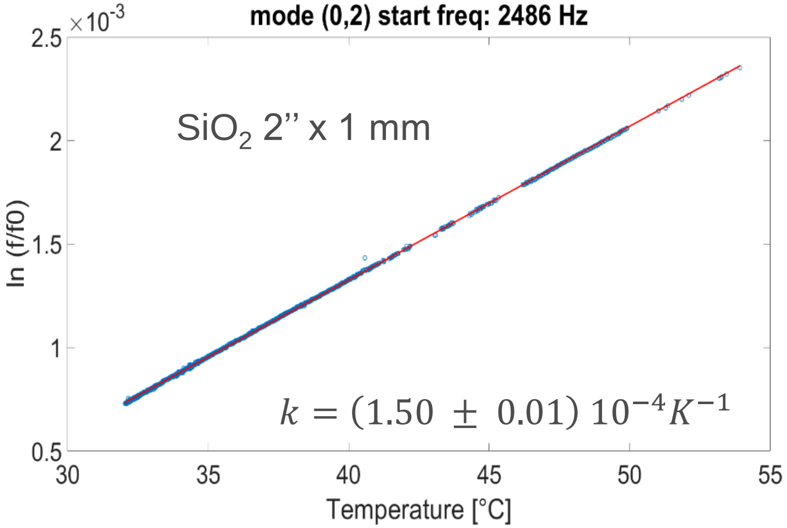
Strong assumption: the mass variation (m_0 ; m) and the frequency variation (f_0 ; f) are only due to the coating deposition.



$$f \sim \sqrt{E} = f_0 e^{\frac{kT}{2}}$$

$$\frac{1}{E}\frac{dE}{dT} = \mathrm{k}$$





Solution:

- Precise measurement of the temperature, by a twin sample
- Correction of the frequency values by using the estimated value of k

- See talk M. Bischi: "Optical and mechanical characterization of ion-beam-sputtered MgF2 and AlF3 thin films"
- Granata et al. P2100113

Metrological issue: Frequency shift vs curvature

Issue:

- **One-side coating** produces a **curvature** on the sample, inducing a spurious **frequency shift** -> systematic error in dilution factor

Coating ↑

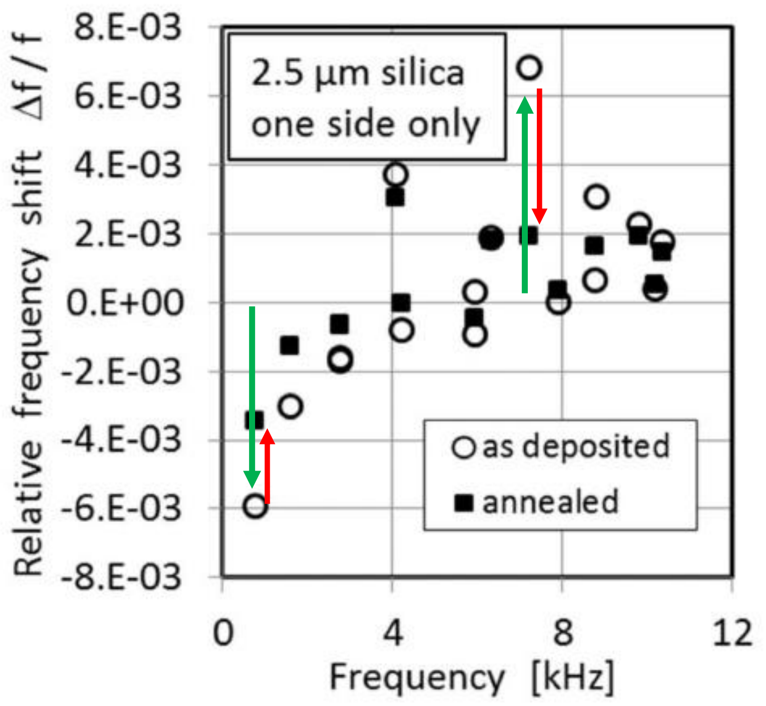
Butterfly modes goes down

Mixed modes goes up

Annealing ↑

Butterfly modes goes up

Mixed modes goes down



Granata, M., et al. *Archives of Metallurgy and Materials* 60 (2015).

Solution:

- **Coating** deposition in **both sides.**

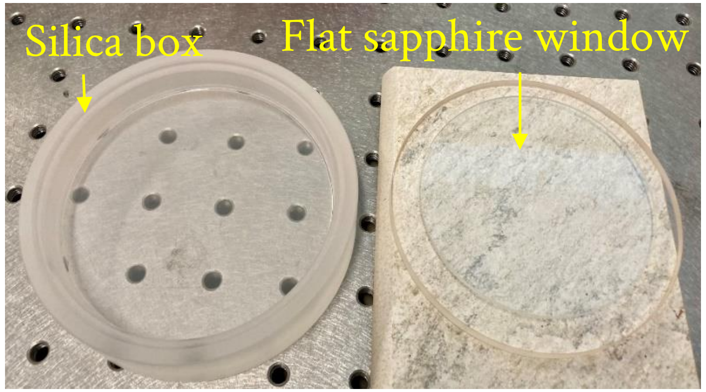
Metrological issue: Frequency shift vs curvature/annealing

