SiN films: Characterization workflow and examples from analysis

work-in-progress report

Michele Magnozzi
for the Virgo Coating R&D Collaboration

The scientific case

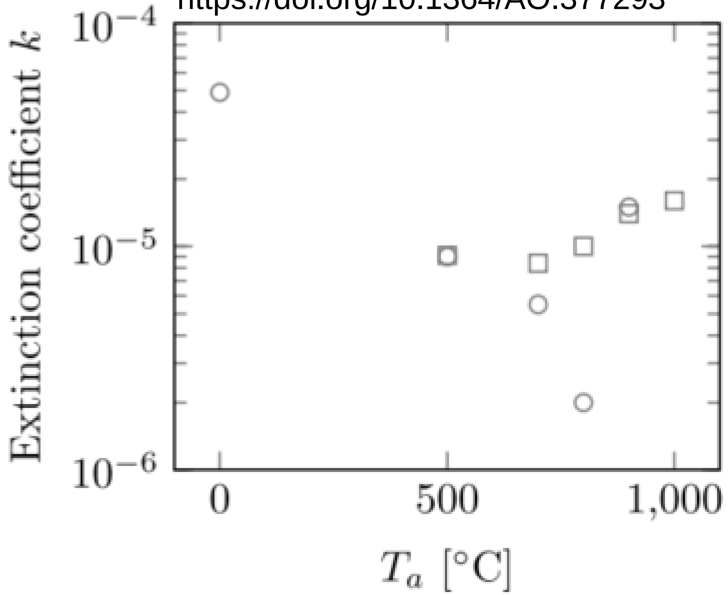
- Silicon Nitride coatings
  - Losses are about 3 times lower than present Ta_2O_5-TiO_2 coatings
  - Can be annealed up to 900 °C without crystallization
  - Optical absorption is relatively high and has to be reduced

Challenge:
increase n, *decrease* k

Table 2. Tentative Projection of Requirements for Optical and Mechanical Properties of Coating Layers for Future GW Interferometers, Based on Current Standards (from Refs. [4,30] and This Work)

Refractive Index	$n_H > 2.09$
	$n_L < 1.45$
Extinction	$10^{-7} < k < 10^{-6}$
Scattering	$\alpha_s \leq 10$ ppm
Internal friction	$\phi_c < 10^{-4}$ at 100 Hz
Coated diameter	$d \geq 35$ cm
Thickness uniformity	$\Delta t_c \leq 0.1\%$
Surface roughness	≤ 0.1 nm rms

M. Granata et al., Appl. Opt. 59 (2020)
https://doi.org/10.1364/AO.377293



Origin of absorption

- Factors affecting the optical absorption & refractive index
  - Stoichiometry (S/N ratio)
  - Oxygen
  - Hydrogen
  - Contaminants
  - Coating (in)homogeneity
  - Density

Literature studies help
(only up to a certain point…)

Example: silicon-rich SiNx films have higher absorption [Paule et al., Vacuum 37:395, 1987]

- Characterization is required to track ALL the above factors, so that they can be optimized during the next fabrication campaign

Tracking the factors affecting absorption

Optical properties

Quantity	Technique	Group
n	Spectrophotom. Ellipsometry	LMA Genova
k	Spectrophotom. Phototherm. defl. Ellipsometry	LMA " Genova
gradient	Spectrophotom. Ellipsometry	LMA Genova

Chemical/compositional properties

Quantity	Technique	Group
Stoichiometry, H content, O content, Contaminants	EDX XPS " Raman LIBS FTIR RBS SIMS ERDA RBS	Perugia Roma1 Genova g-MAG g-MAG Padova " " SAFIR/ Navier

