

i-CoRe **(innovative Coating Research)**

Innocenzo M. Pinto



Università
degli Studi
del Sannio



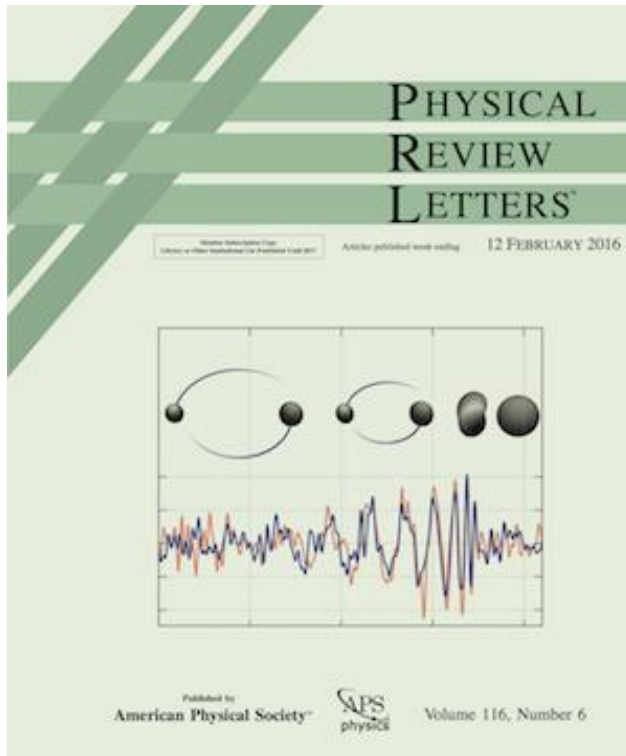
Istituto Nazionale
di Fisica Nucleare



MUSEO
STORICO DELLA FISICA
E
CENTRO
STUDI E RICERCHE
ENRICO FERMI

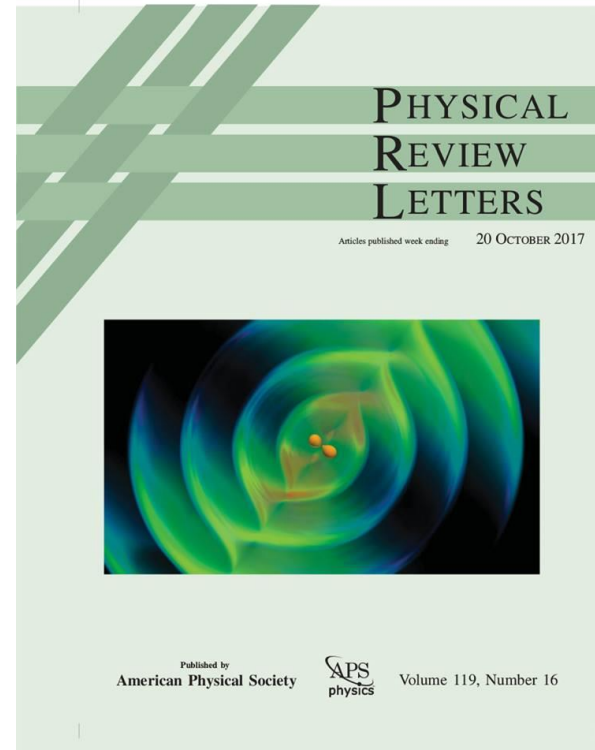
New Eyes / Ears !

GW150914 (2015)



birth of Gravitational Wave
Astronomy

GW170817 (2017)

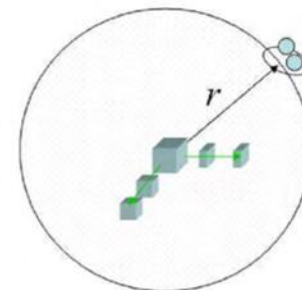
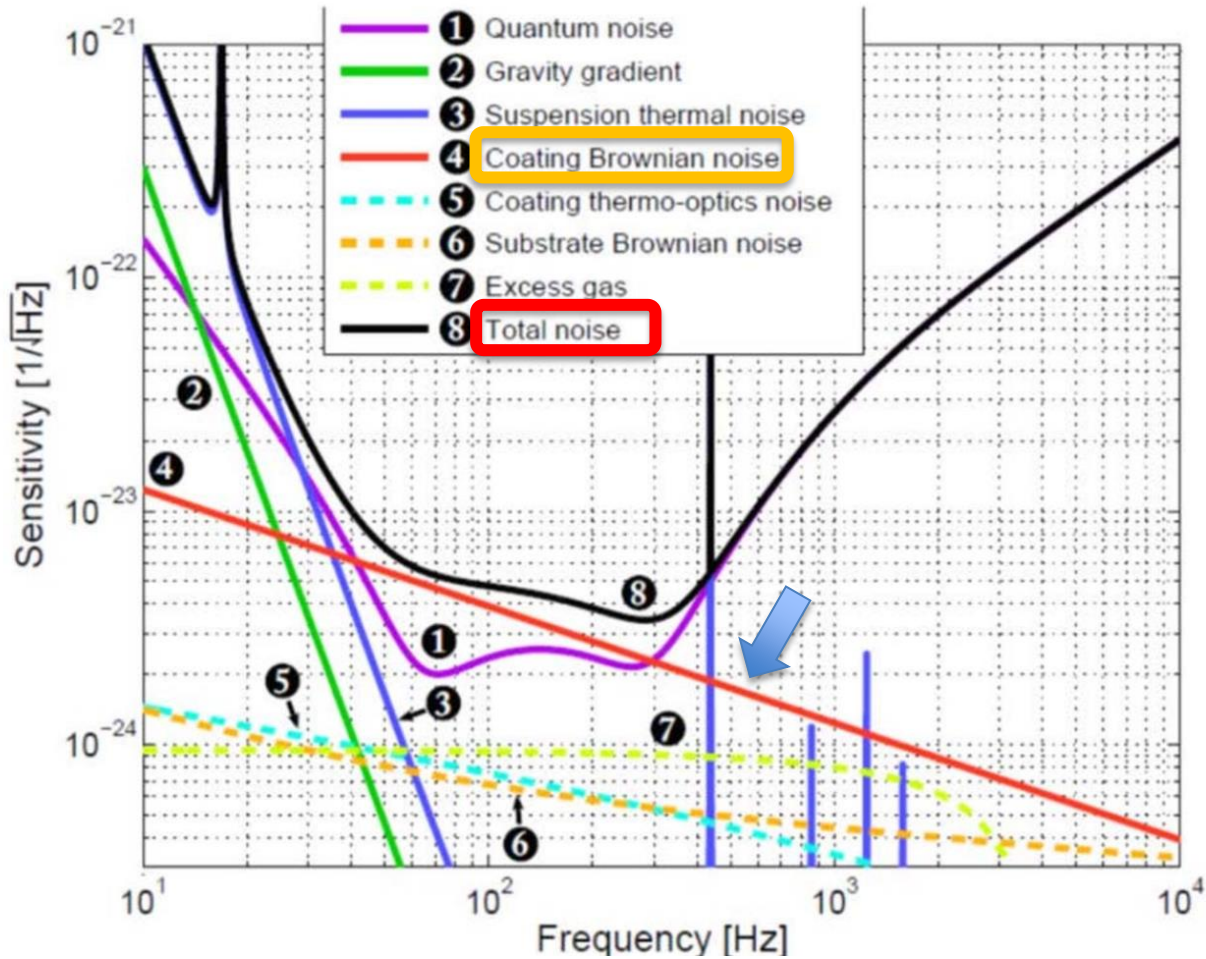