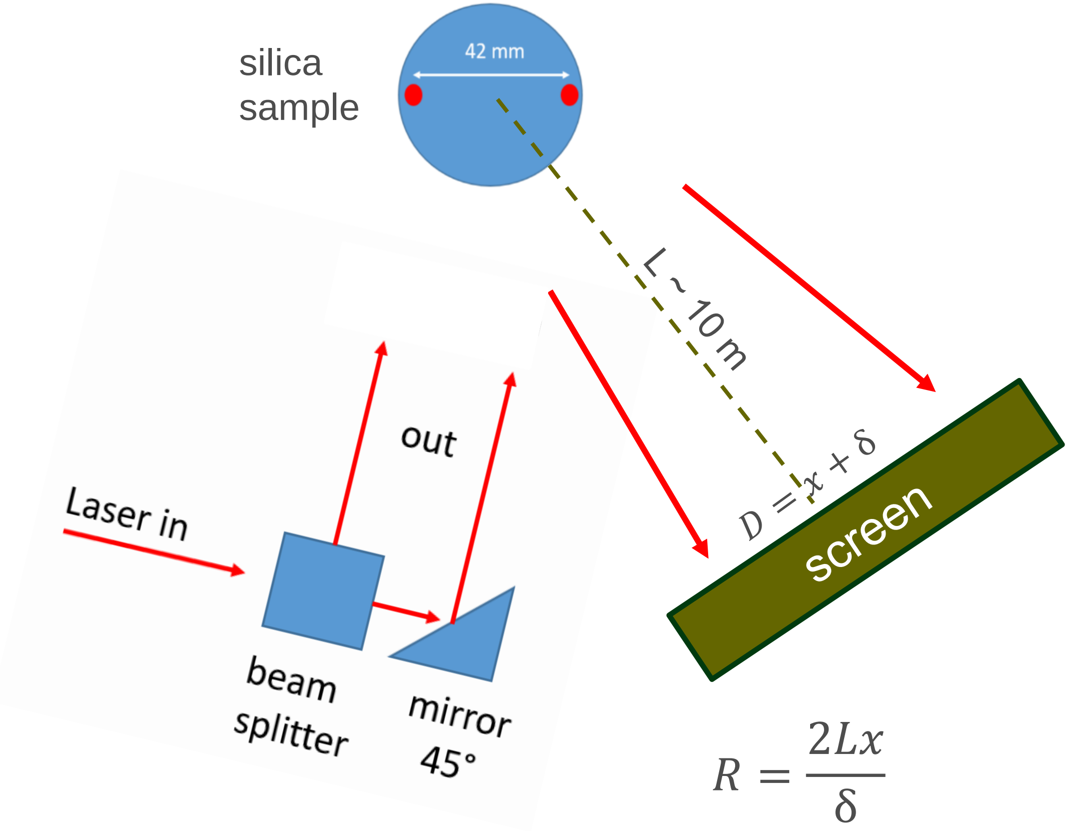
Issue:

- **Annealing** can change sample **curvature**, and produces **frequency shifts**

Experiments in Urbino, Roma ToV, LMA, ILM

- Annealing of a 2”, 0.1mm substrate for 20 h at 900 °C, T ramp (up and downwards) = 100 °C/h





$$R = \frac{2Lx}{\delta}$$

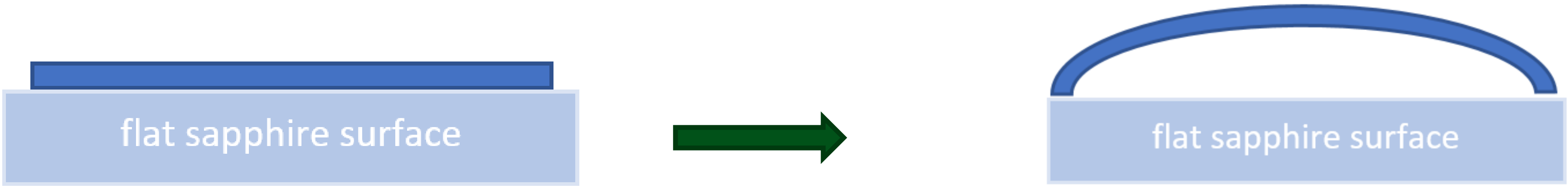
Metrological issue: Frequency shift vs curvature/annealing

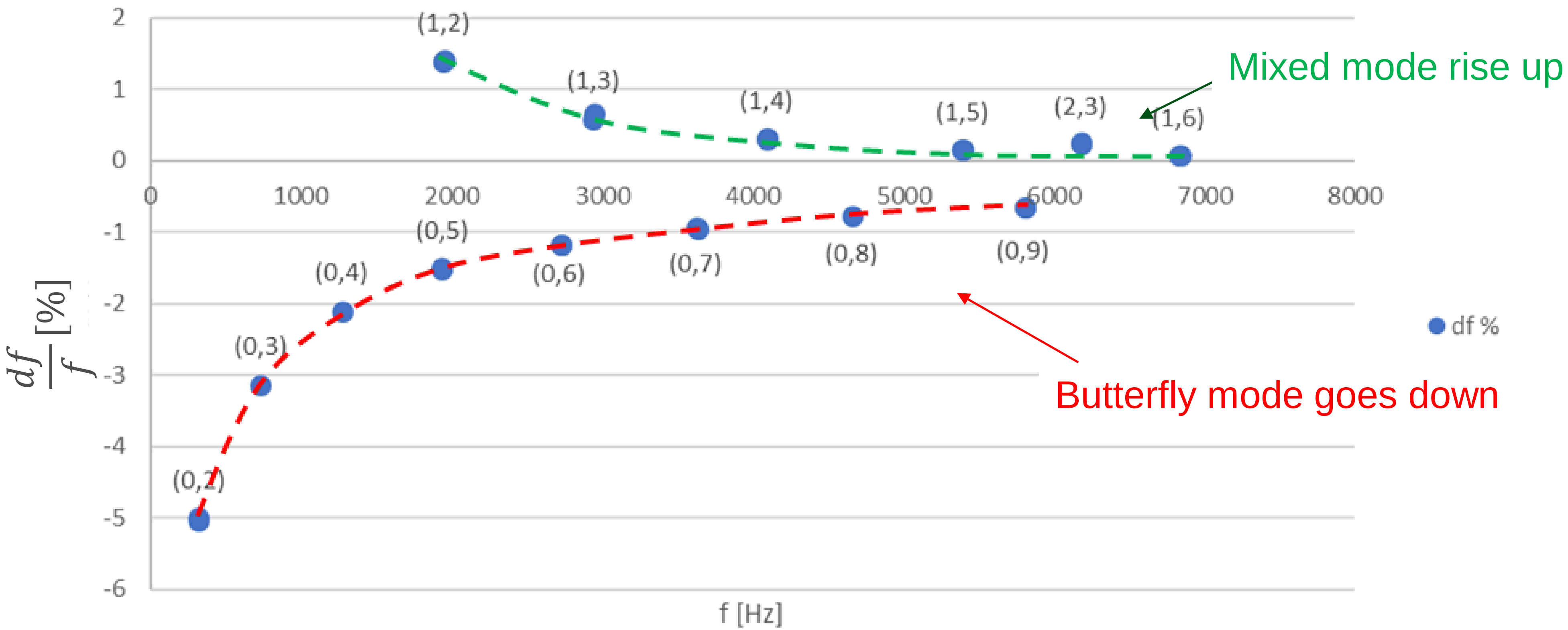
Initial situation (Before Annealing):

