

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

Guillaume Freychet

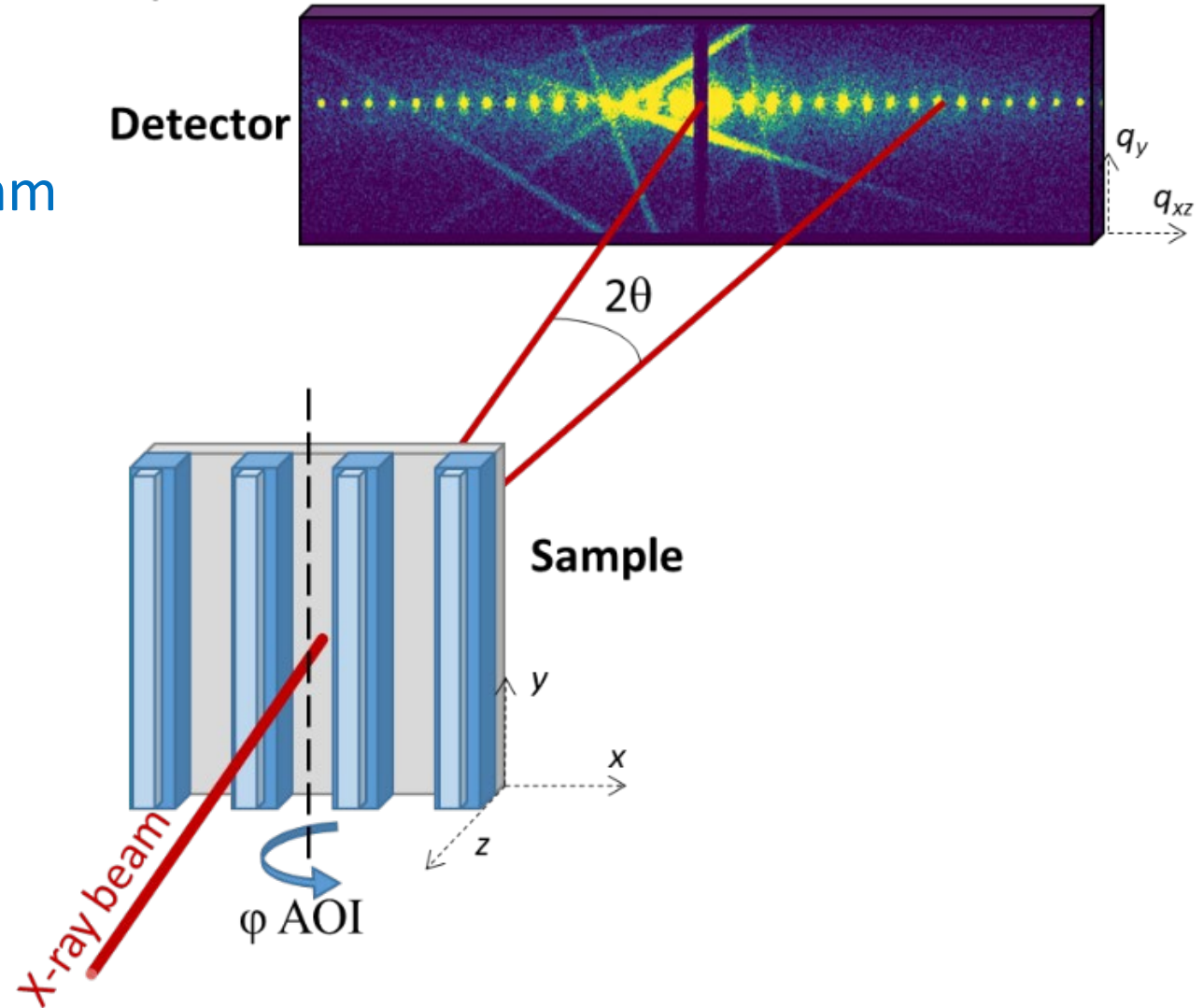
Université Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France

# Small Angle x-ray scattering (SAXS)

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

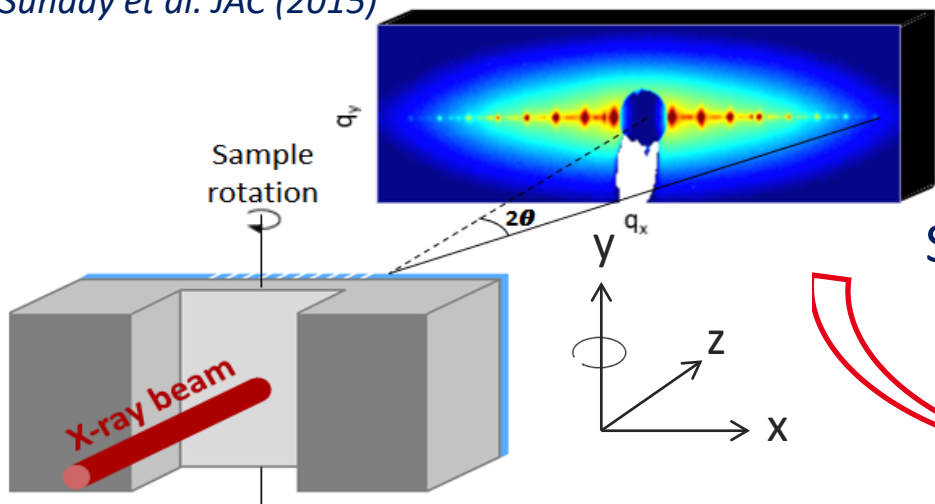
$$q = \frac{2\pi}{\lambda} \sin 2\theta$$

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

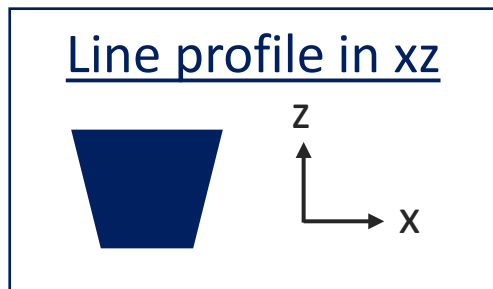
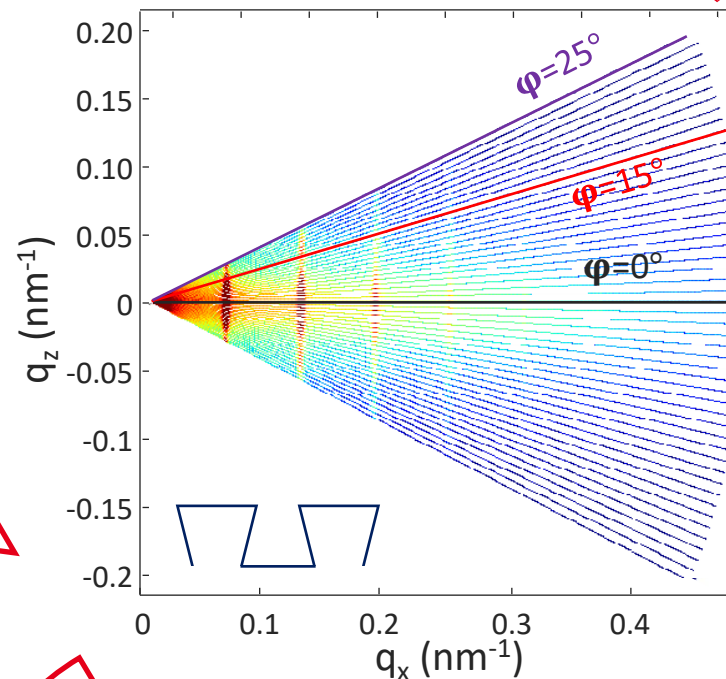


# CD-SAXS : principle

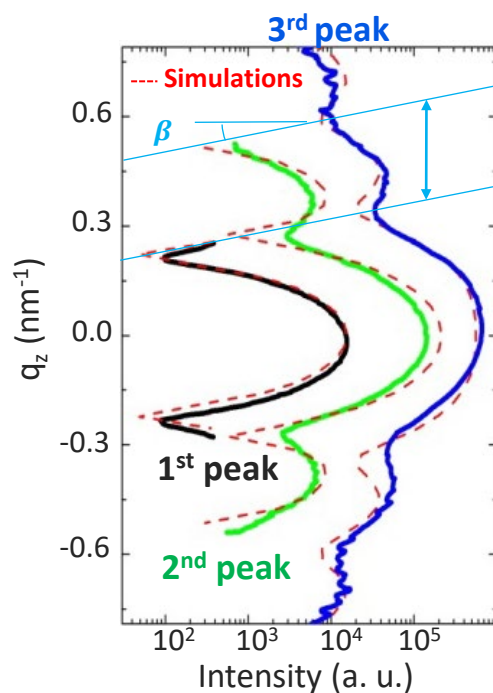
D. Sunday et al. JAC (2015)