birth of multi-messenger
Astronomy

Visibility distance (aLIGO, 2017) : 130Mpc for BNS coalescence

i-CoRe – Science Case

Advanced Virgo Noise Budget



Visibility volume & event rate } $\propto PSD_{floor}^{-3/2}$

... a 5μ thick film sets the sensitivity of a 5 Km scale instrument ! ...

Coating (Brownian) Noise

HR coatings consist of cascaded doublets of high/low index material. Each doublet is $\lambda/2$ thick (Bragg) ; the *total number of doublets depends on the prescribed transmittance and the optical contrast...*

Temperature \rightarrow

$$S_{coat}^{(B)}(f) = \frac{2k_B T}{\sqrt{\pi^3} f} \frac{1 - \sigma^2}{w_m Y} \phi_c$$

Coating loss angle (mechanical, F/D theorem) \rightarrow

Beam spot-size \rightarrow

Act on the thicknesses \rightarrow

Act on the materials \rightarrow

$$\phi_c = \frac{\lambda_0}{w\sqrt{\pi}} (\eta_L d_L + \eta_H d_H), \quad \eta_{L,H} = \frac{\phi_{L,H}}{n_{L,H}} \left(\frac{Y_{L,H}}{Y_s} + \frac{Y_s}{Y_{L,H}} \right)$$

total (H,L)-index material thickness, in units of local wavelength

L,H material noisyness per unit thickness



2nd Generation Coatings (aLIGO, AdvVirgo 2018)

Material downselection

SiO₂ and Ta₂O₅::Ti were chosen as coating materials after extensive trial and error experiments ...

500nm thick films

	Refraction index	Absorption (ppm)	Mechanical losses
Ta ₂ O ₅	2.035	1.22	3·10 ⁻⁴
Ta ₂ O ₅ : Co	2.11	5000	11·10 ⁻⁴
Ta ₂ O ₅ : W	2.07	2.45	7,5·10 ⁻⁴
Ta ₂ O ₅ : W+Ti	2.06	1.65	3,3·10 ⁻⁴
→ Ta ₂ O ₅ : Ti	2.07	0.5	2,4·10 ⁻⁴

Coating	Refraction index	Absorption (ppm)	Mechanical losses
ZrO ₂	2.10	11	2,3·10 ⁻⁴
ZrO ₂ : Ti	2.15	37	6,8·10 ⁻⁴
ZrO ₂ : W	2.12	10	2,8·10 ⁻⁴

...

[Flaminio et al., CQG 27 (2010) 84030]

2nd Generation Coatings (aLIGO, AdvVirgo 2018)

Multilayer Optimization

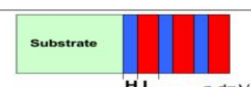
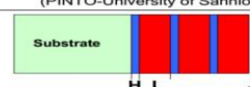
(I.M. Pinto, M. Principe, R. DeSalvo, Ch. 12 in, "Optical Coatings and Thermal Noise in Precision Measurements," Cambridge Un Press, 2012)

Coating designs for Advanced Detectors
 (Nazario MORGADO – LMA Lyon)

Thermal Noise Workshop – 23 february 2012

Optimized coatings : Gain for the Thermal Noise
 Innocenzo PINTO (University of SANNIO) [Optimized Coating (LSC 12-17 August 2005, LIGO Hanford Observatory LIGO G-050363-00-R)]

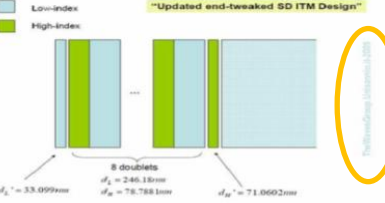
Goal : Modify the physical stack without changing the optical response

Mirror transmission : 278 ppm H:Ta ₂ O ₅ (n : 2.035-i.3.10 ⁻⁸ , Φ : 3.10 ⁻⁴) L:SiO ₂ (n:1.465-i.4.10 ⁻⁸ , Φ : 5.10 ⁻⁵)	
Coatings for the TNI QWL mirror  (HL) ₁₃ HLL	Lowest noise end tweaked stacked doublet (PINTO-University of Sannio)  0.56H(1.38 L0.62H) ₁₀ 0.16L
Ta ₂ O ₅ thickness : 1830 nm	Ta ₂ O ₅ thickness : 1347 nm
SiO ₂ thickness : 2722 nm	SiO ₂ thickness : 4032 nm
Relative PSD (Power Spectral Density) : 1	Relative PSD : 0.83

