

# Overview of Critical Dimension Small Angle X-ray Scattering (CD-SAXS)

Guillaume Freychet

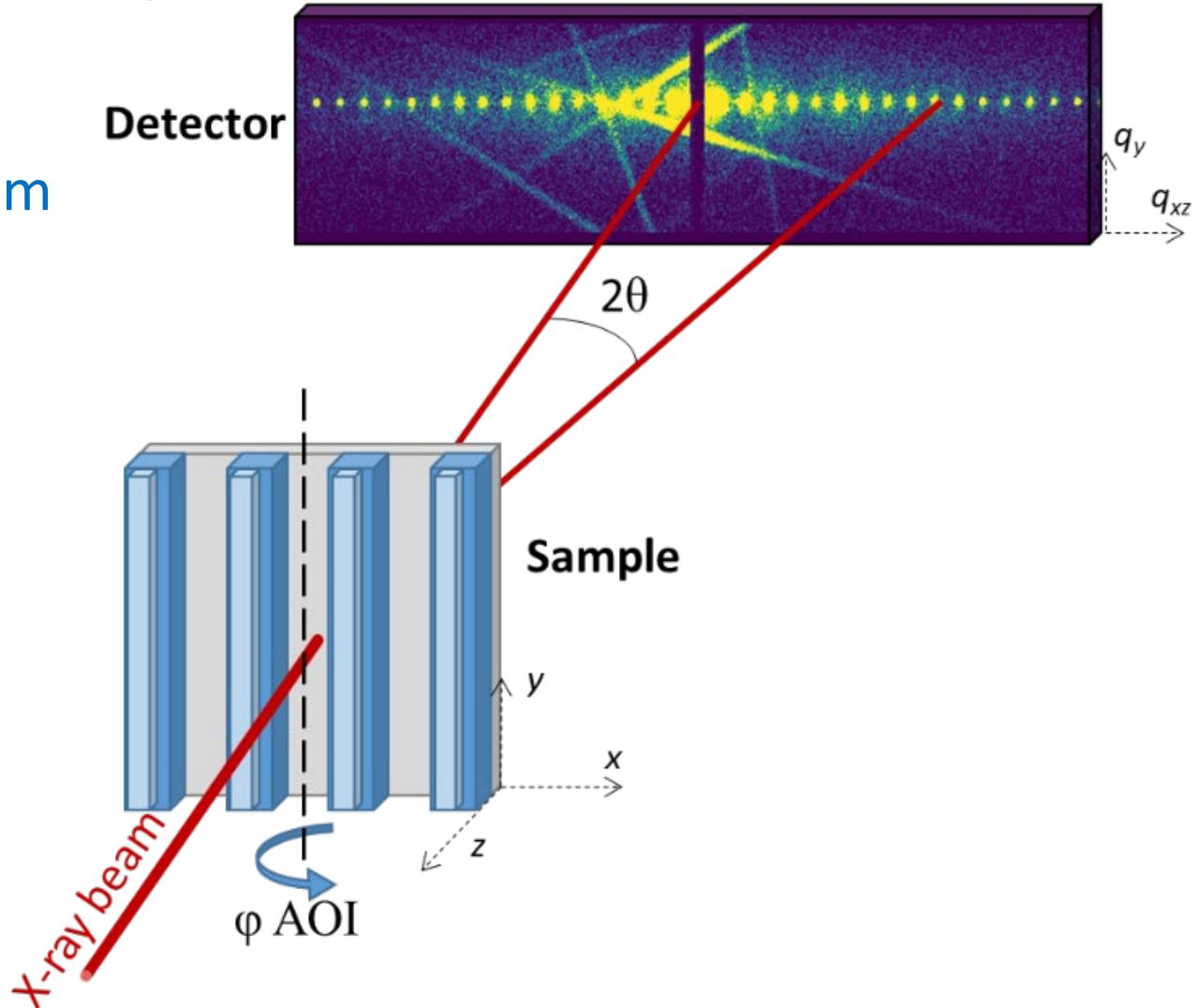
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# Small Angle x-ray scattering (SAXS)

- ✓ X-ray => Wavelength from  $\sim 0.1$  nm
- ✓ Small angle scattering: probe 0.1-1000 nm

$$q = \frac{2\pi}{\lambda} \sin 2\theta$$

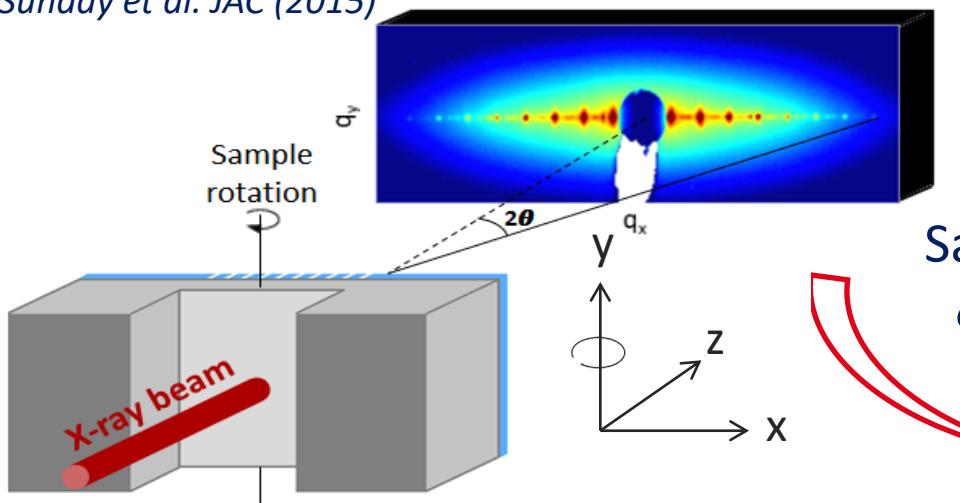
- ✓ Probe electronic density contrast
- ✓ Transmission geometry
- ✓ Indirect technique
- ✓ Averaging over the beam



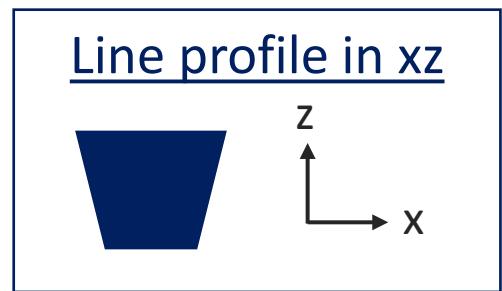
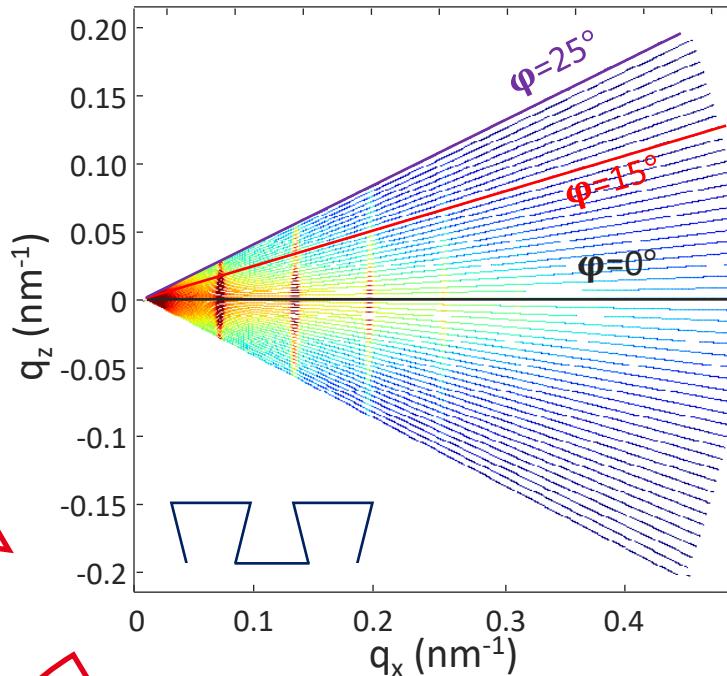
# CD-SAXS : principle



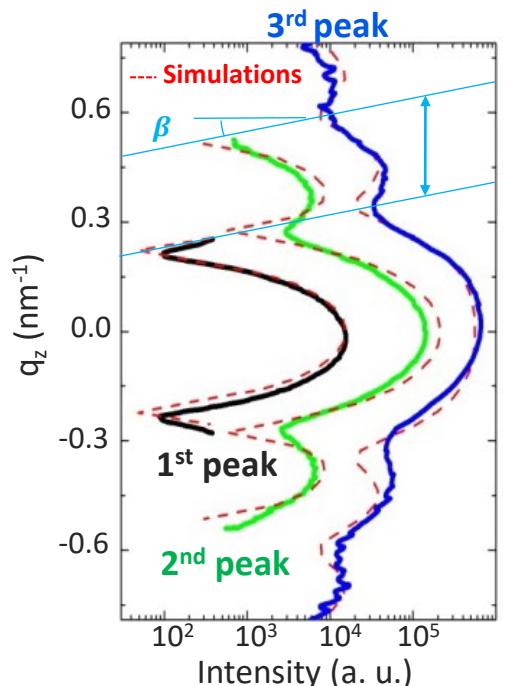
D. Sunday et al. JAC (2015)



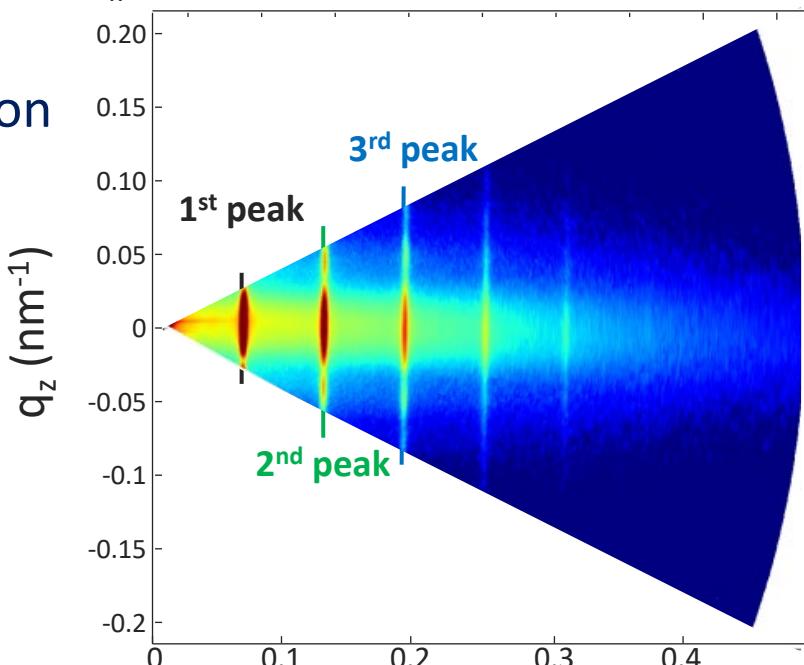
Sample rotation  
 $\varphi: -25 \Rightarrow 25^\circ$



