Temperature-dependent optical characterization of materials for mirrors: from cryogenic to annealing temperatures

Michele Magnozzi on behalf of the Genova RU

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Ministero dell'Università e della Ricerca

Italiadomani PIANO NAZIONALE DI RIPRESA E RESILIENZA

The role of temperature in GWD mirrors

High Temperatures
during post-deposition annealing

- How does annealing improves the performance of GWD mirrors?
- What are the optimal annealing parameters to obtain the best performing GWD mirror?

Low Temperatures
during mirrors operation

- How do the optical properties of materials for GWD mirrors change at cryogenic temperatures?
- How can we measure those properties?
- Do we need to take into account spurious effects, such as ice growth?

Motivation

High Temperatures

Optimization of the post-deposition annealing parameters has been so far based on ex-post evaluations, that is, comparing the properties of coatings before and after the annealing. This approach is time-consuming and does not guarantee optimization. How can we improve this?

M. Rezac et al., *Appl. Opt.*, 62, B97 (2023)

Low Temperatures

Unless special measures are taken, ice overlayer is very likely to grow on cold surfaces even in UHV. For example, KAGRA reported issues of icing on the main mirrors.

Hasegawa et a., *Phys. Rev. D* 99, 022003 (2019); Steinlechner et al., *Phys. Rev. Res.* 013008 (2019); Tanioka et al., *Phys. Rev. D* 102, 022009 (2020).

Beside the on-site performance of the GWD mirrors, if ice grows on cold coatings, then any sensitive measurement of their properties during preliminary R&D activities should take into account the presence of ice. How can we do this?

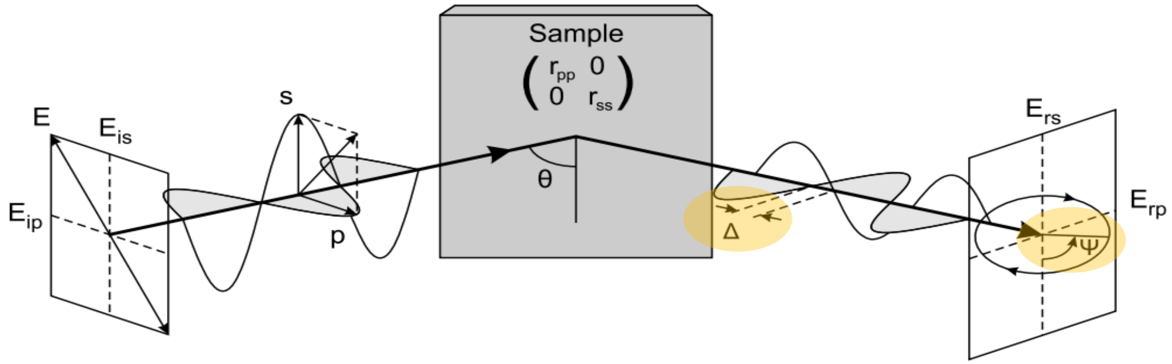
M. Magnozzi et al., *J. Phys. D: Appl. Phys.*, 56, 475105 (2023)

Spectroscopic ellipsometry

$$\frac{\tilde{r}_p}{\tilde{r}_s} = \tan\Psi \exp(i\Delta)$$

$$\psi = \tan^{-1}\left(\frac{|r_p|}{|r_s|}\right)$$

$$\Delta = \delta_{rp} - \delta_{rs}$$



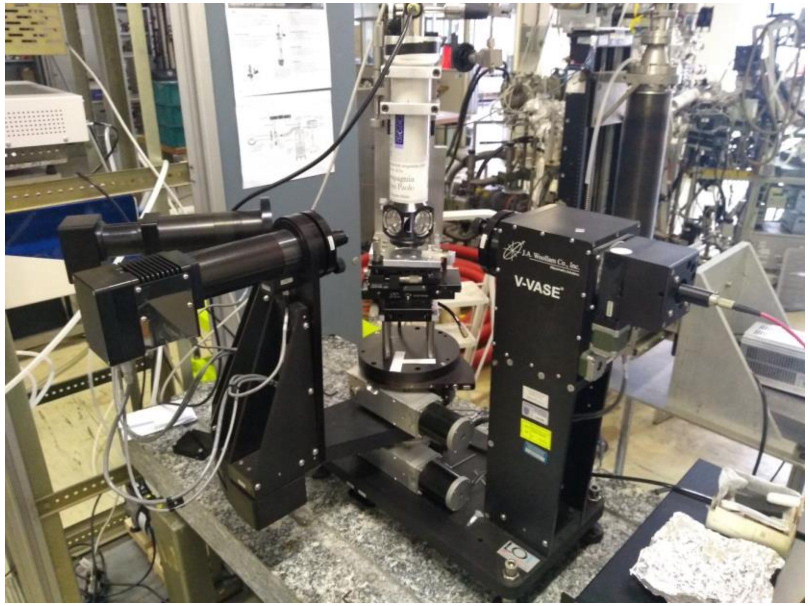
M. Canepa in *Surface Science Techniques*, Springer 2013