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

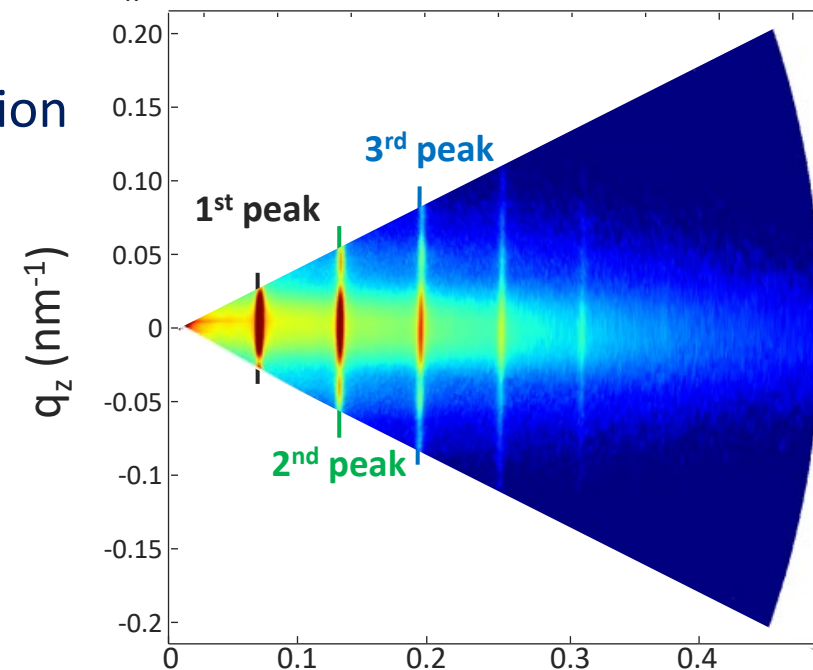


Fit the experimental data



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

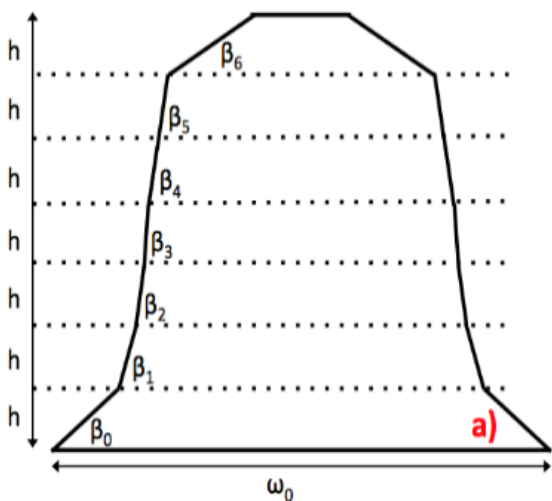


D. Kumar





# CD-SAXS : principle



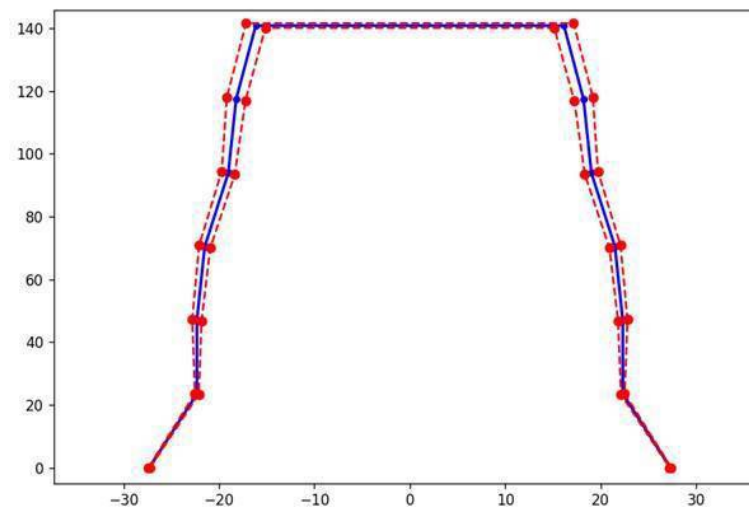
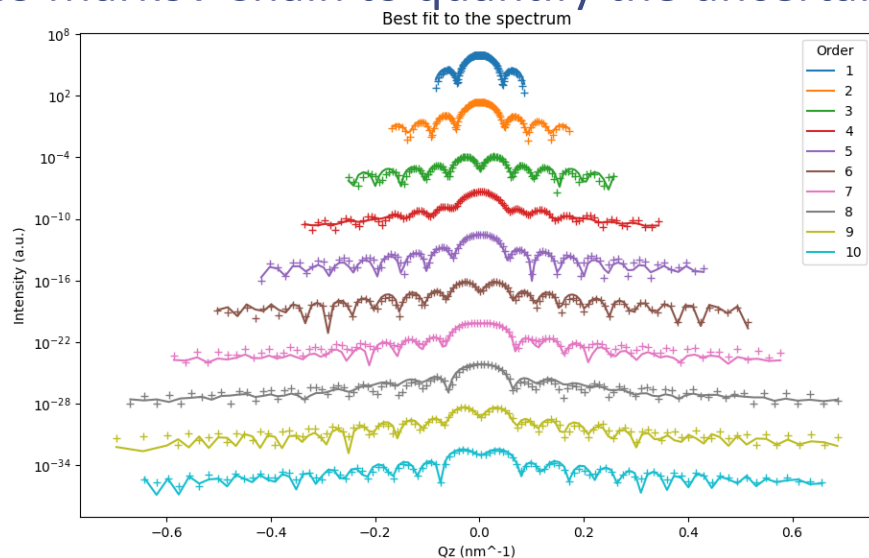
- ✓ Fourier transform of a trapezoid ( $L = \omega_0$  and  $m = \tan \beta$ ):

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

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

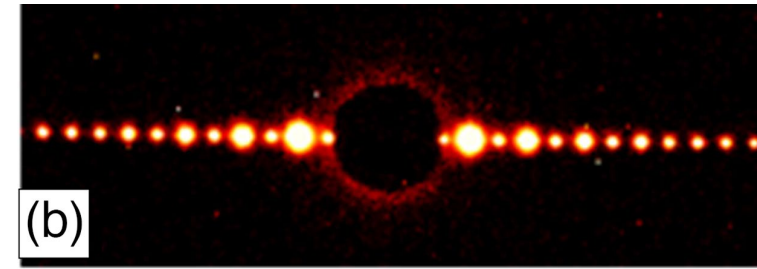
Genetic algorithm to converge to the best profile

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



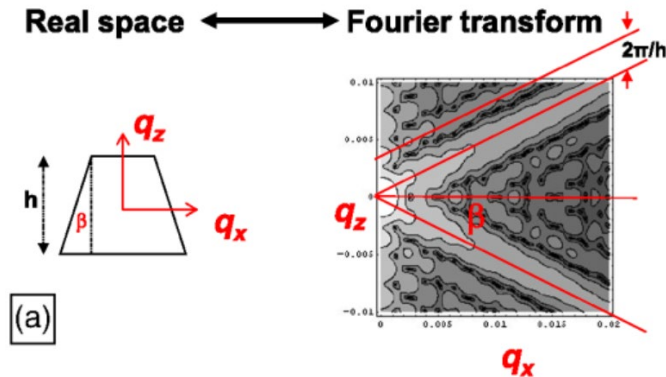
# CD-SAXS : History

✓ Early 2000s: First measurements at APS by NIST

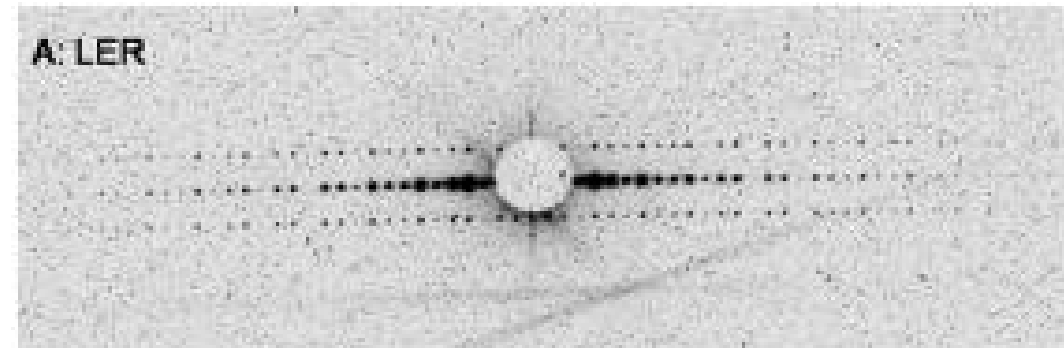


W-L. Wu et al. JM3, Vol. 22, Issue 3, 031206 (2023)

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



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



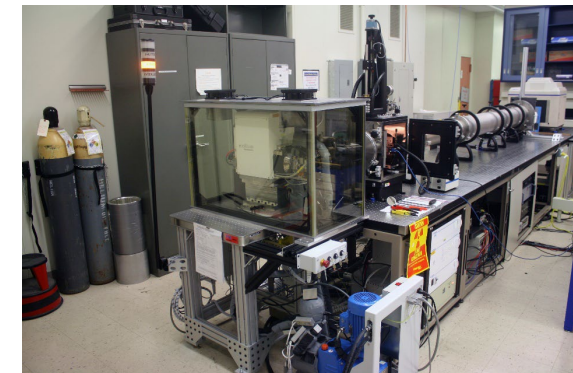
Wang et al. J. Appl. Phys. 102, 024901 (2007)

✓ 2013-2020:

Strengthen the robustness of the analysis (genetic algorithm)

