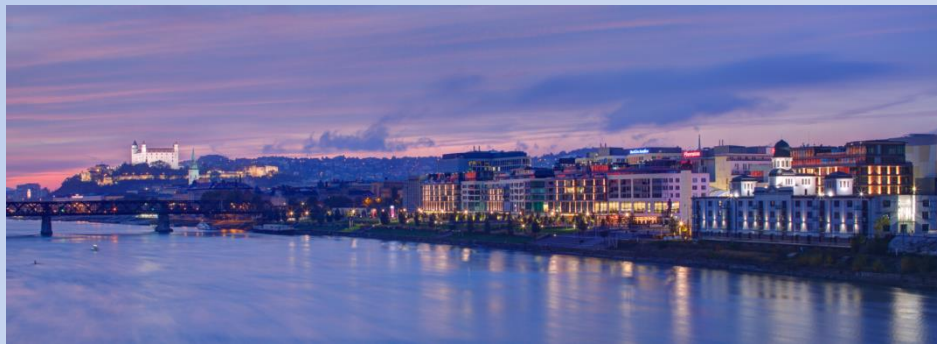


# Tungsten growth on silicon dioxide and boron carbide and additional role of spacer in the ultrashort period multilayer X-ray mirrors

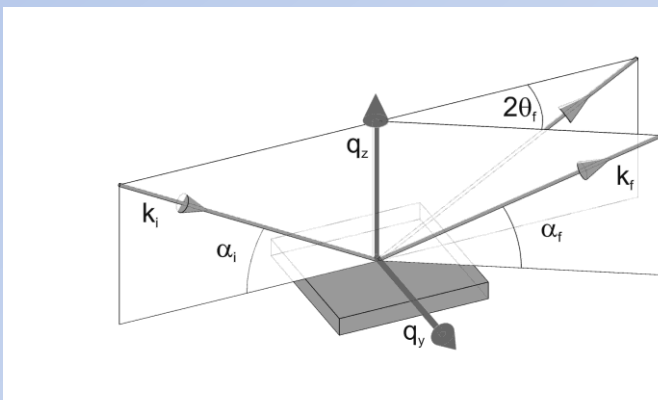
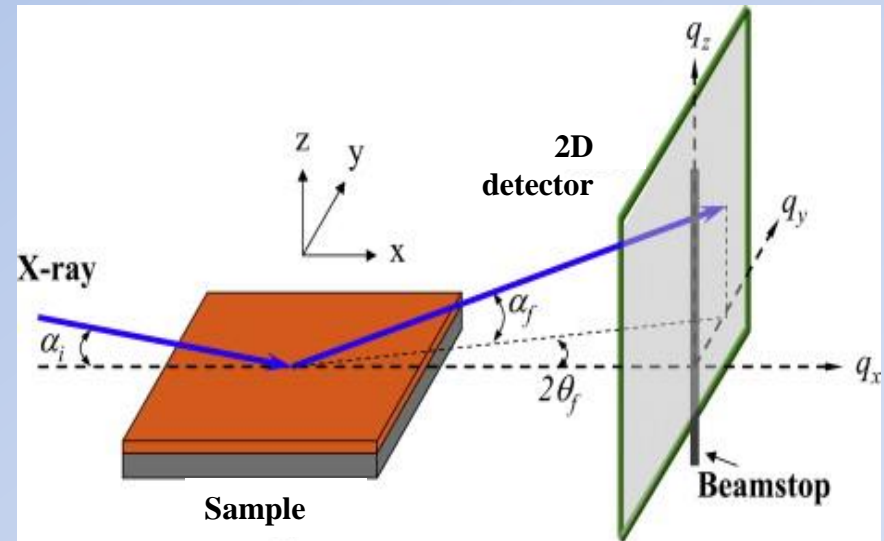
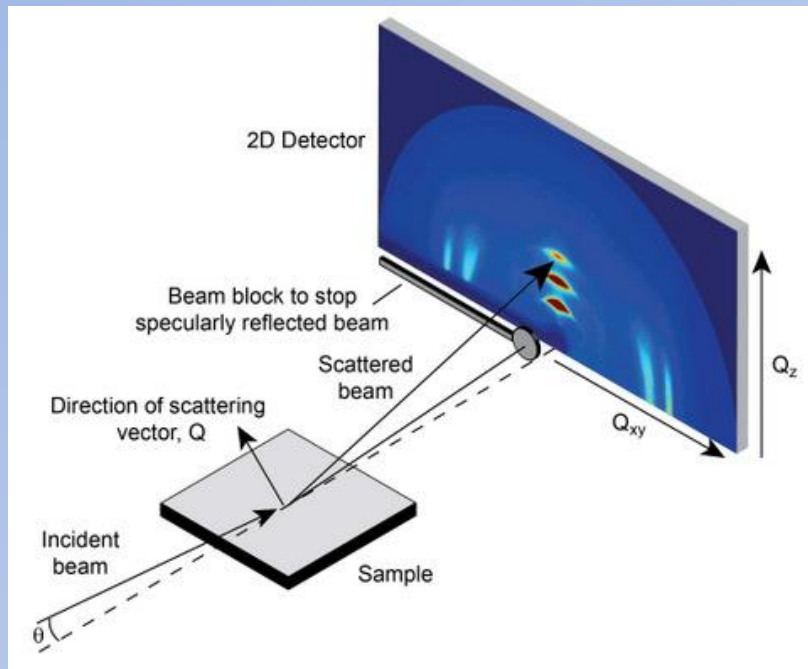
Matej Jergel