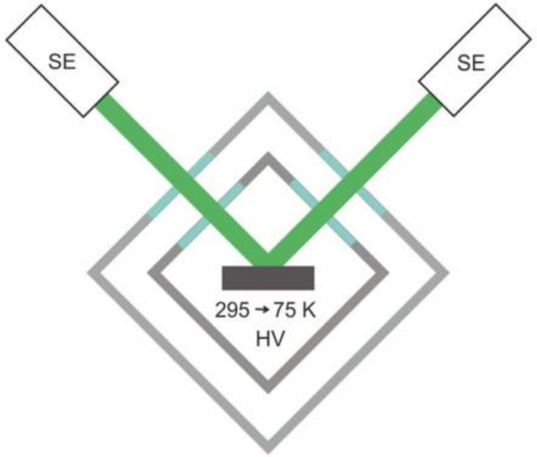
- Intensity *ratios* are measured
  → sensitivity to ambient noise is almost null
- Two quantities are obtained: Ψ, Δ
- Model-based approach

- Excellent sensitivity to **refractive index n** - down to 10^{-3}
- Good sensitivity to extinction coefficient k - down to 10^{-3}
- Outstanding sensitivity to **film thickness d** - less than 1 nm

Other physical quantities can be extracted: energy gap, surface roughness, Urbach energy…