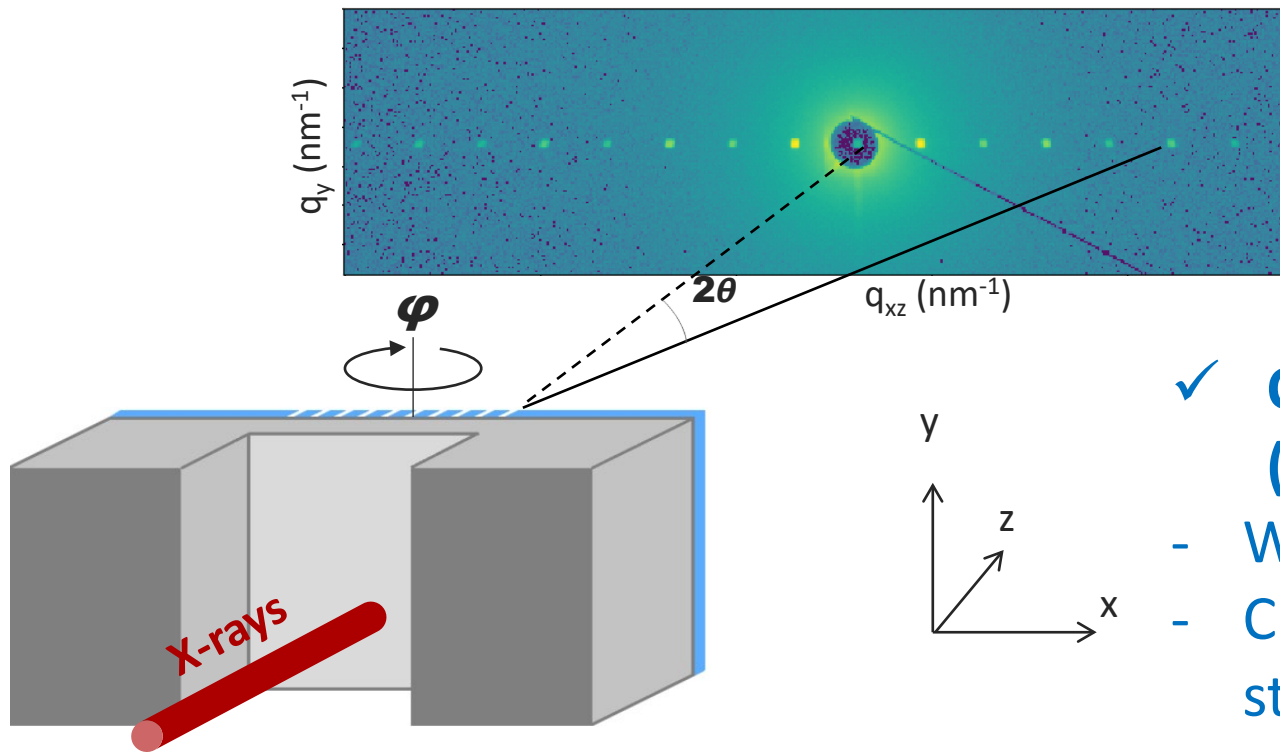
Test measurements with laboratory sources

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

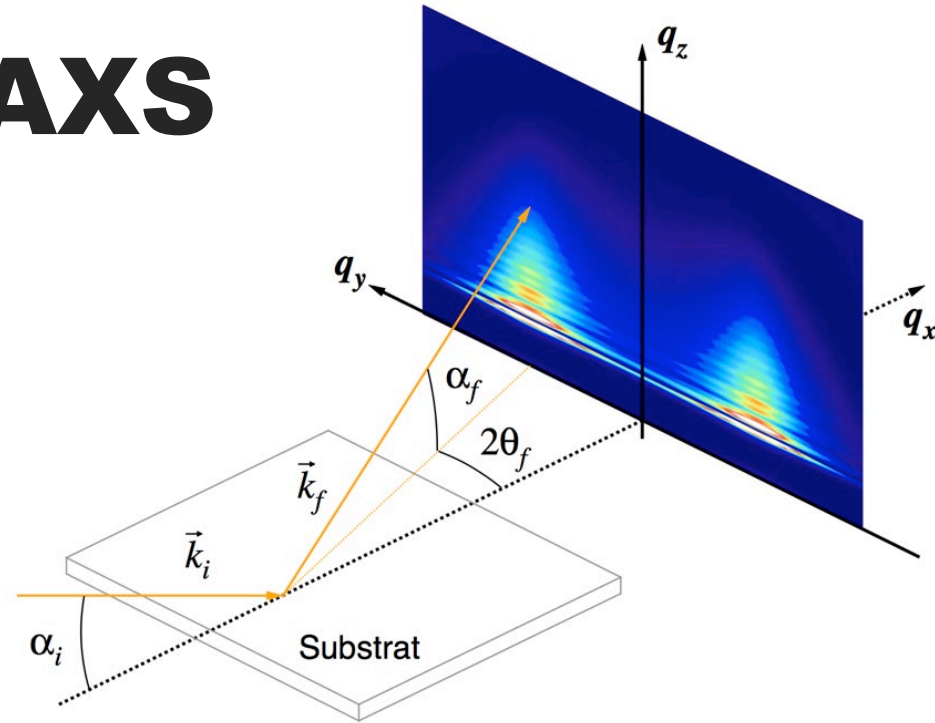
## Measurement in grazing incidence: CD-GISAXS



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

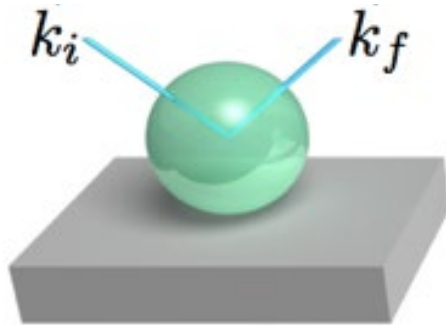
# Grazing-incidence SAXS (GISAXS)

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

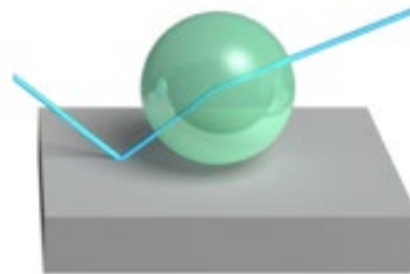


## Distorted Wave Born Approximation (DWBA)

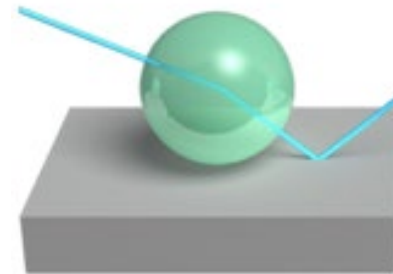
$$\mathcal{F}(q_{||}, k_z^i, k_z^f) = F(q_{||}, q_z^1) + r(\alpha_f)F(q_{||}, q_z^2) + r(\alpha_i)F(q_{||}, q_z^3) + r(\alpha_i)r(\alpha_f)F(q_{||}, q_z^4)$$



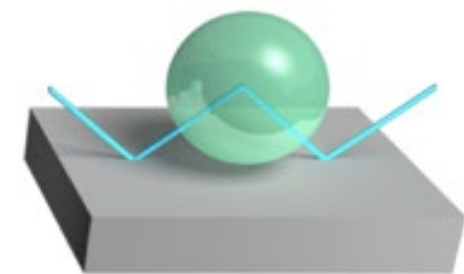
$$\vec{q}_1 = \vec{k}_f - \vec{k}_i$$



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



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



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

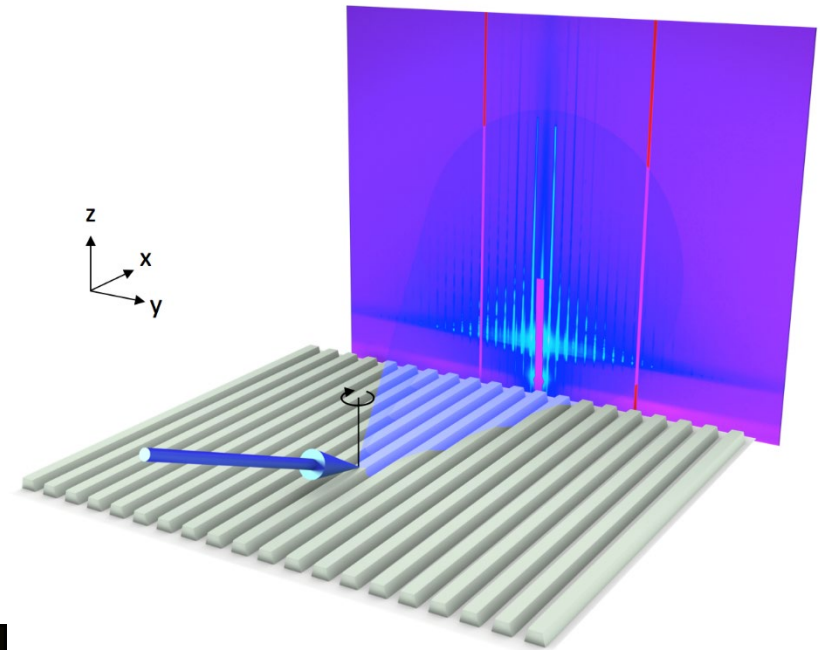
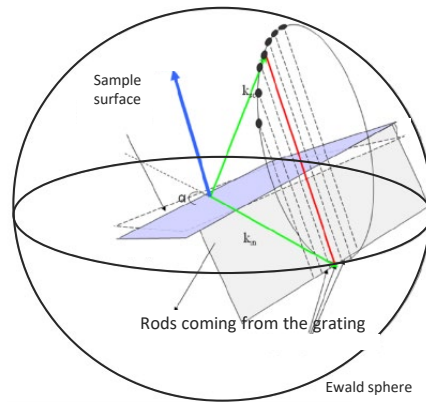
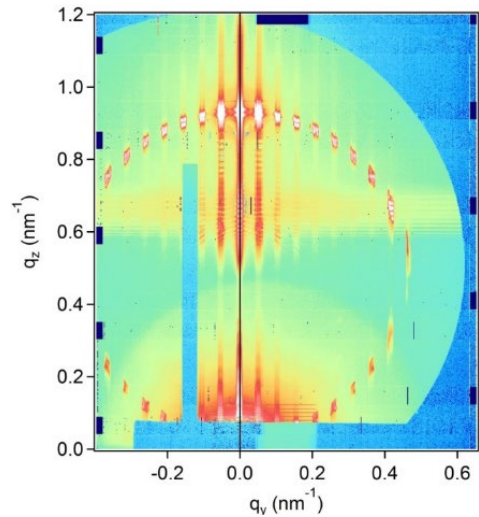


