ICORE : **I**nnovative **CO**ating **RE**search

Innocenzo M. Pinto

University of Sannio, Centro Fermi, INFN, LVC and KAGRA

Rome, December 19, 2018

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Outlook

- Science case
- Research Goals & Trends
- Nanolayered Composites
    - Rationale
    - What/How
- iCoRe
    - Results 2018-19
    - Work planned for 2019-2020
- Funding requests

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Scientific Case

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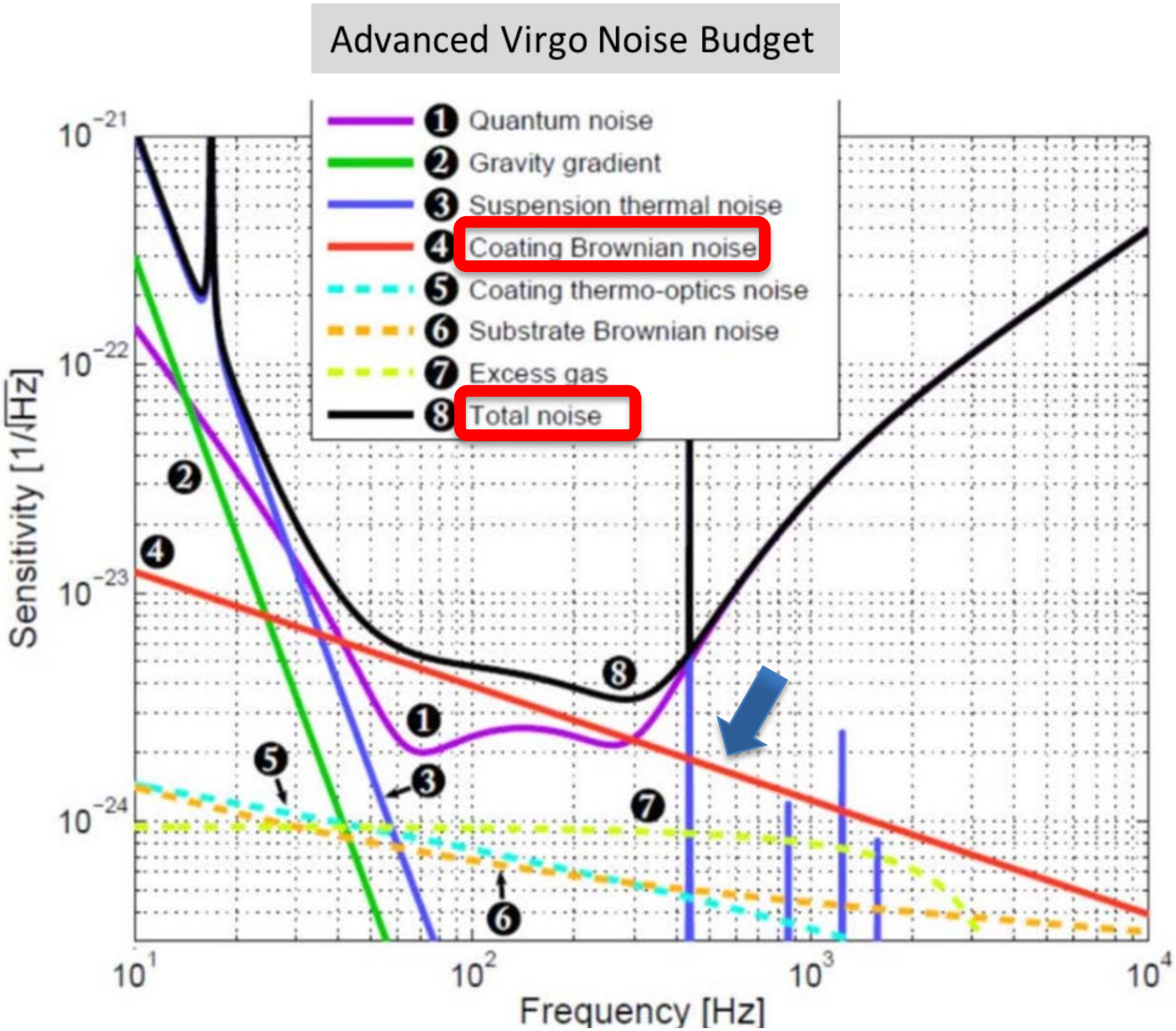

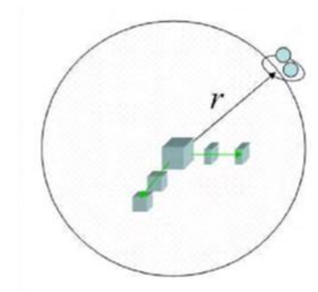

Visibility volume & event rate $\Big\} \propto PSD_{floor}^{-3/2}$

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

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Coating Noise PSD

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HR coatings consist of cascaded doublets of low/high index materials. Each doublet Is $\lambda/2$ thick (Bragg); the total number of doublets (and hence the total coating thick-Ness incrases for higher reflectance, and lower high/low index ratio (contrast)

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

Temperature

Coating loss angle (mechanical, F/D theorem)

Beam spot-size

Thickness optimization [Villar et al, PRD 81, 122001 (2010)]

Act on the thicknesses

Act on the materials

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

Ti-doped Tantala [Harry et al, CQG 24 (2006) 40]

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

L,H material noisyness per unit thickness

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Coating Design Optimization
(2005-2015)

