6th ET Symposium - Coating Session

LIGO-G1401358

nm-Layered Amorphous Glassy Oxide Composites for 3rd Generation Interferometric Gravitational Wave Detectors

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Lyon, November 19-20 2014

Outlook

Cold Mirrors aren’t Cool

Cryo Friendly Glassy Oxides

Doping vs Thinning

nm-Layered Composite Design

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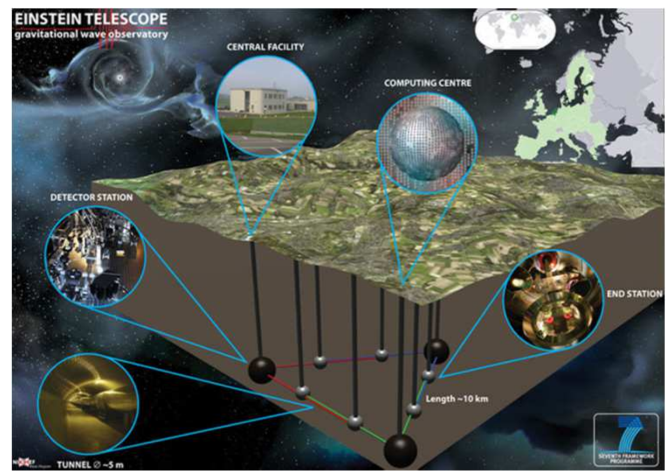
3rd Generation GW Detectors

Potential advantages of cryogenic IFOs early recognized since pioneering work by Kuroda and co-Workers [K. Kuroda et al., Int. J. Mod. Phys. D08 (1999) 557].

Medium scale cryogenic interferometer demonstrated (CLIO, 100m) [T. Uchiyama et al., J. Phys.: Conf. Ser. 32 (2006) 259]

KAGRA [K. Somiya et al., CQG **29** (2012) 124007] and ET [M. Punturo et al., CQG **27** 194002] will be cryogenic.

Many ideas (e.g., *He-II*) under discussion for a *cryo*-LIGO [*Workshop on Next Generation LIGO Detectors*, Caltech, 2012, see, e.g., LIGO-G120025] .

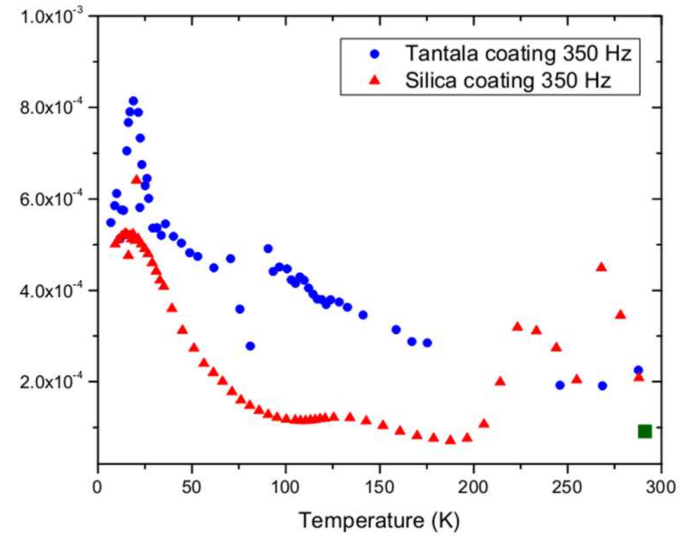


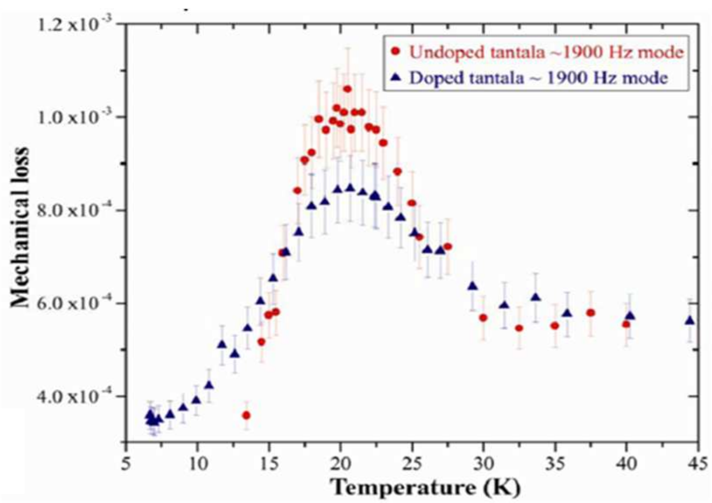
Cold Coatings aren't Cool

A cryogenic peak (up to 10 x loss angle increase) in the range 10 - 20 K is observed in many coating materials, consistent with (a spectrum of) thermally activated relaxation processes, with

$$\phi(\omega) \propto \frac{\omega\tau}{1+(\omega\tau)^2}\ ,\ \tau^{-1} = \tau_0^{-1}\, e^{-\frac{E_a}{k_B T}}$$

[I. Martin et al., CQG 25 (2008) 055005]





Ti-doped Tantala has lower losses compared to plain Tantala. Also at cryo temperatures.

[I. Martin et al., LIGO-G080313]

Both Silica (~ 10x loss angle increase) and Ti-doped Tantala (~ 4x loss angle increase) suffer from this.

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The Cryo-Peak Issue – Multilayers

Mechanical loss measurements on multi-layer Titania-doped-Tantala/Silica coatings on Silicon (annealed at 400 C~ 600 C) show a cryo-peak at ~ 30K **[Granata et al., Opt. Lett. 38, 5268 (2013)].**

Mechanical loss measurements on multi-layer Tantala/Silica coatings on Sapphire do *not* show such peak, yielding almost temperature-independent losses **[Yamamoto et al., PRD-74 022002 (2006); Hirose et al., LIGO-P1400107]** .

Reasons behind these discrepancies yet to be understood .

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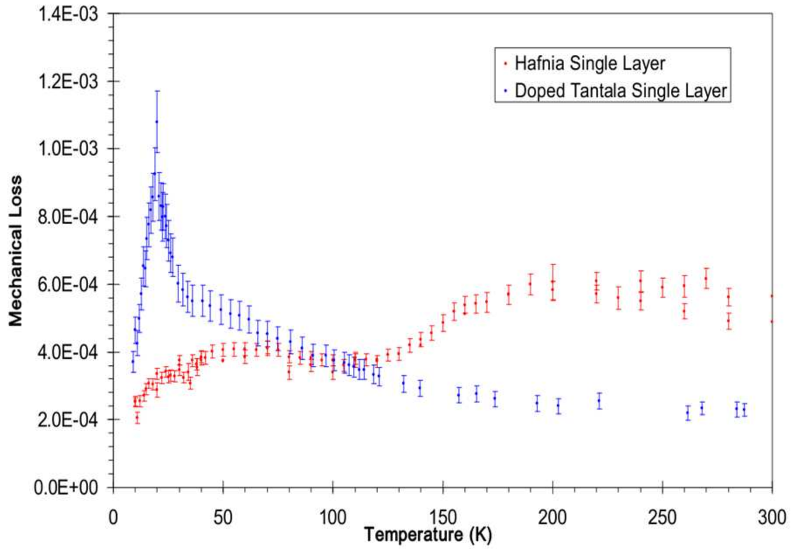
Hafnia



" Initial results of the mechanical loss of HfO_2 do not show a large peak in dissipation at T ~ 20K in contrast to Ta_2O_5 "

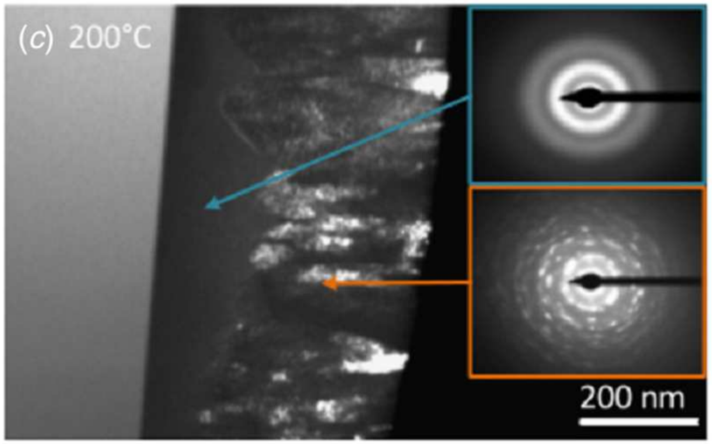
" Broad peaks do appear to occur at ~50K and ~200K – could this indicate a different dissipation mechanism? "

[E. Chalkley et al., LIGO-G080314]

Hafnia coatings crystallize upon annealing.

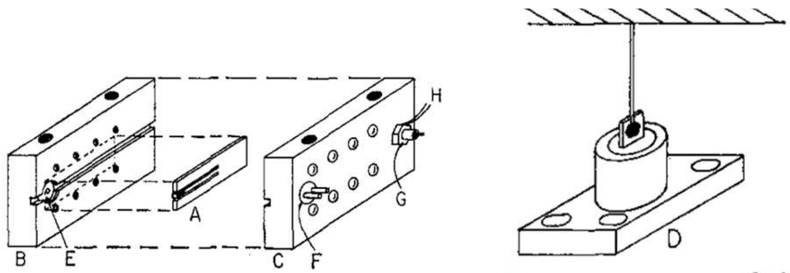
"Doping of the hafnia coatings with silica has been suggested as a viable method for preventing the crystallization of the hafnia coatings, possibly improving their mechanical loss and optical properties."

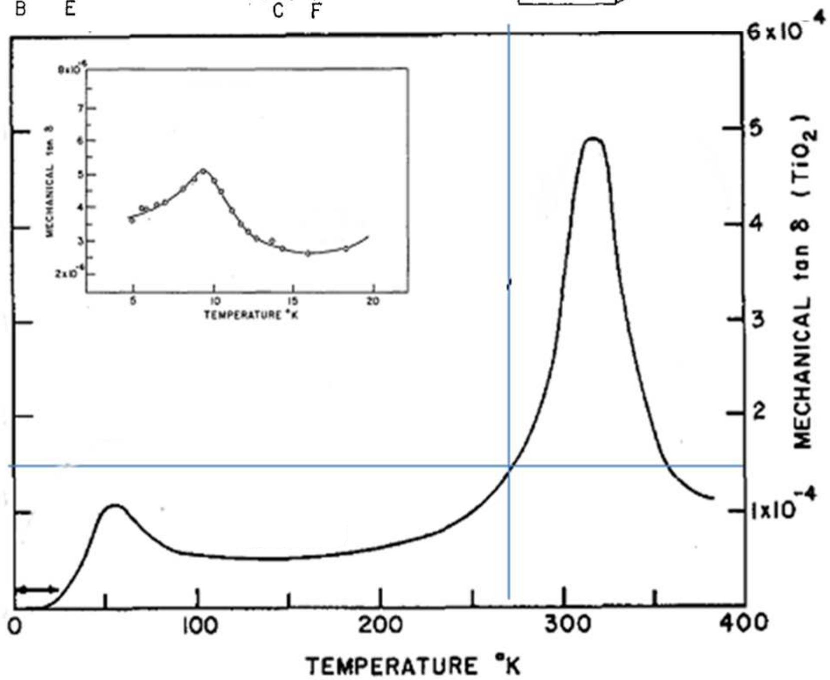
[M. Abernathy et al., CQG 28(2011) 195017]



α-Titania

A—sample; B, C—steel holders; D—steel supporting yoke; E—driver coil; F—capacitive pickup; G—germanium thermometer; H—copper-constantan thermocouple.





Results in Scott and MacCrone, Rev. Sci. Instr. 39 (1968) 821 obtained from a cantilever - like based ring-down measurement setup suggest that (amorphous) Titania may be almost exempt from a cryogenic mechanical loss peak.

Fresh measurements needed !

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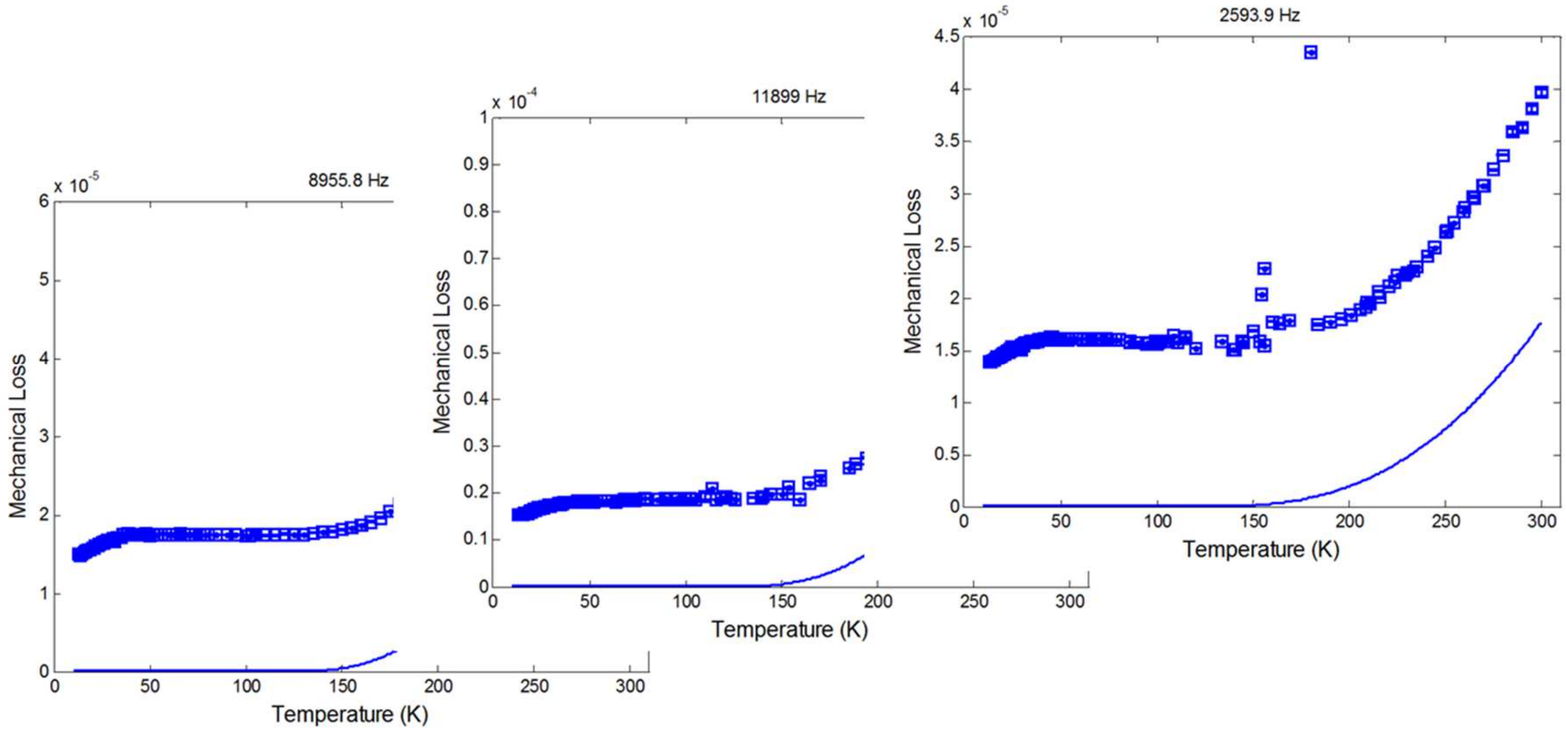
a-Titania, contd.