# Critical Dimension GISAXS (CD-GISAXS)

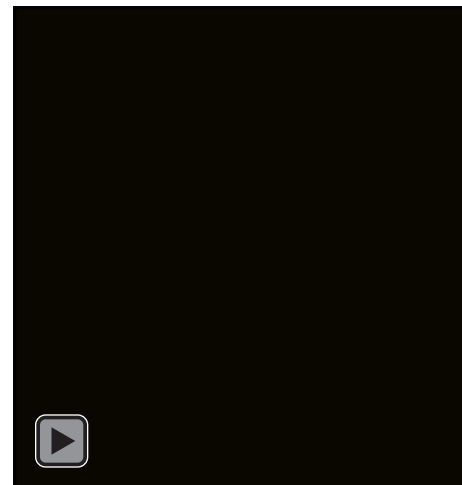
*Hofmann et al. J. Vac. Sci. Tech. (2009)*

*Lu et al. App. Cryst. (2012)*

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

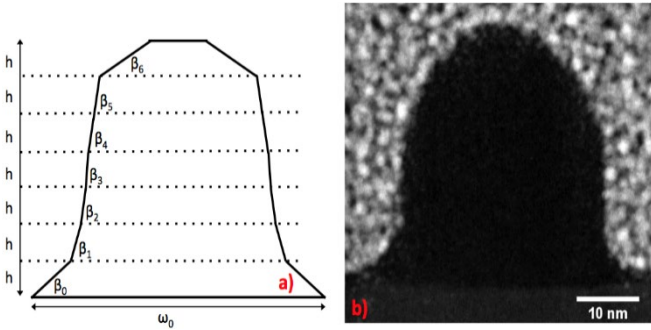


Rotation of the gratings  
(along z)



Full intersection of the Bragg rods  
and the Ewald sphere recorded

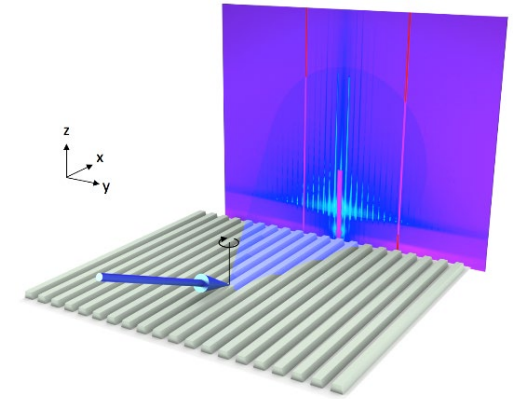
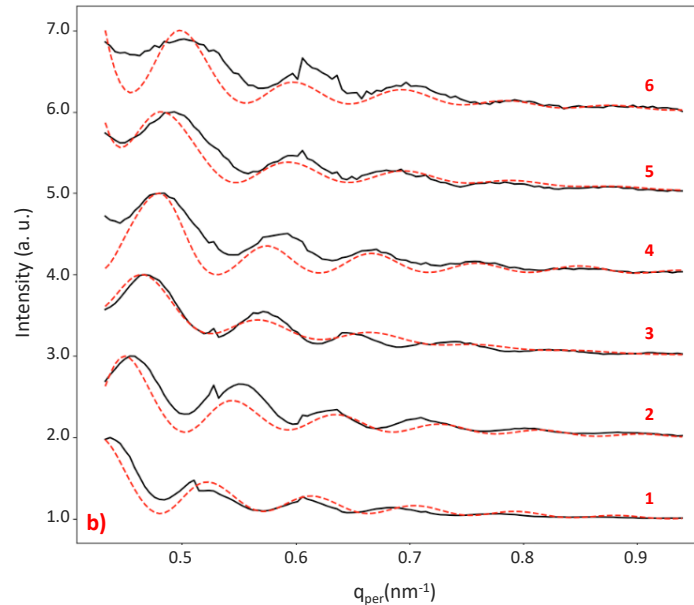
# Critical Dimension GISAXS (CD-GISAXS)



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

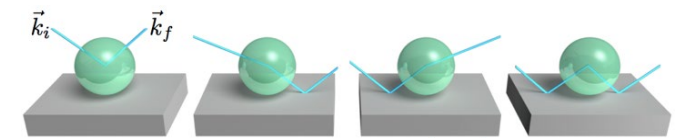
*Freychet et al. Proc. SPIE (2018)*

*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



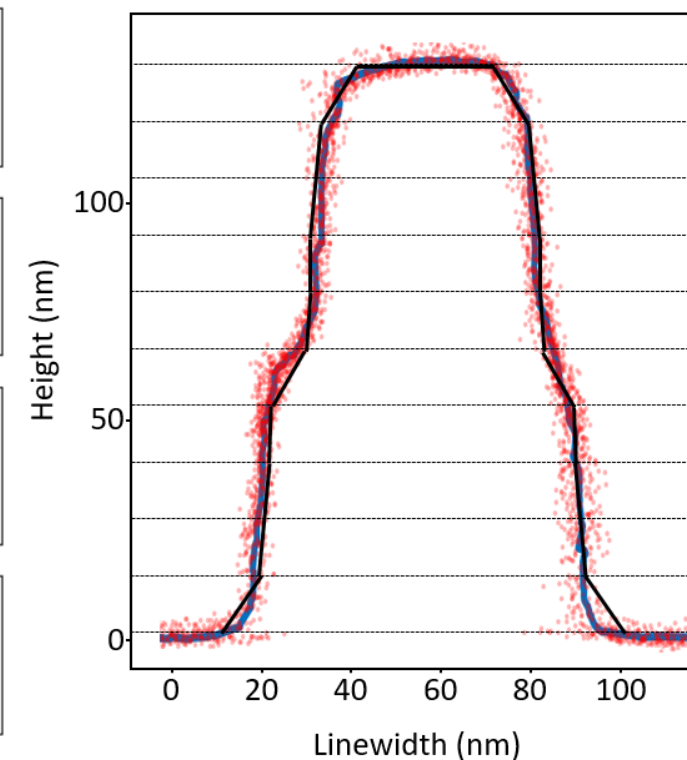
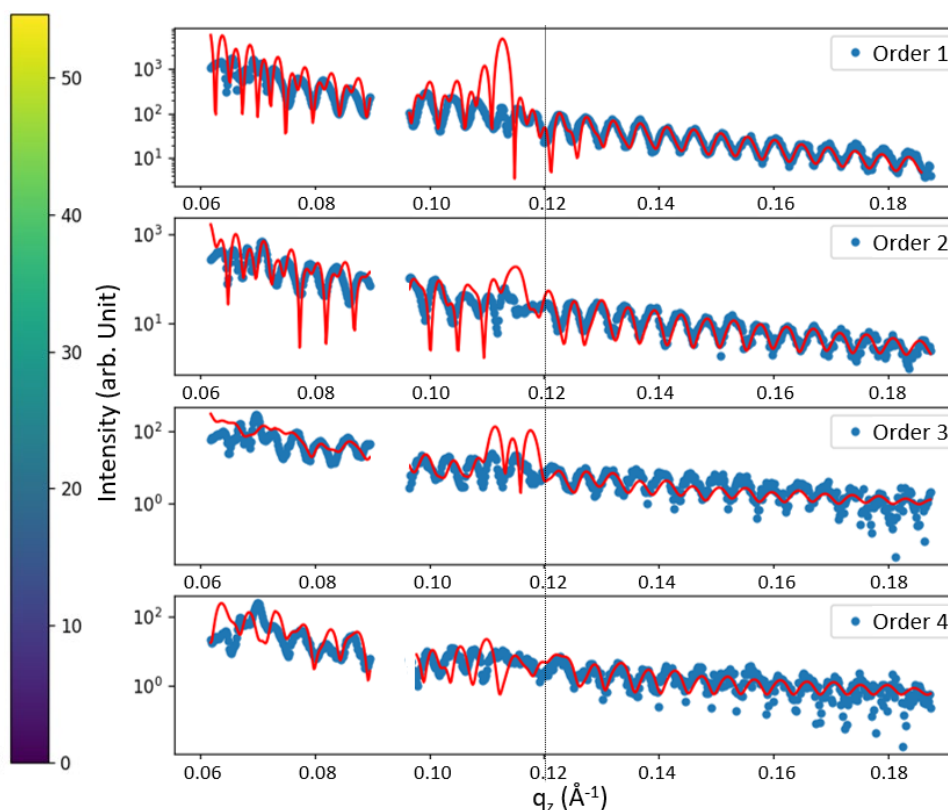
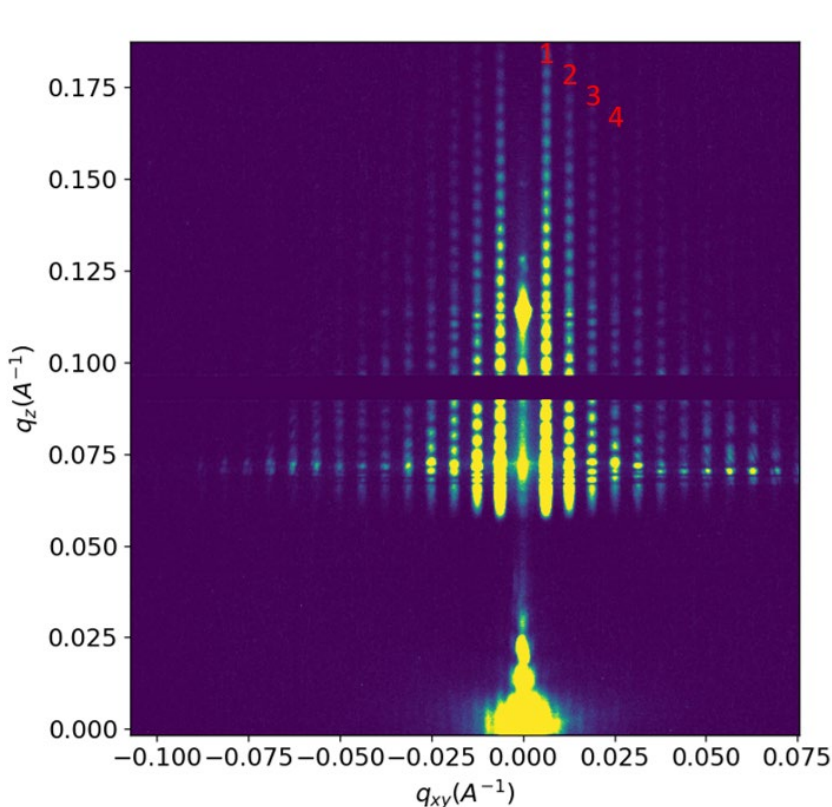
- ✓ Source Cu K- $\alpha$  (8.047 keV)
- ✓ Sample to detector distance: 1.8 m
- ✓ Transmission and Reflection