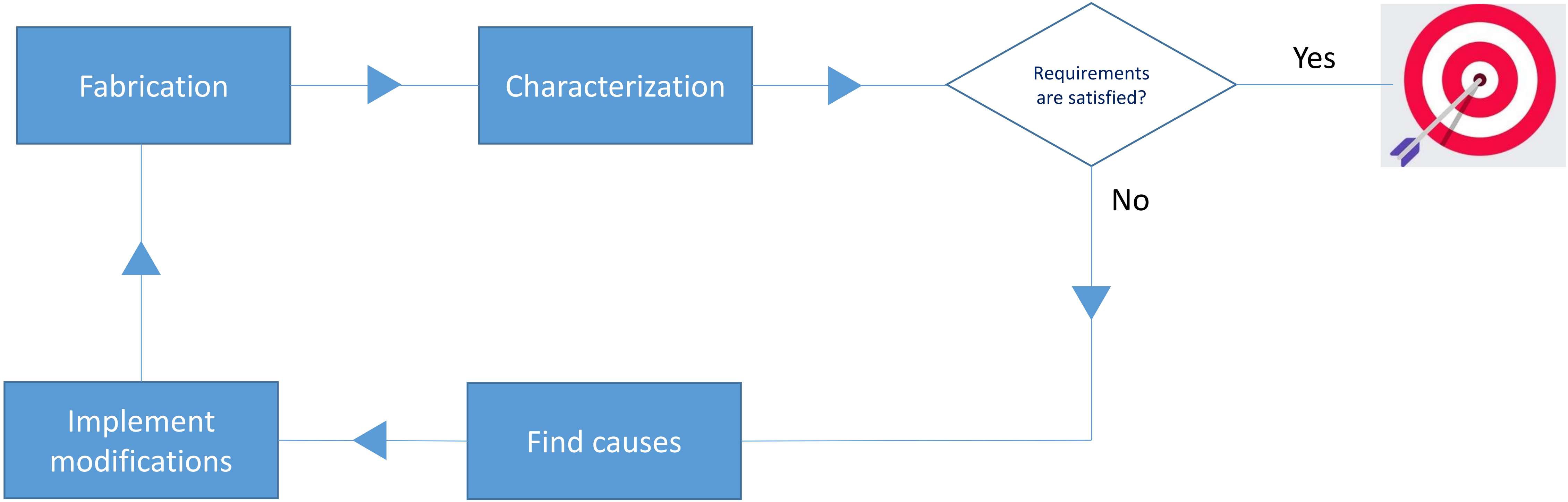
Morphological/structural properties

Quantity	Technique	Group
Thickness	Spectroph. Ellipsom.	LMA Genova
Surface analysis	SEM AFM	Perugia "
Density, roughness	XRR	Padova
Cryst. content	GIXRD	"

The results from the characterization are evaluated and discussed by considering the theory describing the amorphous coatings (Navier)

*NOTE: concerning the thermo-optic effect in SiNx coatings, see the poster by **Matteo Bischi** on 20 May*

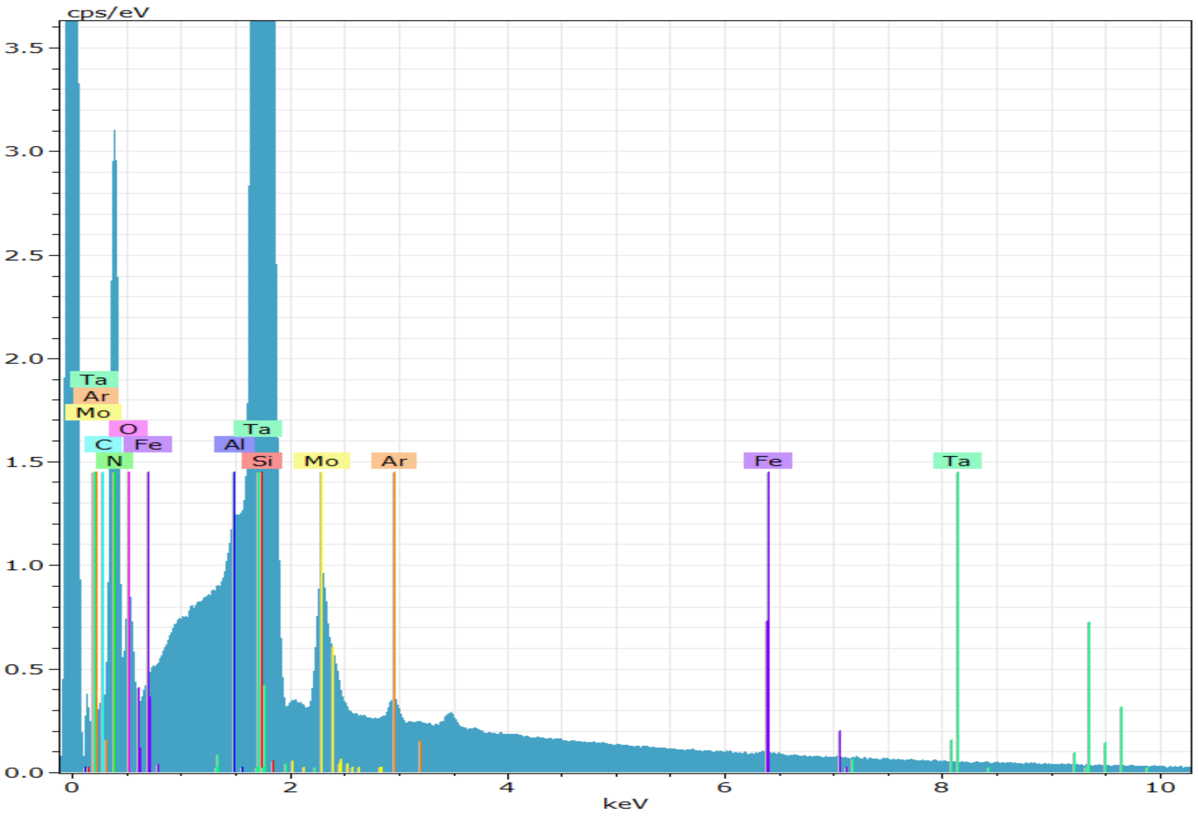
Workflow



So far: **4 batches of SiN films** have been produced by LMA and distributed to VCR&D for characterization.
Other samples will be available soon.