The sample is **nearly flat.**

Final situation (After Annealing):

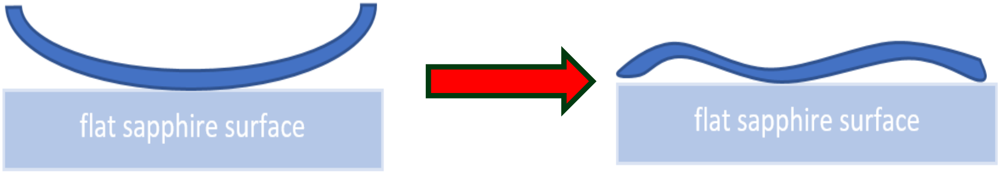
The sample is **curved**, like a dome with a radius of curvature: $R \sim (3.5 \pm 0.4)m$





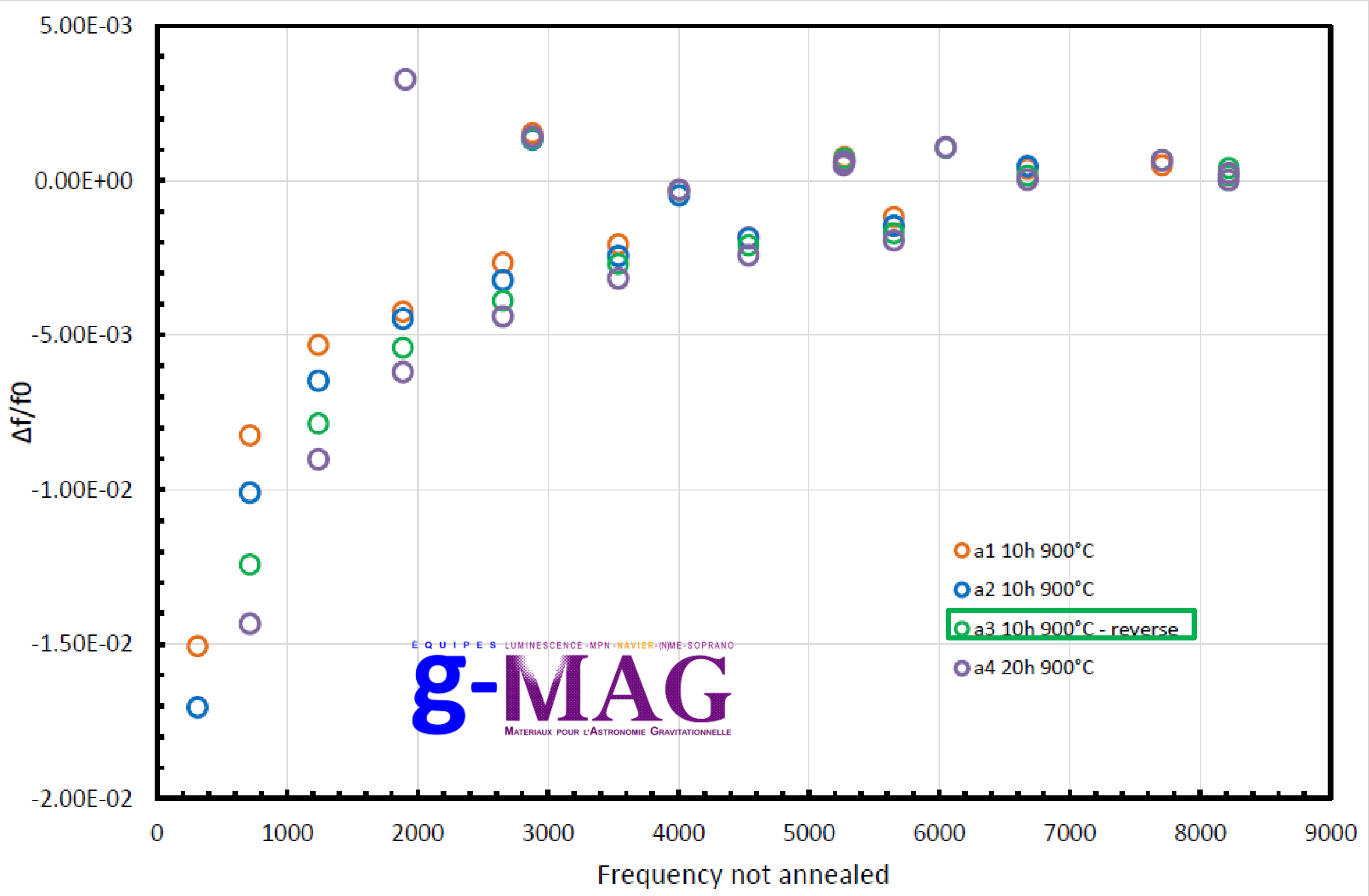
Metrological issue: Frequency shift vs curvature/annealing

After a further annealing of the **flipped** sample global curvature is cancelled (irregularities are still present)