Thermal Noise Workshop – 23 february 2012

Optimized ITM mirror

Updated end-tweaked SD ITM Design



Goals :

- @ 1064 nm : Transmittance = 1.3 % - 1.5 %
- @ 532 nm : 0.5 % < Transmittance < 2 %
- Minimize the Electric Field

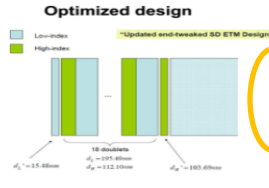
Always have in mind : ROBUST design (the less sensitive to manufacturing errors)

Thermal Noise Workshop – 23 february 2012

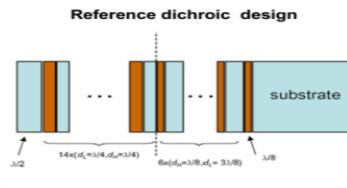
ETM mirror Designs

Optimized design

Updated end-tweaked SD ETM Design



Reference dichroic design



Goals :

- @ 1064 nm : Transmittance = 5 +/- 1 ppm
- @ 532 nm : 3 % < Transmittance < 15 % with goal 5% desired
- Minimize the Electric Field < 0.01 V/m

Always have in mind : ROBUST design (the less sensitive to manufacturing errors)

Thermal Noise Workshop – 23 february 2012

GOALS

- “Better” materials (2) : *high contrast, low optical absorption, low mechanical losses*
- Cryo – compatibility (→ Einstein Telescope; KAGRA)
- “Easy” technology, scalable

R&D LINES (see I. Pinto, LIGO-G1700171)

- **Microscopic/molecular modeling** (dangling bonds, TLS models - *UFL, Stanford, Glasgow*)
- **High-temperature deposition** (enhanced surface mobility, ultrastable glasses - *Stanford*)
- **Ion Plating** (*Glasgow, Jena*)
- **More glassy oxides and glassy oxide mixtures** (*H&WSC, LMA, CSIRO*)
- **Multimaterial (m-ary) coatings** (*Glasgow, MIT*)
- **Nanolayered composite materials** (*USannio, NTHU, UFL*)
- **Crystalline (GaAlAs, GaAlP) materials** (*CMS & LLC, Stanford, Glasgow, LMA*)
- **Silicon Nitrides** (*NTHU, H&WSC, USannio*)
- **Diffraction & Metamaterials** (*Mie, gratings - USannio, Braunschweig*)

FUNDING

US NSF (3M US\$, 3 years collaborative plan) - LIGO-LSC

GOALS

- “Better” materials (2) : *high contrast, low optical absorption, low mechanical losses*
- Cryo – compatibility (→ Einstein Telescope; KAGRA)
- “Easy” technology, scalable to meter-size mirrors

RESEARCH LINES (as of 2017-2018 – see I. Pinto, LIGO-G1700171)

- Microscopic/molecular modeling (dangling bonds, TLS models - *UFL, Stanford, Glasgow*)
- High-temperature deposition (enhanced surface mobility, ultrastable glasses - *Stanford*)
- Ion Plating (*Glasgow, Jena*)
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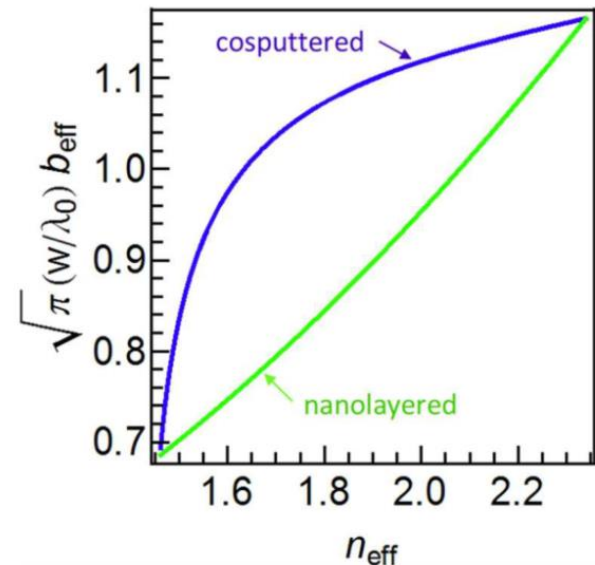
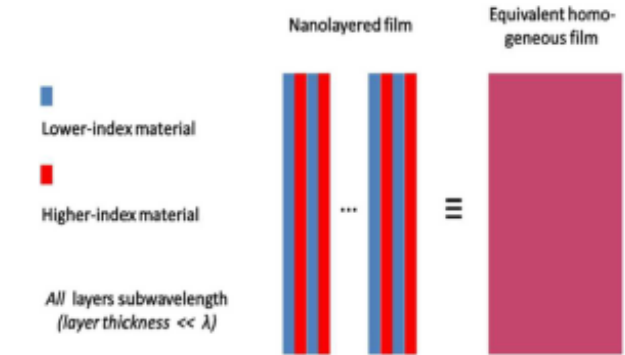
US NSF (3M US\$, 3 years collaborative plan) - LIGO-LSC

nm-Layered Composites

- Thin layers of a glass-former (e.g. Silica) alternated to a crystallization-prone material (e.g., Titania) hinder crystallite formation during the annealing phase, that spoils mechanical and optical losses

[Sankur et al., J Appl Phys 66, 4747 (1989);
 Pan, Principe et al., Opt. Expr. 22, 29857 (2014)]

- Amenable to simple modeling (*effective medium theory*) ➡ easily engineerable;
 [Principe, Opt; Expr. 23, 10938 (2015)]
- Less noisy than amorphous mixtures w. the same (effective) refractive index
 [Principe, Opt. Expr. 23, 10938 (2015)]