Mechanical loss of (as deposited) *a*-TiO_2 on silicon has been measured at Glasgow [**Preliminary data**, **courtesy Iain Martin & Peter Murray, 2014**] .



…almost no cryopeak found

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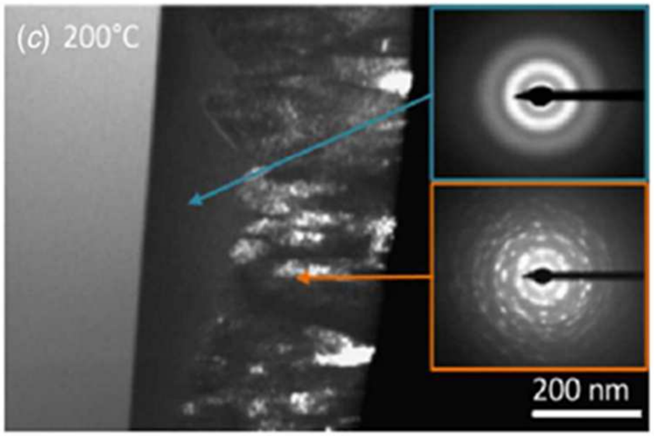
Si-Doping Contrasts Crystallization

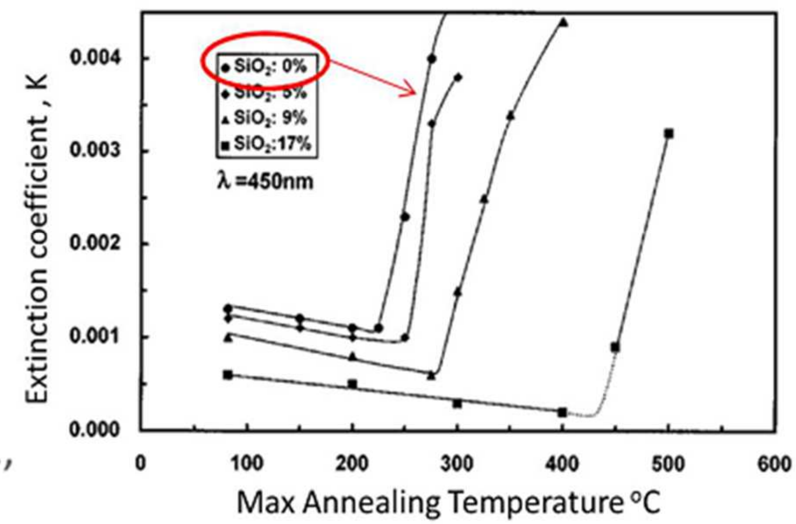
Long since known result:

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



[Abernathy, CQG 28(2011) 195017]

SiO_2 *doping stabilizes* HfO_2 (and ZrO_2) against *crystallization* [Ushakov et al., Phys. Stat. Sol. B241 (2004) 2268].



SiO_2 *doping stabilizes* TiO_2 against *crystal-lization* [Wang and Chao, Opt. Lett. 23 (1998) 1417; Chao, JOSA A16 (1999) 1477; Chao et al., Appl. Opt. 40 (2002) 2177]

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Si-Doped Titania

ThD2.pdf © 2007 OSA/OIC 2007

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

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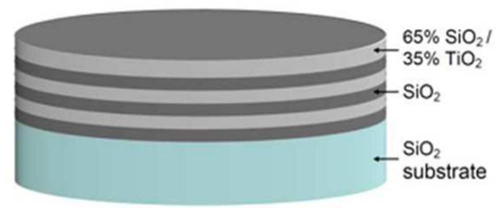
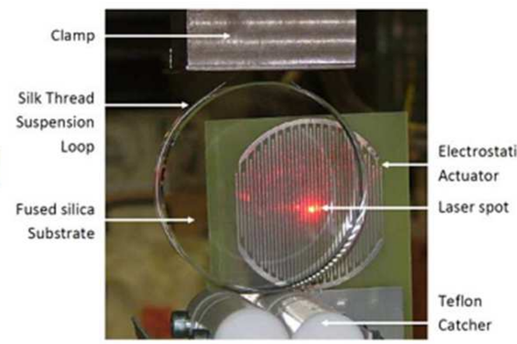
Abstract: Ion-beam-sputtered mixtures of silica and titania are investigated as potential coating materials for use in gravitational wave interferometer optics. Such coatings must have both low optical and mechanical loss to maximize detection sensitivity.

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

Loss angle measurements made by P. Murray in his PhD thesis.





$(\lambda/4, \lambda/4)^{15}$	$10^4 \times$ (residual loss angle)	n_H
Ta_2O_5 / SiO_2	4.4 ± 0.2	2.02
TiO_2:: Ta_2O_5 / SiO_2 (15::85)	2.4 ± 0.2	2.07
SiO_2:: Ta_2O_5 / SiO_2 (35::65)	2.5 ± 0.4	1.83
TiO_2:: Si_2O_2 / SiO_2 (35::65)	1.7 ± 0.4	1.77

Almost as good as Titania dopd Tantala, but lower index.

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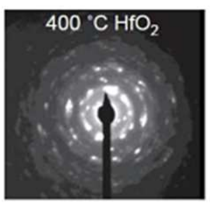
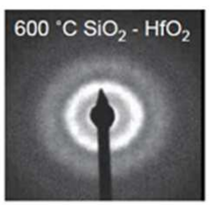
No Cryo Peak in Si-Doped Hafnia

Silica doping, besides halting crystallization upon annealing, does **not** spoil nice (no cryo-peak) behaviour of undoped Hafnia.

Mechanical losses 4 times lower @ 20 K compared to Titania doped Tantala



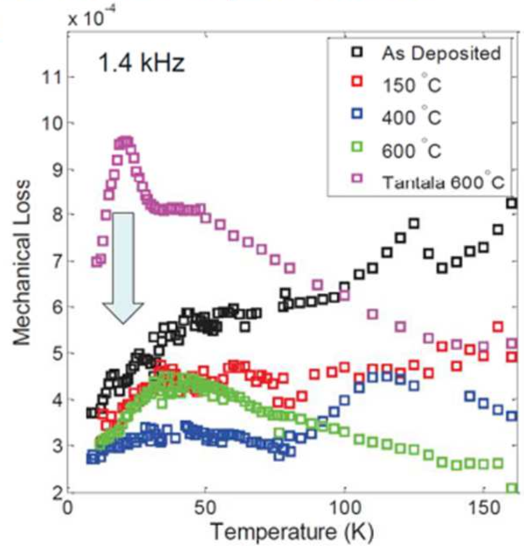


30% SiO_2 doped HfO_2 (0.5 μm thick on Silicon cantilever)

University of Glasgow

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]

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A Different Option: *Thin(ner)* Films…

Thinn(er) films crystallize at *high(er)* temperatures

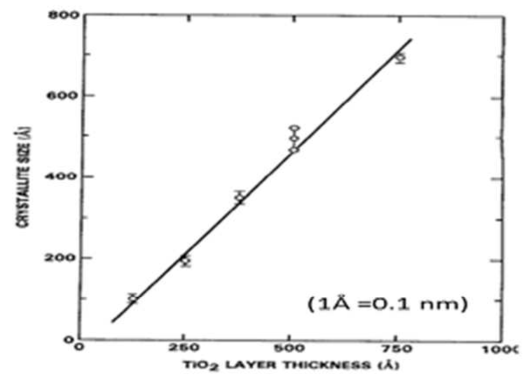
Seminal work on ***thin – layer Titania*** films by Sankur & Gunning [J. Appl. Phys. 66 (1989) 4747]

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

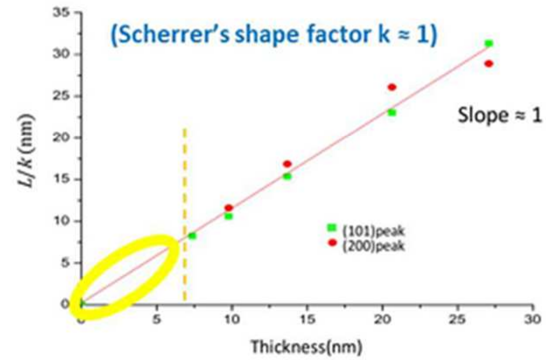
"Grain size, as deduced from diffraction line broadening, was comparable to the layer thickness"

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

"Below a certain critical thickness crystallization in pure TiO_2 films is inhibited"



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



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

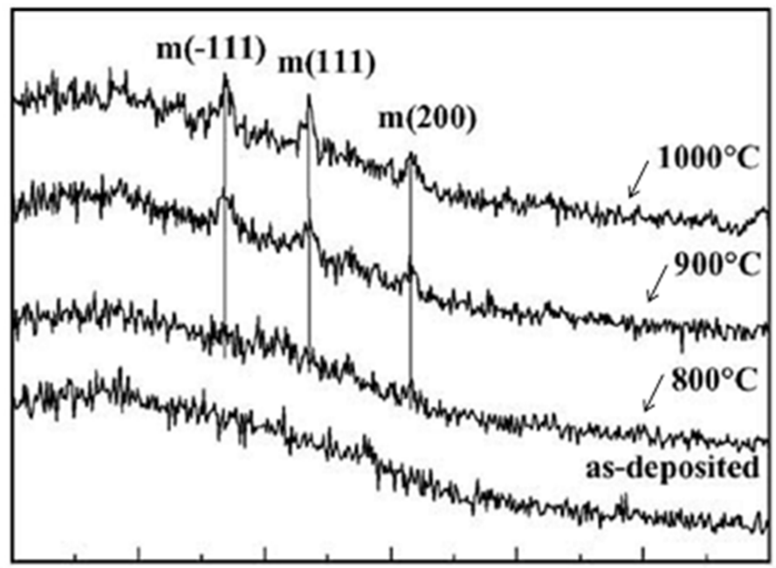
nm Layered Hafnia-Alumina Composites

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 anneal-ing temperature of 800 °C"

"FTIR indicates that no interface layer forms during annealing up to 800 °C"



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Sub-wavelength (nm) Layered Composites

SWL composite properties depend *only* on the constituents' properties and the thickness ratio $d_{H,tot}/d_{L,tot}$

$$n_{eff} = \left[r_H {n_H}^2 + (1 - r_H){n_L}^2\right]^{1/2}$$

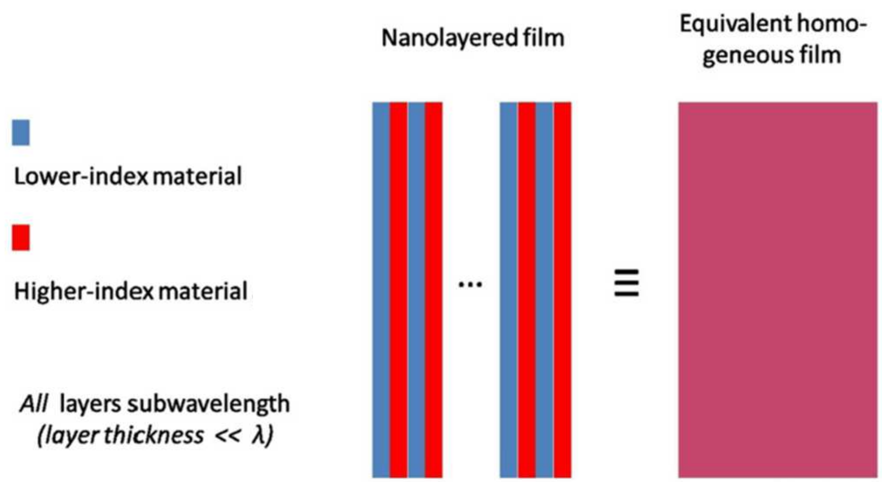
Drude's formula

$${Y_f}^{\parallel} = r_H Y_H + (1 - r_H) Y_L$$

Voigt formula

$${Y_f}^{\perp} = [r_H/Y_H + (1 - r_H)/Y_L]^{-1}$$

Reuss formula



$$\phi_f = \frac{\left(\frac{Y_s}{Y_H} + \frac{Y_H}{Y_s}\right) r_H \phi_H + \left(\frac{Y_s}{Y_H} + \frac{Y_H}{Y_s}\right)(1 - r_H)\phi_L}{Y_s[r_H/Y_H + (1 - r_H)/Y_L] + {Y_s}^{-1}[r_H/Y_H + (1 - r_H)/Y_L]^{-1}}$$

$$r_H = \frac{d_{H,tot}}{d_{L,tot} + d_{H,tot}}$$

Harry's formula

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nm-Layered Composite Design

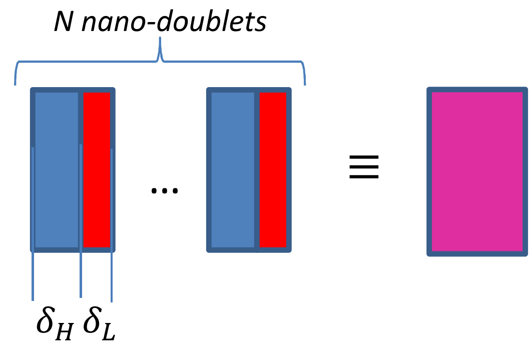
The simplest geometry uses **cascaded nano-doublets**, and is thus specified by $(\boldsymbol{N}, \boldsymbol{\delta_H}, \boldsymbol{\delta_L})$.