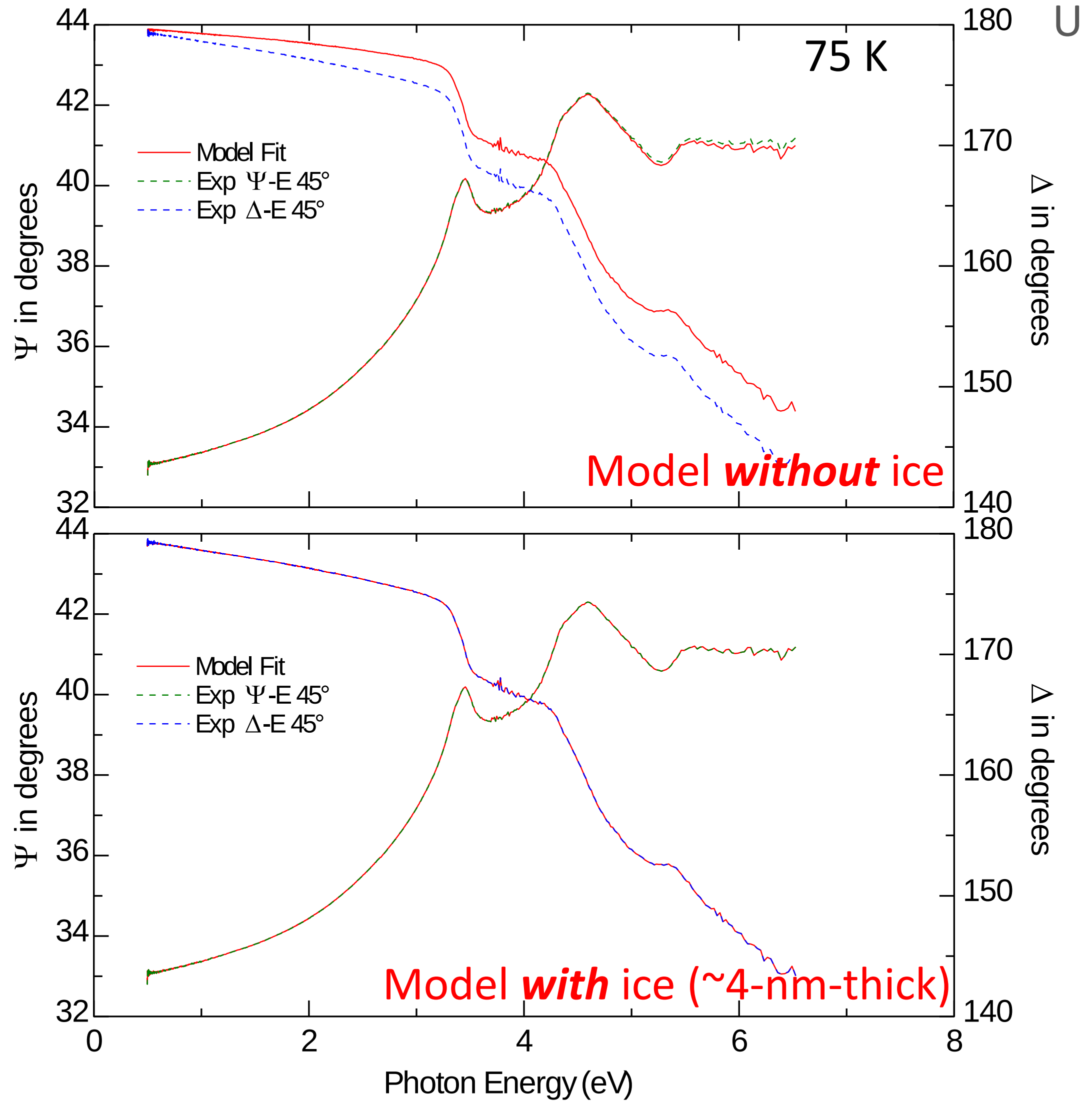
Ellipsometry at cryogenic temperatures





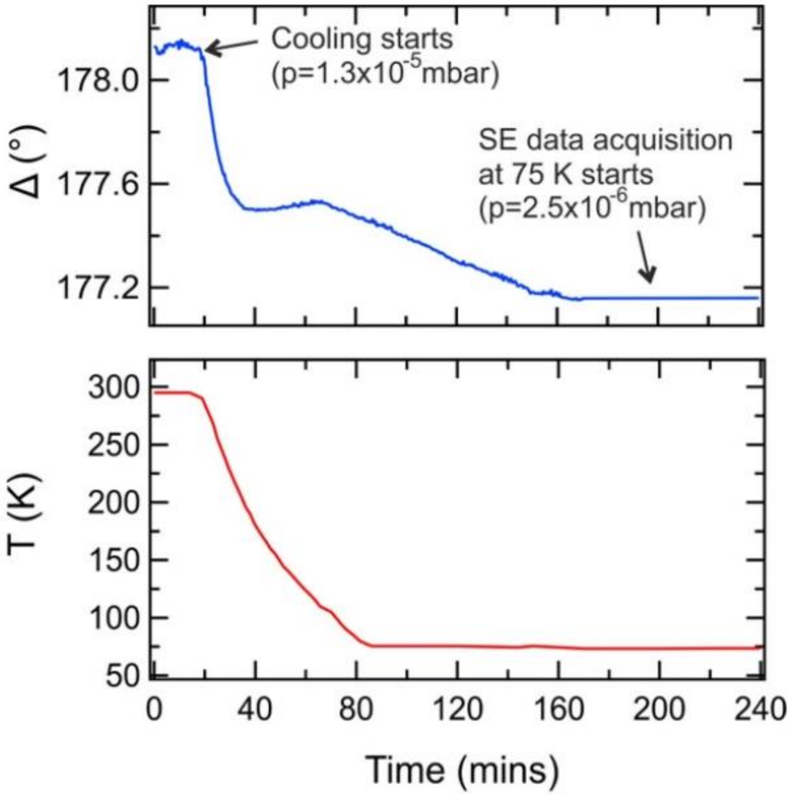
- Custom modified optical cryostat coupled with a Woollam VASE spectroscopic ellipsometer
- Very small cryostat volume (<1 l), allows 'fast' cryo and vacuum operations
- P_{base}: ~10^{-6} mbar below 100 K
- Angle of incidence: 45° (fixed)
- Spectral range available: 190-2500 nm (UV+Vis+NIR)
- Operating temperatures: 75 K or 4 K (hydrogen & helium compatible)
- Customization necessary to ensure geometry compliance with the ellipsometry measurements and alignment of the whole cryostat

