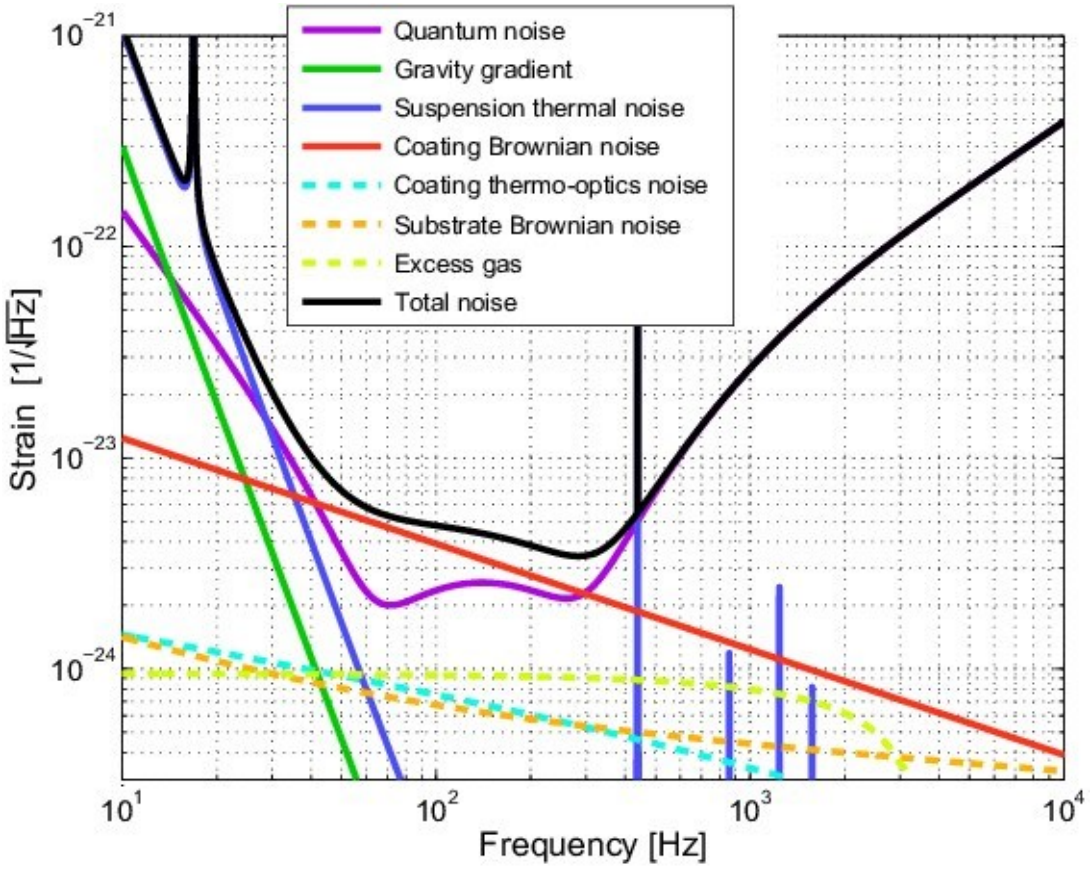
A structural study of the properties of amorphous silica coatings for low internal friction optics

Laura Silenzi
on behalf of Perugia-Camerino Virgo group

Overview

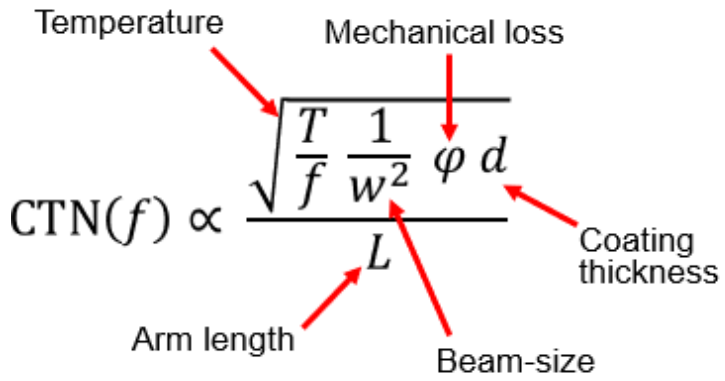
- Coating thermal noise for interferometric mirrors
- Investigation of the structure with different techniques
  - Experiments and results
- Conclusions

Coating thermal noise



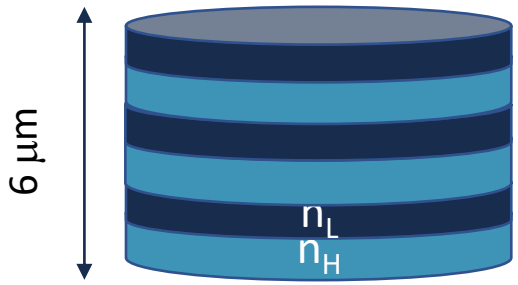
EPJ Web of Conferences **182**, 02003 (2018)

Coating thermal noise (CTN) dominates mid-band frequency sensitivity (40-350 Hz)

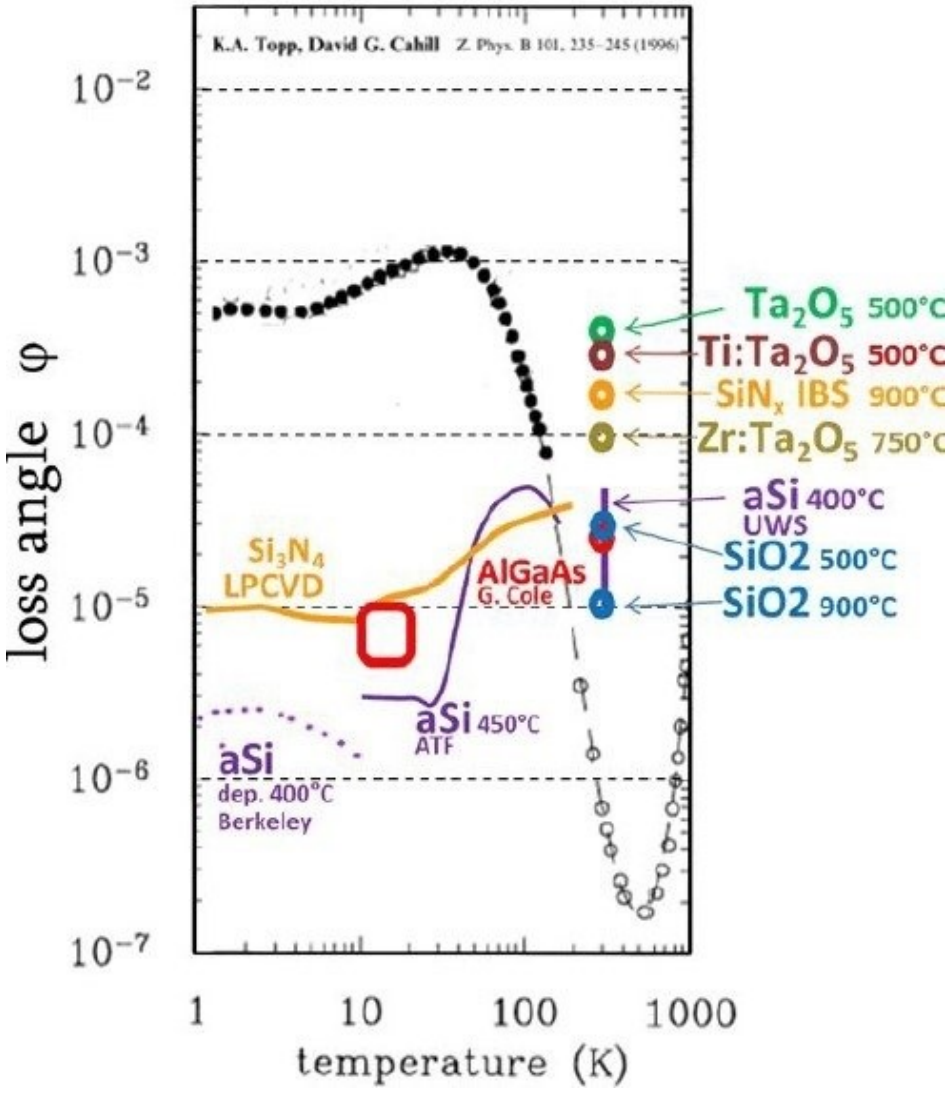


CTN related to ''intrinsic'' mechanical loss (fluctuation-dissipation theorem)