*Freychet et al. JM3 2023*

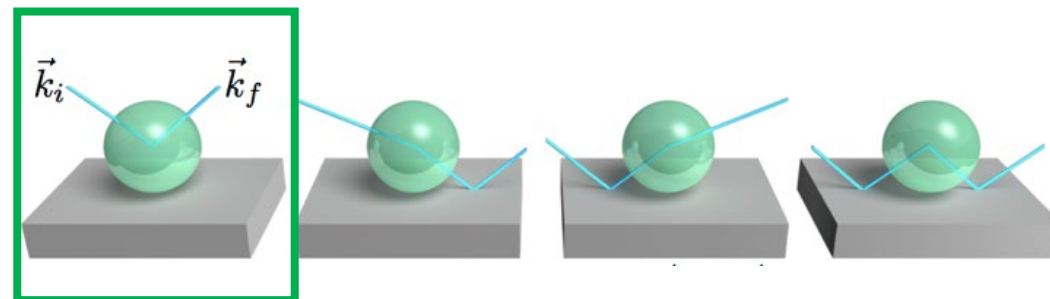
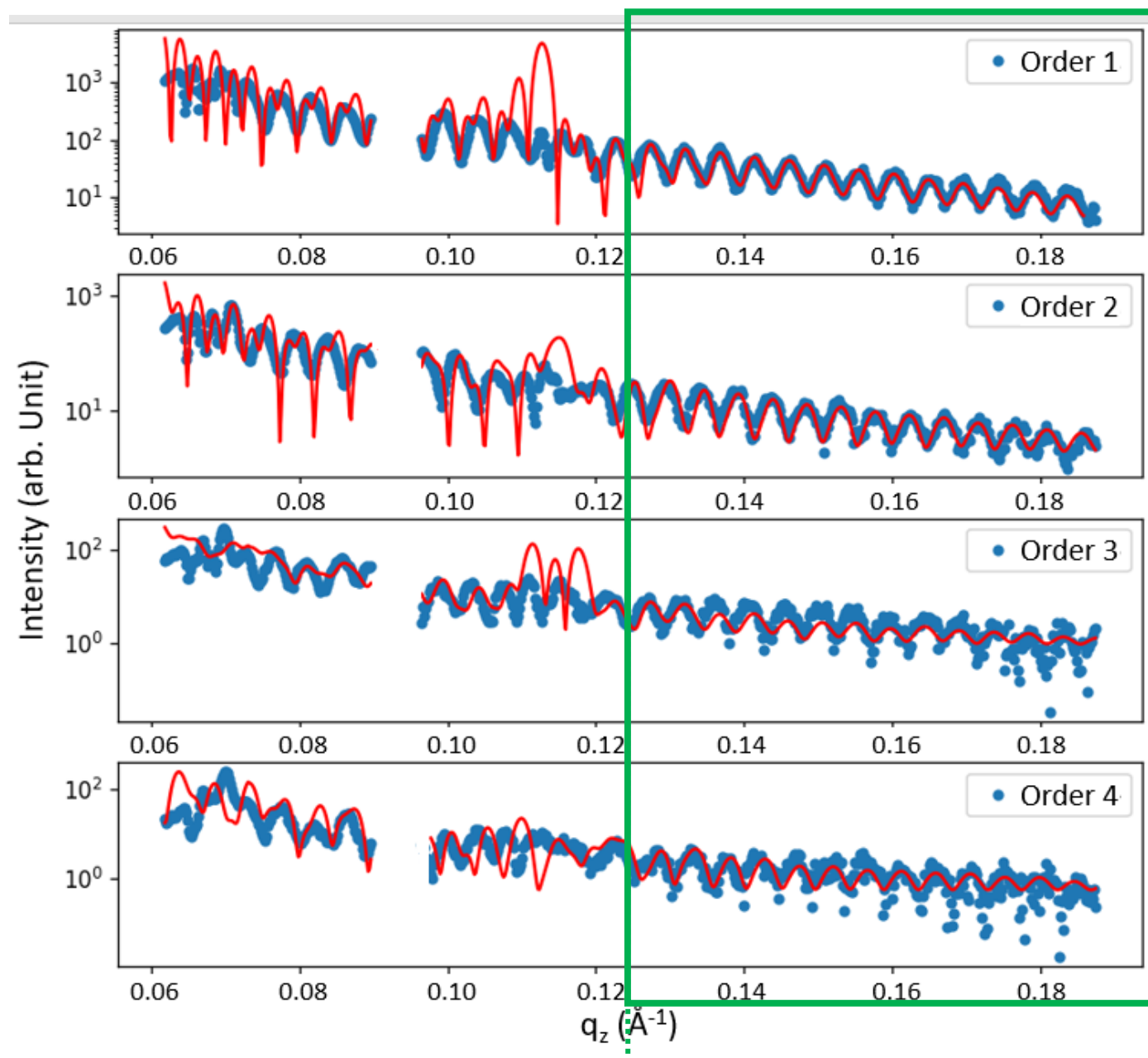
# CD-GISAXS with a Laboratory equipment



*Freychet et al. JM3 2023*



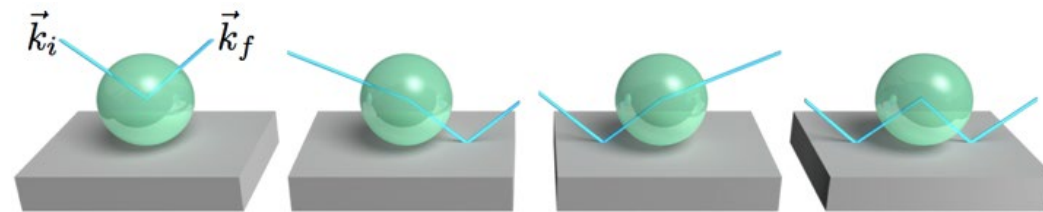
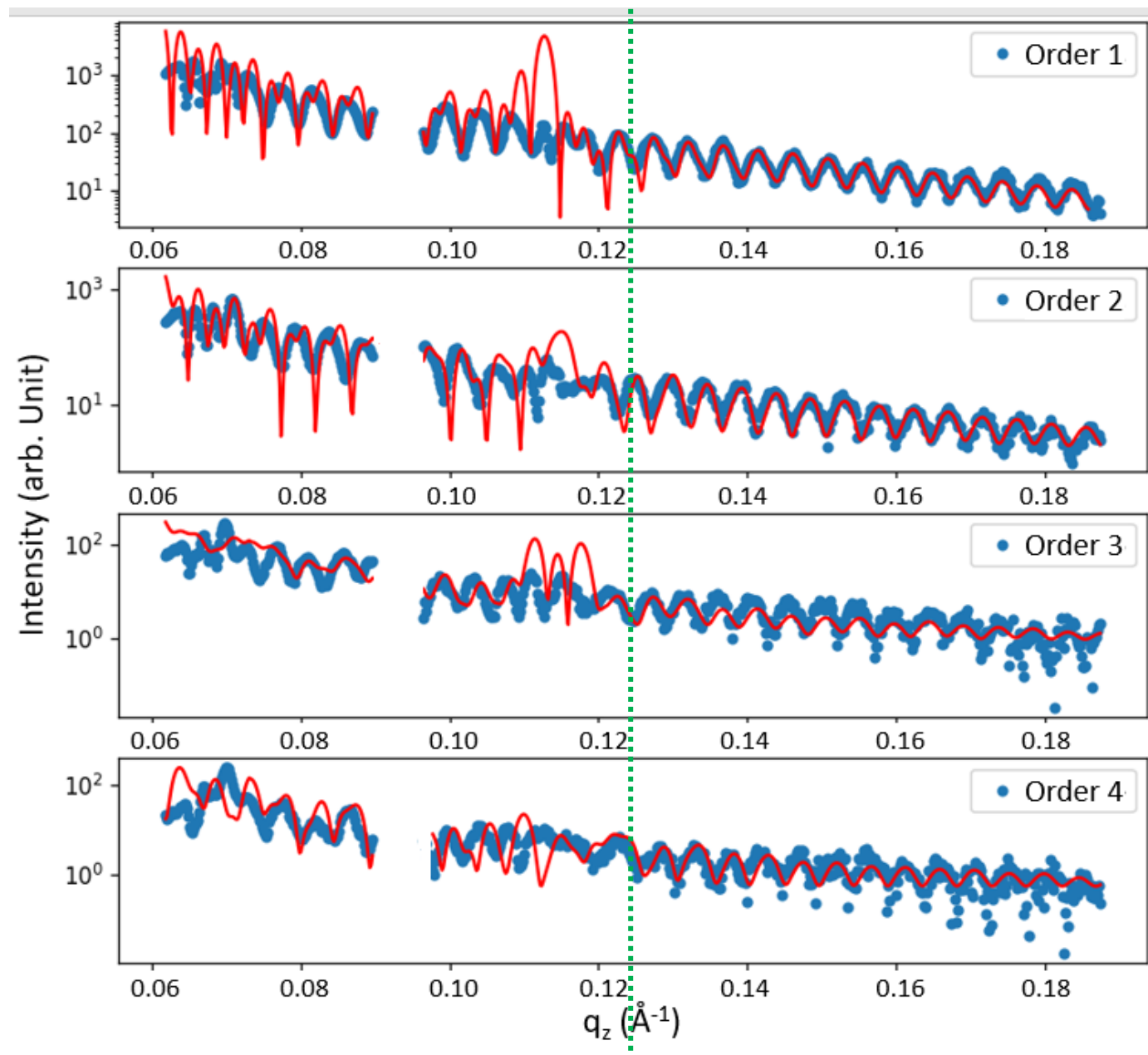
# CD-GISAXS with a Laboratory equipment