Ultrathin ice on cold coatings



Ultrathin ice layer can be easily observed in SE in *real-time* during cooling

M. Magnozzi et al., *J. Phys. D: Appl. Phys.*, 56, 475105 (2023)

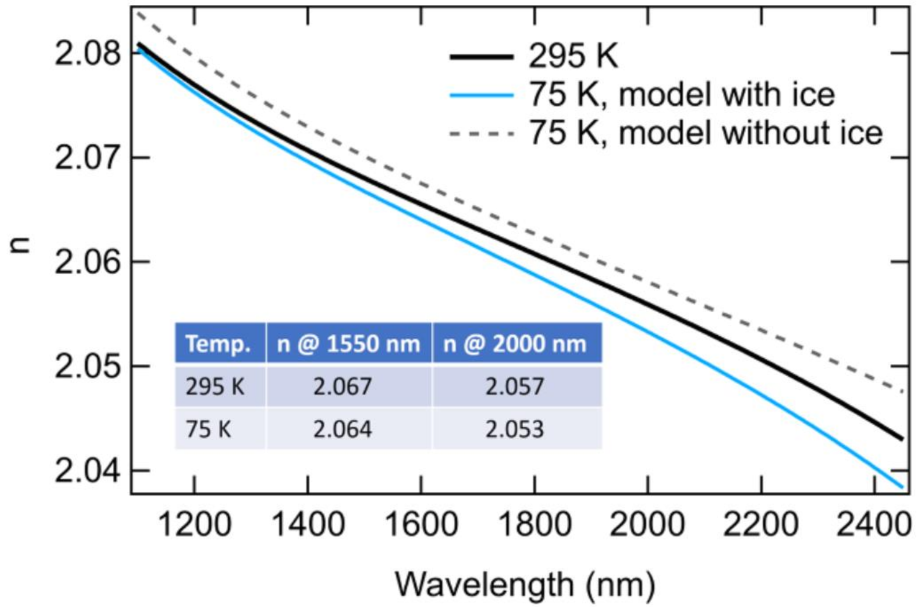


Once taken into account, it no longer affects the analysis of the underlying coating.

Optical properties of materials for mirrors at low temperatures

First material investigated was titania-doped tantala, currently used in GWD mirrors.

M. Magnozzi et al., *J. Phys. D:Appl. Phys.*, 56, 475105 (2023)



Temp.	n @ 1550 nm	n @ 2000 nm
295 K	2.067	2.057
75 K	2.064	2.053

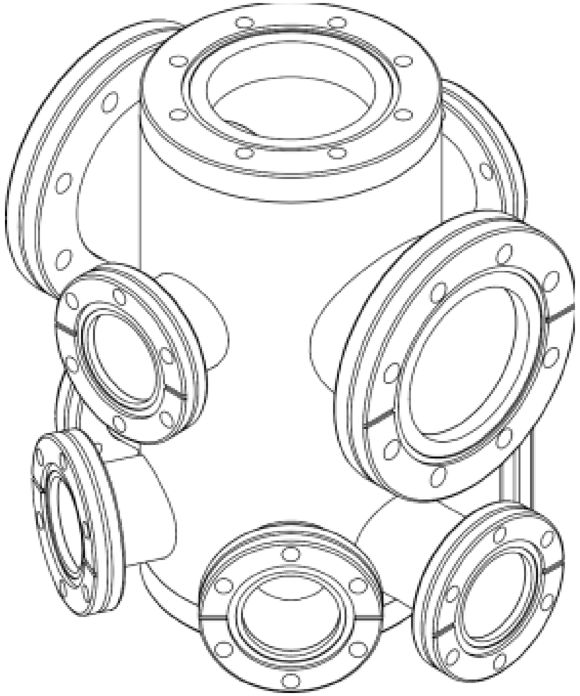
- ✓ Modelling the ice layer is necessary to properly evaluate the sign of temperature-induced variations.
- ✓ Temperature-induced variations in the high-refractive index of TiO_2-doped Ta_2O_5 were assessed.