Mirror coatings

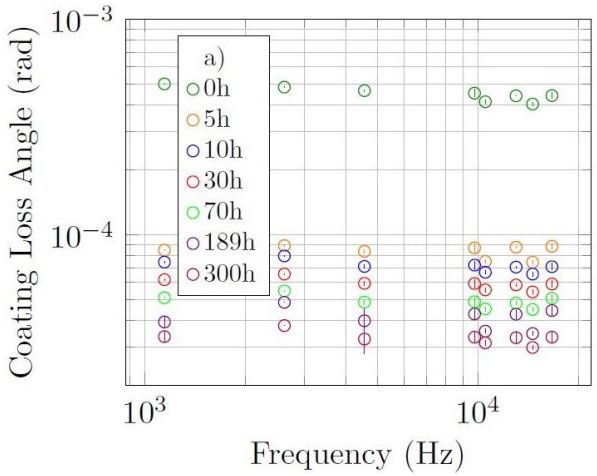


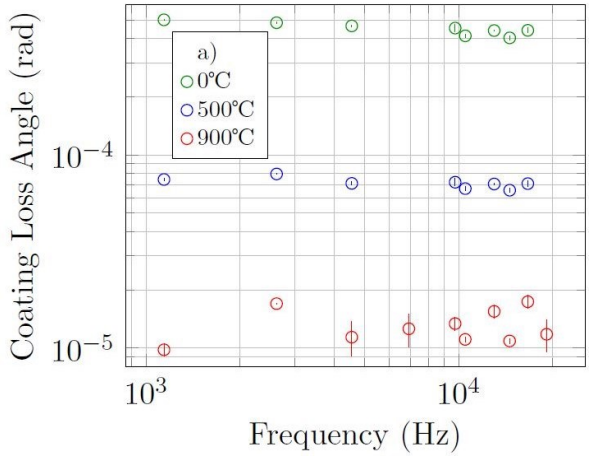
- Alternating materials with high/low refractive index
- Current adVirgo coatings: amorphous Ti(20%):Ta_2O_5 (n_H=2.07) and amorphous SiO_2 (n_L= 1.45)
- Deposition and post-deposition treatments



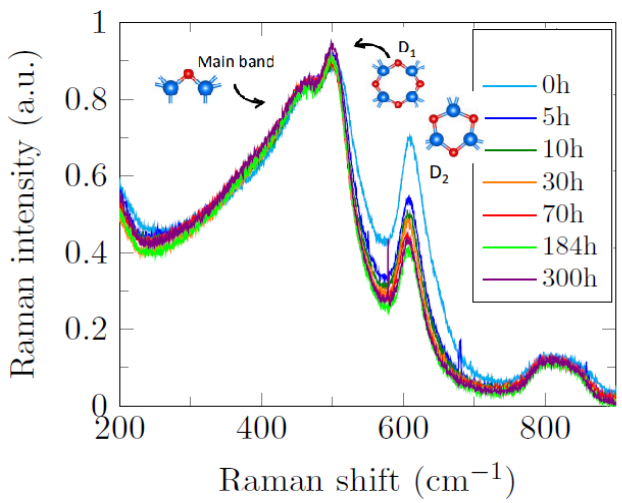
G.Cagnoli on behalf of VCR&D collaboration

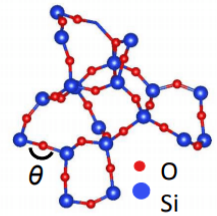
SiO_2: status

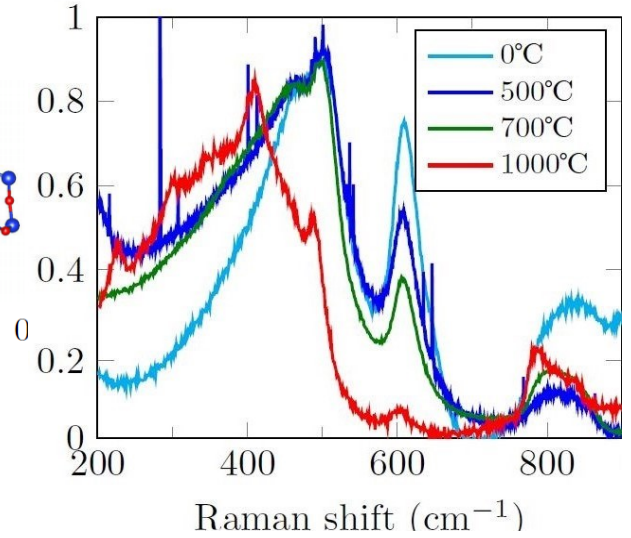




A. Amato et al., *2018 J. Phys.: Conf. Ser*. **957** 012006







M. Granata et al., *Physical Review Materials* **2**, 053607 (2018)

A. Amato, *Low Thermal Noise Coating for New Generation Gravitational-Wave Detectors, Univ. de Lyon* (2019)

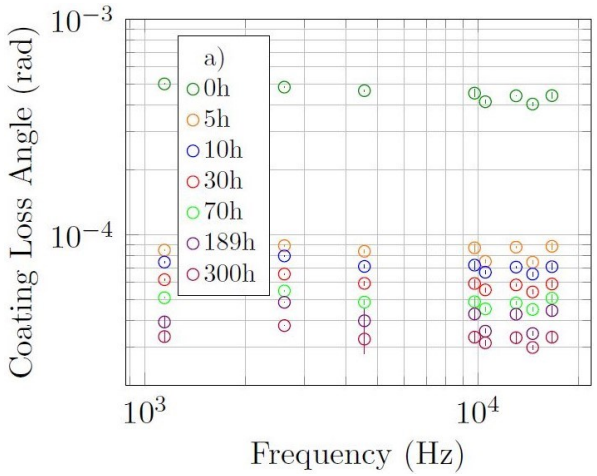
Reduction of mechanical losses at:

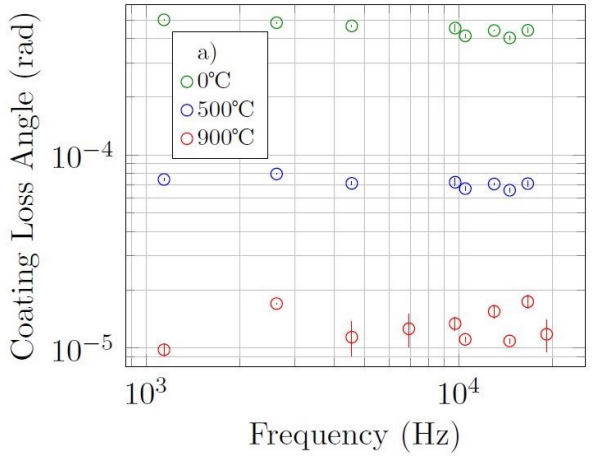
- Increasing annealing time (fixed temperature T= 500°C)
- Increasing annealing temperature (fixed time t= 10 hours)

Structure by Raman spectroscopy:

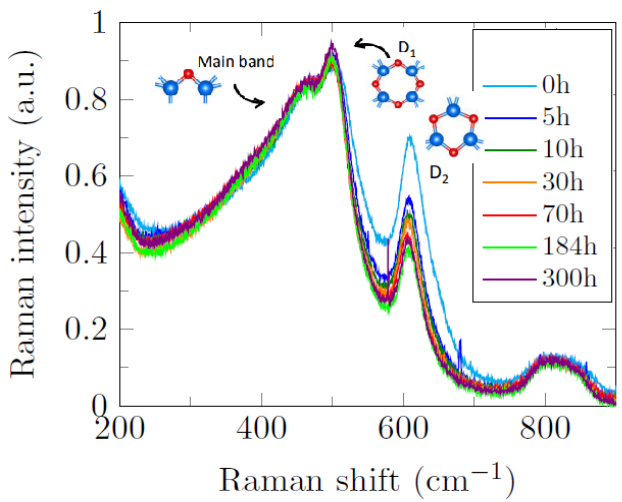
- Shift of the main band to lower frequencies → less dense structure
- Relative area of D2/D1 bands → decrease in the 3-fold ring population

