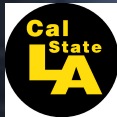


# Status of Nano-layered Coating Developments



**Riccardo DeSalvo & the INFN AdCOAT Team**

*Cal State University Los Angeles, Uni-Sannio, INFN, LVC and KAGRA*



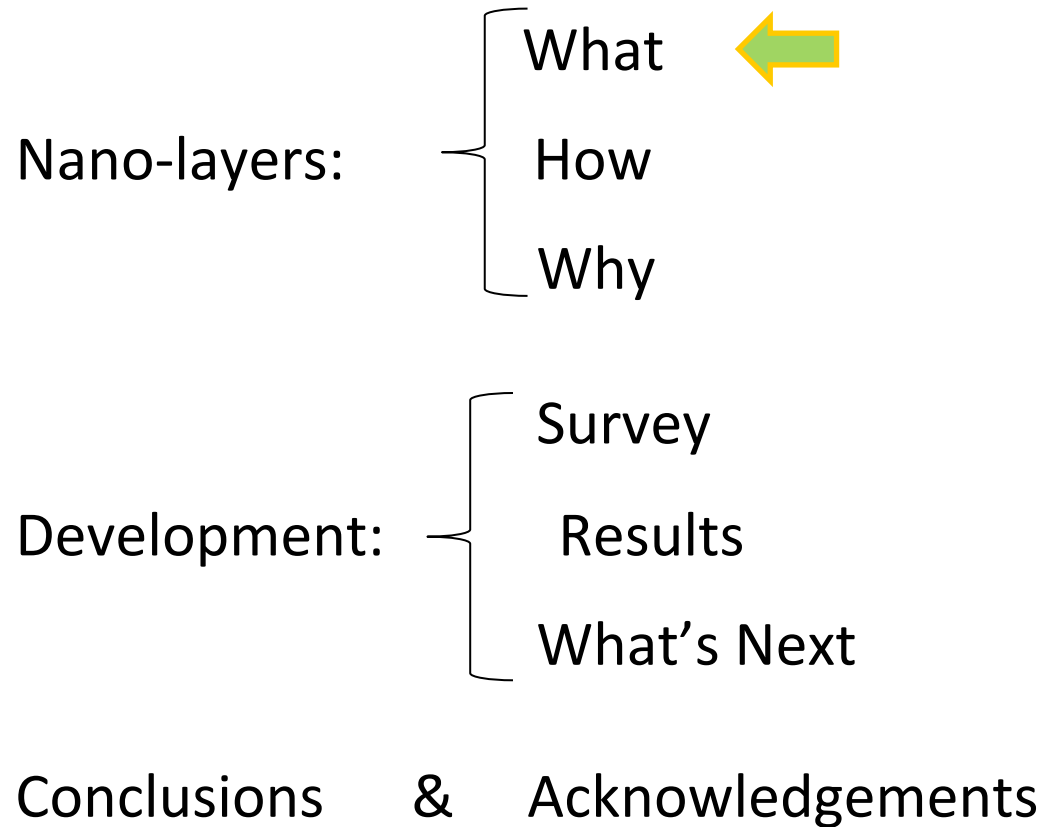
**Shiuh Chao, Huang-Wei Pan, Ling-Chi Kuo**

*National Tsing-Hua University, Taiwan (ROC) and LVC*



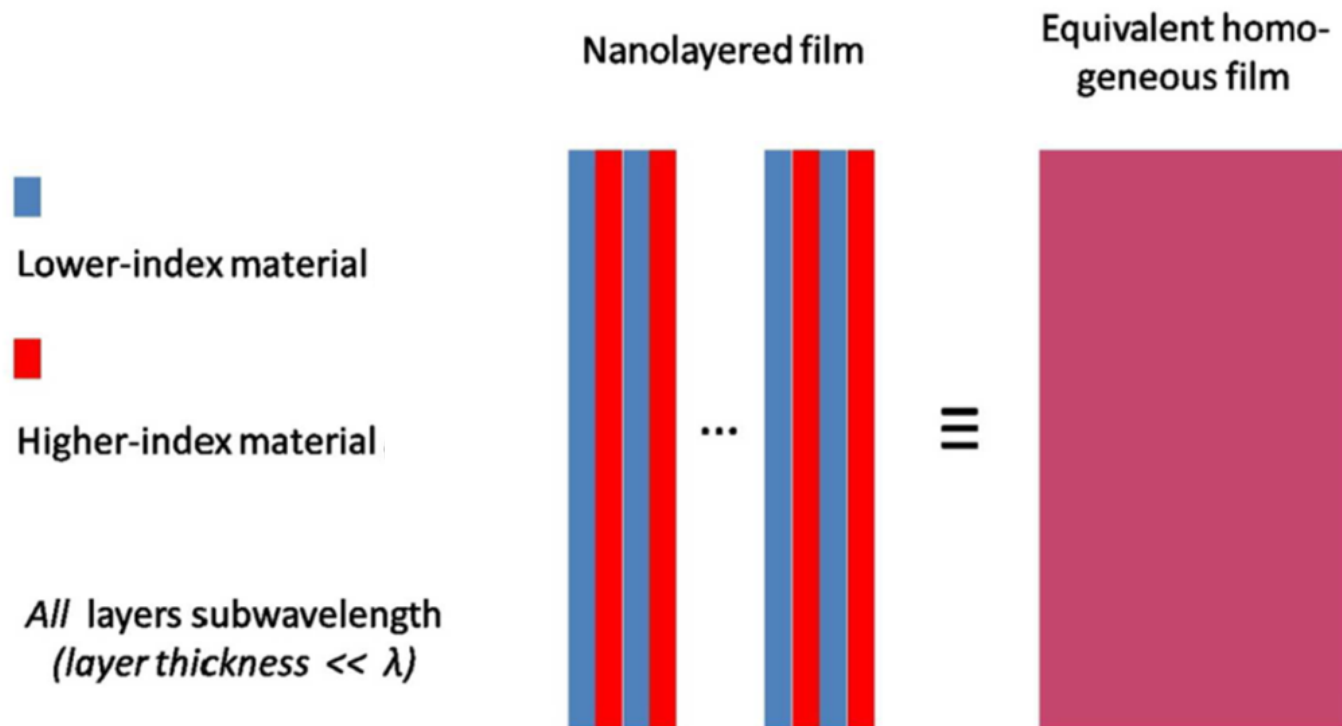
# Outlook

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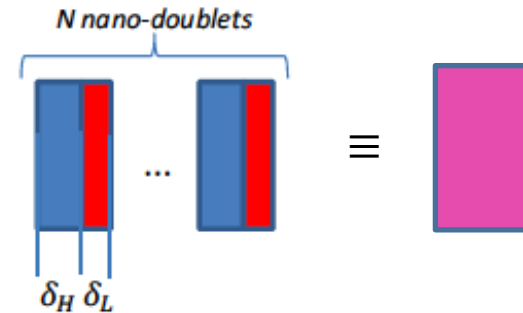
# Sub-Wavelength Nano-layers

Stratified composites, whose properties depend in a *very simple way* on the constituents' properties and the thickness ratio



# Nano-layer Prototypes

Consist of nominally identical **cascaded nano-doublets**, and are thus specified by  $(N, \delta_H, \delta_L)$ .



Can adjust refractive index by changing the nano-layer thickness ratio

Can adjust the super-layer optical thickness by adjusting the number of layers

Example of segmentation of a  $\frac{1}{4}$  wavelength (qwl.) super-layer

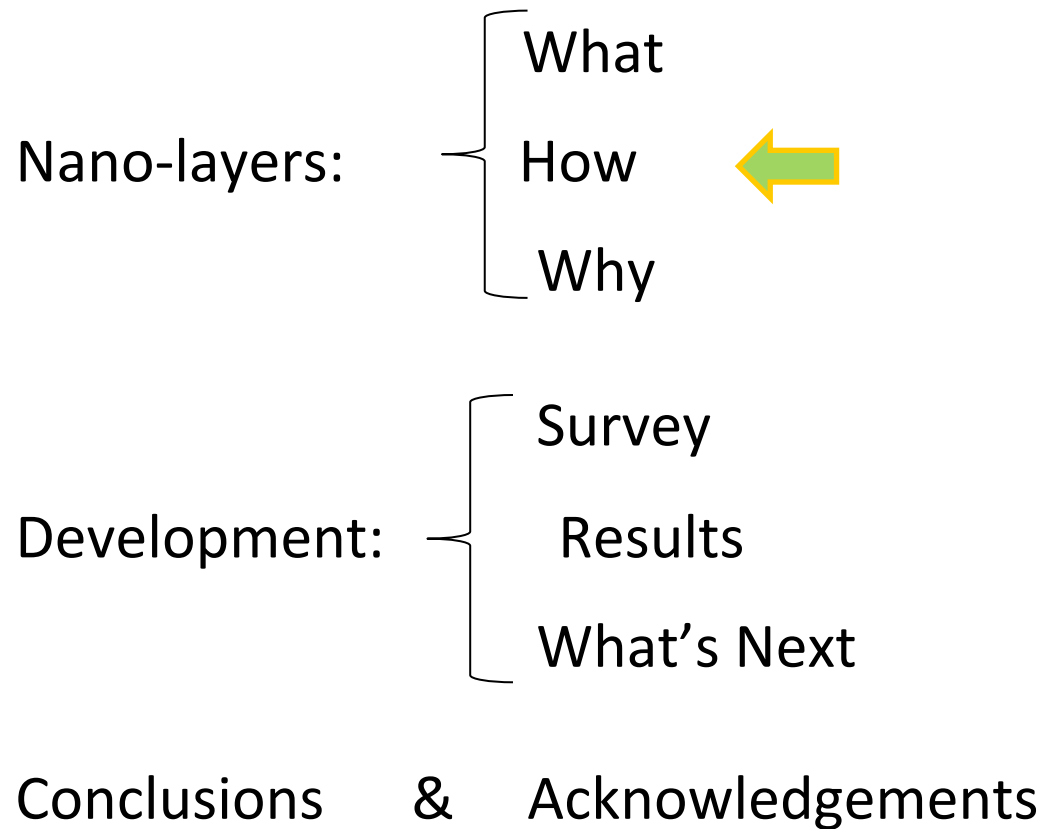


**Equivalent  $\text{TiO}_2/\text{SiO}_2$  subwavelength doublet based, QWL thick composites with  $n_{eff}=2.09$**

$N$	$\delta_{\text{TiO}_2}[\text{nm}]$	$\delta_{\text{SiO}_2}[\text{nm}]$	$N$	$\delta_{\text{TiO}_2}[\text{nm}]$	$\delta_{\text{SiO}_2}[\text{nm}]$
1	78.0559	49.2168	14	5.57542	3.51549
2	39.0279	24.6084	15	5.20373	3.28112
3	26.0186	16.4056	16	4.87849	3.07605
4	19.514	12.3042	17	4.59152	2.89511
5	15.6112	9.84337	18	4.33644	2.73427
6	13.0093	8.20281	19	4.1082	2.59036
7	11.1508	7.03098	20	3.90279	2.46084
8	9.75699	6.1521	21	3.71695	2.34366
9	8.67288	5.46854	22	3.548	2.23713
10	7.80559	4.92168	23	3.39373	2.13986
11	7.09599	4.47426	24	3.25233	2.0507
12	6.50466	4.1014	25	3.12224	1.96867
13	6.0043	3.78591			

# Outlook

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# Predecessors

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# X-Ray Nano-layered Coatings

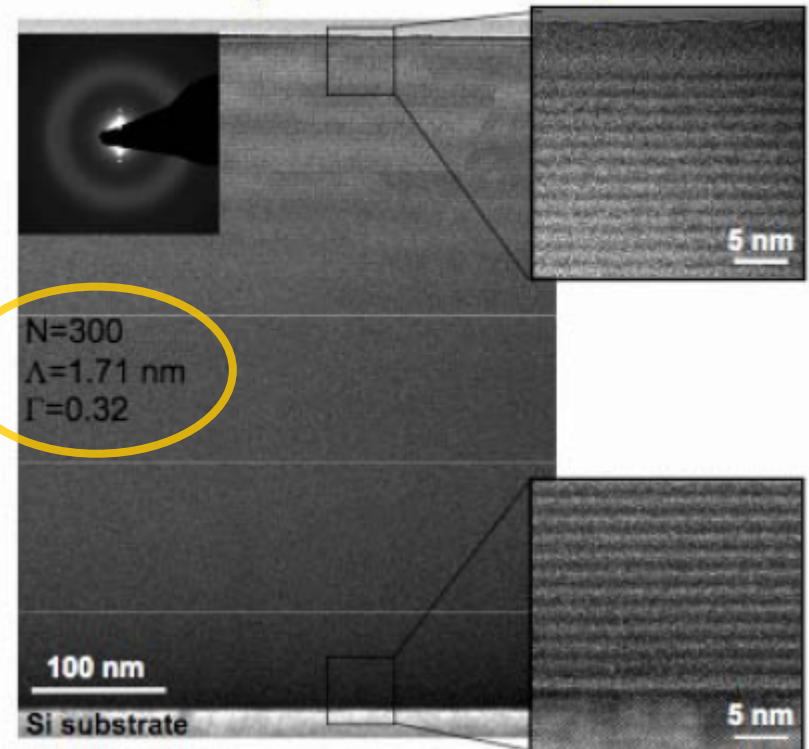
X-ray interference mirrors consisting of hundreds/thousands of nm scale layers, With sub - nm precision [see, e.g., Proc . 10<sup>th</sup> PXRMS Conf. (2008)] , using

- Interleaved nm-scale “buffering” layers prevent to crystallization & maintain flatness [E. Gullikson, Proc. 8<sup>th</sup> PXRMS (2006)]
- Ion assisted (modulated) magnetron sputtering [N. Ghafoor et al., Thin Sol. Films 516 (2008) 982]



Control of stress, crystallite size, and roughness [D.L. Windt, Proc. SPIE (2007) vol 6688]

Interleaved B<sub>4</sub>C- Cr/Sc multilayer

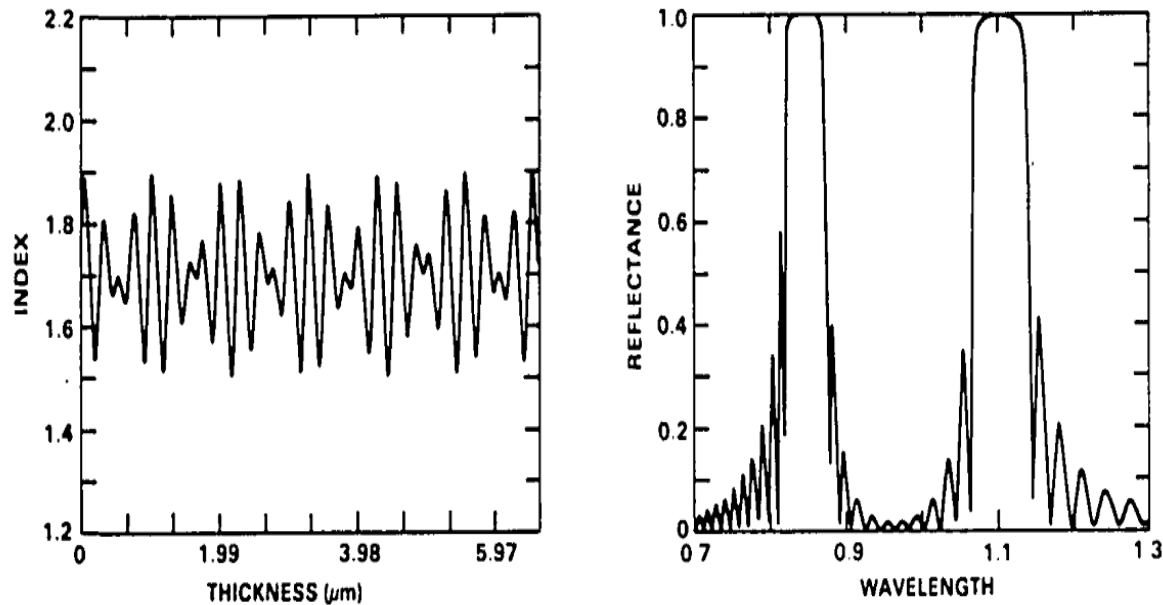


See also [R. DeSalvo, LIGO-G080106] for discussion.