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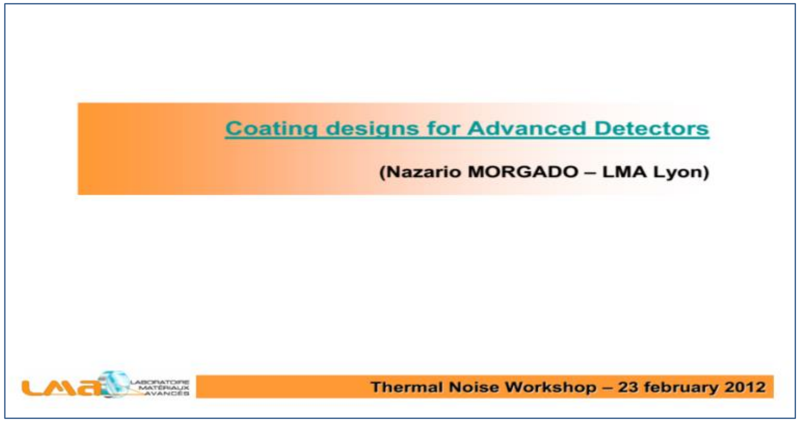

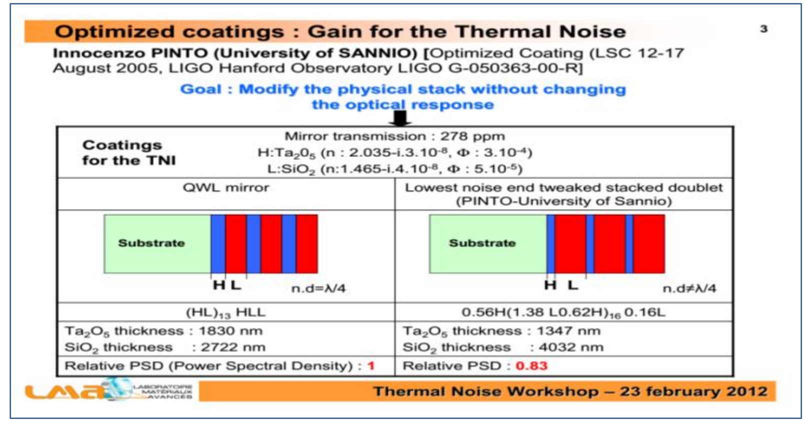

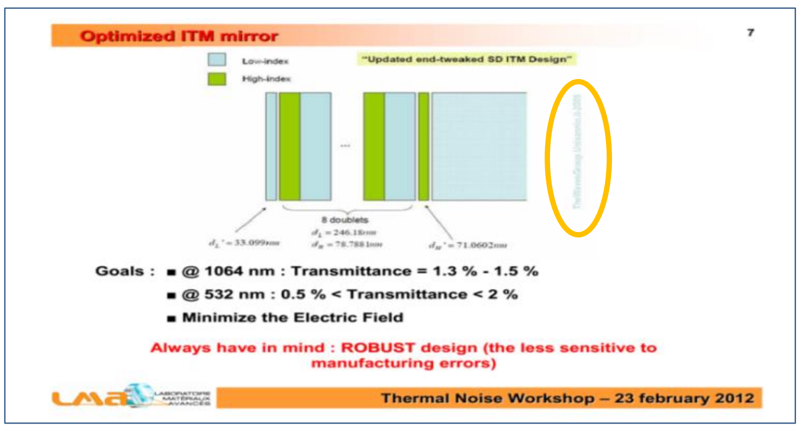

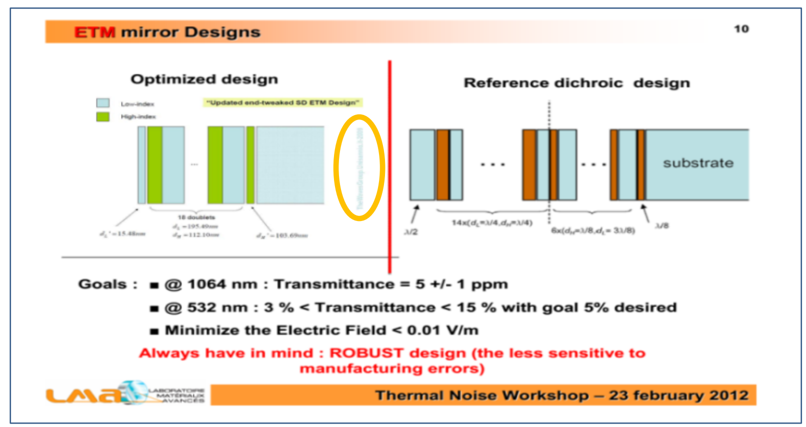

(I.M. Pinto, M. Principe, R. DeSalvo, Ch. 12 in, "*Optical Coatings and Thermal Noise in Precision Measurements*," Cambridge Univ Press, 2012; M. Principe, Opt. Expr. 23 (2015) 10938)

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Research Goals and Trends

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GOALS

- “Better” materials : ***high contrast, low optical absorption, low mechanical losses***
- **Cryo – compatibility (3G detectors, Einstein Telescope)**
- **“Easy” technology, scalability**

RESEARCH LINES (see I. Pinto, LVC Document G1700171)

- **Microscopic/molecular modeling** (TLS models - UFL, Stanford, Glasgow)
- **High-temperature deposition** (enhanced surface mobility, ultrastable glasses - Stanford)
- **Ion Plating** (Glasgow)
- **Glassy oxide mixtures and more** (LMA, H&WSC, CSIRO)
- **Multi (>2) material coatings** (Glasgow, MIT)
- **Nanolayered composite materials** (USannio, NTHU, UFL) ⇨ **iCoRe**
- **Crystalline (GaAlAs, GaAlP) materials** (CMS & LLC, Stanford, Glasgow, LMA)
- **Silicon Nitrides** (NTHU)
- **Diffractive mirrors and metamaterials** (UBraunschweig, USannio)

FUNDING

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

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Nanolayered Films : Rationale

Dense (→ high contrast, fewer doublets, lower noise) coating materials, including Titania (n=2.33@1064nm) Zirconia (n=2.12) and Hafnia (n=2.08), featuring almost no mechanical loss-peak at cryo-temperatures, crystallize upon annealing, with observed subsequent blow-up of optical & mechanical losses (annealing in Silica reduces losses).