DWBA:

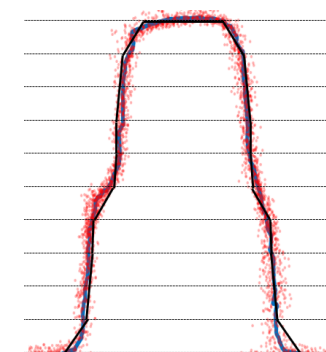
✓  $\alpha_i$  and  $\alpha_f > 3 \times \alpha_c$ : minimize the multi-reflection effect

# CD-GISAXS with a Laboratory equipment



DWBA:

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



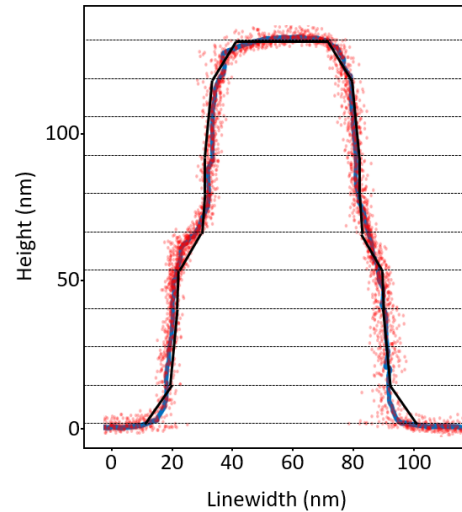
Substrate

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)

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

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

Soft x-ray (200 eV):

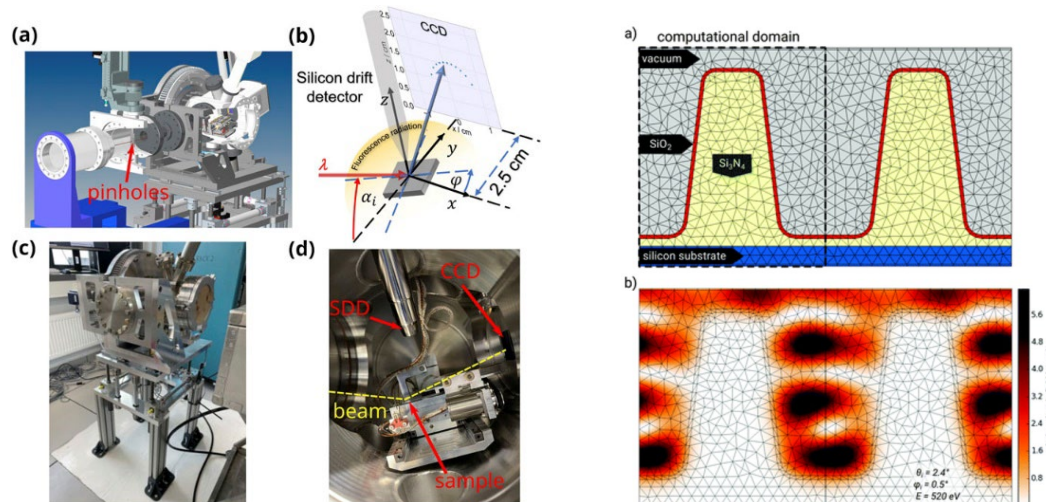
$\alpha_c(\text{Si}) = 10 \text{ deg}$

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

**No footprint issue**

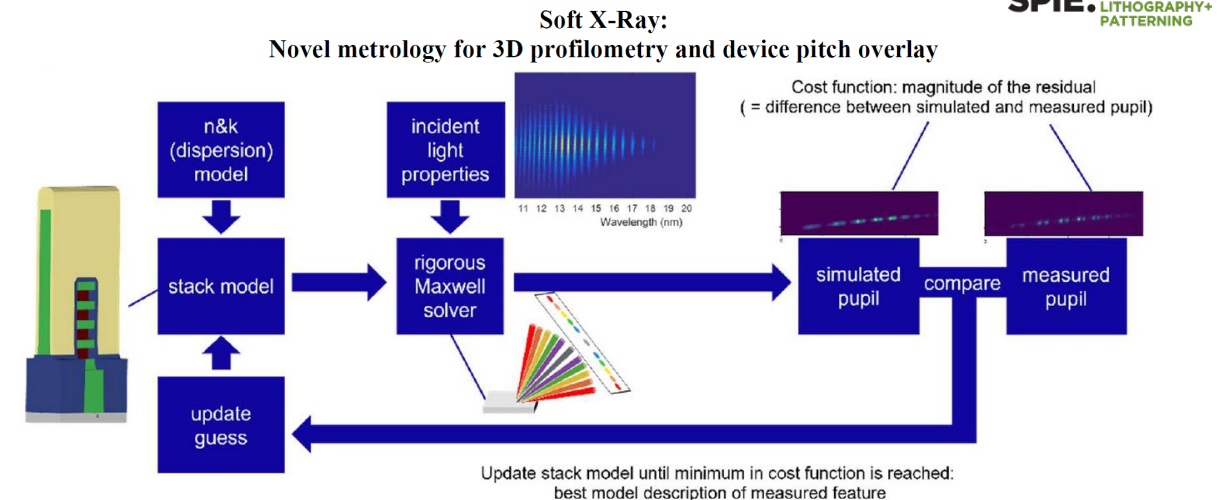
PTB is working with EUV source since 2014

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



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

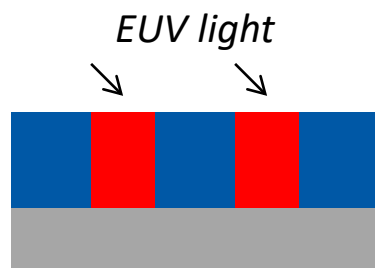
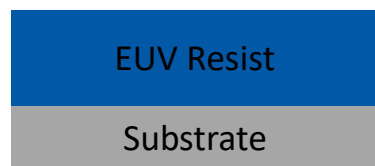
- ✓ Footprint  $< 50 \mu\text{m}$
- ✓ Acquisition time around 200 s



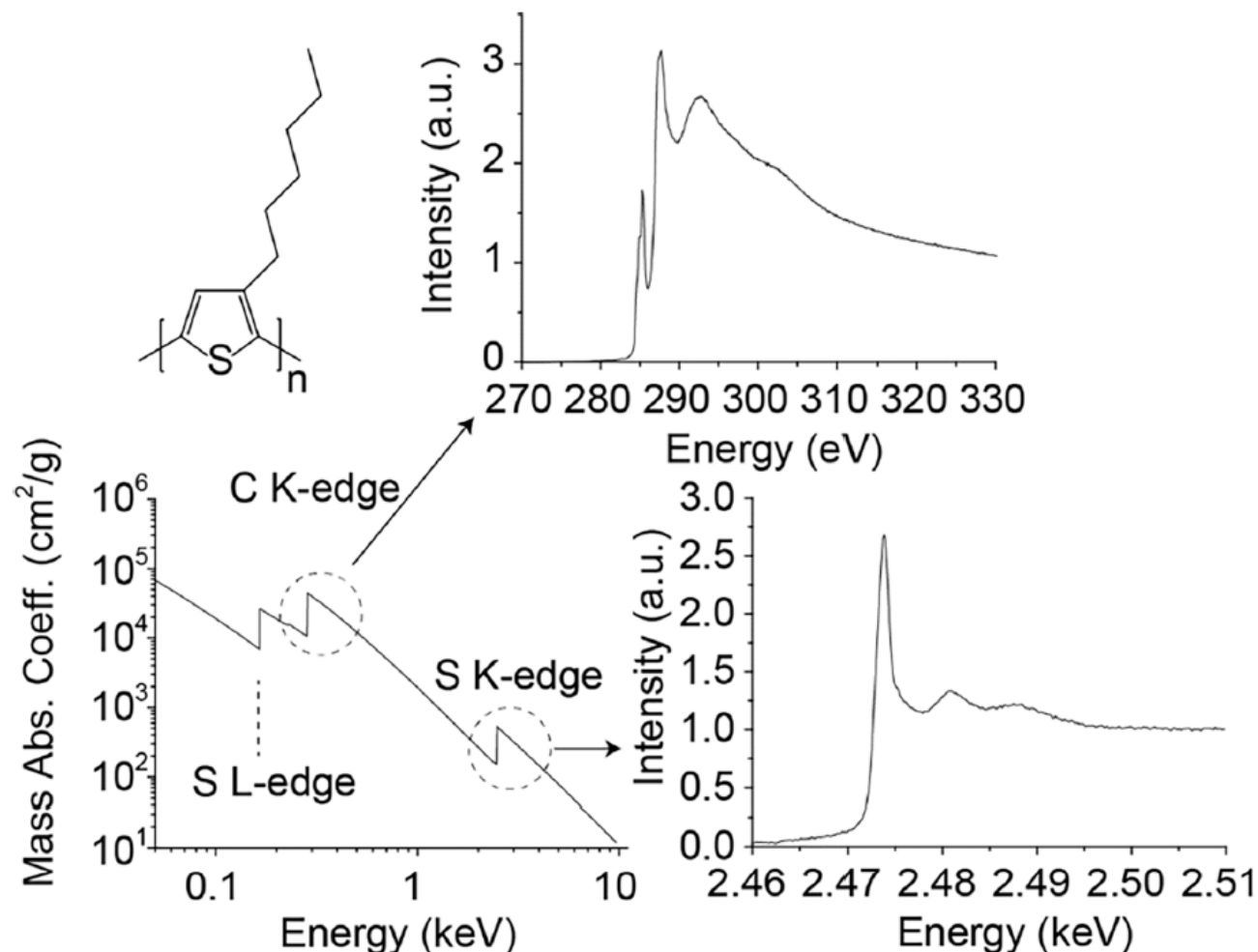
# Resonant Soft X-Ray Scattering (RSoXS)

