Monitoring in real time the annealing of optical coatings

Michele Magnozzi
OptMatLab, Dipartimento di Fisica, Università di Genova, Italy

michele.magnozzi@unige.it

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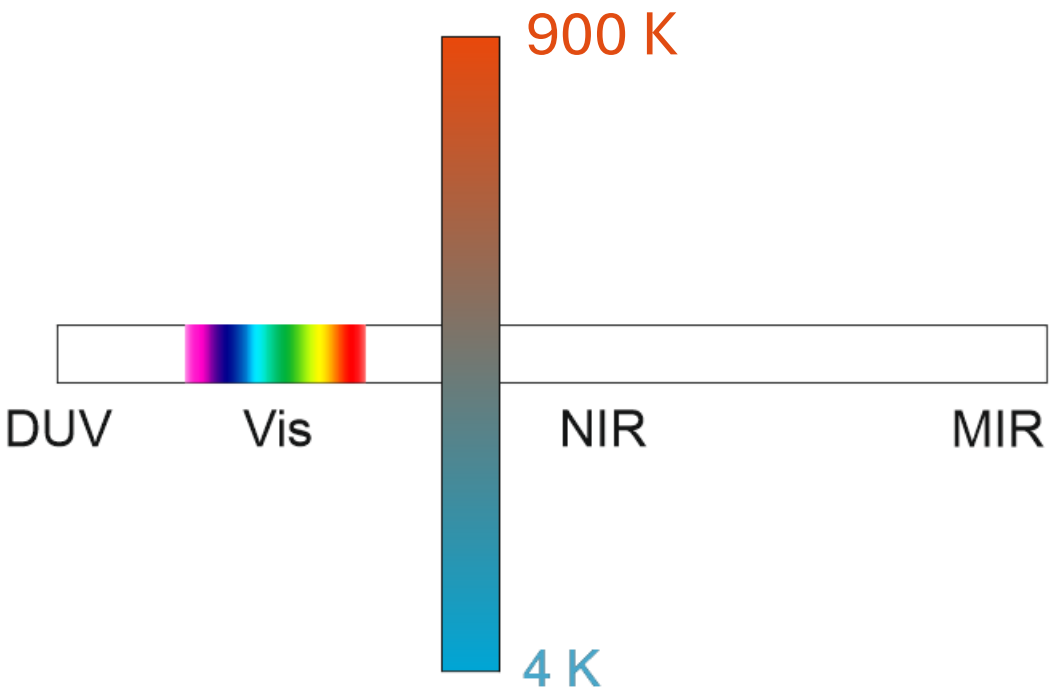
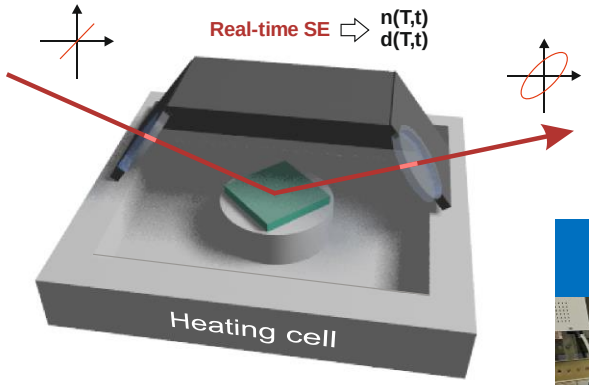