# Rugate Optical Filters

Rugates have been around since long. They use a *continuously varying* index distributions, to synthesize, e.g., a *dichroic response* [see, e.g., W. H. Southwell, Appl. Opt. 24 (1985) 457-460]

*Rugate dichroic mirror coating*



***Mimic a continuous, periodically-changing refractive index function by a staircase of stacked, optically-thin homogeneous layers...***



# Requirements / Technological Challenges

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*relatively large thickness errors  
in the individual low/high index nanolayer thicknesses  
are Irrelevant.*

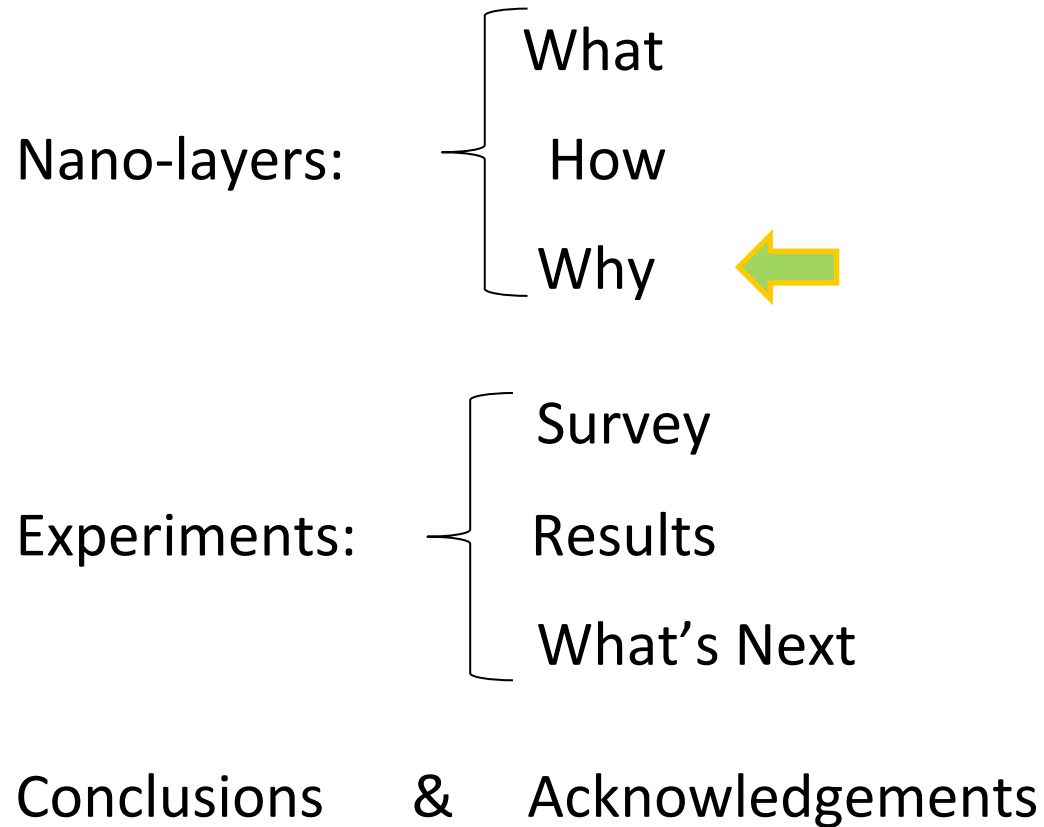
*IF each layer is sub-wavelength and  
total thickness ratio has the design value*

**Are there any other technological issues ?**

*Try and see ...*

# Outlook

---



# Doping Hinders Crystallization

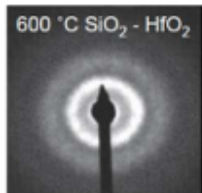
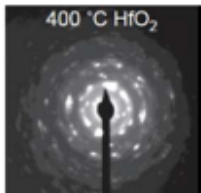
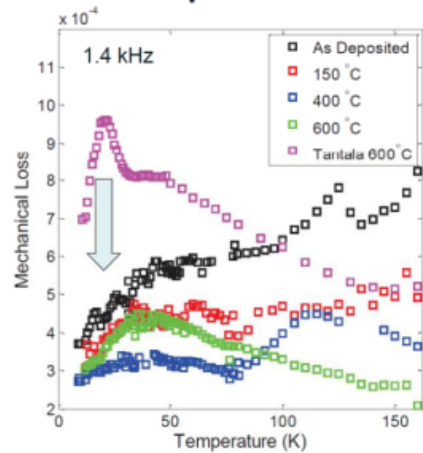
- Silica doping contrasts crystallization [S. Pond, Appl. Optics, 28 (1989) 2800]



30% SiO<sub>2</sub> doped HfO<sub>2</sub>  
(0.5 μm thick on Silicon cantilever)

## Loss measurements of IBS silica-doped hafnia

- Heat treatment reduces loss with 400 °C giving lowest loss
- Heat treated silica-doped hafnia coatings have a loss roughly a factor of 3-4 lower than 600 °C tantala at 20K



[P. Murray, LIGO G-1400275]

ThD2.pdf

© 2007 OSA/OIC 2007

## Investigation of Ion-Beam-Sputtered Silica-Titania Mixtures for Use in Gravitational Wave Interferometer Optics

Roger P. Netterfield, Mark Gross

CSIRO Division of Industrial Physics, P.O. Box 218, Lindfield, NSW 2070, Australia,  
roger.netterfield@csiro.au

**Abstract:** Ion-beam-sputtered mixtures of silica and titania are investigated as potential coating materials for use in gravitational wave interferometer optics. Such coatings must have both low optical and mechanical loss to maximize detection sensitivity.

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OCIS codes: (230.4170) Multilayers; (160.4670) Optical Materials; (240.0310) Thin Films.

$(\lambda/4, \lambda/4)^{15}$	$10^4 \times (\text{residual loss angle})$	$n_H$
Ta <sub>2</sub> O <sub>5</sub> / SiO <sub>2</sub>	$4.4 \pm 0.2$	2.02
TiO <sub>2</sub> :: Ta <sub>2</sub> O <sub>5</sub> / SiO <sub>2</sub> (15::85)	$2.4 \pm 0.2$	2.07
SiO <sub>2</sub> :: Ta <sub>2</sub> O <sub>5</sub> / SiO <sub>2</sub> (35::65)	$2.5 \pm 0.4$	1.83
→ TiO <sub>2</sub> :: Si <sub>2</sub> O <sub>2</sub> / SiO <sub>2</sub> (35::65)	$1.7 \pm 0.4$	1.77

# Thin(ner) Layers ..

... crystallize at *high(er)* annealing temperatures

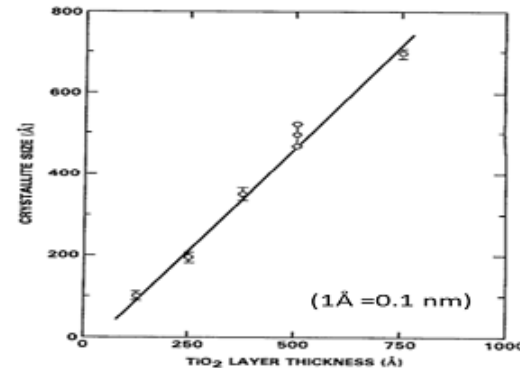
Seminal work on **thin – layer Titania** films by Sankur & Gunning [J. Appl. Phys. 66 (1989) 4747]

“Thinner layers (< 250 Å) required higher temperatures [to crystallize]. 65 Å layer films exhibit diffraction only after annealing at 600°C.”

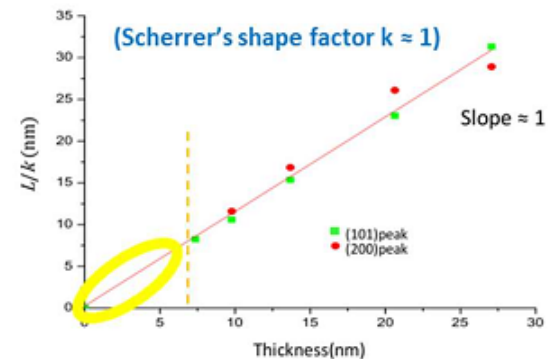
➔ **“Grain size, as deduced from diffraction line broadening, was comparable to the layer thickness”**

“Thicker layers remain in the Anatase phase and never transform into Rutile, even for prolonged (72 h) annealing at the highest temperatures (1100°C). Thinner layers (65 Å) convert into Rutile starting at 900°C”

➔ **“Below a certain critical thickness crystallization in pure TiO<sub>2</sub> films is inhibited”**



[Gluck et al., J. Appl. Phys. 69 (1991) 3037]



[S. Chao et al., LIGO-G1300921]

Fraction of silica can be reduced, thus increasing  $n_{high}/n_{low}$  contrast =>

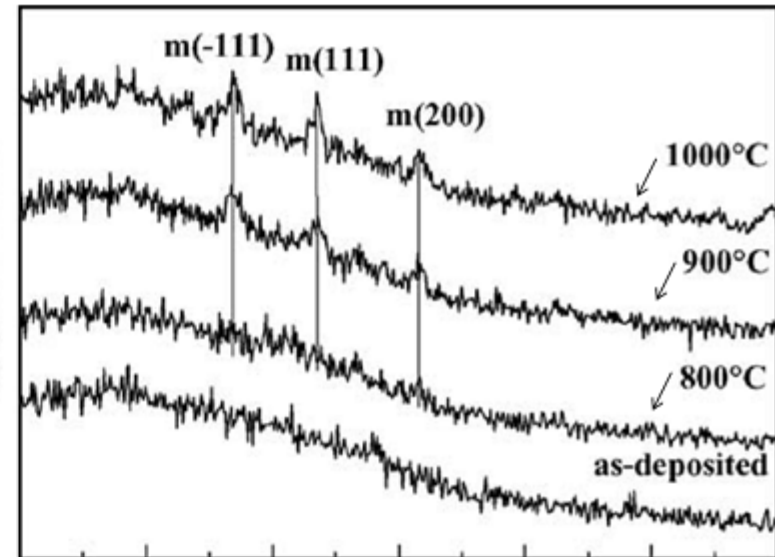
=> For same reflectivity reduce number of layers / thermal noise

# Hafnia-Alumina Nano-layers

**Nanometer-layered Hafnia (12nm)/Alumina (3nm) composites do not crystallize upon annealing, up to temperatures of 800 °C**

[M. Liu et al., Appl. Surf. Sci. 252 (2006) 6206].

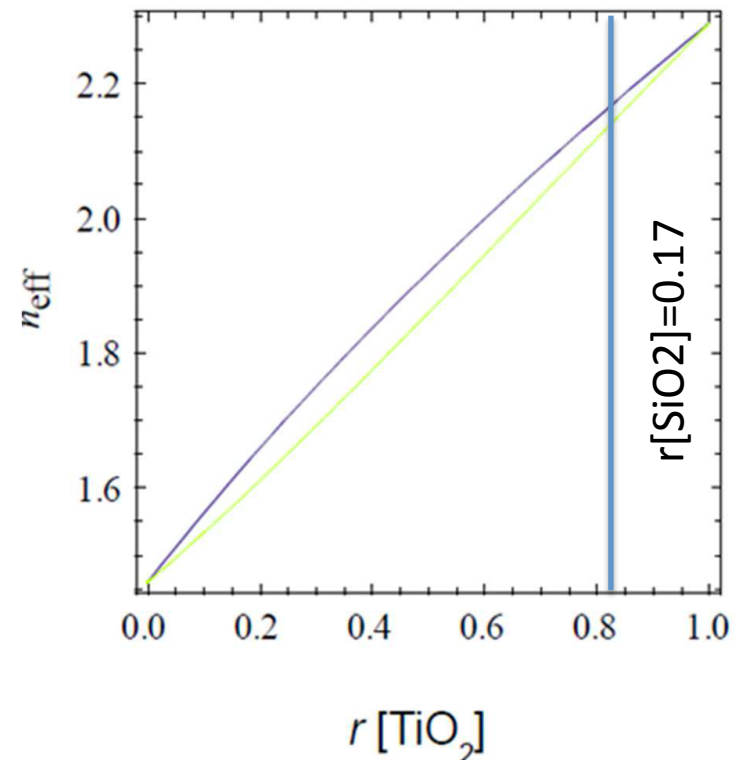
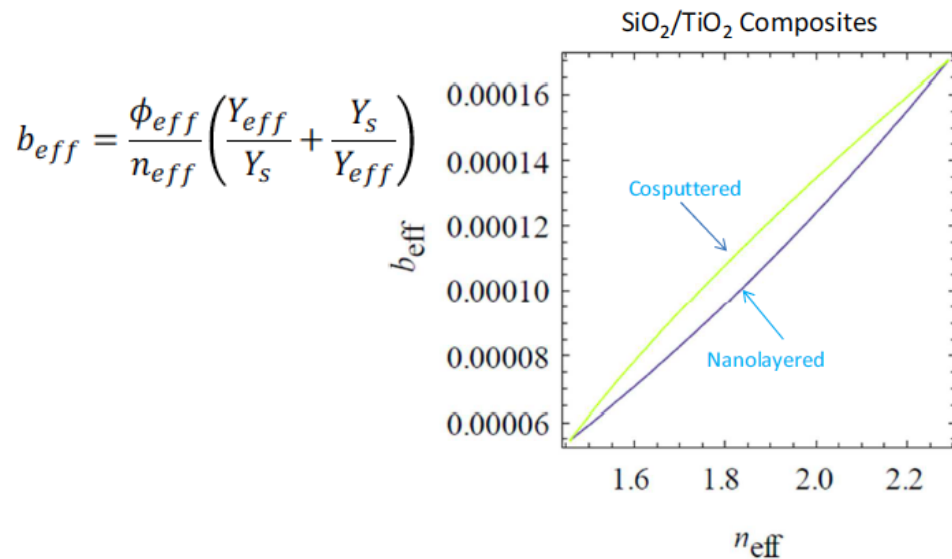
- ➔ “XRD analysis shows that the films remain amorphous up to an annealing temperature of 800 °C”
- ➔ “FTIR indicates that no interface layer forms during annealing up to 800 °C”



Our own results shown later

# Nano-layered vs. Doped

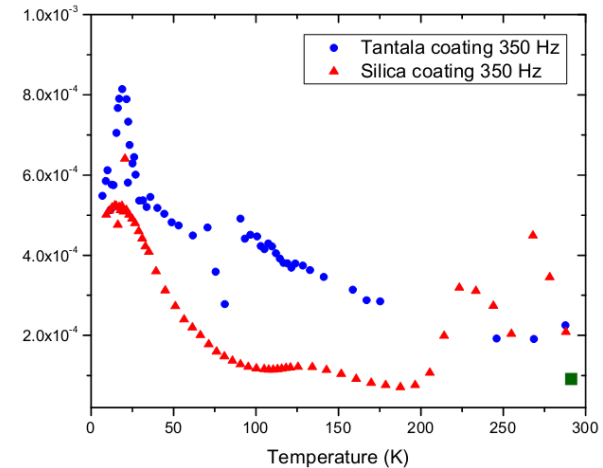
- Nano-layered can be better in terms of noise compared to doped with the same optical density