For given $n_{L,H}$, **prescribing the composite index** $\boldsymbol{n_{eff}}$ **determines uniquely the thickness ratio** of the low / high index materials in it (from Drude's equation),

$$\frac{\boldsymbol{\delta_L}}{\boldsymbol{\delta_H}} = \left(\frac{\boldsymbol{n_H}^2 - \boldsymbol{n_{eff}}^2}{\boldsymbol{n_{eff}}^2 - \boldsymbol{n_L}^2}\right)$$

Prescribing the **optical thickness** ***z*** **of the composite material** (in units of the local wavelength), and the *minimum* thickness of the nano-layers) yields *all equivalent* slab design parameters (N, δ_H, δ_L), from

$$\boldsymbol{N(\delta_H + \delta_L) = z\lambda_0 n_{eff}^{-1}}$$



Equivalent TiO_2/SiO_2 subwavelength doublet based, QWL thick composites with n_{eff}=2.09

N	$\delta_{TiO_2}[nm]$	$\delta_{SiO_2}[nm]$
1	78.0559	49.2168
2	39.0279	24.6084
3	26.0186	16.4056
4	19.514	12.3042
5	15.6112	9.84337
6	13.0093	8.20281
7	11.1508	7.03098
8	9.75699	6.1521
9	8.67288	5.46854
10	7.80559	4.92168
11	7.09599	4.47426
12	6.50466	4.1014
13	6.0043	3.78591

N	$\delta_{TiO_2}[nm]$	$\delta_{SiO_2}[nm]$
14	5.57542	3.51549
15	5.20373	3.28112
16	4.87849	3.07605
17	4.59152	2.89511
18	4.33644	2.73427
19	4.1082	2.59036
20	3.90279	2.46084
21	3.71695	2.34366
22	3.548	2.23713
23	3.39373	2.13986
24	3.25233	2.0507
25	3.12224	1.96867

Co-sputtered Composites (EMT Model)

Mesoscopic (*Effective Medium Theory*) approach [I. Pinto et al., LIGO-G100372, LIGO-G1100937] – **see** [D. Aspnes, Am. J. Phys, 50 (1982) 704] **for foundations**.

- Bruggemann dielectric mixture formula [D.A. Bruggeman Ann. Phys. 24 (1935) 636; C.C. Lee, C.J. Tang, Appl. Opt., 45 (2006) 9125]

$$\eta_2 \frac{{n_2}^2 - n^2}{\gamma {n_2}^2 + (1-\gamma) n^2} = (1-\eta_2) \frac{n^2 - {n_1}^2}{\gamma {n_1}^2 + (1-\gamma) n^2}$$

- Barta's extension of Bruggemann formulas to viscoelastic properties [S. Barta, J. Appl. Phys. 75 (1994) 3258]

Define : $y = \sigma - 2 \ , \ X = \dfrac{\sigma Y}{\sigma + 1}$

$$\begin{cases} (1-\eta_2)\dfrac{X - X_1}{2X + (X_1/y_1)(\sigma_1 + 1)} + \eta_2 \dfrac{X - X_2}{2X + (X_2/y_2)(\sigma_2 + 1)} = 0 \\ (1-\eta_2)\dfrac{(X/y) - (X_1/y_1)}{2X + (X_1/y_1)(\sigma_1 + 1)} + \eta_2 \dfrac{(X/y) - (X_2/y_2)}{2X + (X_2/y_2)(\sigma_2 + 1)} = 0 \end{cases} ,$$

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Co-sputtered vs Nanolayered, I – Optical

Cosputtered (Bruggemann formula)

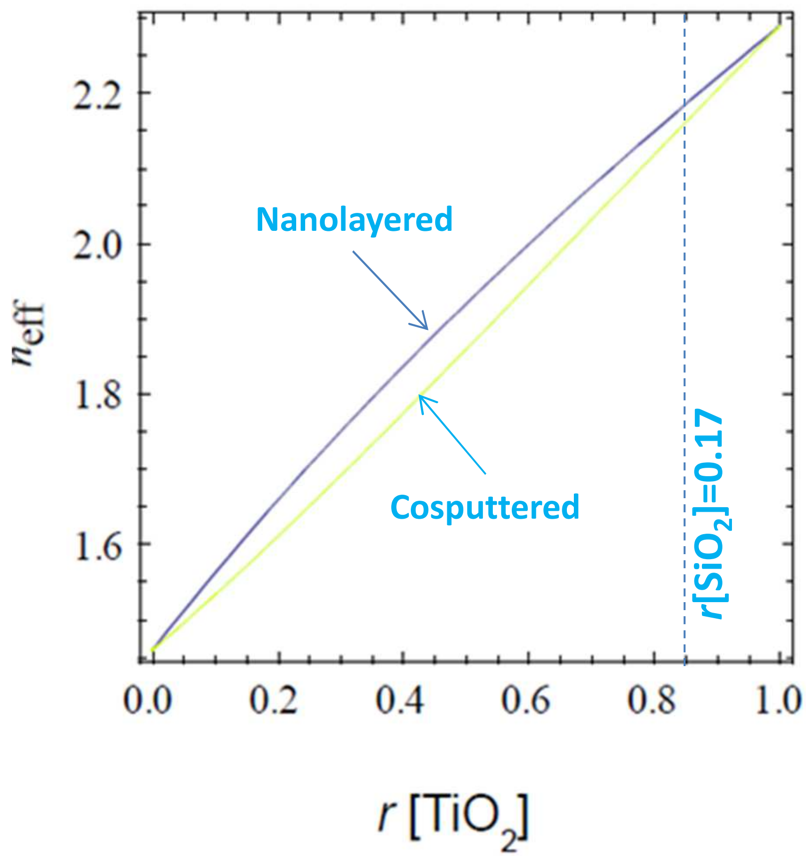
$$r_H \frac{n_H{}^2 - n_{eff}{}^2}{\gamma n_H{}^2 + (1-\gamma) n_{eff}{}^2} = (1 - r_H) \frac{n_{eff}{}^2 - n_L{}^2}{\gamma n_L{}^2 + (1-\gamma) n_{eff}{}^2}$$

γ is Bruggemann shape-factor (1/3 for sph. incl.)

Nanolayered (Drude's formula)

$$n_{eff} = \left[r_H n_H{}^2 + (1 - r_H) n_L{}^2 \right]^{1/2}$$

SiO_2/TiO_2 Composites



Co-sputtered vs Nanolayered, II - Mechanical

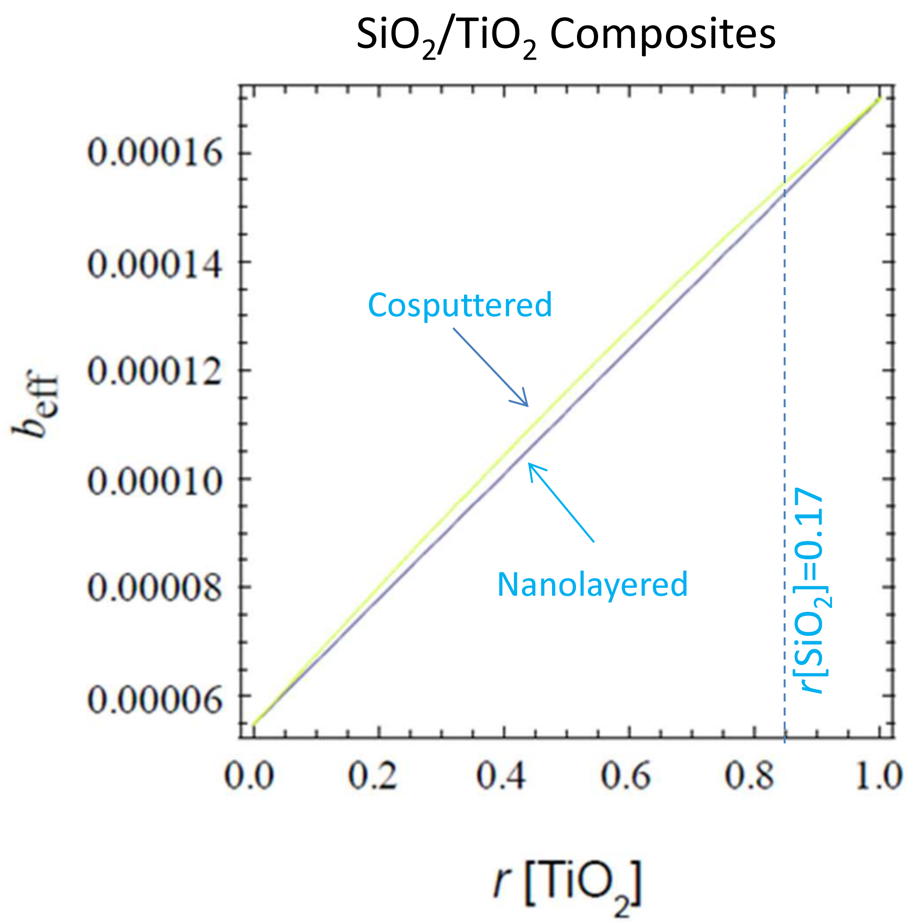
Material noisyness embodied in coefficients

$$b_{eff} = \frac{\phi_{eff}}{n_{eff}} \left(\frac{Y_{eff}}{Y_s} + \frac{Y_s}{Y_{eff}} \right)$$

to be used in

$$\phi_c \cong \frac{\lambda_0}{w_m \sqrt{\pi}} (b_L d_L + b_H d_H)$$

(Silica substrate assumed)



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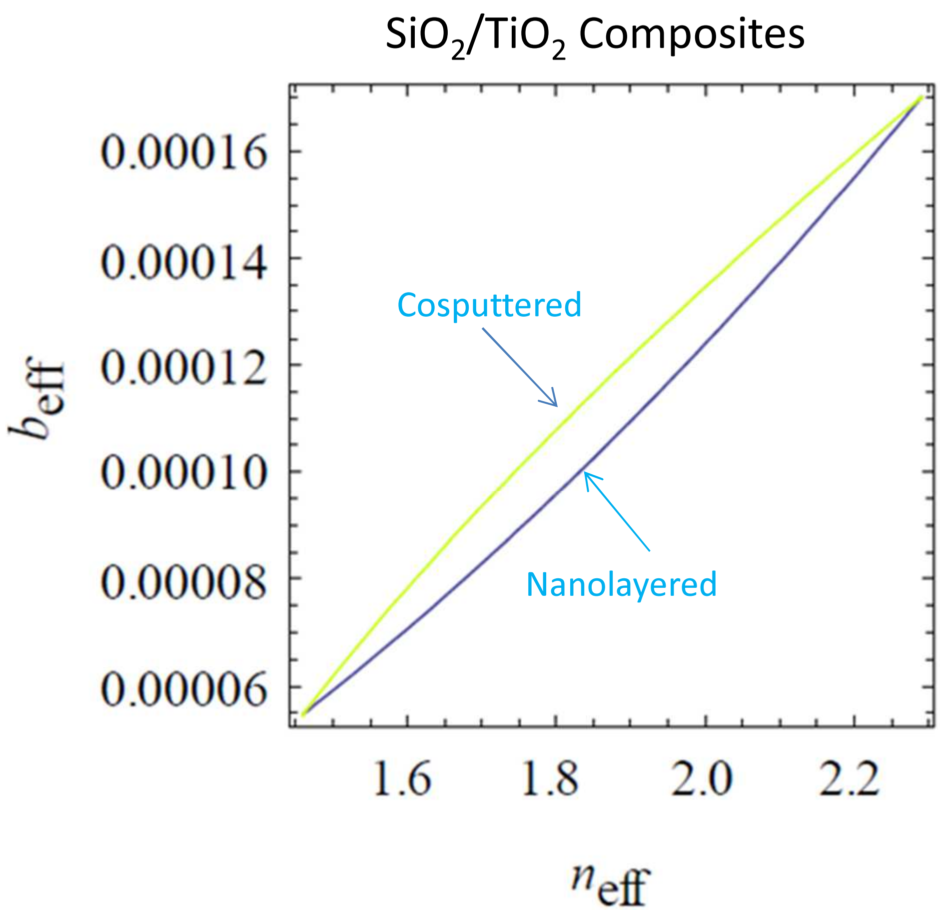
Co-sputtered vs Nanolayered, III



Nanolayered SiO2/TiO2 composites are *less noisy* , compared to co-sputtered SiO2/TiO2 composites having *the same refraction index*

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Technological Challenges ?

Even *relatively large thickness` errors* in the *individual* low/high index layer *thicknesses* are *irrelevant*, provided *each layer is sub-wavelength* and *the total thickness ratio has the design value*.

Other ?

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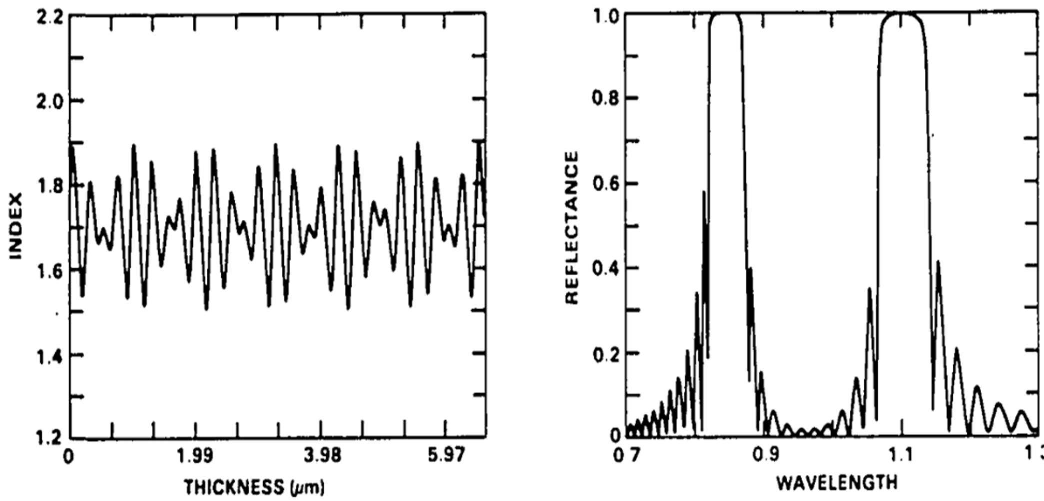
Good Old Technologies I: Rugates

Rugate dichroic mirror coating



Rugates are widely used in coating designs. They offer, e.g., the simplest option for *N*-chroic operation, obtained from the superposition of *N* sinusoidal *index modulations*, viz:

$$n(x) = n_0 + \sum_{k=1}^{N} \Delta n_k sin\left(\frac{2\pi x}{\Xi_k}\right) \quad (1)$$

In practice, one uses ***staircase approximations*** for the sine functions in (1), where each *sub-wavelength-thick* layer can be obtained by *sandwiching two nm-thick layers made of a low (L) and high (H) index material, such that*

$$\frac{d_L}{d_H} = \left(\frac{{n_H}^2 - n^2}{n^2 - {n_L}^2}\right)$$

[W. H. Southwell, Appl. Opt. 24 (1985) 457-460]