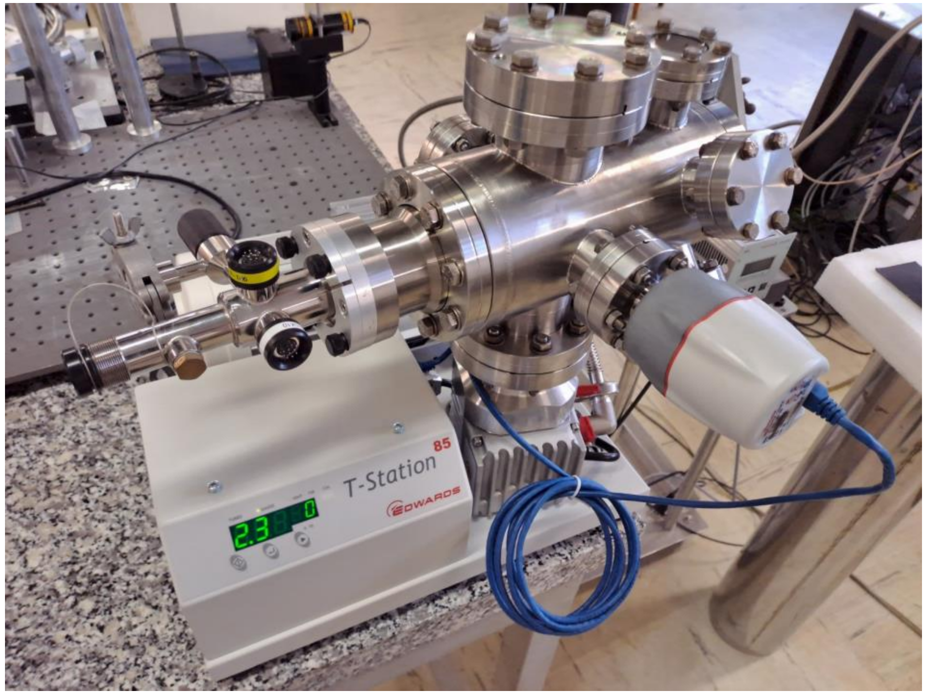
New samples are welcome!
Contact me for proposals
magnozzi@fisica.unige.it

Ellipsometry at cryo temperatures: work in progress

An **upgraded setup** is currently under development setup and will be operational in 2024

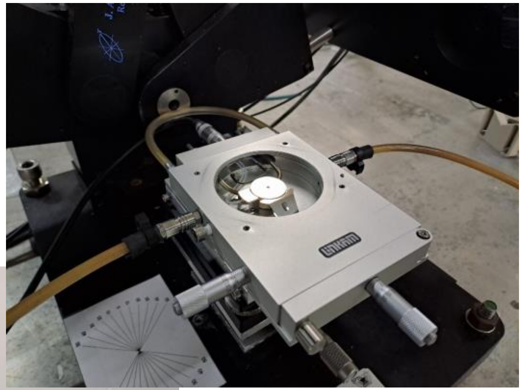
- improved pumping efficiency within the cryostat and possibly reduced base pressure
- allows measurements at 65° angle of incidence, better suited for spectroscopic ellipsometry