Optical properties of materials
in wide spectral and temperature ranges

High temperature, in air



High temperature, in vacuo

Low temperature, in vacuo

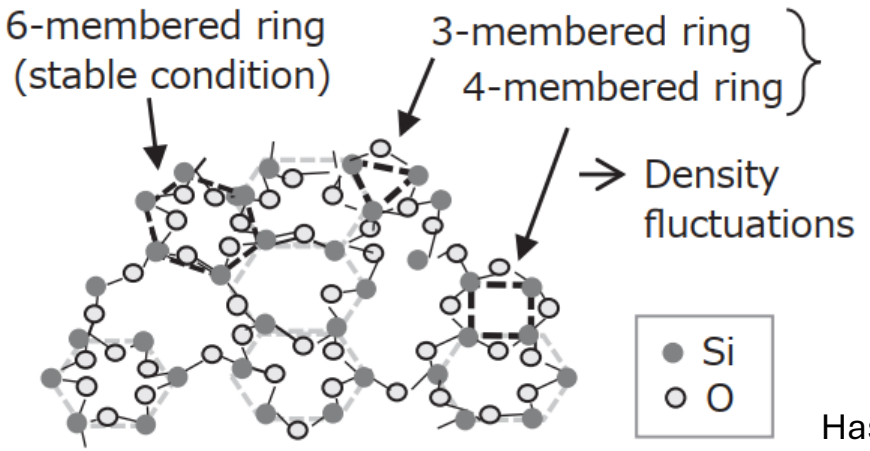
- **state-of-the-art suite of ellipsometers**
- **home-made** as well as **commercial add-ons to vary temperature & atmosphere**
- **in situ & in operando capabilities**

M. Magnozzi et al., *Appl. Surf. Sci* **421** 651 (2017)
M. Magnozzi et al., *J. Phys. D* 475105 (2023)

The need for thermal annealing in IBS-produced coatings

Films produced by Ion Beam Sputtering (IBS) suffer from **compressive stress**, meaning that their structure is not in a state of equilibrium.

As a consequence, the performance of **as-deposited IBS-produced coatings** is not satisfactory.
This means that mechanical losses and optical absorption are too high.



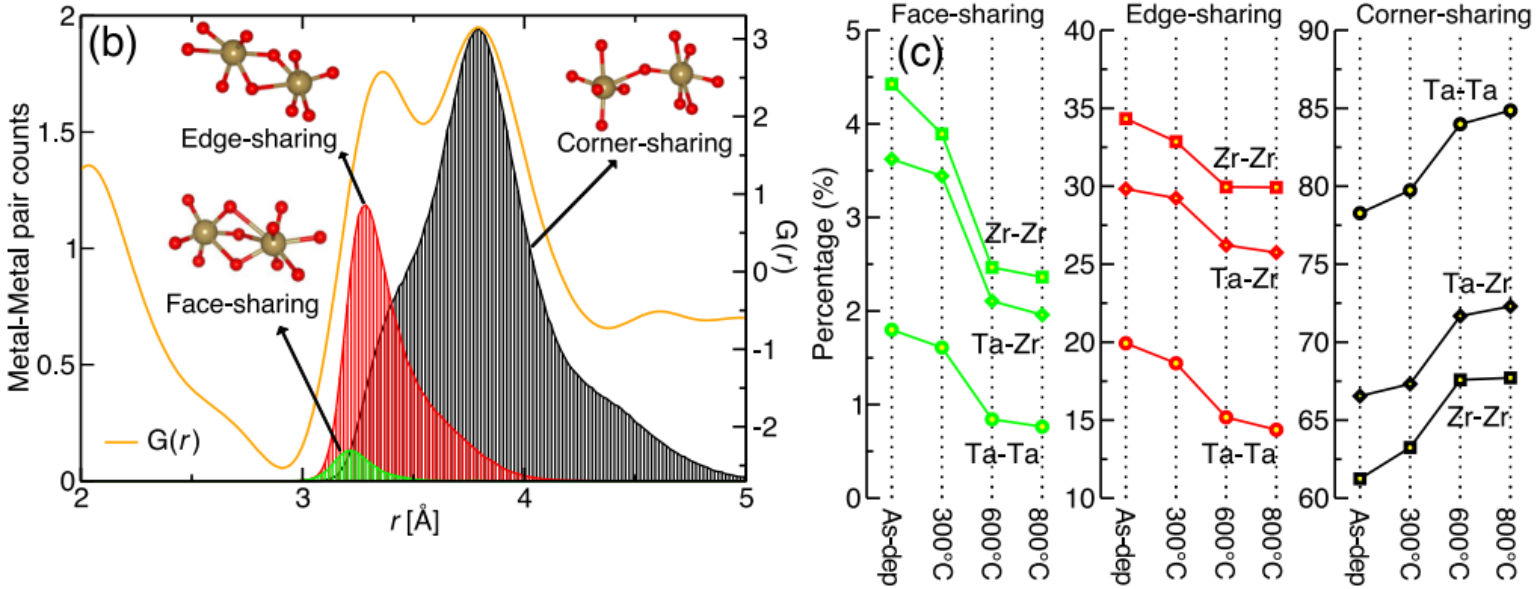
Annealing modifies the microscopic structure of the coating materials