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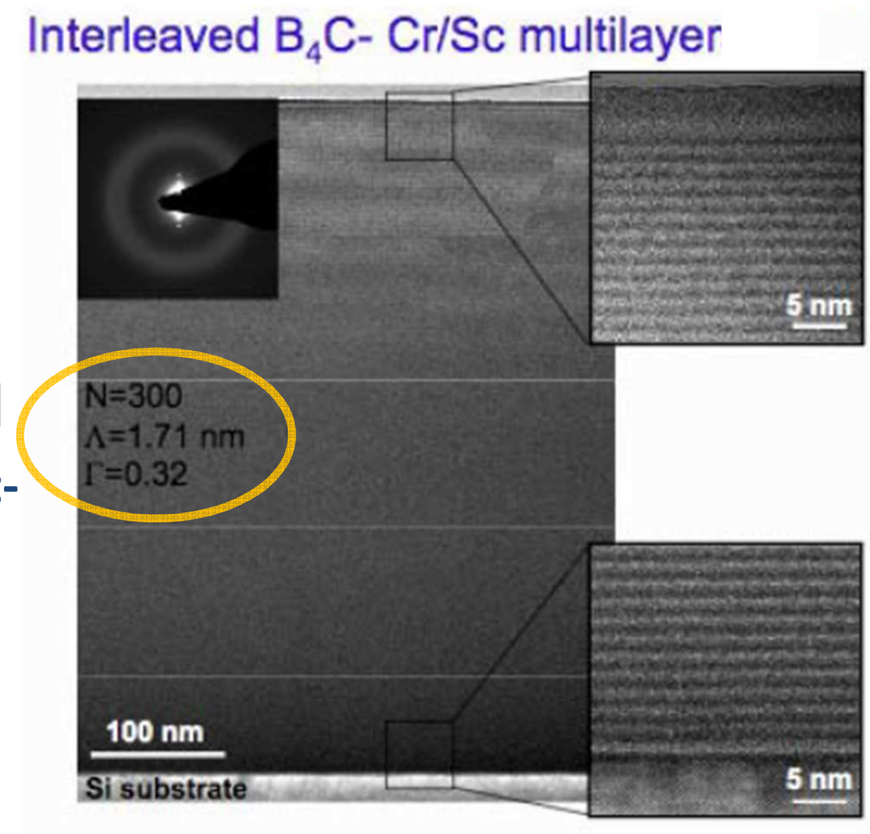
Good Old Technologies II: X-Ray Mirrors

Interference mirrors consisting of hund-reds/thousands of nm scale layers, with sub - nm precision [see, e.g., Proc . 10th PXRMS Conf. (2008)] **, using**

- **Interleaved nm-scale "buffering" layers to prevent crystallization & maintain flat-ness** [E. Gullikson, Proc. 8th PXRMS (2006)]
- **Ion assisted (modulated) magnetron sput-tering** [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]



See also [R. DeSalvo, LIGO-G080106] **for a survey.**

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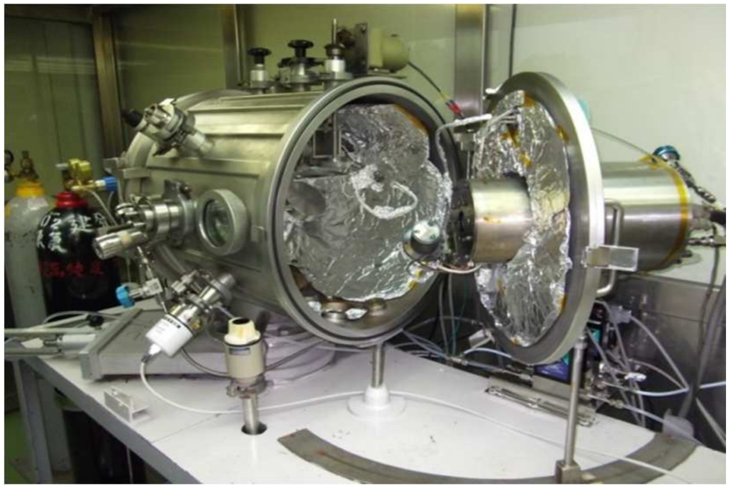
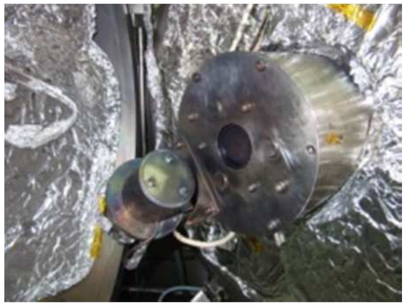
NTHU Deposition Facility

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

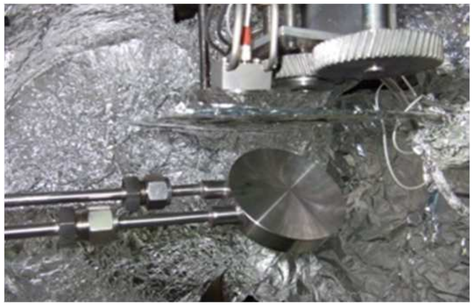
[S. Chao et al, LIGO-G1101083, G1200489, G1300921]]



Kaufman gun & neutralizer

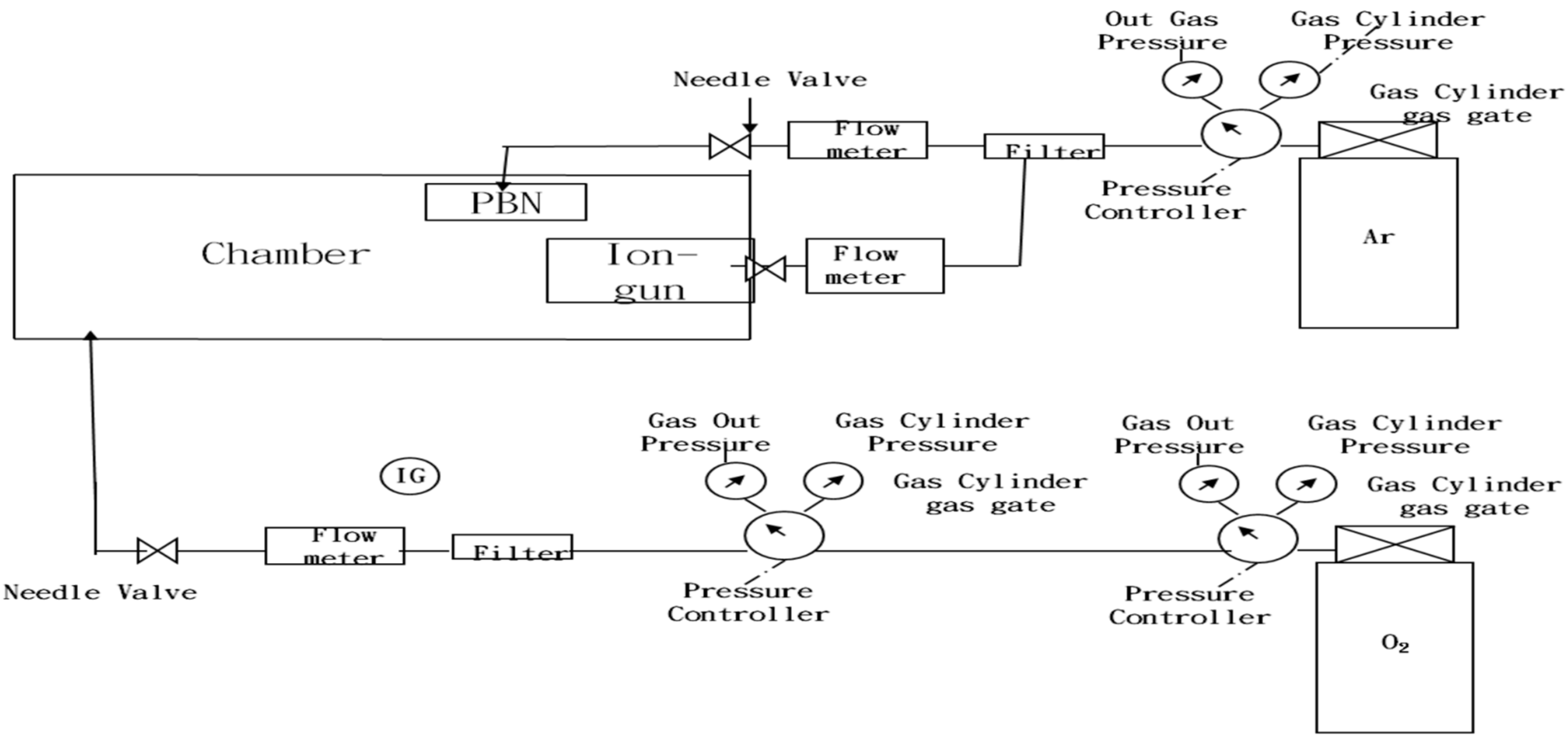


Sputter target and rotator



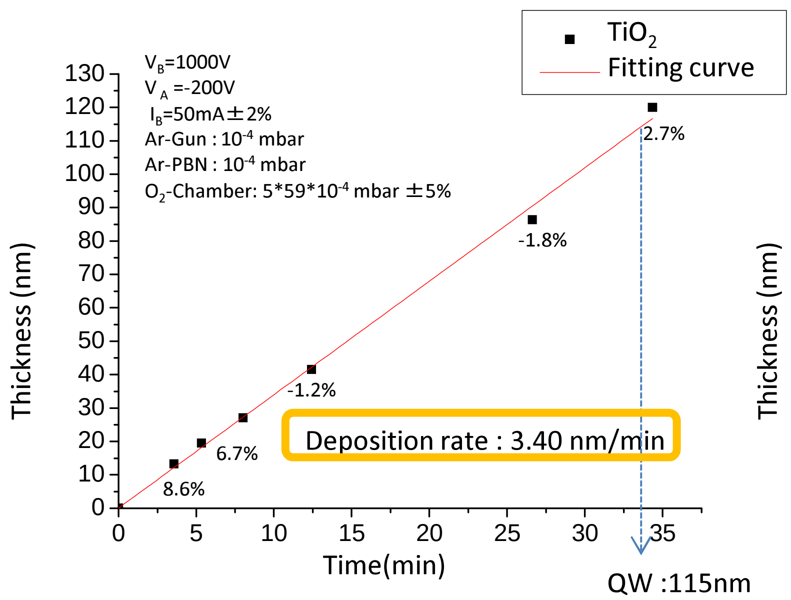
Twin exch. target holder

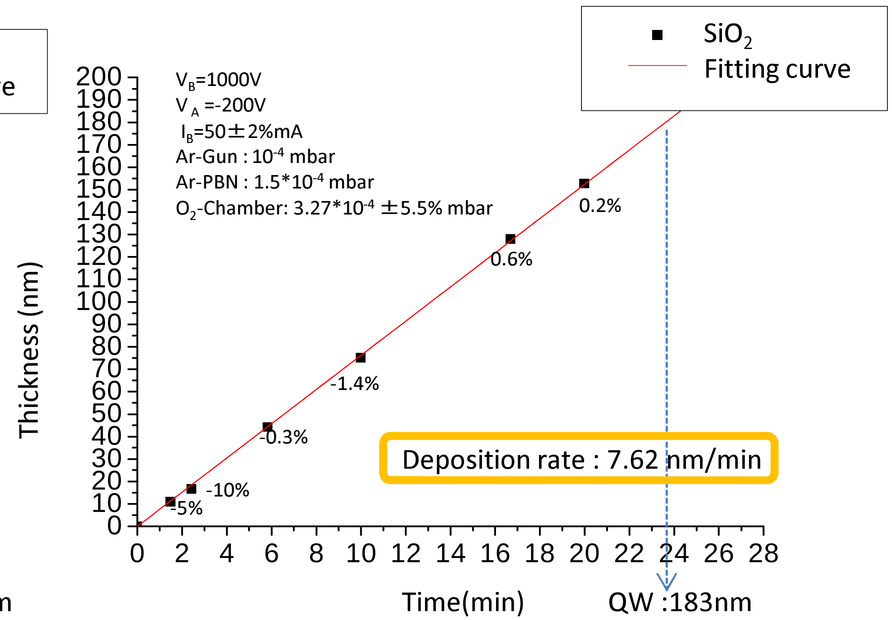
NTHU Deposition Facility



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

Deposition Rate / Calibration





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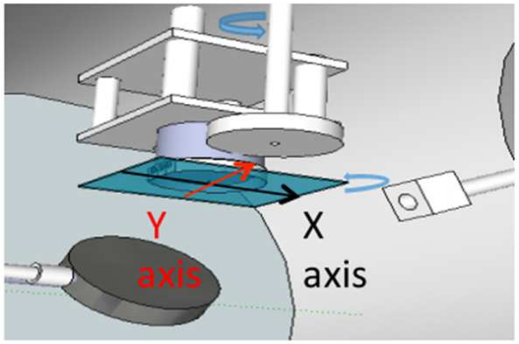
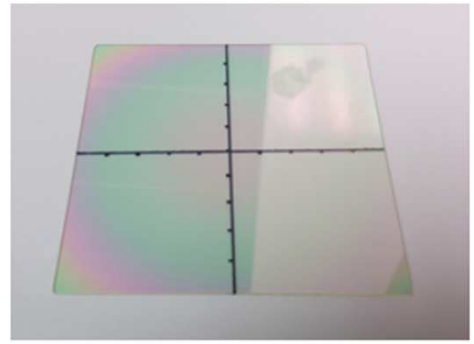
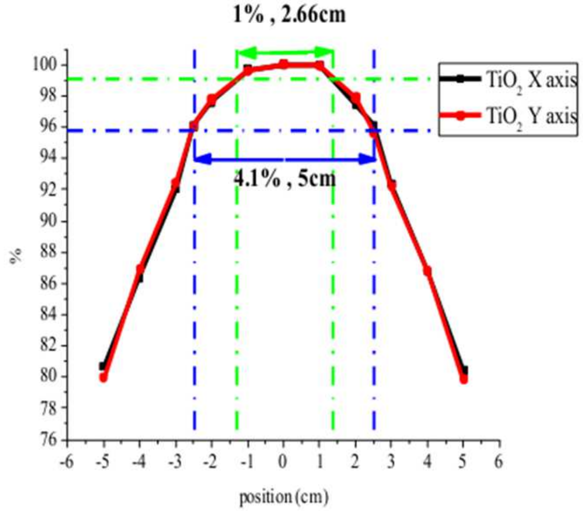
Deposition Uniformity