Fit the experimental data



Interpolation  
1D Vertical cut  
(Extraction of the form factor)



# Python package for CD-SAXS (Xicam)



*Collaboration with NIST (J. Kline, D. Sunday, C. Liman, D. Delongchamp)*



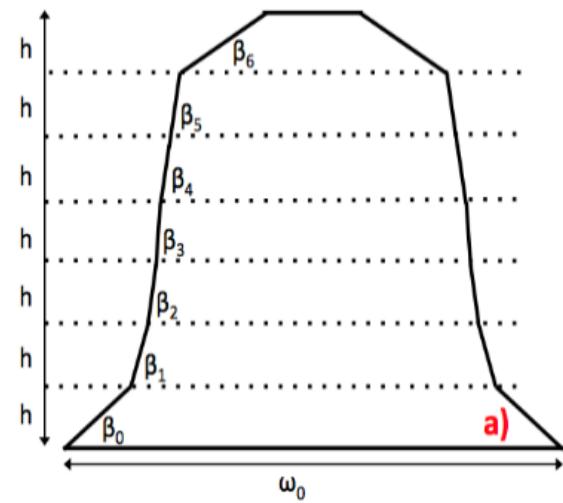
R. Pandolfi



D. Kumar



# CD-SAXS : principle



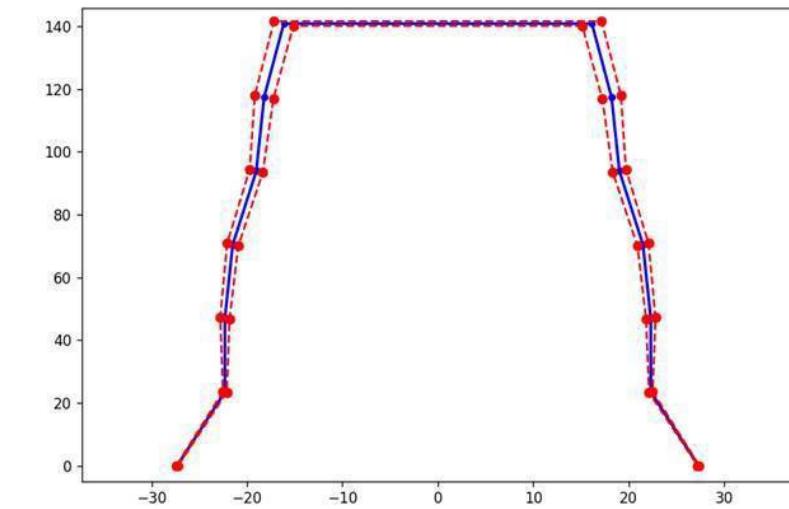
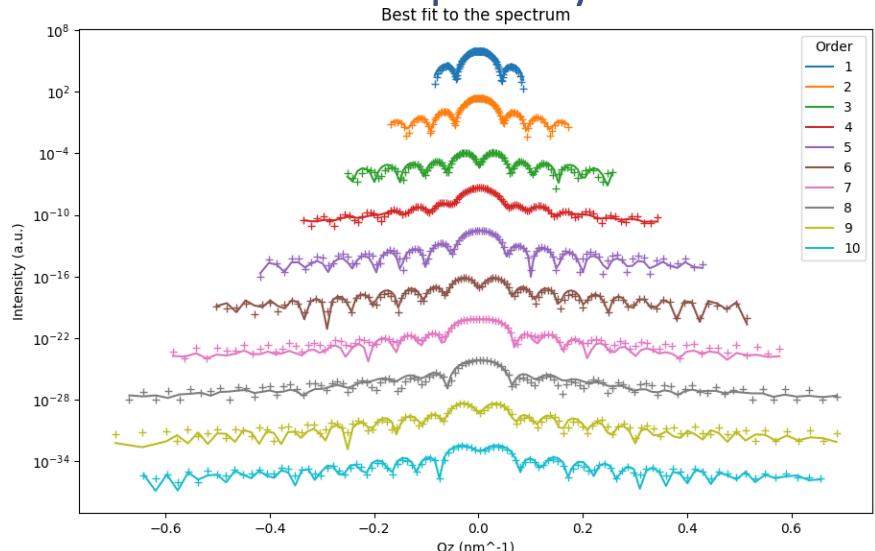
✓ Fourier transform of a trapezoid ( $L = \omega_0$  and  $m = \tan \beta$ ):

$$F(q_y, q_z) = \frac{1}{q_y} \left[ -me^{jh(q_y L/2)} \left( 1 - e^{-jh[(q_y + m q_z)/m]} \right) + me^{-jh(q_y L/2)} \left( 1 - e^{-jh[(q_y + m q_z)/m]} \right) \right],$$

✓ Minimize the difference between  $I_{exp}$  and  $I_{sim} = |F(q_y, q_z)|^2$

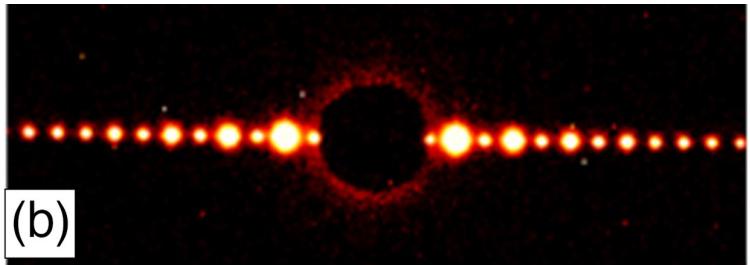
Genetic algorithm to converge to the best profile

Monte-Carlo Markov Chain to quantify the uncertainty on each profile



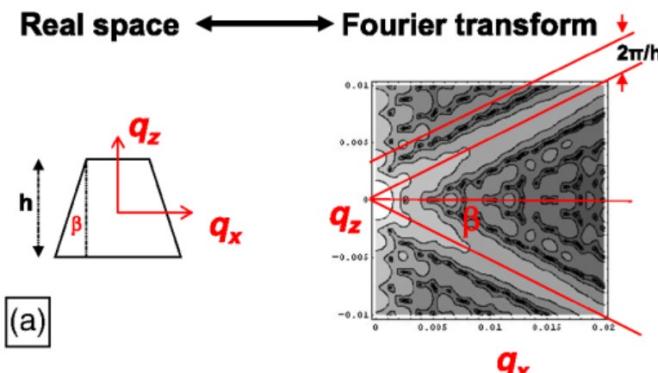
# CD-SAXS : History

- ✓ Early 2000s: First measurements at APS by NIST

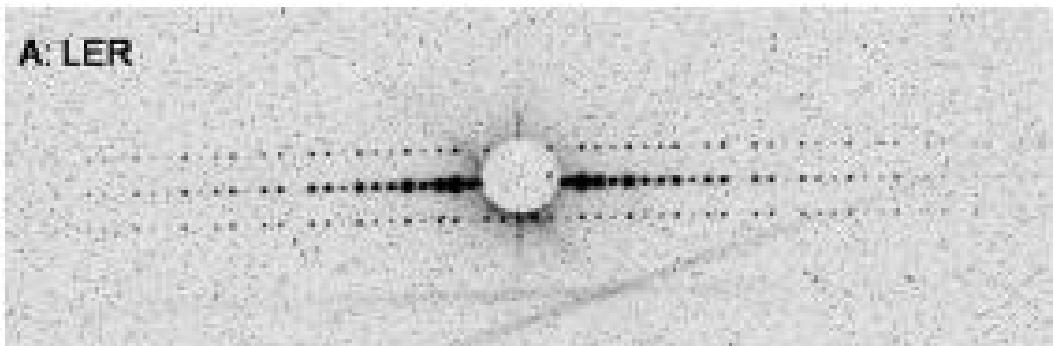


W-L. Wu et al. JM3, Vol. 22, Issue 3, 031206 (2023)

- ✓ 2000-2013: Exploration of possibilities with CD-SAXS: Sidewall angle, roughness, ...



W-L. Wu et al. JM3, Vol. 22, Issue 3, 031206 (2023)



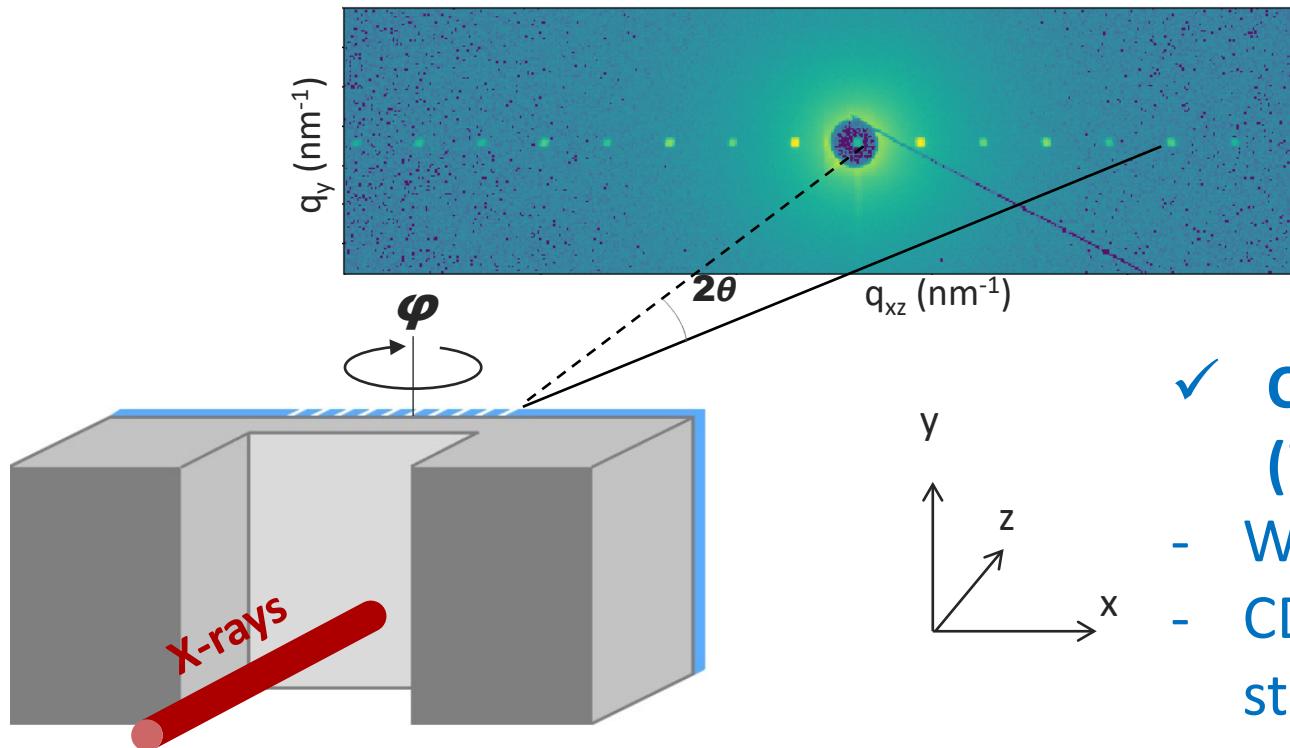
Wang et al. J. Appl. Phys. 102, 024901 (2007)

- ✓ 2013-2020:
  - Strengthen the robustness of the analysis (genetic algorithm)
  - Test measurements with laboratory sources
  - Explore more complex system (BCP polymers, contacts, ...)