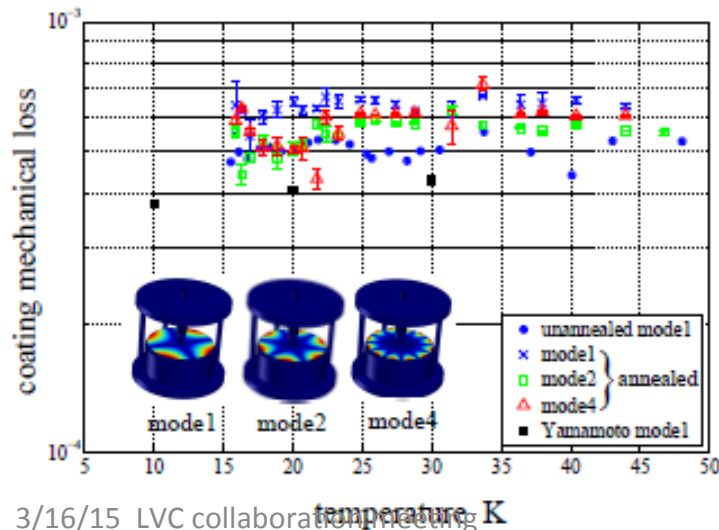
- Nano-layered are much easier modeling compared to doped [Pinto et al., LIGO-G100372]

# The Cryo-Peak Puzzle

Mechanical loss measurements on *single-layer Titania-doped-Tantala and Silica films* show a mechanical loss peak at  $\sim 30\text{K}$  [Martin et al., CQG 25 (2008) 055005]



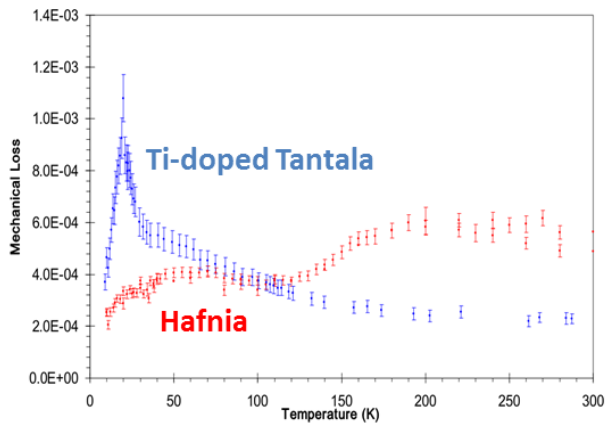
Mechanical loss measurements on *multilayer Titania-doped-Tantala coatings on Silicon* (annealed at  $400\text{C} \sim 600\text{C}$ ) also show a cryo-peak at  $\sim 30\text{K}$  [Granata et al., Opt. Lett. 38, 5268 (2013)].



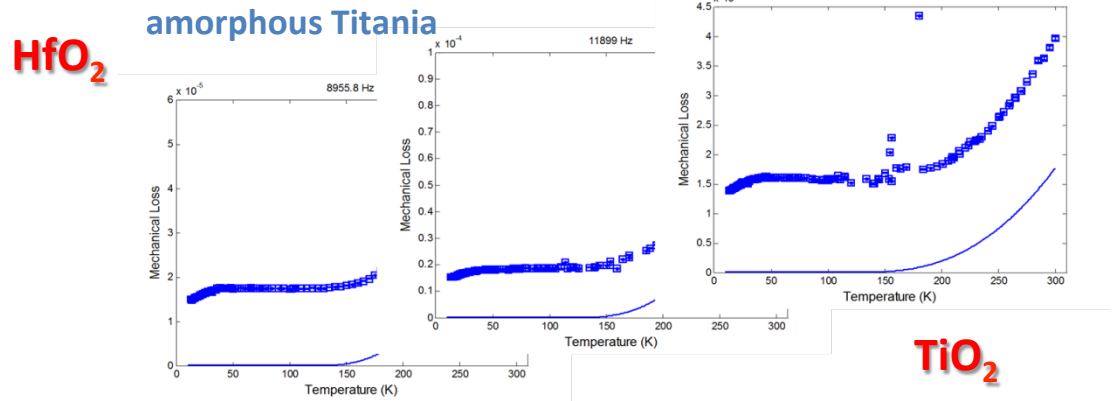
Mechanical loss measurements on *multi-layer Tantalum/Silica coatings on Sapphire* do not show such peak, yielding almost temperature & annealing schedule independent losses

[Yamamoto et al., PRD-74 022002 (2006); Hirose et al., LIGO-P1400107].

# Cryo – Friendly Oxides...



[Chalkley et al., LIGO-G080314]

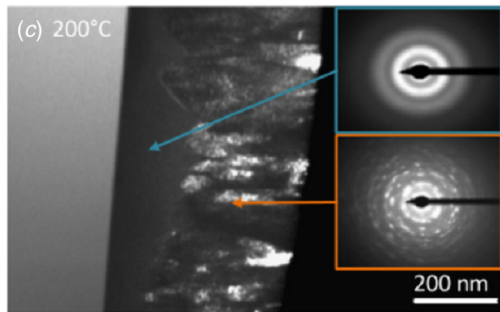


[IMartin & Murray, GWADW 2014, preliminary]

... *crystallize upon annealing*

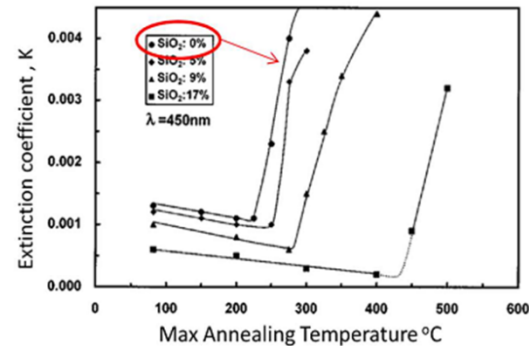
*(needed for optical properties)*

[Abernathy et al., CQG 28(2011) 195017]



HfO<sub>2</sub>

Chao and Wang, Appl. Opt 23 (1998) 1417



TiO<sub>2</sub>



# Outlook

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Nanolayers: {  
What  
How  
Why

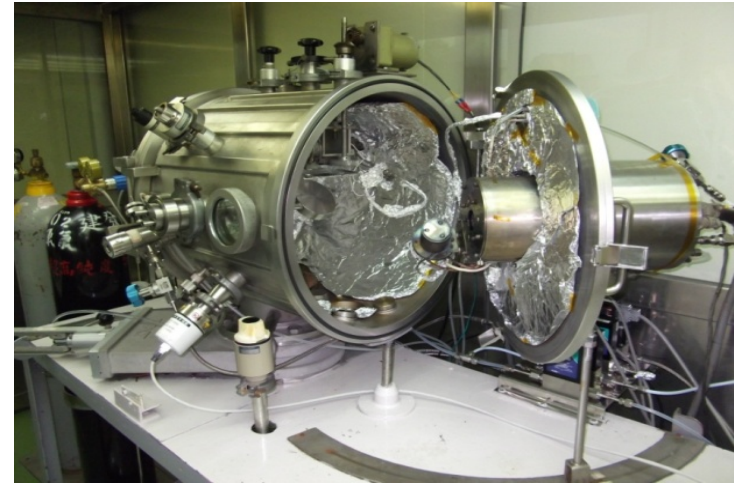
Development: {  
Survey of Technical means ←  
Results  
What's Next

Conclusions & Acknowledgements

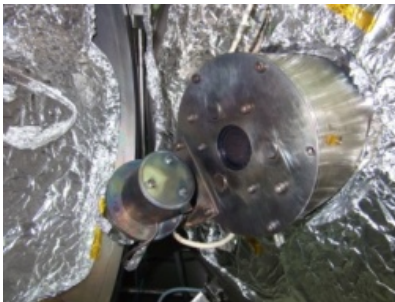
# NTHU Deposition Facility

Kaufman-type ion beam sputterer  
in Class 100 clean compartment  
within Class 10000 clean room.

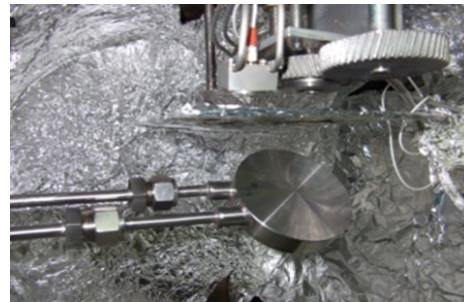
[S. Chao et al, LIGO-G1101083, G1200489, G1300921]



Kaufman gun & neutralizer



Sputter target and rotator

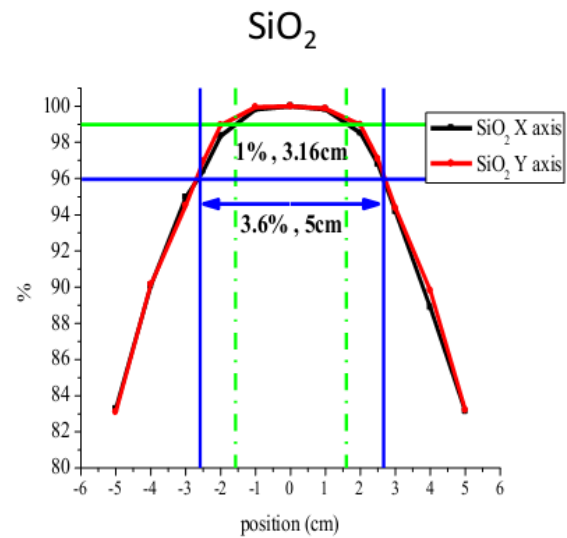
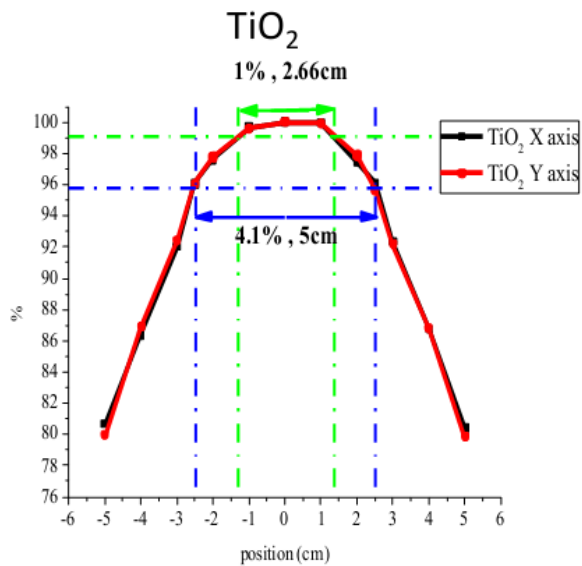
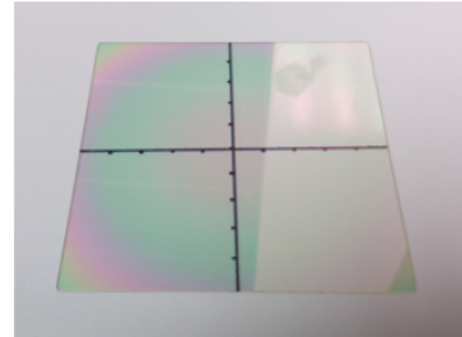
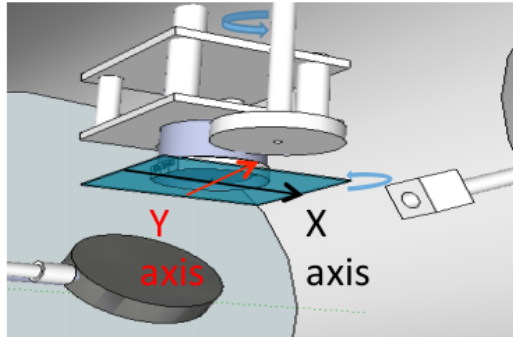


Twin exch. target holder



- **Several witness samples are deposited together with a few cantilevers in each run for structural/optical characterization.**

# Deposition Uniformity

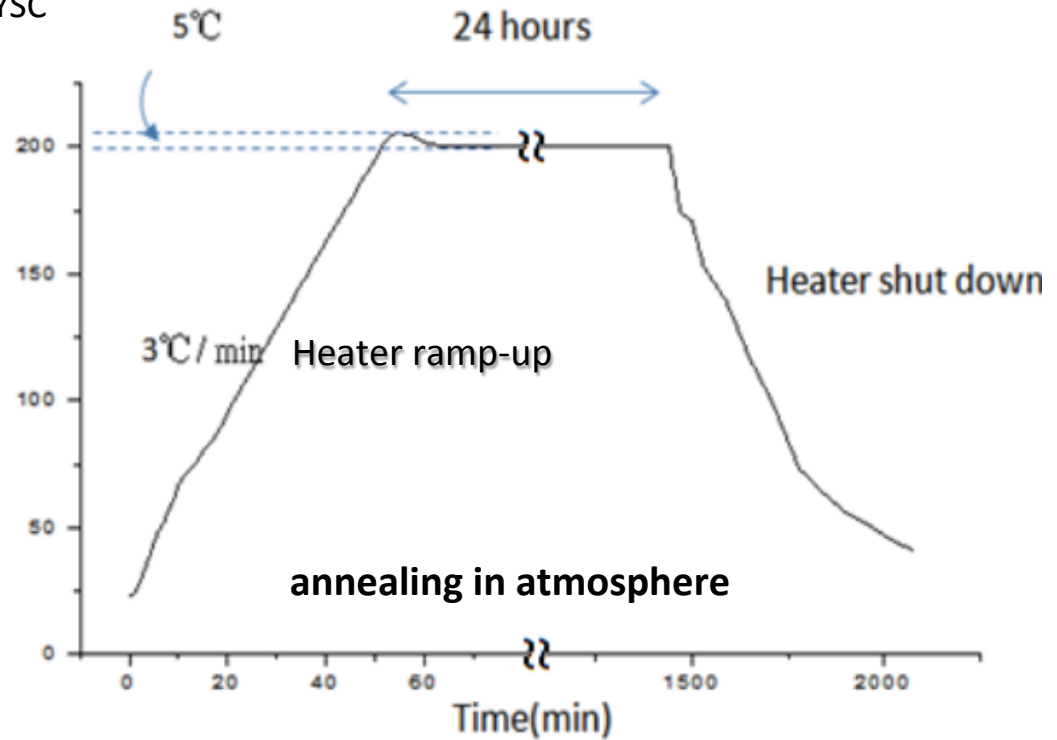
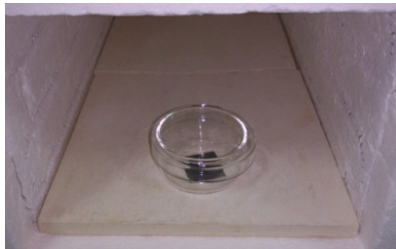


[S. Chao et al., LIGO-G1300921]