Example of analysis (sample prod. date: dec2020-jan2021)

EDX data and analysis (**Perugia**)

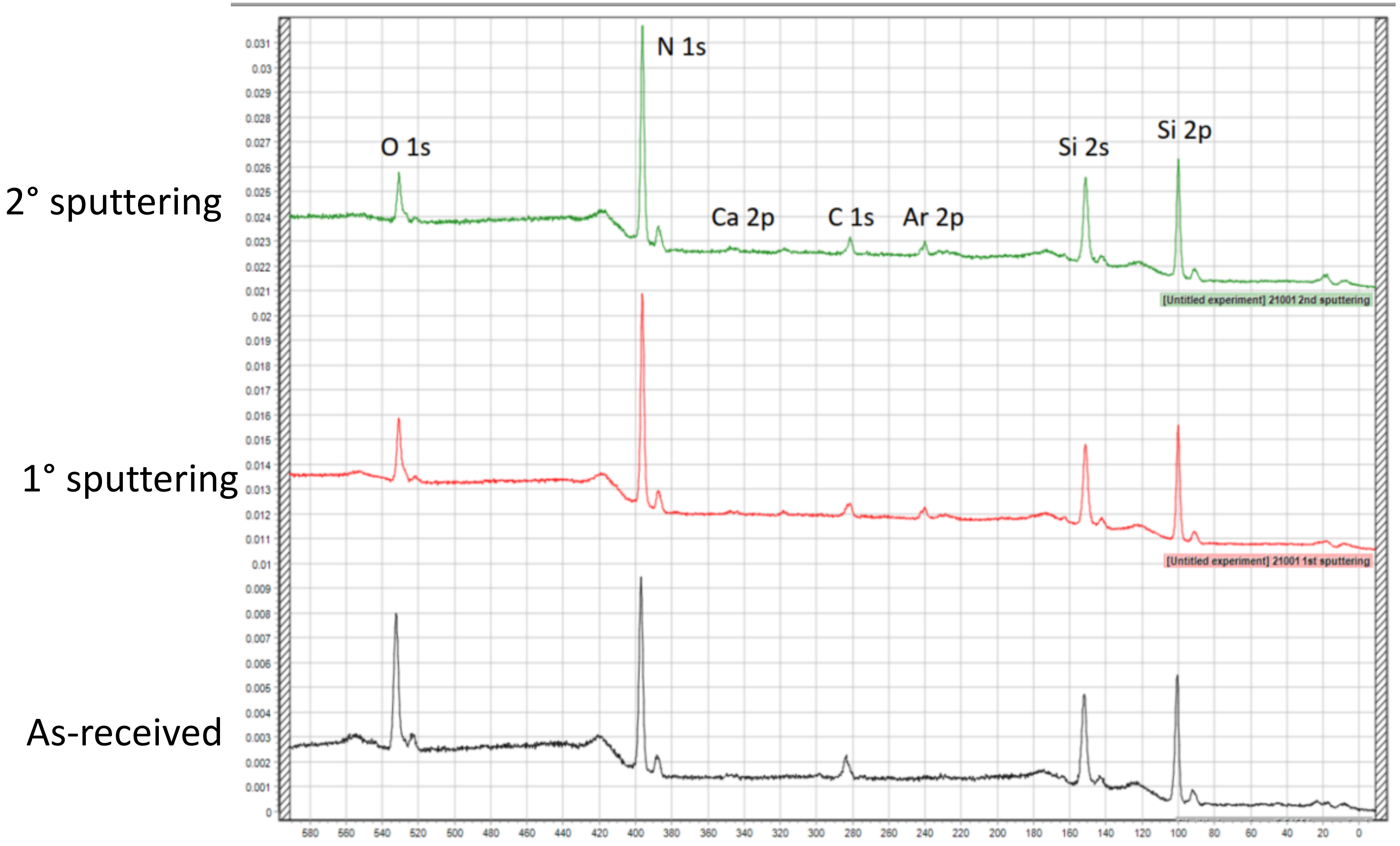


Element	series	[norm. wt.%]	[norm. at.%]	Error in wt.% (1 Sigma)
Silicon	K-series	62.43918541	48.89649742	2.608289506
Nitrogen	K-series	27.58287787	43.31181983	3.094118152
Aluminium	K-series	0.425638601	0.346958026	0.044989492
Carbon	K-series	0	0	0
Oxygen	K-series	4.548775018	6.253072217	0.581343653
Molybdenum	L-series	3.105090425	0.711831517	0.134403896
Argon	K-series	0.359018787	0.197662736	0.037264495
Tantalum	L-series	LOD	LOD	LOD
Iron	K-series	0.349075697	0.137474594	0.037868634
	Sum:	100	100	

Si/N ratio: 1.129

Example of analysis (sample prod. date: dec2020-jan2021)