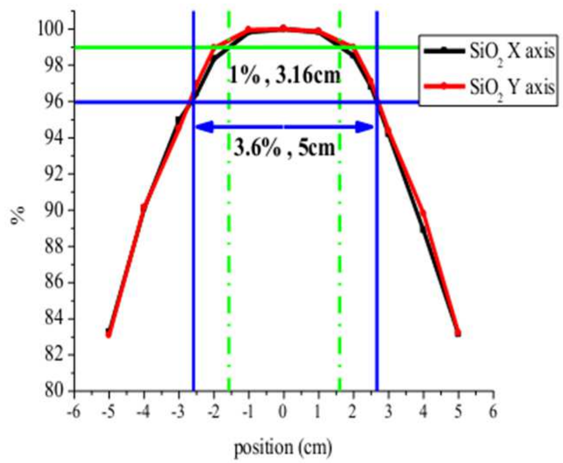




TiO_2



SiO_2



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

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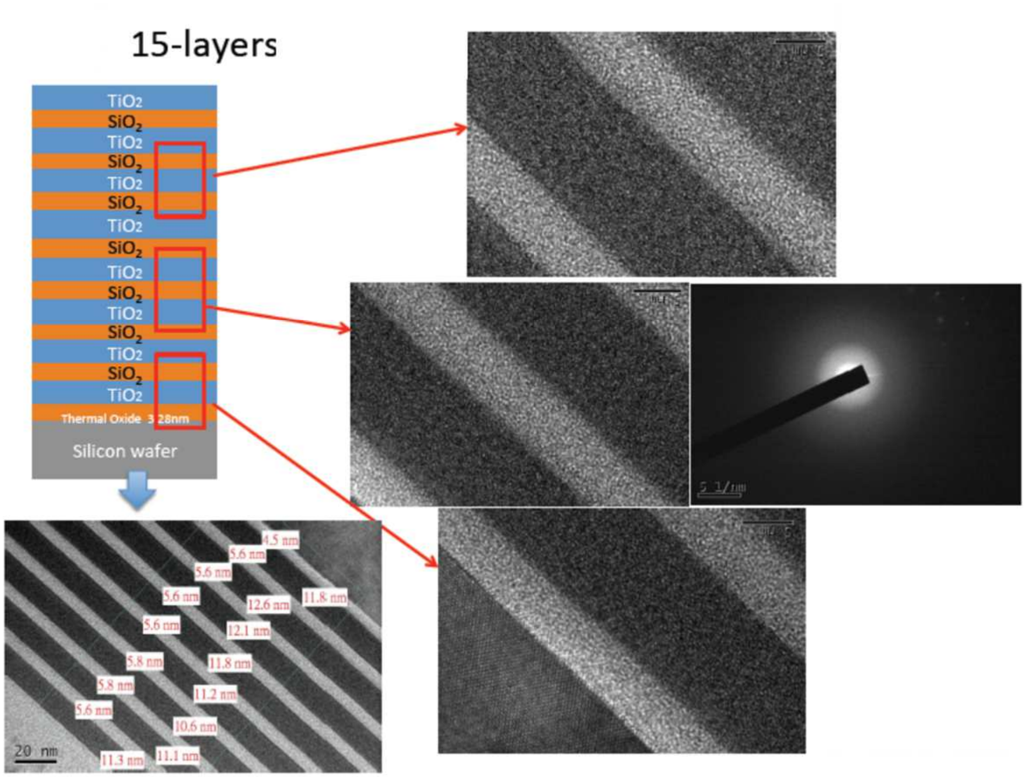
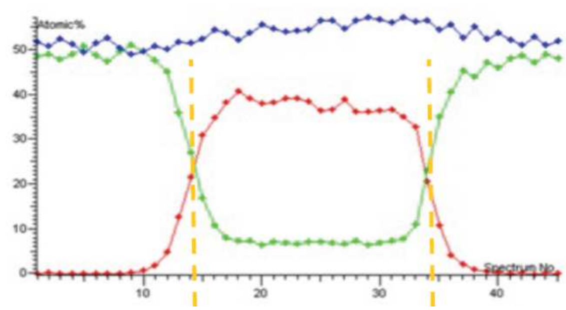
nm-Layered Prototypes



Morphology of witness samples investigated using TEM and electron diffraction.

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



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

1st Generation nm-Layered Prototypes

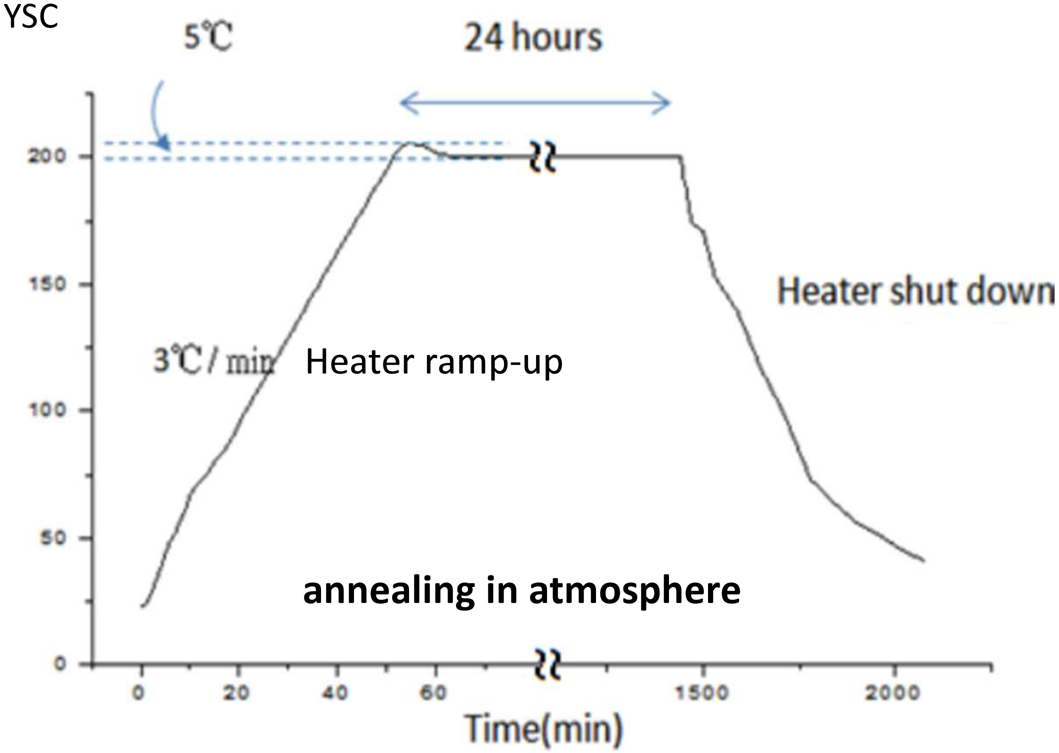
	Total thickness (nm)	Averaged thickness of TiO_2 and SiO_2 layer(nm)	
		TiO_2	SiO_2
single TiO2	121.9	121.9	0
3 layer	119.8	40.9	40.7
5 layer	119.2	26.2	20.3
7 layer	120.0	20.6	12.5
11 layer	119.3	13.7	7.4
15 layer	112.4	9.8	4.8
19 layer	112.6	7.4	4.3

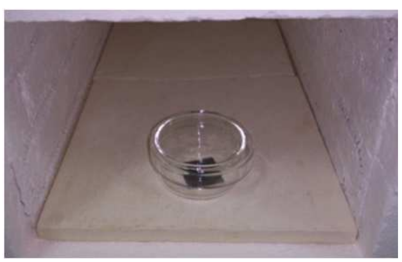
[S. Chao et al., LIGO-G1300921]

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

NTHU Annealing Facility

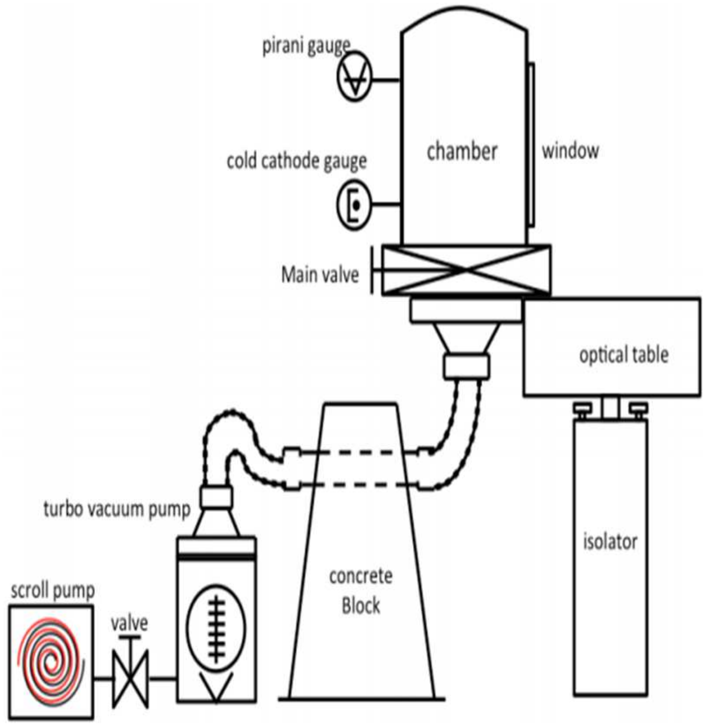
Model : YF-4
Company : YSC

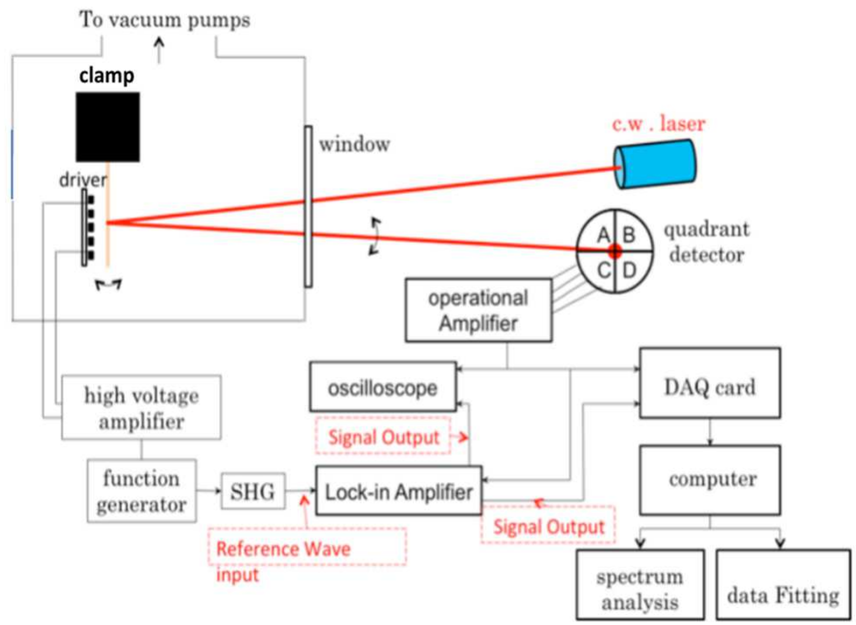




[S. Chao et al., LIGO-G1300921]

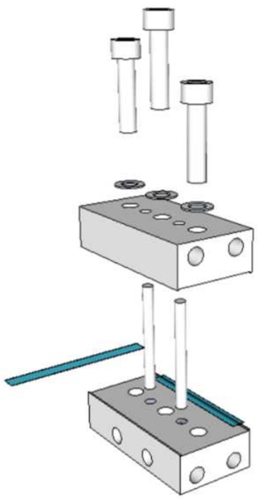
NTHU Cantilever Setup

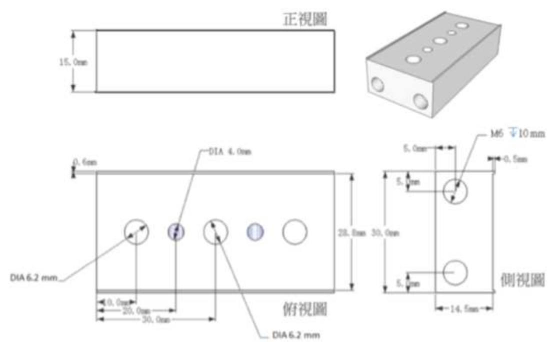


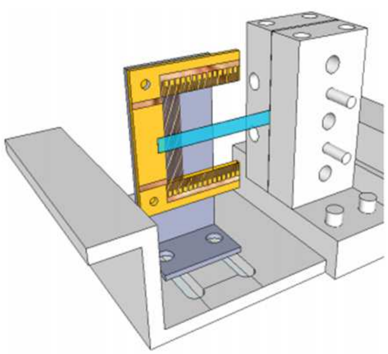


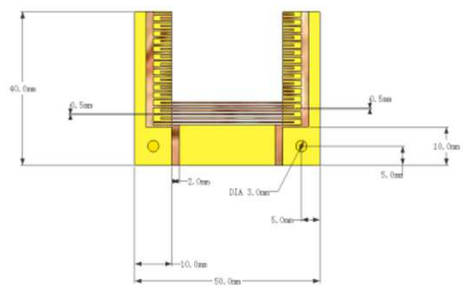
[S. Chao, LIGO-G1200489]

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]

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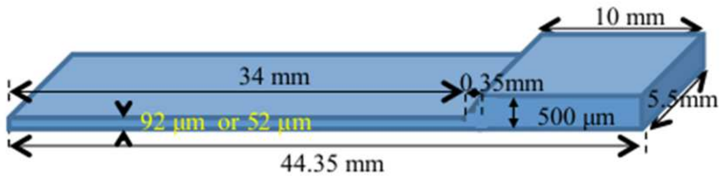
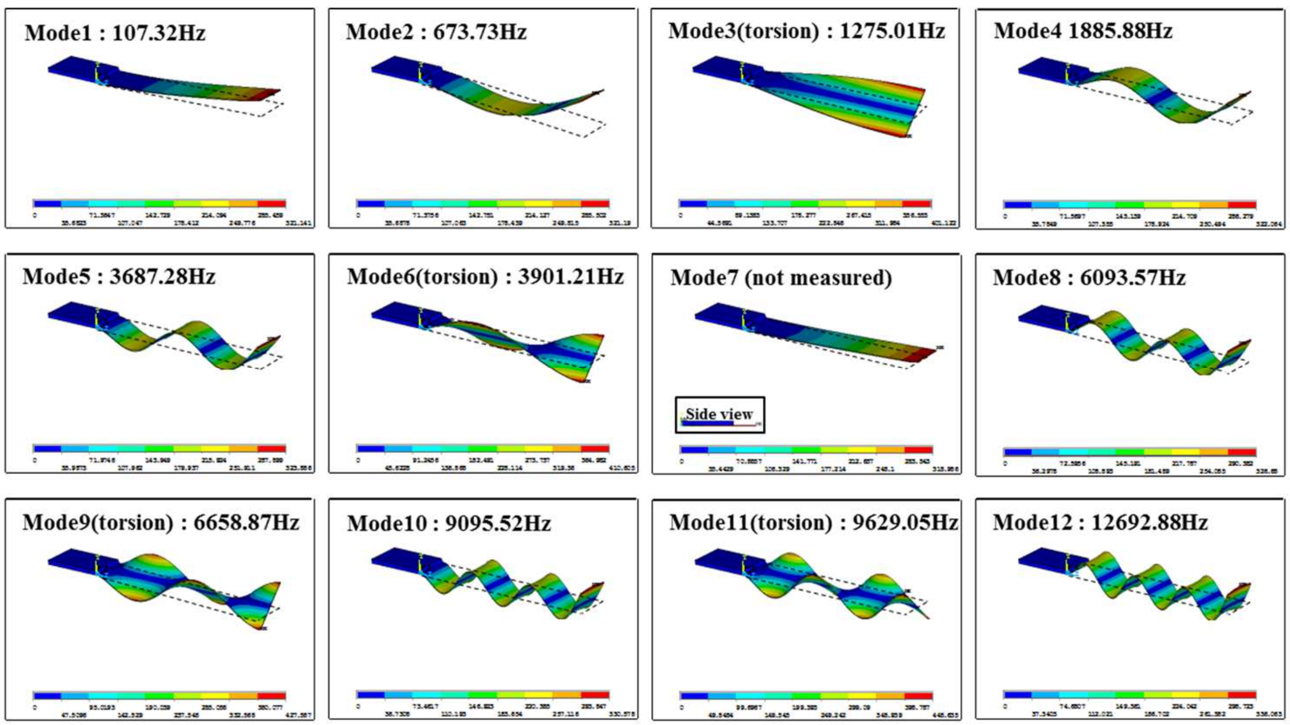
Cantilever Design



