ET Coating R&D @UniSa/UniSannio

F. Bobba, G. Carapella, F. Chiadini, C. Di Giorgio, O. Durante,

R. Fittipaldi, V. Fiumara, J. Neilson, V. Pierro, I.M.Pinto, M. Principe

Summary

- UniSa/UniSannio Team
- Scientific Case and Goals of UniSa/UniSannio group
  - *Coating noise relevance, and the quest for cryo-friendly coating materials*
  - *Design and Fabrication of optical coatings based on amorphous nano-layered oxides*
- Optical Coating -Our Deposition Facility
  - *Ion-Assisted Electron Beam from OptoTech*
- Optical Coating – Our Characterization Facility
  - *Scanning probe microscopes, X-Ray diffractometers, Scanning Electron Microscopes*
- Collaborations
- Preliminary results, on-going work, and future plans

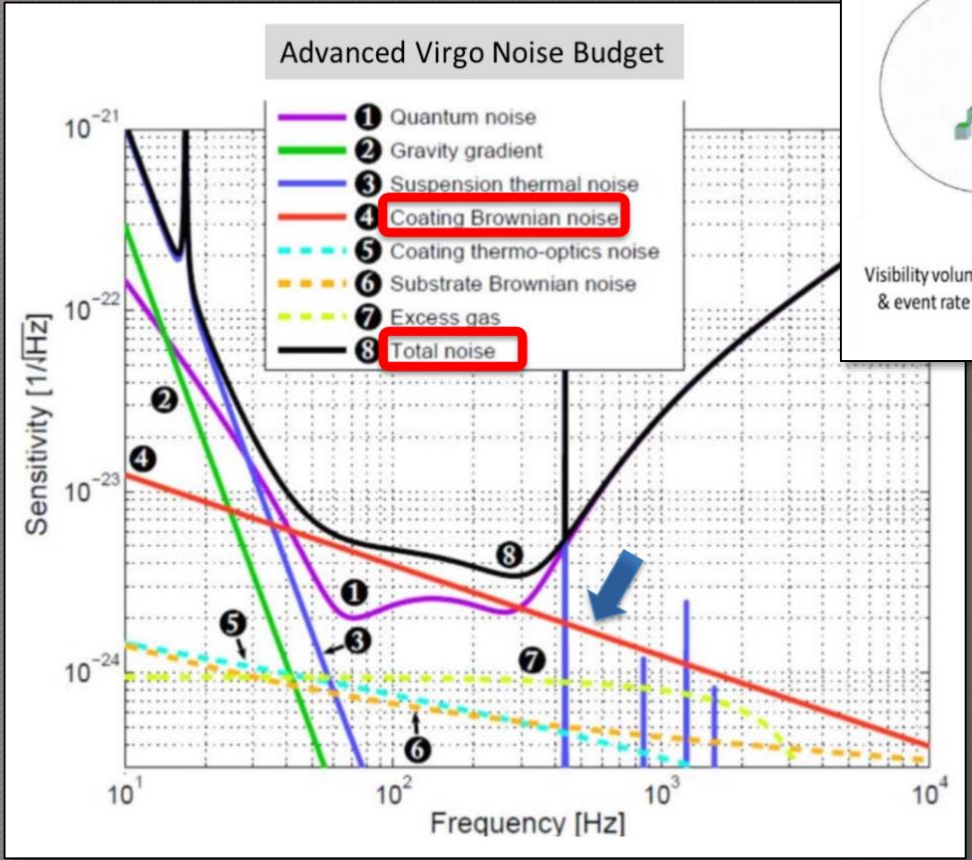
UniSa/UniSannio Team

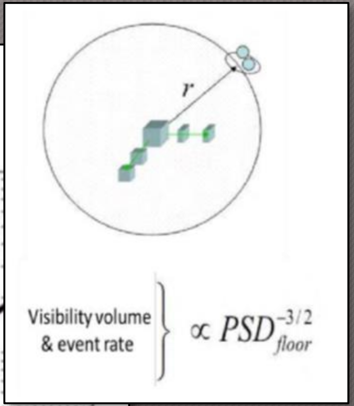
Name	Affiliation	Specialization
Fabrizio Bobba	UniSa - INFN	Material Science
Giovanni Carapella	UniSa - INFN	Material Science
Francesco Chiadini	UniSa - INFN	Optics/Metamaterials
Cinzia Di Giorgio	UniSa - INFN	Material Science
Ofelia Durante	UniSa - INFN	Ph.D. Student
Rosalba Fittipaldi	CNR-SPIN - INFN	Material Science
Vincenzo Fiumara	UniBas - INFN	Optics/Metamaterials
Joshua Neilson	UniSannio - INFN	Ph.D. Student
Vincenzo Pierro	UniSannio - INFN	Optical Modelling
Innocenzo Pinto	UniSannio - INFN	Optics, OSA Fellow
Maria Principe	UniSannio - INFN	Optical Engineering

7 permanents
2 Post-Docs
2 Ph.D. students

Scientific Case

Noise Spectrum of Virgo GW interferometer





Power spectrum of mirror thermal noise:

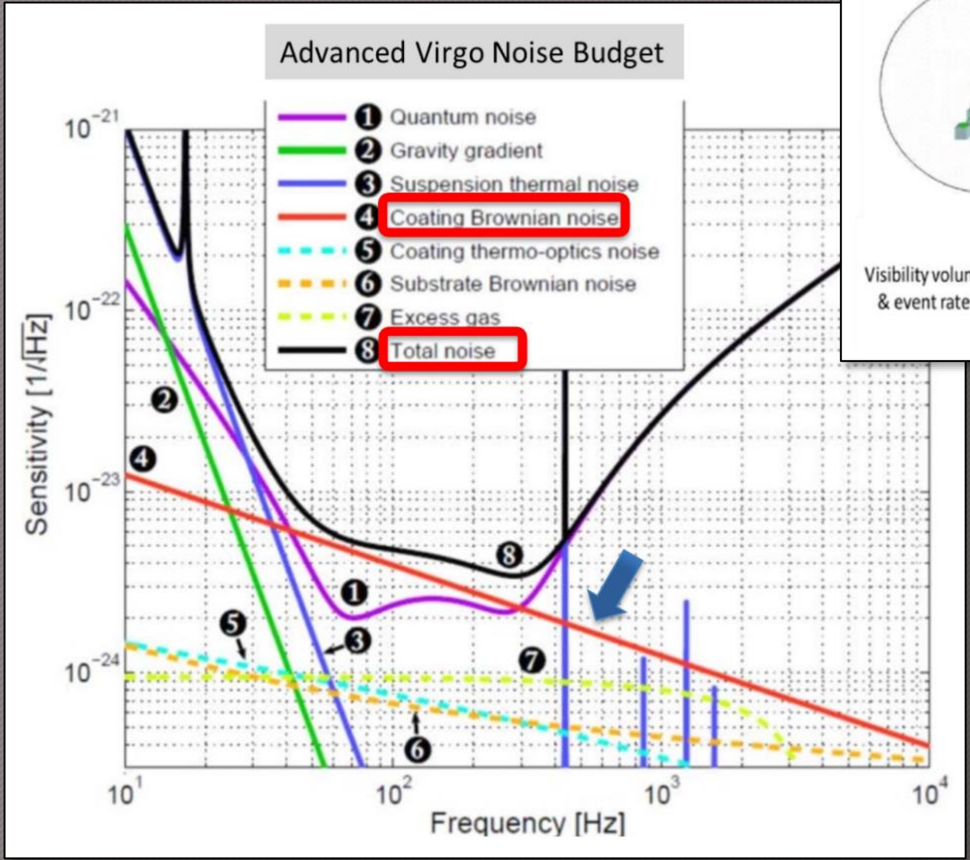
Temperature

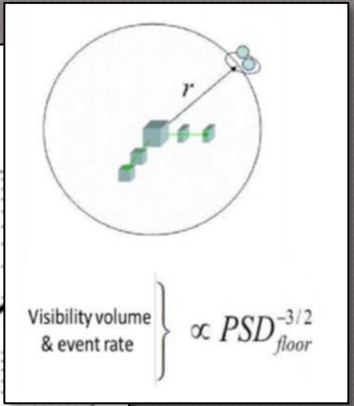
$$S_B(f) = \frac{2\, k_B T}{\pi^{3/2} f} \frac{(1-\sigma_s{}^2)}{w\, Y_s} \varphi_c$$

Mechanical loss angle

Scientific Case

Noise Spectrum of Virgo GW interferometer





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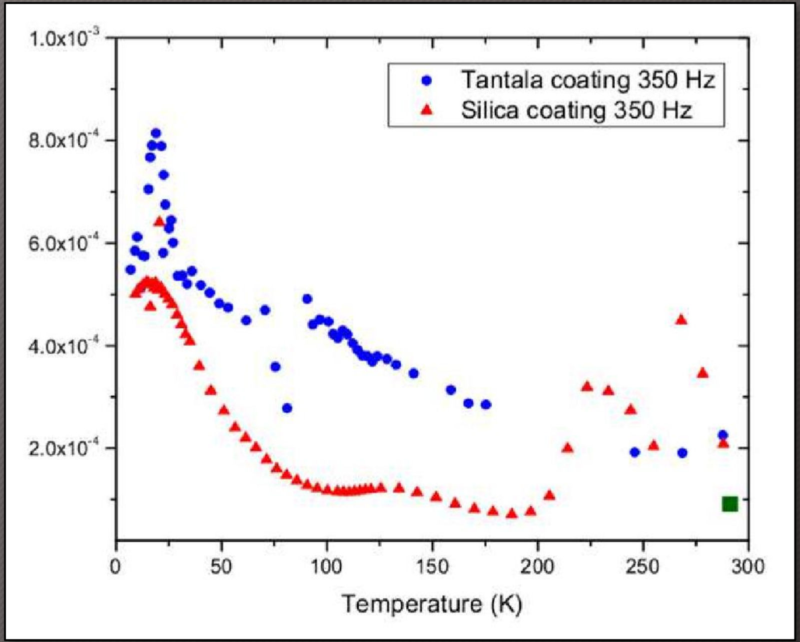
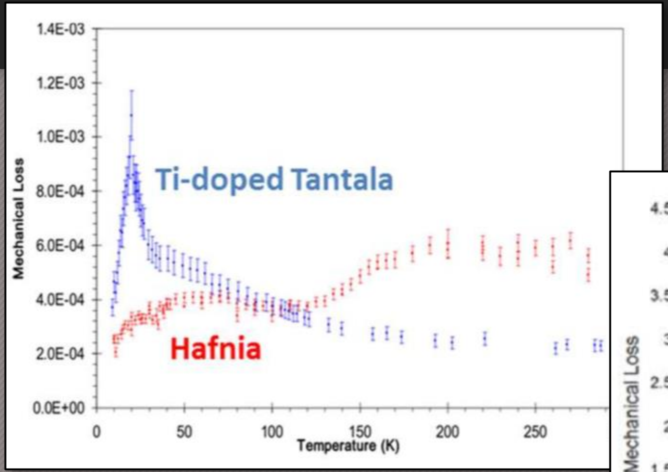
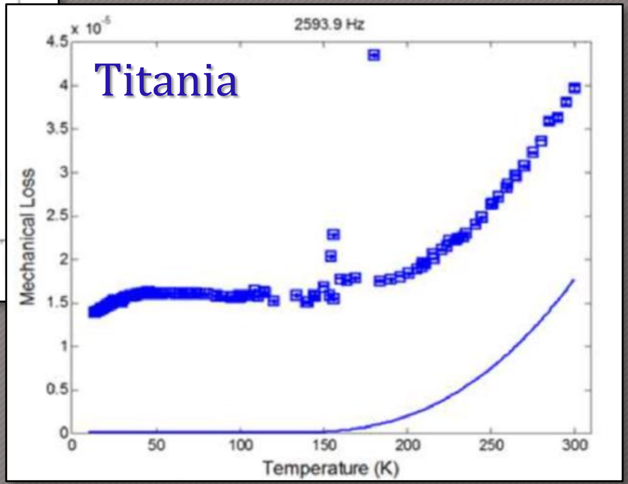


Mechanical loss behavior in nowadays used coating materials!

Scientific Case



Cryo-friendly coating materials exist..