Silica doped Titania tolerates higher annealing temperature before the onset of crystal-lization [Chao et al, Appl. Opt 40 (2001) 2177]; same behaviour observed in Silica doped Zirconia and Hafnia [Ushakov, Phys. Stat. Sol. B241 (2004) 2268 (2004)].

Nanolayered Titania/Silica composites were first studied in [Gluck et al., J. Appl. Phys. 69 (1991) 3037]. Thinner layers were shown to tolerate higher annealing temperatures. Similarly, nanolayered Hafnia/Alumina composites tolerate high annealing temperatures [Liu et al., Appl. Surf. Sci. 252 (2006) 6206].

We introduced the idea of nanolayered Silica/Titania composites for GW detectors [LIGO-G], developed in collaboration w. Chao [NTHU] and DeSalvo's Groups.

Nanolayering e.g. Zirconia/Titania or Hafnia/Titania may hinder crystalization *in both materials*, due to crystalline mismatch (unproved yet, needs to be checked).

Nanolayered Films : What/How

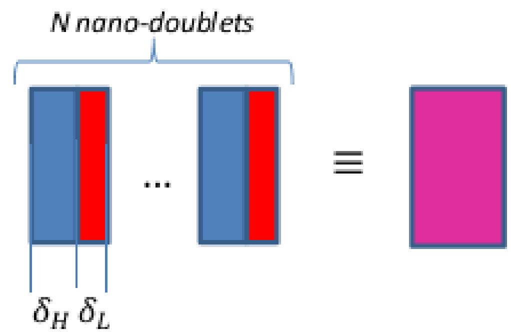
… The simplest geometry uses **cascaded nano-doublets**, and is thus specified by (N, δ_H, δ_L).

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

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

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

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



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

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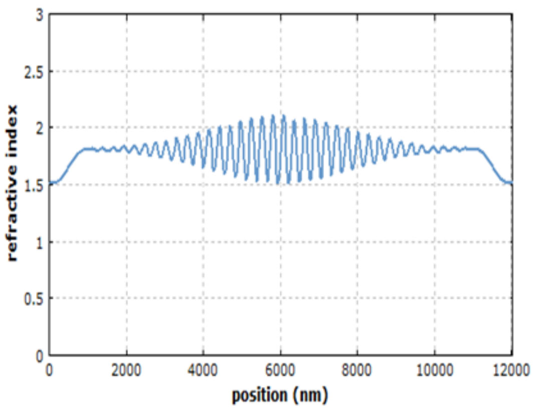
Technology

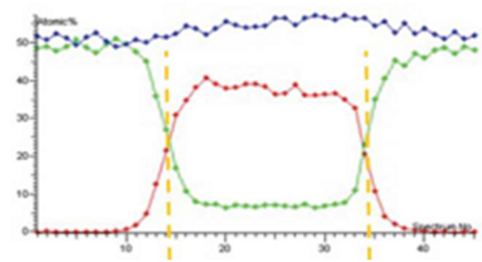
iCoRe



Very thin layers are not new in applied optics: rugate filters [Southwell, Appl. Opt. 24 (1985) 457] and X-ray mirrors are routinely made using nm-scale layers [Gullikson, Proc. 8th PXRMS (2006)]

Thickness inaccuracies in the deposited individual low/high index layers are irrelevant provided the total thickness ratio has the design value

Using thinner nanolayers entails an incrasing number of interfaces, making interfacial irregularities potentially more and more relevant. Preliminary measurements show that film surface rugosity and thickness uni-formity do not increase with # layers



energy-dispersive X-ray diffraction (EDXRD) of 2.2/4.8nm 19 layers film interfaces

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The iCoRe Project

iCoRe

Coordinator: Innocenzo M. Pinto (professor, OSA Fellow)

Participants: Elisabetta Cesarini (CF postdoc)
Maria Principe (L'Oreal UNESCO Women in Science Fellow; PhD)
Joshua Neilson (PhD student @ University of Sannio)

Place of Work : University of Salerno
(TEM, STM, AFM, XRD film characterization)
University of Sannio at Benevento
(film deposition Lab, ion-assisted e-beam evaporator)
University of Rome "Tor Vergata"
(film mechanical loss measurement)

Collaborations : NTHU (National Tsing Hua University), Taiwan, ROC
Virgo VCR&D (Genoa, Pisa, Rome, Rome ToV, Perugia, Urbino)
LIGO CCR (AU, H&W-S, UFL, CSULA,
CNR - IMM (Naples, Lecce)

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2018 Summary of Activities

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- We started operating a coating deposition facility based on a custom version of the OPTOTECH-OAC75F ion-assisted e-beam evaporator in November 2017, thanks to substantial funding (> 600 KEUR) by Regione Campania.
- We established an extremely fruitful collaboration with the SPNM and CNR-Spin Laboratories of the University of Salerno for thin-film characterization usingSEM/TEM, AFM and XRD. Our group at USannio merged with the Salerno group to form a single INFN Virgo working group.
- Collaborations with other Virgo groups involved (Genoa, Pisa, Rome “Tor Vergata”, Perugia, Urbino) was promoted in the frame of the Virgo Coating R&D (VCR&D) effort.
- Collaboration with the Photonics Lab of the National Tsing Hua University of Taiwan, (prof. Chao), our first partner in the development of nanolayered composite optical films, continued.