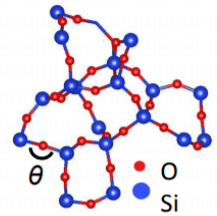
SiO_2: status

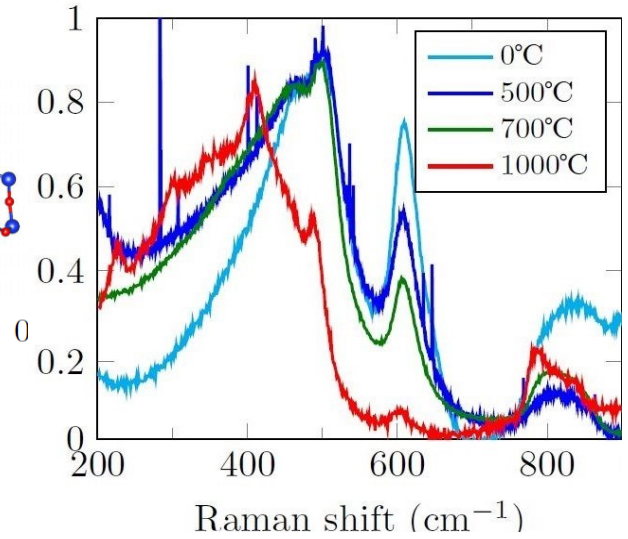




A. Amato et al., *2018 J. Phys.: Conf. Ser*. **957** 012006







M. Granata et al., *Physical Review Materials* **2**, 053607 (2018)

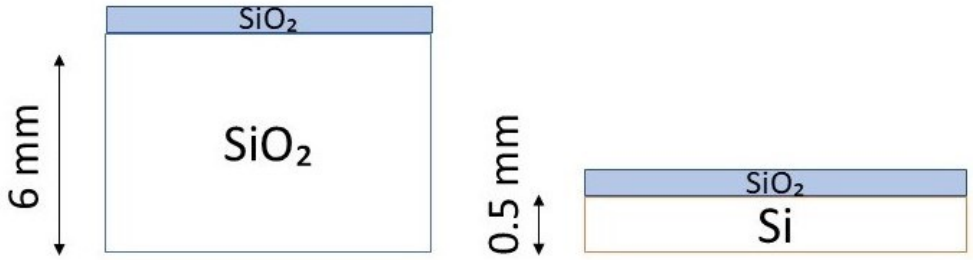
A. Amato, *Low Thermal Noise Coating for New Generation Gravitational-Wave Detectors, Univ. de Lyon* (2019)

AIM OF THIS STUDY

- Possible correlation between mechanical and structural properties of silica, investigating the structure at different length scales and with techniques complementary to Raman

SiO_2 study

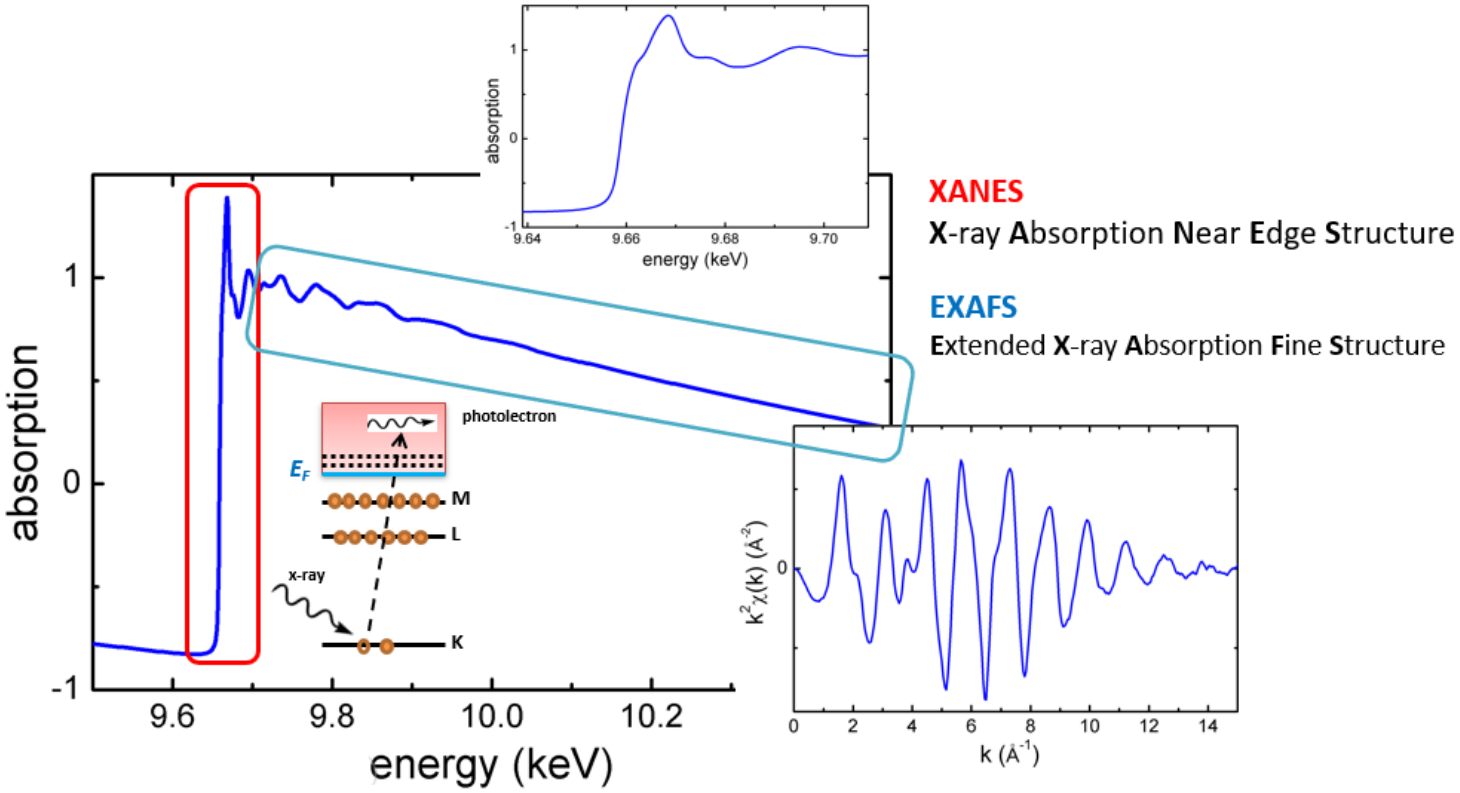
Samples:
- SiO_2 coatings deposited by IBS (LMA, Lyon)
- Si and SiO_2 substrates
- Annealing in air 10 hours up to 1000°C
- Coating nominal thickness: 500-720 nm



LUCIA beamline
Si K-edge (1839 eV)

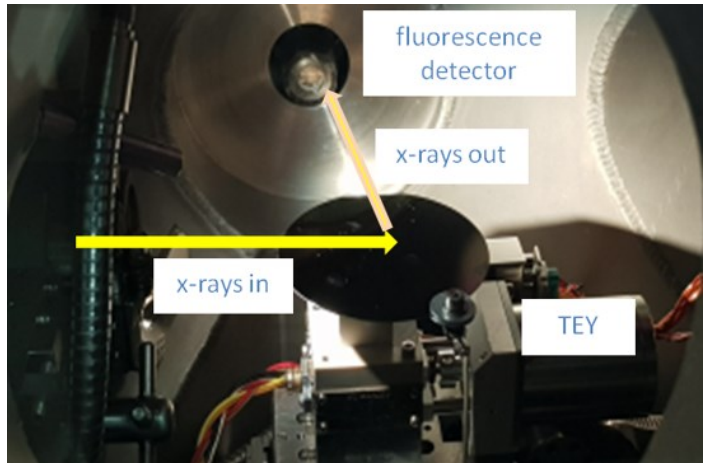
Techniques:
- X-Ray Absorption Spectroscopy
  - XANES (oxidation state and medium range order)
  - EXAFS (local order)
- Grazing Incidence X-Ray Diffraction (structure, crystallization)
- X-Ray Reflectivity (density)
- Fourier Transform Infrared Spectroscopy (short and medium range order)