Hasegawa et al., *Sei Technol. Rev*. 86, 19 (2018)

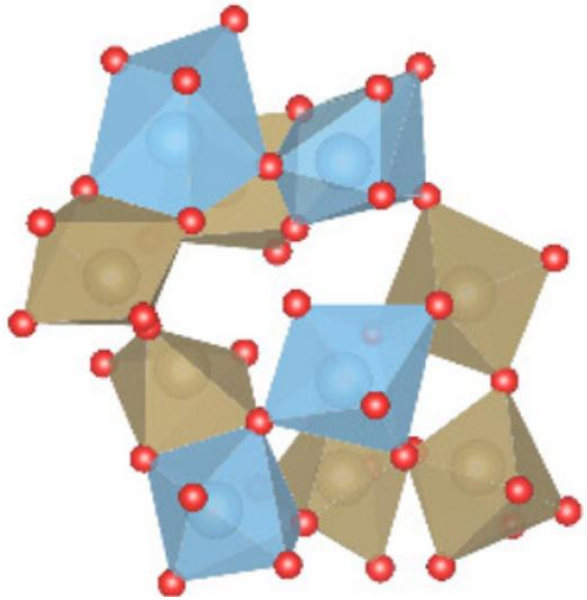
Case in point:
The mechanical losses of silica produced by IBS can be up to *four orders of magnitude higher* than those of fused silica. [M. Granata et al., *Phys. Rev. Mat*. 2 053607 (2018)].

Effects of thermal annealing on mirror coatings

Transition-metal oxides, too, show **annealing-induced microscopic variations** in their atomic structures.



Prasai et al., *Phys. Rev. Lett*. 123, 045501 (2019)



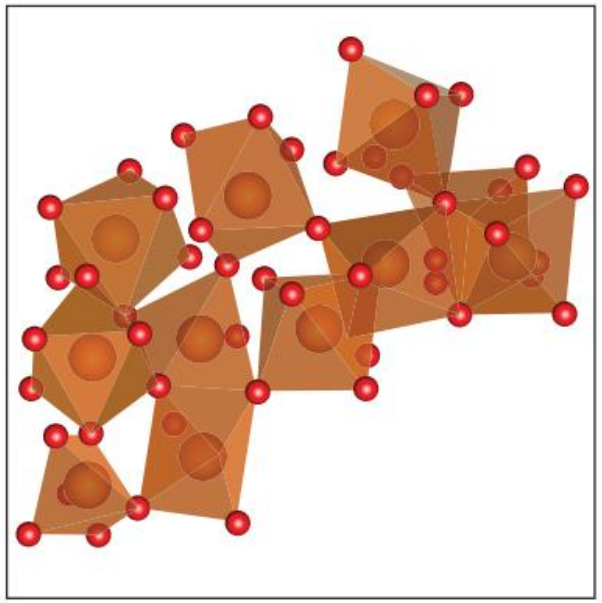
Damart et al., *J. Appl. Phys.*119, 175106 (2016)

Variations in the face-sharing / edge-sharing / corner-sharing ratios
Possibly related to a variation in the oxygen content in the coating following annealing
(Fazio et al., *Opt. Expr*. 10, 1687 (2020))