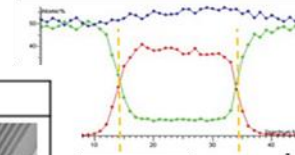
Hindering Crystallization

All nanolayered composite prototype films :
 $n_{eff} = 2.065$, QWL thick @1064nm

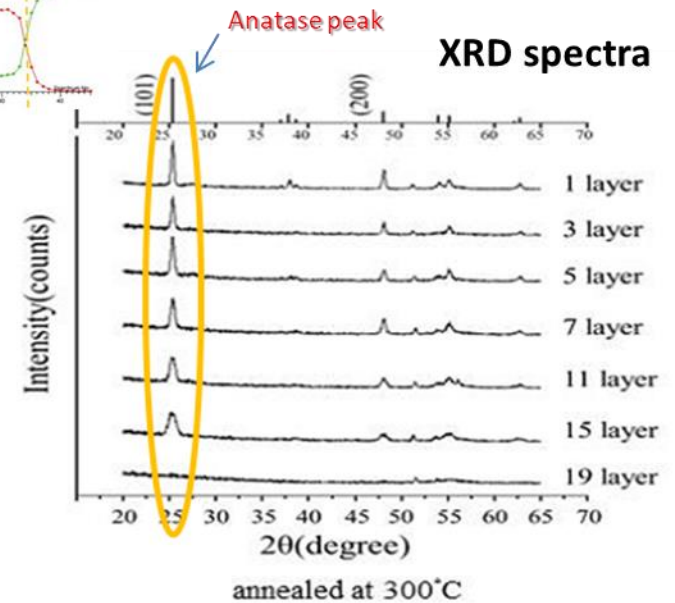
TEM & electron diffraction

	Single TiO ₂	3-layer	15-layer	19-layer
Before anneal				
Anneal at 300°C 24hrs				

EDXRD investigation of interfacial diffusion before/after annealing (negligible)



(S. Chao, I. Pinto et al., Opt. Expr. 22 (2014) 29847)



- **Glass-former (silica, alumina) nanolayers inhibit crystallite growth upon annealing;**
- **Allows using cryofriendly materials, like Titania or Hafnia, that otherwise crystallize;**
- **The nanolayered composites exhibit very low mechanical losses, and no cryopeak**

i-CoRe - People

Coordinator: *Innocenzo M Pinto (CF associate, LVC PI)*

OSA Fellow (2017) “For fundamental contribution to the thermal noise reduction in the mirror coatings of the LIGO interferometric gravitational wave detectors and for diverse contributions to the science of Electromagnetics.”

Participants (CF related):

Elisabetta Cesarini (CF postdoc fellow 2017)

Maria Principe (CF associate - postdoc at UniSA; former Fulbright Scholar)

Places of Work & Collaborations:

- Virgo** {
- University of Sannio (*Coating Deposition Laboratory, M. Principe, I. Pinto, J. Neilson*)
 - University of Salerno (*AFM, SFM, TEM, STM, XRD – F. Bobba, C. DiGiorgio, R. Fittipaldi*)
 - University of Rome “Tor Vergata” (*GeNS System – M. Lorenzini, E. Cesarini, D. Lumaca*)
 - University of Genoa (*Ellipsometric characterization – M. Canepa*)
 - University of Perugia (*cryo-GeNS ‘H. Vocca, F. Travasso*)
 - LMA – CNRS [Lyon, FR] (*IBS deposition and testing; glassy oxide mixture; film metrology*)
 - CNR IMM [Na-Le] (*Ellipsometry; MM; SiN_xH_y PECVD – F. Quaranta, E. De Tommasi*)
 - National Tsing-Hua University (NTHU) Optics and Photonics Lab [Taiwan]
(IBS nanofilms; PECVD - SiN_xH_y; cryo characterization - S. Chao)

i-CoRe Activity 2016-2017

- **New coating prototyping facility (IAD) set up at USannio**
(OAC75-F custom configured; 700KEUR funding from “Regione Campania” - POR 2007-2013; delivered 2016; fully operational 2017)
- **New Salerno/Benevento Virgo group established (formerly in LIGO); collabs with other Virgo coating R&D groups fostered**
(MOU with Virgo approved ; INFN funding approved)
- **Work on mechanical (loss) characterization of nm-layered Silica/Titania films based on GeNS** *(Rome-TV ; published, Int. J. Chem. Mol. Eng. 10 (2016) 1)*
- **Work on optical (ellipsometric) characterization of nm-layered Silica/Titania films** *(Genoa; published, Opt. Materials 75 (2018) 94101)*

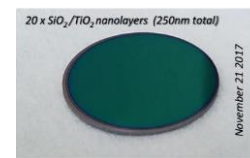
i-CORE Activity 2016-2017

New Coating Deposition facility @ USannio

(Optotech OAC75-F custom configured; 700KEUR funding from “Regione Campania” - POR 2007-2013; delivered 2016; fully operational 2017)



- High vacuum chamber (cryo + rotative pumps)
- 1 EB-gun with 6 pockets (a second source will be installed)
- Plasma source (IAD)
- Argon and Oxygen in chamber feeds
- Fully controllable from GUI
- Rotating substrate support to enhance uniformity
- Ceramic lamps to heat the substrate



(courtesy Maria Principe, Josh Neilson)

i-CoRe Activities 2016-2017

GeNS Facility @ Rome “Tor Vergata”

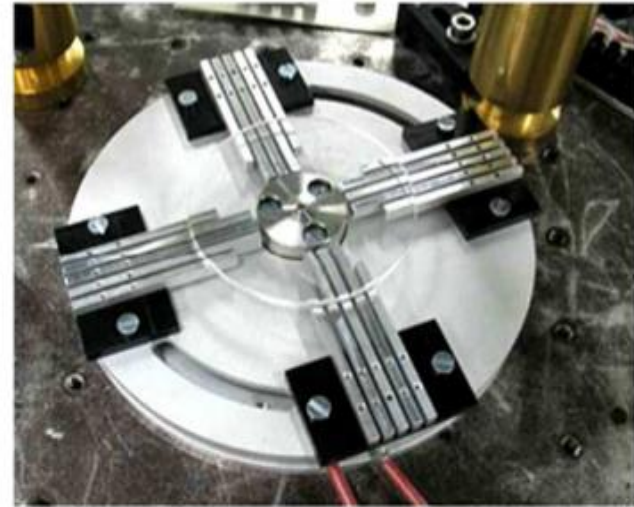
(courtesy Viviana Fafone, Elisabetta Cesarini)

The Gentle nodal Suspension (GeNS) is to date the most reliable mechanical loss estimation setup based on ringdown measurement.