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USannio Coating Lab

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Coating machine (front view) and laminar-flow hood

Coating machine (rear view)

The Venue (CeRICT/MUSA Labs)

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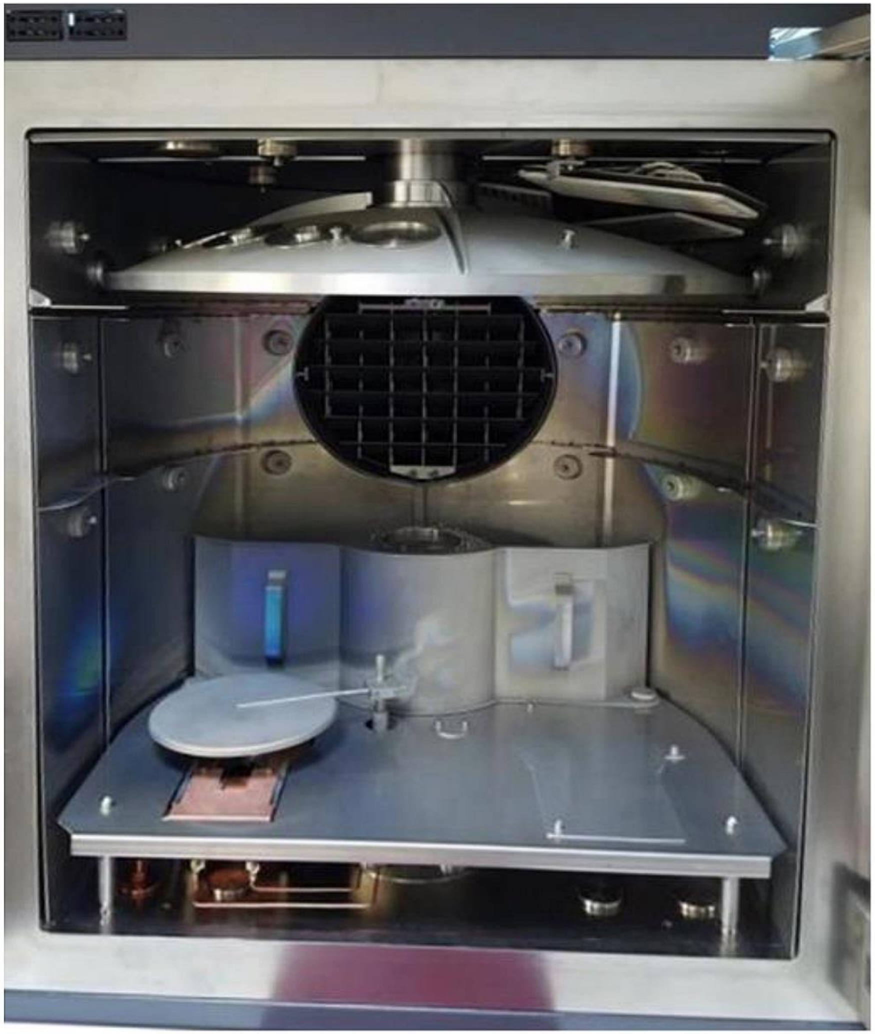
USannio Coating Lab

iCoRe



- 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 ehnance uniformity
- Ceramic lamps to heat the substrate

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iCoRe UniSA Facilities

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The UniSA WG has several facilities for morphological, structural and compositional characterization of optical thin films:

- Three room temperature AFM/AFS (*Bruker, Multimode V; Jpk, Nanowizard III; Nanite, Nanosurf*);
- Two UHV ($P < 10^{-9} mbar$) AFM/STM (*Omicron*), one working in a temperature range from T_{amb} down to $T = 5K$;
- A SEM station (*Zeiss, Leo EVO 50*) w. energy dispersive spectroscopy (EDS) ;
- A field-emission-SEM *(Zeiss, Σigma GEMINI)*;
- an X-ray Diffractometer *(Philips, X'Pert MRD-PRO @* CNR-Spin Lab*)*;
- A FeG-SEM (*FEI, INSPECT F*) w. integrated nanolithography system (*Raith Elphy Plus*);
- A photolithography station.

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iCoRe UniSA Facilities

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SPnM (Scanning **P**robe Microscopy and **n**ano**M**atter) lab.

MUSA (**Mu**ltifunctional materials **S**ynthesis & **A**nalysis) lab (CNR - SPIN)
(R. Fittipaldi)

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iCoRe Pipeline

- Depositions @ USannio
- Heat-treatment
- XRD (crystallization study)
- AFM, TEM, STM (morphology, interfaces, etc)

(Heat-treatment, XRD, AFM/TEM/STM) @ UniSA

- Raman/Brillouin @ CNR-IMM (Naples)
- Mechanical loss (ringdown, GeNS) @ RomeToV
- Ellipsometry measurements @ Genoa

➡ ***Missing** measurement facilities (as of August 2018)*
- ***optical losses***
- ***thermooptic coeffs***
- ***direct TN***

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AFM Surface Roughness Analysis