# NTHU Annealing Facility

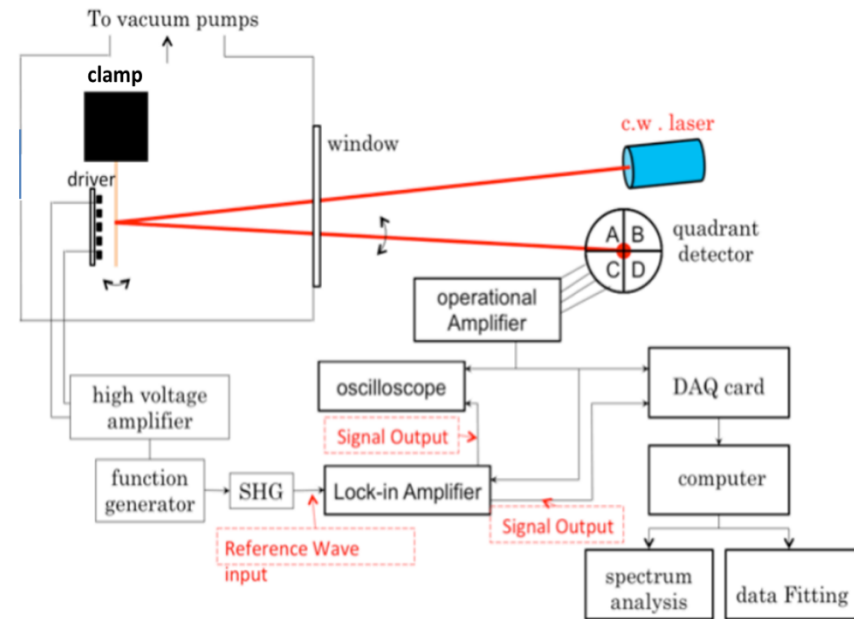
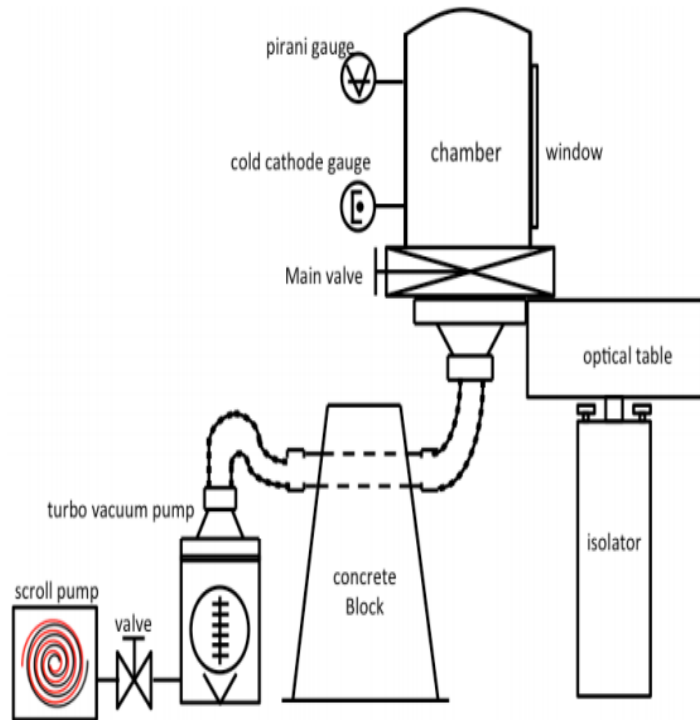


Model : YF-4  
Company : YSC



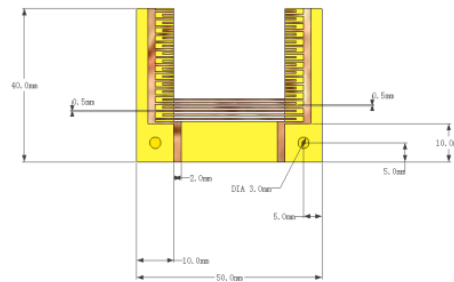
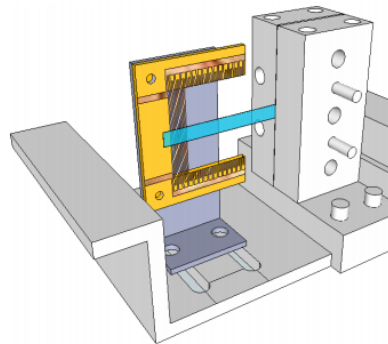
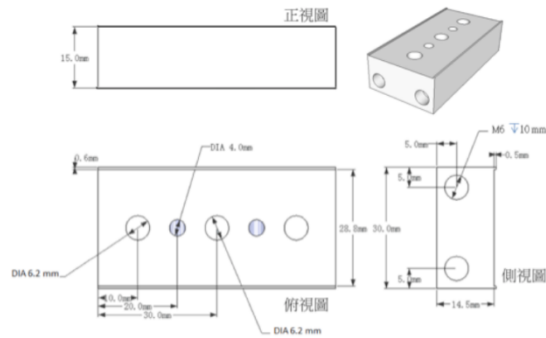
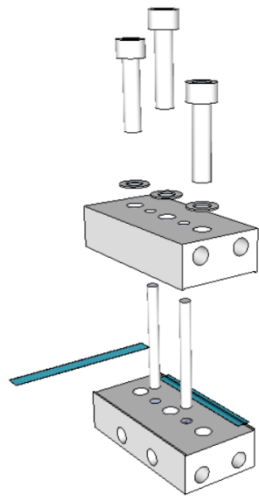
[S. Chao et al., LIGO-G1300921]

# NTHU quality factor control Cantilever Setup



[S. Chao, LIGO-G1200489 ]

# Clamp/Exciter Design



國立清華大學  
碩士論文

題目：應用於雷射干涉重力波偵測器開發工作  
之單晶矽懸臂樑之機械震動性質研究

Study of mechanical vibration and loss of silicon  
cantilever for development of the high-reflection  
mirror in the laser interference gravitational  
wave detector

系所：光電工程研究所

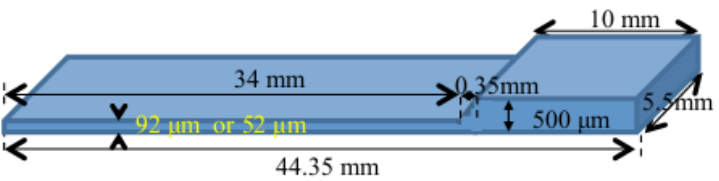
學號姓名：9966701 王薇雅 (Wei-Ya Wang)

指導教授：趙煦 教授 (Prof. Shiuh Chao)

中華民國一百零二年八月

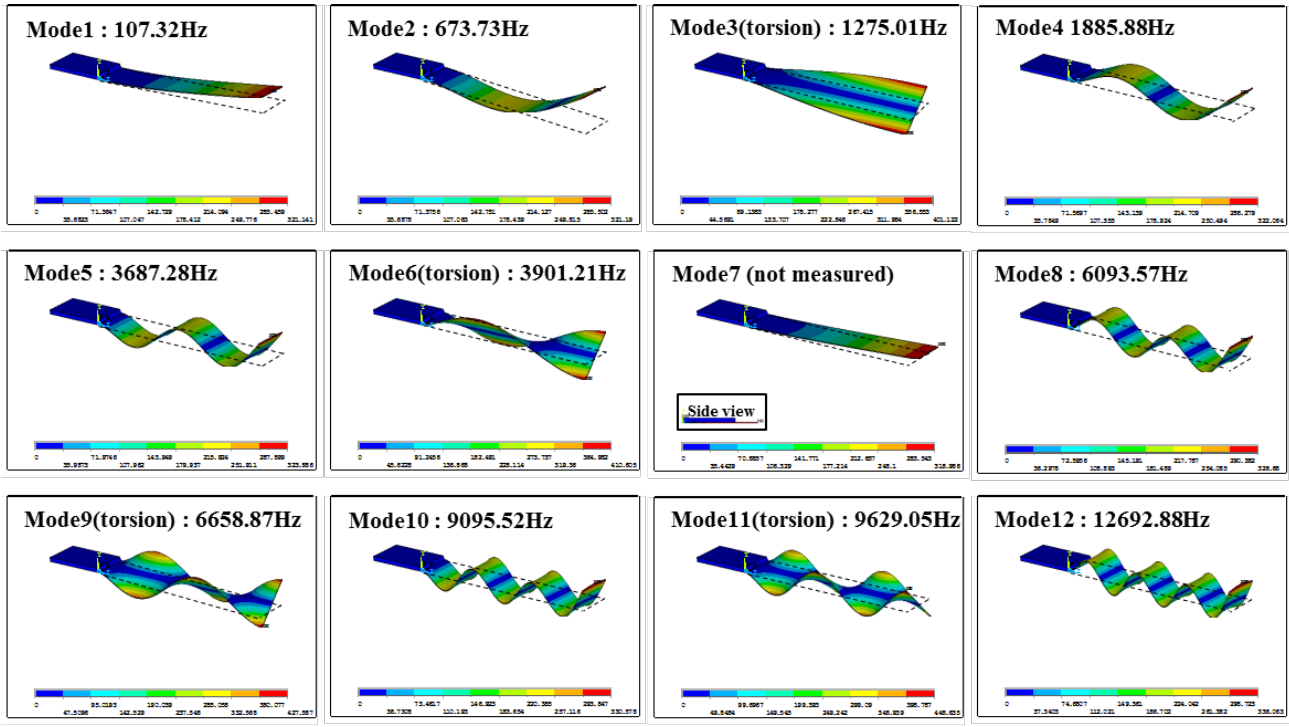
[J. Wang, MA Thesis, NTHU, 2012]

# Cantilever Design



Cantilever fabricated from (100, undoped) 4" silicon wafer by KOH wet etching.

[S. Chao, LIGO-G1200849]



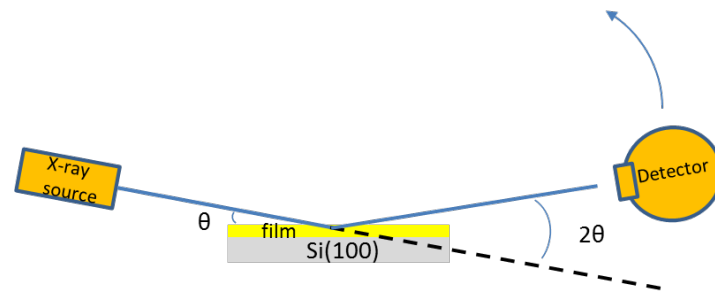
[J. Wang, MA Thesis, NTHU, 2012]

# NTHU XRD Facility



Model : X'Pert Pro (MRD)  
Company : PANalytical  
X-ray source : Cu ( $K\alpha$  ;  $\lambda= 0.154$  nm)  
Generator voltage : 45kV  
Tube current : 40mA  
Detector : Proportional Counter  
Beam size : 12 mm  $\times$  0.4 mm  
Sample size : 10mm X 10mm

Incidence angle( $\theta$ ) : 0.5  $^\circ$   
Scan range ( $2\theta$ ): 20  $^\circ$  ~65  $^\circ$   
Scan step size : 0.02  $^\circ$   
Time per step : 0.5s





# The INFN AdCOAT Project (2014-15)

---

## *Mission:*

➔ “Investigating, characterizing and comparing the properties (morphological, structural, optical and viscoelastic) of Silica::Titania and Silica::Hafnia mixtures, both nm-layered and co-sputtered, at ambient and cryogenic temperatures.”

## *Working Groups:*

**USannio** (PI, nanolayer modeling and design);

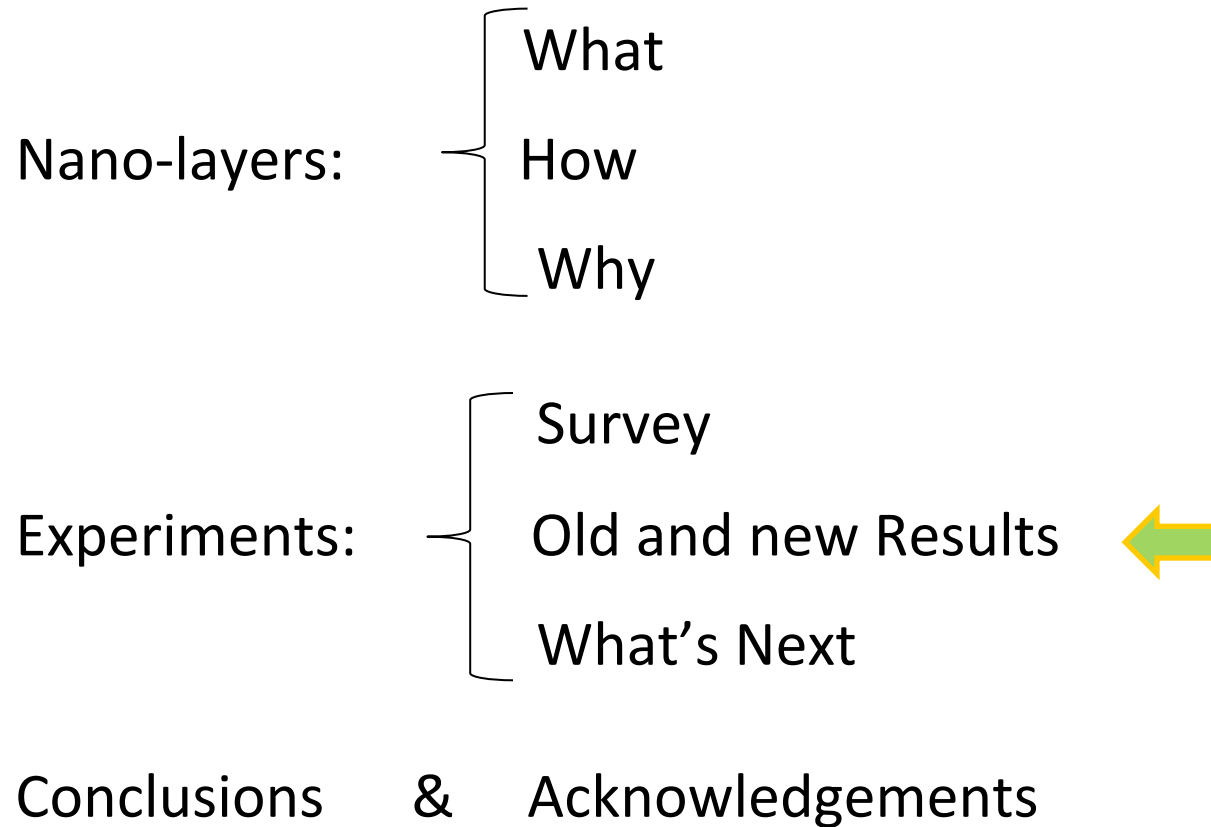
**Genoa** (structural/optical characterization);

**Perugia** (dissipation modeling in glasses; viscoelastic parameter measurements);

**Rome** (cryogenic GeNS based loss angle measurement setup).

# Outlook

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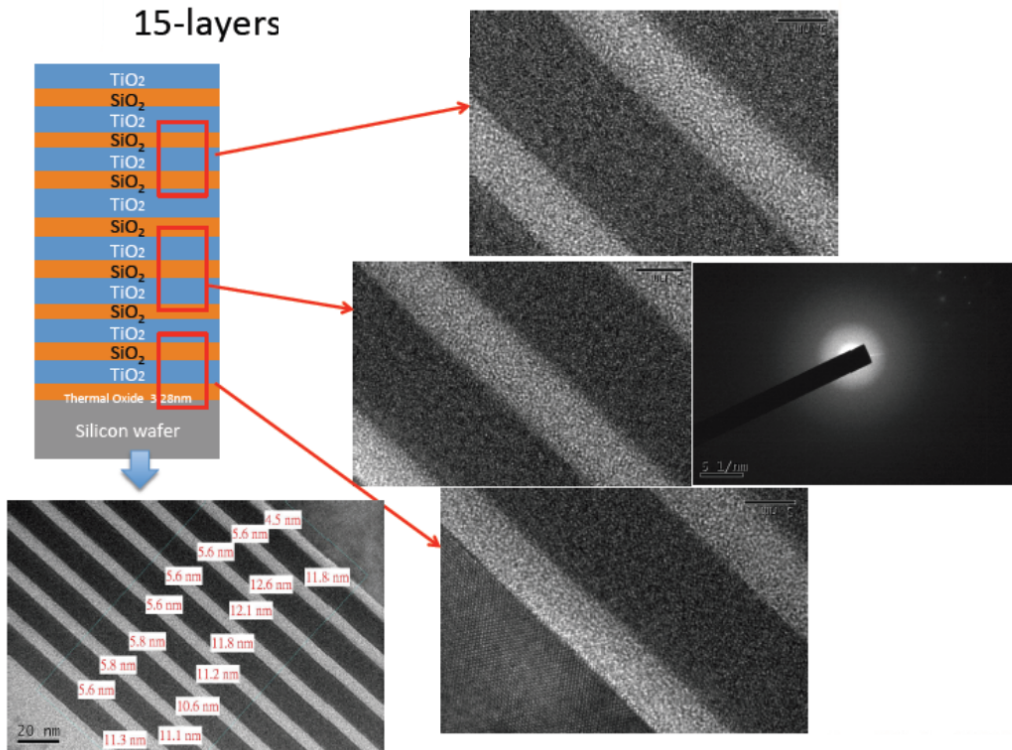
# 1<sup>st</sup> Generation Prototypes