Several improvements compared to clamped cantilever systems;

Designed to be exempt from re-clamping issues, yields nicely repeatable results.

Multimode operation should allow measurement of bulk/shear loss angles (TBD).



GeNS facilities (2018)

- LMA (G. Cagnoli)
- Rome-TV (E. Cesarini)
- Caltech (G. Vajente)
- Perugia (cryo, H. Vocca)

[Cesarini et al., Rev. Sci. Instrum. 80 (2009) 053904.]

i-CORE Activities 2016-2017

Loss angle Measurements using GeNS

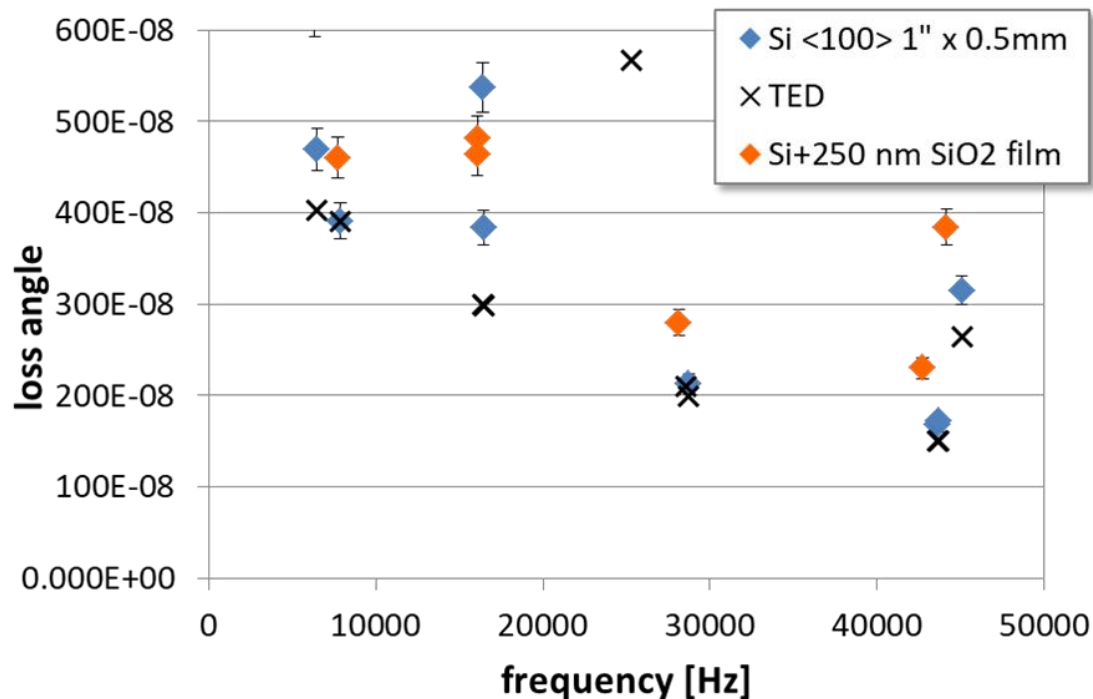
Film deposited @ USannio



Y_{Si}	Y_{SiO_2}
1,60E+10	7,3E+10
t_{Si}	t_{SiO_2}
5,00E-04	2,50E-07

$$\phi_{film} = \frac{Y_{Si} t_{Si}}{3Y_{SiO_2} t_{SiO_2}} \cdot \Delta\phi$$

(courtesy Elisabetta Cesarini, Daria Lumaca)



$$\phi_{film} = (1.02 \pm 0.10) \cdot 10^{-4}$$

(as deposited)