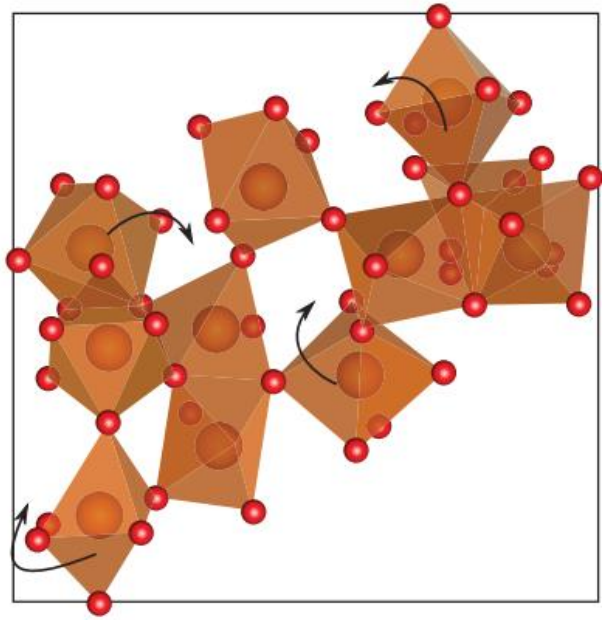
Effects of thermal annealing on mirror coatings

Annealing-induced variations can (and typically do) have a counterpart in the **macroscopic properties** of the coatings, such as **density** and **refractive index**.

Dense (as-deposited)



Less dense (annealed)



Adapted from E. Coillet, PhD Thesis, Université de Lyon 1 (2017)

Thermal annealing requisites

A thermal annealing is defined by a **few key parameters**, such as:

- heating rate
- maximum temperature