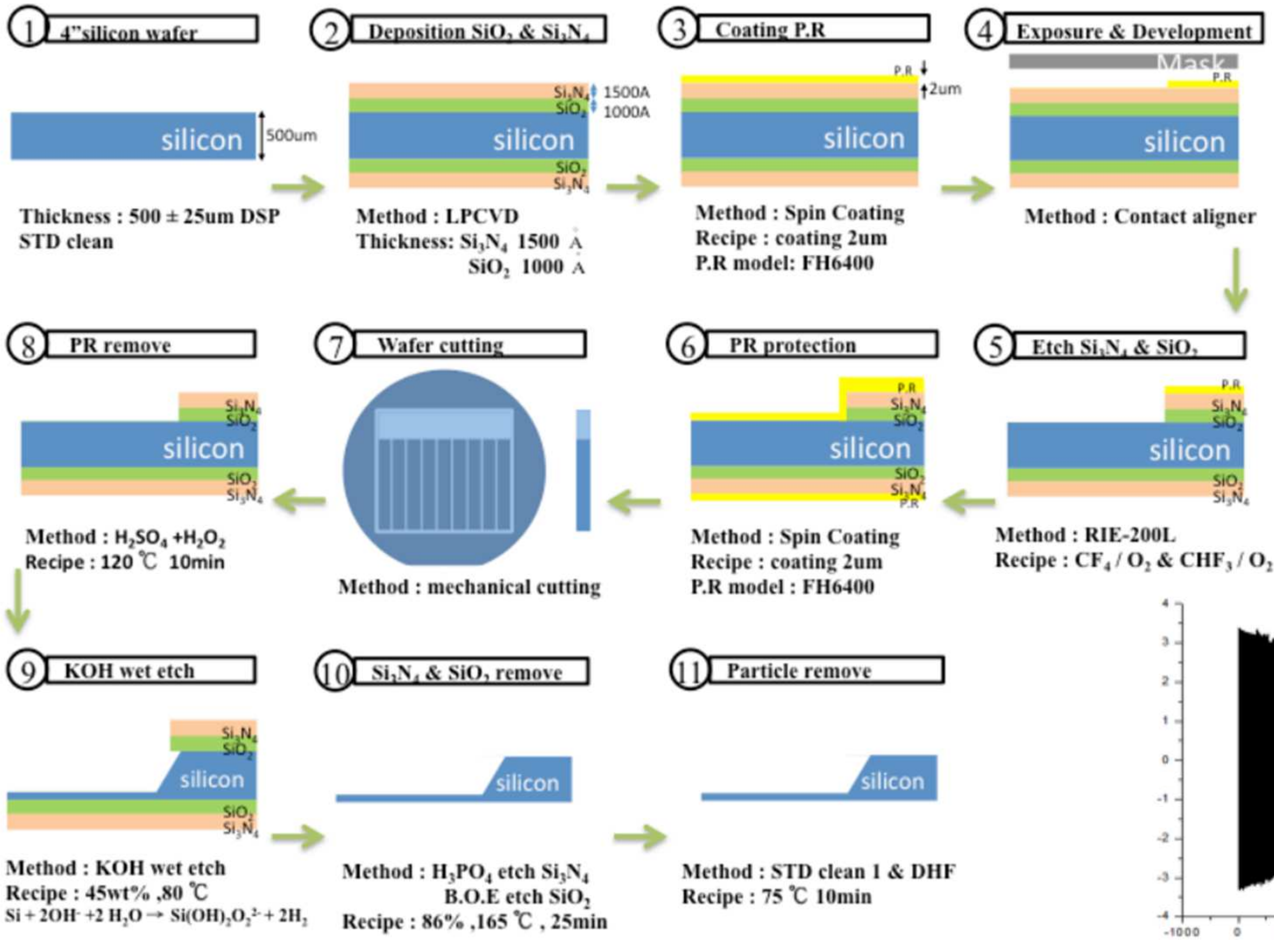
Cantilever fabricated from (100, undoped) 4” silicon wafer by KOH wet etching.

[S. Chao, LIGO-G1200849]

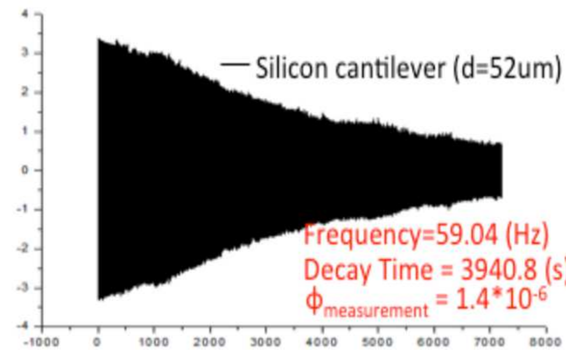


[J. Wang, MA Thesis, NTHU, 2012]

Cantilever Production



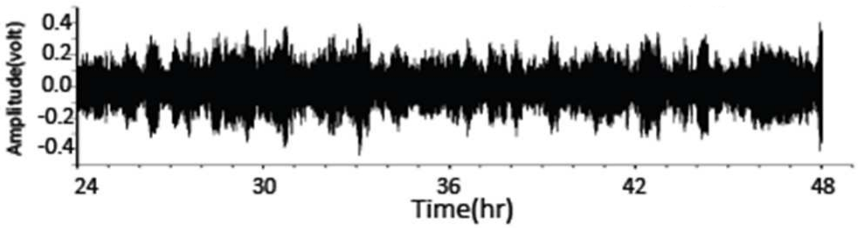
2-side polished wafer – use non KOH etched face for coating dep.

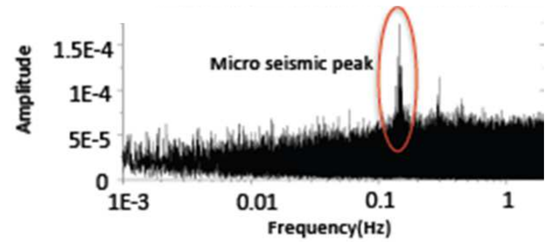


(collab. W. Taiwan National Nanotech Device Lab (NDL)

[S. Chao, LIGO-G1200849]

Ringdown Error Analysis





48 hours background noise (measured) and its low frequency spectrum

Error simulation method

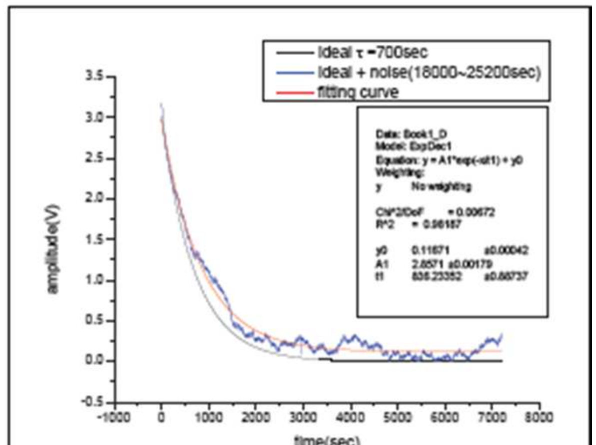
(1) Pick up a segment of continuous 2 hours background noise from the measurement

(2) Add the noise to an ideal noiseless ring-down curve

(3) Fit the curve to find the decay time

(4) Repeat the simulation many times

(5) Obtain the statistics for the decay time



A typical simulation result

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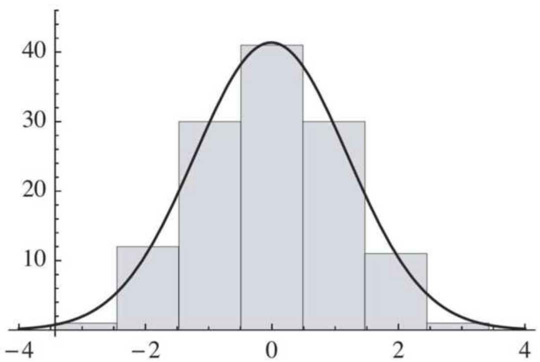
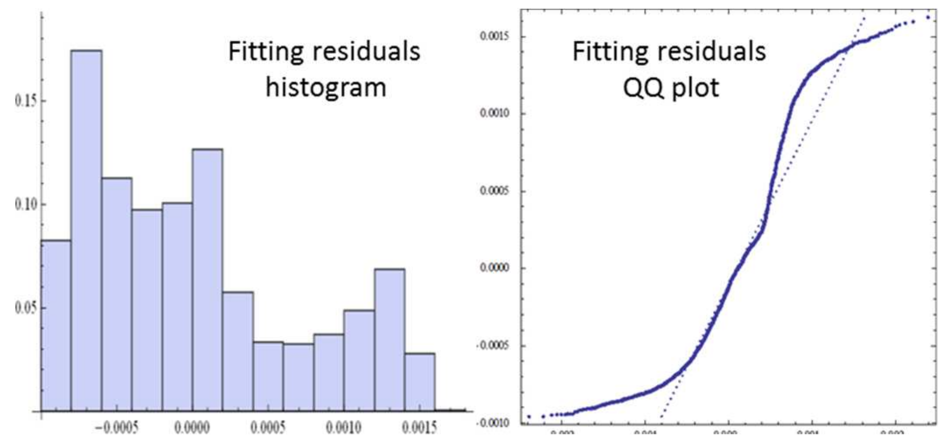
Digression : Fitting Residuals

Typical loss angle fitting residuals, TNI measure-ments



[Villar et al., PRD 81 (2010)]

Typical loss angle fitting residual, clamped cantilever based ring-down measurement



[data courtesy N. Morgado (2008)]

Confidence intervals must be robustly estimated in the non Gaussiann case.

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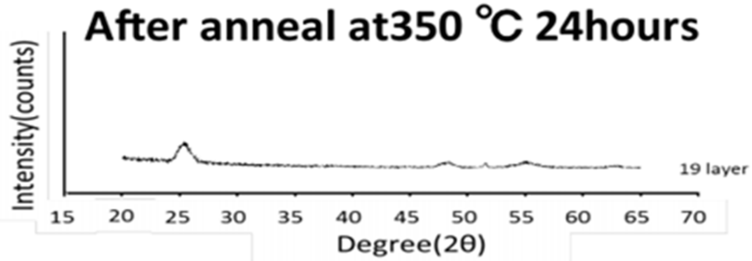
XRD Spectra after Annealing

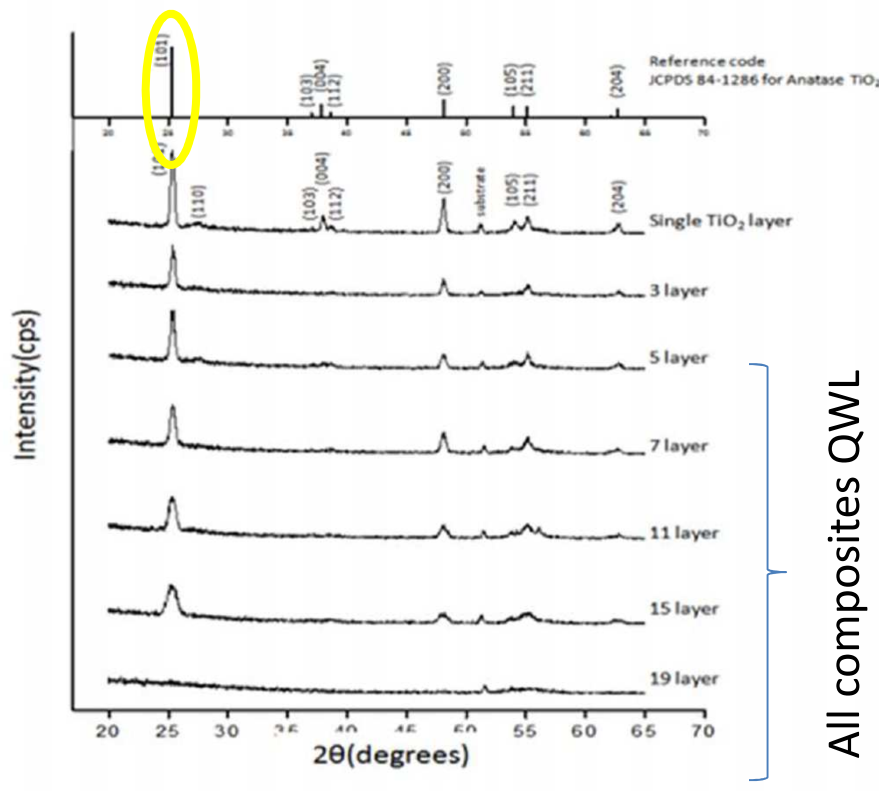
There is a threshold anneal temperature for the onset of crystallization (Anatase). The threshold temperature increases with the number of layers, or equivalently, with decreasing the (Titania) layer thickness.