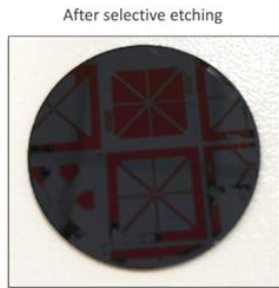
i-CORE Activities 2016-2017

Prototype Morphology @ UniSA

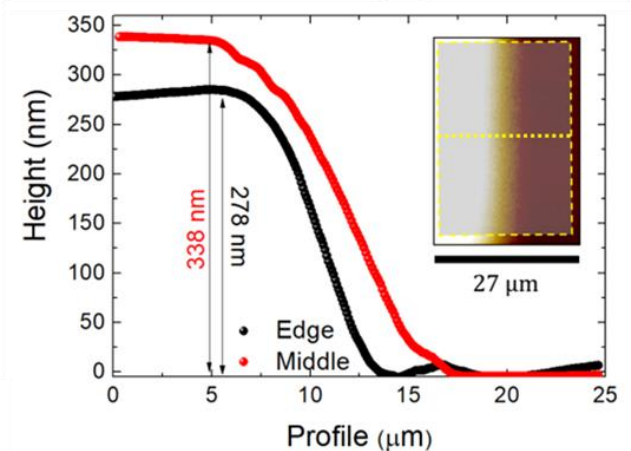
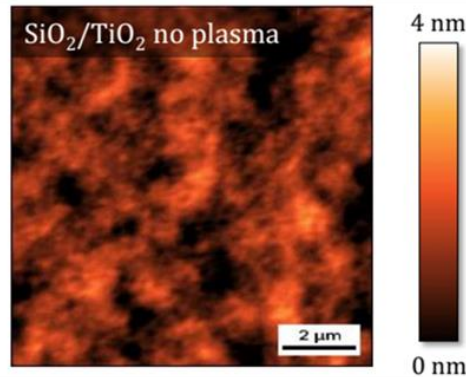
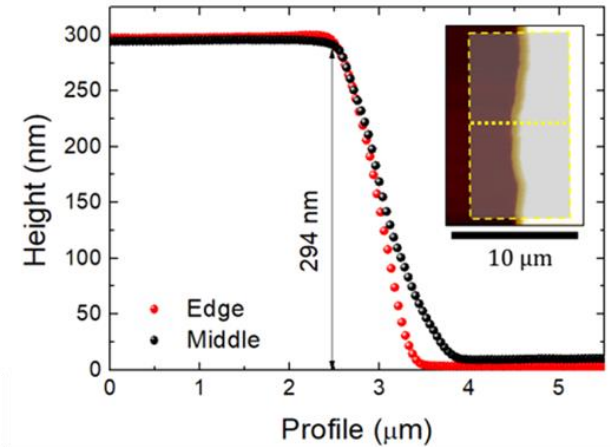
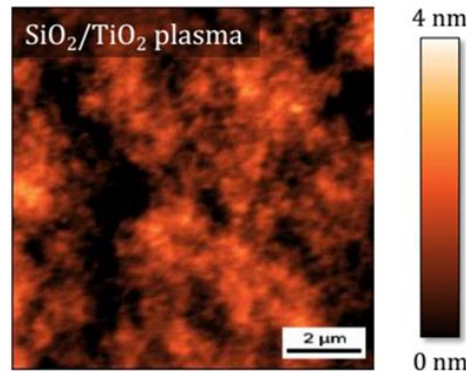
(courtesy Cinzia DiGiorgio, Giovanni Carapella)

USannio deposited
 nm-layered $\text{SiO}_2/\text{TiO}_2$
 Film (19nanolayers)

With IAD:



Without IAD:



i-CORE Activities 2016-2017

Creating a “distributed” Coating Lab in Italy

LIGO-G1701529



Toward an Integrated/Distributed Optical Coating Research Infrastructure in Italy

F. Baldaccini¹, F. Bobba², M. Canepa³, G. Castaldi⁴, E. Cesarini^{5,6}, D. D'Agostino², C. Di Giorgio², V. Fafone⁶, R. Fittipaldi², V. Galdi⁴, G. Gemme³, M. Lorenzini⁶, M. Magnozzi³, M. Moccia⁴, M. Principe⁴, V. Pierro⁴, I.M. Pinto⁴, A. Rocchi⁶, S. Terreni³, F. Travasso¹, H. Vocca¹

¹Dept of Physics, University of Perugia, IT, INFN, and LVC

²Dept. of Physics “E.R. Caianiello,” University of Salerno, IT, INFN and LVC

³Dept of Physics, University of Genoa, IT, INFN and LVC

⁴Dept. of Engineering, University of Sannio at Benevento, IT, INFN, LVC and KAGRA

⁵Centro Studi e Ricerche Enrico Fermi, Rome, IT, INFN and LVC

⁶University of Rome “Tor Vergata”, IT, INFN and LVC

Collaboration seeded in the frame of the AdCOAT project funded by INFN-CSN5 (2014-2015).

Coating Design/Deposition @UniSannio

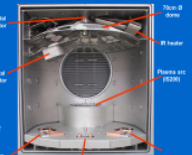
Group background: introduced/developed coating geometry optimization [PR D81 (2010) 122001] used in aLIGO & AdvVirgo; nm-layered glassy oxide composites [Opt. Expr. 22 (2014) 29847]; EMT - based mixture modelling [PR D91 (2015) 022005].

Facilities: coater

- Plasma assisted deposition
- Dual e-beam
- Multimaterial (up to 6)
- Fully programmable Q...
- nm accuracy
- 2 in. blank size
- IBS as a possible future option

Funded by Regione Campania, PON FESR grant.

- Coating material/geometry optimization
- Small-scale prototyping



Structural Characterization @ UniSA

Group background: researchers from the UNISA, SANM and MUSA Labs are leading experts in thin film characterization in the frame of international collaborations [Drexel Univ., Temple Univ., Argonne CNISM etc.], AIP Adv. 7 (2017) 055010; Sci. Rep. 6 (2016) 38557; PRB-89 (2014) 214505].

Facilities: SpMM; UHV room temperature and cryo AFMSTM (Omicron); AFM/AFS Multimode - Nanoscope - V w. conductive AFM and Kelvin Probe Force Microscopy; AFM/AFS Nanoscope - 3 w. Raman spectroscopy and

Inverted optical microscope; MUSA; TEM Techni TM G2; SEM Leo EVO 50 w. EDS system; FocSEM Sigma GEMINI; XPert MRD-PRO Diffractometer.

- In-situ surface & interface morphology and topography;
- Hourly modulus meas.
- Crystallization study via Wulff-Schramm-Hall method.



Spectroscopic Ellipsometry @ UniGE

Group background: microscopic/structural and optical characterization of Ta₂O₅ [Thin Solid Films 519 (2011) 2877] and TiO₂ [J.Phys. D47 (2014) 485301] films; ellipsometric characterization of nm-layered SiO₂/TiO₂ composites [LIGO-P170015, in print on Opt. Mat. (2017)].

Facilities: Woollam 2000 spectroscopic ellipsometer (190 - 1700 nm); VASE spectroscopic ellipsometer (190 -1100 nm); scanning monochromator; access to several structure/morphology characterization tools (HR-TEM, SEM

FE-SEM, AFMSTM, XRD and synchrotron light source @ ESRF).



- Measurement of optical film properties vs thickness and temperature;
- Optical uniformity and isotropy assessment;
- Study of film porosity.

Mechanical Losses @ UniRomeTV and Centro Ricerche “Enrico Fermi”

Group background: developed the Gentle Nodal Suspension (GeNS) concept [Rev. Sci. Instrum. 80 (2009) 053904; CQG 27 (2010) 084031]; characterized loss angle of several coating materials, including nm-layered SiO₂/TiO₂ composites [Int. J. Chem. Mol. Nud. Mat. 10 (2016) 597].