<https://www.nist.gov/programs-projects/metrology-nanolithography>

# CD-SAXS: Advantages and drawbacks



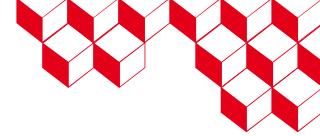
- ✓ Provide the line profile with a sub-nm resolution
- ✓ Challenges link to the transmission geometry (i.e. cross a silicon wafer):
  - Work well with synchrotron
  - CD-SAXS in-line equipment => focused on HAR structure
  - Requires significant improvement of laboratory high energy source in term of flux

## Measurement in grazing incidence: CD-GISAXS



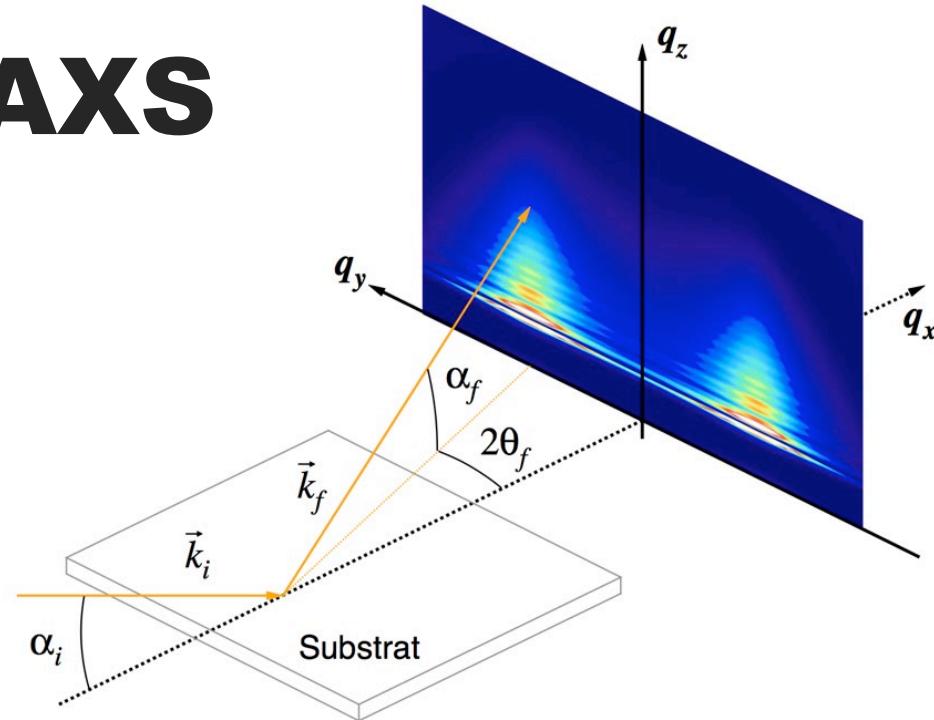
# 2.

## Critical Dimension Grazing Incidence SAXS (CD-GISAXS)



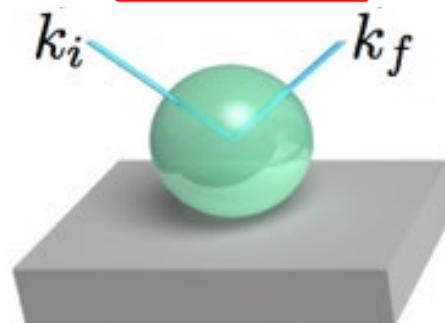
# Grazing-incidence SAXS (GISAXS)

- ✓ Grazing incidence geometry
- ✓ Thin films
- ✓ Large footprint



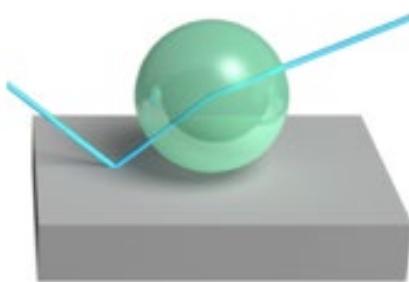
## Distorted Wave Born Approximation (DWBA)

$$\mathcal{F}(q_{||}, k_z^i, k_z^f) = \boxed{F(q_{||}, q_z^1)} +$$



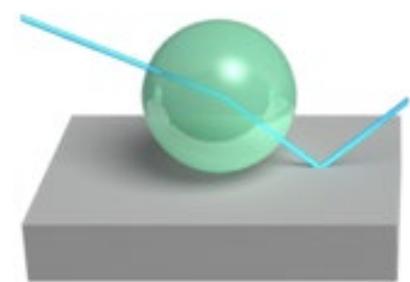
$$\vec{q}_1 = \vec{k}_f - \vec{k}_i$$

$$+ \boxed{r(\alpha_f)F(q_{||}, q_z^2)}$$



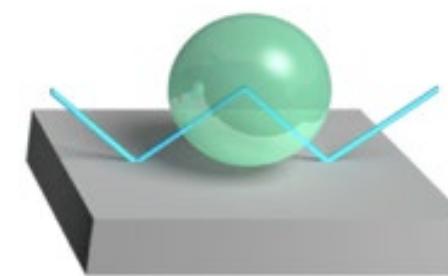
$$\vec{q}_3 = \vec{k}_f + \vec{k}_i$$

$$+ \boxed{r(\alpha_i)F(q_{||}, q_z^3)}$$



$$\vec{q}_2 = -\vec{k}_f - \vec{k}_i$$

$$+ \boxed{r(\alpha_i)r(\alpha_f)F(q_{||}, q_z^4)}$$



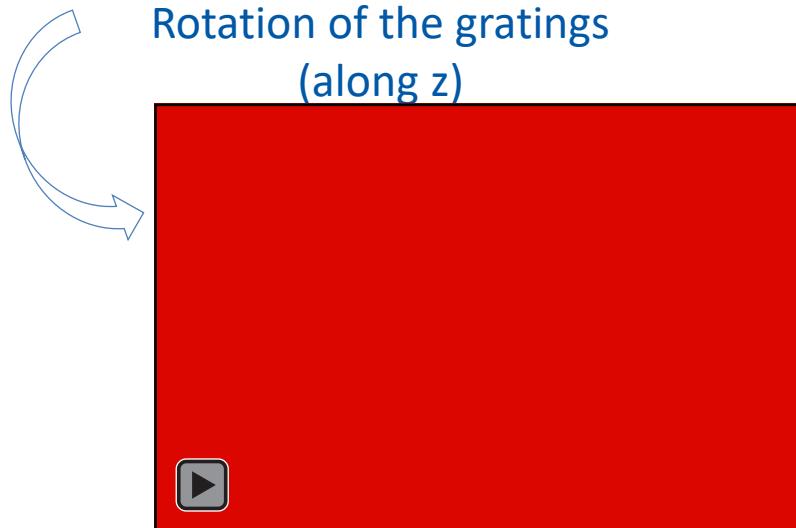
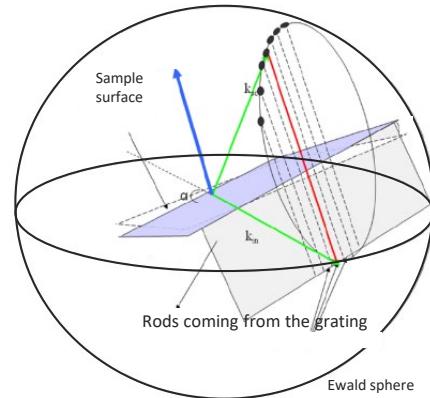
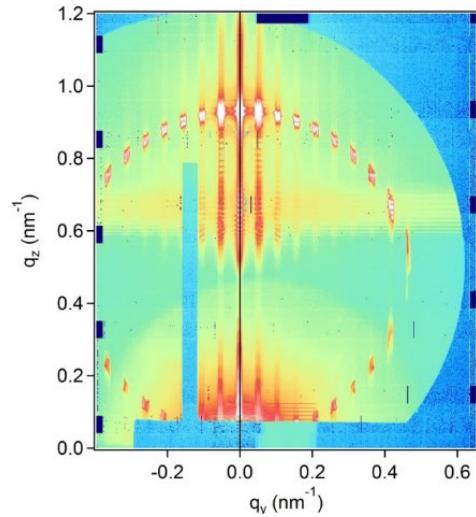
$$\vec{q}_4 = -\vec{k}_f + \vec{k}_i$$