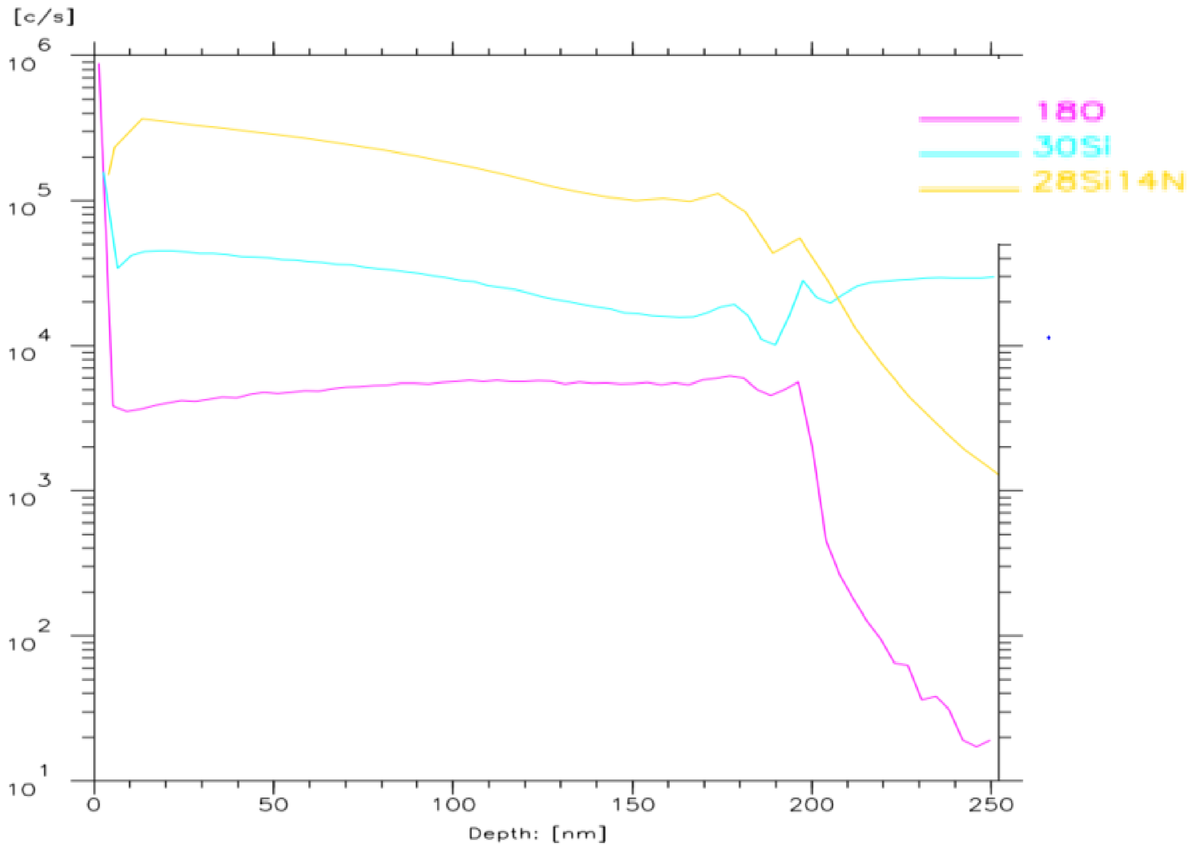
XPS data before and after sputtering (**Roma1**)



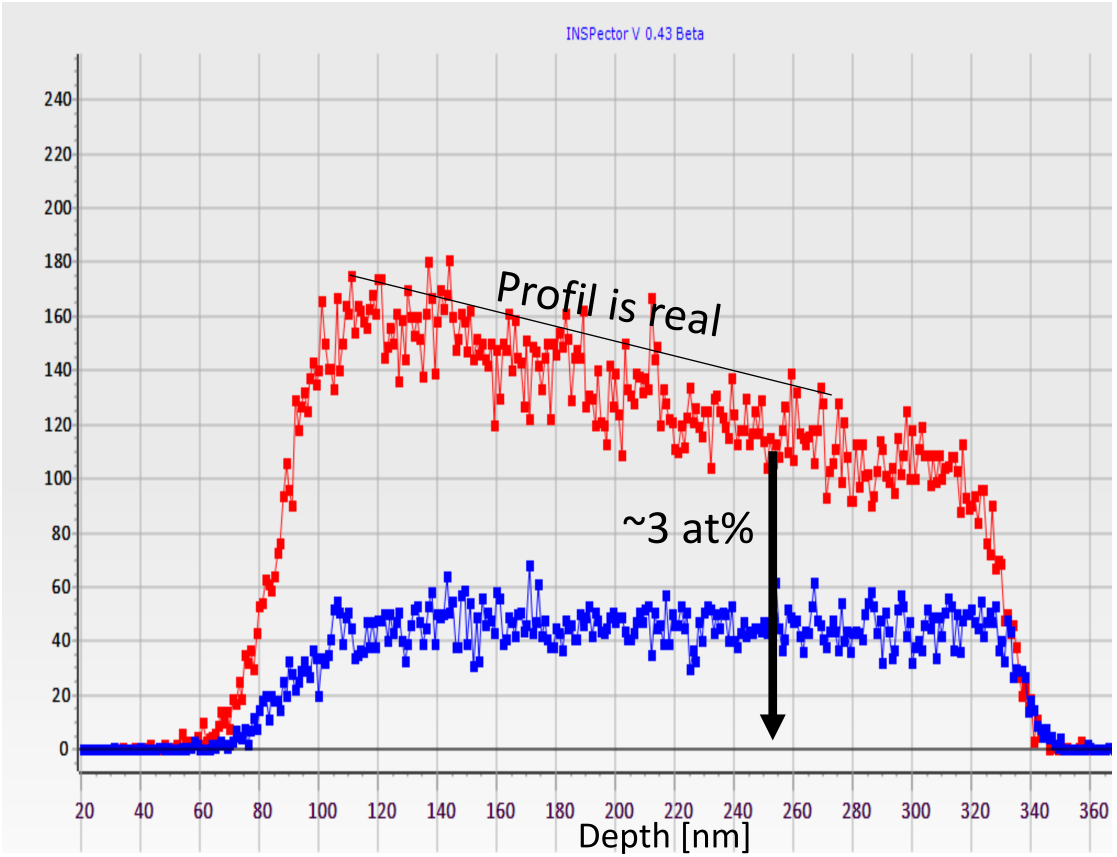
Note:
O content decreases
as depth is increased