	Total thickness (nm)	Averaged thickness of TiO <sub>2</sub> and SiO <sub>2</sub> layer(nm)	
		TiO <sub>2</sub>	SiO <sub>2</sub>
single TiO <sub>2</sub>	121.9	121.9	0
3 layer	119.8	40.9	40.7
5 layer	119.2	26.2	20.3
7 layer	120.0	20.6	12.5
11 layer	119.3	13.7	7.4
15 layer	112.4	9.8	4.8
19 layer	112.6	7.4	4.3

[S. Chao et al., LIGO-G1300921]

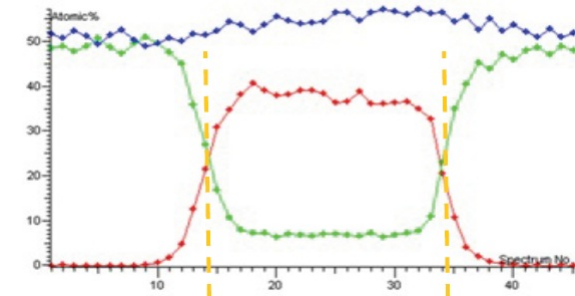
All prototypes QWL thick @ 1064nm, all with  $n = 2.065$

# Morphology (As Deposited)



Morphology of witness samples investigated using TEM and electron diffraction.

Interface profiles characterized via energy-dispersive X-ray diffraction (EDXRD)



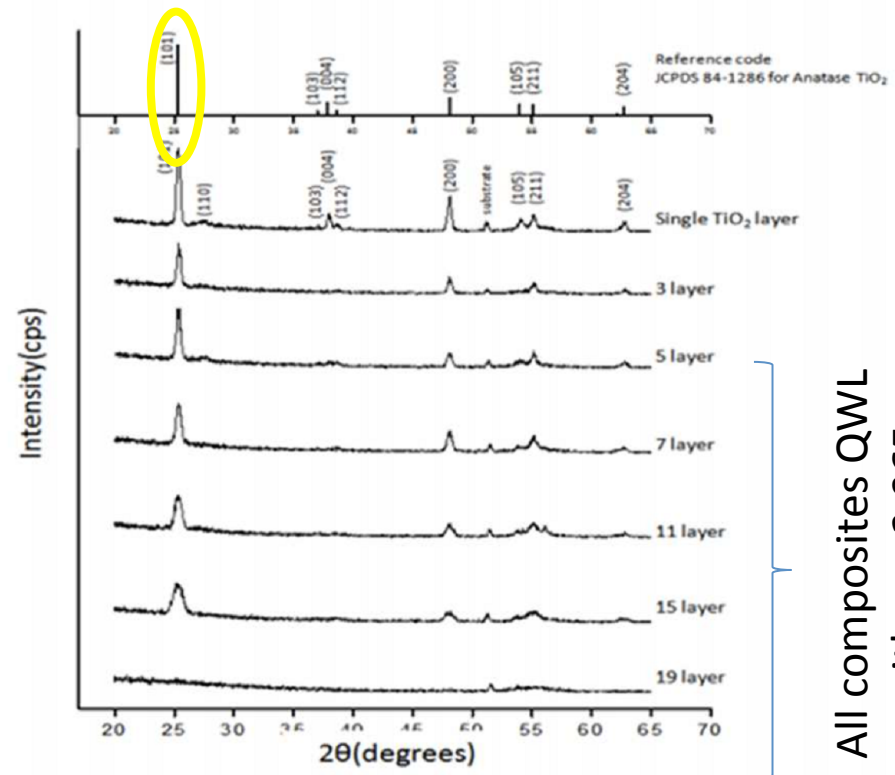
[S. Chao et al., LIGO-G1200489]

# XRD Spectra after Annealing

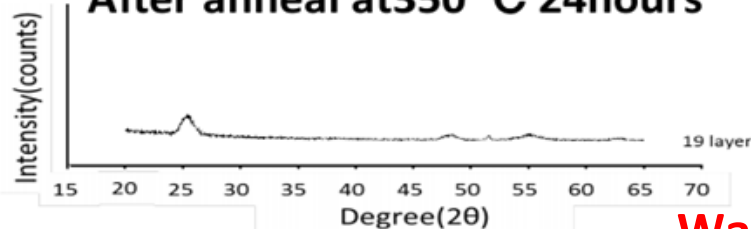
Threshold anneal temperature  
For crystallization  
increases with :  
the number of layers  
decreasing the Titania layer thickness.

At 300C the Anatase peak gets smaller and broader as the nanolayer thickness decreases (and the nanolayer number increases), signaling progressive crystallization frustration, until it disappears for N=19.

After anneal at 300°C 24hours



After anneal at 350 °C 24hours



[Chao et al., LIGO-P1400122;  
Optics Express Nov. 2014]

**Warning:**  
**Appearance of crystallites is not a step function**

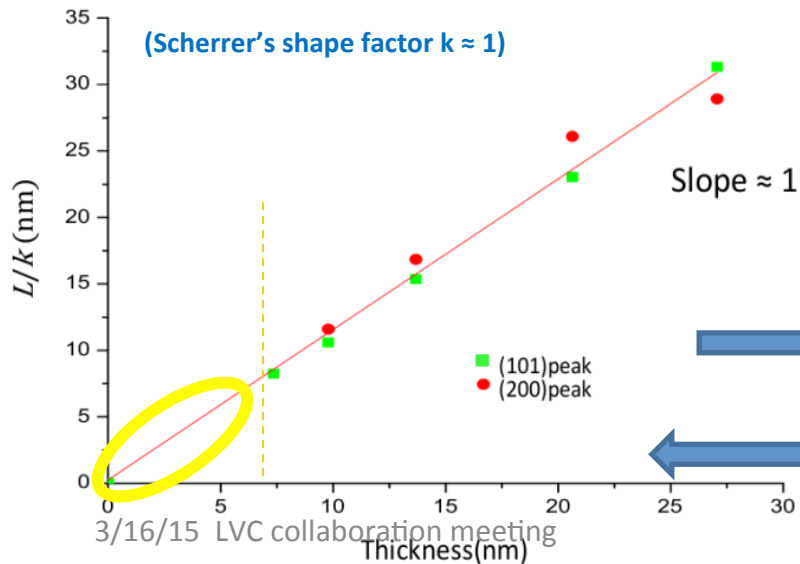
# Crystallization

Crystallization Sample	Anneal Condition	Before annealing	225 °C 24hr	250 °C 24hr	300 °C 24hr	350 °C 24hr
Single TiO <sub>2</sub>		No	No (explained)	Yes FWHM=0.37 °	Yes FWHM=0.37 °	--
3 layer		No	Yes FWHM=0.37 °	Yes FWHM=0.40 °	Yes FWHM=0.35 °	--
5 layer		No	No	Yes FWHM=0.50 °	Yes FWHM=0.44 °	--
7 layer		No	No	Yes FWHM=0.53 °	Yes FWHM=0.50 °	--
11 layer		No	No	Yes FWHM=0.81 °	Yes FWHM=0.65 °	--
15 layer		No	No	No	Yes FWHM=0.93 °	--
19 layer		No	No	No	No	Yes FWHM=1.32 °

# Crystallite Size vs Nano-thickness

Crystallite sizes  $L/k$  of the nano-layers after anneal at 300C 24hr

	Thickness of TiO2 (nm)	Anatase TiO2 Peak (101)				Anatase TiO2 Peak (200)			
		$2\theta$	$\beta_{exp}$ (degree)	$\beta_s \cos\theta$ (degree)	$L/k$ (nm)	$2\theta$	$\beta_{exp}$ (degree)	$\beta_s \cos\theta$ (degree)	$L/k$ (nm)
Single TiO2	121.9	25.344	0.356	0.160	55.162	48.088	0.392	0.180	49.087
3-layer	40.9	25.371	0.390	0.223	39.569	48.108	0.424	0.233	37.942
5-layer	26.2	25.351	0.428	0.282	31.330	48.096	0.476	0.305	28.916
7-layer	20.6	25.355	0.504	0.383	23.034	48.099	0.502	0.338	26.097
11-layer	13.7	25.330	0.668	0.574	15.366	48.094	0.666	0.524	16.854
15-layer	9.8	25.315	0.910	0.833	10.597	48.048	0.899	0.761	11.602
19-layer	7.4	25.416	1.140	1.068	8.258	--	--	--	--



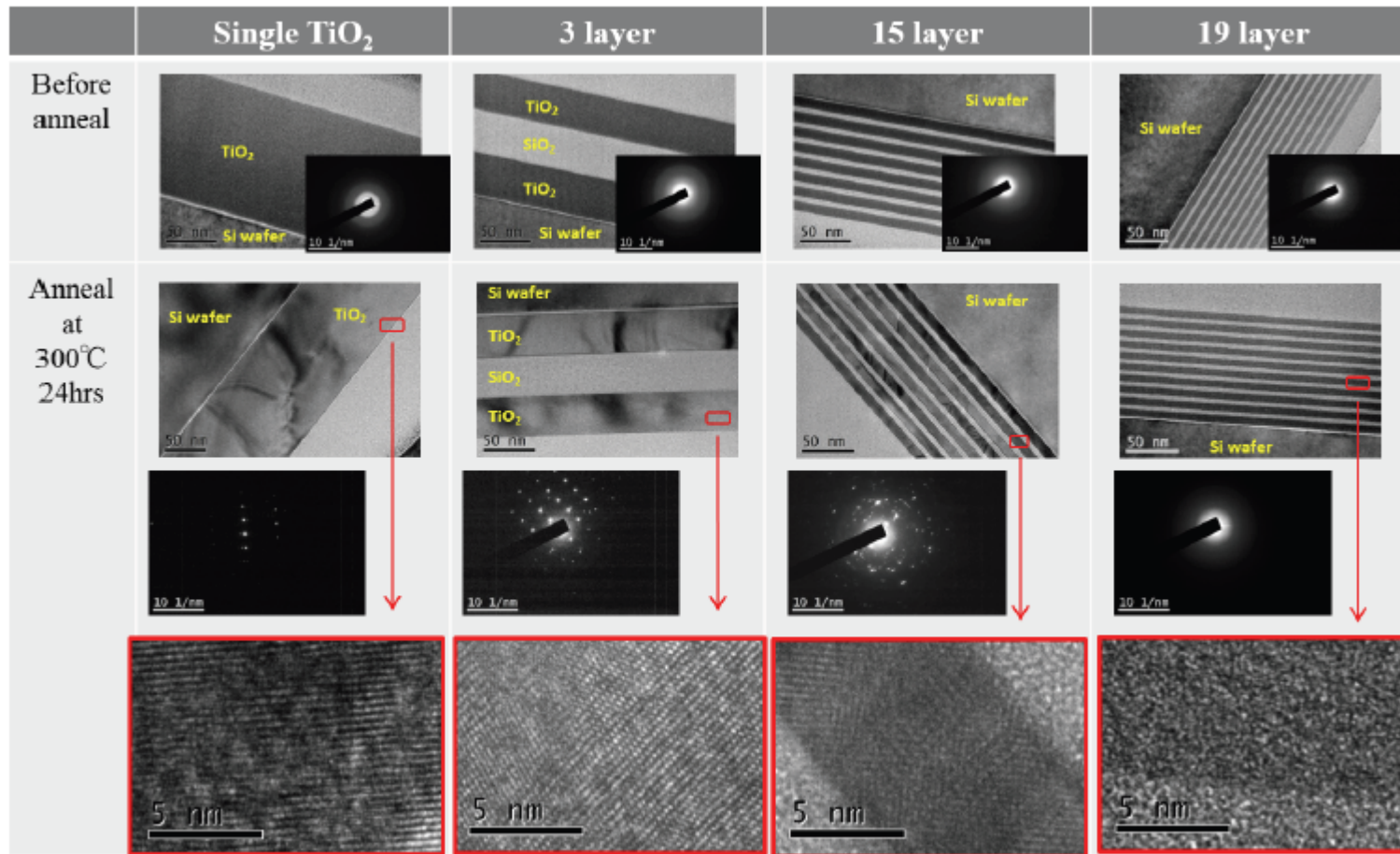
Crystallite size decreases almost linearly with thickness down to thickness  $\approx 7$ nm

[S. Chao et al., LIGO-G1300921]

Sankur-Gunning results confirmed

Next to be tried layers  $\sim 1$  nm, no interdiffusion in X-ray mirrors

# TEM Before/After Annealing

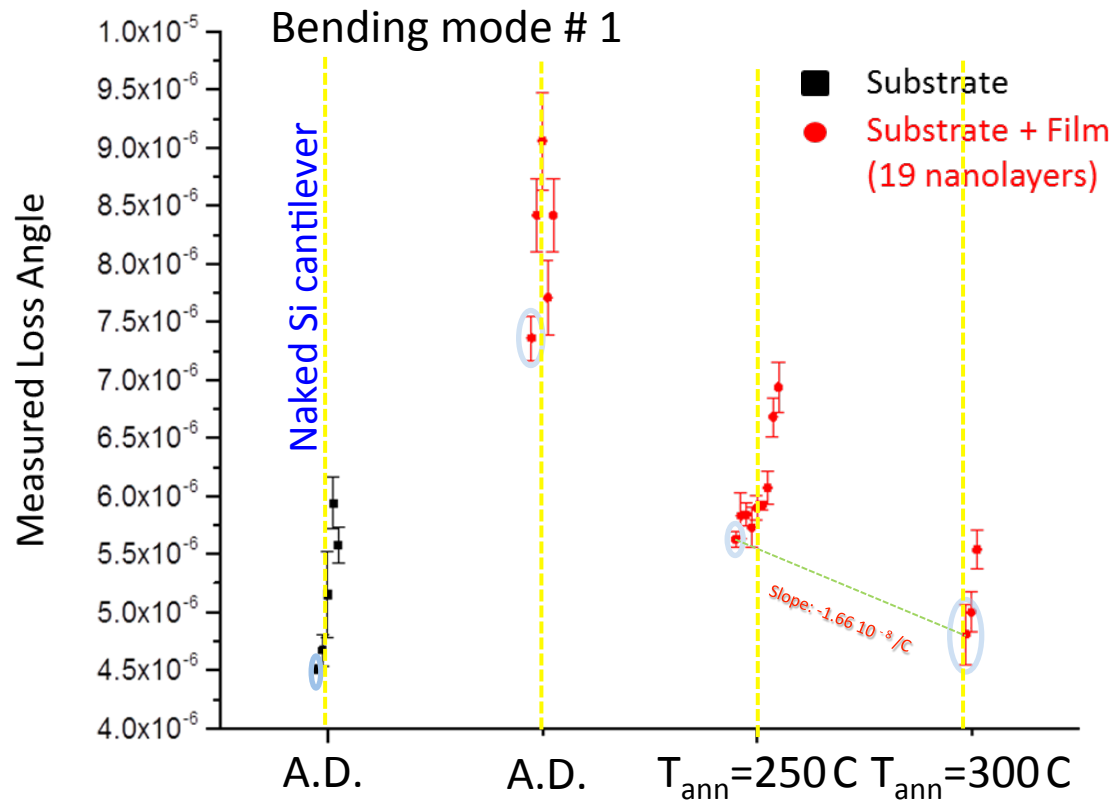


[S. Chao et al., LIGO-G1300921]

➡ **TEM shows that no significant cross-interface diffusion occurs during annealing**



# Loss Angle Before/After Annealing



Different bars correspond to different re-clampings.