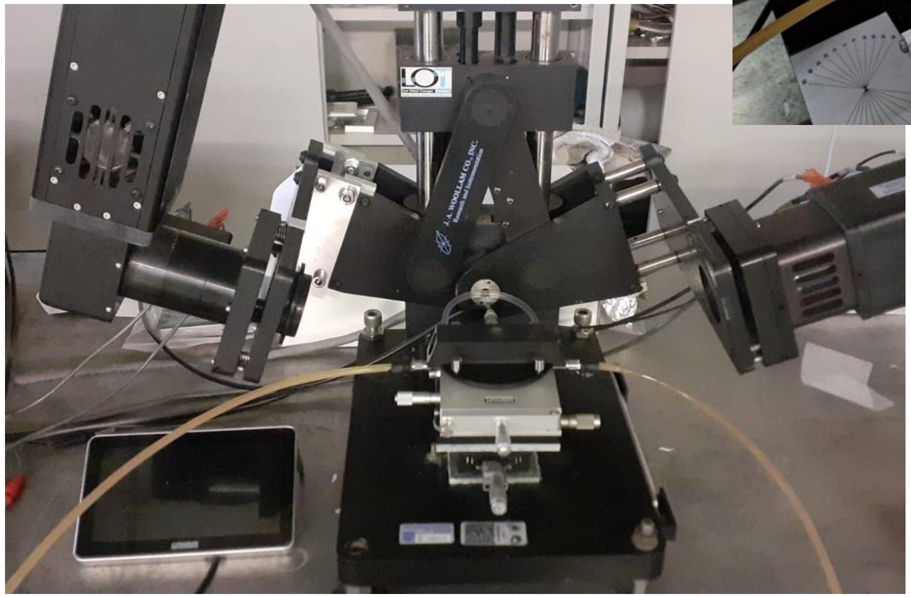




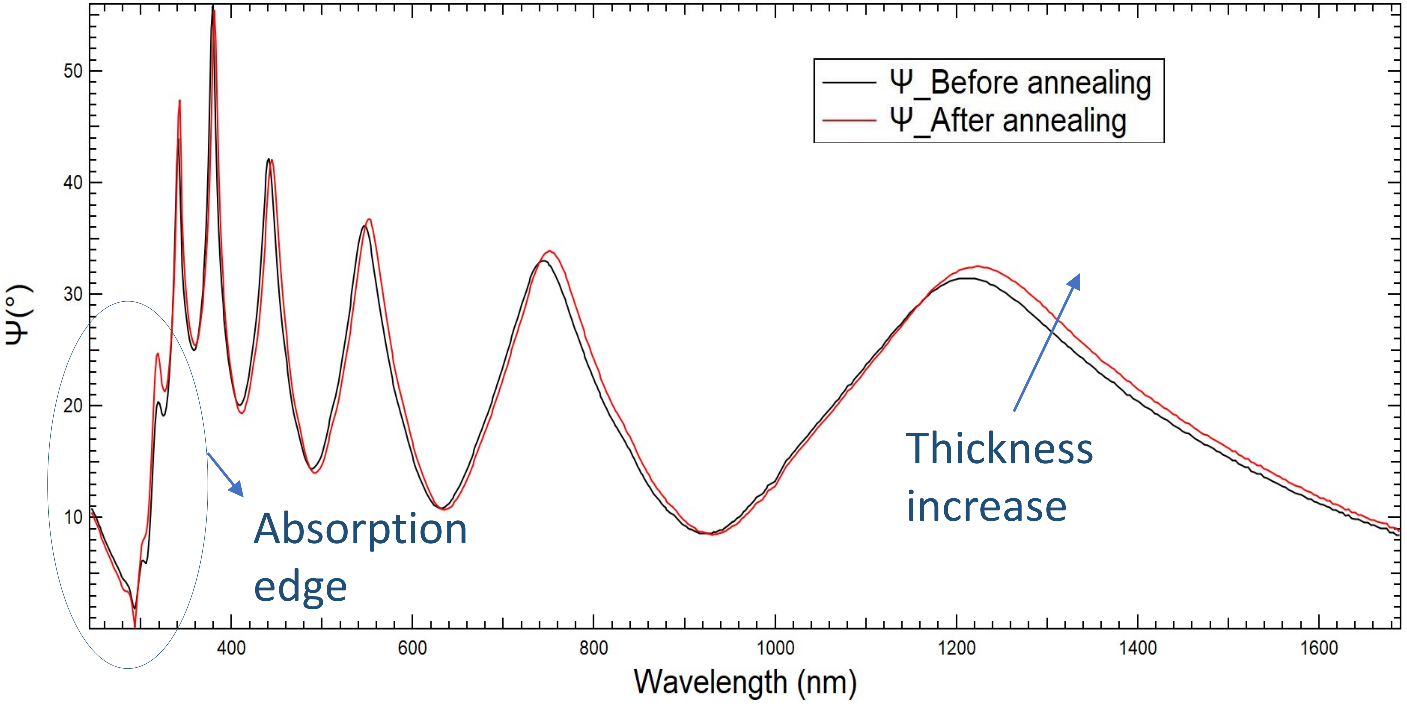
Monitoring in real time the annealing of the coatings

- Woollam M2000 ellipsometer with a Linkam heating cell
- Precise control over heating rate, maximum temperature and cooling rate
- Spectral range: 245-1685 nm
- Allows very long annealing (>10 h)
- Measurement time is very fast (~1 s)
- Air or Ar or N atmosphere