Example of analysis (sample prod. date: dec2020-jan2021)

Compositional analysis across the coating with SIMS (**Padova**)

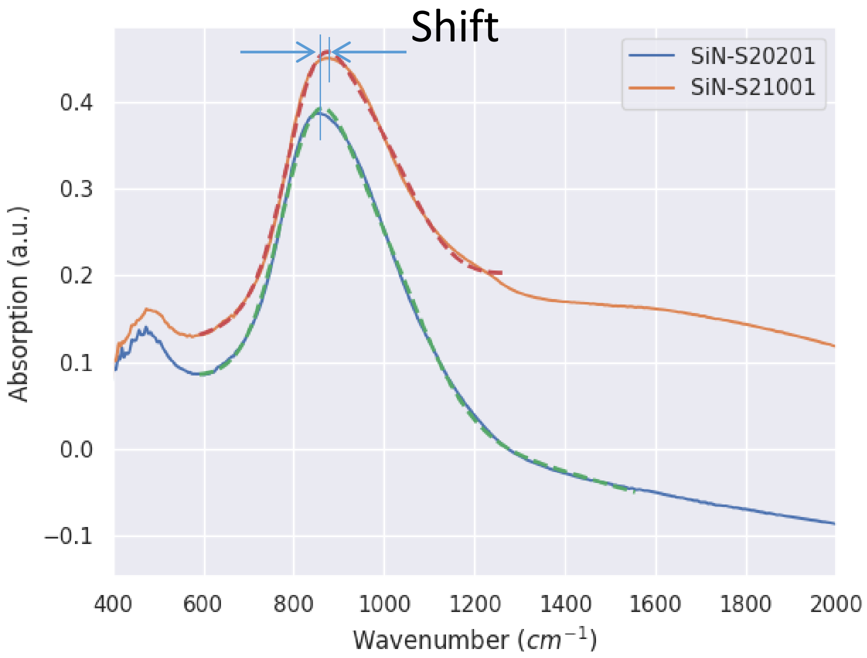


H content and vertical gradient measured with ERDA (**SAFIR/Navier**)



Example of analysis (sample prod. date: dec2020-jan2021)

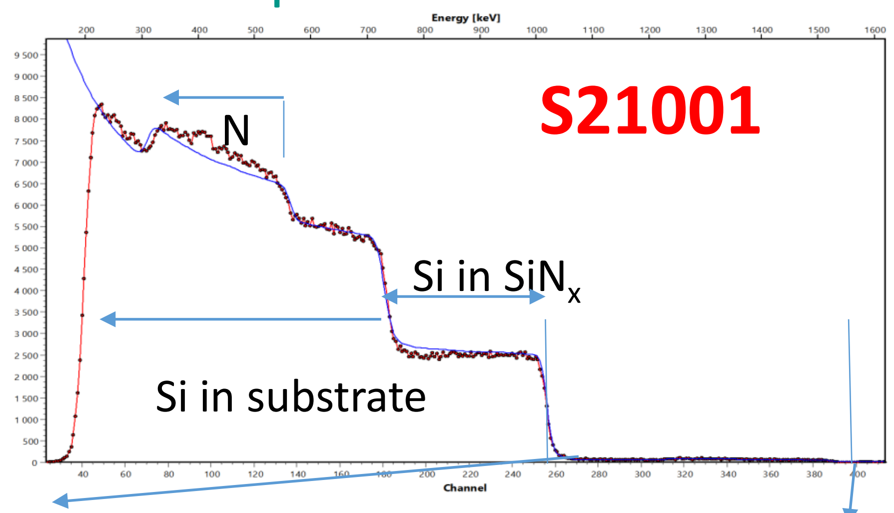
Presence of Oxygen evaluated by FTIR (**Padova**)