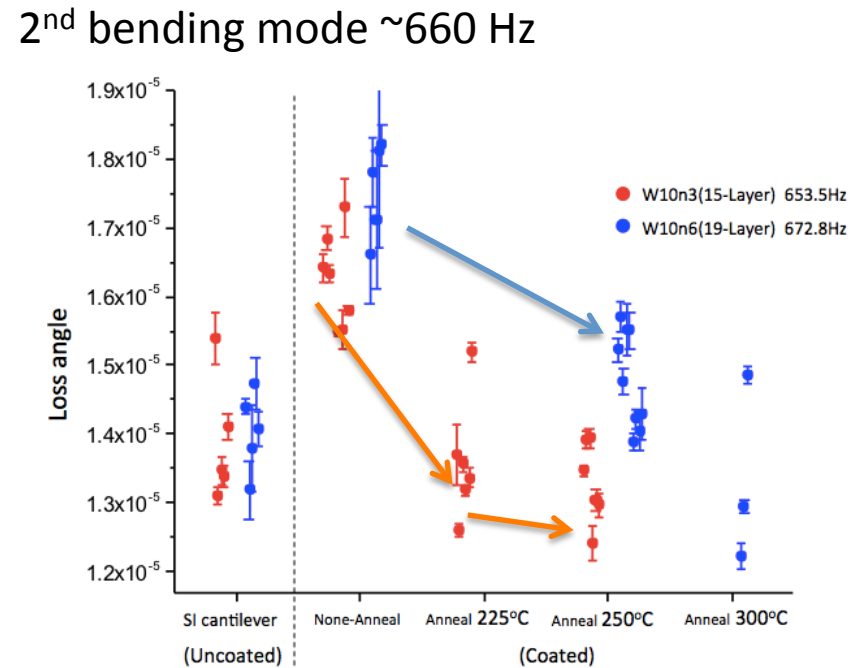
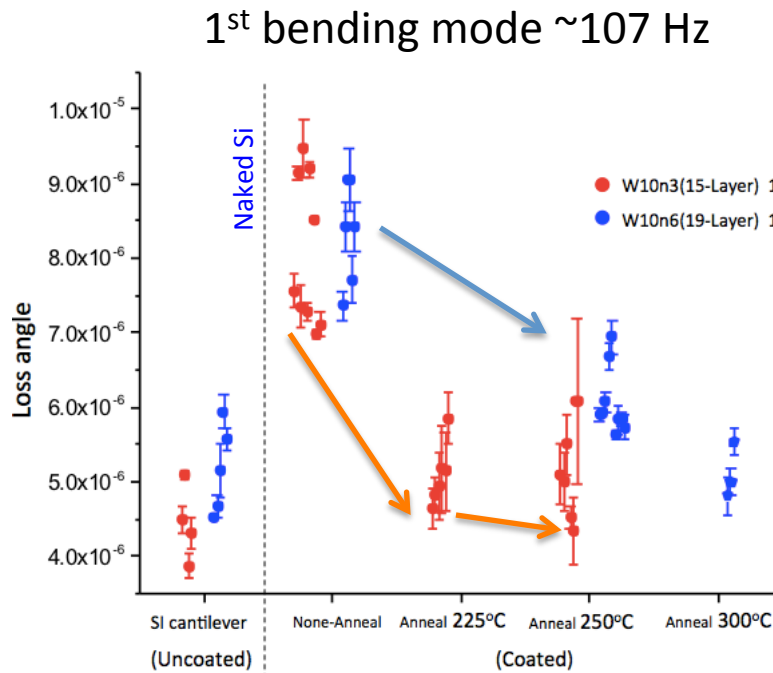
Lowest-average bar yields most trustable value.

Multiple measurements taken for each re-clamping, yielding error bars.

Loss Angle		
$T_{ann}$	$\mu$	$\sigma/\mu$
A.D.	$7.36 \cdot 10^{-6}$	0.082
250	$5.64 \cdot 10^{-6}$	0.074
300	$4.81 \cdot 10^{-6}$	0.074
substrate + film (19 layers)		

[preliminary results , S. Chao et al., LIGO-G1401055]

# Puzzles?

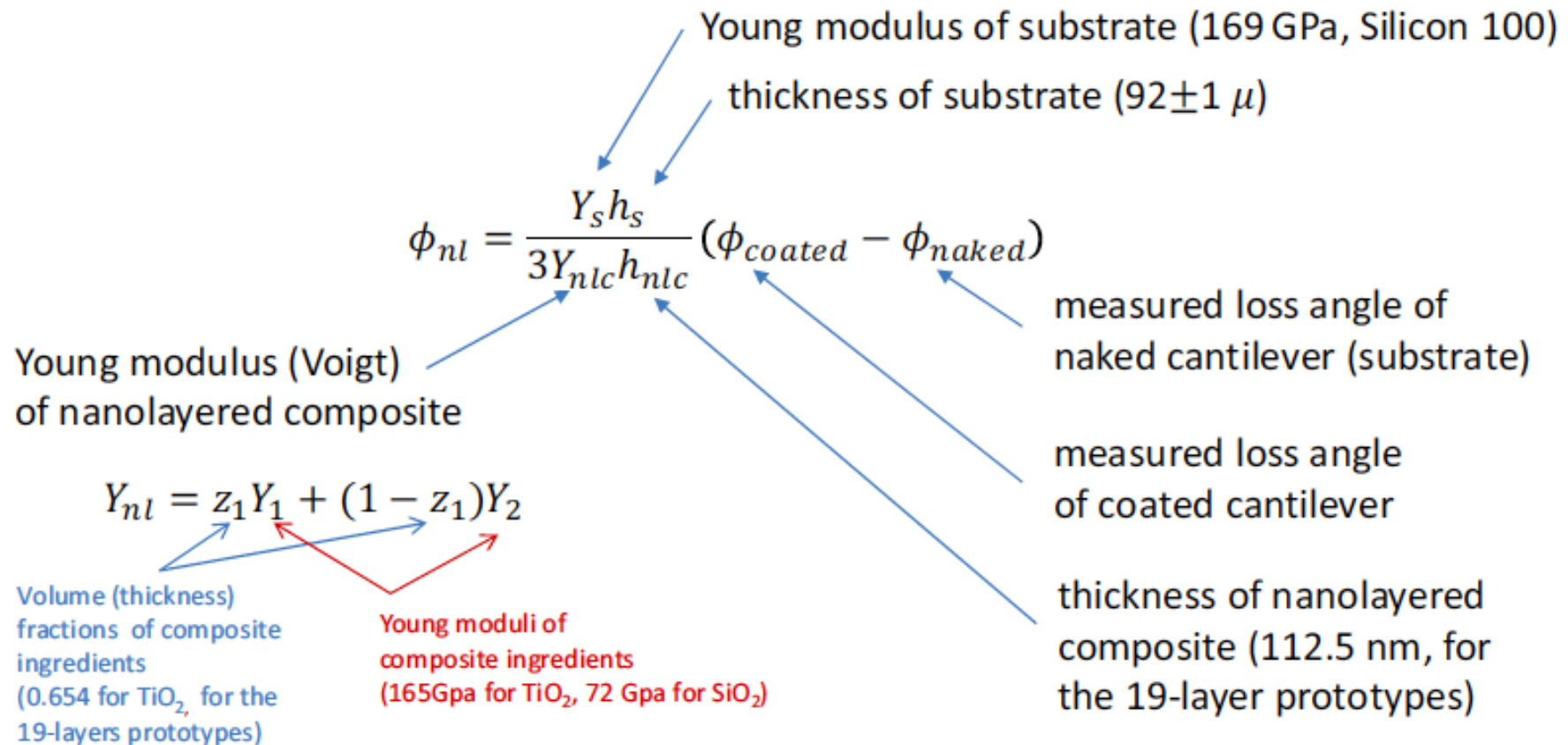


Is there an optimal Ti thickness (or distributed stress) to reduce mechanical losses?

Is there a physical saturation effector simply because coating losses are already almost indistinguishable from Si substrate's

Need improved measuring system, make one free of re-clamping noise.

# Nanolayer Loss Angle



[Pierro et al., LIGO-T060173]

# TiO<sub>2</sub> Loss Angle in Nanolayer

---

... Use fiducial value of Silica loss angle ( $5 \cdot 10^{-5}$ ) to retrieve loss angle of  $\alpha$ -Titania *in the nm-layered composite* ....

$$\phi_{nlc} = \frac{\left(\frac{Y_s}{Y_1} + \frac{Y_1}{Y_s}\right) z_1 \phi_1 + \left(\frac{Y_s}{Y_2} + \frac{Y_2}{Y_s}\right) (1 - z_1) \phi_2}{Y_s \left[\frac{z_1}{Y_1} + \frac{1 - z_1}{Y_2}\right] + Y_s^{-1} \left[\frac{z_1}{Y_1} + \frac{1 - z_1}{Y_2}\right]^{-1}}$$

Yields loss angle values  $\sim 10^{-4}$  for  $\alpha$ -TiO<sub>2</sub>, consistent with [Scott and MacCrone, Rev. Sci. Instr. 39 (1968) 821].

# TiO<sub>2</sub> Loss Angle in Nanolayer

---

...yielding for our 19-layers nm-layered composite the following estimates for the loss angle as a function of the annealing temperature (with typical 10% uncertainties)

$\phi = 1.04 \cdot 10^{-3}$	(as deposited)
$\phi = 0.43 \cdot 10^{-3}$	(after annealing 24h @ 250°C)
$\phi = 0.13 \cdot 10^{-3}$	(after annealing 24h @ 300°C)

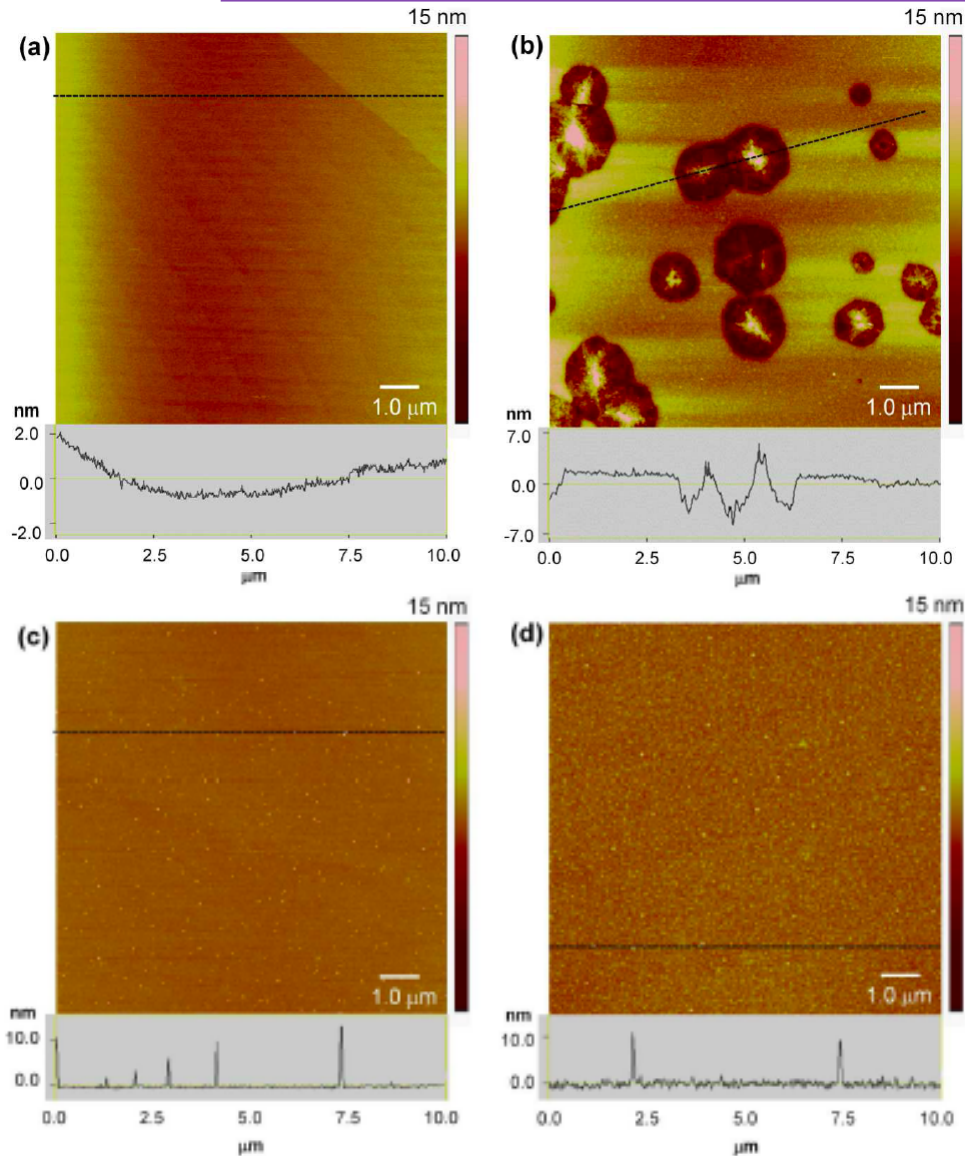
Preliminary

...comparable to or better than Ti-doped Tantalum !

Note: the effective refractive index of our nm-layered composite is (Drude formula):

$$n_{nl} = [z_1 n_1^2 + (1 - z_1) n_2^2] \approx 2.063 \text{ (@1064nm)}$$

# AFM Characterization (AdCOAT)



AFM (Dimension 3000)

AFM images of single Ti layer qwl.  
on silicon

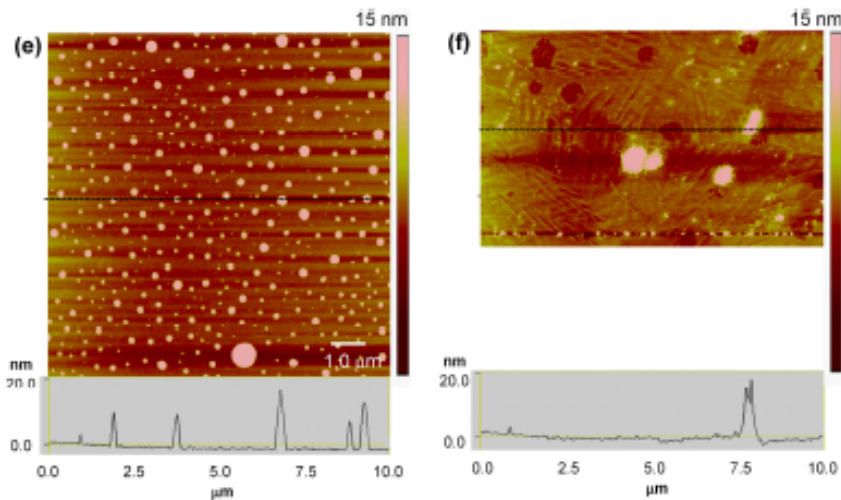
(a) as-deposited  
(b) as-annealed at 250°C,

AFM images of single silica layer qwl.  
on silicon

(c) as-deposited  
(d) as-annealed at 300°C.