# Critical Dimension GISAXS (CD-GISAXS)

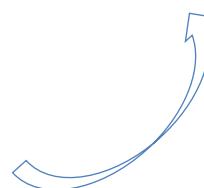
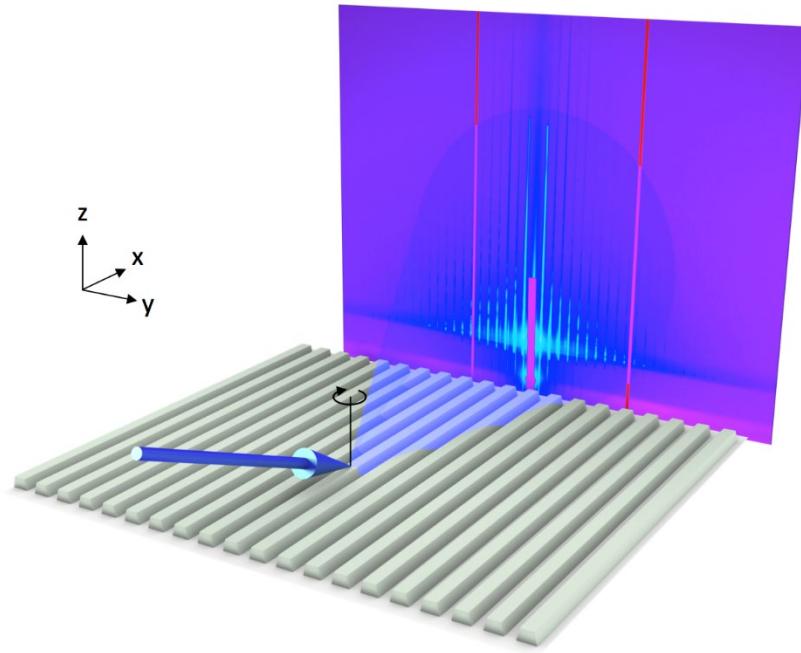
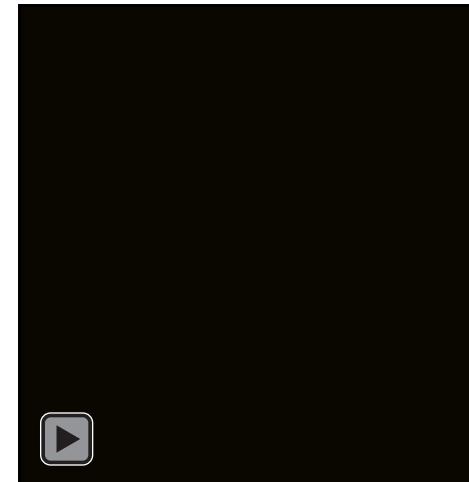
Hofmann et al. J. Vac. Sci. Tech. (2009)

Lu et al. App. Cryst. (2012)

Suh et al. J. Appl. Cryst. (2016)

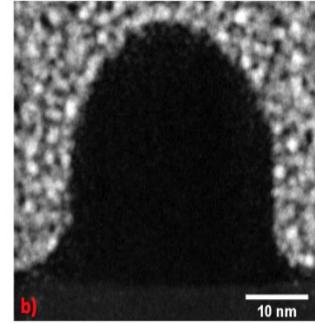
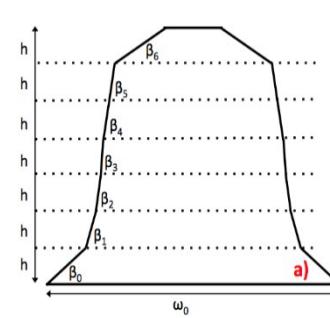


Rotation of the gratings  
(along z)



Full intersection of the Bragg rods  
and the Ewald sphere recorded

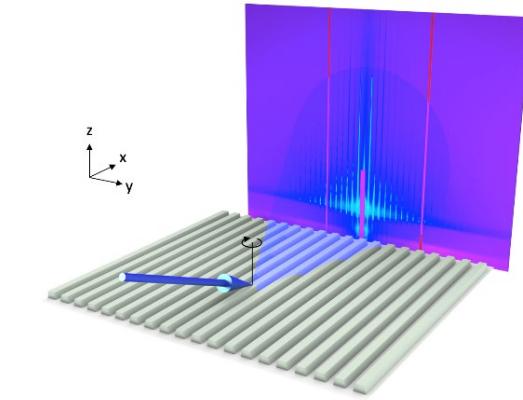
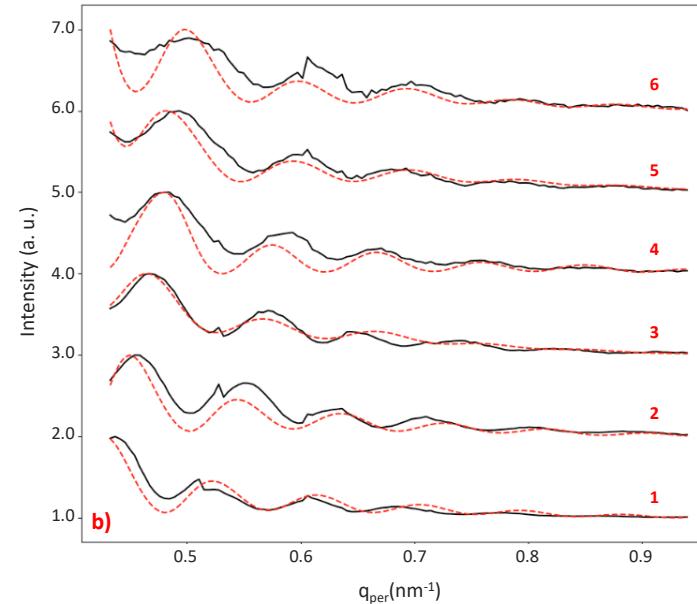
# Critical Dimension GISAXS (CD-GISAXS)



Good agreement between the cross-section TEM and the CD-GISAXS

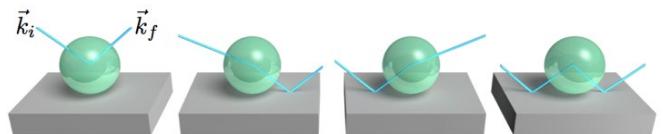
Freychet et al. Proc. SPIE (2018)

Freychet et al. Phys. Rev. Appl. (2019)

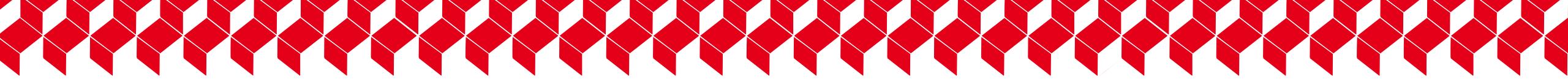


1D vertical cut

Distorted Wave Born Approximation



- ✓ Complexity of the model
- ✓ Bigger footprint on the sample
- ✓ No need to cross a silicon wafer => doable with a laboratory / Soft x-ray sources



# 3. CD-GISAXS with a Laboratory equipment

# CD-GISAXS with a Laboratory equipment



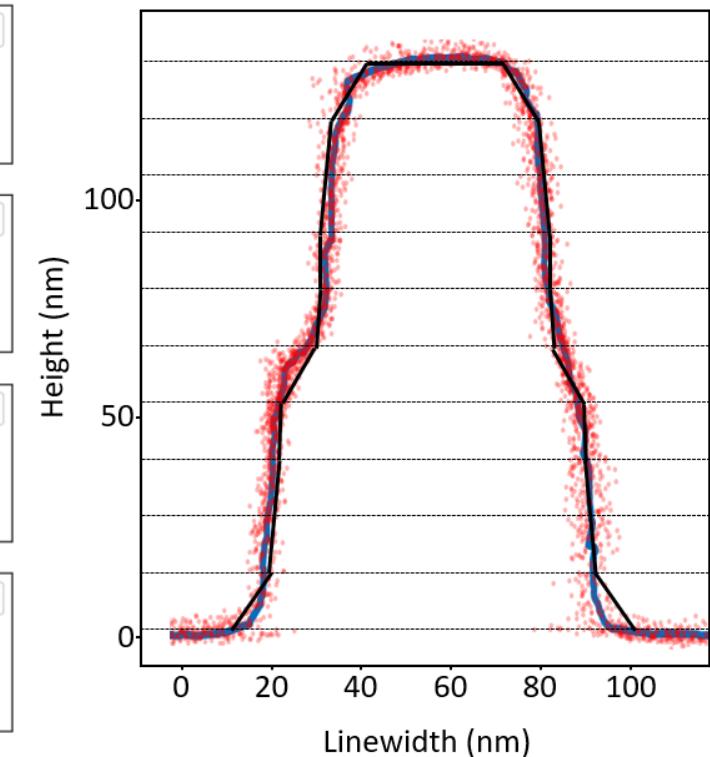
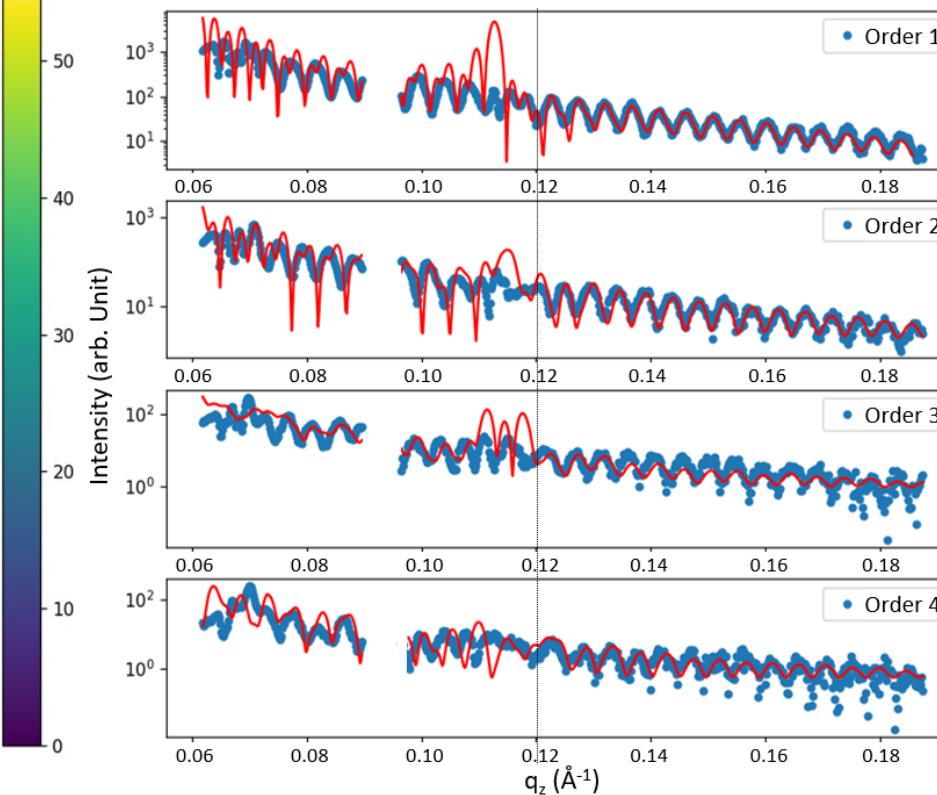
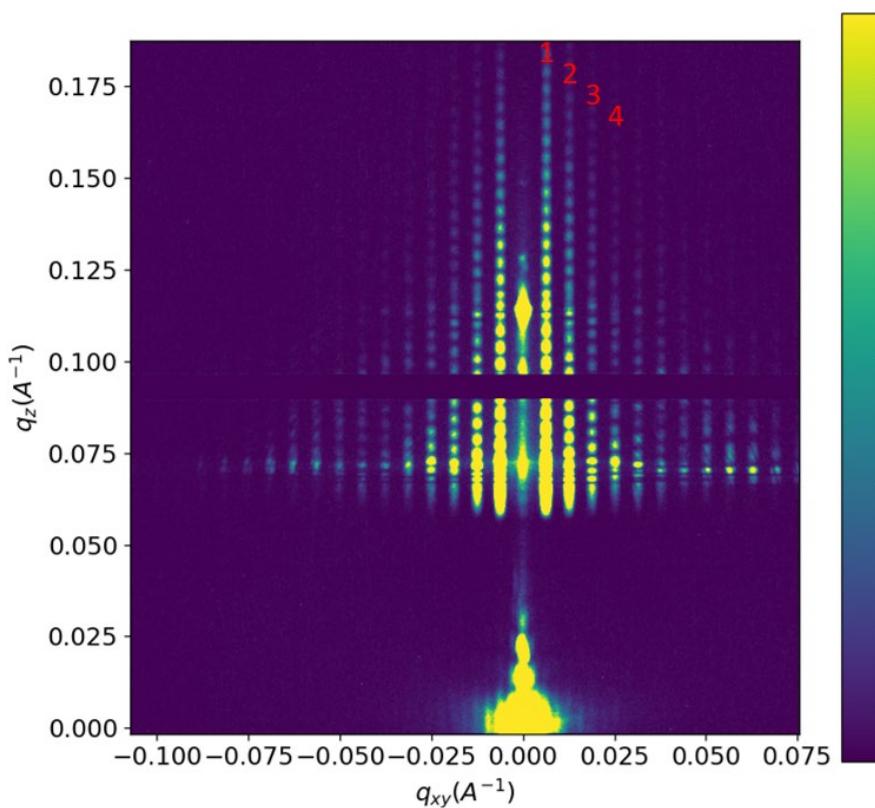
- ✓ Source Cu K- $\alpha$  (8.047 keV)
- ✓ Sample to detector distance: 1.8 m
- ✓ Transmission and Reflection