*Institute of Physics, Slovak Academy of Sciences  
Bratislava, Slovakia*



# Grazing-incidence small-angle X-ray scattering (GISAXS)



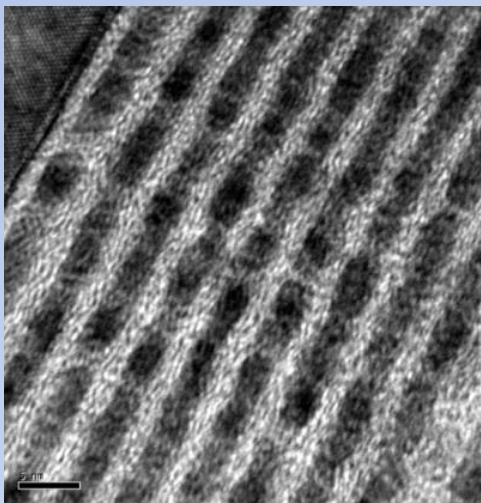
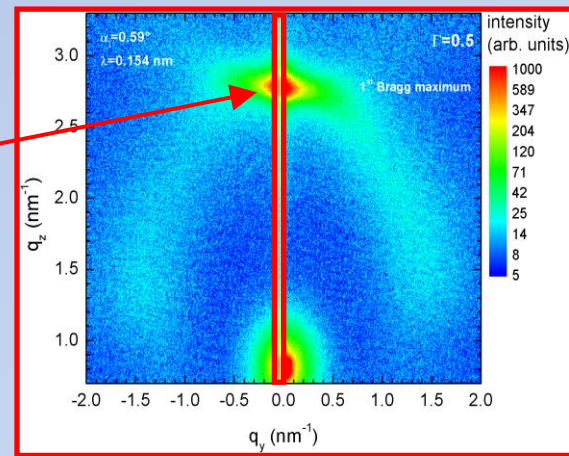
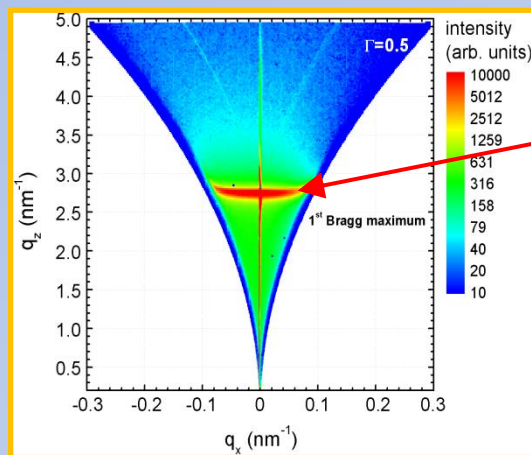
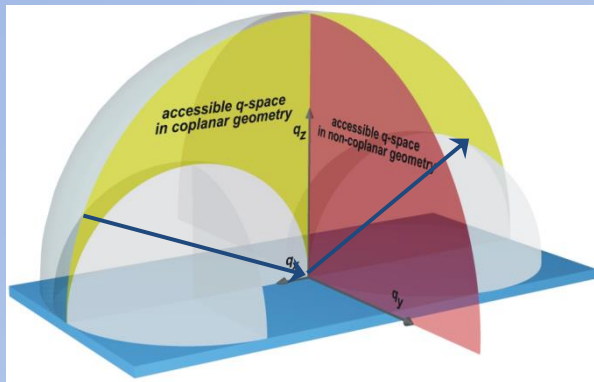
$$\vec{k} = \vec{k}_f - \vec{k}_i \quad \text{scattering vector}$$