*(M. Canepa, INFN Ge)*

# AFM Characterization (AdCOAT)

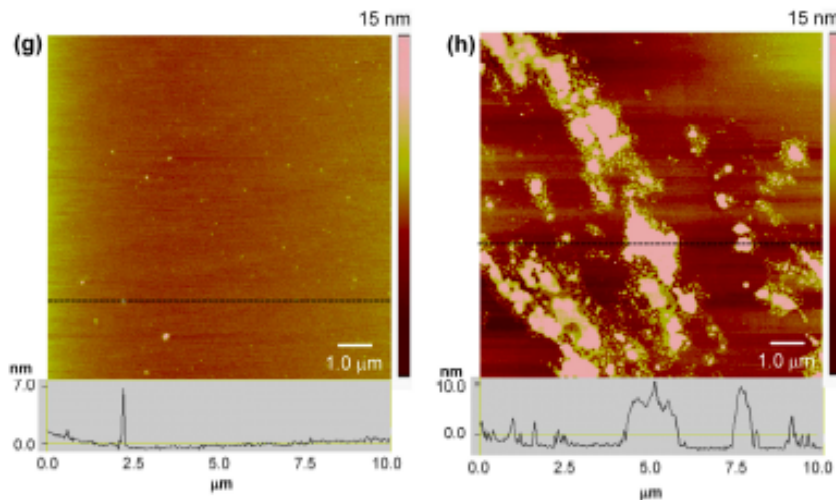


AFM (Dimension 3000)

AFM images of 1 QWL (5 layers) Ti/Si on silicon

(e) as-deposited

(f) as-annealed at 300°C,



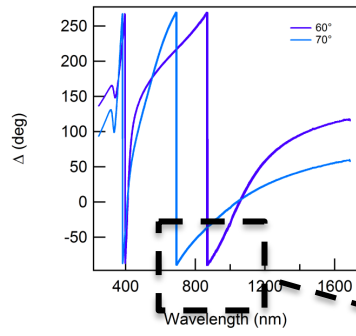
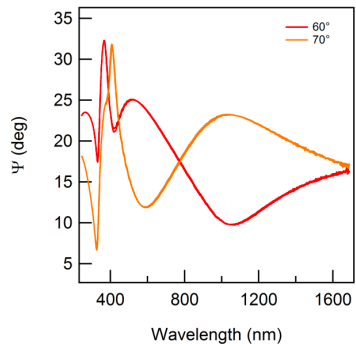
AFM images of 1 QWL (19 layers) Ti/Si on silicon

(g) as-deposited

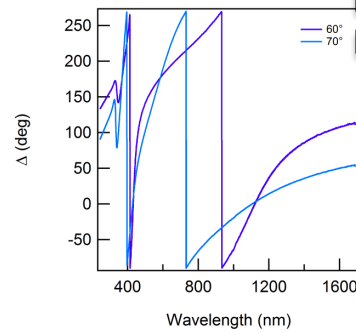
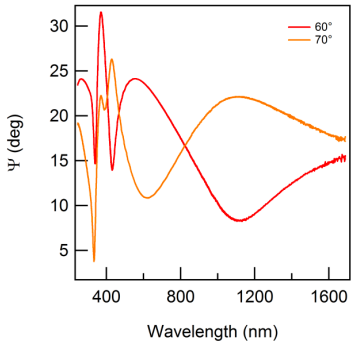
(h) as-annealed at 300°C.

*(M. Canepa, INFN Ge)*

# Ellipsometry-Optical Characterization (AdCOAT)

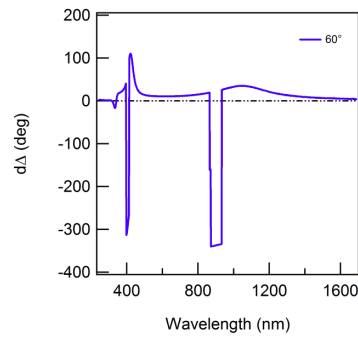
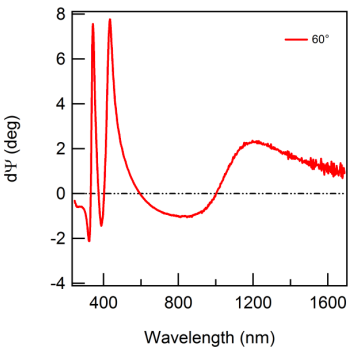


Single Titania layer on silicon  
Un-annealed



Very Good  
Uniformity

Crystallized (250° C)

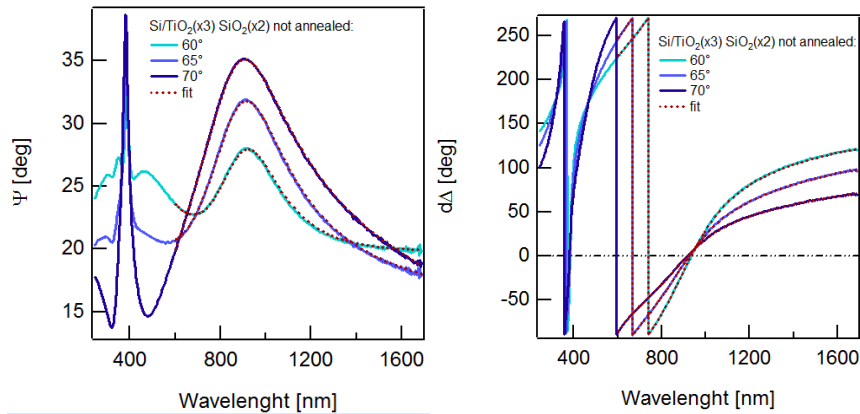


Difference not-annealed/annealed

Ellipsometric spectroscopy  
(Woollam 2000, VASE)  
(M. Canepa, INFN Ge)



# Optical Characterization (AdCOAT)



5 layer Titania/Silica  
nominal thickness  
27.1 nm (Titania x3) ;  
19.9 nm (Silica x2)

Un-annealed

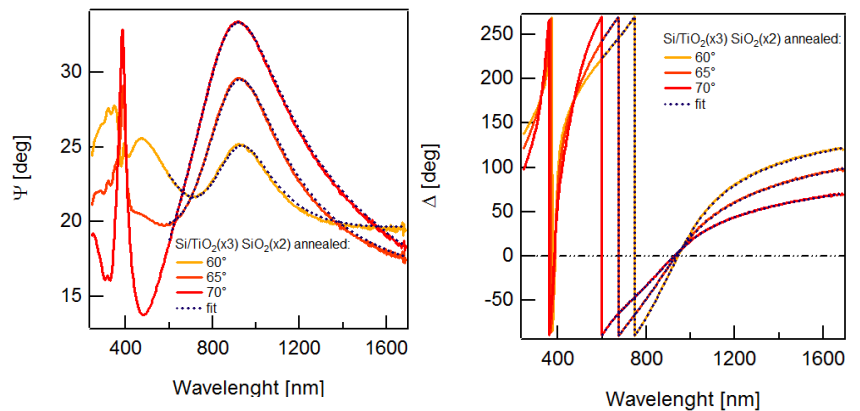
$d_{\text{cauchy\_TiO}_2} = 27.30 \pm 0.02 \text{ nm}$   
 $d_{\text{cauchy\_SiO}_2} = 20.15 \pm 0.02 \text{ nm}$   
 $d_{\text{roughness}} = 1.20 \pm 0.04 \text{ nm}$   
 $\text{MSE} = 8.575$   
 $n_{\text{TiO}_2}[\lambda=1064 \text{ nm}] = 2.319$

$A_{n\_TiO_2} = 2.275$      $B_{n\_TiO_2} = 0.05$   
 $A_{n\_SiO_2} = 1.478$      $B_{n\_SiO_2} = 0.0048$   
 $d_{\text{SiO}_2\_jaw} = 1.64 \pm 0.07 \text{ nm}$   
 $n_{\text{SiO}_2}[\lambda=1064 \text{ nm}] = 1.482$

Annealed (300° C)

Very good fit using EMT  
(BruggeMann and/or  
Drude formulas)

Optical losses are below  
measurement sensitivity  
(1ppm)

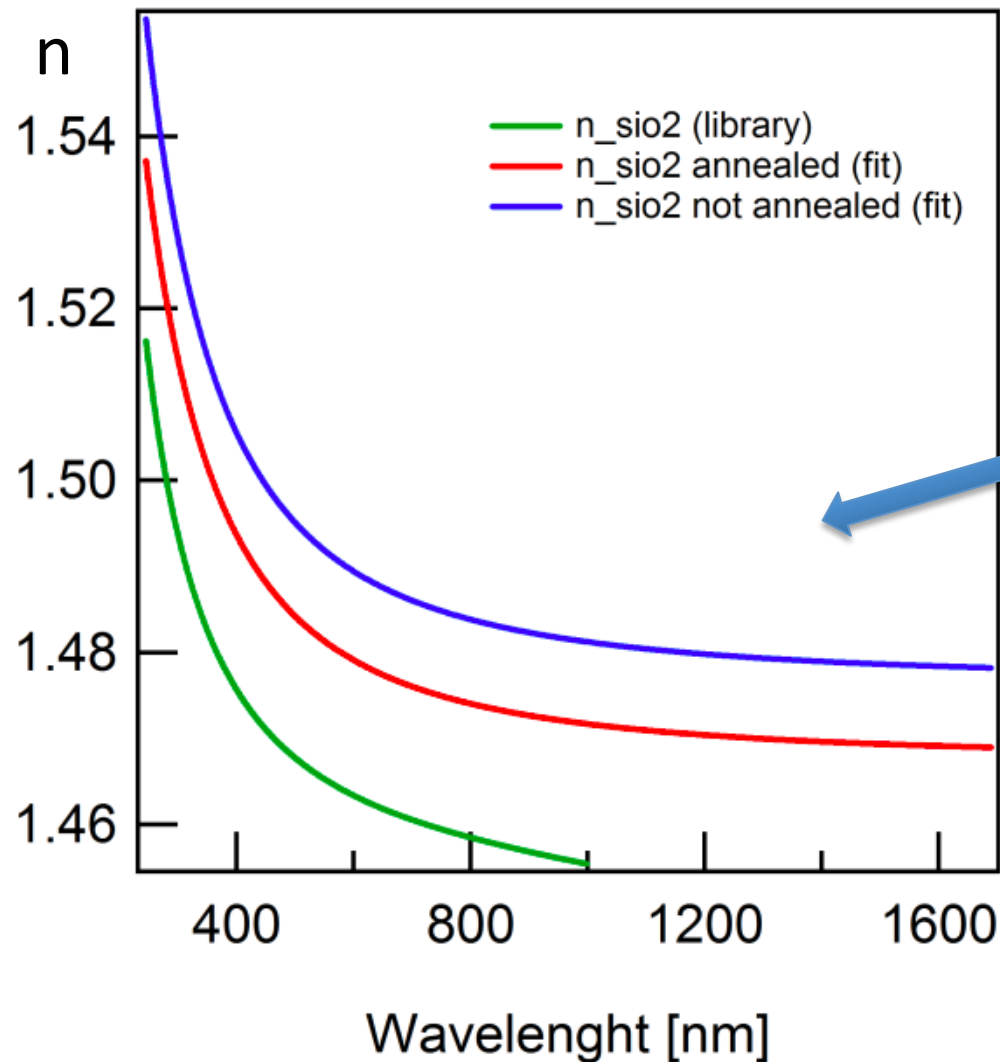


$d_{\text{cauchy\_TiO}_2} = 26.98 \pm 0.02 \text{ nm}$   
 $d_{\text{cauchy\_SiO}_2} = 19.01 \pm 0.02 \text{ nm}$   
 $d_{\text{roughness}} = 0.265 \pm 0.04 \text{ nm}$   
 $\text{MSE} = 9.78$   
 $n_{\text{TiO}_2}[\lambda=1064 \text{ nm}] = 2.354$

$A_{n\_TiO_2} = 2.31$      $B_{n\_TiO_2} = 0.05$   
 $A_{n\_SiO_2} = 1.453$      $B_{n\_SiO_2} = 0.0048$   
 $d_{\text{SiO}_2\_jaw} = 3.16 \pm 0.07 \text{ nm}$   
 $n_{\text{SiO}_2}[\lambda=1064 \text{ nm}] = 1.457$

Ellipsometric spectroscopy  
(Woollam 2000, VASE)  
(M. Canepa, INFN Ge)

# Optical Characterization (AdCOAT)



REFRACTION INDEX  
SINGLE SILICA LAYER  
ON SILICON

Visibly annealing relaxes  
deposited silica towards  
the molten silica glass  
state

Ellipsometric spectroscopy  
(Woollam 2000, VASE)  
(*M. Canepa, INFN Ge*)

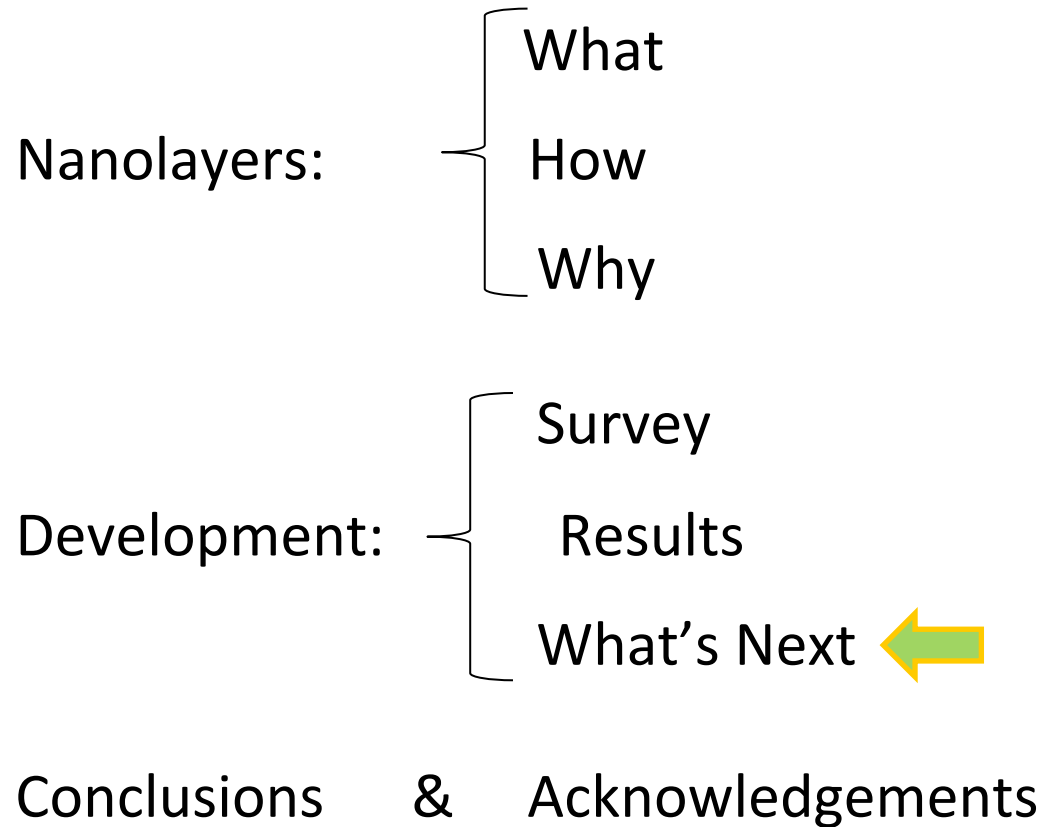
# Characterization tools

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We have good eyes to see !