*Freychet et al. JM3 2023*

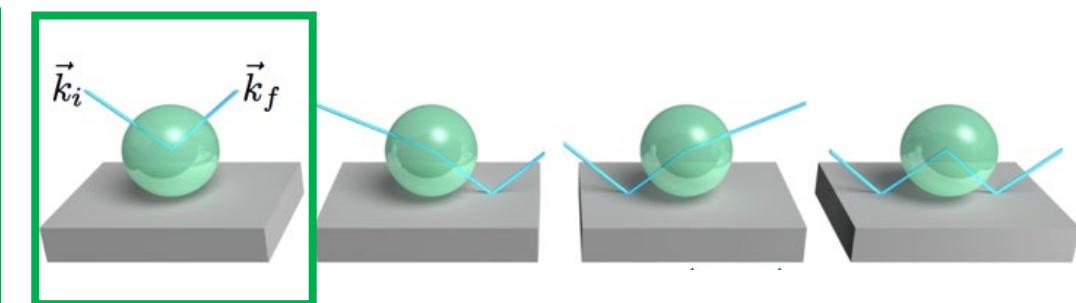
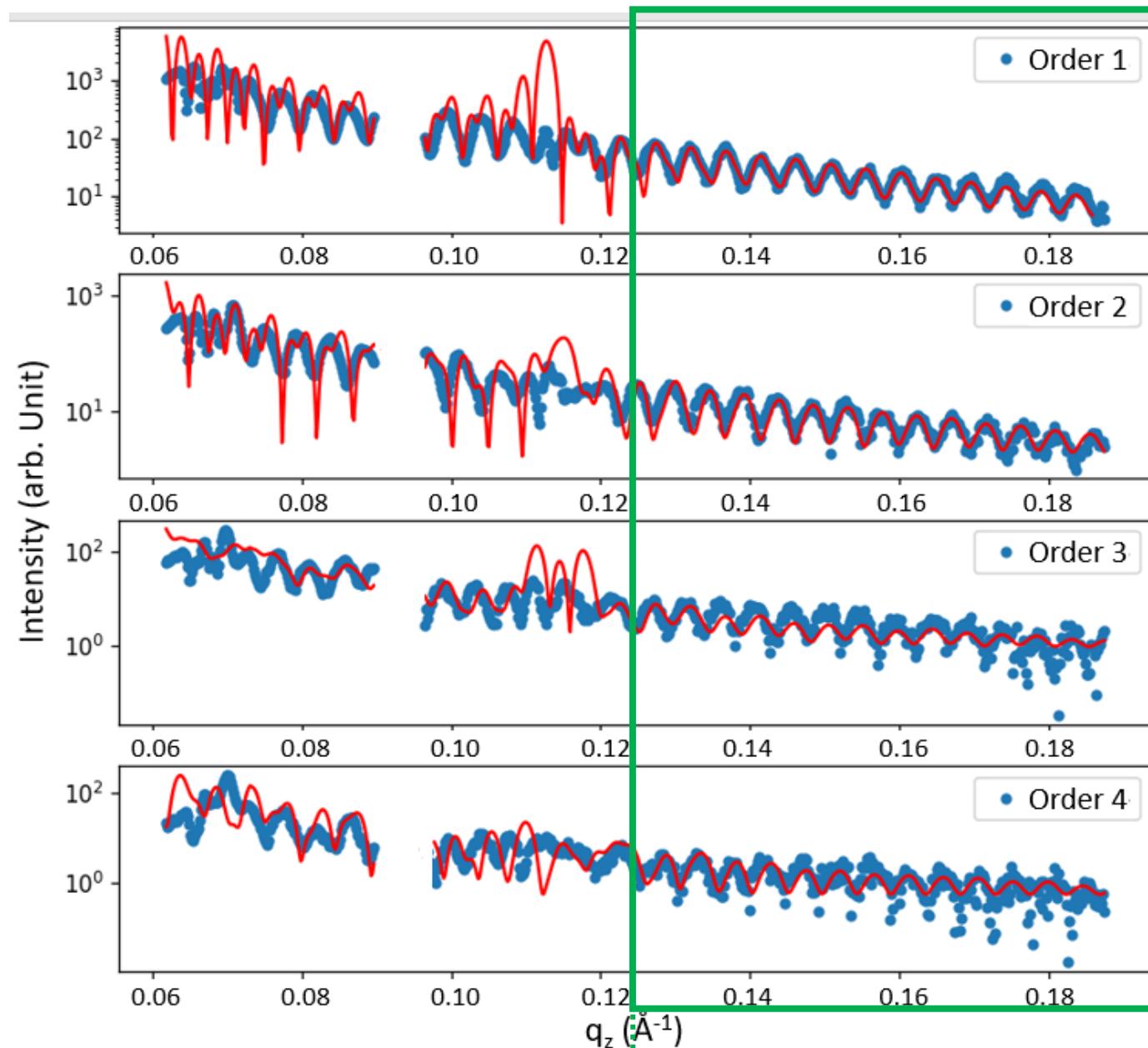
# CD-GISAXS with a Laboratory equipment



Freychet et al. JM3 2023



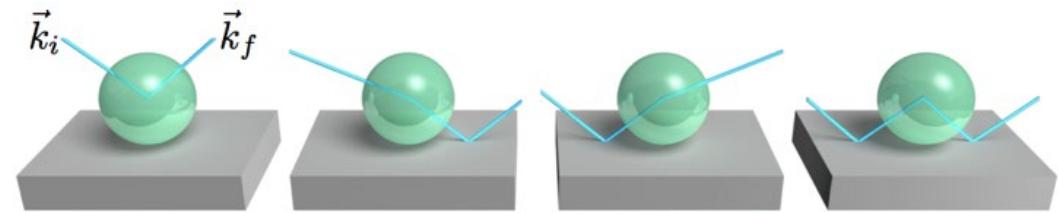
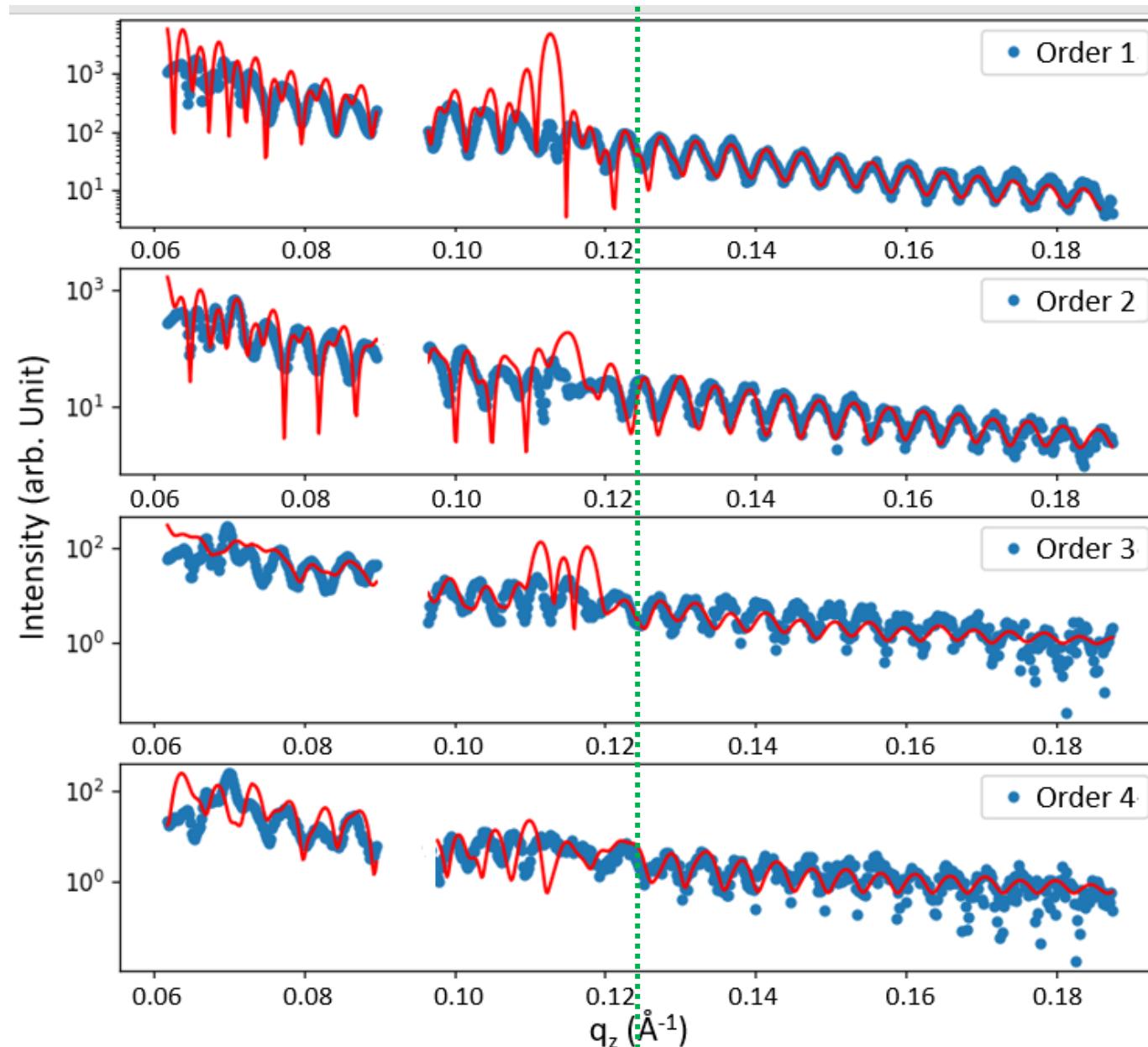
# CD-GISAXS with a Laboratory equipment