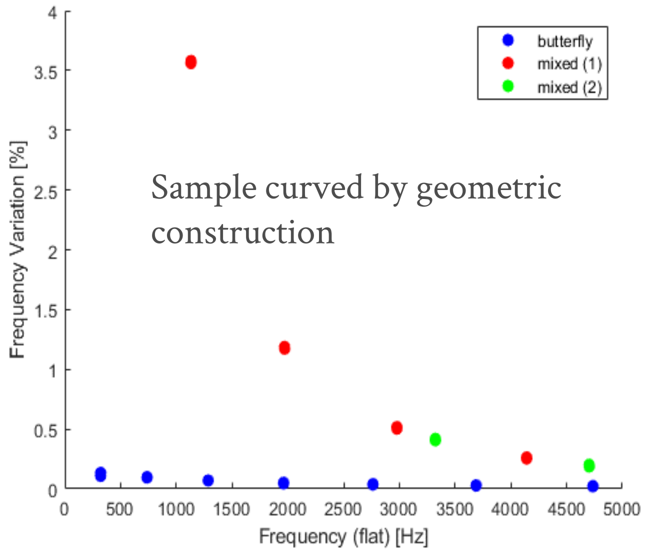
But:

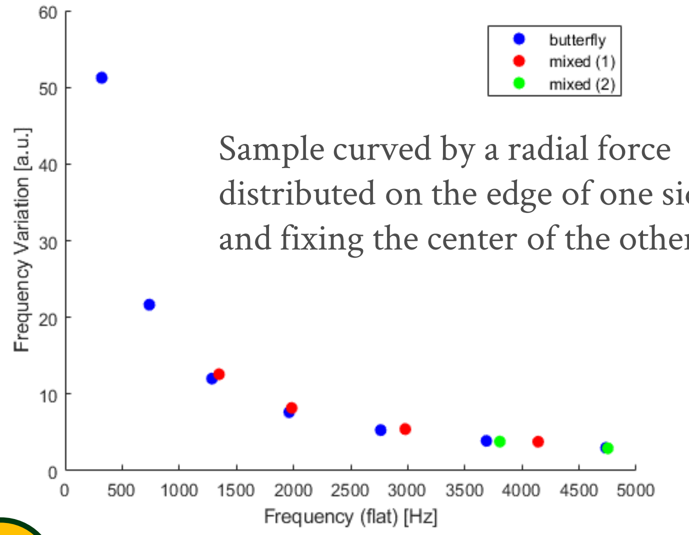
Each new annealing induce the **same** effect on the mode frequencies, regardless the sample is **flipped** or not

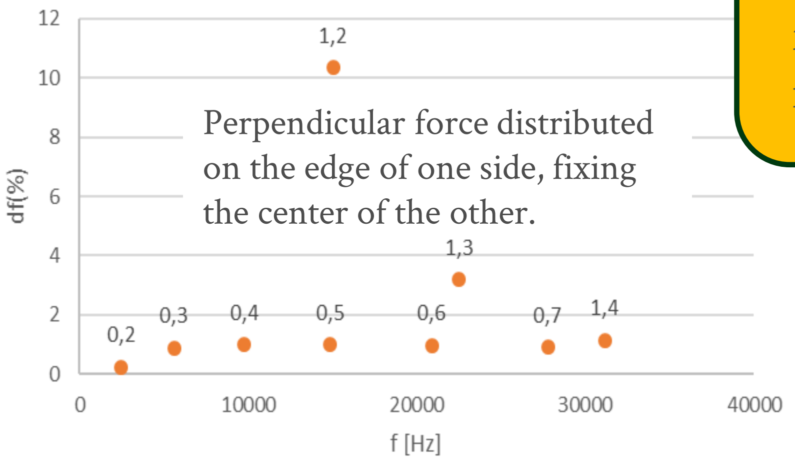


Metrological issue: Frequency shift vs curvature/annealing

Different **FEA** simulations of curved samples, seems **to do not reproduce** the observed frequency shifts







All FEMs give an increment of the frequencies for all the modes

Next steps:

- Try to avoid curvature
- Investigate on annealing effects on shear/bulk parameters

Metrological issue: Thermoelastic shift

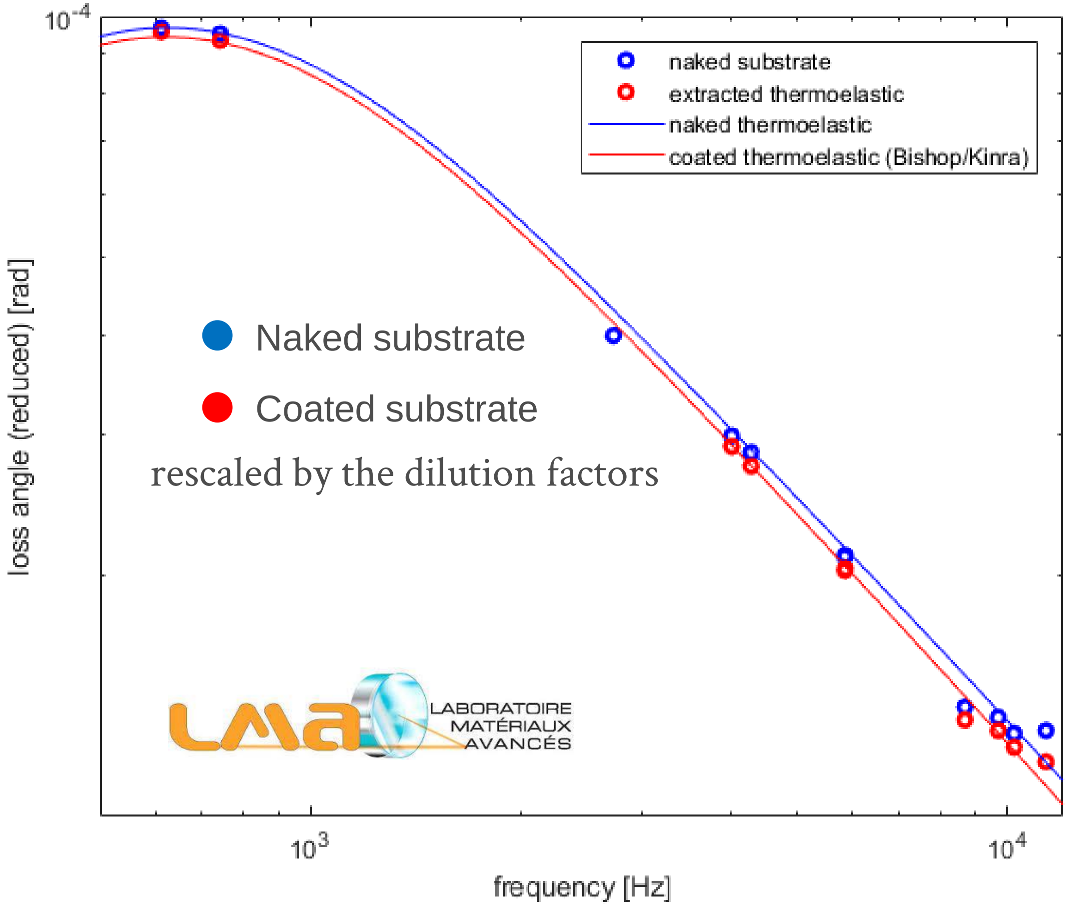
Issue:
Coating deposition influences the thermoelastic dissipation of the substrate