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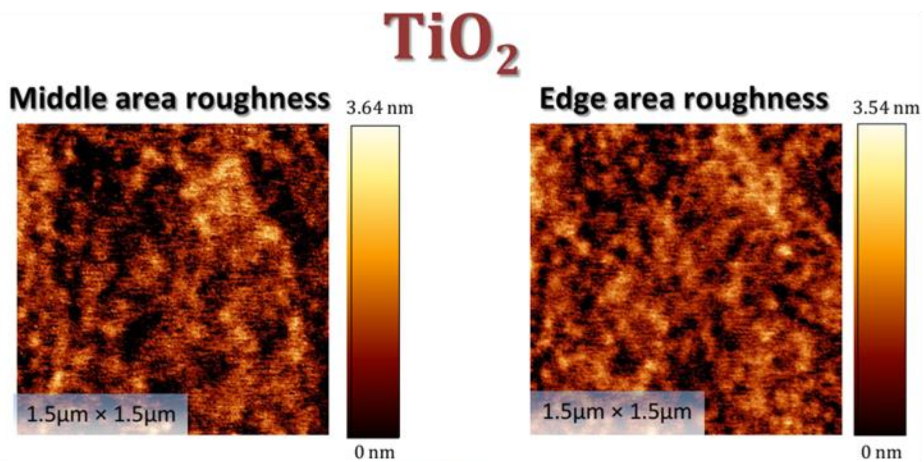
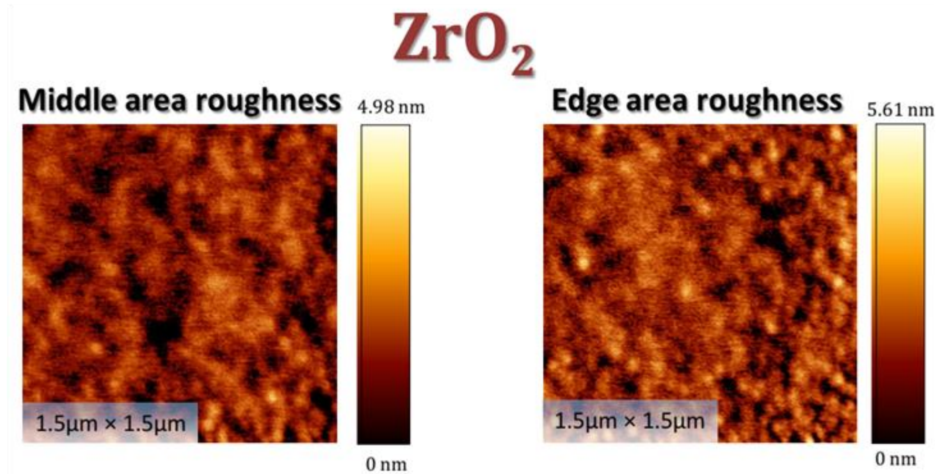
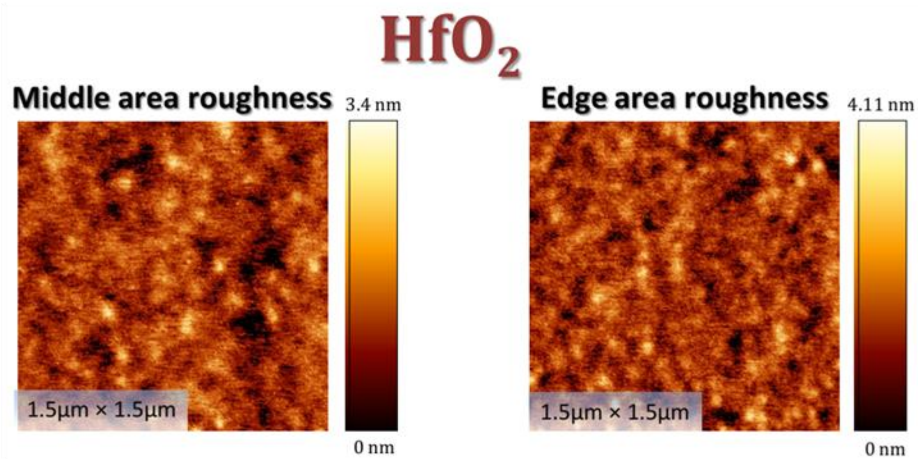
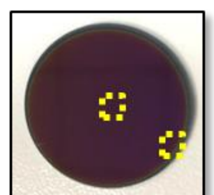
(single layer 1in Ø, 200nm thick, with IAD)









	Max P-V roughness [nm]	Max RMS roughness [nm]
TiO_2	3 ± 0.1	< 1
ZrO_2	4 ± 0.1	< 1
HfO_2	3.5 ± 0.1	< 1
SiO_2	1.5 ± 0.1	< 1

(C. DiGiorgio, O. Durante)

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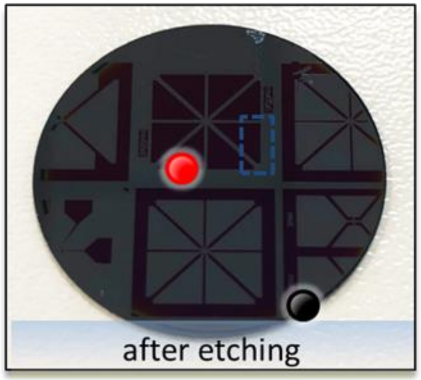
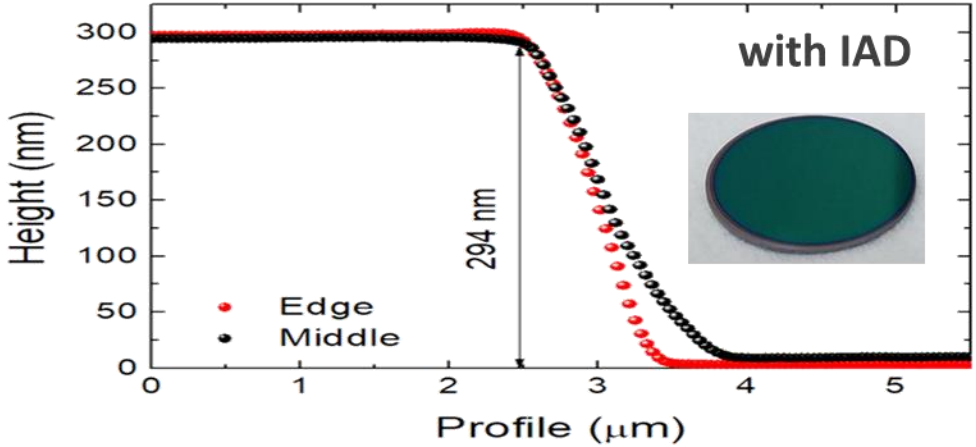
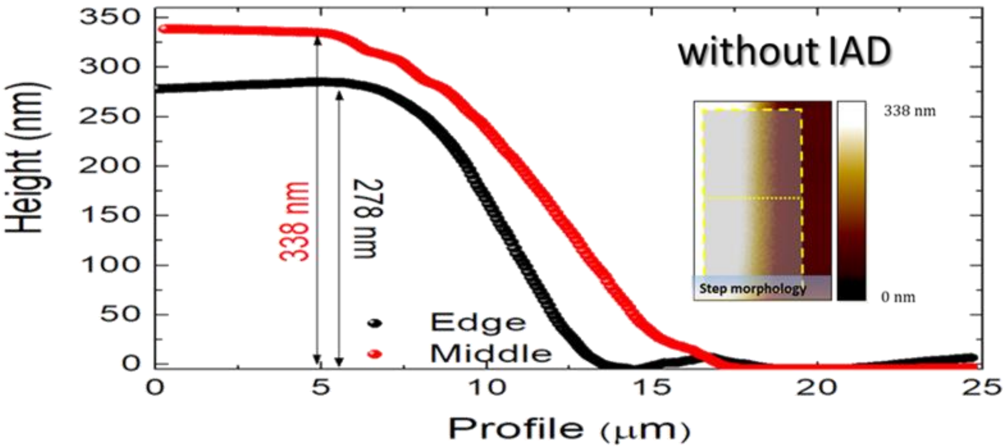
AFM Thickness Uniformity Analysis iCoRe