$$k_x = 2\pi / \lambda (\cos \alpha_f \cos 2\theta_f - \cos \alpha_i)$$

$$k_y = 2\pi / \lambda \cos \alpha_f \sin 2\theta_f$$

$$k_z = 2\pi / \lambda (\sin \alpha_f + \sin \alpha_i)$$

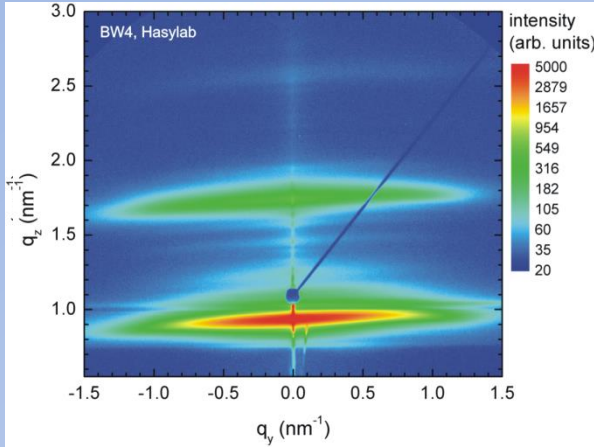
# Coplanar versus non-coplanar geometry



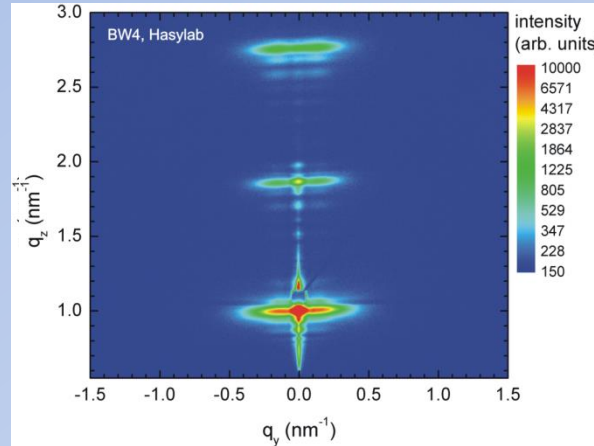
Co/C mirror for water window ( $d = 2.3$ nm,  $N = 200$ )  
cross-sectional TEM reveals granular multilayer