Facilities: mechanical loss measurement system based on (multimode) GeNS ringdown system.



- Best ringdown system
- Exempt from redamping problems
- Laser assisted centering
- Optical lever readout
- Butterfly-mode exciters
- Lowest measured loss (fused Silica) : $q=4.8 \cdot 10^{-4}$
- Cryogenic upgrade in prog.

Si₃N₄ Framed Films (T_{amb} & cryo) @ UniPG

Group background: theoretical and experimental work on mechanical loss mechanisms in amorphous glasses [Mat. Sci. Eng. A521 (2009) 268; Euro Phys. Lett. 80 (2007); J. Phys. Conf. Ser., 32 (2006) 413].

Facilities: Experimental (interferometric) setup for measuring mechanical loss angles and resonant frequencies in (NORCADATM) Si₃N₄ frame deposited films; N₂/He and pulse-tube cryostats; HR-FE-SEM.



- Si₃N₄ membranes have large Q (~10⁷) at relatively high frequencies (~10⁵ Hz). They are near - ideal substrates (dilution factors ~1) for precise measurement of very low mechanical losses in optical films;

Outlook

More Science Partners

LVC-OWG Groups; LMA, Lyon, FR; Braunschweig & Jena Univ. Groups; CSULA; NTHU, Taiwan, ROC.

- Goals: Small scale prototype testing of proposed ideas, e.g.:
- plasma-assisted deposition
 - hot-substrate deposition
 - nanolayered composites
 - new materials/mixtures

Missing Facilities (as of 2017)

- Optical loss measurement (→ LMA, Glasgow)
- Scattering measurement (→ CSULA)
- Direct thermal noise measurement (→ MIT)
- Thermo-optic noise measurement (→ Whitman)



Ligo-Virgo Collaboration Meeting (2017)

Milestones 2018

- Steady operation of our deposition and characterization chains
- Characterization of IAD nm-layered SiO_2/TiO_2 film prototypes
- Formulation of process/design optimization criteria

Plan of activities 2018 - 2020

Deposition (IAD) & Characterization of nm-layered SiO_2/TiO_2 composites
(morphological, optical, viscoelastic properties, down to $T=10K$)

Optimization of nm-layered composites *design*

(optimum thickness and number of nanolayers for fixed n and d/λ)

Optimization of *deposition & annealing parameters/schedule*

(IAD tuning; substrate temperature; annealing protocol and T_{max})

SiN_x development

(comparison with optimized doped [LMA] and nanolayered composites)

Preliminary study/prototyping of Mie Metalayers

New measurement facilities in Italy (CTN, PCI) ?

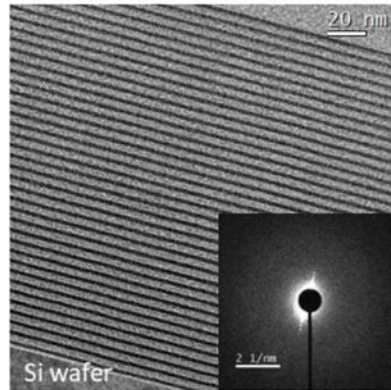
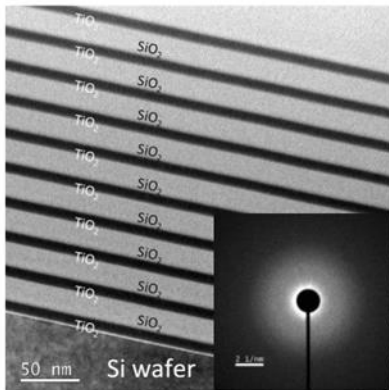
i-CoRe Activity Plan (2018-2020)

Preliminary (new) results

➔ 2nd generation nm-layered Silica/Titania composites (collab. w. NTHU)