Selective wet-etching by photolithographic masking.
Average of hundreds profiles along the film-substrate step, middle region (red) and edge (black)

10 x TiO2/SiO2 doublets
25nm-thick each







(G. Carapella, C. DiGiorgio)

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Optical/Mechanical Props

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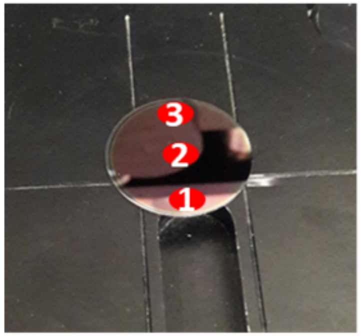
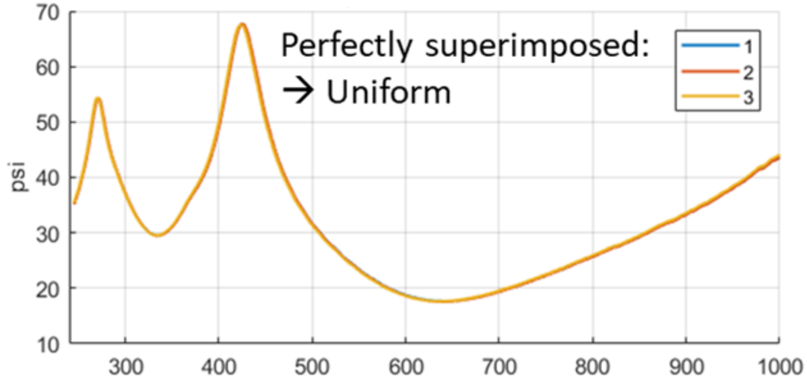
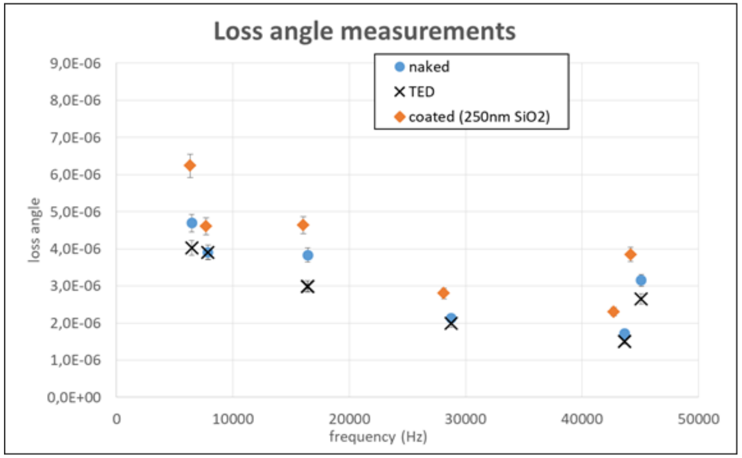
(USannio single Silica layer 1 in Ø, 250 nm thick, with IAD, as deposited)



SiO_2 film single layer 250nm thick



(M. Canepa, UniGE)



$$\varphi_{coating} = (1{,}1 \pm 0{,}4) \cdot 10^{-4}$$

in line w. results in the Literature

(E. Cesarini, D. Lumaca, UniRm ToV)

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Mechanical Loss Measurements

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Main Results,contd.

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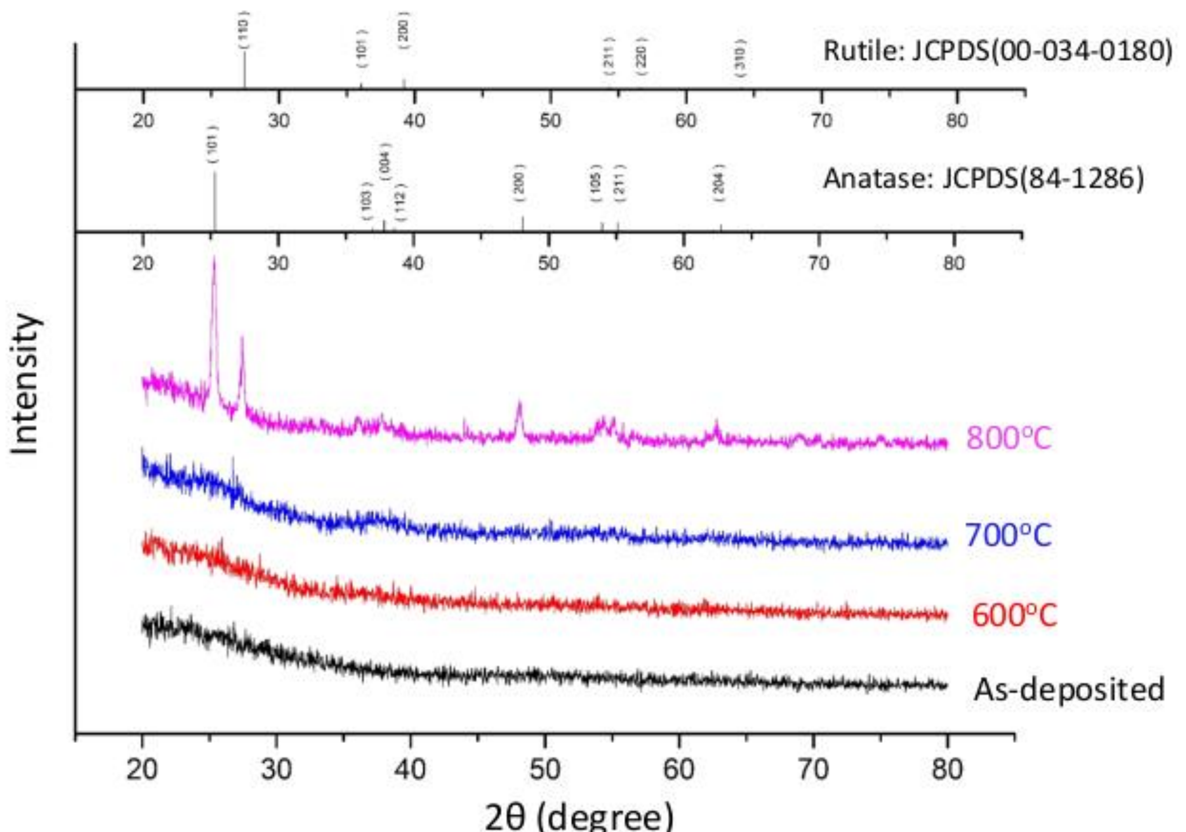
Silica/Titania nanolayered composites with individual layer thicknesses below 3nm were annealed at 600C (24h) withour crystallization (collaboration with NTHU)