DWBA:

✓  $\alpha_i$  and  $\alpha_f > 3 \times \alpha_c$ : minimize the multi-reflection effect

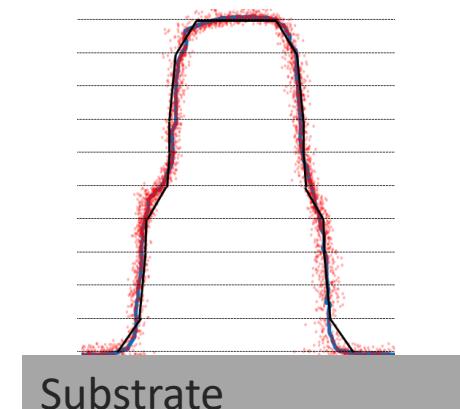
# CD-GISAXS with a Laboratory equipment



DWBA:

✓  $\alpha_i$  and  $\alpha_f > 3 \times \alpha_c$ : minimize the multi-reflection effect

✓ Multi-reflection

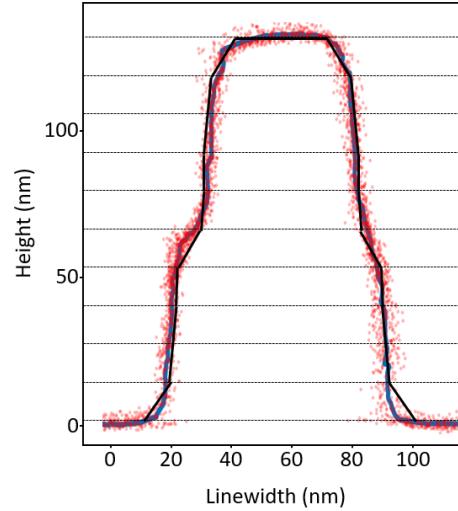


Substrate

Perfect interface (No roughness)

# CD-GISAXS: Advantages and drawbacks

- ✓ CD-GISAXS brings a sub-nm resolution
- ✓ In grazing incidence configuration, Cu K- $\alpha$  x-ray source can be used



- ✓ Quick measurements (currently 10s minutes with a standard micro-source Cu K- $\alpha$  source)
- ✓ Large footprint (couple mms)
- ✓ HAR samples



# 4. CD-GISAXS with lower energy source



# CD-GISAXS with a soft x-ray source

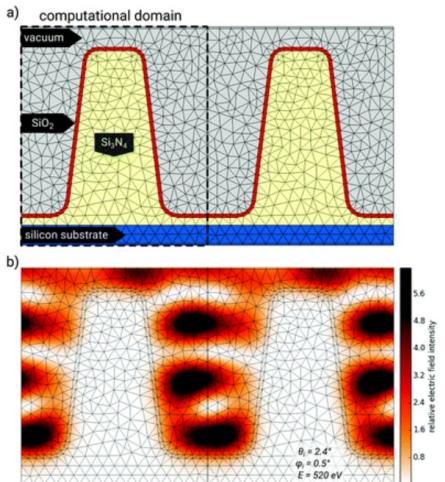
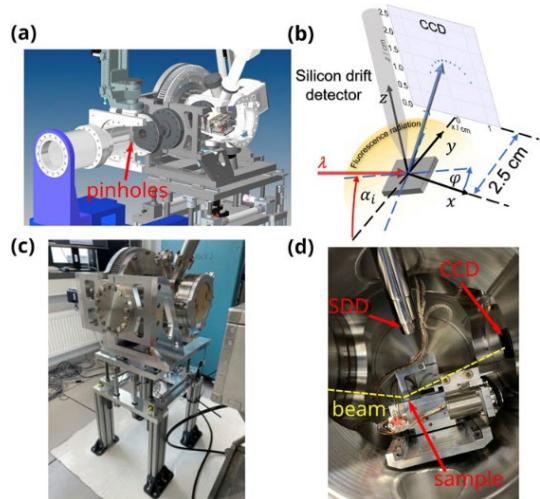
Hard x-ray (8 keV)

$$\alpha_c(\text{Si}) = 0.2 \text{ deg}$$

$$\text{Beam footprint } \propto 1/\sin(\alpha_i) \propto 200$$

PTB is working with EUV source since 2014

- ✓ Synchrotron device
- ✓ Major development in terms of analysis software



cea

Soft x-ray (200 eV):

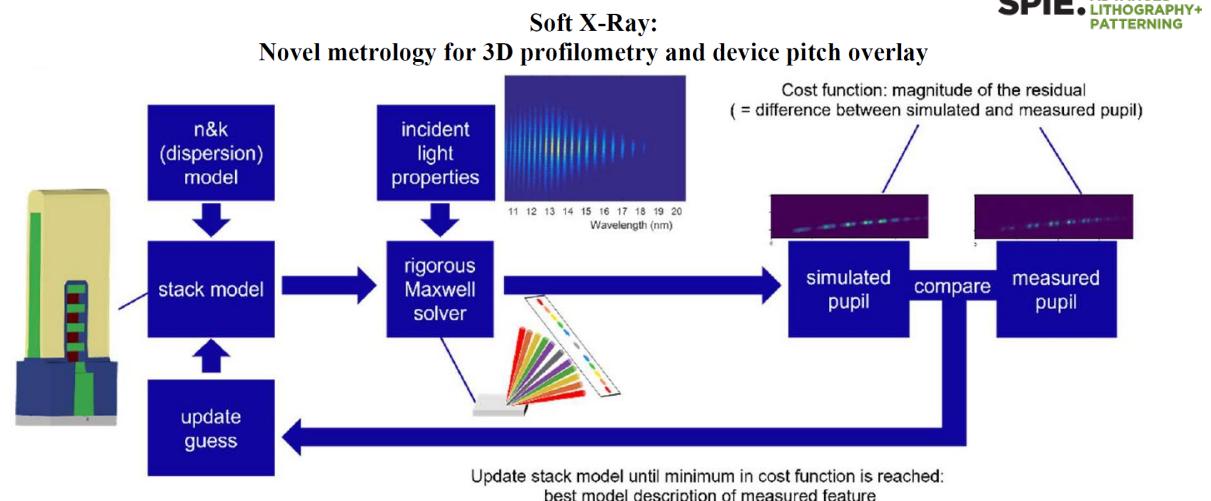
$$\alpha_c(\text{Si}) = 10 \text{ deg}$$

$$\text{Beam footprint } \propto 1/\sin(\alpha_i) \propto 4$$

**No footprint issue**

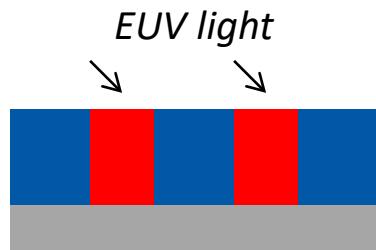
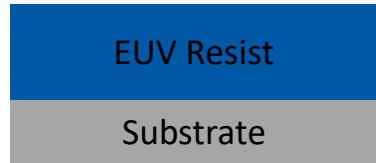
ASML (C. Porter) proposed the 1<sup>st</sup> laboratory equipment results in 2023 using HHG source