- Dissipation of **substrates** (disks) for cryogenic measurements (**silicon / sapphire**) is dominated by **thermoelastic damping** for most of the temperature range

- Silicon disc 0.468 mm thick, with 1.11 μm SiO2 coating on each surface
- Room temperature measurements

Loss angles are **reduced** by the presence of the **coating**

Not expected



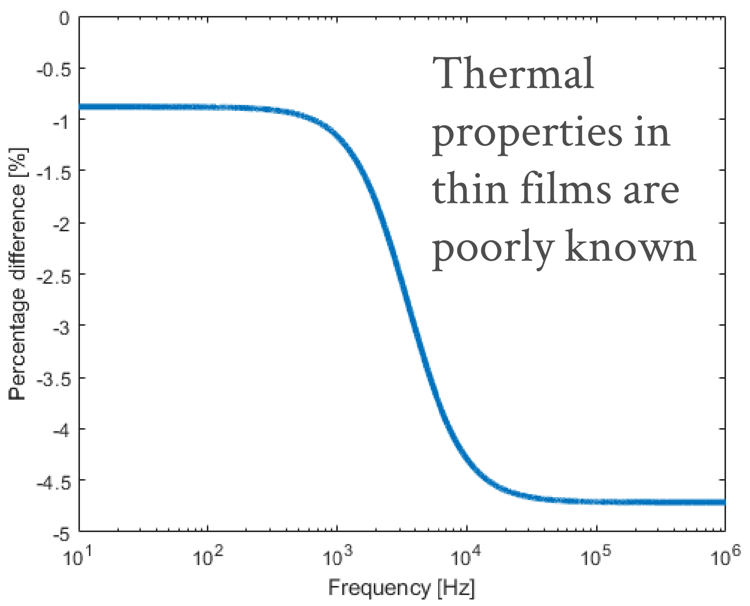
Metrological issue: <u>Thermoelastic shift</u>

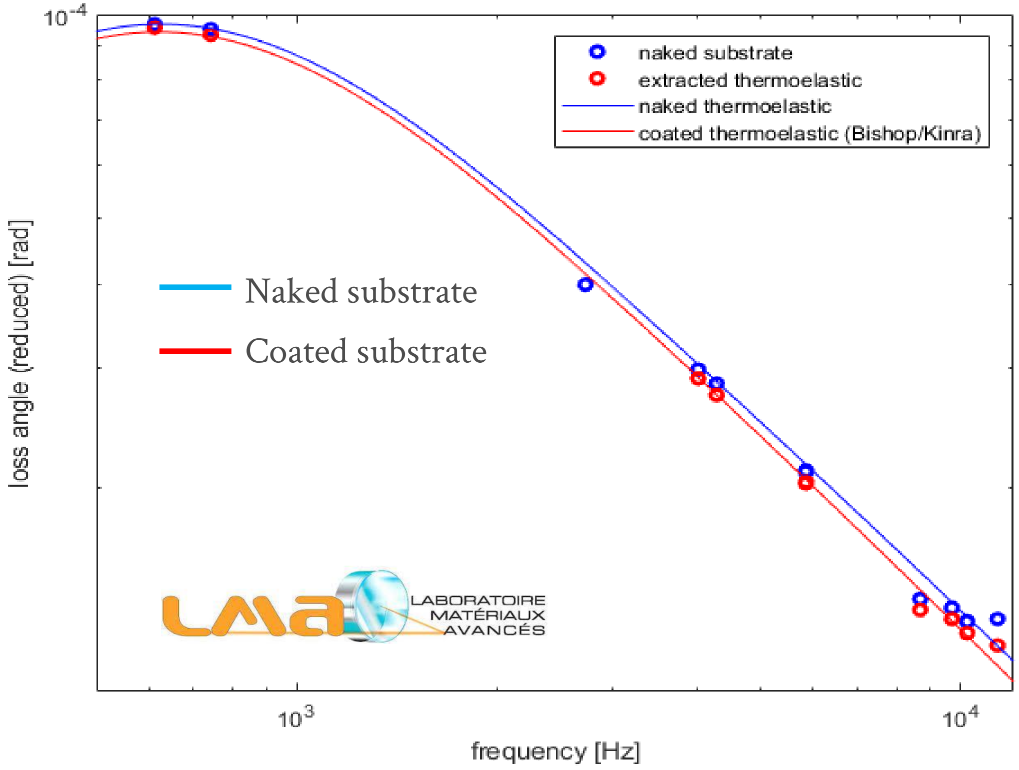
The thermoelastic dissipation of the substrate changes by the presence of the coating

- TED of coated samples is modeled as a series of Debye peaks

J. E. Bishop and V. K. Kinra, Comp. Mater. Struct. 3 83-95 (1996)
S. Vengallatore et al., Jour. of Micromech. and Microen. (2005)

- It depends on the thermo-mechanical properties of the coating (thermal conductivity, linear thermal expansion, specific heat)





Cesarini et al. VIR-0128A-18

Fabrizi et al. VIR-0271A-21 - G2100541

Metrological issue: Thermoelastic shift

Naked sample

t = 0

Coating sample

t (coating thickness)

$$\phi_{meas}(0) = \phi_S^{intr} + D^{TE}(0)\phi_S^{TE}(0)$$

$$\phi_{meas}(t) = \big(1 - D_C(t)\big)\phi_{meas}(0) + \Delta\phi^{TE}(t) + D_C(t)\phi_C^{intr}$$