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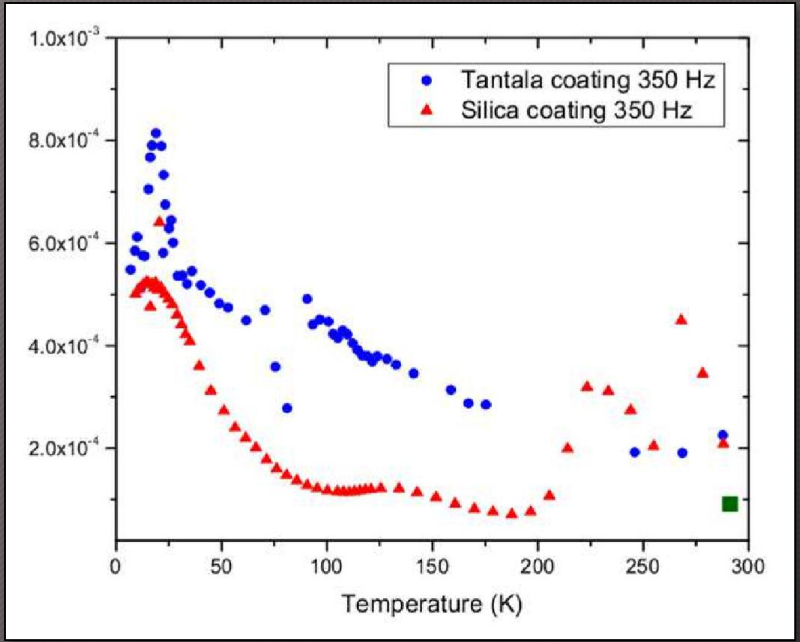
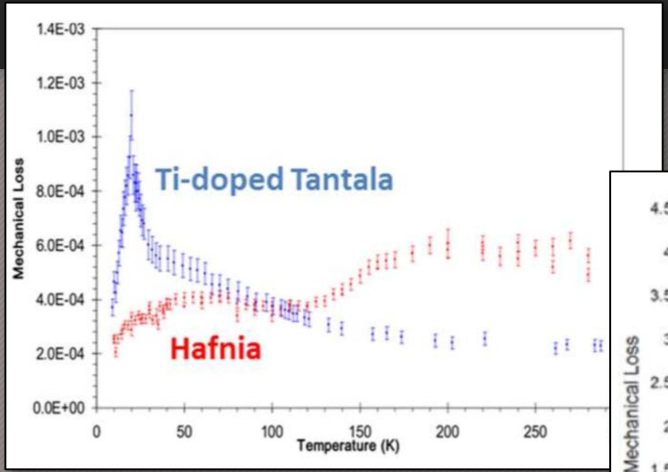


Scientific Case



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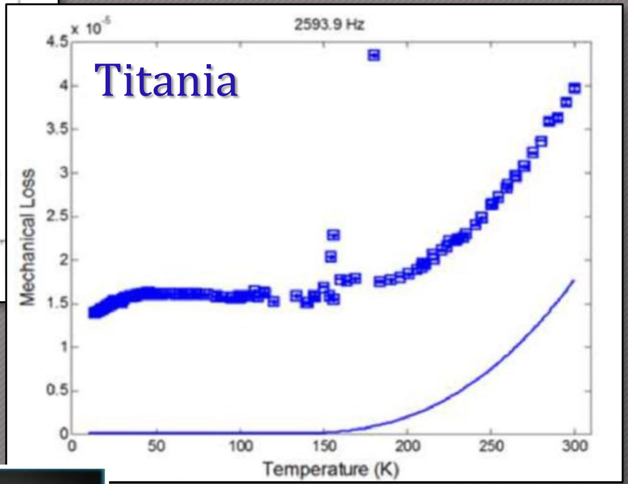

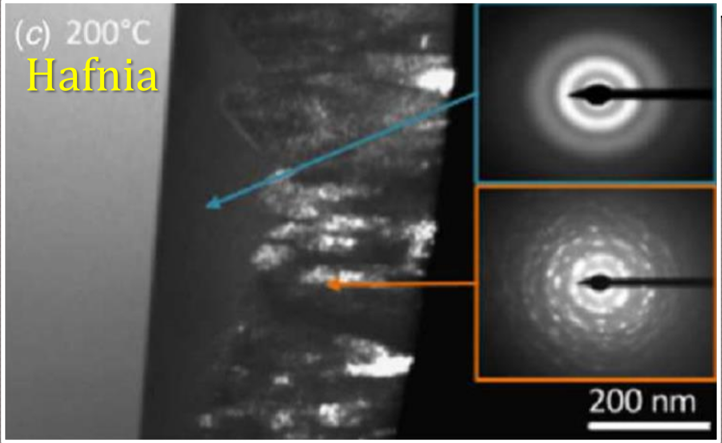

..but they crystalize very soon upon annealing!

Power spectrum of mirror thermal noise:

Temperature

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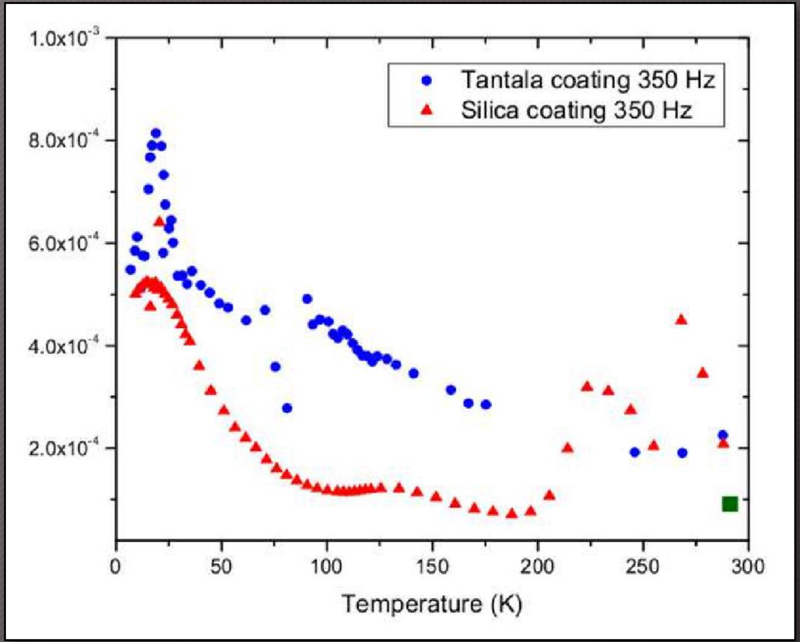
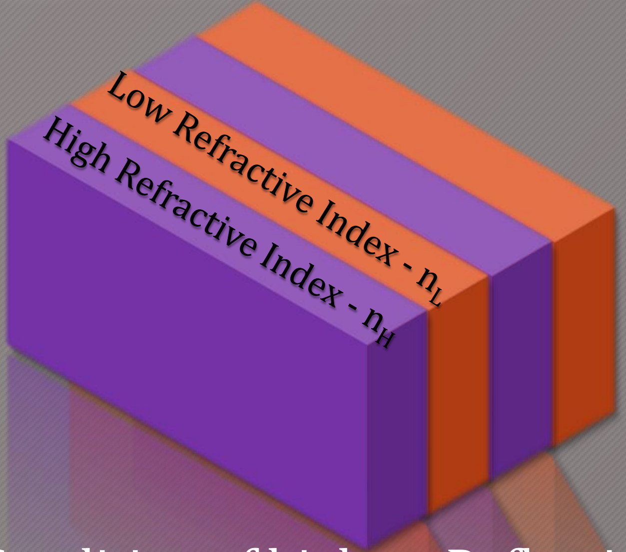


Background of UniSa/UniSannio group

Design, production & characterization of innovative optical coatings

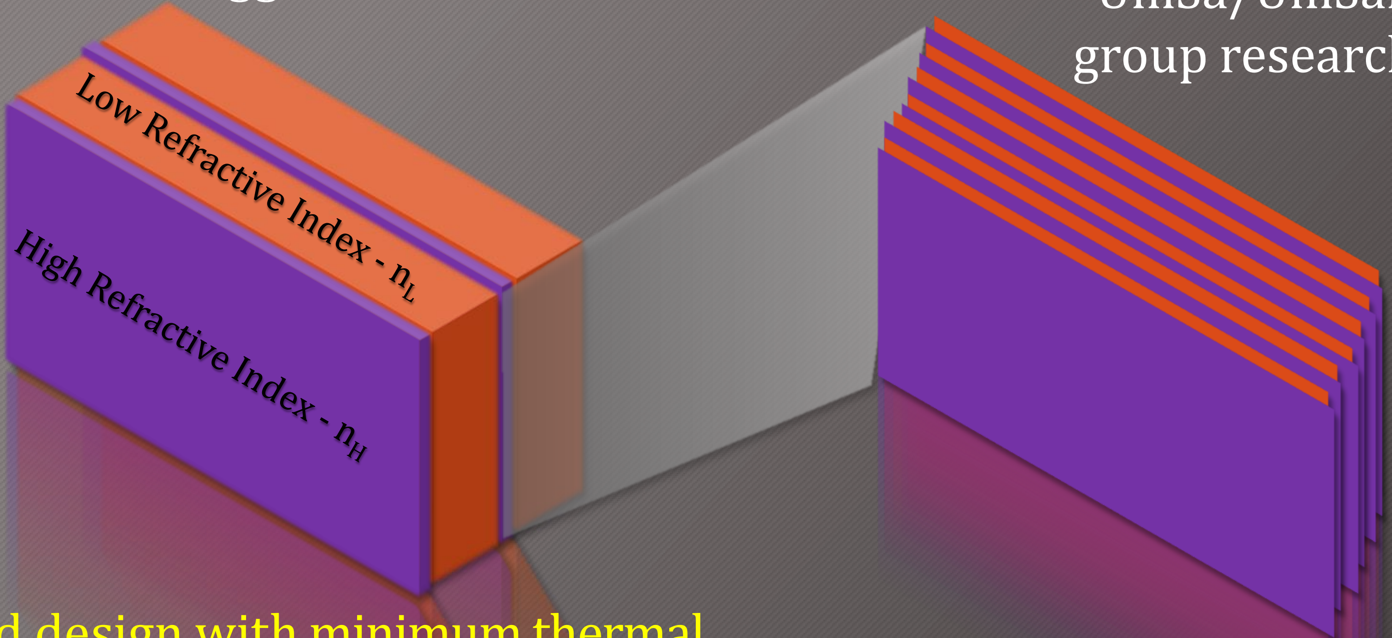
Starting point: Bragg Reflector



Condition of highest Reflectivity

$$t_H = t_L = \frac{\lambda}{4}$$

Modified Bragg Reflector

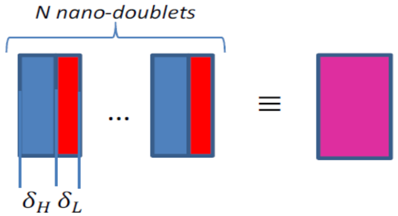


Optimized design with minimum thermal noise [PRD 81 (2010) 122001]

UniSa/UniSannio group research line

Replace homogeneous layers with *stratified nano-composites*

Background of UniSa/UniSannio group : Nanolayered Composites



Prescribing the nanolaminate index n_{eff} **determines uniquely** **the thickness ratio** of the low / high index materials

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

Prescribing the optical thickness z **of the nanolaminate** (in units of λ_0/n_{eff}) and a fiducial *minimum* thickness of the nanolayers **yields *all* *feasible*** (N, δ_H, δ_L) **designs**, 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

Many equivalent designs, with different N, δ_L, δ_H

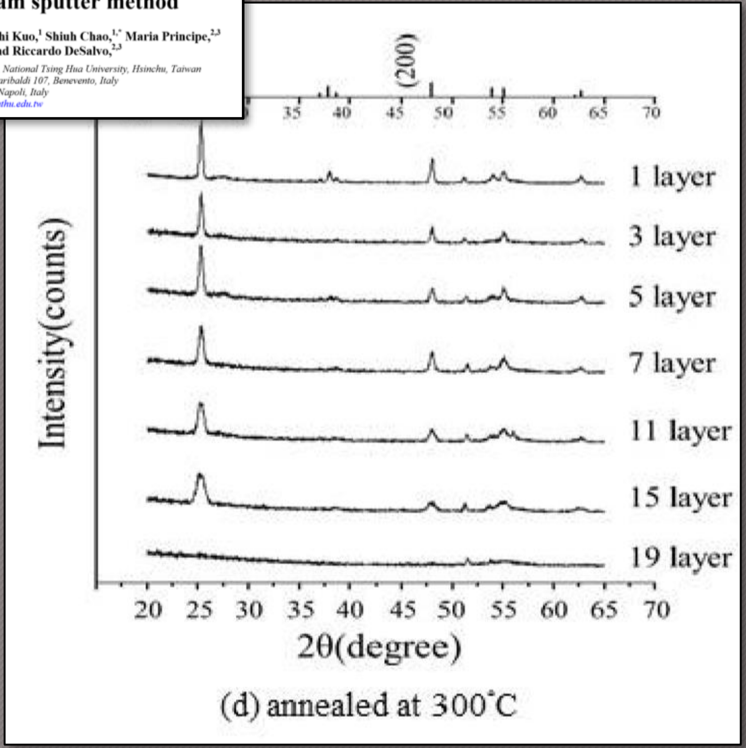
Background of UniSa/UniSannio group

Main Advantage: Nano-layering inhibits crystallization upon annealing!