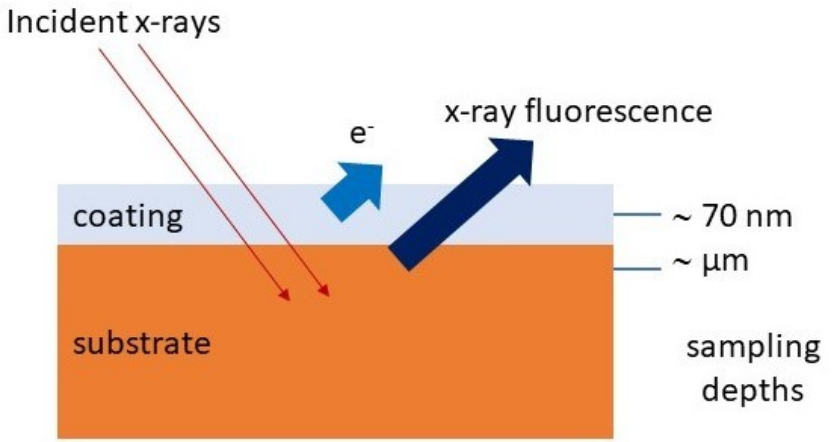
X-ray Absorption Spectroscopy



XANES
X-ray **A**bsorption **N**ear **E**dge **S**tructure

EXAFS
Extended **X**-ray **A**bsorption **F**ine **S**tructure

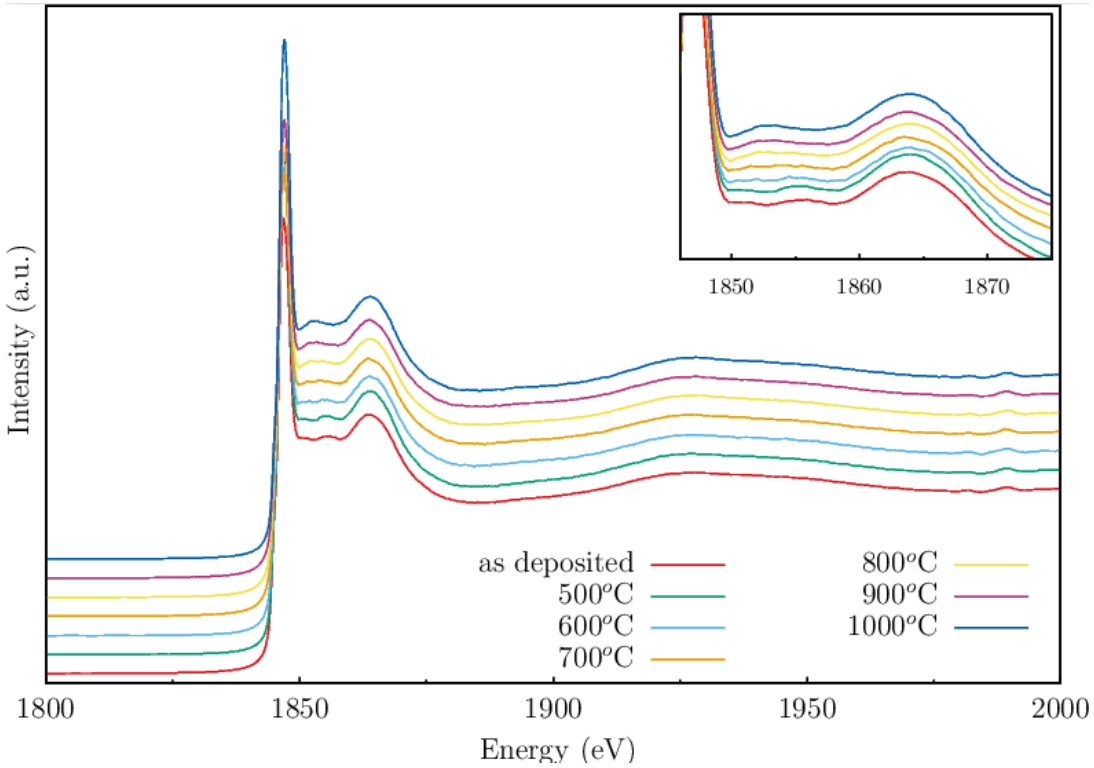




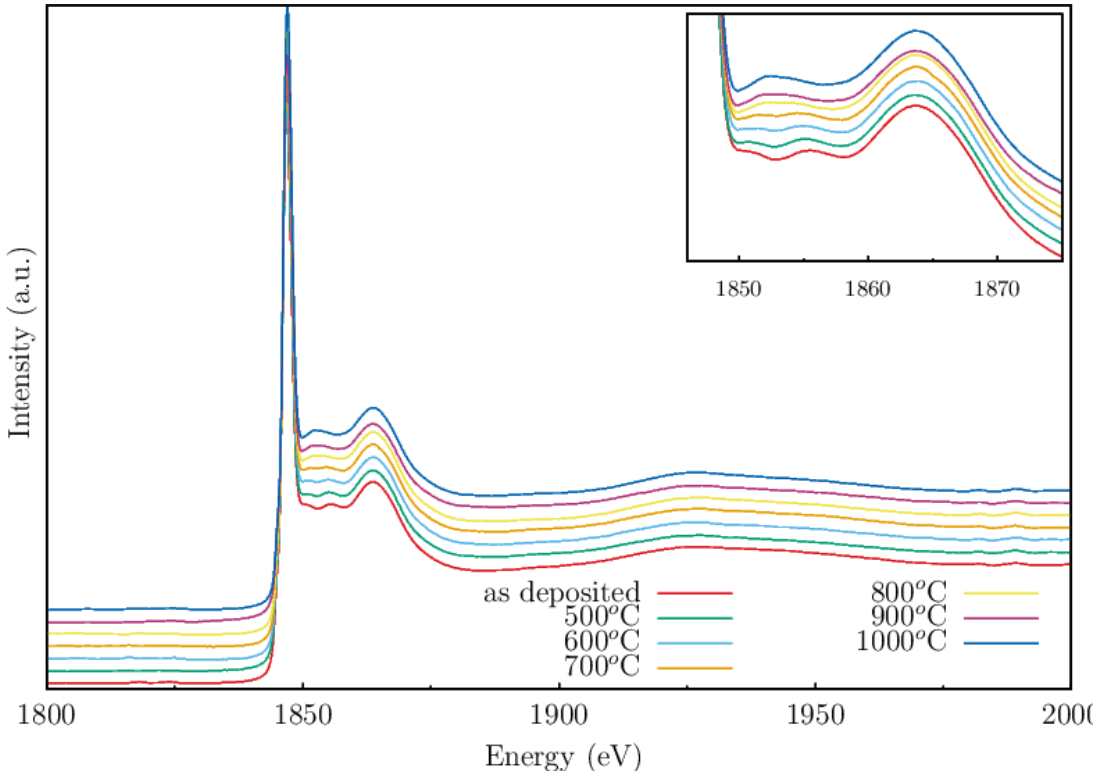
- Syncrothron based technique
- X-ray absorption coefficient µ as a function of the photon energy across the (photoelectric) absorption edge of a specific atom in the material