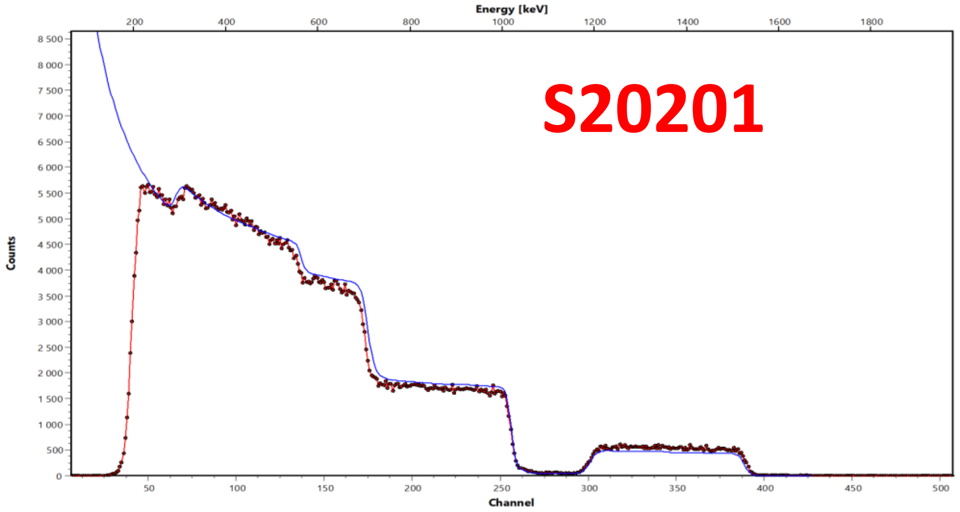


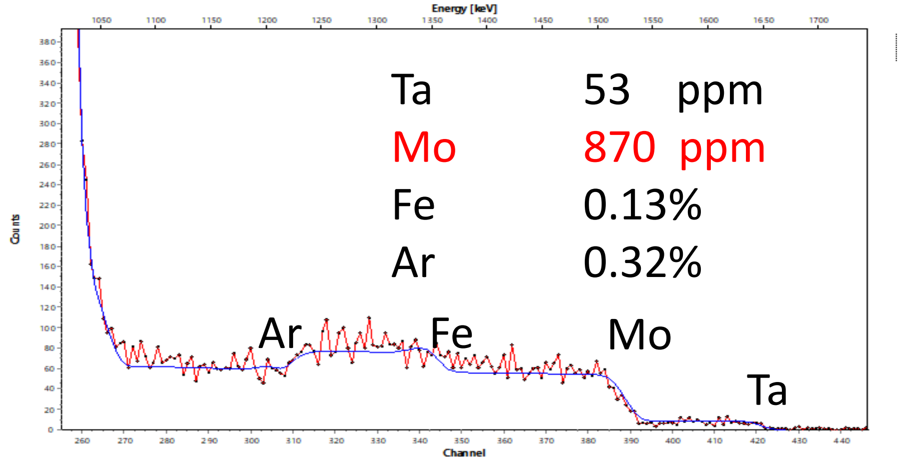
- Dominating band between 800 and 1200 cm^-1 is attributed to the group of Si-N and Si-O bonds.
- The main peak position shifts to larger wavenumbers by increasing the O content as the predominant bond type passes from the Si-N stretching to the Si-O stretching.
- Sample S21001 is richer in oxygen.

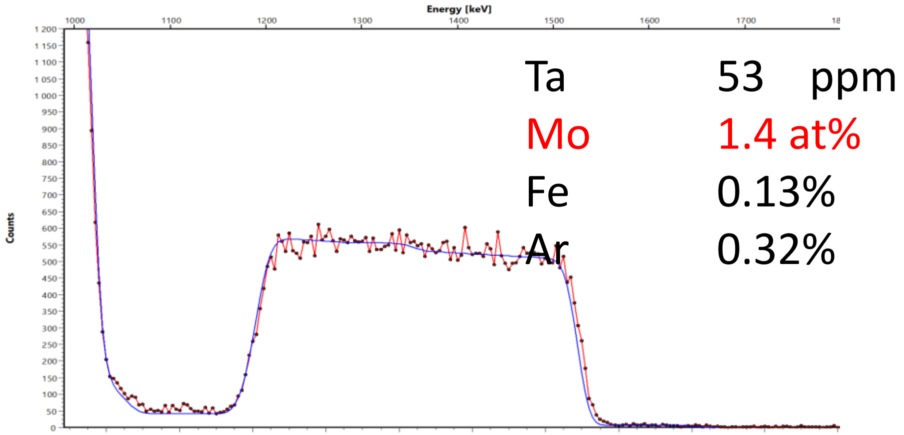
Example of analysis (sample prod. date: dec2020-jan2021)

SiN composition measured with RBS (**SAFIR/Navier**)