Thermoelastic shift

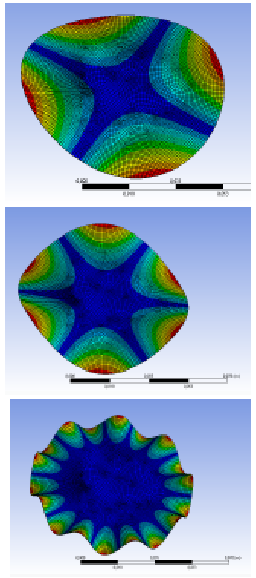
We have two unknown quantities

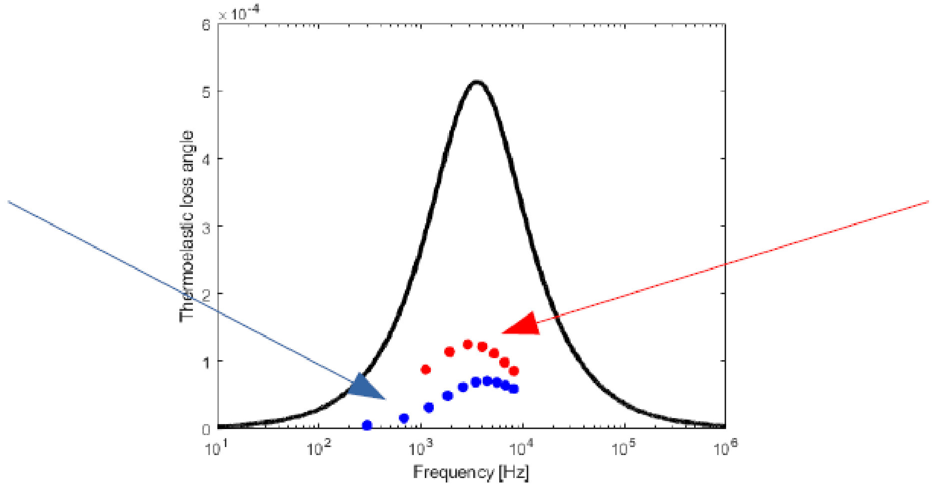
We need two equations!

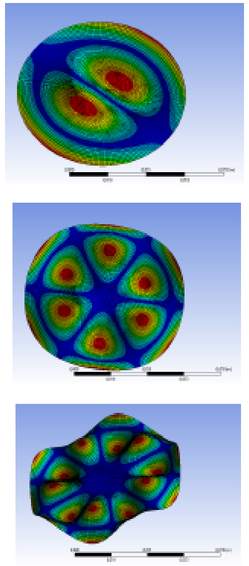
Metrological issue: Thermoelastic shift

Possible experimental approach: exploit mode shape and frequency dependence

(F. Fabrizi, F. Piergiovanni)







butterfly modes

mixed modes

The factor D^{TE} is highly dependent on the mode especially for modes of different families

$$\phi_{meas}(t;m1) = \big(1 - D_C(t,m1)\big)\phi_{meas}(0) + \Delta\phi^{TE}(t;m1) + D_C(t;m1)\phi_C^{intr}$$

$$\phi_{meas}(t;m2) = \big(1 - D_C(t,m2)\big)\phi_{meas}(0) + \Delta\phi^{TE}(t;m2) + D_C(t;m2)\phi_C^{intr}$$

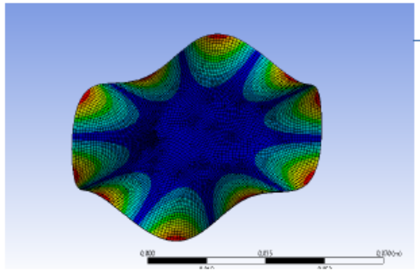
Now we have three unkowns, but we can write a relation between $\Delta\phi^{TE}(t;m2)$ and $\Delta\phi^{TE}(t;m1)$

Metrological issue: Thermoelastic shift

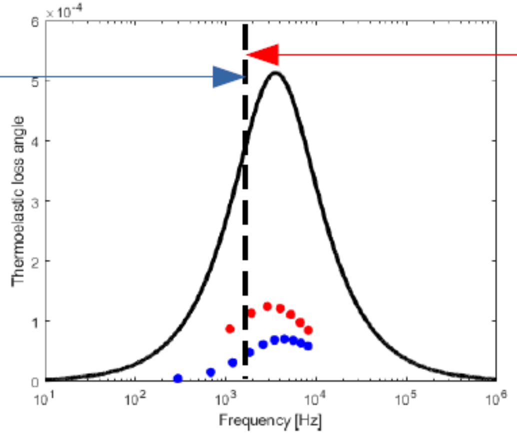
Possible experimental approach: exploit mode shape and frequency dependence

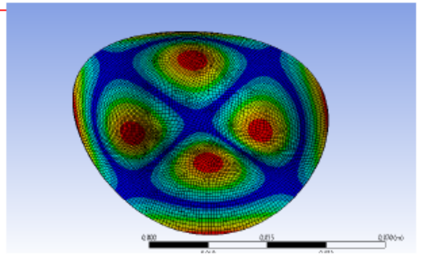
We can select two modes:

- as close in frequency as possible
- as different as possible in D^{TE}



Butterfly mode (0, 5)





Mixed mode (1, 2)

If we suppose $D^{TE}(t;m) \approx D^{TE}(0;m)$ then:

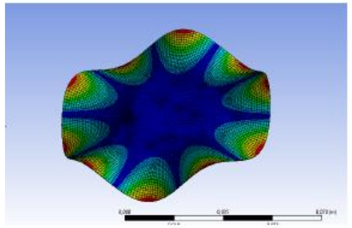
$$\frac{\Delta\phi^{TE}(t;m2)}{\Delta\phi^{TE}(t;m1)} = \frac{\big(1 - D_C(t,m2)\big)D^{TE}(m2)}{\big(1 - D_C(t,m1)\big)D^{TE}(m1)}$$

We have the third equation!