# GISAXS – fast probe of interface quality

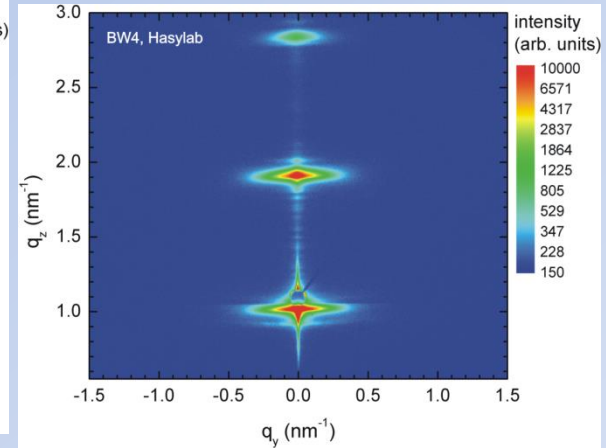
PVD



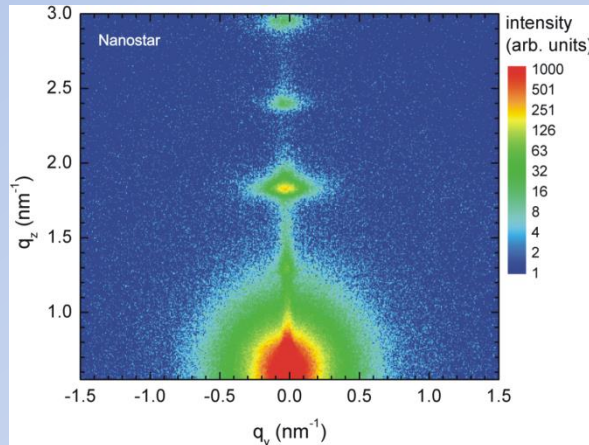
PVD@T=170°C



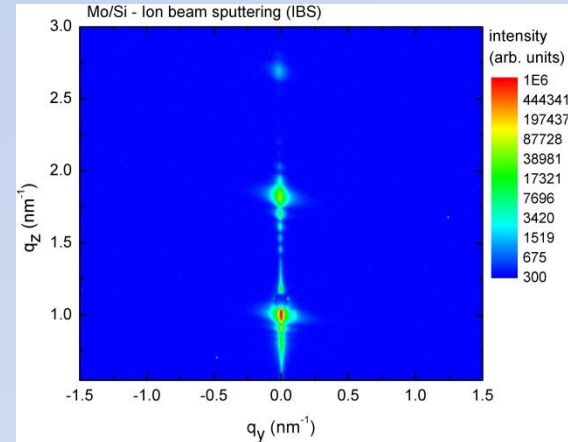
PVD/IB polishing



RF sputtering



ion beam sputtering



Mo/Si mirrors for EUV lithography

# Grazing-incidence small-angle X-ray scattering (GISAXS)

$$\text{CuK}_\alpha \quad \alpha_i = \alpha_f = 0.7^\circ \quad 2\theta_f = 1^\circ$$

$$k_x \hat{=} -0.006 \text{ nm}^{-1} \rightarrow \Lambda_x \hat{=} 1011 \text{ nm}$$

$$k_y \hat{=} 0.7 \text{ nm}^{-1} \rightarrow \Lambda_y \hat{=} 8.8 \text{ nm}$$

$$k_z \hat{=} 1 \text{ nm}^{-1} \rightarrow \Lambda_z \hat{=} 6.3 \text{ nm}$$

- ❖ nanometer-scale in-plane and in-depth resolutions are easily accessible
- ❖ lateral  $k_y$  and vertical  $k_z$  components of the scattering vector are independent of each other => simple BA theory is fully sufficient
- ❖ simple experimental arrangement
- ❖ intuitive shape of GISAXS pattern => modelling is not always necessary