X-ray Absorption Spectroscopy

Fluorescence



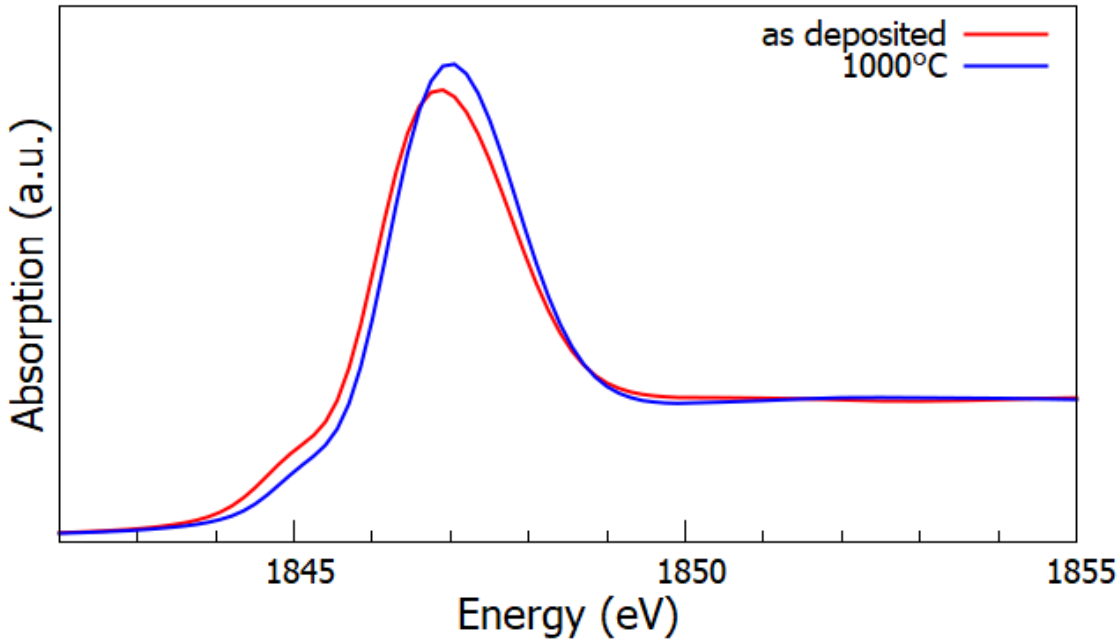
Total electron yield

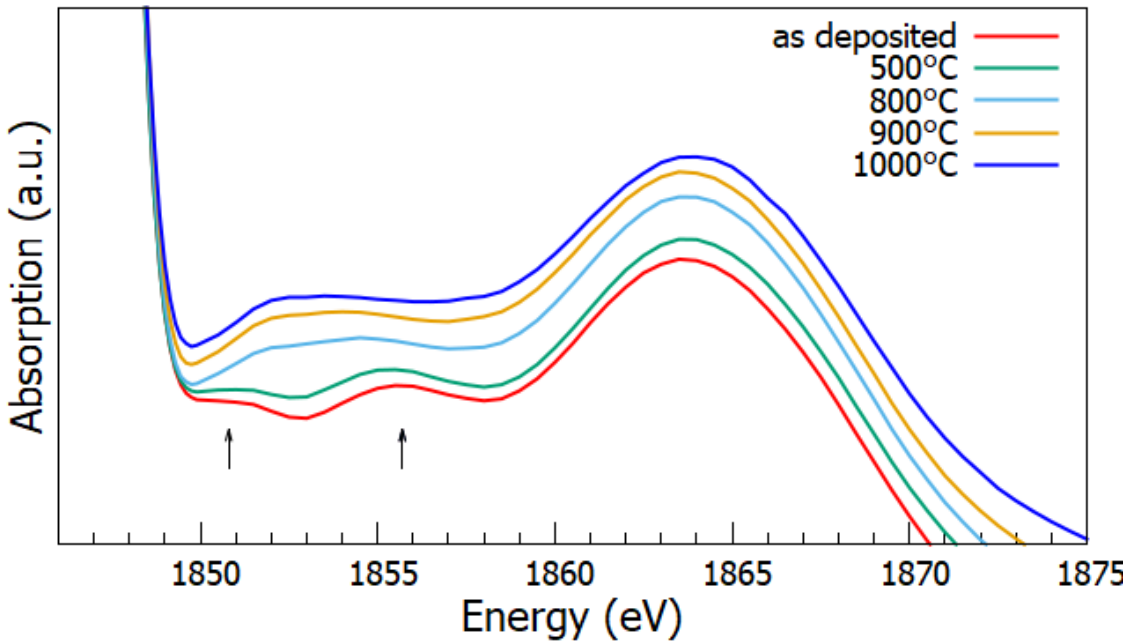


No differences in the result for different sampling depths

➢ Change occurs within the whole coating depth

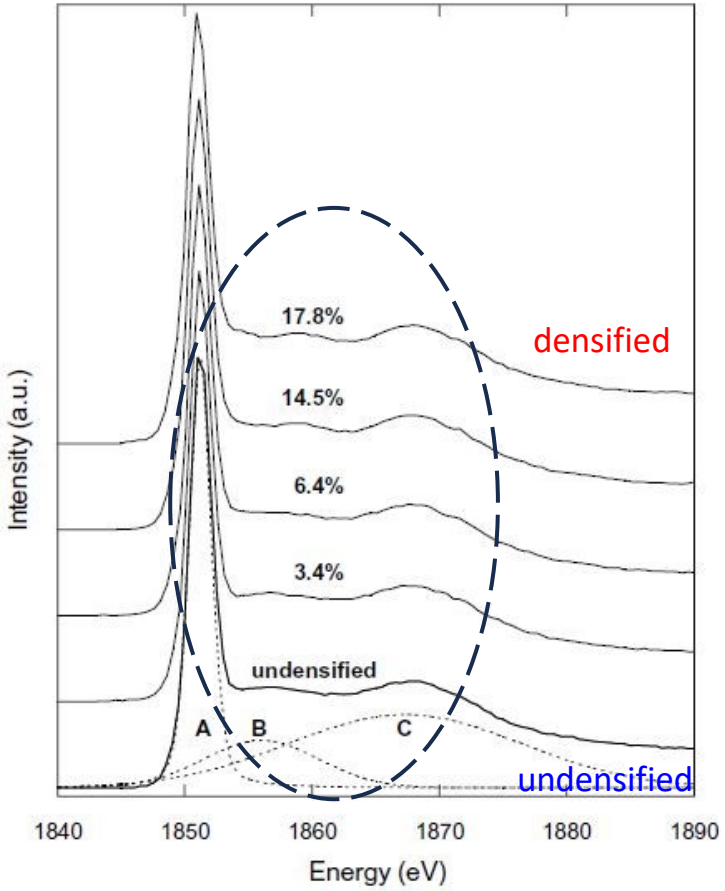
XANES



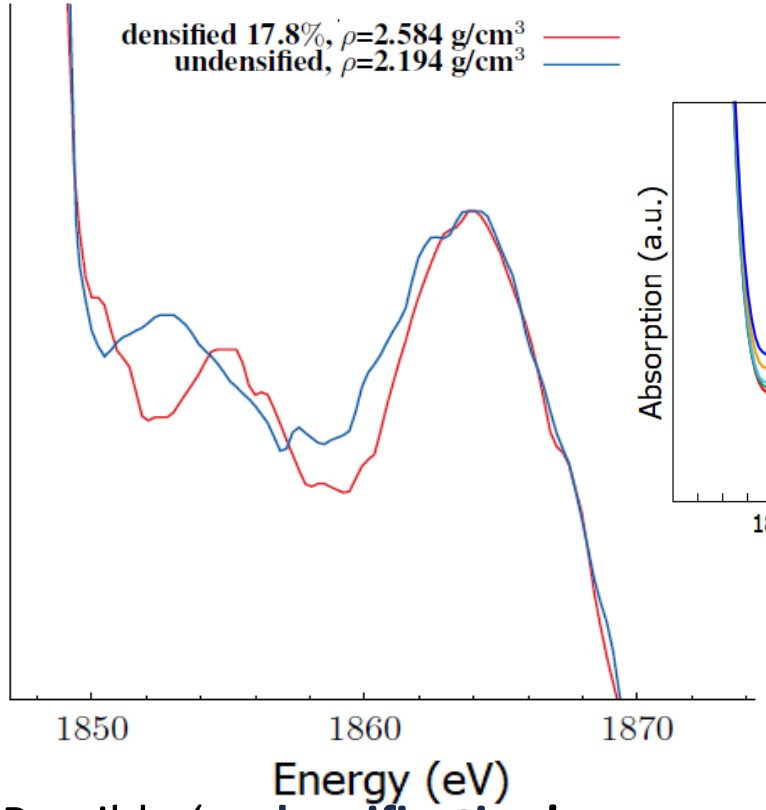


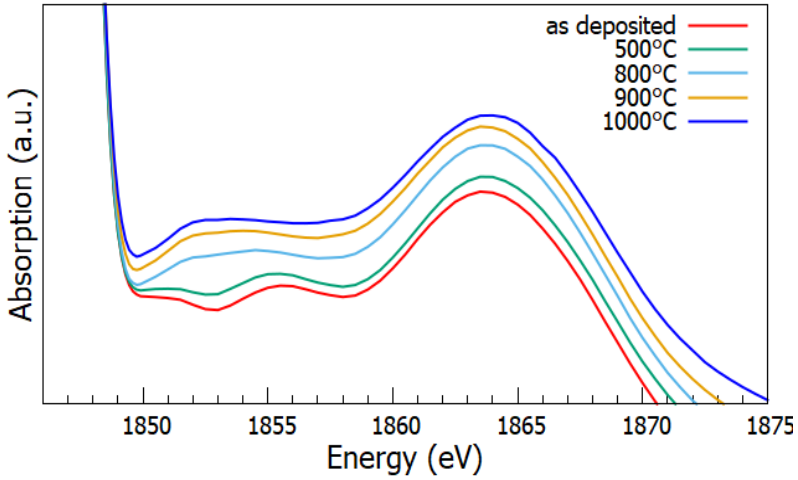
- **Shift of the white line** (~0.2 eV) at increasing annealing temperature
- **Change in post-edge region**: the two marked features merge into a single one
- Independent of the sampling depth
- **Independent of the substrate** (Si or SiO_2)