- time spent at the maximum temperature (a.k.a. soaking, plateau)
- cooling rate

A good set of parameters is one which brings the coatings in a state where their properties (mechanical, optical) are suitable for GWD applications.
-> relaxation typically requires high temperatures

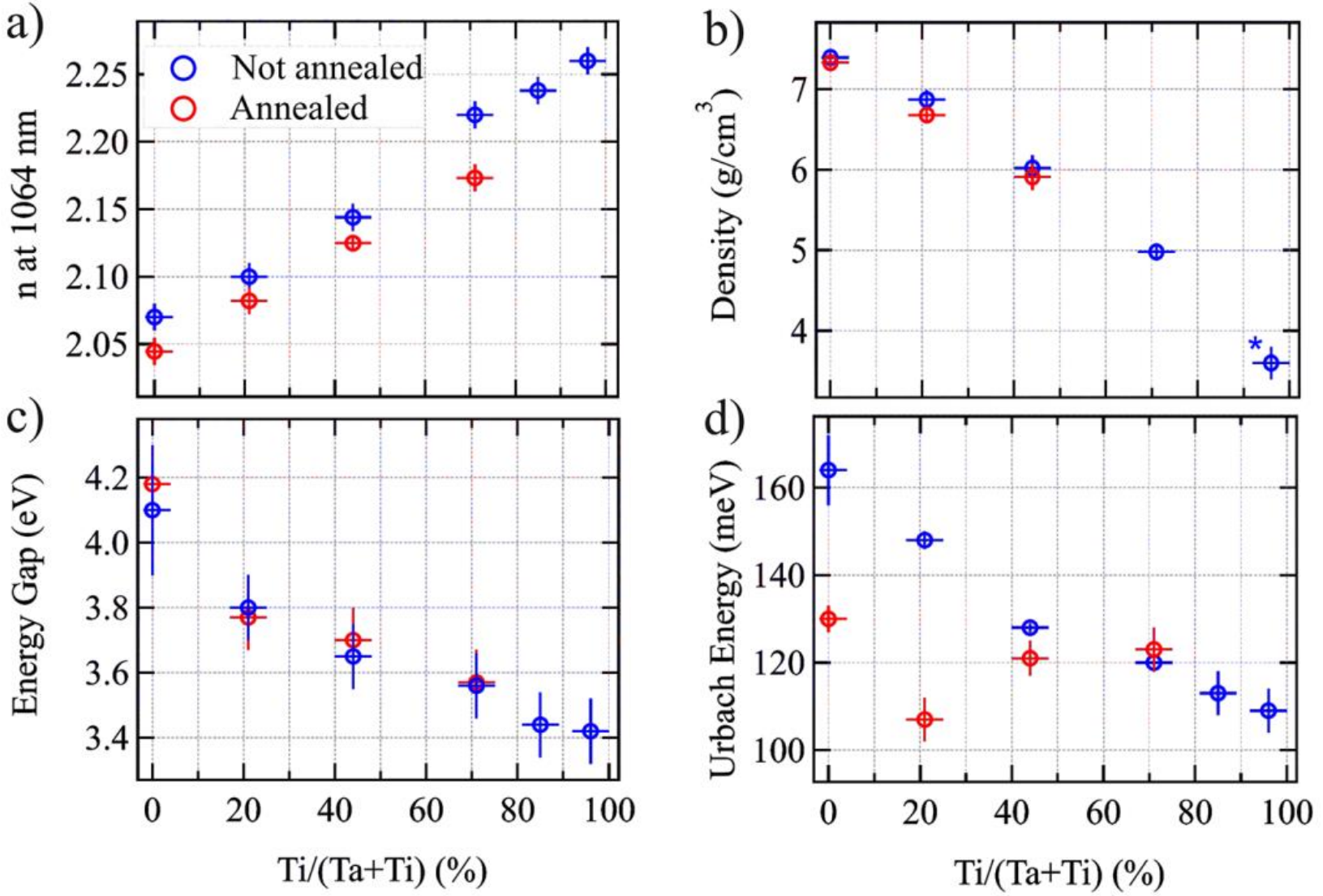
Fundamental requirement: amorphous coatings should remain amorphous – crystallization must be avoided (partial crystallization still to be properly understood..)
-> crystallization occurs at high temperatures