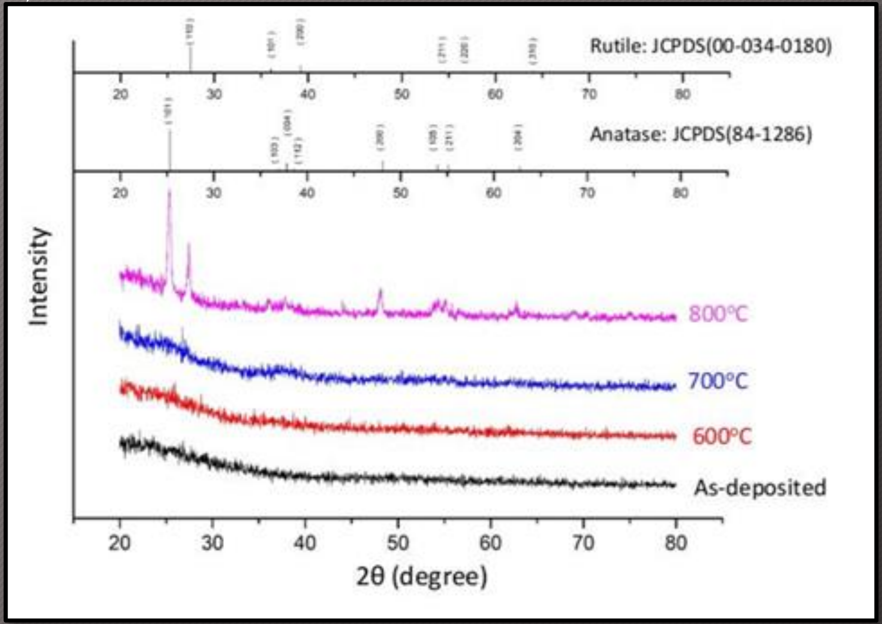
Thickness-dependent crystallization on thermal anneal for titania/silica nm-layer composites deposited by ion beam sputter method

Huang-Wei Pan,[1] Shun-Jin Wang,[1] Ling-Chi Kuo,[1] Shiuh Chao,[1,*] Maria Principe,[2,3] Innocenzo M. Pinto,[2,3] and Riccardo DeSalvo,[2,3]

[1] *Institute of Photonics Technologies and E.E. Dept., National Tsing Hua University, Hsinchu, Taiwan*
[2] *University of Sannio, Corso Garibaldi 107, Benevento, Italy*
[3] *INFN Sez.di Napoli, Italy*
[*] schao@ee.nthu.edu.tw



2014 → 2019

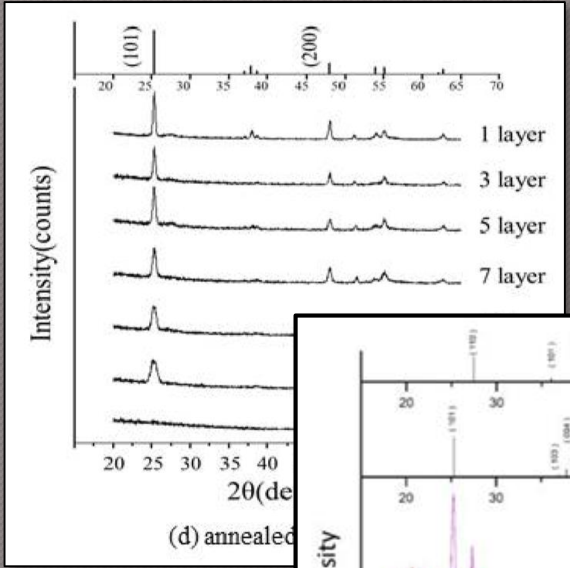


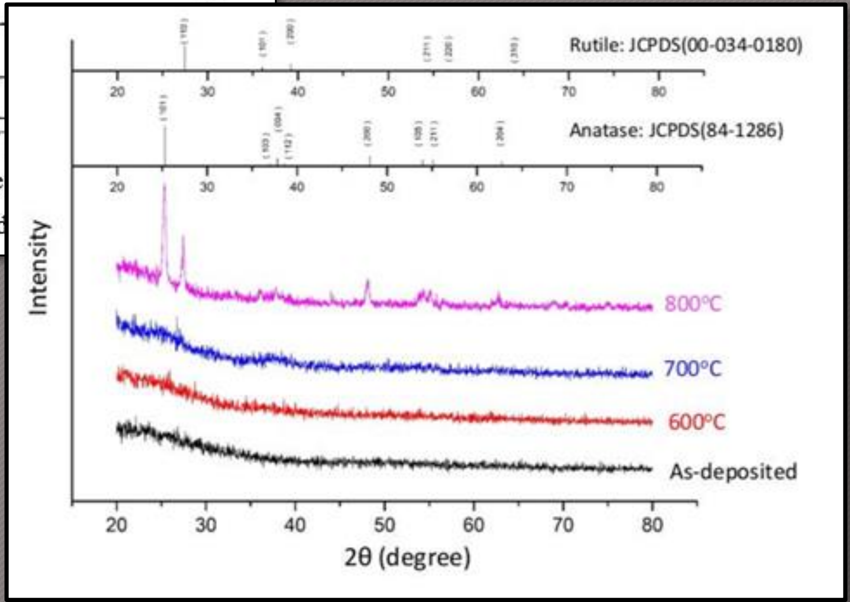
TiO_2 does not crystallize upon annealing up to 300°C when stack with SiO_2, in multilayers structure (10 nm each layer).

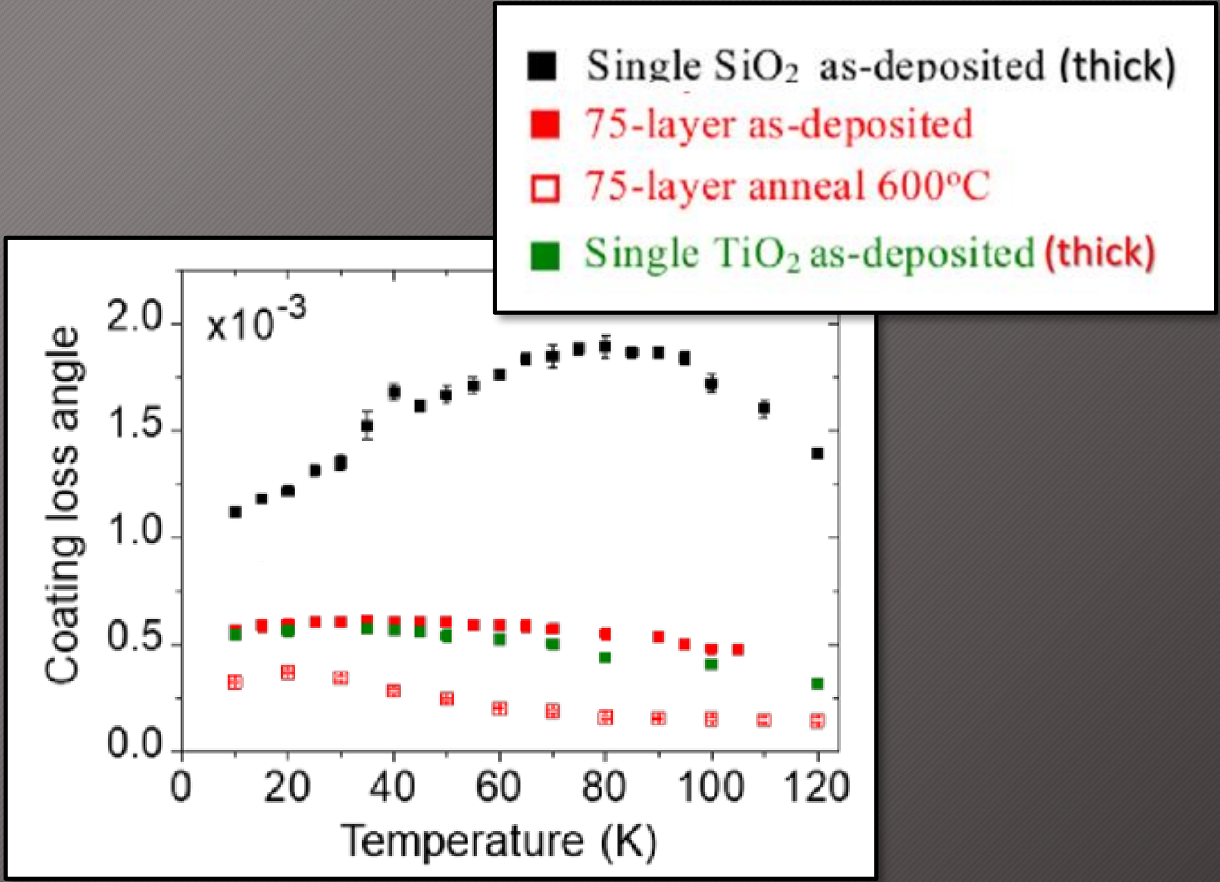
TiO_2 does not crystallize upon annealing up to 700 ° C when stack with SiO_2, in multilayers structure (3 nm each layer).

Background of UniSa/UniSannio group

Main Advantage: …thus resulting into lower losses even at cryo-T!







Background of UniSa/UniSannio group

Main Advantage of nano-layering strategy:

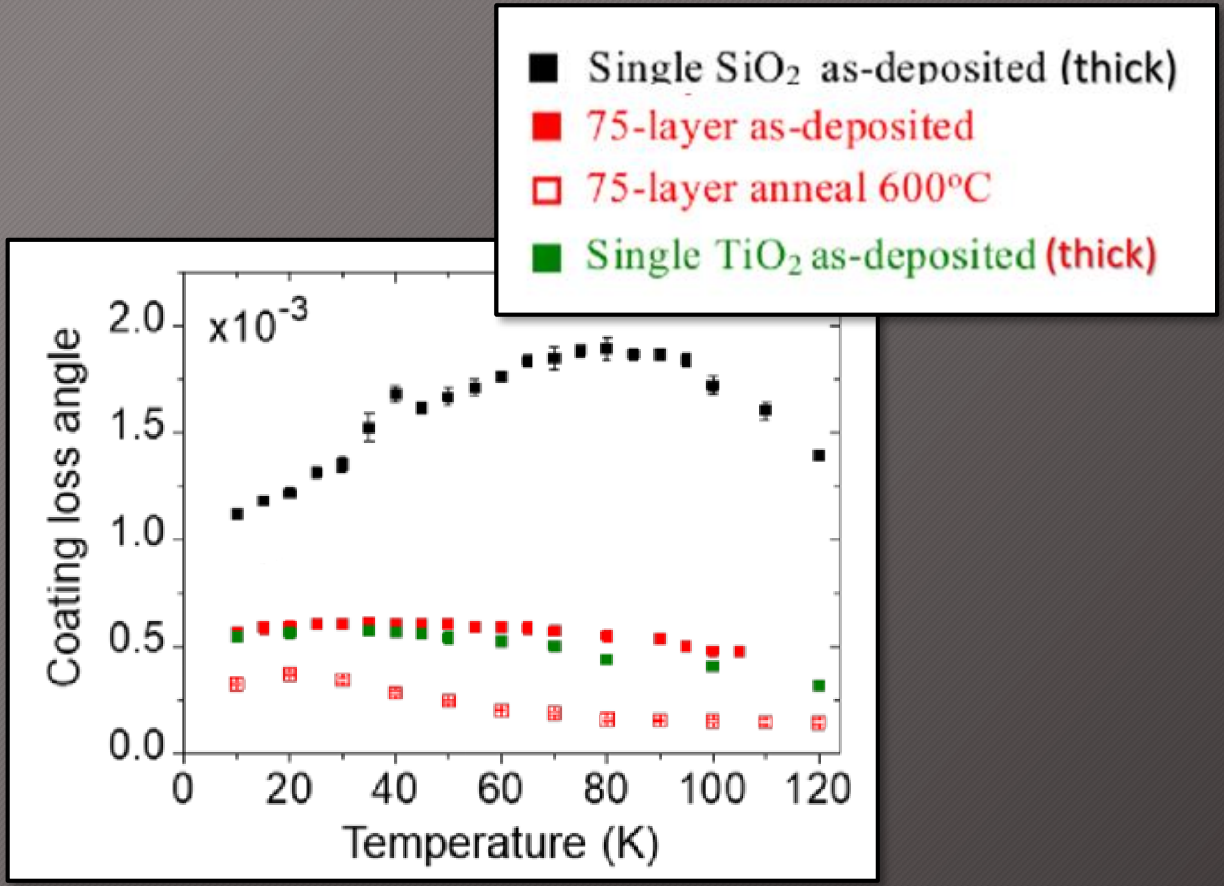
Higher crystallization temperature.