XANES: densified silica glasses



Poe B.T. et al., *Journal of Non-Crystalline Solids* 341 (2004) 162–169

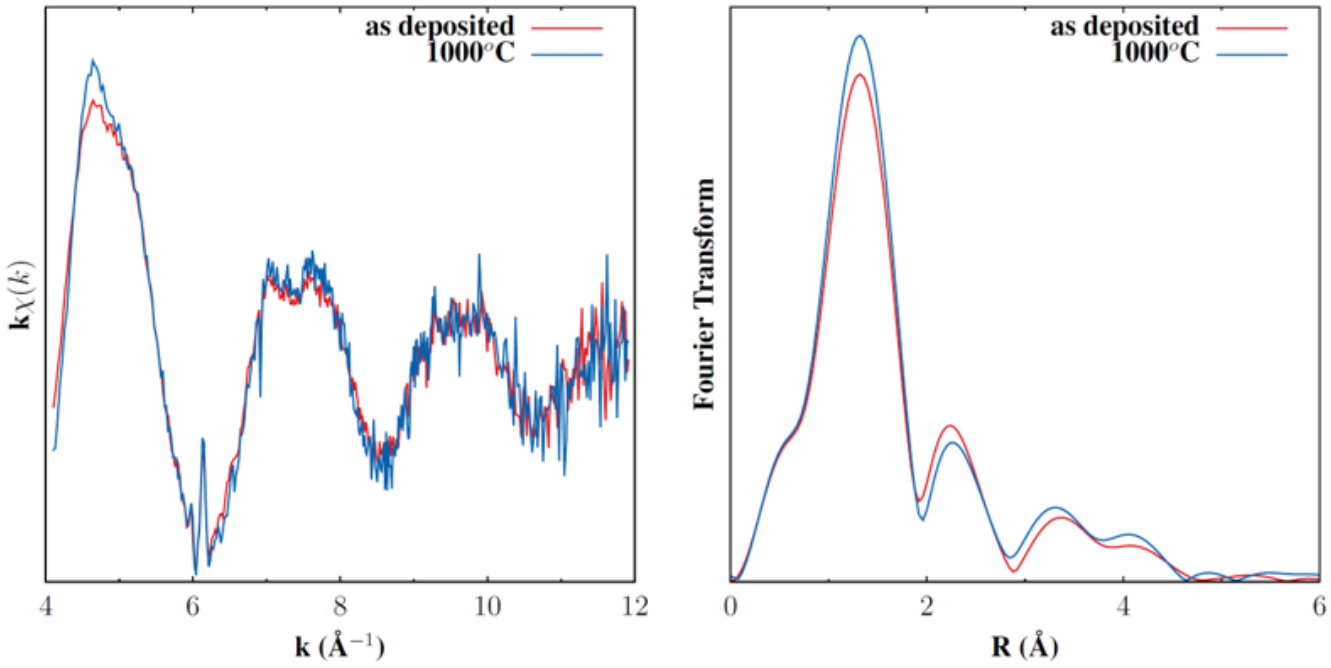




Possible '**undensification**' process associated to **medium range order** effects

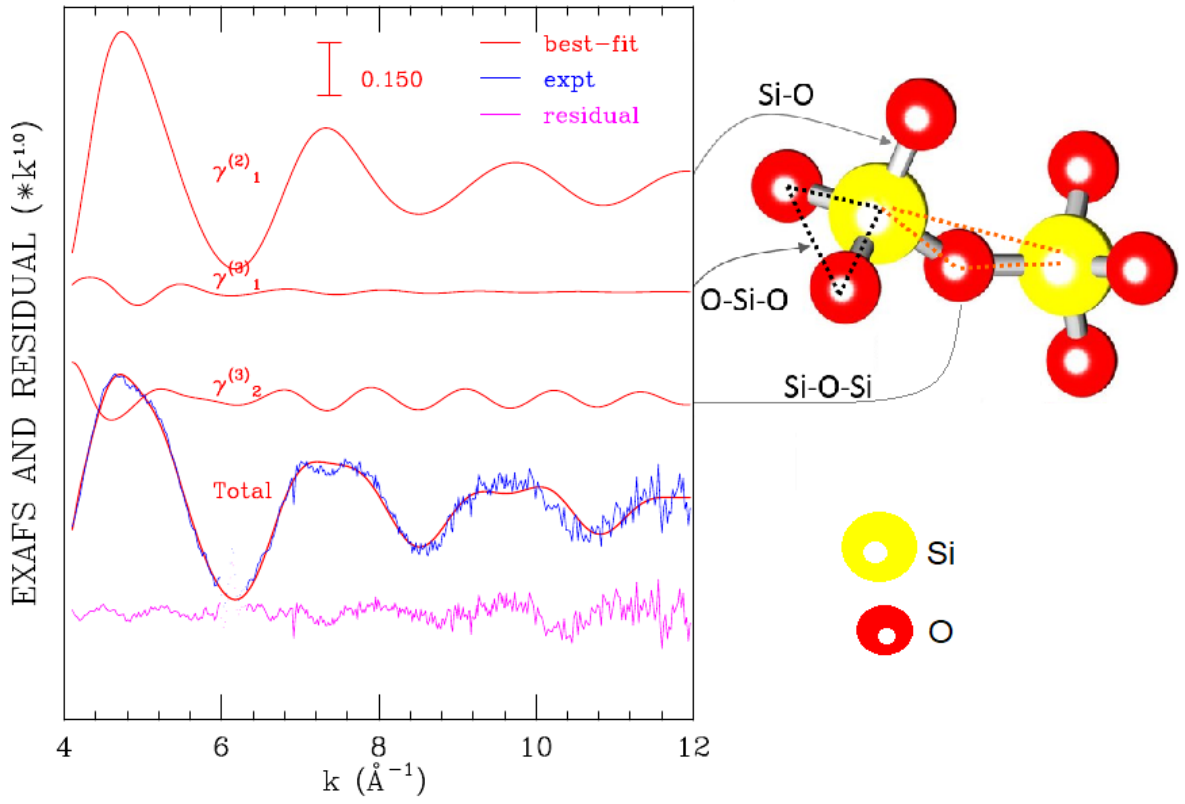
- XANES calculations for different densification levels for a complete interpretation

EXAFS



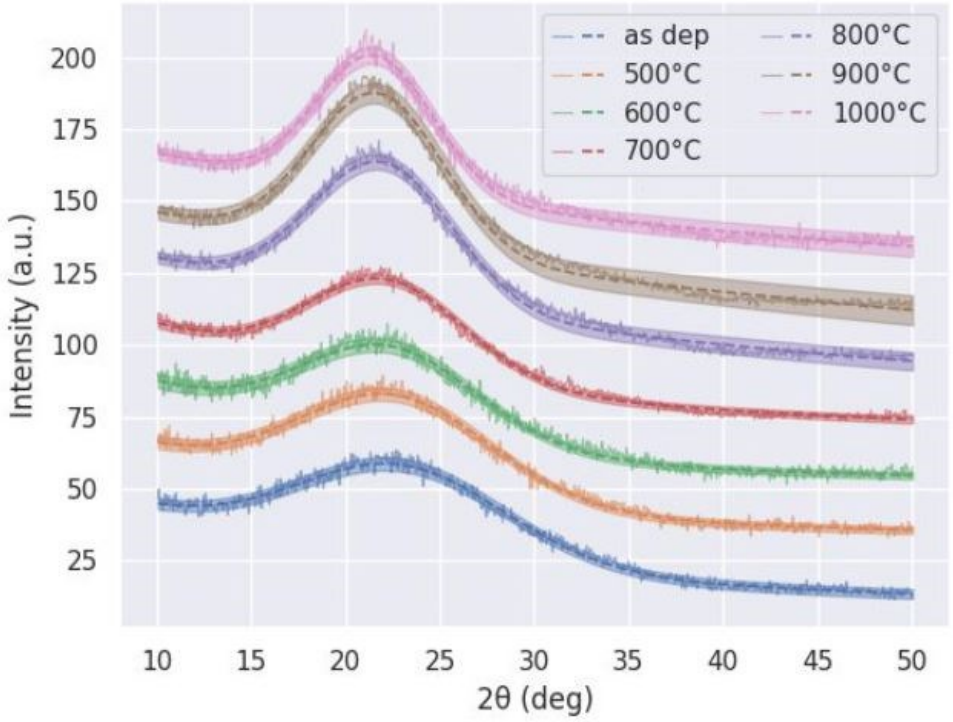
Experimental EXAFS spectra do not change in a significant way with annealing