# Grazing-incidence small-angle X-ray scattering (GISAXS)

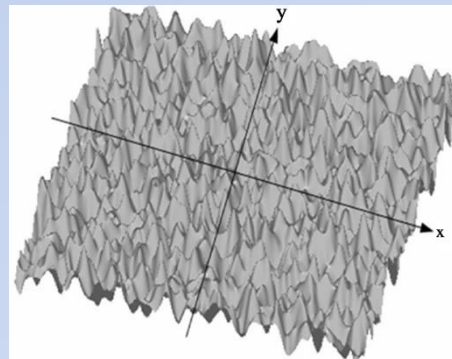
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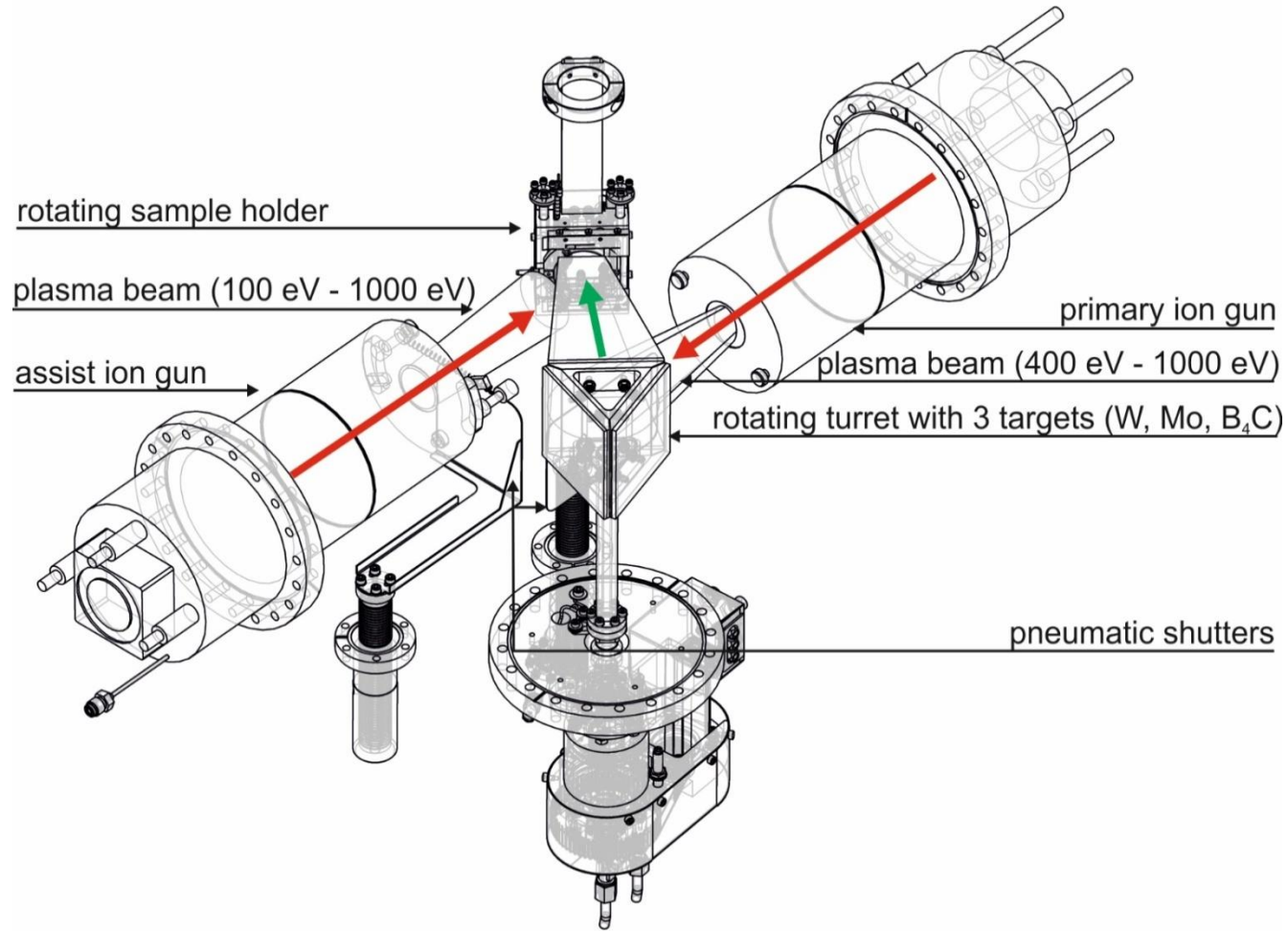
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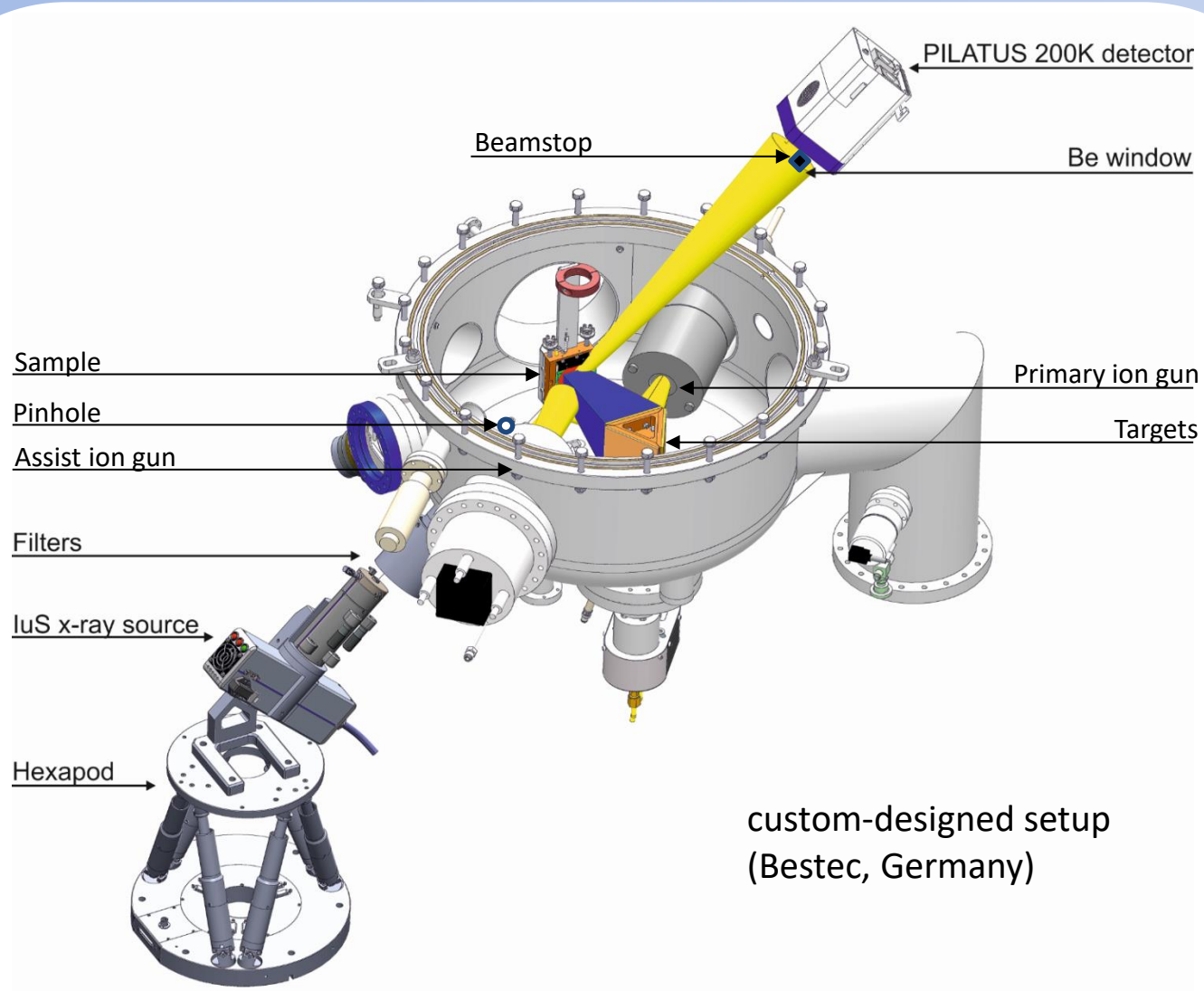
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- ❖ simple experimental arrangement
- ❖ intuitive shape of GISAXS pattern => modelling is not always necessary
- ❖ GISAXS patterns provides FT of 2D autocorrelation function of the probed surface