XRD



75-layer (TiO_2 =1.8nmx38, SiO_2 =3.6nmx37)

(courtesy S. Chao)

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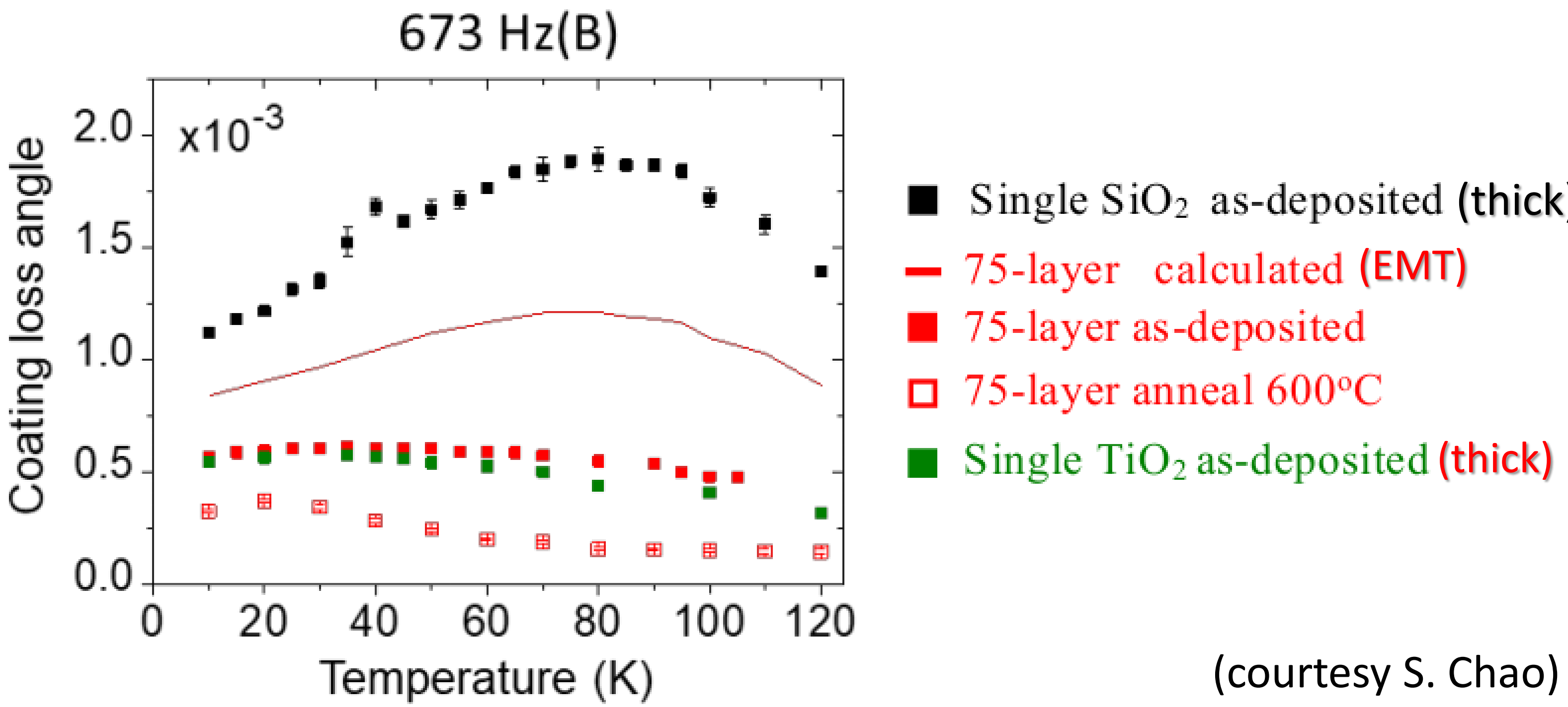
Main Results, contd.

- Nanolayering Silica/Titania suppresses Silica's mechanical loss peak at cryogenic temperatures. Does this apply to other glassy oxides (e.g., Tantala) ?



(courtesy S. Chao)

- Annealing at 600C (24h) reduces coating loss angle by a factor ~2 .

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2018 Achieved Milestones

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- Production of nanolayered SiO2/TiO2 films (optically equivalent, e.g., same refractive index and optical thickness but different individual layer thicknesses and total number of nanolayers).