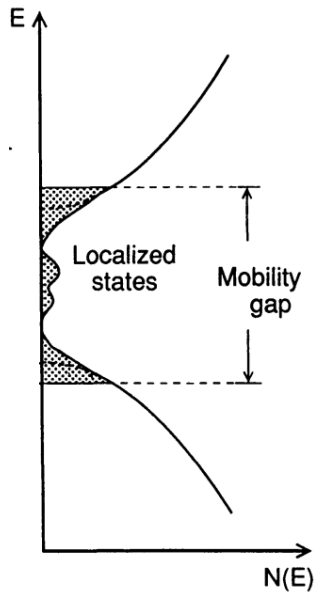
Where is the optimum between the two requirements?

How are the annealing parameters chosen?

Example of ex-situ analysis on titania-tantala

Amato A., Magnozzi M. et al., *ACS Appl. Opt. Mater.* 1, 395 (2023)





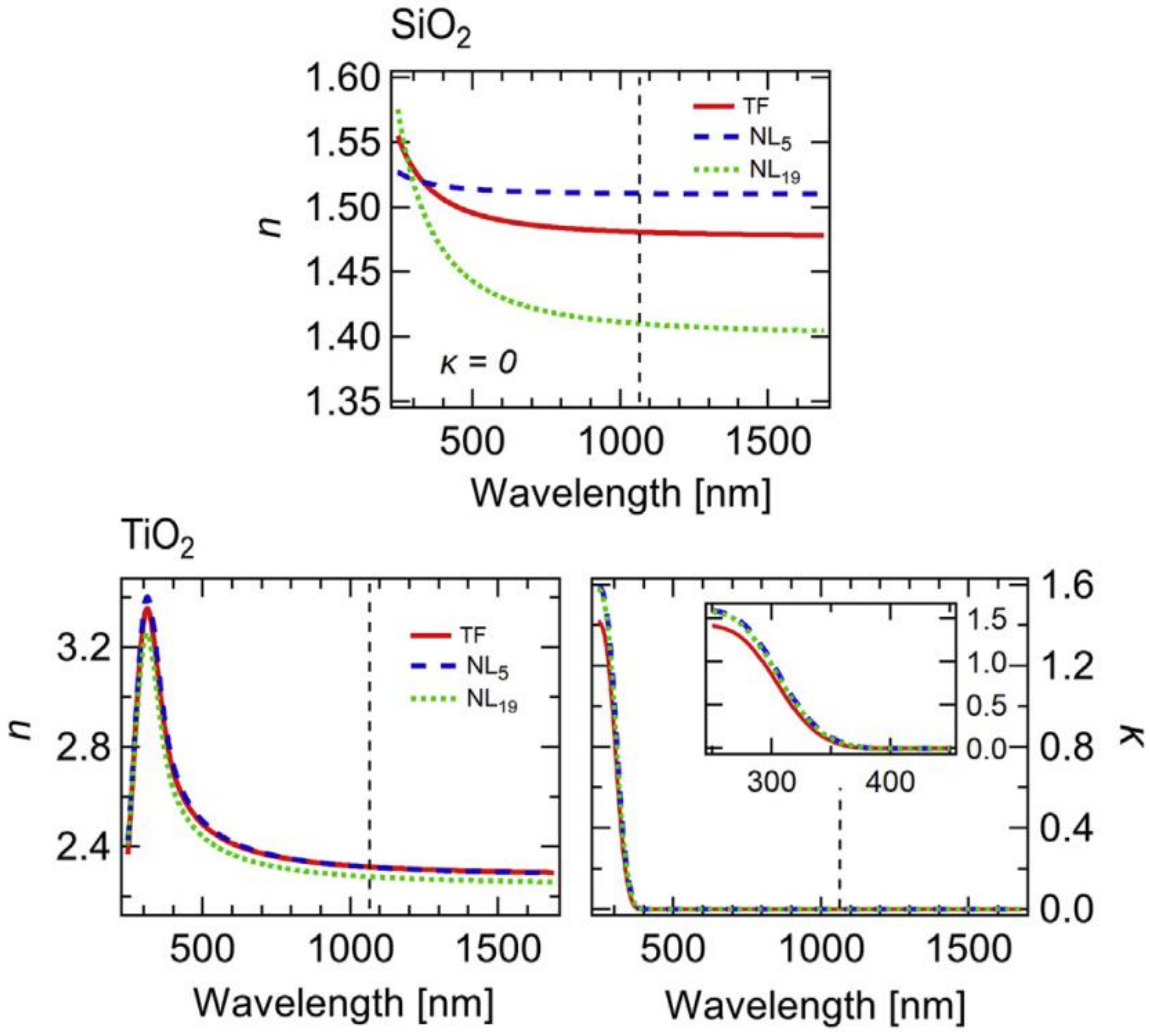
Urbach energy is related to the degree of 'disorder' within the material.