No changes in the short range structure



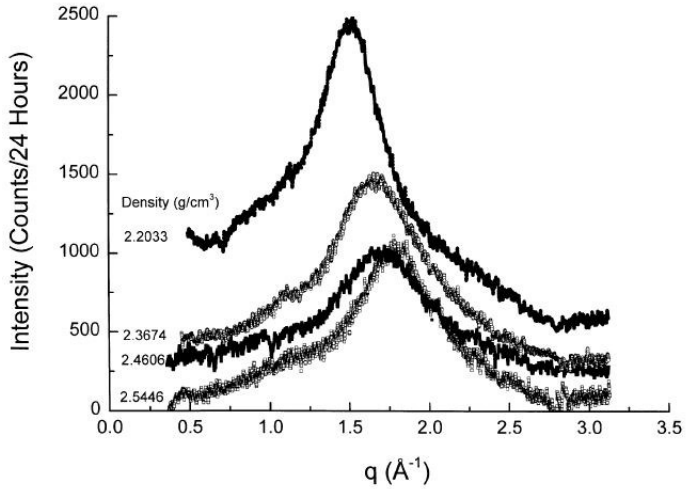
Grazing Incidence X-Ray Diffraction

M. Bazzan
G. Favaro



- Collected at University of Padova
- Cu energy (λ= 1.54 Å)
- Patterns: 2θ range 10°-50°

- No detectable peaks of SiO_2 crystalline polymorphs → samples remain amorphous after annealing

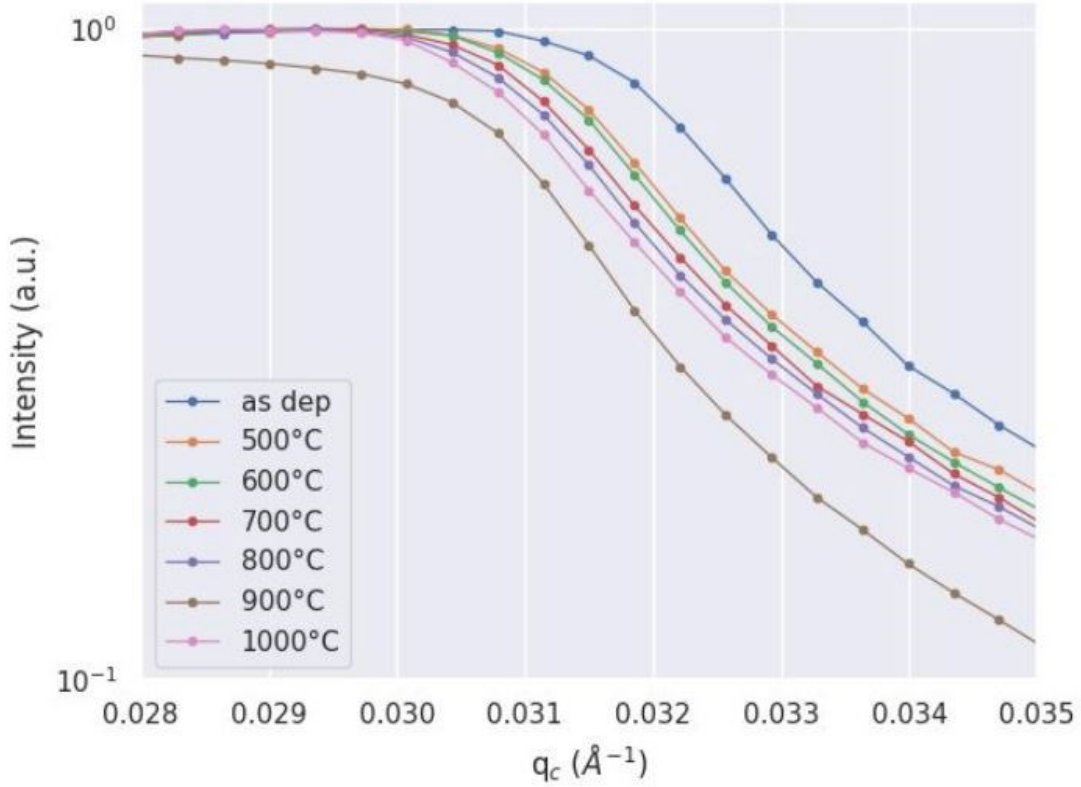


C.Z. Tan, J. Arndt, *Jour. Of Non-Cryst. Solids* ***249*** *(1999) 47-50*

- The main peak around 22° undergoes a change during annealing
- Same changes (height and peak position) versus density observed for permanently densified silica glasses

M. Bazzan
G. Favaro

X-Ray Reflectivity

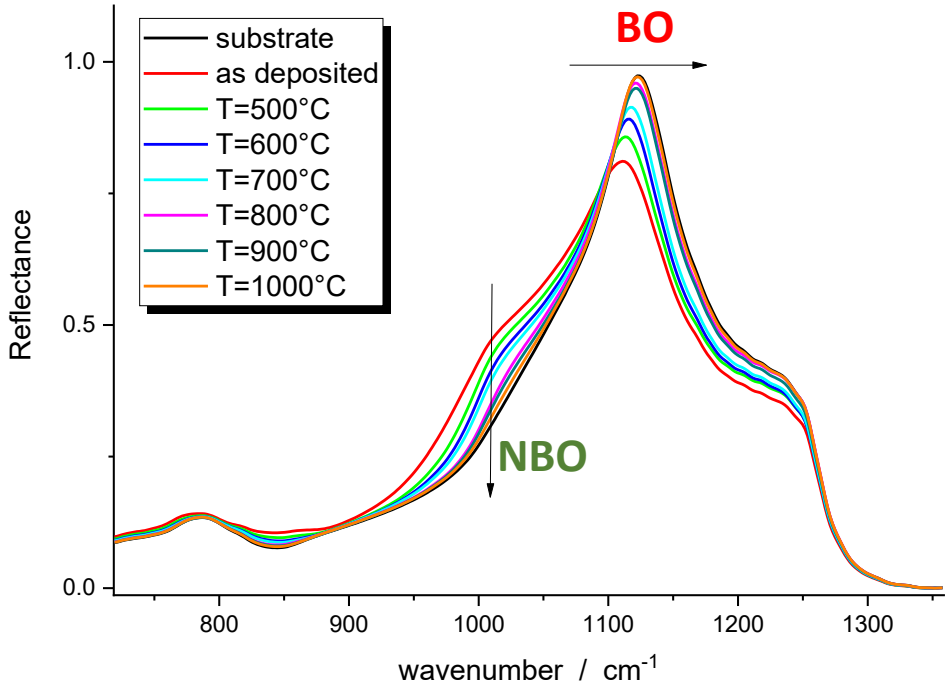


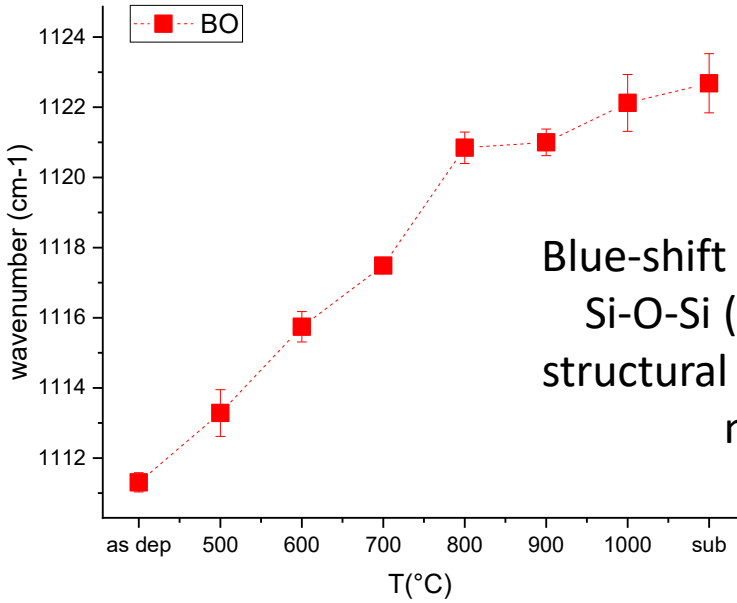
	Density [g/cm³]
As Deposited	2.376
500°C	2.291
600°C	2.278
700°C	2.251
800°C	2.234
900°C	2.212
1000°C	2.215