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

EUV lithography process  
simplified



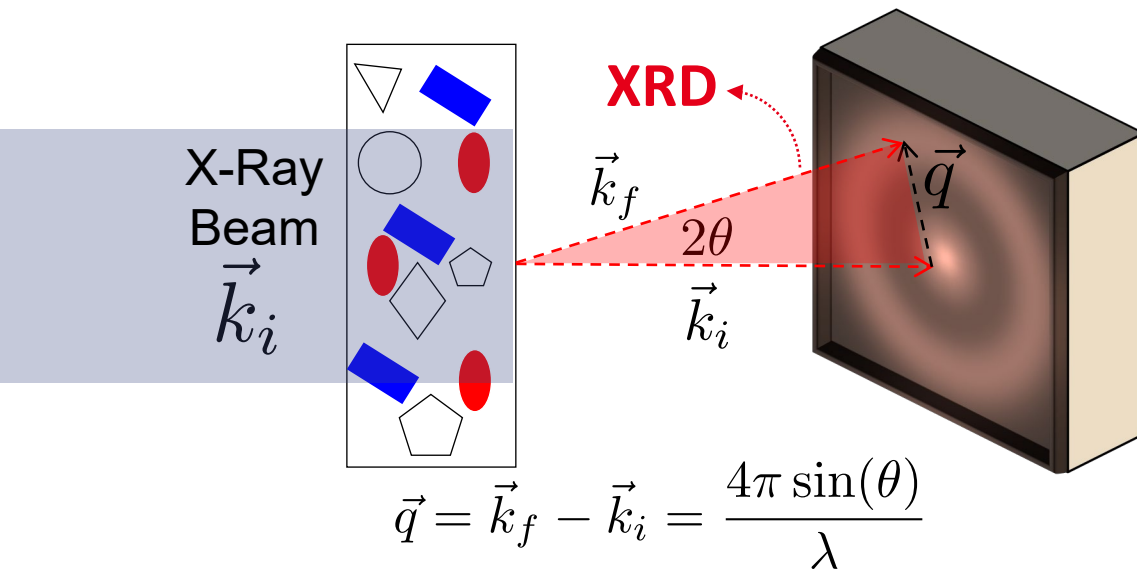
Exposed resist  
Unexposed resist



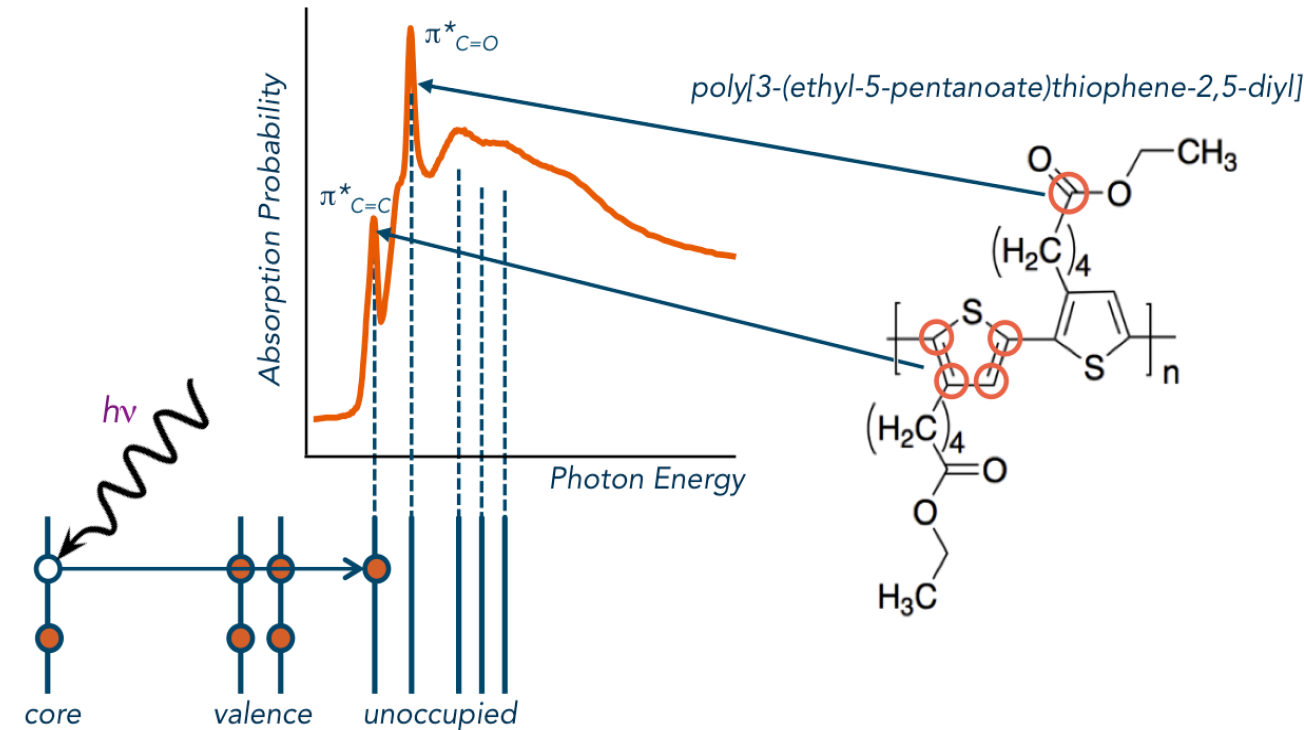
# Resonant Soft X-Ray Scattering (RSoXS)

*Resonance (Anomalous) Effect  $f = f_0 + f' + if''$*

## Small Angle X-Ray Scattering

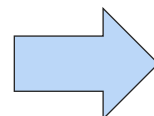


## X-ray Absorption Spectroscopy ( $\beta$ )



## Refractive Index

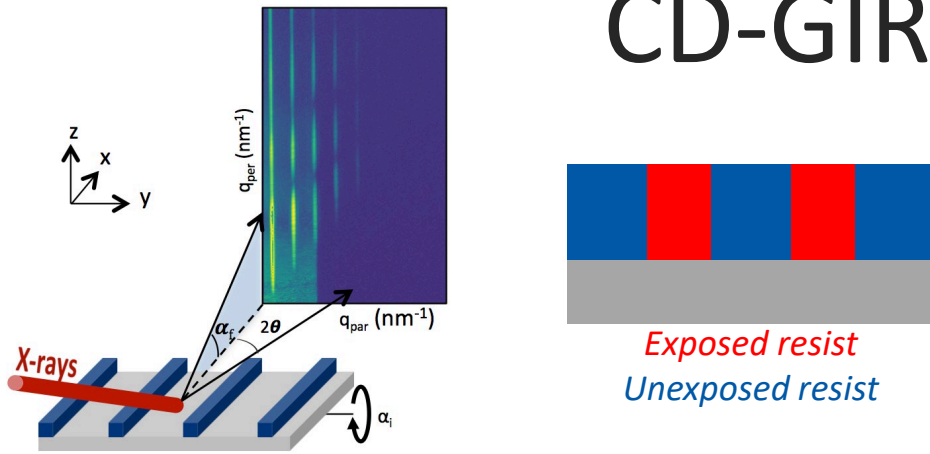
$$n(E) = 1 - \delta + i\beta$$



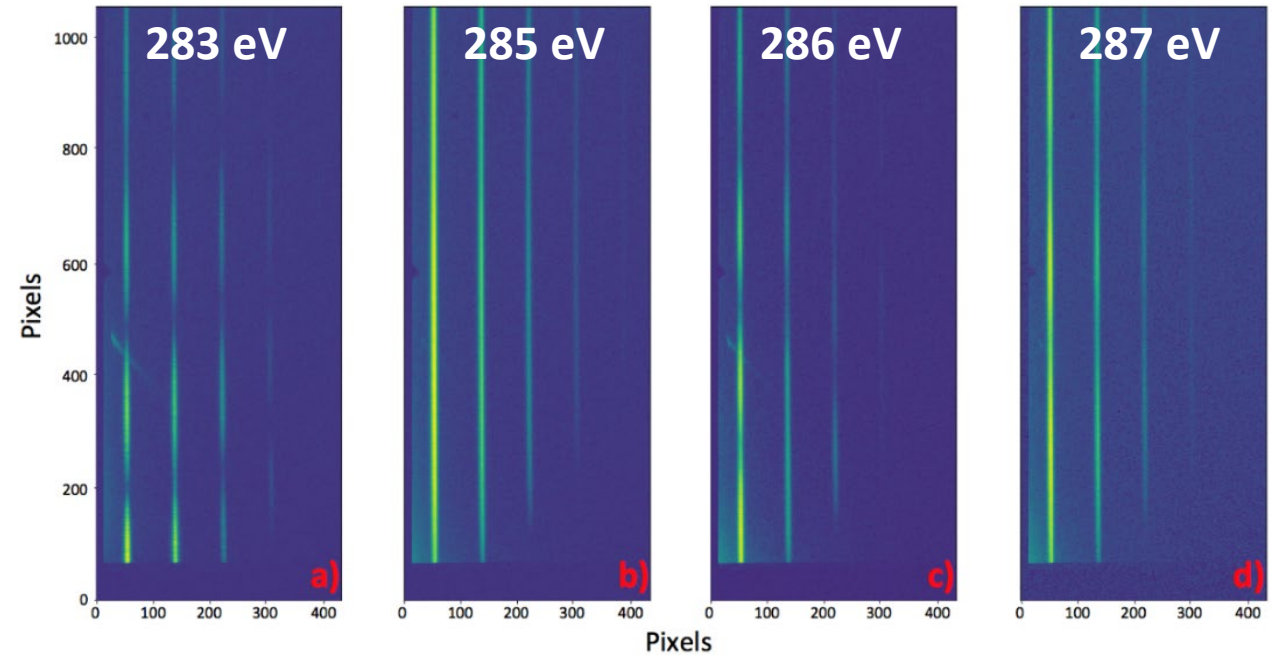
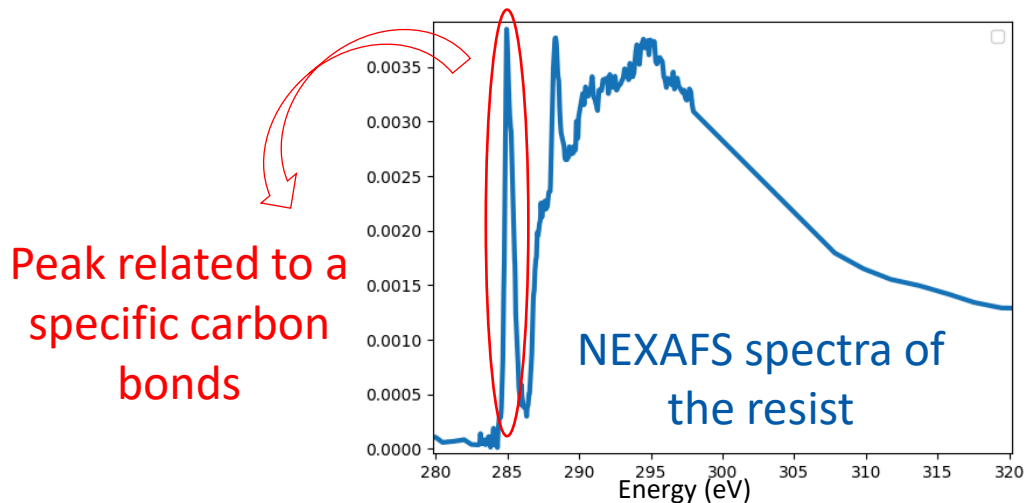
## Scattering Intensity