guaranteeing

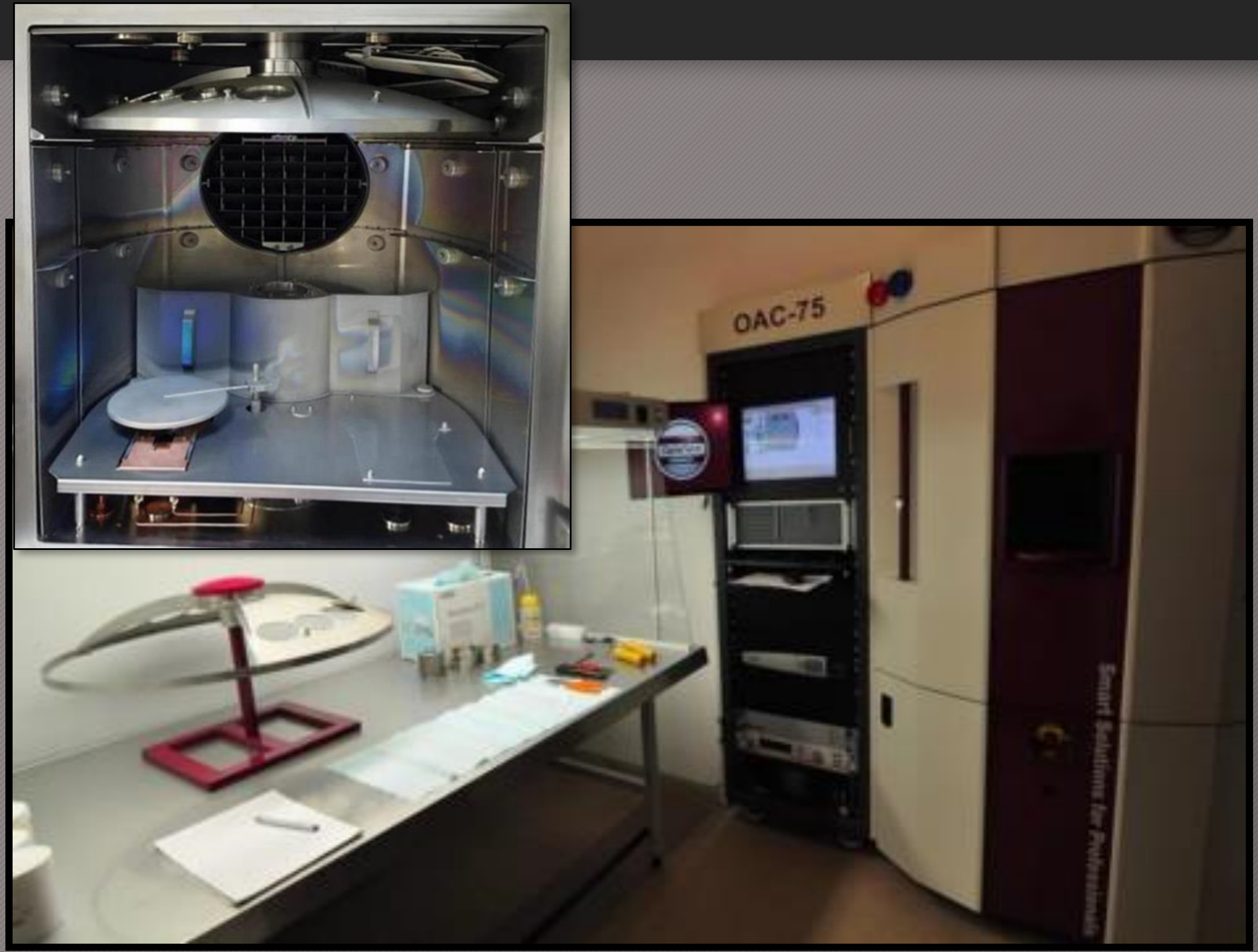
Higher temperature of post-deposition annealing.

leading to

1. Reduction of optical absorption and/or mechanical losses;
2. Smoother surfaces and interfaces.
3. Material stress and strain release.



Optical Coatings – Deposition Facility

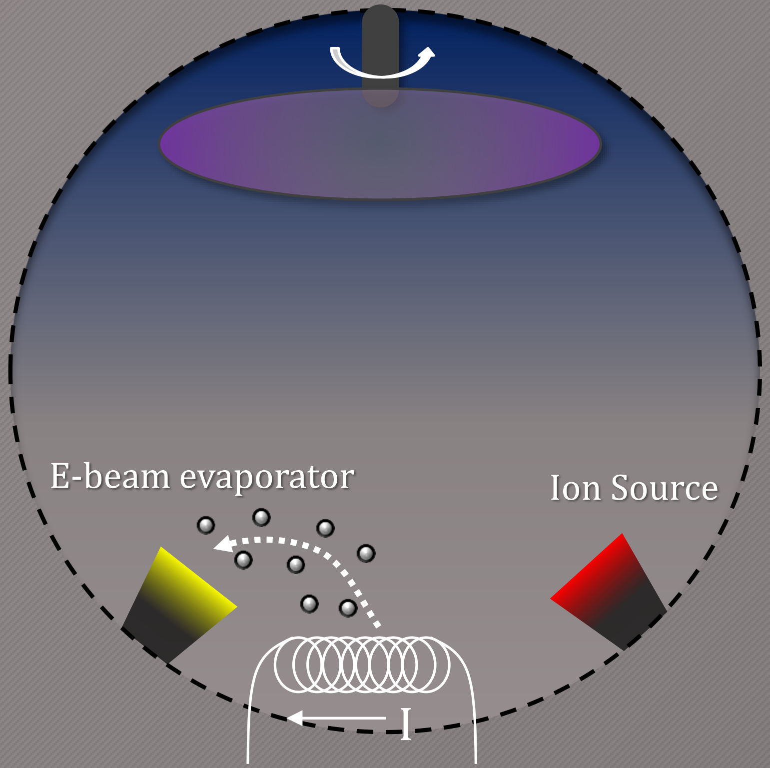


Ion Assisted Deposition Facility

- High-vacuum deposition chamber (10^{-7} mbar)
- Electron-beam source equipped with 6 crucibles
- Plasma source (IAD)
- Multiple samples deposition
- Sample size (from 1 to 5 inches)
- Ceramic lamp for possible high temperature depositions (up to 350°C)
- Rotary substrate holder for enhanced uniformity – with sub-nm accuracy/repeatability
- Fully programmable via GUI

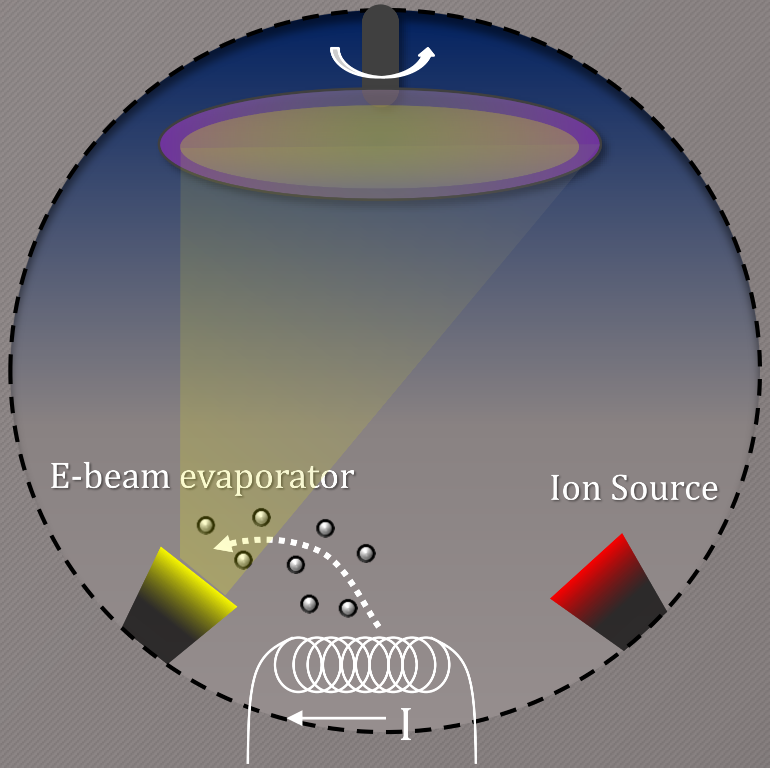
Optical Coatings – Deposition Facility

Ion Assisted – Electron Beam



- Electron-beam source: Electrons are emitted from a filament due to thermionic effect, thus deflected and accelerated against the target.

Optical Coatings – Deposition Facility



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- The target reaches the melting temperature (e.g. about 1700°C for SiO_2 and 1800°C for TiO_2) thus releasing atoms, which travel toward the substrate.

Optical Coatings – Deposition Facility

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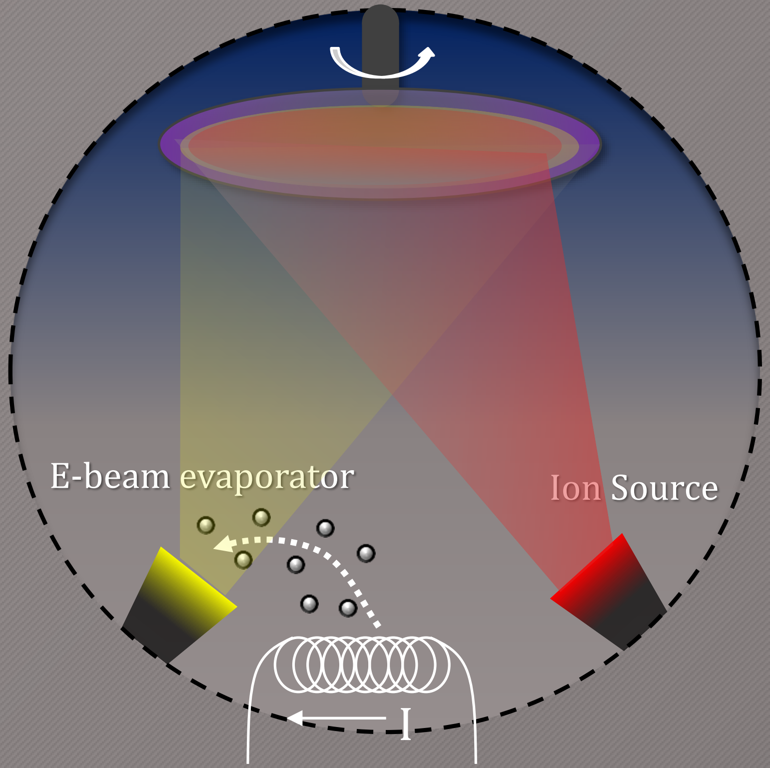

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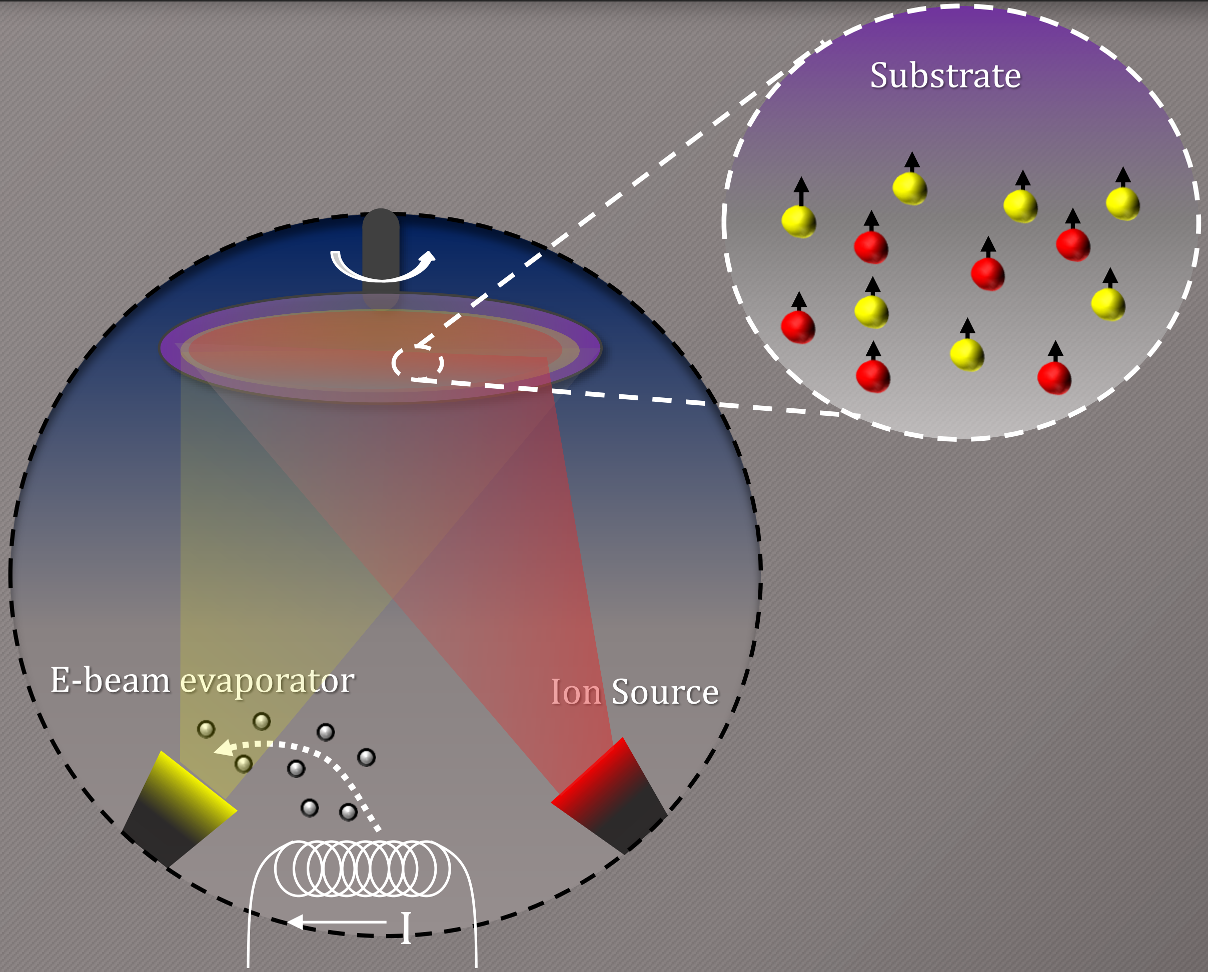
- In ion-assisted deposition, high energy ion beam (Ar, O_2) is focused toward the substrate

Optical Coatings – Deposition Facility



Ion Assisted – Electron Beam

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- The target reaches the melting temperature (e.g. about 1700°C for SiO_2 and 1800°C for TiO_2) thus releasing atoms, which travel toward the substrate.
- In ion-assisted deposition, high energy ion beam (Ar, O_2) is focused toward the substrate
- Thermally evaporated atoms adsorb kinetic energy by hitting the ions, thus increasing their own energy when reaching the substrate, giving rise to a more dense coating.