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

After anneal at350 °C 24hours



After anneal at 300°C 24hours



All composites QWL with *n* = 2.065

[S. Chao et al., LIGO-P1400122; Optics Express (2014) in print]

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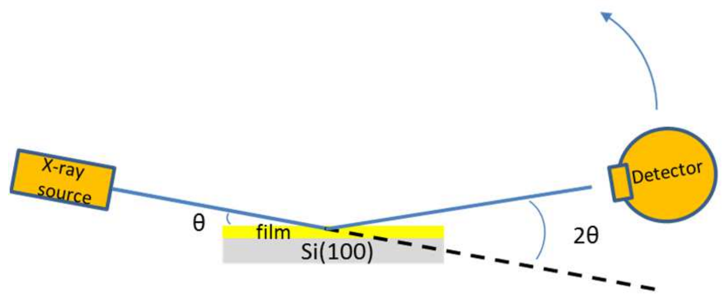
NTHU XRD Facility

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

Incidence angle(θ) : 0.5 °
Scan range (2θ): 20 ° ~65 °
Scan step size : 0.02 °
Time per step : 0.5s



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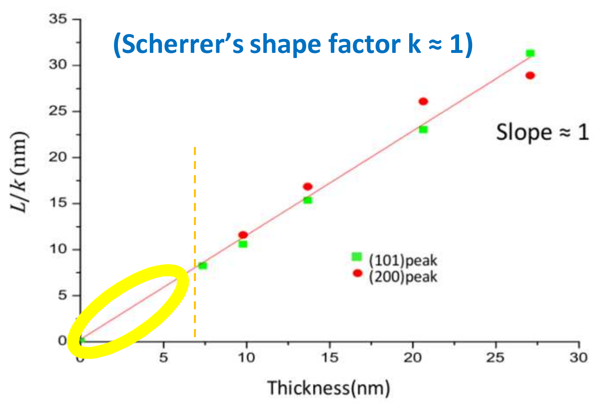
Crystallite Size vs Nanolayer Thickness

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)			
		2θ	β_{exp} (degree)	$\beta_s\cos\theta$ (degree)	L/k (nm)	2θ	β_{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 ≈ 7nm (and hopefully below)

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

→ Sankur-Gunning results confirmed

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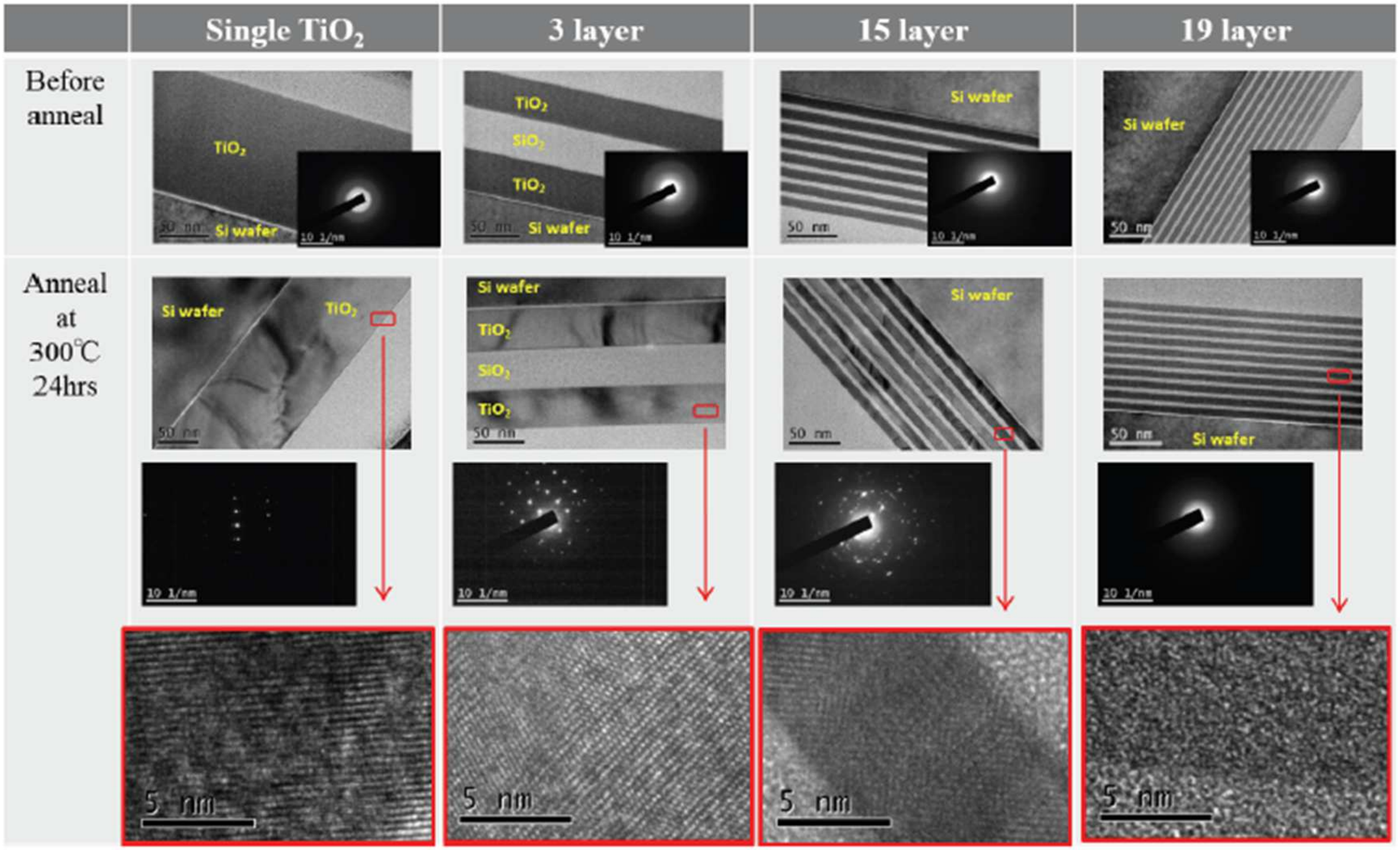
TEM Imagery Before/After Annealing



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

TEM shows that no significant across-interface diffusion occurs during annealing

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Crystallization After Annealing

Anneal Condition / Crystallization / Sample	Before annealing	225 °C 24hr	250 °C 24hr	300 °C 24hr	350 °C 24hr
Single TiO_2	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 °

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AFM Imagery

AFM scans of top (TiO_2) layer surface

	Before annealing	225°C 24hr	250°C 24hr	300°C 24hr	350°C 24hr
Single TiO_2	Roughness: 0.24 ±0.07 nm	Roughness: 0.35 ±0.04 nm	Roughness: 1.62 ±0.42 nm	Roughness: 3.20 ±0.50 nm	- -
3 layer	Roughness: 0.22 ±0.54 nm	Roughness: 0.48 ±0.61 nm	Roughness: 0.62 ±0.06 nm	Roughness: 0.39 ±0.06 nm	- -
5 layer	Roughness: 0.17 ±0.03 nm	Roughness: 0.34 ±0.06 nm	Roughness: 0.39 ±0.05 nm	Roughness: 0.32 ±0.03 nm	- -
7 layer	Roughness: 0.25 ±0.07 nm	Roughness: 0.17 ±0.05 nm	Roughness: 0.26 ±0.06 nm	Roughness: 0.26 ±0.03 nm	- -
11 layer	Roughness: 0.15 ±0.03 nm	Roughness: 0.12 ±0.02 nm	Roughness: 0.35 ±0.02 nm	Roughness: 0.26 ±0.03 nm	- -
15 layer	Roughness: 0.26 ±0.07 nm	Roughness: 0.51 ±0.02 nm	Roughness: 0.25 ±0.05 nm	Roughness: 0.30 ±0.02 nm	- -
19 layer	Roughness: 0.17 ±0.01 nm	Roughness: 0.28 ±0.05 nm	Roughness: 0.21 ±0.02 nm	Roughness: 0.32 ±0.07 nm	Roughness: 0.29 ±0.10 nm

Amorphous
Crystallized

[S. Chao, LIGO-M1300282]

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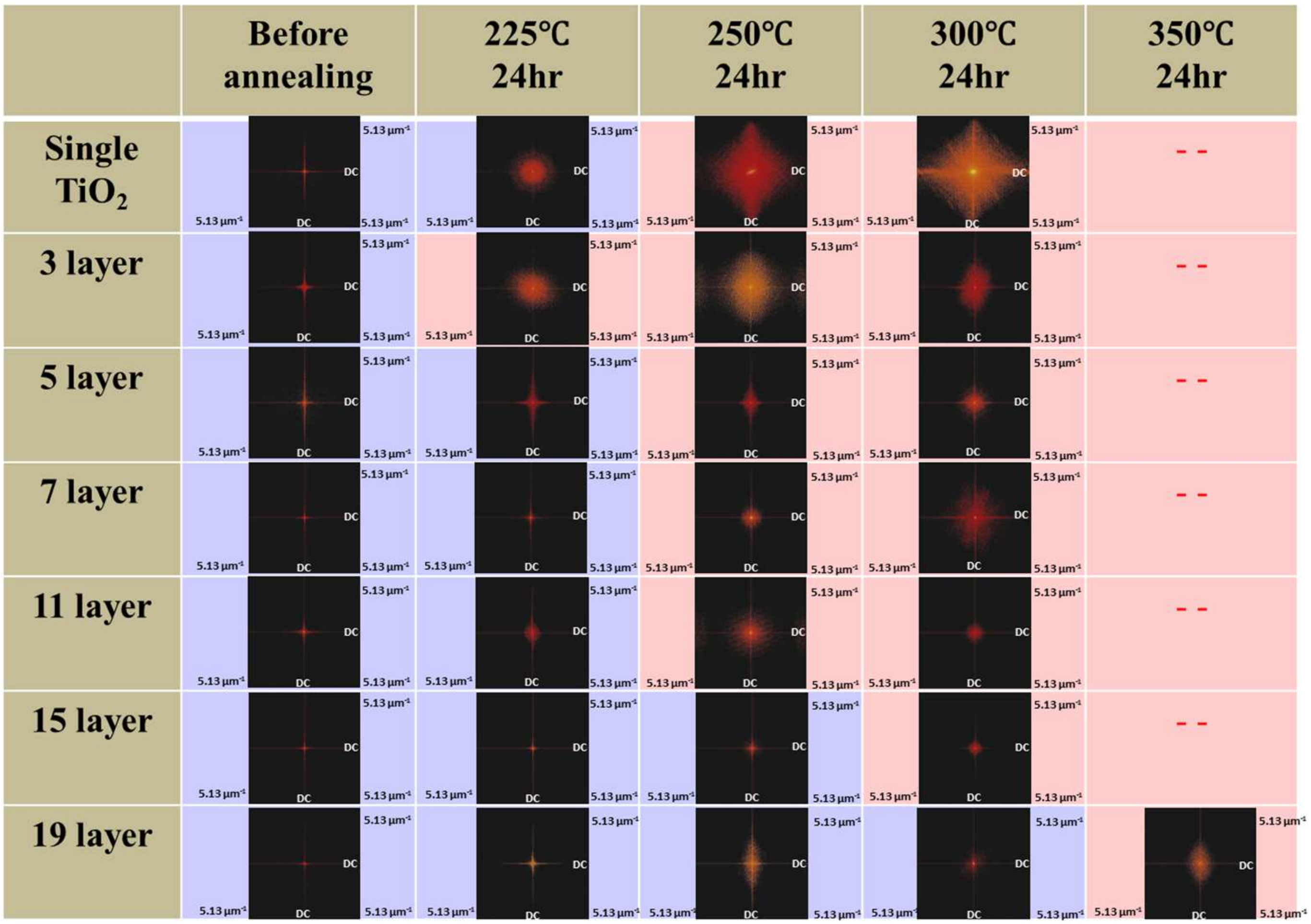
AFM Spectra

Spatial spectra of AFM scans. Tile sidelength is (5.14 μ)$^{-1}$



Amorphous
Crystallized

[S. Chao, LIGO-M1300282]

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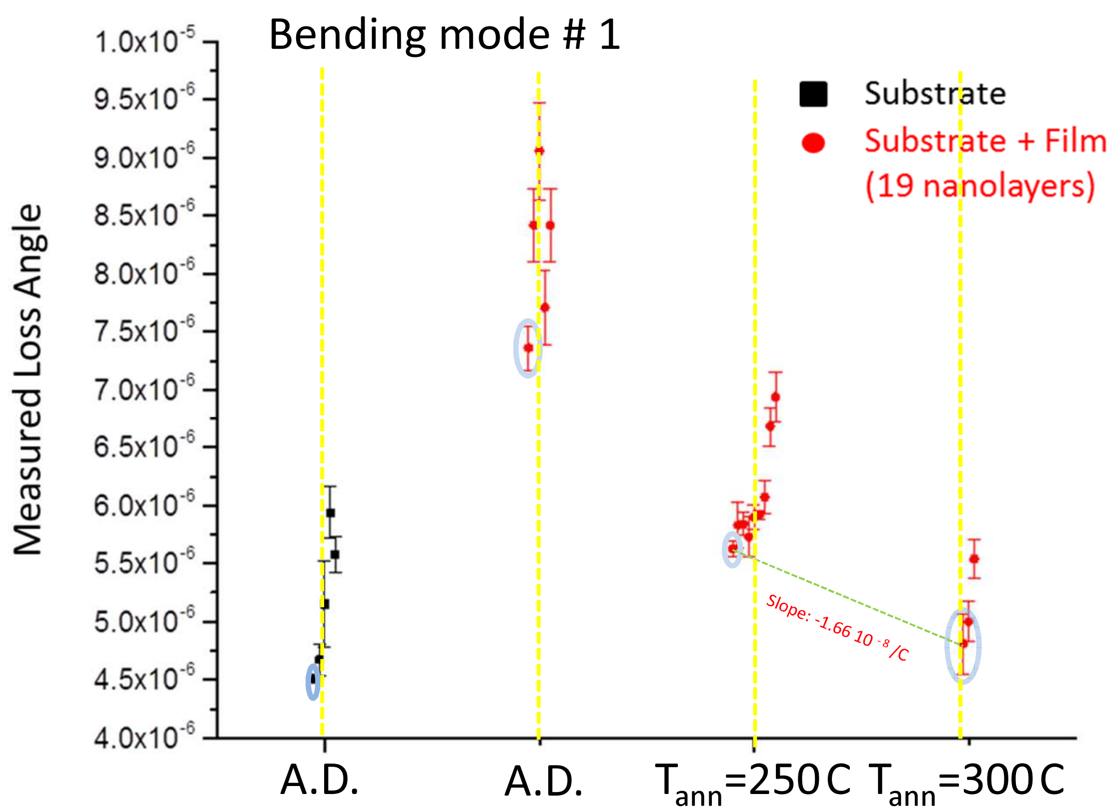
Loss Angle Before/After Annealing



Different bars correspond to different re-clampings.