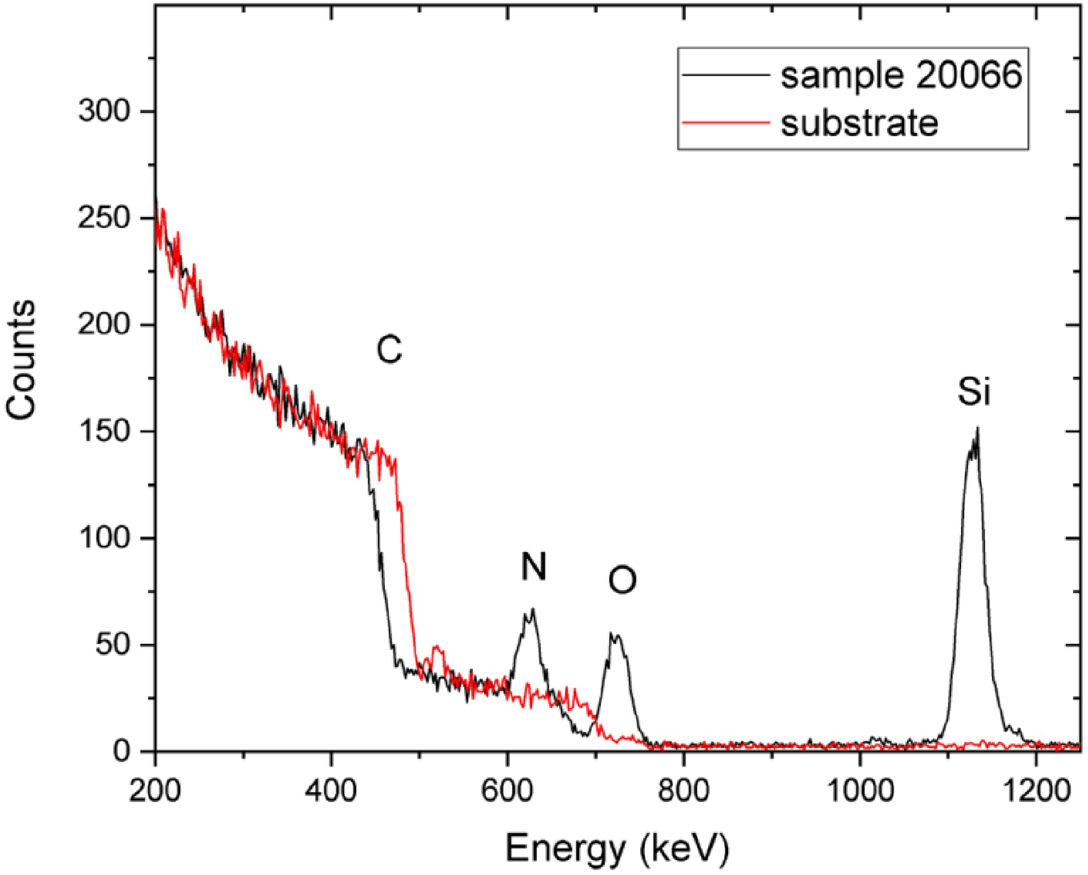






Example of analysis (sample prod. date: spring 2020)

SiN composition measured with RBS **(Padova, previous batch)**



After background subtraction:

Si = 1.40e17 atoms/cm2

O = 1.44e17 atoms/cm2

N = 1.20e17 atoms/cm2

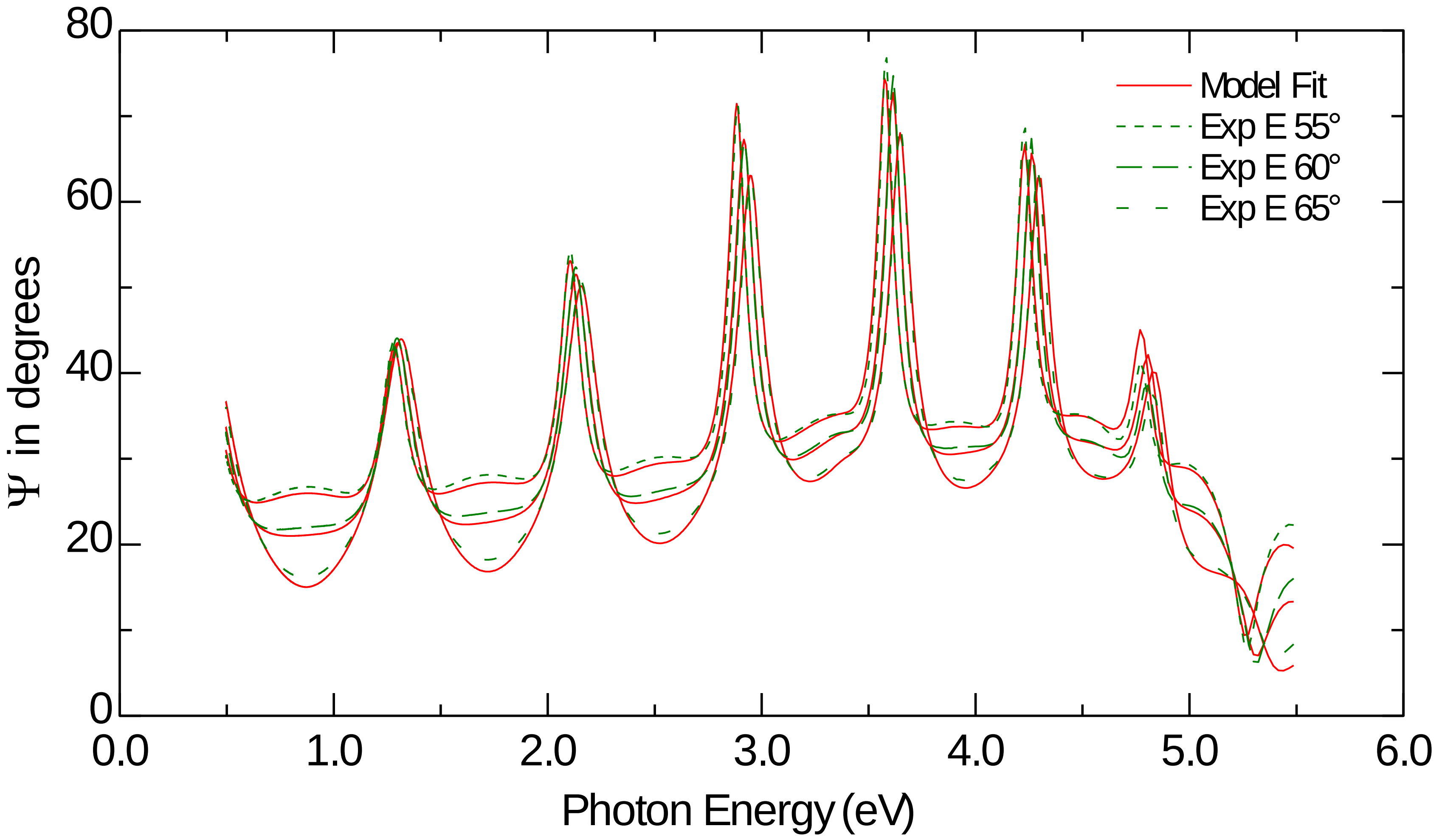
From these data one can obtain the corresponding atomic ratios:

N/Si=0.86

O/Si=1.03

Example of analysis (sample prod. date: dec2020-jan2021)

Presence of vertical gradient evaluated by ellipsometry (**Genova**)



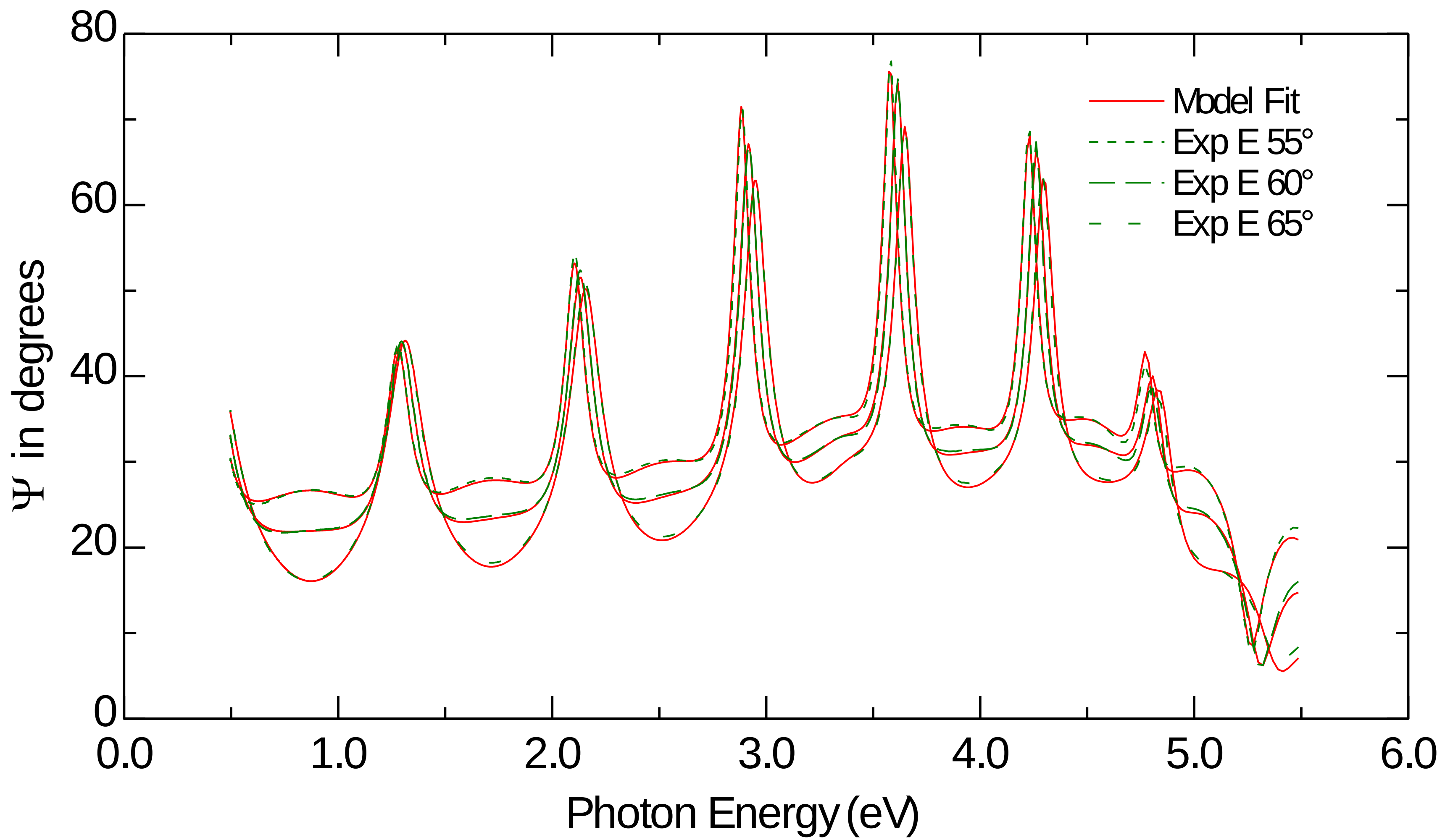
Model without grading

Results from SE analysis:
n@ 1064 nm: 1.90
Thickness: 417 nm

Results from spectrophotometry:
n@1064 nm: 1.94
Thickness: 415 nm

Example of analysis (sample prod. date: dec2020-jan2021)

Presence of vertical gradient evaluated by ellipsometry (**Genova**)



Results from SE analysis:

5.7% grading

Bottom to surface, n@1064nm: 1.85-1.96

Summary

- An **intensive, cooperative effort** is ongoing to provide a comprehensive characterization of the **optical, chemical, morphological/structural properties** of the SiN films produced by LMA

- 4 SiN batches produced and characterized so far; the ''performances'' of the films have been continuously improved thanks to optimizations in the fabrication process and feedback provided by characterization. Examples of negative factors that have been significantly reduced:
  - water content
  - vertical inhomogeneity

- New samples will be ready soon; the collaboration is ready to characterize them

- Best results so far on SiNx films and perspectives: **see next talk by Massimo Granata**