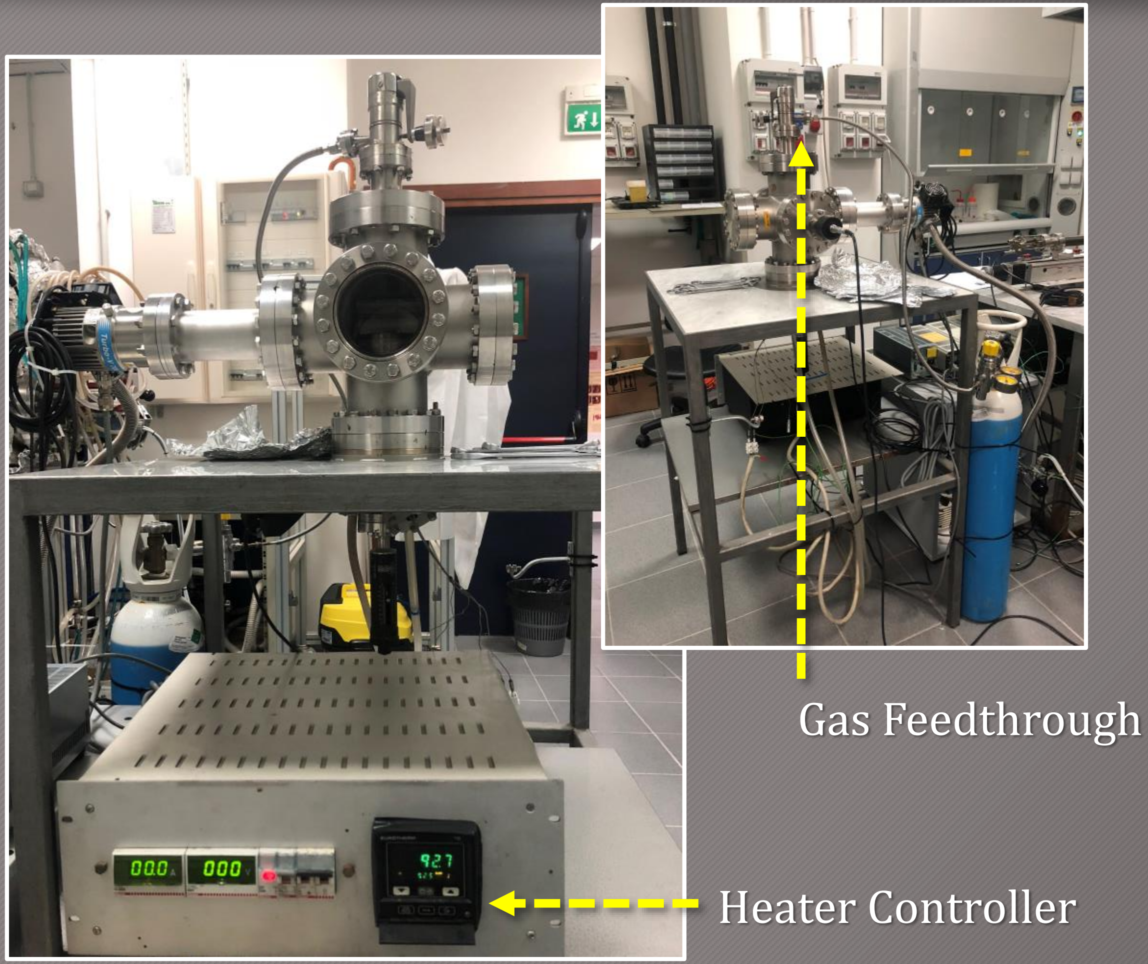
Optical Coatings – Characterization Facility

Scanning Probe Microscopes

Scanning Electron Microscopes

X-Ray Diffractometers

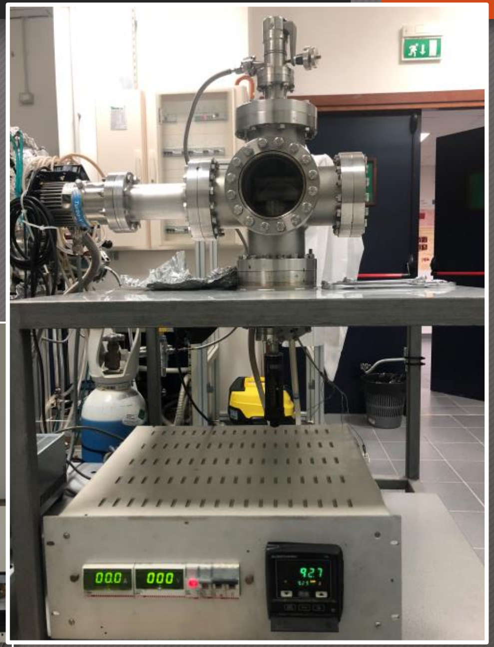
Optical Coatings – Characterization Facility



Recent addition: Annealing Facility

- Adjustable annealing temperature from room temperature up to 900°C;
- PID Feedback controlled power supply heater – with stability of 0.05°C;
- Adjustable annealing pressure from room pressure down to 3×10^{-4} mBar;
- Controllable annealing atmosphere.

Optical Coatings – Characterization Facility



Main Tasks: Contribute to structural and morphological characterization of coatings, before and after heat treatment.

Collaborations

Besides structural and morphological analysis, our group collaborates with:

Università di Roma
Tor Vergata

CENTRO FERMI
MUSEO STORICO DELLA FISICA E CENTRO STUDI E RICERCHE ENRICO FERMI

UNIVERSITÀ DEGLI STUDI DI PERUGIA

UNIVERSITÀ DEGLI STUDI DI GENOVA

1506 UNIVERSITÀ DEGLI STUDI DI URBINO CARLO BO

Lyon 1

..for optical characterization and mechanical loss measurements.

Results and Future Plans

Oct.-Nov.-Dic. 2017	Jen.-Feb.-Mar. 2018	Apr.-May-June 2018	July-Aug.-Sept. 2018	Oct.-Nov.-Dic. 2018	Jen.-Feb.-Mar. 2019	Apr.-May-June 2019	July-Aug.-Sept. 2019	Oct.-Nov.-Dic. 2019

Setting-up of the coater
Production of test samples:
SiO_2 and SiO_2/TiO_2 multilayers

Production of TiO_2/ZrO_2 multilayers

Coater Off

Production of SiO_2/TiO_2 and SiO_2/Al_2O_2 multilayers

Characterization of test sample to feedback production activity

Characterization of TiO_2/ZrO_2 before heat treatment

Characterization of TiO_2/ZrO_2 after heat treatment

Characterization of new samples

Preliminary Tests

Oct.-Nov.-Dic. 2017	Jen.-Feb.-Mar. 2018	Apr.-May-June 2018	July-Aug.-Sept. 2018	Oct.-Nov.-Dic. 2018	Jen.-Feb.-Mar. 2019	Apr.-May-June 2019	July-Aug.-Sept. 2019	Oct.-Nov.-Dic. 2019

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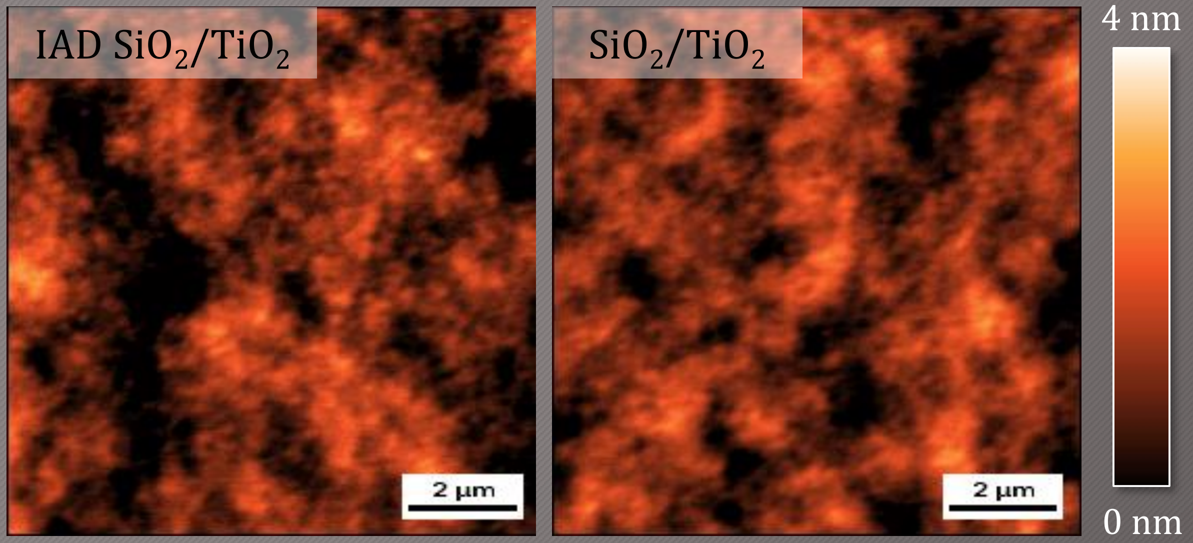

AFM Topography.
RMS roughness is 0.6nm

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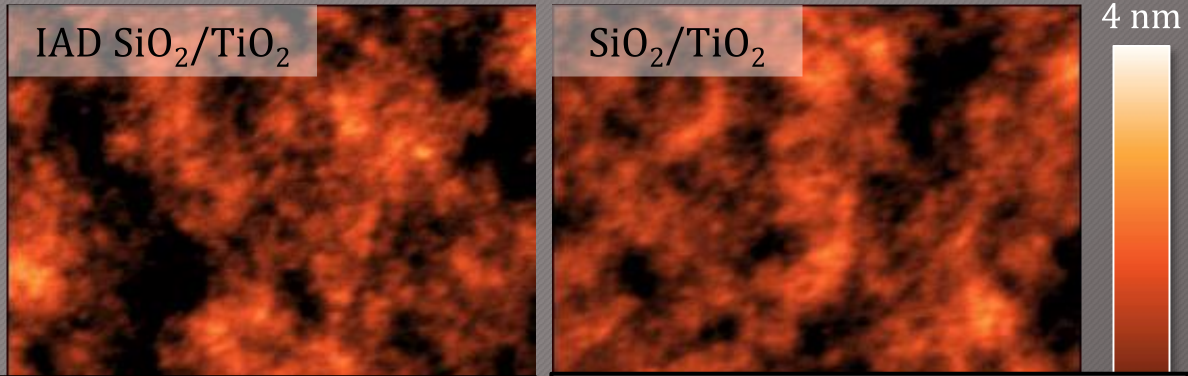

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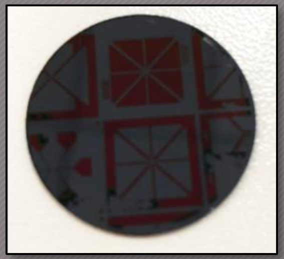
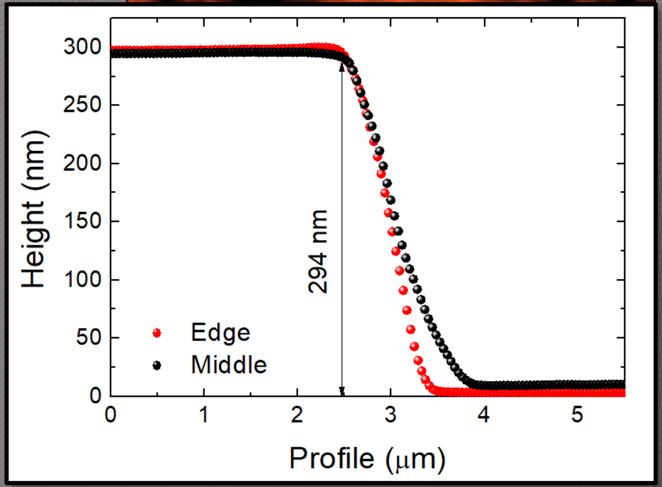
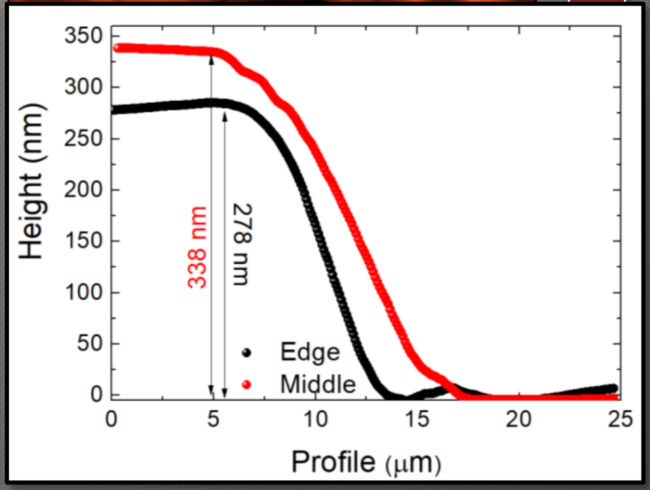






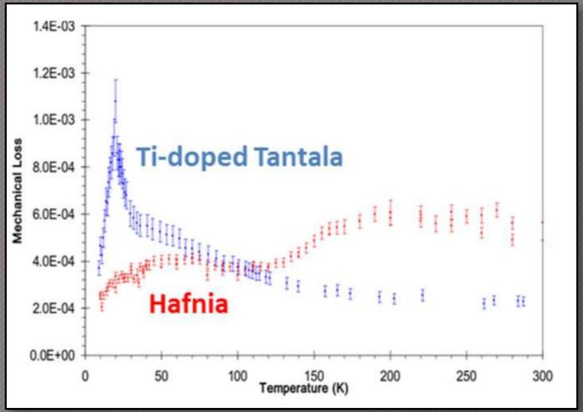
Deposition Uniformity Evaluation.