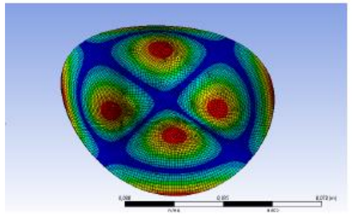
- D_C are measured
- D^{TE} can be found G. *Cagnoli et al., Phys. Lett. A 382 (2018)*

Metrological issue: Thermoelastic shift

$$\phi_{meas}(t;m1) = \big(1 - D_C(t,m1)\big)\phi_{meas}(0) + \Delta\phi^{TE}(t;m1) + D_C(t;m1)\phi_C^{intr}$$



$$\phi_{meas}(t;m2) = \big(1 - D_C(t,m2)\big)\phi_{meas}(0) + \Delta\phi^{TE}(t;m2) + D_C(t;m2)\phi_C^{intr}$$



- We have assumed the coating loss angle to be the same for the two modes, but for modes of different families the contributions of bulk and shear will be different

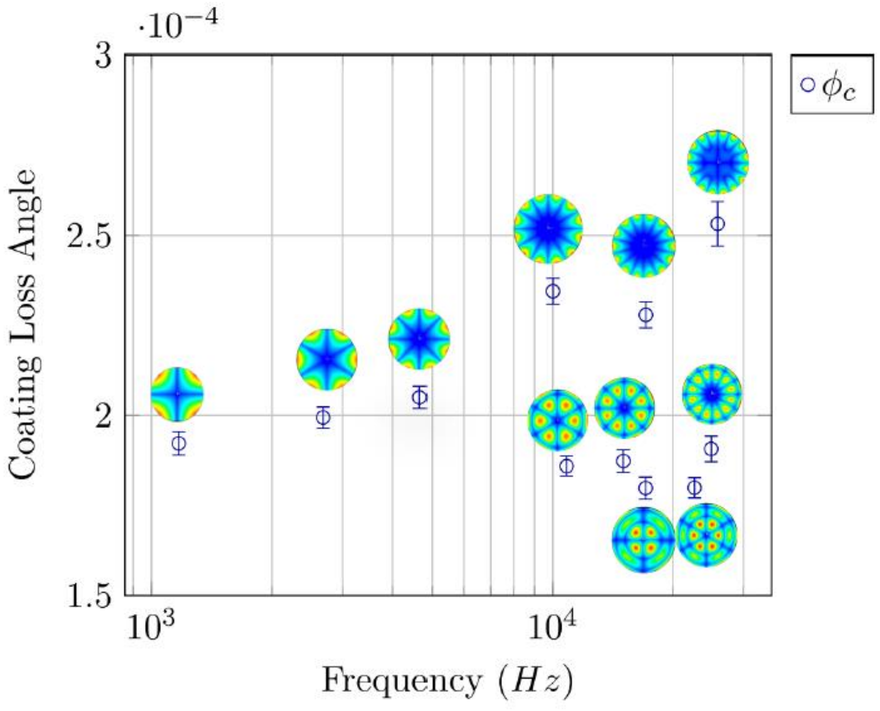
- Conversely, any measurement hoping to determine bulk and shear losses on silicon substrates must contend with the difference in thermoelastic background

Possible systematic in bulk/shear disentangle

Metrological issue: Coating edge effect

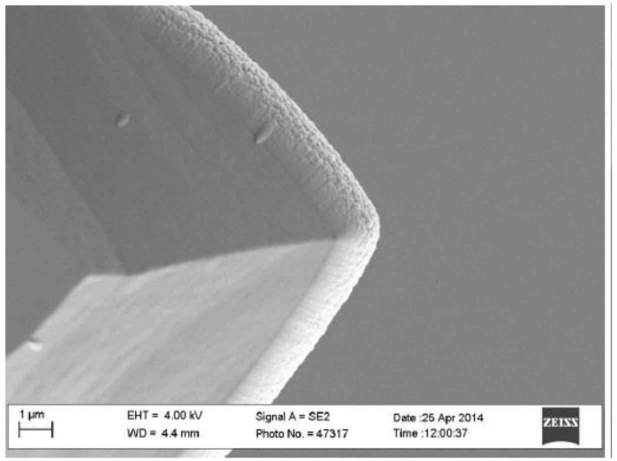
Mode families observed in silica coating measurement

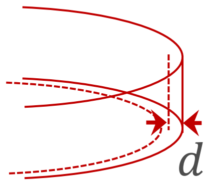
A. Amato G1801791



Spurious contribution coming from the edge of the disks, which was also coated during deposition (masking the edge would have induced uncontrolled, undesired shadowing effects on the main surfaces) .