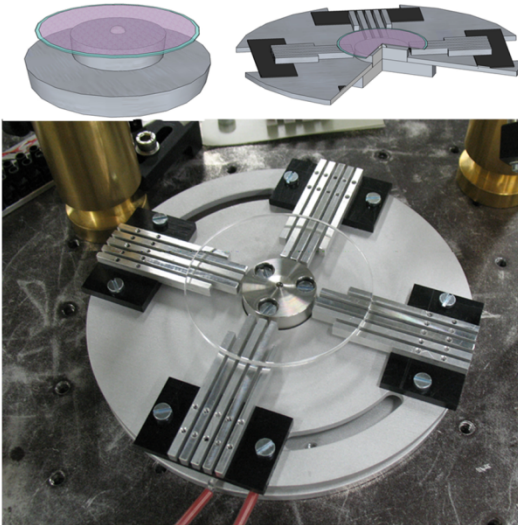
# Outlook

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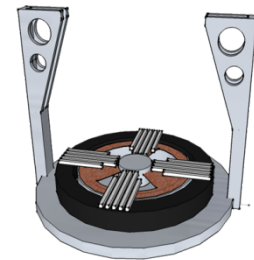
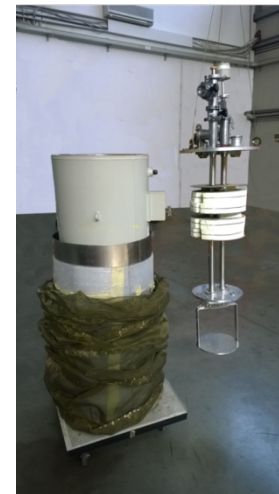


# GeNS Setup, $T_{amb}$ & Cryo (AdCOAT)

GeNS (Gentle Nodal Suspension)



**AdCOAT INFN Rome WG** - is building a GeNS based setups for mechanical Q measurement at ambient and (soon) cryogenic temperatures.



mechanical drawings by Matteo Lorenzini

1" Silicon disks being sent to Chao for coating  $T_{amb}$  measurements to start soon (June 2015).

*(courtesy A. Rocchi, INFN U-Rome-Tor Vergata)*

# Si<sub>3</sub>N<sub>4</sub> Substrates (AdCOAT)

AdCOAT INFN U-Perugia Group is setting up a new MM interferometer for high Q (Norcada) Si<sub>3</sub>N<sub>4</sub> membrane characterization (naked first, then nanolayer-coated).

Designed for cryogenic operation.

Lightwave 126



Thorlabs BS-011 & BB1-E03



Vacuum: Pfeiffer TSH 071E

- Turbomolecular Drag Pumping Station TSH 071 E
- 1 Turbomolecular Drag Pump TMH 071 P
  - 2 Diaphragm Pump MVP 015-2
  - 2a DN/DTF main switch diaphragm pump
  - 3 Display And Operating Unit CCU 001
  - 8 Electronic Drive Unit TC 600
  - 28 DN/DTF Pumping Station (located internally)
  - 36 Air Cooling



Norcada QX10500CS



NF 2011-FC



DAC NI-SB 6221



Cryomech PT 405



SR-844 Lock-In



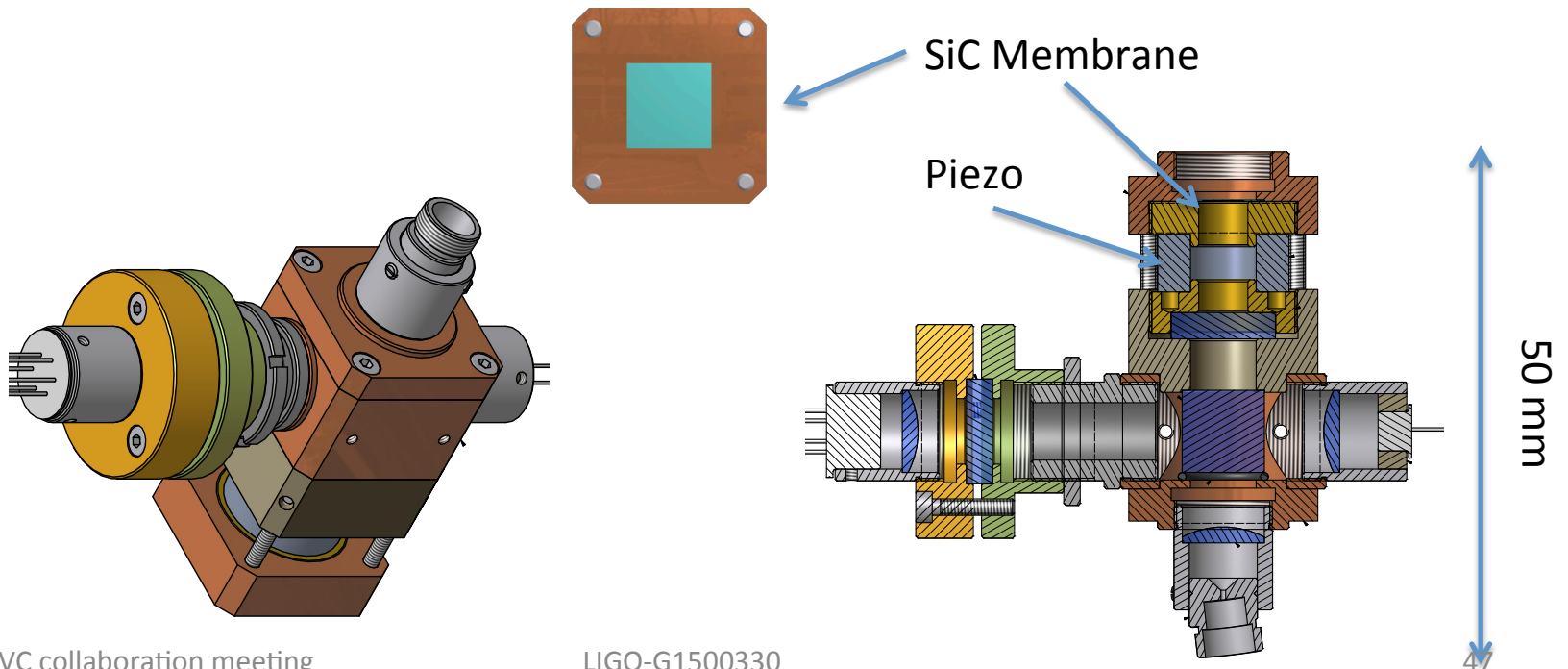
*(courtesy H. Vocca, INFN, U-Perugia)*

# More on $\text{Si}_3\text{N}_4$ Substrates

## UniSannio / NTHU / CSULA

Designed for cryogenic operation in NTHU and Sannio cryostats.

- Michelson readout
- Piezo excitation/resonance track
- Fiber light feeding

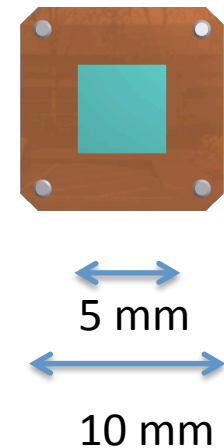


# Si<sub>3</sub>N<sub>4</sub> Substrates (AdCOAT)

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## Advantages / disadvantages of mechanical loss measurement on thin Silicon Nitride substrates

- Advantages:
  - Ultra-thin (~100nm), very low mechanical loss substrate
  - Coating losses by far dominating
  - Cheap modular substrate (e-microscope windows)
- Limitations:
  - High frequency operation
  - Membrane will bow under surface stress of deposited layer
    - + Directly measure surface tension variations during annealing
  - Probably limited to several nanolayers
- Possible problems:
  - Low reflectivity from membrane, low contrast

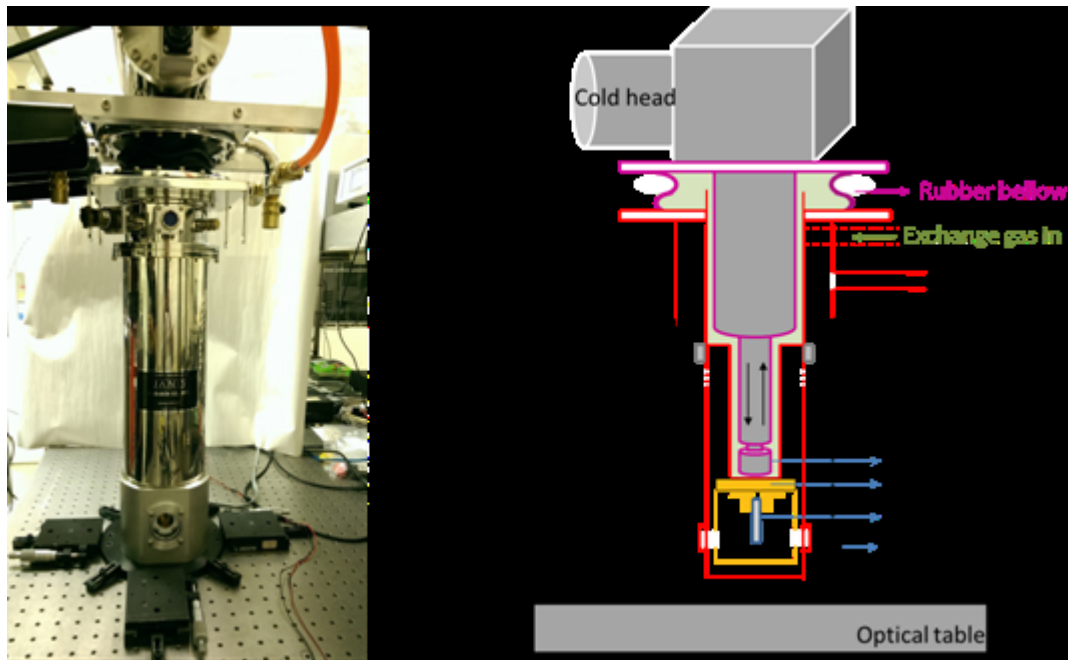


Zwickl, B. M., et al. "High quality mechanical and optical properties of commercial silicon nitride membranes." *Applied Physics Letters* 92.10 (2008): 103125-103125.



# NTHU Cryo Upgrade (2014-15)

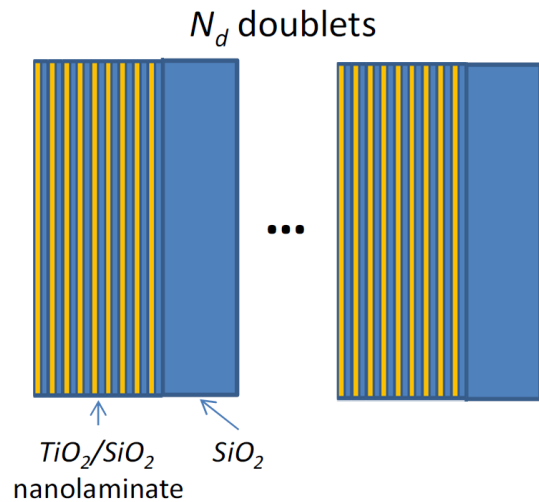
S. Chao at NTHU is upgrading his cantilever based Q measurement facility for cryogenic operation. New funding received from Taiwan National Research Council.



[S. Chao, LIGO-G1400806]

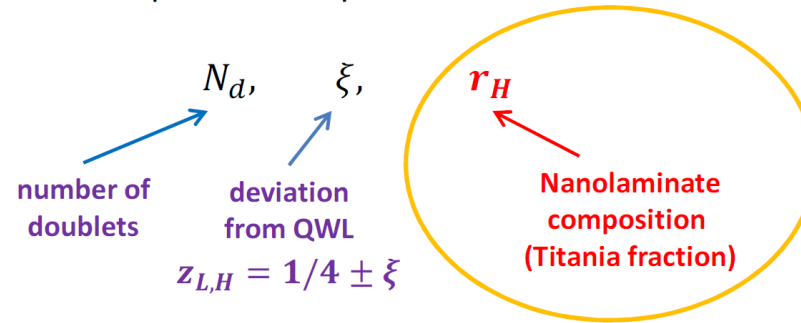
# Design Optimization (AdCOAT)

Generalizing results in [Principe et al, ch 12 in Harry et al, "Thermal Noise in Precision Measurements," Cambridge Un. Press, 2012] to *nanolayer based coatings*



Constrained transmittance @ 1064nm  
(e.g., 278ppm, TNI)

Free optimization parameters:



... comparison (on paper) to optimized TNI mirrors based on plain and Ti-doped Tantalum...



Little chance of being able to prototype such animal in Chao's Lab...

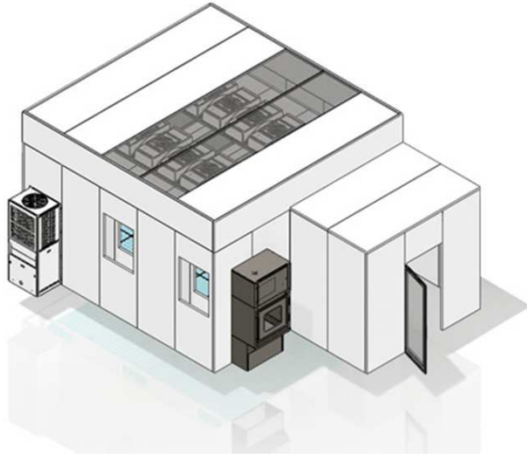
(courtesy M. Principe, INFN, U-Sannio)

# New Coating Facility (AdCOAT)

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**AdCOAT Usannio WG** – new funding from Regione Campania for new “big” Laboratory Facilities

➔ Includes, among others,



MITEC 45 m<sup>2</sup> class 10000  
reconfigurable clean room  
9 m<sup>2</sup> class 100 room.



Optotech OAC-75F  
coater

# New Coating Facility (AdCOAT)

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Optotech OAC-75F  
coater



- 700 mm  $\phi$  dome
- 75 mm  $\phi$  useful coating area
- Cryo pumping 90 kl/s
- Op. temp 30 to 300°K
- Multiple targets
- Ion gun 10 eV up to 300 eV
- Ar/O<sub>2</sub> atmosphere controls
- 6 MHz Quartz crystal (multiple) thickness control
- Shutters
- De-ionizing gun
- 2 replaceable wall liners
- Sander/cleaner

Make *thinner* layers (and hence more layers) to allow for *higher annealing temperatures*, and measure loss angle.

**Practical thickness limit  $\approx 2\text{nm}$ , may allow  $T_{\text{ann}} \approx 400\text{ C}$ ;**  
**Better (higher Q) substrates may be needed (Norcada's ?);**

Complete optical characterization and investigate correlation with macroscopic structural (TEM, AFM) features (Genoa).

**Need to characterize optical scattering losses of nanolaminates;**  
**Collaboration w. other LVC/KAGRA Groups sought.**

Investigate behaviour of nm-layered composites at cryogenic temperatures

**Cryogenic ringdown measurement facilities are being implemented at NTHU, Rome (GeNS) and Perugia ( $\text{Si}_3\text{N}_4$  membranes).**

# Upcoming upgrades

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We are getting

Better eyes to see, and

Better hands to do !

# Acknowledgements

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This work has been sponsored in part by EU through the EU-FP-7 ELiTES (IRSES) Project; Italian National Institute for Nuclear Physics (INFN) under the AdCOAT grant; National Science Council of Taiwan under NSC-1002221-E-007-099.