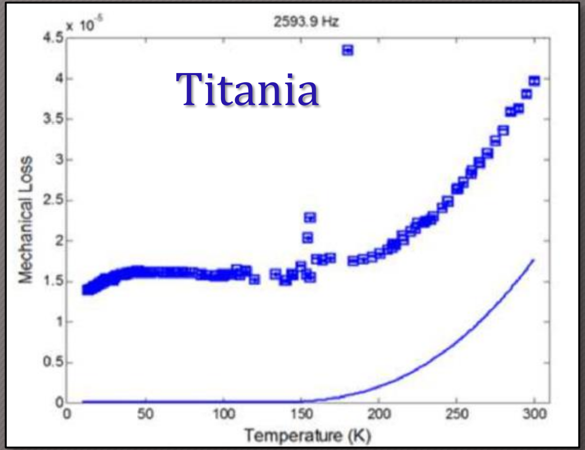
Preliminary Results: Fabrication

Deposition of 200-nm single materials

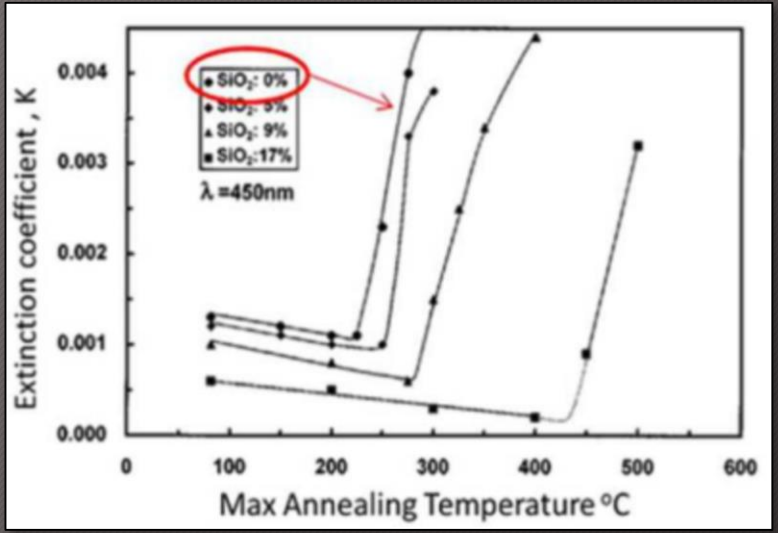
Material	Plasma	Total Thickness (nm)
SiO_2	Yes	200
TiO_2	Yes	200
ZrO_2	Yes	200
HfO_2	Yes	200

What do we know about these materials?





TiO_2 and HfO_2 do not show cryo-peak in mechanical losses BUT crystalize upon annealing at low temperature (200-300°C) with consequent blow-up of optical properties!



Preliminary Results: Fabrication

Deposition of multilayers

Sample	Plasma	n. Layers	Single layer thickness (nm)	Total Thickness (nm)
TiO_2/ZrO_2	Yes	8	25.6	205
TiO_2/ZrO_2	Yes	16	12.6	202
TiO_2/ZrO_2	Yes	32	6.4	205
TiO_2/ZrO_2	Yes	64	3.2	205
TiO_2/ZrO_2	Yes	122	1.6	195
TiO_2/ZrO_2	Yes	128	1.6	205

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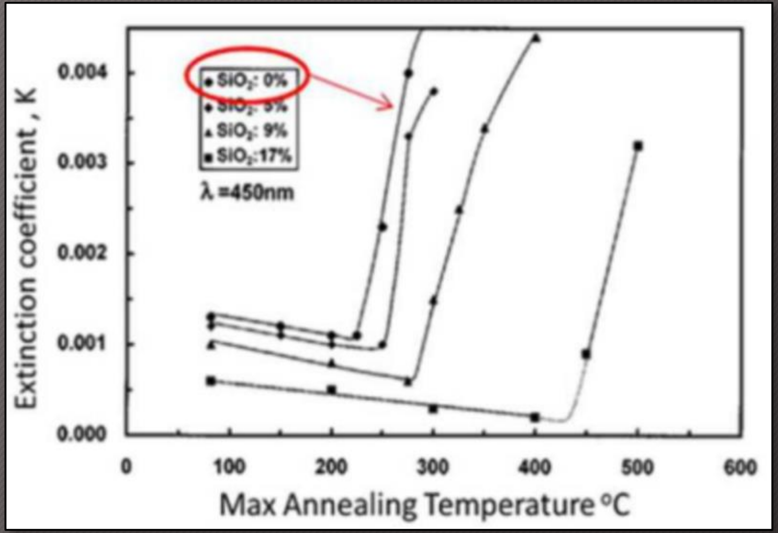
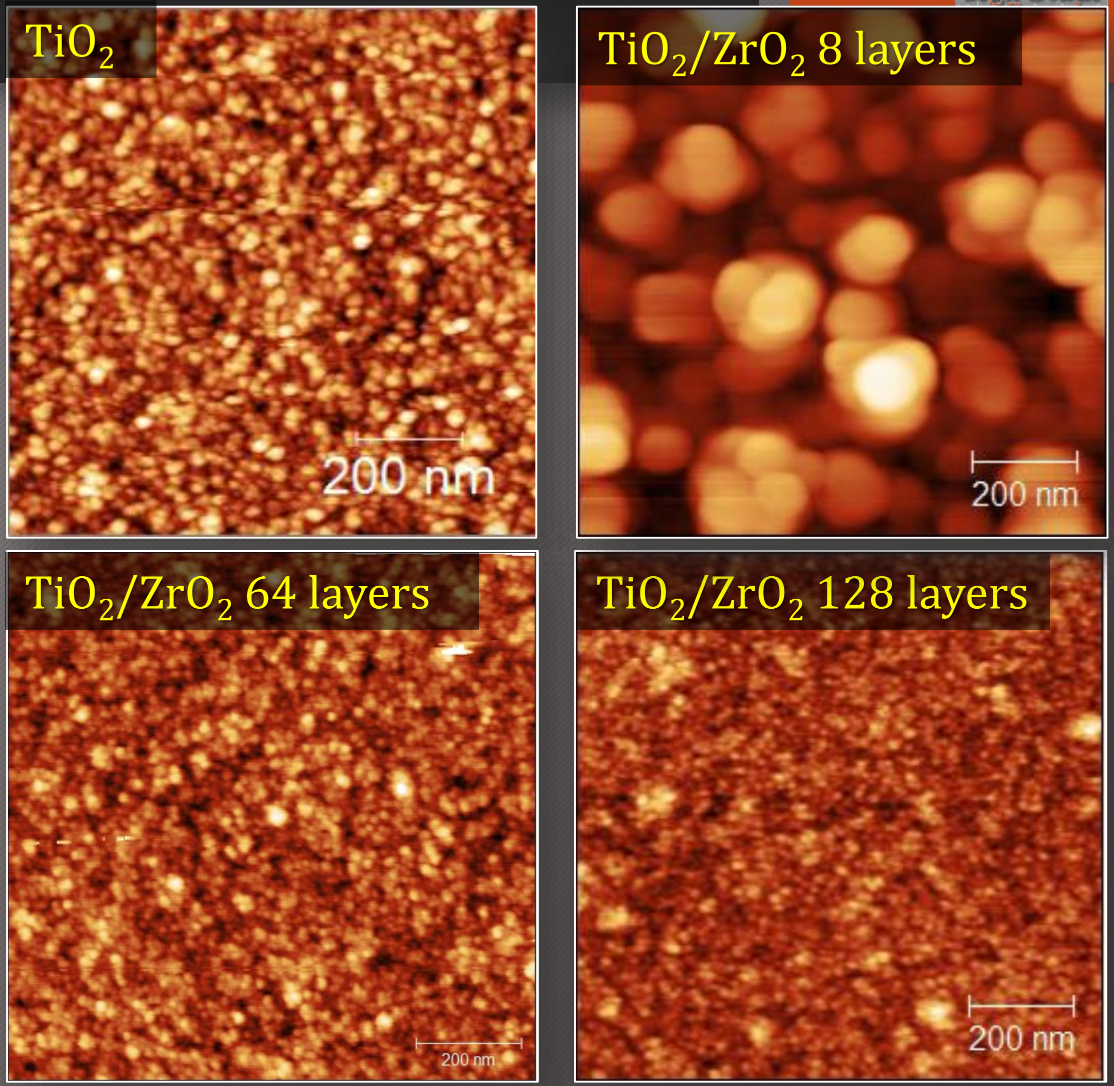


Preliminary Results - Characterization

Before Annealing – as deposited:

- We measured the AFM topography to qualify the morphology and quantify surface roughness and uniformity of top-most TiO_2 surface



Preliminary Results - Characterization

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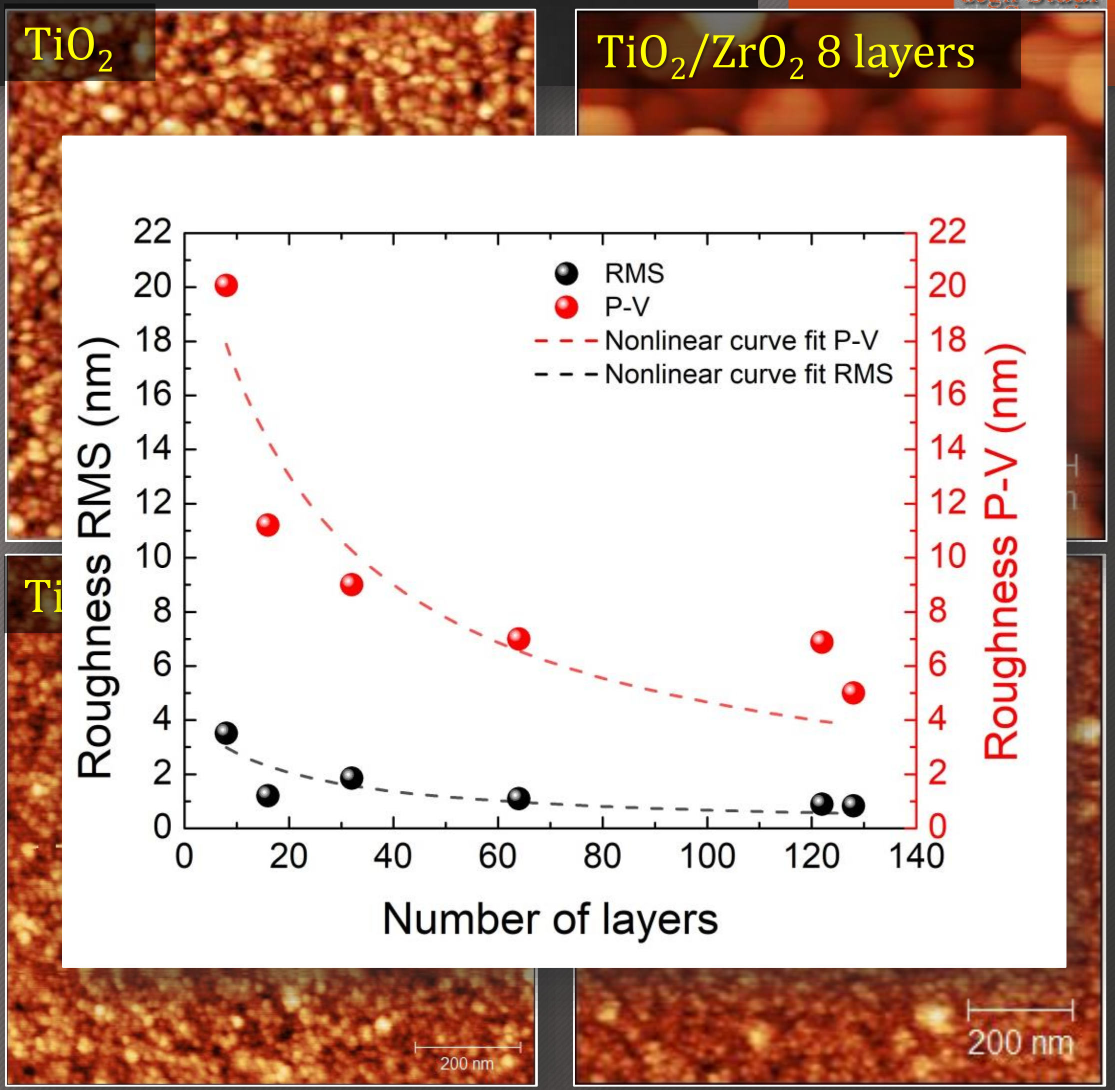
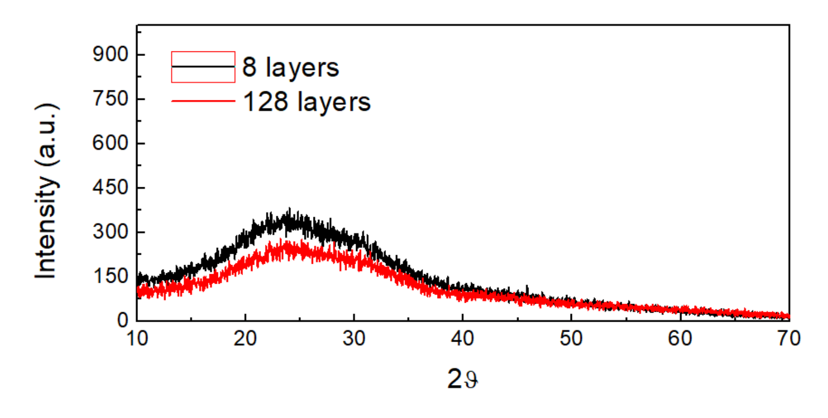
- We found a reduction of surface roughness as the layer thickness decreases (increased segmentation);