- ✓ Footprint < 50 μm
- ✓ Acquisition time around 200 s

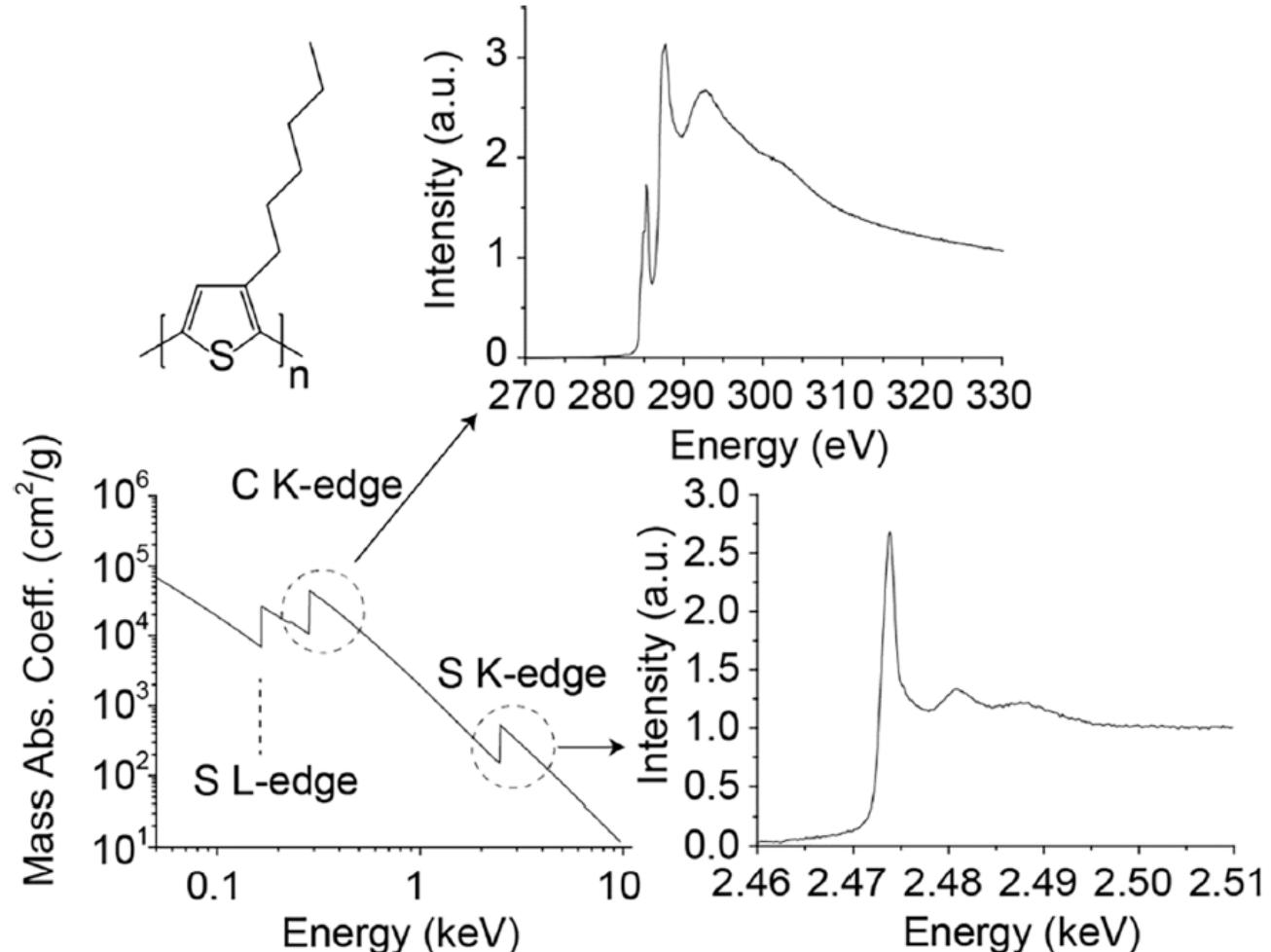


# Resonant Soft X-Ray Scattering (RSoXS)

EUV lithography process simplified



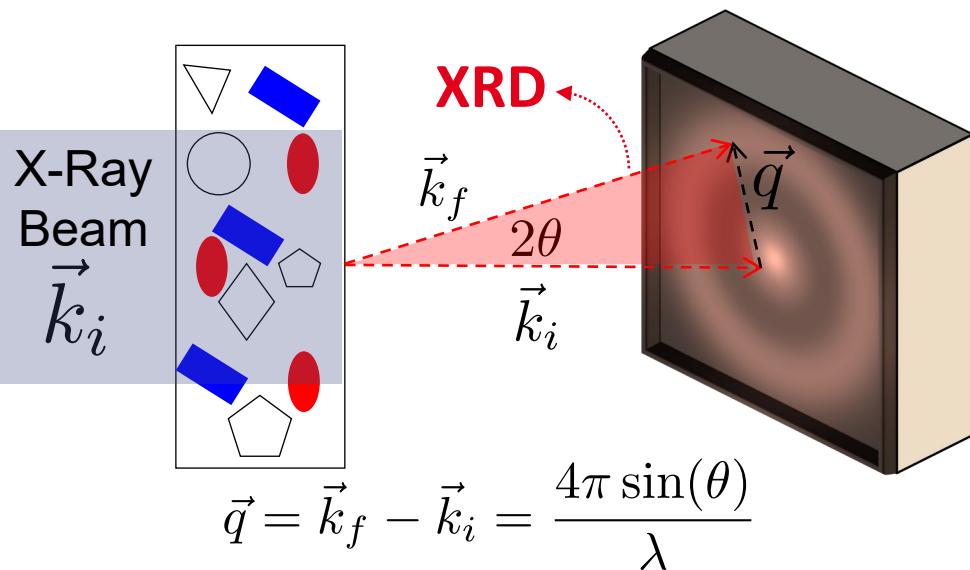
Latent imaging => Electronic density contrast between exposed and unexposed resist?



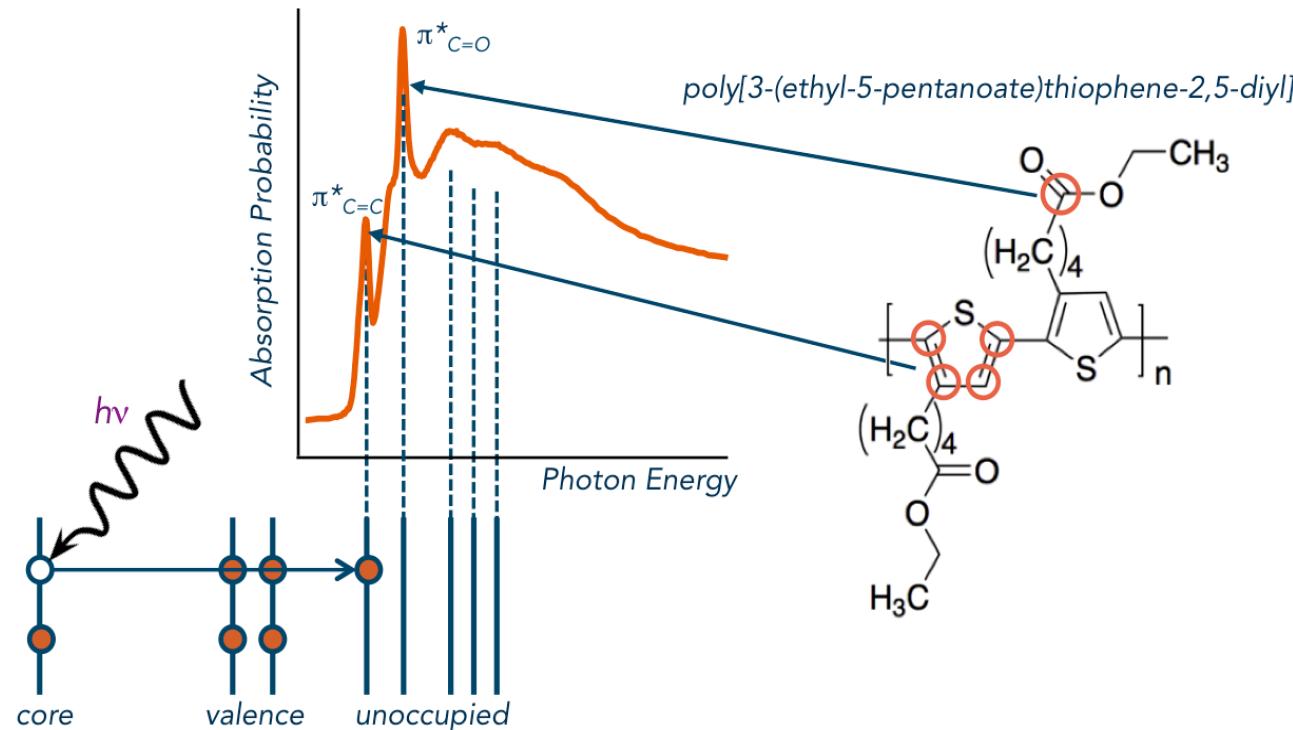
# Resonant Soft X-Ray Scattering (RSoXS)

*Resonance (Anomalous) Effect  $f = f_0 + f' + i\beta$*

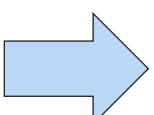
## Small Angle X-Ray Scattering



## X-ray Absorption Spectroscopy ( $\beta$ )



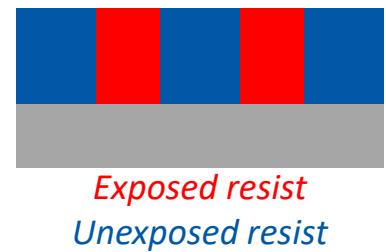
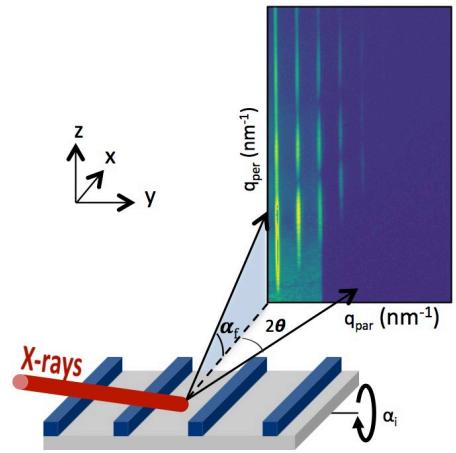
**Refractive Index**  
 $n(E) = 1 - \delta + i\beta$