# Dual ion-beam sputtering system

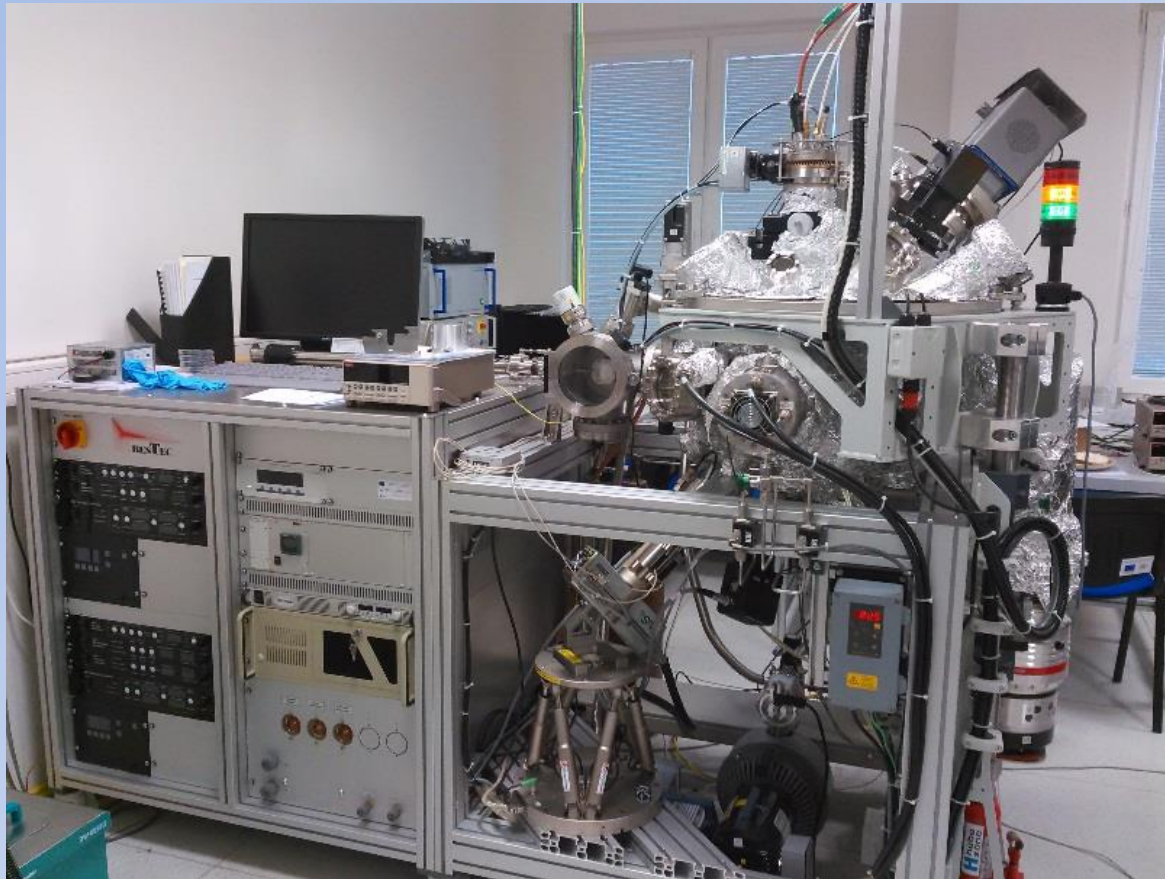


# Dual ion-beam sputtering system



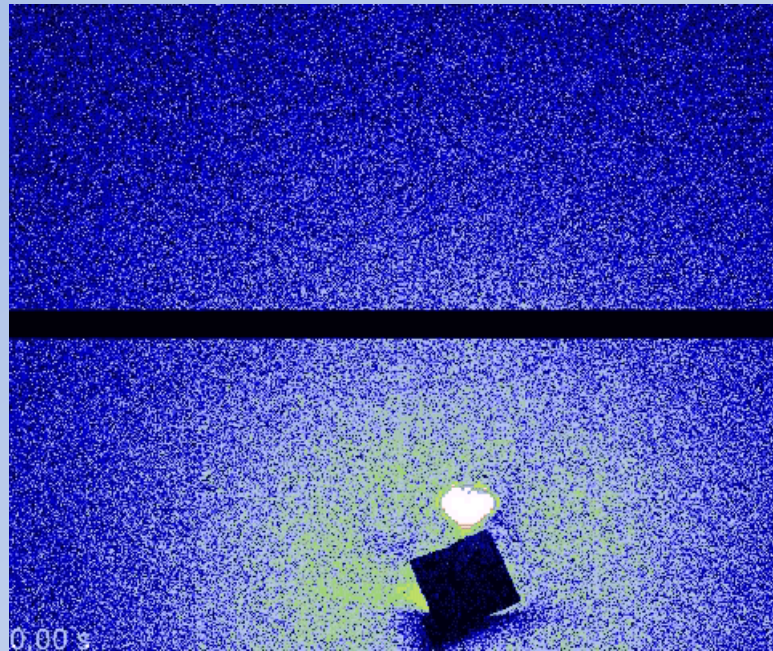


## Dual ion-beam sputtering system



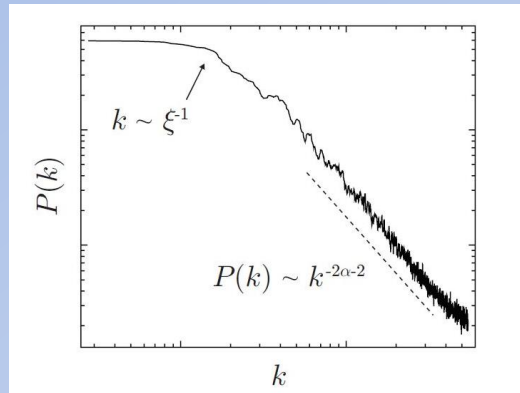
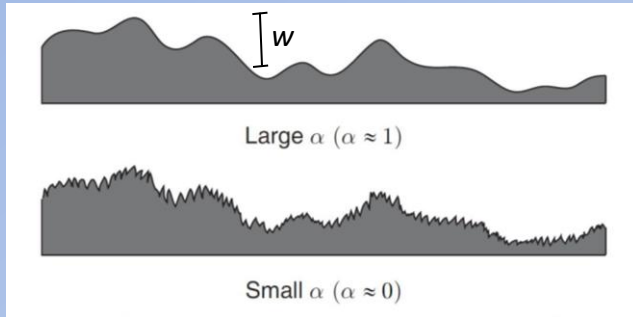
- ❖ 30 W microfocus X-ray source  $I\mu S$  (Incoatec),  $Cu K_{\alpha}$  radiation
- ❖ X-ray detector Pilatus 200K (Dectris)
- ❖ total X-ray flux  $1 \times 10^8$  ph/s

# Multilayer mirror growth by in-situ GISAXS



W/B<sub>4</sub>C mirror, 1.5 nm period, N=15  
( $d_W=0.6$  nm,  $d_{B_4C}=0.9$  nm)  