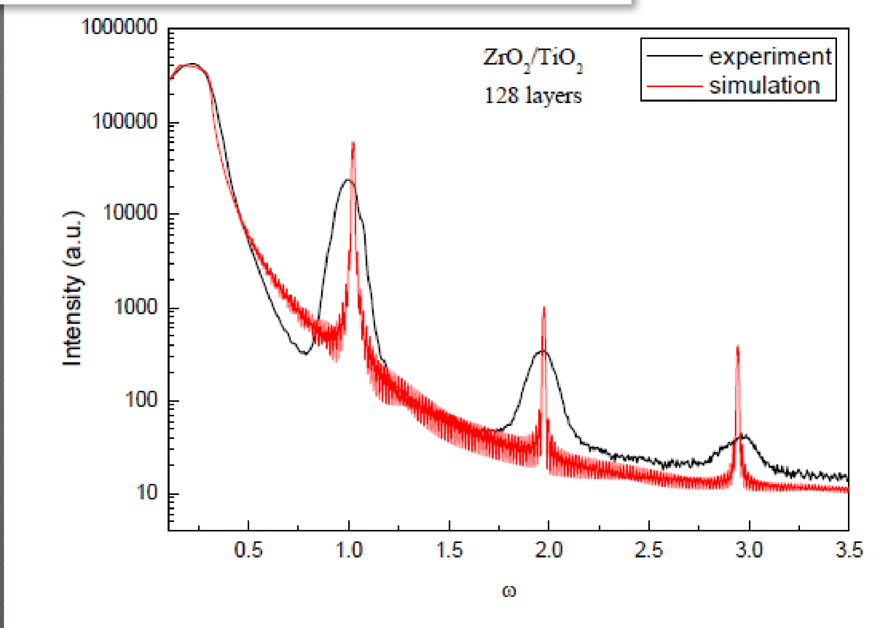


Preliminary Results - Characterization

Before Annealing – as deposited:

- We measured the AFM topography to qualify the morphology and quantify surface roughness and uniformity of top-most TiO_2 surface
- We found a reduction of surface roughness as the layer thickness decreases (increased segmentation);
- We performed X-Ray diffraction and checked the amorphic nature of our samples;
- We performed SAXS on multilayered samples and confirmed the presence of segmentation;

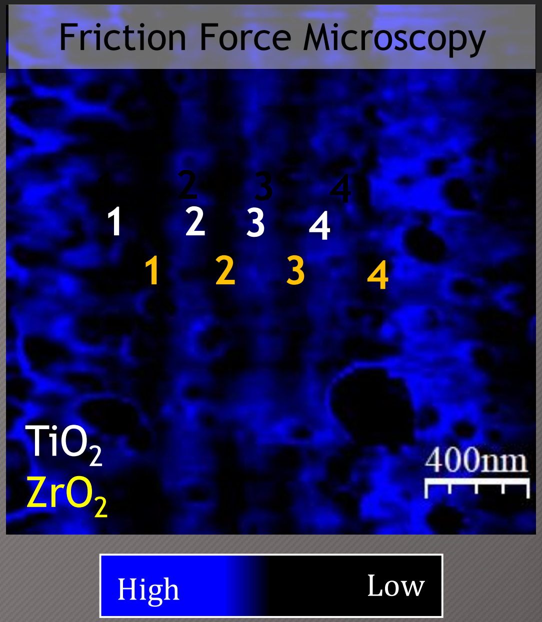




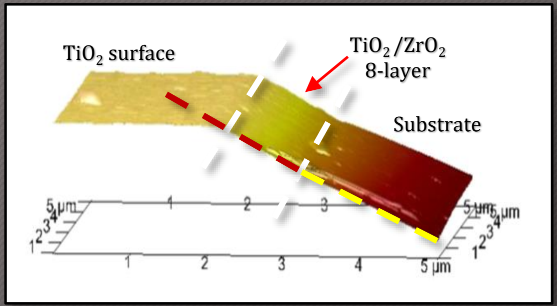
Preliminary Results - Characterization

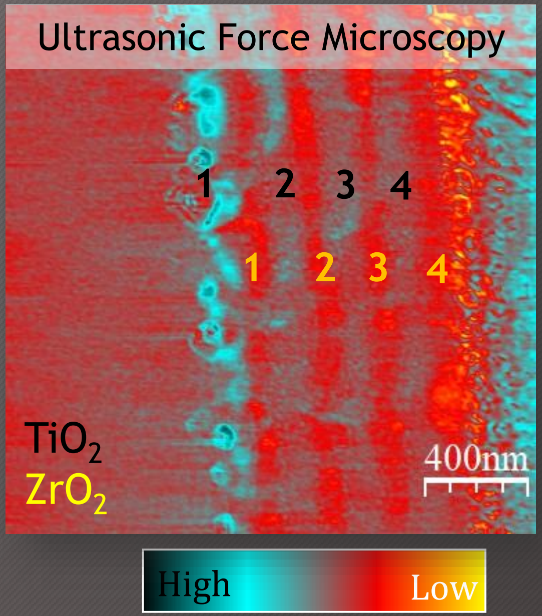
Before Annealing – as deposited:

- We measured the AFM topography to qualify the morphology and quantify surface roughness and uniformity of top-most TiO_2 surface
- We found a reduction of surface roughness as the layer thickness decreases (increased segmentation);
- We performed X-Ray diffraction and checked the amorphous nature of our samples;
- We performed SAXS on multilayered samples and confirmed the presence of segmentation;
- We cross-sectioned the multilayered samples and imaged the layers by profiting of advances scanning probe techniques.



Colour contrast is representative of different friction coefficient.



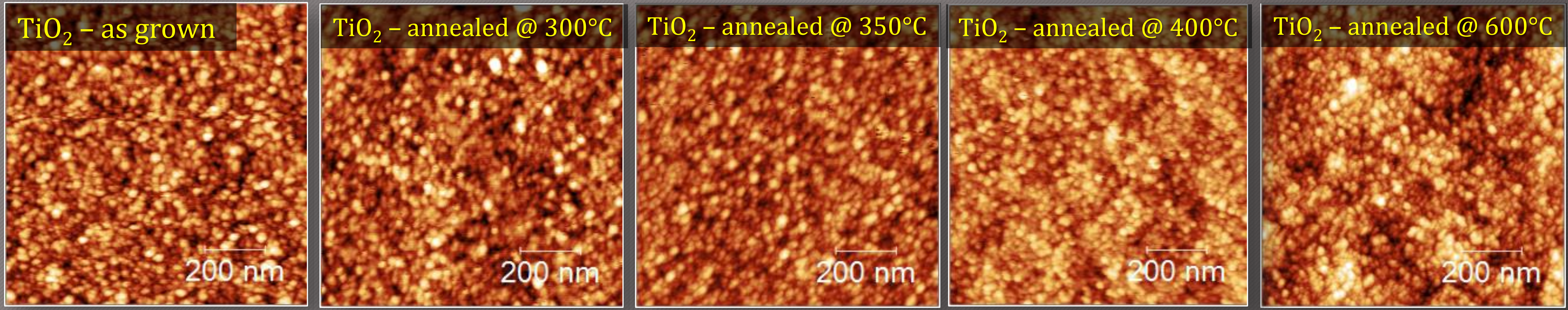


Colour contrast is representative of different stiffness.

TiO$_2$ – 200nm – single material

Preliminary Results - Characterization

After Annealing (12 hours – 10^{-4} mBar):



- Granular morphology is preserved after heat treatment.

TiO$_2$ – 200nm – single material

Preliminary Results - Characterization

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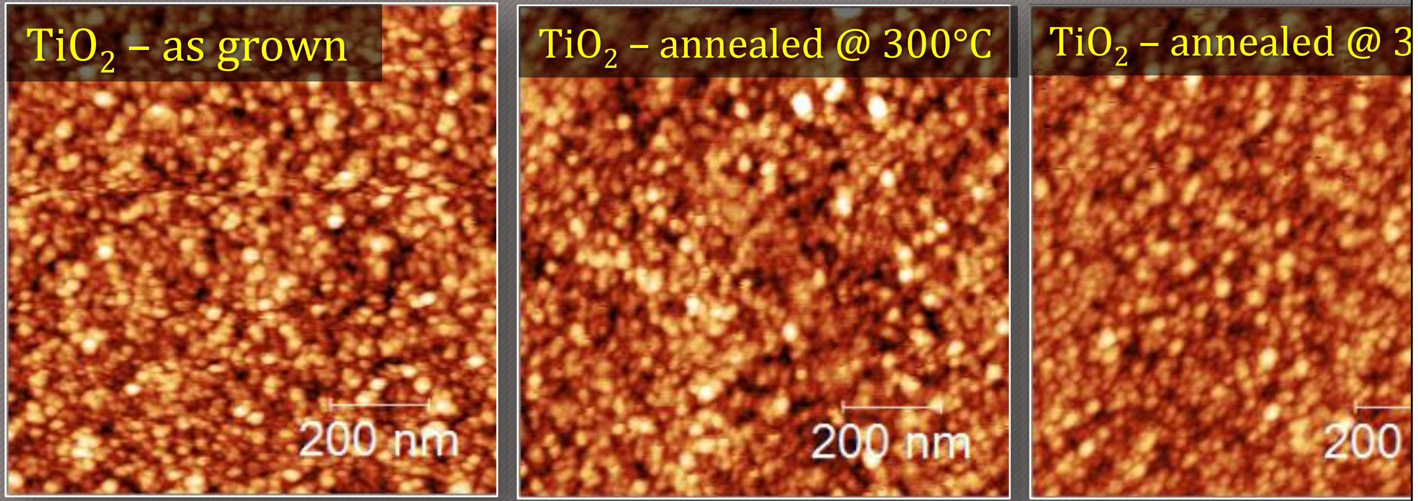
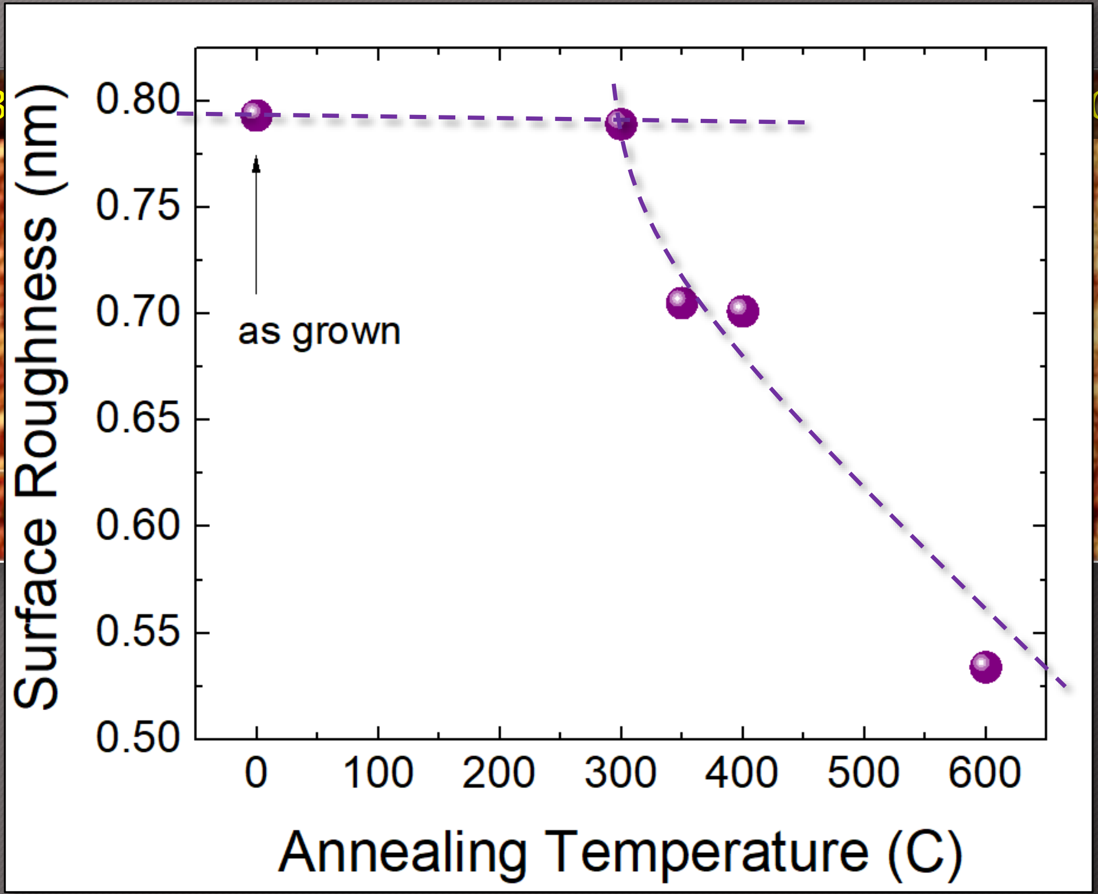
After Annealing (12 hours – 10^{-4} mBar):