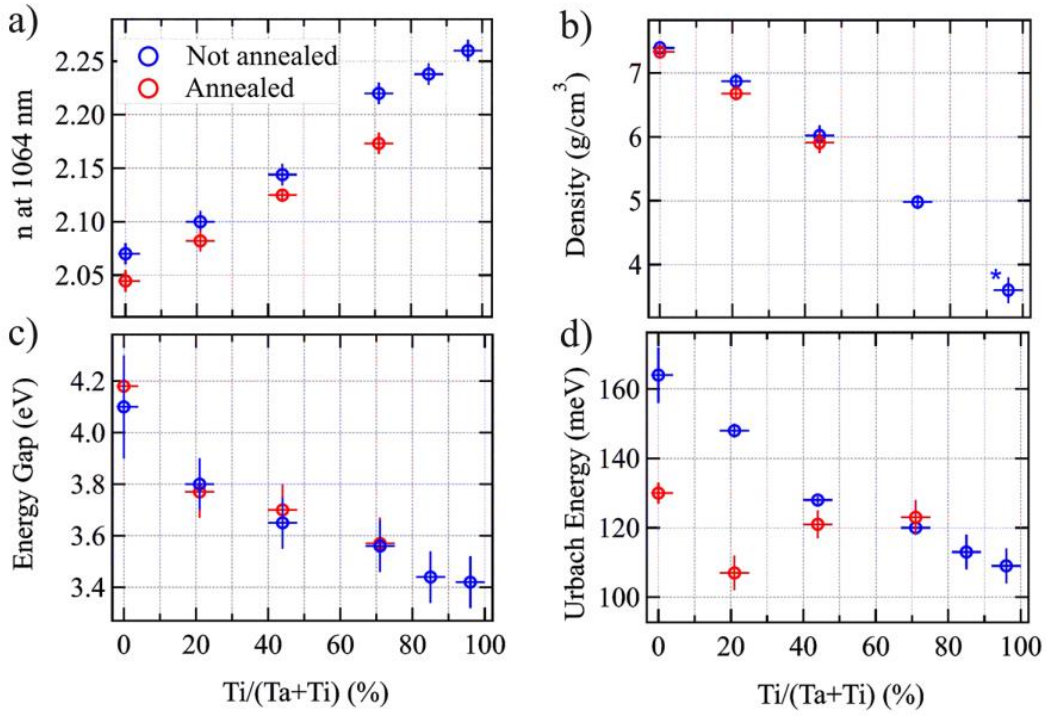




The static picture: before and after the annealing



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



By probing different EM regions of the spectrum (UV-vis-NIR) we can determine different properties

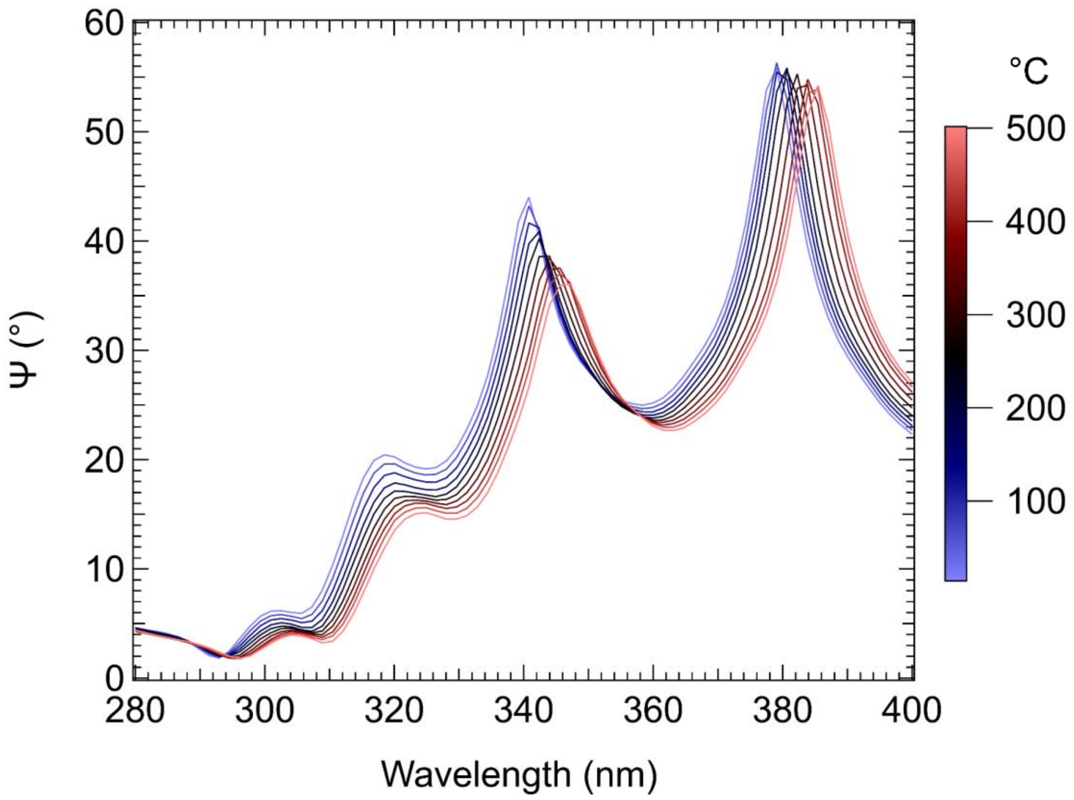
- thickness
- refractive index
- absorption edge
- energy gap
- Urbach energy
- surface roughness