Lowest value of Urbach energy

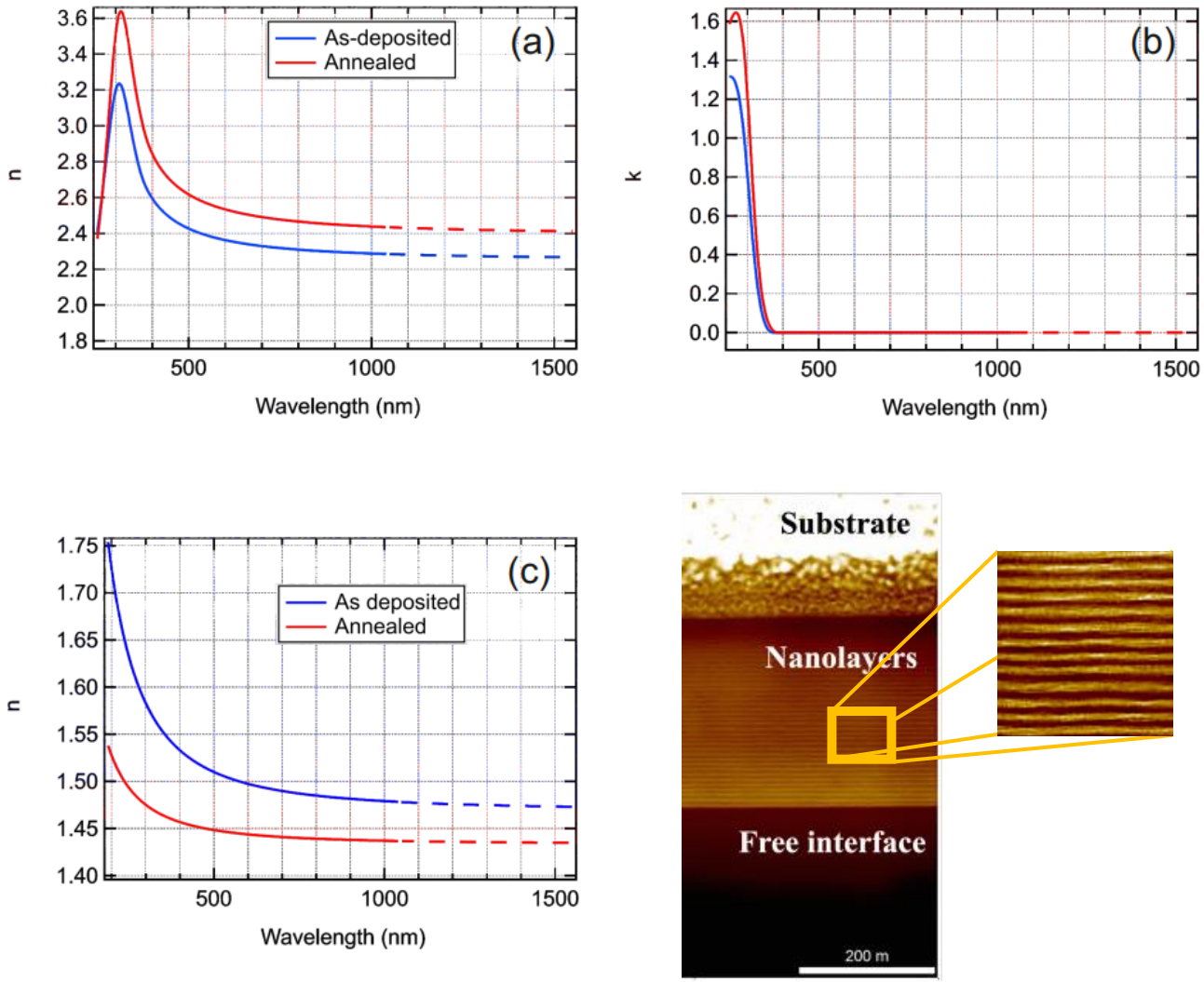
better performing coating

Example of ex-situ analysis on multi-nanolayers

Magnozzi M. et al., *Opt. Mater.* **75**, 94 (2018)