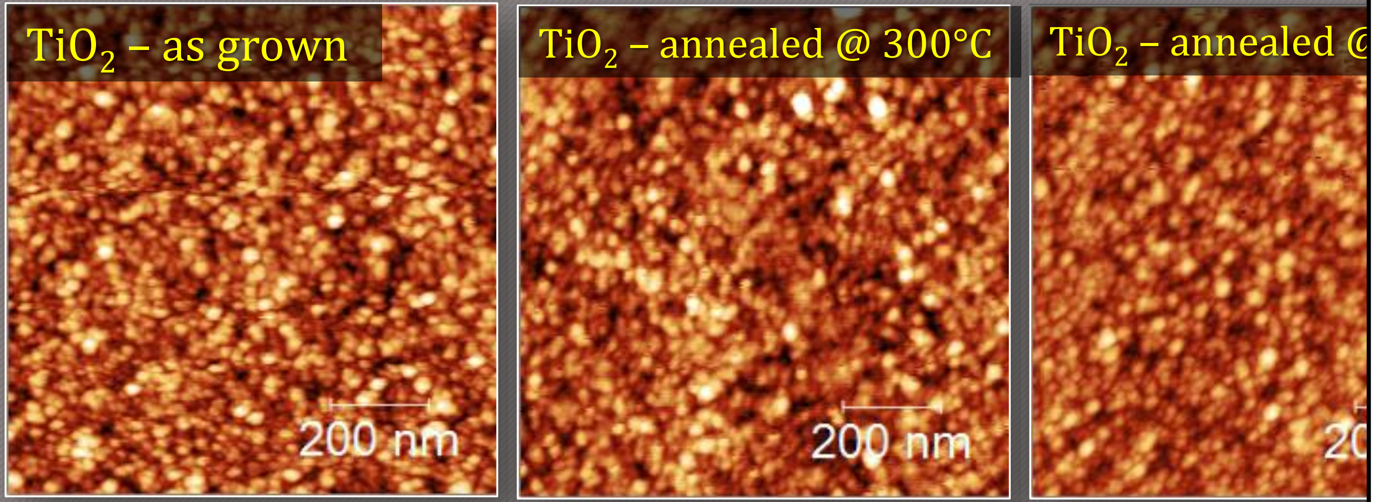


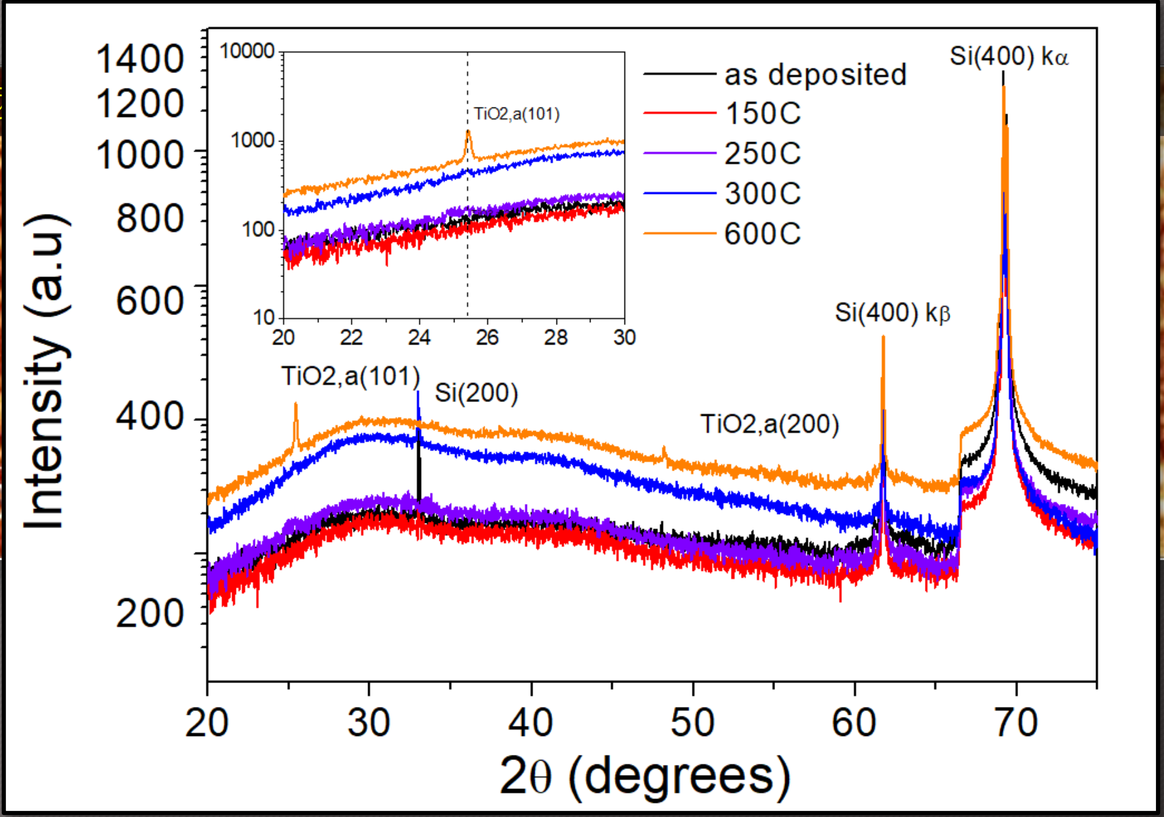
- Granular morphology is preserved after heat treatment.
- A decreasing trend of surface roughness is observed.

TiO$_2$ – 200nm – single material

Preliminary Results - Characterization

After Annealing (12 hours – 10^{-4} mBar):

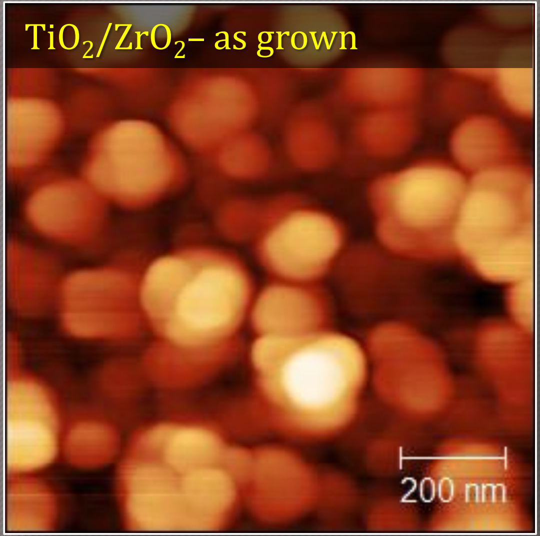


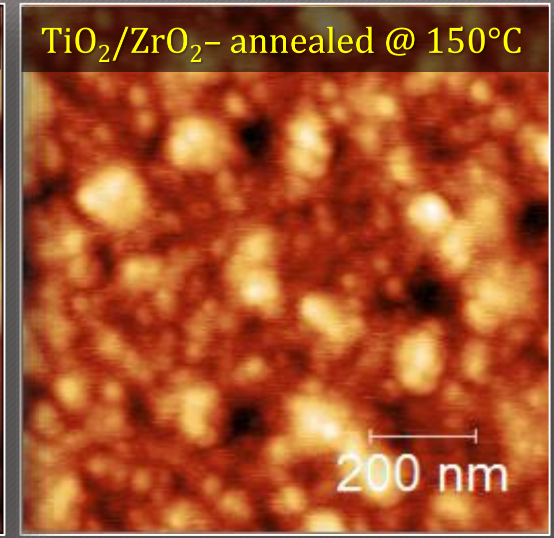


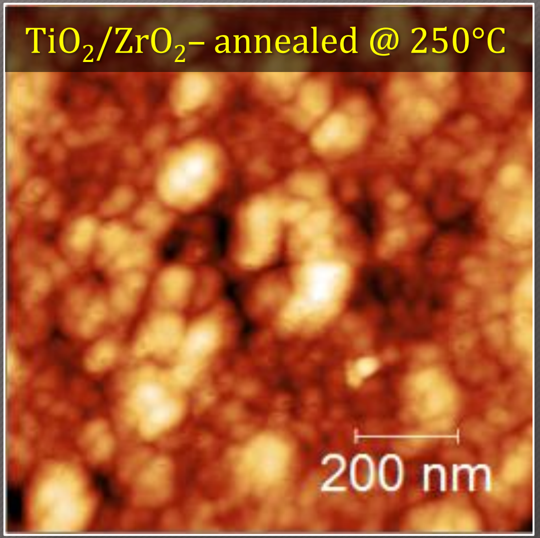
- Granular morphology is preserved after heat treatment.
- A decreasing trend of surface roughness is observed.
- XRD demonstrate that T-crystallization is higher than 300C, given the used annealing conditions.

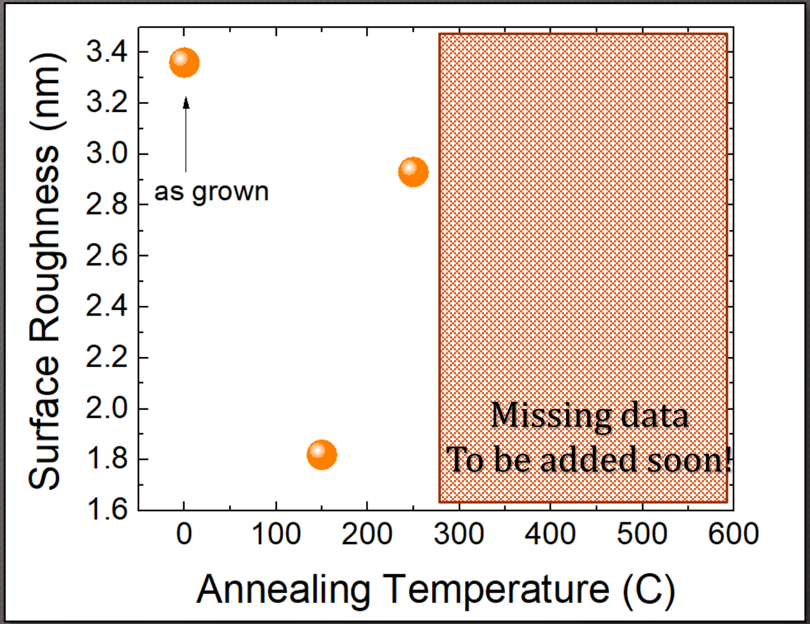
Preliminary Results - Characterization

Annealing at higher temperatures, AFM and XRD analyses on annealed TiO_2/ZrO_2 are on-going!









Results and Future Plans

Oct.-Nov.-Dic. 2017 | Jen.-Feb.-Mar. 2018 | Apr.-May-June 2018 | July-Aug.-Sept. 2018 | Oct.-Nov.-Dic. 2018 | Jen.-Feb.-Mar. 2019 | Apr.-May-June 2019 | July-Aug.-Sept. 2019 | Oct.-Nov.-Dic. 2019

Setting-up of the coater
Production of test samples:
SiO_2 and SiO_2/TiO_2 multilayers

Production of TiO_2/ZrO_2 multilayers

Coater Off

Production of SiO_2/TiO_2 and SiO_2/Al_2O_2 multilayers

Characterization of test sample to feedback production activity

Characterization of TiO_2/ZrO_2 before heat treatment

Characterization of TiO_2/ZrO_2 after heat treatment

Characterization of new samples

Thank you for the attention.