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

# CD-GIRSOXS of latent image



## Chemically amplified resist:



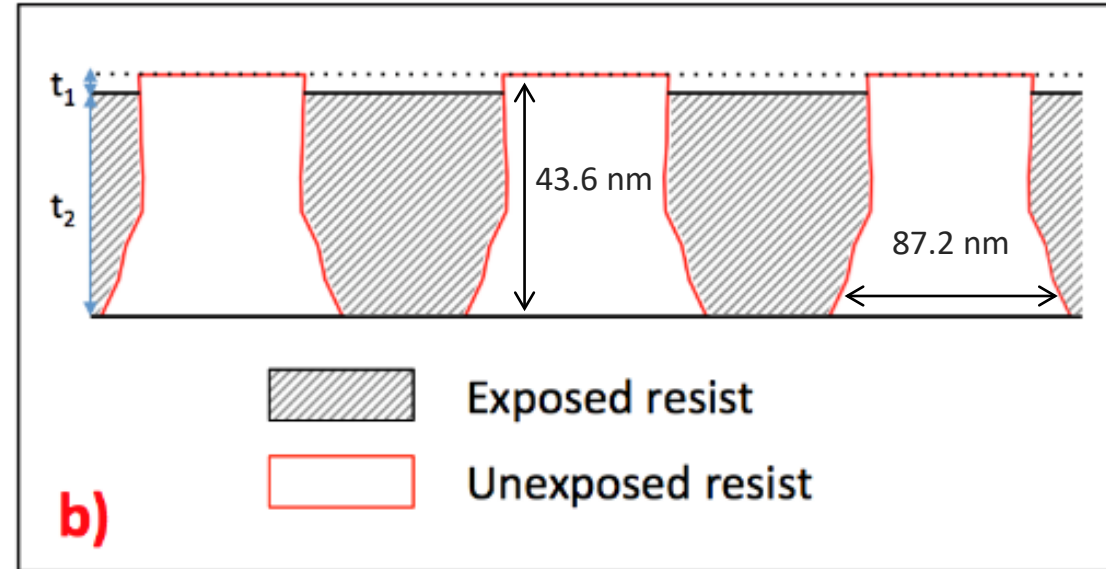
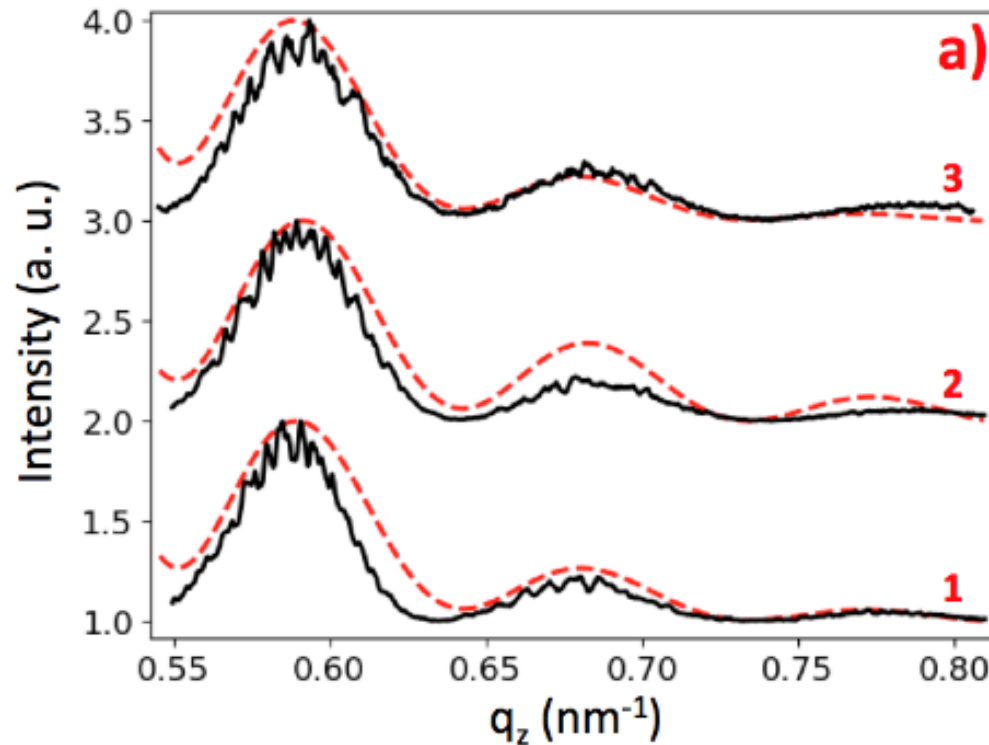
✓ 283 and 286 eV: Modulations in  $Q_z$ =contrast!

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

**Contrast between exposed/unexposed resist**

# CD-GIREXS of latent image

## 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)*

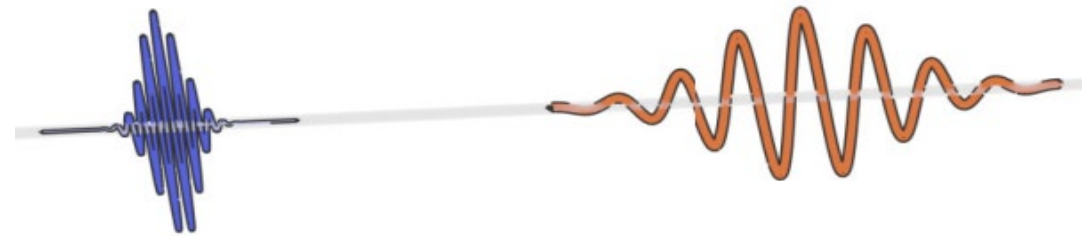
*Freychet et al. J. Micro. Nanolith. MEMS, and MOEMS (2019)*



# 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
1 H 1.00794	2 He 4.002602						
3 Li 6.941	4 Be 9.00918	5 B 10.811	6 C 12.011	7 N 14.00643	8 O 15.999	9 F 18.998403	10 Ne 20.1797
11 Na 22.98976928	12 Mg 24.304	13 Al 26.9815385	14 Si 28.0855836	15 P 30.973761998	16 S 32.06	17 Cl 35.45	18 Ar 39.948
19 K 39.0983	20 Ca 40.078	21 Sc 44.955912	22 Ti 47.88	23 V 50.9415	24 Cr 51.9961	25 Mn 54.938045	26 Fe 55.845
37 Rb 85.4678	38 Sr 87.62	39 Y 88.90584	40 Zr 91.224	41 Nb 92.90638	42 Mo 95.94	43 Tc [98]	44 Ru 101.07
55 Cs 132.90545196	56 Ba 137.327	57-71 Lanthanides	72 Hf 178.49	73 Ta 180.94788	74 W 183.84	75 Re 186.207	76 Os 190.23
87 Fr [223]	88 Ra [226]	89-103 Actinides	104 Rf [261]	105 Db [262]	106 Sg [266]	107 Bh [264]	108 Hs [277]
			109 Mt [268]	110 Ds [271]	111 Rg [272]	112 Uub [285]	113 Uut [284]
			114 Uuq [289]	115 Uup [288]	116 Uuh [292]	117 Uus [294]	118 Uuo [293]
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			164 Uub [289]	165 Uut [288]	166 Uuq [292]	167 Uus [294]	168 Uuo [293]
			169 Uue [289]	170 Uub [288]	171 Uut [292]	172 Uuq [294]	173 Uuq [293]
			174 Uub [289]	175 Uut [288]	176 Uuq [292]	177 Uus [294]	178 Uuo [293]
			179 Uue [289]	180 Uub [288]	181 Uut [292]	182 Uuq [294]	183 Uuq [293]
			184 Uub [289]	185 Uut [288]	186 Uuq [292]	187 Uus [294]	188 Uuo [293]
			189 Uue [289]	190 Uub [288]	191 Uut [292]	192 Uuq [294]	193 Uuq [293]
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			259 Uue [289]	260 Uub [288]	261 Uut [292]	262 Uuq [294]	263 Uuq [293]
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