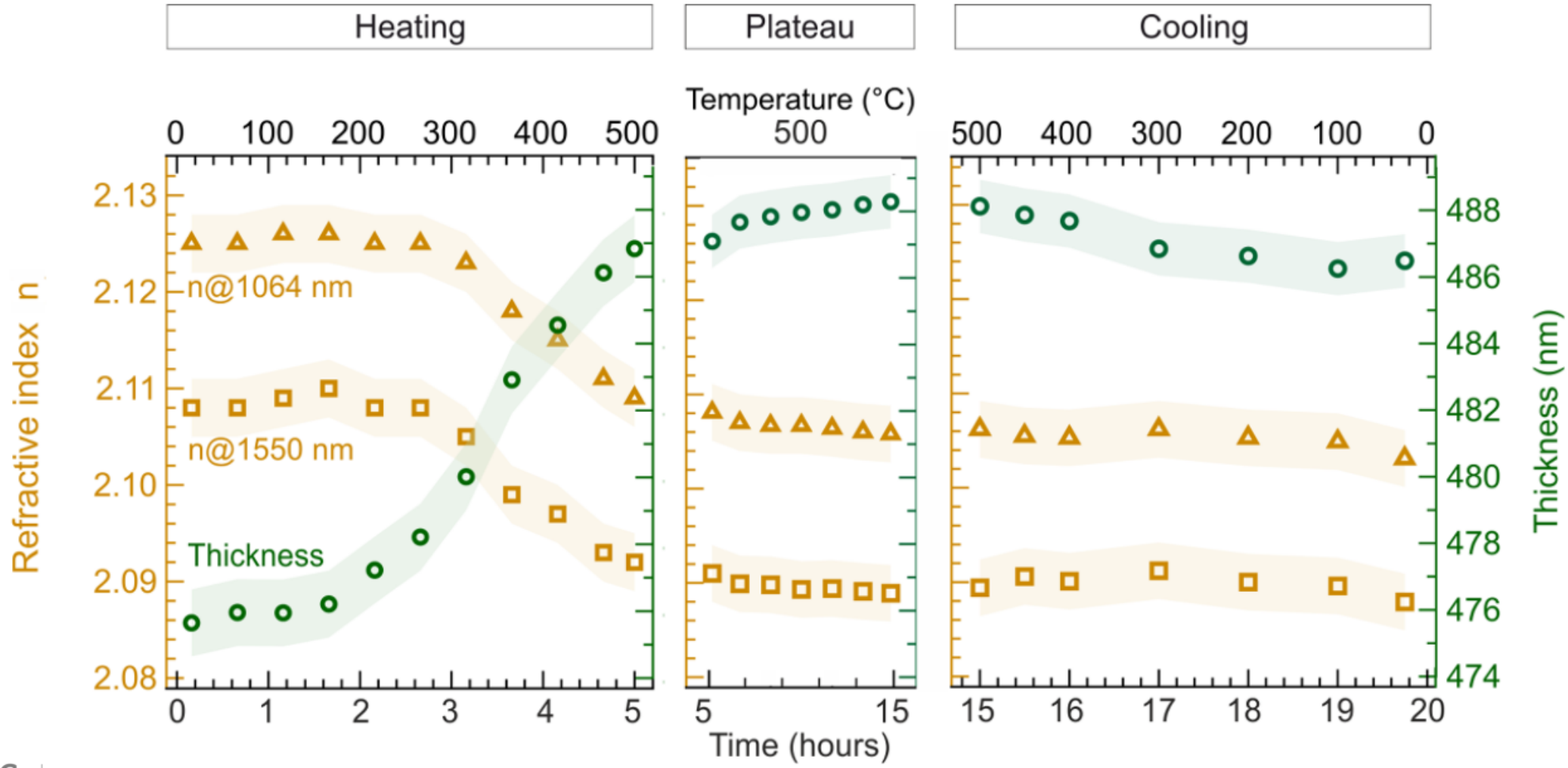
O. Durante et al., *Opt. Mater. accepted manunscript* (2024)



Tracking macroscopic optical properties of coatings during annealing

The composition of the Ti:Ta_2O_5 coating and the annealing protocol are **the same employed in current GWD mirrors**.

S. Colace *et al.*, *Class. Quant. Grav.* **41** 175016 (2024)



Key takeaways from the in-situ annealing of titania-tantala

The data indicate that:

- **the largest variations occur during the heating ramp -** not during the plateau
- the onset of the variations occur at temperatures slightly higher than the deposition temperature
- **the evolution** of the considered coating properties **is still ongoing** after 10 hours at the maximum temperature

Implications for the annealing protocol of titania-tantala:

- increasing the soaking time could bring the coating in a different state – probably one having better performances for GWD mirrors
- **deposition parameters** (incl. substrate temperature) **and annealing should be optimized together** when searching for the best performing coatings.

S. Colace *et al.*, *Class. Quant. Grav.* **41** 175016 (2024)

Summary and perspectives

Monitoring in-situ & in real time the annealing of optical coatings can provide **unique insights** into the properties, initial state and possible evolution of the coatings for GWD mirrors.

Ex-situ and in-situ characterization approaches are **complementary and both necessary**.

Each choice of coating composition (Ti:GeO_2, Ti:SiO_2, SiN, etc.) requires a **dedicated optimization** of the annealing parameters.

In-situ, real-time diagnostics tools can facilitate the identification of the best annealing parameters and therefore **facilitate the search for the best performing coatings.**

Ongoing work at OptMatLab:
apply the in-situ monitoring tools to study other high-index candidate materials.