Density decreases

- Films too thick → only density estimation from the critical angle

Fourier Transform Infrared Spectroscopy

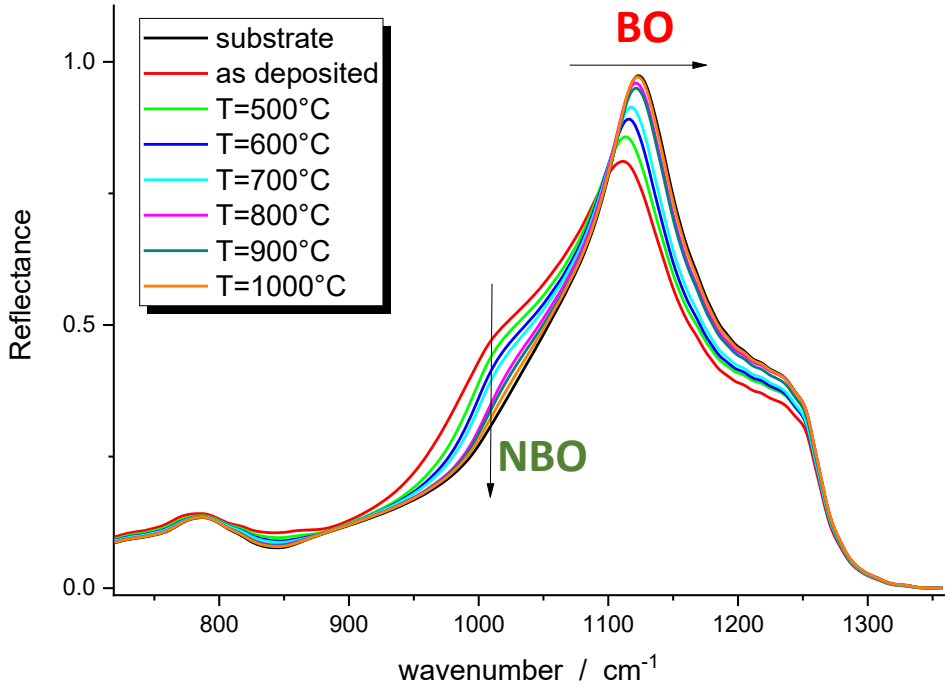


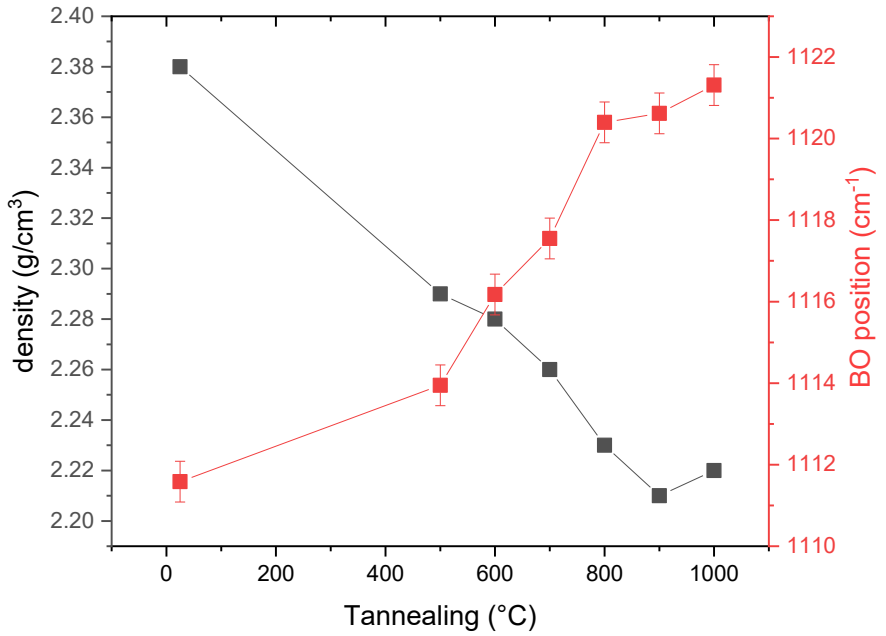


Blue-shift of antisymmetric Si-O-Si (BO) vibrations: structural variation of silica network

- Shift of the fundamental band , correlated to Si-O-Si bond angle, at higher frequencies with increasing annealing temperature → **Medium range order** structural rearrangement, evident at 800°C
- The shoulder at ~1020 cm^{-1} may suggest the presence of some structural defects (OH groups or **$Si-O^-$ non bridging oxygens**) for low annealing temperatures

Fourier Transform Infrared Spectroscopy





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Conclusions

- SiO_2 coatings have been investigated for different annealing temperatures and with several complementary techniques
- Spectral changes occur in the XANES region, associated to medium range order effects
- The local structure remains unchanged from EXAFS analysis
- No crystallization peaks of SiO_2 polymorphs with annealing, so the samples remain amorphous. Moreover, a change in the peak of amorphous SiO_2 at 22° is observable
- All the samples exhibit a decrease of the density with respect to the annealing temperature, with XRR
- Medium range order rearrangement is observable from FTIR. The presence of possible defects is under study

Less dense structure at increasing annealing temperature
Local structure unchanged

Collaborators

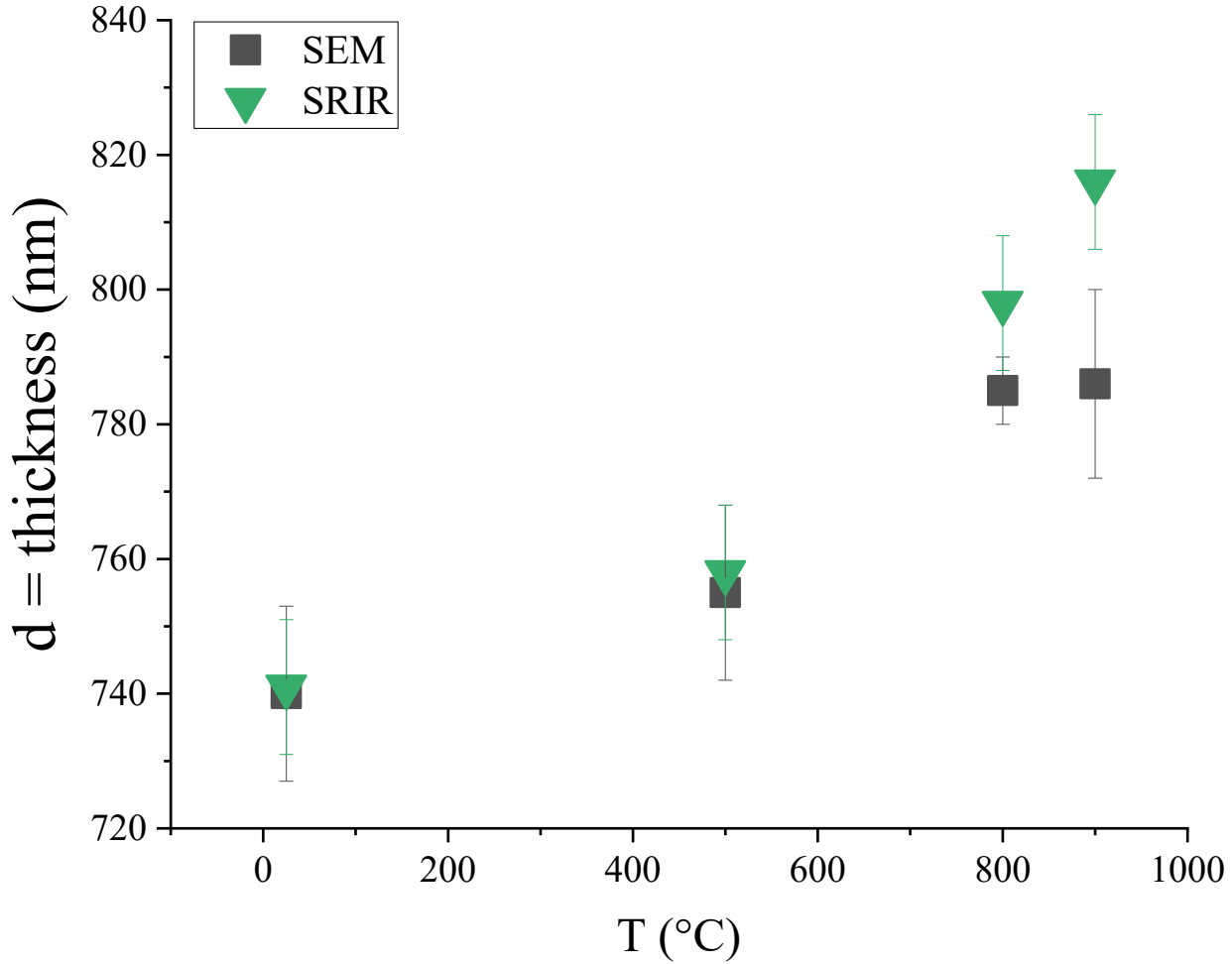
A. Trapananti
F. Travasso

B. Bracco
S. Corezzi
A. Di Michele
P. Sassi
H. Vocca

M. Bazzan
G. Favaro

M. Granata
C. Michel
L. Pinard

G. Cagnoli


SEM
SRIR
d = thickness (nm)
840
820
800
780
760
740
720
0
200
400
600
800
1000
T (°C)