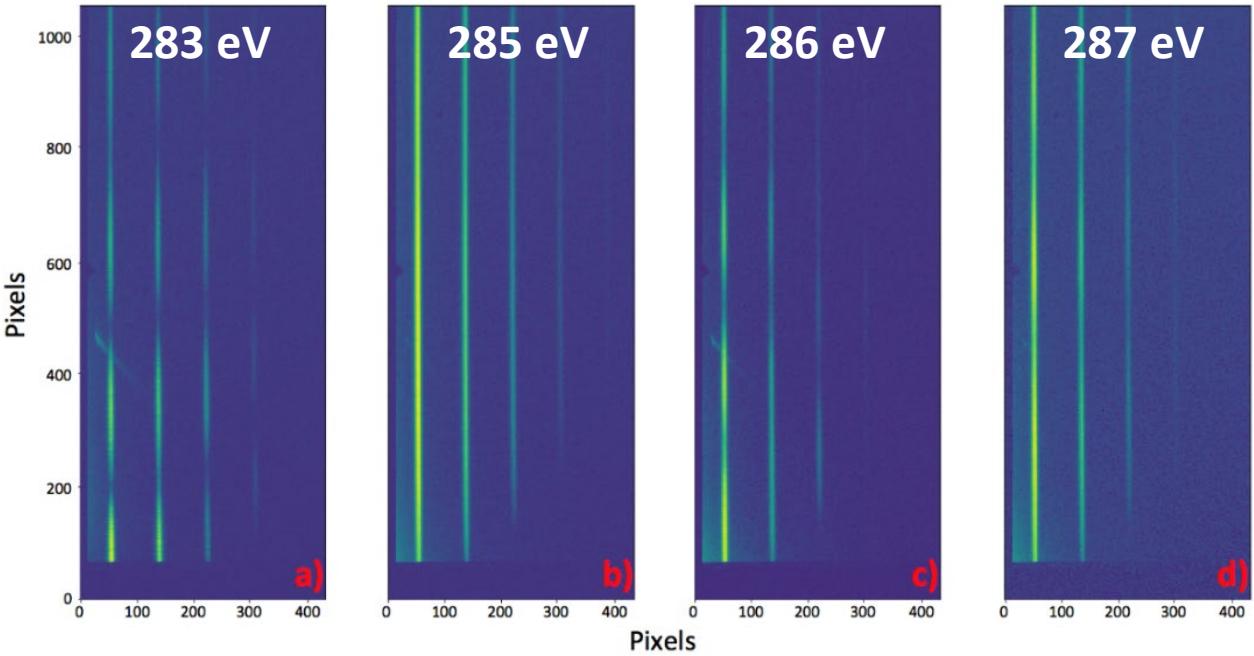
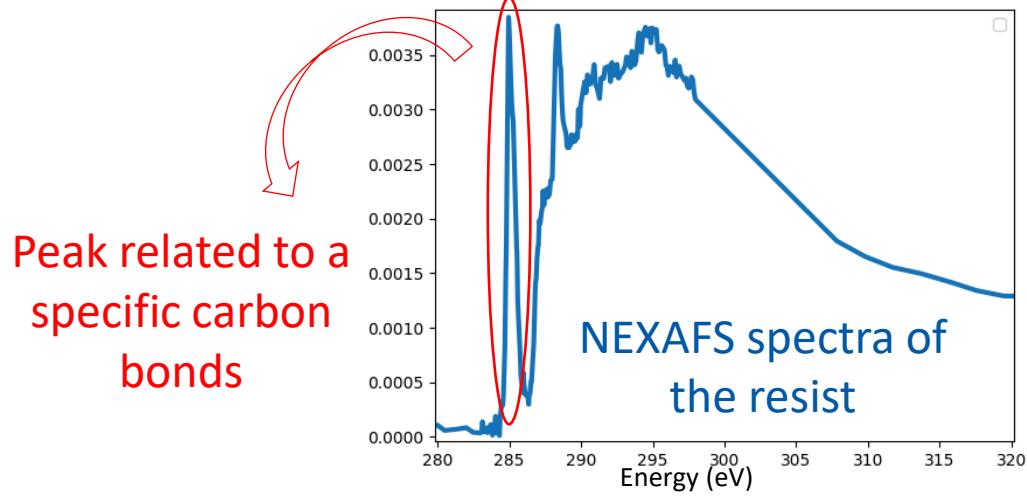
**Scattering Intensity**  
 $I(q) \propto |\Delta n|^2 = |\Delta\delta^2 + \Delta\beta^2|E^4$



# CD-GIRSOXS of latent image



## Chemically amplified resist:



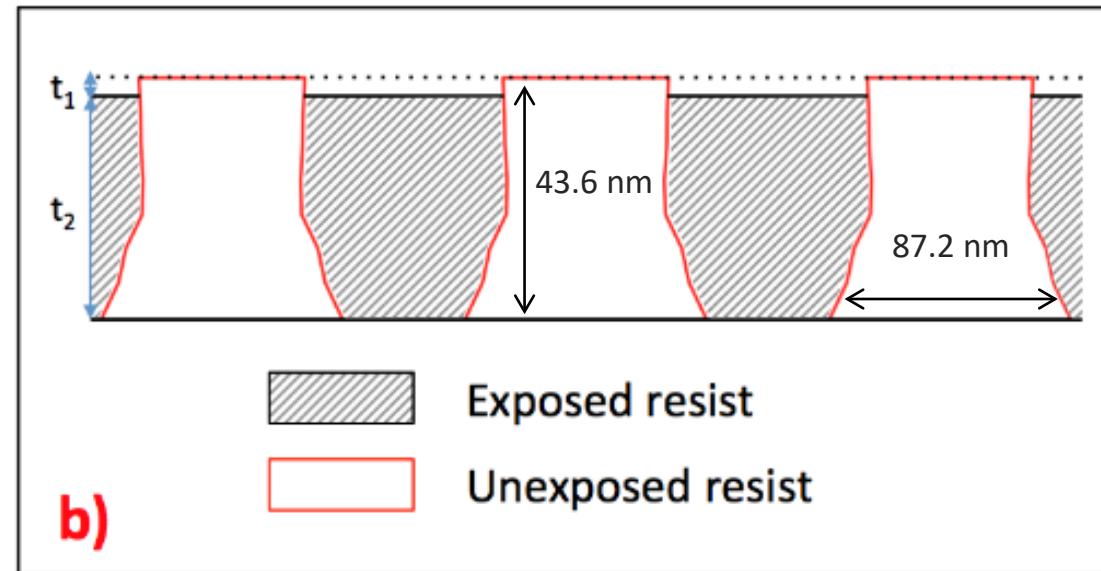
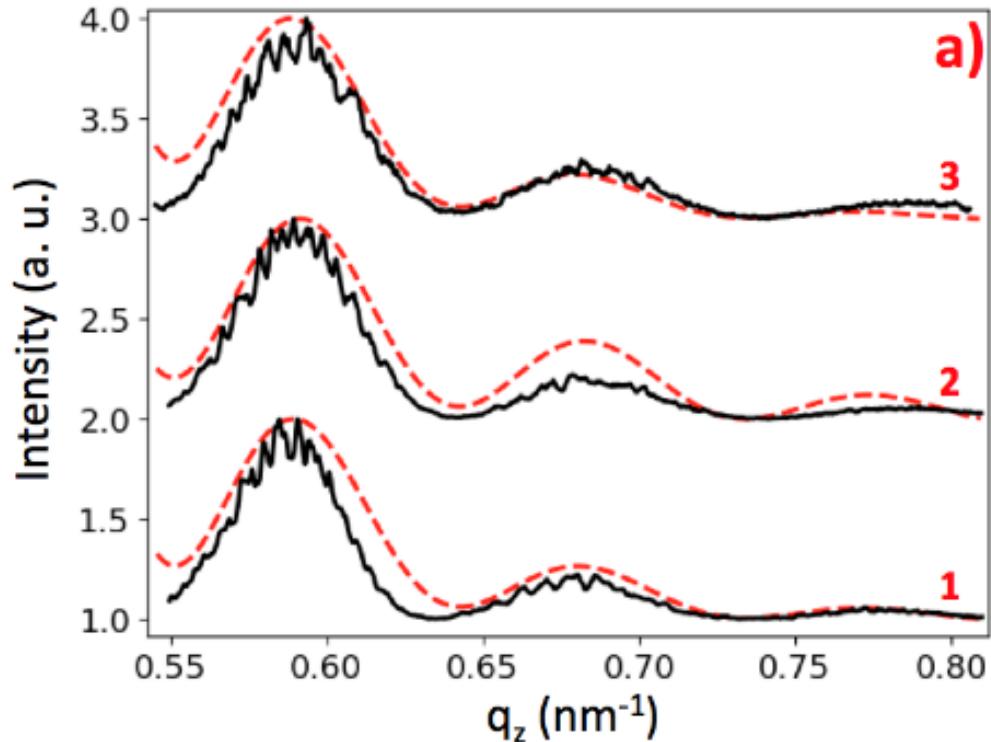
✓ **283 and 286 eV: Modulations in  $Q_z$ =contrast!**

✗ **285 and 287 eV: No contrast observed...**

**Contrast between exposed/unexposed resist**



## Reconstruction of the 3D shape of a latent image (283 eV)



- ✓ New perspectives to control the 3D shape of a latent image
- ✓ Short measurement time: 10 seconds

Freychet et al. Proc. SPIE EUV lithography (2019)

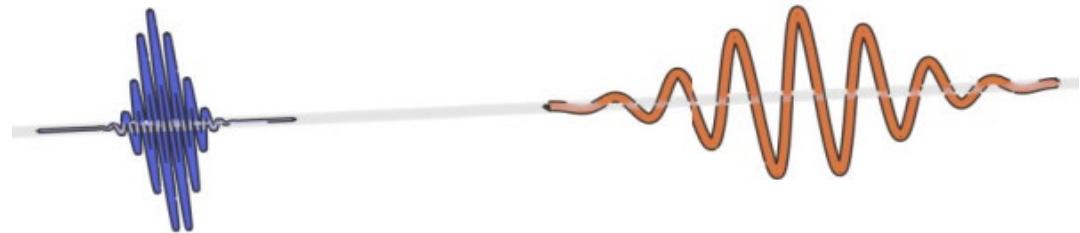
Freychet et al. J. Micro. Nanolith. MEMS, and MOEMS (2019)



# Perspectives / Challenges

Hardware improvement requirements towards in-line equipment:

- ✓ For transmission CD-SAXS: Gain of an order of magnitude on flux in hard x-ray laboratory
- ✓ For Grazing Incidence CD-SAXS: Development in soft x-ray sources => smaller size and more stable over time



Challenges to tackle for SAXS in the future:

- ✓ Can line roughness be extracted?
- ✓ Hybrid/Combine model based methods to gain certainty and unicity of solution
- ✓ Explore and develop soft x-ray scattering at the C-edge to probe latent imaging and extend to new edges for new resists (Sn, ...)

Periodic Table of the Elements

http://chemistry.about.com  
©2008 Todd Helmenstine

1A	2A	3A	4A	5A	6A	7A	8A										
H	Be						He										
Li	Mg																
Na																	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56
Rb	Sr	Y	Zr	Nb	Mn	Tc	Ru	Pd	Ag	Cd	In	Sn	St	Te	I	Xe	
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72
Cs	Ba	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn	
87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104
Fr	Ra																
223	224																
Lanthanides																	
57	58	59	60	61	62	63	64	65	66	67	68	69	70	71			
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu			
139	140	141	142	143	144	145	146	147	148	149	150	151	152	153			
Actinides	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103		
	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105		
	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lu		
	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241		



# Acknowledgements



Patrice Gergaud  
Nicolas Vaxelaire  
Jerome Reche  
Guido Rademaker  
Yoann Blancquaert  
Timothée Choisnet



Ronal Pandolfi  
Dinesh Kumar  
Jamie A. Sethian



Isvar Cordova  
Greg Su  
Cheng Wang  
Alexander Hexemer



Patrick Naulleau  
Luke Long