M Granata *et al* 2020 *CQG* **37** 095004

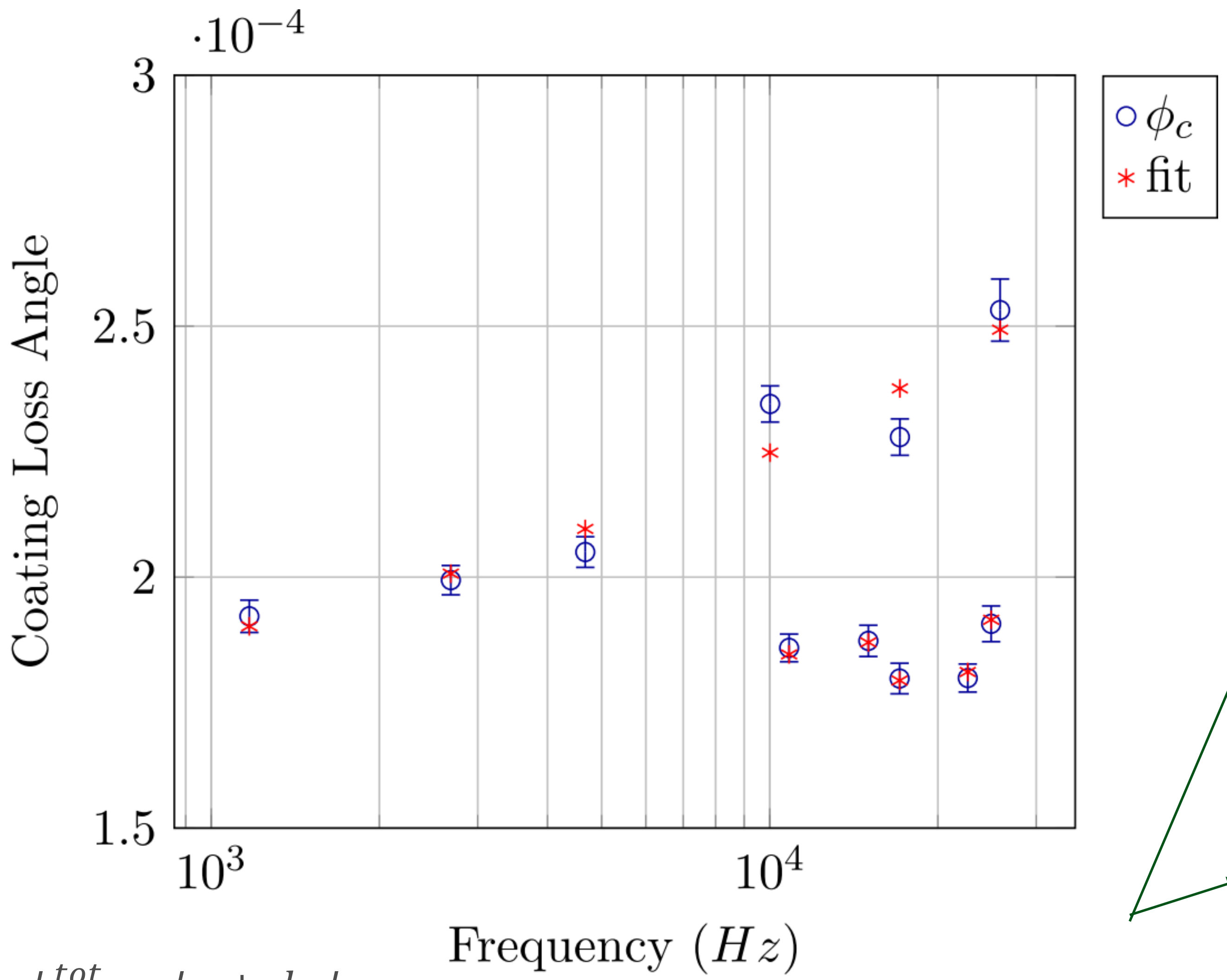


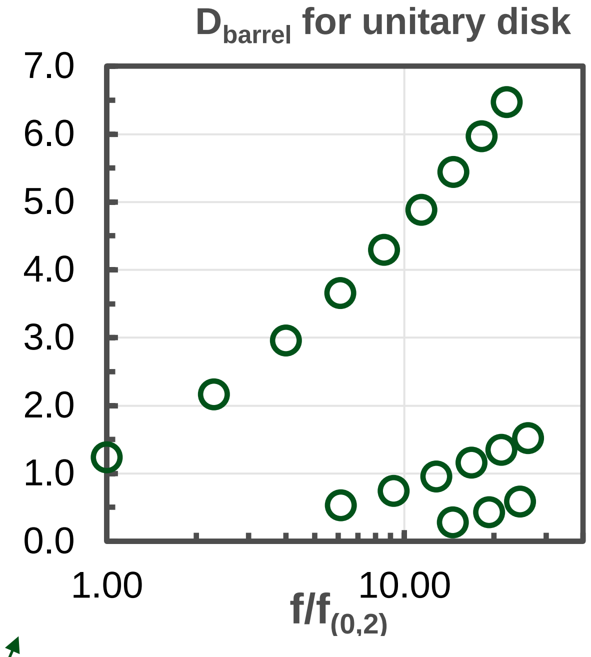


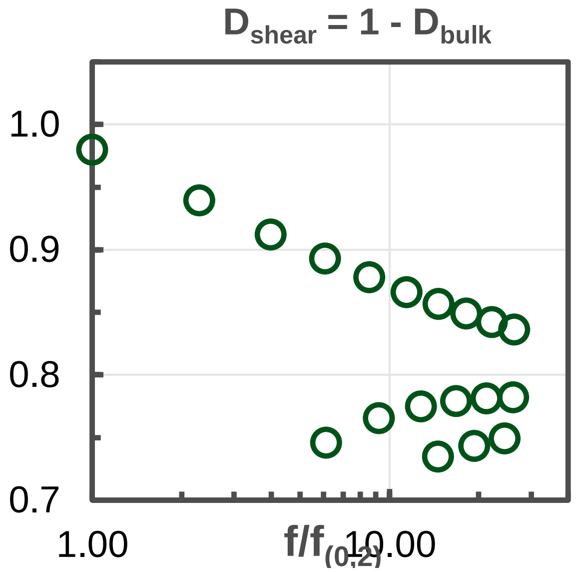
$$\phi_C^{tot} = \phi_C + d\epsilon\phi_B$$

$$\epsilon = \lim_{d\to 0} \frac{1}{d}\frac{E_{barrel}}{E_{tot}}$$

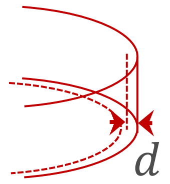
Metrological issue: Coating edge effect







$$\phi_C^{tot} = \phi_C + d\epsilon\phi_B$$



$$\epsilon = \lim_{d \to 0} \frac{1}{d} \frac{E_{barrel}}{E_{tot}}$$

- If we admit a frequency-dependent loss angle, it is difficult to discriminate the two contributions

Conclusions

- GeNS has proven to be a powerful instrument/technique for mechanical spectroscopy
- Measurement protocols for cleaning and sample preparation are well established
- Some issues have been solved as aging, substrate edge effects, temperature effects…
- Some issues are still open:
  - ✓ Frequency shifts induced by annealing and their connection with the sample curvature
  - ✓ The possible systematics caused by coating edge inhomogeneities, in particular for measuring bulk and shear loss angle separately.
  - ✓ Substrate thermoelastic shift given by coating deposition adds an unknown to cryogenic measurement