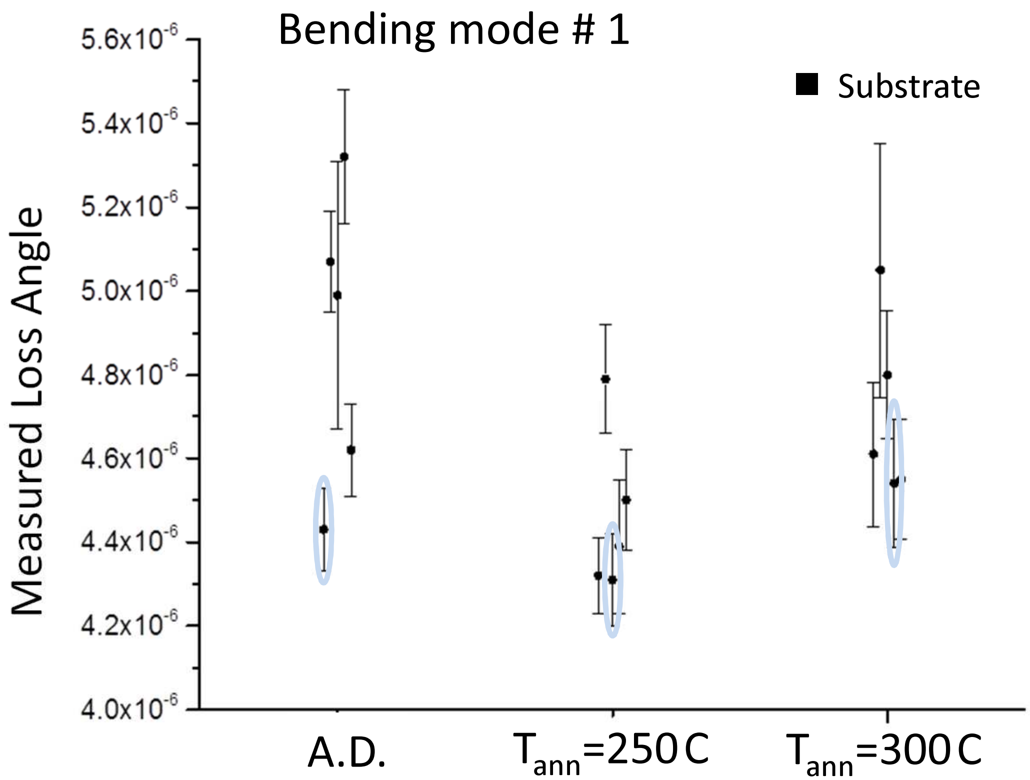
Lowest-average bar yields most trustable value.

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

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

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

Loss Angle Before/After Annealing



Uncoated substrate loss angle shows no significant change after annealing.

	Loss Angle	
T_{ann}	μ	σ/μ
A.D.	$4.48\ 10^{-6}$	0.073
250	$4.32\ 10^{-6}$	0.044
300	$4.55\ 10^{-6}$	0.046
substrate only (Silicon)		

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

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Loss Angle of nm-Layered Material

Young modulus of substrate (169 GPa, Silicon 100)

thickness of substrate (92$\pm$1 μ)

$$\phi_{nlc} = \frac{Y_s h_s}{3Y_{nlc} h_{nlc}} (\phi_{coated} - \phi_{naked})$$

measured loss angle of naked cantilever (substrate)

measured loss angle of coated cantilever

thickness of nanolayered composite (112.5 nm, for the 19-layer prototypes)

Young modulus (Voigt) of nanolayered composite

$$Y_{nlc} = z_1 Y_1 + (1 - z_1) Y_2$$

Volume (thickness) fractions of composite ingredients (0.654 for TiO_2, for the 19-layers prototypes)

Young moduli of composite ingredients (165Gpa for TiO_2, 72 Gpa for SiO_2)

[Pierro et al., LIGO-T060173]

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Loss Angle of *a*-Titania in nano-Composite

… Use fiducial value of Silica loss angle (5 10^{-5}) to retrieve loss angle of *a*-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 *a*-TiO_2 , consistent with **[Scott and MacCrone, Rev. Sci. Instr. 39 (1968) 821].**

Loss Angle Before/After Annealing

…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)

$\phi = 1.04\ 10^{-3}$ (as deposited)
$\phi = 4.3\ 10^{-4}$ (after annealing 24h @ 250 C)
$\phi = 1.3\ 10^{-4}$ (after annealing 24h @ 300 C)

Preliminary

…comparable to or better than Ti-doped Tantala !

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

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

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Outlook

Cold Mirrors aren’t Cool

Cryo Friendly Glassy Oxides

Doping vs Thinning

nm-Layered Composite Design

Good Old Technologies we Can Trust in

Experiments: What we Learned Sofar

What’s Next - The INFN AdCOAT project

Conclusions

Next Steps

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

Practical thickness limit $\approx 2nm$, may allow $T_{ann} \approx 400\ C$;

Better (higher Q) substrates may be needed (Norcada's ?);

Available evidence suggests that (many) interfaces are irrelevant as far as mechanical losses are concerned. **But may be relevant for optical diffusion.**

Need to characterize optical scattering of nanolayered prototypes;

This task is part of the INFN-AdCOAT proj. **[Pinto et al., LIGO-G1400810].**

Investigate behaviour of nm-layered composites at cryogenic temperatures

Cryogenic ringdown measurement facility at NTHU almost completed;

This task is also part of the INFN adCOAT project.

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The INFN AdCOAT Project (2014-15)

Missions:

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

- "Setting up a *coherent interaction* between different Italian Groups working on diverse aspects of coating science and technology, with Specific reference to Interferometric Detectors of gravitational waves."

AdCOAT – WG Background/Tasks

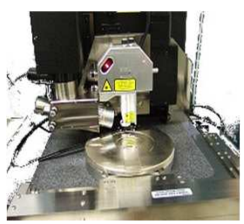
Genoa WG - Coating morphology analysis [Prato et al., J. Phys. Conf. Ser., 228 (2010) 012020]; optical properties characterization [Prato et Al., Thin Solid Films 519 (2011) 2877]

Perugia WG – Dissipation mechanism modeling in glasses **[Travasso et al., Materials Science Eng. A521 (2009) 268;** Euro Physics Lett. 80 (2007) 50008**]** ; viscoelastic parameters measurement techniques [P. Amico et al., J. Phys. Conf. Ser., 32 (2006) 413]

Rome "Tor Vergata" WG - Cryogenic subsystems [Coccia, Physica B 280 (2000) 52]; development of gentle nodal suspension setup [Cesarini et al., Rev. Sci. Instr. 80 (2009) 1.3124800; CQG 27 (2010) 084031].

Sannio WG - Coating design [Villar et al., Phys. Rev.D81 (2010) 122001], EMT modeling of glassy oxide mixtures [Pinto et al., LIGO-G1100372], nm-layered composite modeling and design [Pinto et al., LIGO-G 1100586].

AdCOAT – Experimental Facilities



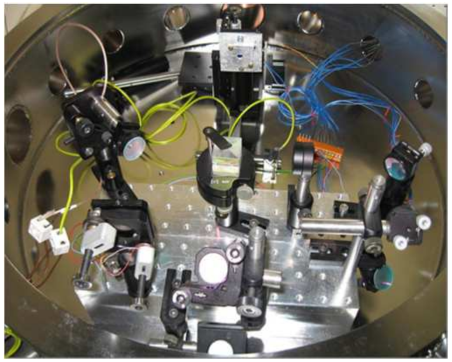
Genoa WG - HR-TEM (JEOL JEM 2010 + accessories); SEM (Zeiss EVO 40 HV + accessories); FE-SEM (Zeiss SUPRA 40 VP + accessories); SPM (Veeco Multimode Picoforce + accessories); AFM (Dimension 3000 +accessories); XRD (Philips XPERT MPD PRO); 2 x OSEs (Woollam M-2000 and VASE); some cryogenic facilities.

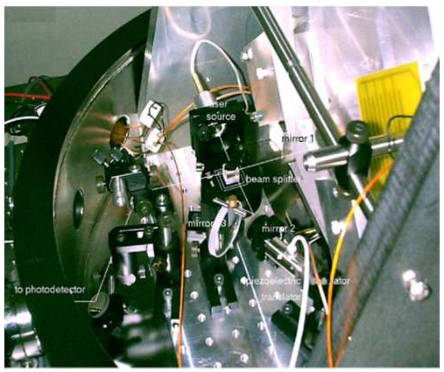
(courtesy M. Canepa, INFN Ge)

AdCOAT – Experimental Facilities, contd.

Perugia WG - Two cryostats (resp. nitrogen/he-lium, and pulse-tube). Three different setups for mechanical Q measurement at ambient tempe-rature; optical lever based setup; frequency sta-bilized Michelson interferometer. FE - SEM for film surface quality analysis down to 2 nm.

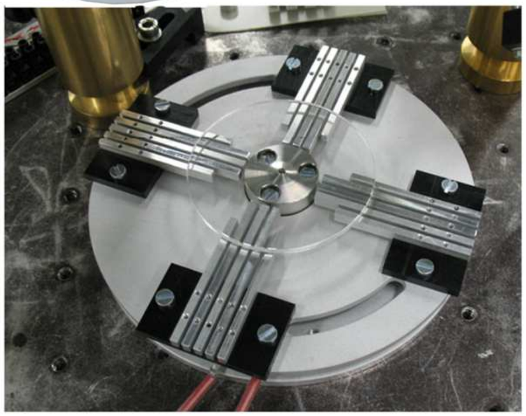
3 optical systems (Michel-son IFO, FP cavity and sha-dow-meter) with stabilized Laser for measuring mech-anical modes of membra-nes and mirrors, at ambi-ent and cryogenic tempe-ratures.





(courtesy H. Vocca, INFN Pg)

AdCOAT – Experimental Facilities, contd.



- Laser assisted centering
- Q independent of suspension point
- coating preserved
- butterfly-mode matched exciters
- lowest loss angle measured so far (fused Silica) : ϕ=4.8 10^{-8}

Rome WG - Gentle nodal suspension (GeNS) based setups for Q measurement at ambient and (soon) cryogenic temperatures.
Some systems for thermal and laser annealing (CO$_2$ laser)

- l-He cooling and radiation shield
- optical lever readout
- alternative (capacitive) readout
- electrostatic (comb) actuators
- cryogenic positioners

(courtesy E. Cesarini, INFN Rm-TV)

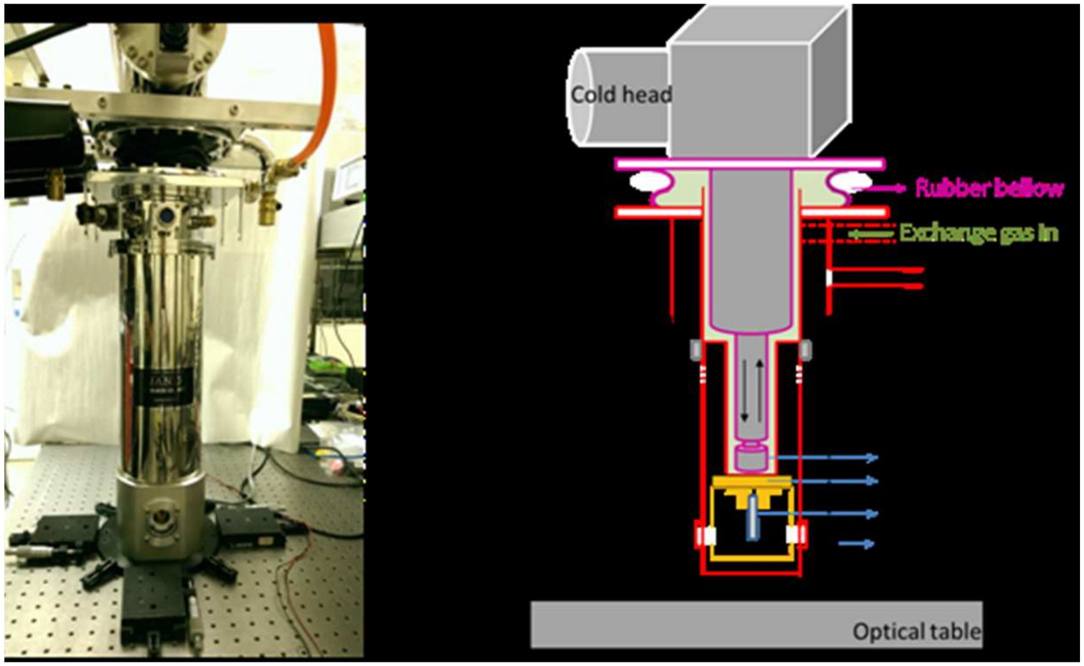
AdCOAT – Experimental Facilities, contd.

Sannio WG – Dual head pulse tube cryocooler (Sumitomo SRP - 052A - W71 D) . Two NVIDIA "Fermi" C2070 GPU based WS.

In house developed SW for coating simulation (thickness optimization, mixture analysis and design, nm-layered composites analysis and design, statistical treatment of measurement residuals and robust estimators).

(courtesy M. Principe, UniSannio and INFN)

NTHU Cantilever Setup Upgrade (2014-15)



[S. Chao, LIGO-G1400806]

ET EINSTEIN TELESCOPE

LMA LABORATOIRE MATÉRIAUX AVANCÉS

Outlook

Cold Mirrors aren't Cool

Cryo Friendly Glassy Oxides

Doping vs Thinning

nm-Layered Composite Design

Good Old Technologies we Can Trust in

Experiments: What we Learned Sofar

What's Next - The INFN AdCOAT project

Conclusions

EINSTEIN TELESCOPE

LABORATOIRE MATÉRIAUX AVANCÉS

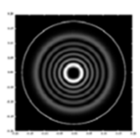
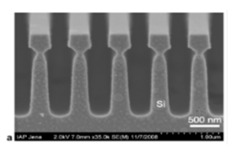
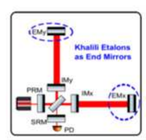
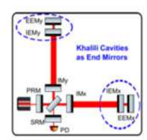
Conclusions

keyword	year	selected reference(s)	Status
Thickness Optimization	2005	LIGO G050176 (2005) ... PRD 81 (2010) 122001	*AdLIGO baseline design*
Doped Tantala	2007	Class. Quantum Grav. 24 (2007) 405 ...	*AdLIGO baseline design*
Cryogenic Coatings	1999	Int. J. Mod. Phys. D8 (1999) 557 ... Opt. Lett. 38(2013) 5268	R&D
"Wide" (or HOGL) beams	2003	PRD 67 (2003) 102004 ... CQG 30 (2013) 035004	R&D
Khalili's Resonator and "Etalon"	2004	Phys. Lett. A334 (2004) 67, Phys. Lett. A375 (2011) 4147	R&D
Coating-less Mirrors	2004	Phys Lett A324 (2004) 345,..., PRD 76 (2007) 053810	R&D
Diffractive "Mirrors"	2006	CQG 23 (2006) 7297 ... LIGO P-1300034 (2013)	R&D
Crystalline Coatings	2012	LIGO G1200948 (2012) ... Nature Phot. 7 (2013) 644	R&D

So far, only doped Tantala and thickness -optimization progressed to the pro-duction stage (and became part of the AdLIGO baseline design).

Most of the above (clever) ideas are still facing *major technological challenges* .

Will nm-layered composites be a viable route ?


Acknowledgements

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