See also:

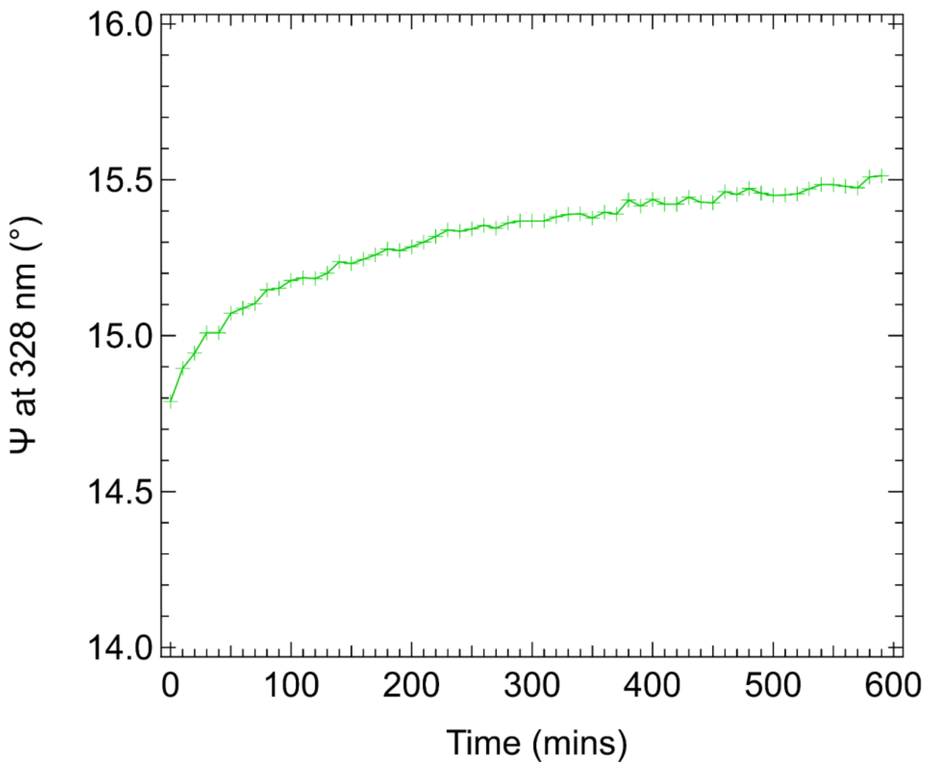
A. Amato et al., *Sci. Rep*. 10:1670 (2020)
A. Amato et al., *J. Phys. Mater. 2*, 035004 (2019)
M. Magnozzi et al., *Opt. Mater*. 75, 94 (2018)

The dynamic picture: during the annealing

Heating ramp 0 → 500 °C @ 1.67 °C/min (100 °C/h)



10-hour plateau @ 500 °C



Real-time monitor of annealing: work in progress

Model-based analysis is mandatory to obtain results from SE data.

Necessary steps include:

- **decoupling** the effects of the **substrate from** those of the **coating**
  requires dedicated measurements and modelling on the bare substrate

- characterize the **relative large variations** in thickness and refractive index, which probably occur during the early stages of the annealing

- study the **fine effects** that modify the **onset of absorption** over long times (many hours)

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Summary and perspectives

Temperature-dependent, real-time investigation of coatings for GWD mirrors is an active field of research in the Genova RU.

- **So far we studied:**

- ultrathin ice growth on cold coatings
- optical properties of titania-doped tantala at 75 K
- real-time annealing of titania-doped tantala (500 °C, 10h)

- **Work in progress:**

- upgrading the cryostat for ellipsometry
- data analysis on real-time annealing of titania-doped tantala

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