- Set up of a characterization pipeline (film morphology, optical properties, mechanical losses) for nanolayered films.

- Optimization of the deposition-process parameters for best quality of the films. We expect further improvements in the film quality (especially as regards morphology) by finer tuning of the plasma-ion assistance unit.

iCoRe Work Plan 2019 - 2020

- We will deposit, and characterize nanolayered films consisting of a good glass-former (SiO_2 or Al_2O_3) paired with an optically dense (but prone to crystalliza-tion upon annealing) material (Zirconia, Hafnia, Tantala, Niobia);
- We will investigate whether /to what extent the temperature of the deposition target (that we can control) affect the quality and structure of the film.
- We will deposit and characterize nanolayered Silica/Alumina films. These could be an appealing option for the low-index coating material of 3G cryogenic de-tectors (ET), in terms of all relevant figures (low optical index, low mechanical and optical losses, no cryo-peak, high annealing temperature).
- On the modeling side, we will investigate the optimal design of m-ary reflective coatings with m > 2, allowing relatively lossy materials but denser materials (e.g., Silicon, Lantanides , Tellurium, etc.).

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Milestones 2019

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1- Deposition of high index nanolayered films based on Silica (or Alumina) alternated with Zirconia, Hafnia, Tantala, Niobia;

2- Characterization of the above composite films : maximum annealing temperature before crystallization vs nanolayer thickness; optical quality; mecha-
nical losses;

3- Deposition of low index Silica/Alumina nanolayered films for cryogenic operation (cryo-testing at NTHU);

4- Characterization of the above low index composite films   (maximum annealing temperature before crystal-lization, optical quality (index, losses), mechanical losses (in particular at cryo- temperatures)

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Fundings

Expected fundings (next 3-years, starting 2019):

-Request of funding by Centro Fermi

 # 2 Grants (2019-2020) – one of the continuation of Dr. Cesarini's

*>> **No budget** requested for consumables or inventory*

-External fundings (yearly)

 # Virgo (INFN-CSN2) funding (consumables & inventory);

-Potential external fundings

 # ERC grant application by Maria Principe @ Usannio

 # PRIN-SUD application to MIUR (1918), worth 1M EUR

-International fundings

 # the NTHU Group is funded by Taiwan NSF

People

USannio

Left to right: Giuseppe Castaldi, Vincenzo Galdi, Joshua Neilson (PhD fellow @ USannio, supported by CSULA & GSSI), Max Moccia, Vincenzo Pierro, Innocenzo M. Pinto, Maria Principe

UniSA

Left to right: Fabrizio Bobba, Giovanni Caraella, Cinzia Diiorgio, Ofelia Durante, Rosalba Fittipaldi, Vincenzo Fiumara (now at UniBAS), Francesco Chiadini

+ Rome ToV Folks… see 2nd part of talk (Elisabetta's)

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People, contd.

iCoRe

Prof. Chao Shiuh (NTHU PI, and our historical research partner)
visited our group and Lab in November 2018
(supported by INFN-FAI)

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iCoRe

Publications (2018)

M.L. Gorodetsky, Y. Levin, **I.M. Pinto**, S.P. Vyatchanin	Editorial – In Memoriam Vladimir Borisovich Braginsky	Physics Letters, Section A	2018	10.1016/j.physleta.2018.05.025
I.M. Pinto, M. Principe et al.	A multi-step approach to assessing ligo test mass coatings	J. Physics: Conference Series	2018	10.1088/1742-6596/957/1/012010
I.M. Pinto, M. Principe et al.	Optical properties of amorphous SiO2-TiO2 multi-nanolayered coatings for 1064-nm mirror technology	Optical Materials	2018	10.1016/j.optmat.2017.09.043
I.M. Pinto, M. Principe et al.	Optical scattering measurements and implications on thermal noise in Gravitational Wave detectors test-mass coatings	Physics Letters, Section A	2018	10.1016/j.physleta.2017.05.050
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	All-sky search for long-duration gravitational wave transients in the first Advanced LIGO observing run	Class. Quantum Grav.	2018	10.1088/1361-6382/aaab76
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	Constraints on cosmic strings using data from the first Advanced LIGO observing run	Phys. Rev. D	2018	10.1103/PhysRevD.97.102002
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	Effects of data quality vetoes on a search for compact binary coalescences in Advanced LIGO's first observing run	Class. Quantum Grav.	2018	10.1088/1361-6382/aaaafa
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	First Search for Nontensorial Gravitational Waves from Known Pulsars	Phys. Rev. Letters	2018	10.1103/PhysRevLett.120.031104
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	Full band all-sky search for periodic gravitational waves in the O1 LIGO data	Phys. Rev. D	2018	10.1103/PhysRevD.97.102003
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	GW170817: Implications for the Stochastic Gravitational-Wave Background from Compact Binary Coalescences	Phys. Rev. Letters	2018	10.1103/PhysRevLett.120.091101
I.M. Pinto, M. Principe, the LIGO Scientific Collaboration	Identification and mitigation of narrow spectral artifacts that degrade searches for persistent gravitational waves in the first two observing runs of Advanced LIGO	Phys. Rev. D	2018	10.1103/PhysRevD.97.082002
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	Prospects for observing and localizing gravitational-wave transients with Advanced LIGO, Advanced Virgo and KAGRA	Living Reviews on Relativity	2018	10.1007/s41114-018-0012-9
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	Search for Tensor, Vector, and Scalar Polarizations in the Stochastic Gravitational-Wave Background	Phys. Rev. Letters	2018	10.1103/PhysRevLett.120.201102
E. Cesarini, I.M. Pinto, M. Principe, the LIGO-Virgo Collaboration	GW170817: Measurements of Neutron Star Radii and Equation of State	Phys. Rev. Lett.	2018	10.1103/PhysRevLett.121.161101

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CENTRO FERMI

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Thanks for Listening!

Questions ?

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