(2014)
19-layers

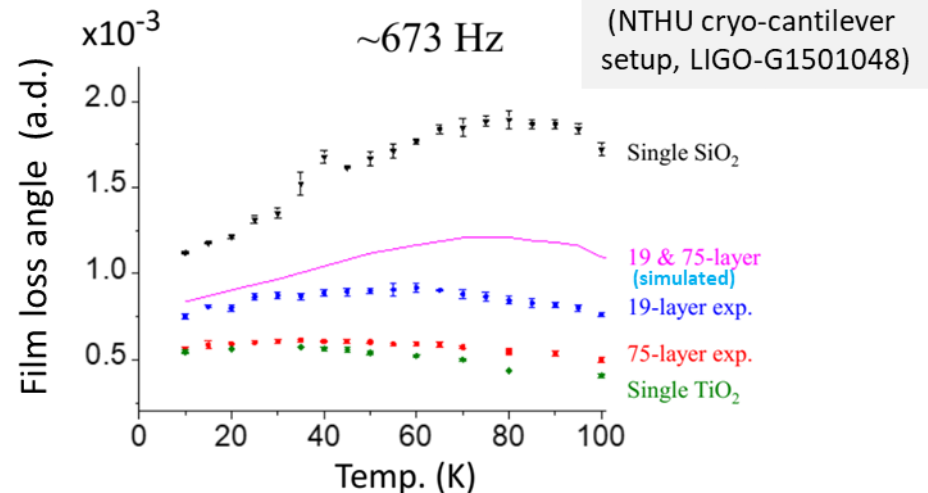
(2018)
75-layers



	thickness (nm)
SiO ₂	19.3 ± 0.1 (x9)
TiO ₂	8.3 ± 0.2 (x10)
$r = 0.32$	

	thickness (nm)
SiO ₂	3.6 ± 0.1 (x37)
TiO ₂	1.8 ± 0.1 (x38)
$r = 0.33$	

Both films QWL, with $n=2.16$



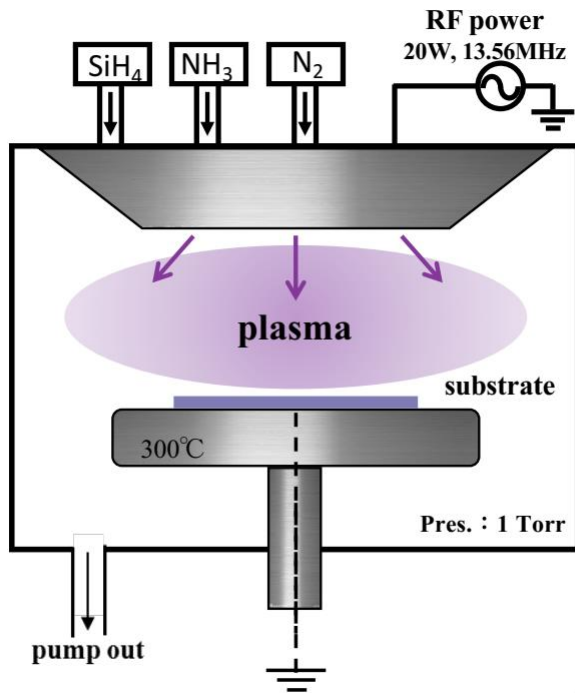
➔ ... as nanolayers become thinner and thinner:
 1. losses *decrease*;
 2. loss peak *shifts to lower temperature*

(courtesy S. Chao, feb. 2018)

i-CoRe Activity Plan (2018-2020)

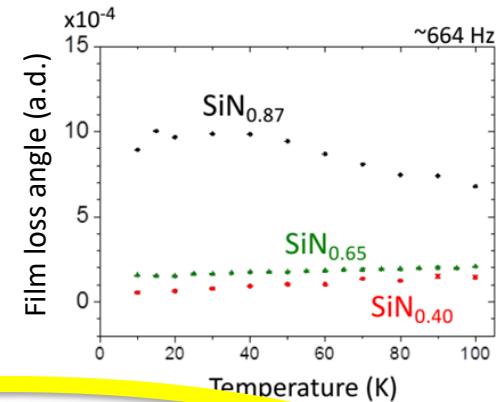
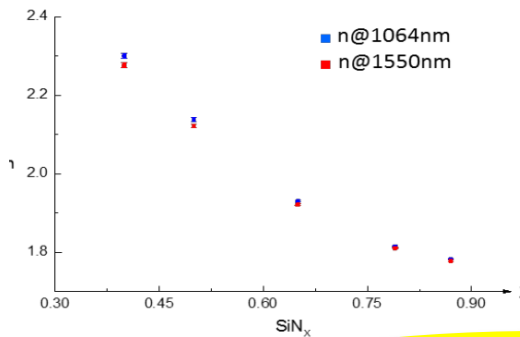
Preliminary (new) results

→ PECVD Silicon Nitrides (SiN_xH_y) films - collab. w. NTHU



Different reactant pressures yield *different stoichiometries*

Refractive index @ 1064 & 1550 nm



PECVD Silicon Nitrides:

- flexible stoichiometry -> index tunability
- mechanical losses on a par with SiO_2
- no cryo peak (for several x)
- scalability poses no problem

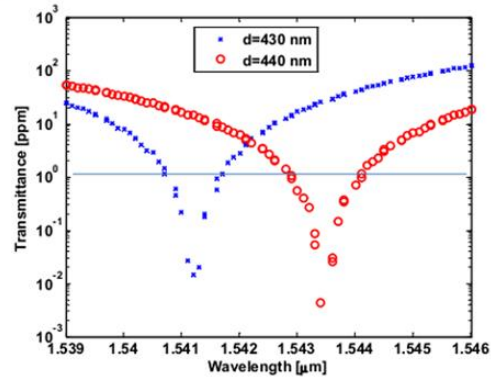
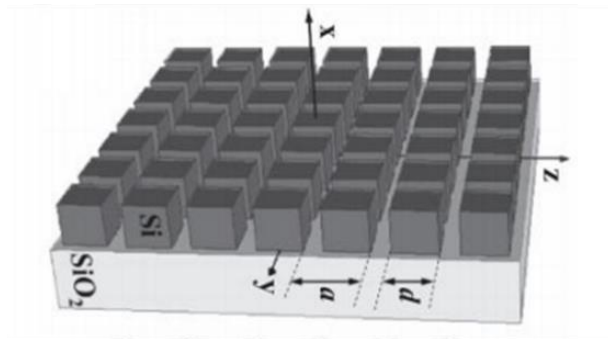
S. Chao et al., LIGO-G1701790

i-CoRe Activity Plan (2018-2020)

Preliminary (new) results

➔ Mie Metalayers (collab. w. Braunschweig Univ)

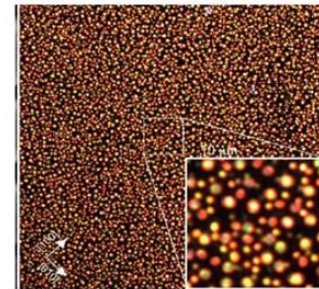
Very thin, monolayer, **fully scalable**



Trade reflectance (exceeding our needs) for bandwidth (insufficient for our needs).

Introduce randomness (or multiple periodicity) in the Mie inclusion pattern ...

Use colloidal self assembly ?



i-CoRe - Fundings

Expected fundings (2018-2020) :

- **Funding from Centro Fermi : 1 postdoc grant (renewal)**
- **External funding : INFN Virgo (2017 given; 2018-2020 expected)**
- **Potential external funding : MIUR-PRIN 2017 (South Line) € 1M**

**“Toward the Next Generation of Optical Coatings for Advanced Virgo:
Consolidating a Distributed Research Laboratory Network in South Italy”**

Participating Institutions: University of Sannio at Benevento
University of Salerno
CNR-IMM (Naples and Lecce)

**Partners: Virgo (Genoa, Rome-TV, Perugia)
LMA-CNRS (Lyon, FR)
Centro Fermi
University Braunschweig (GER)
NTHU (Taiwan, ROC)**