 $\alpha_i = 0.25^\circ$   
deposition time  $\approx 54$  min.

# Self-affine versus mounded surfaces

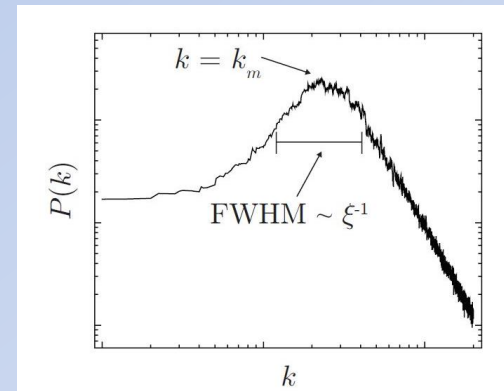
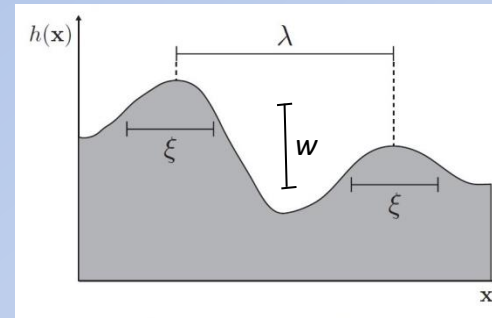


$$R(r) = \exp \left[ - \left( \frac{r}{\xi} \right)^{2\alpha} \right]$$

$\alpha = 1$

$$\text{PSD}(k) = \frac{w^2 \xi^2}{4\pi} \exp \left[ - \frac{k^2 \xi^2}{4} \right]$$

$\alpha$  – fractal (Hurst) parameter



$$\text{PSD}(k) = \frac{w^2 \xi^2}{4\pi} \exp \left[ - \frac{(4\pi^2 + k^2 \lambda^2) \xi^2}{4\lambda^2} \right] J_0 \left( \frac{\pi k \xi^2}{\lambda} \right)$$

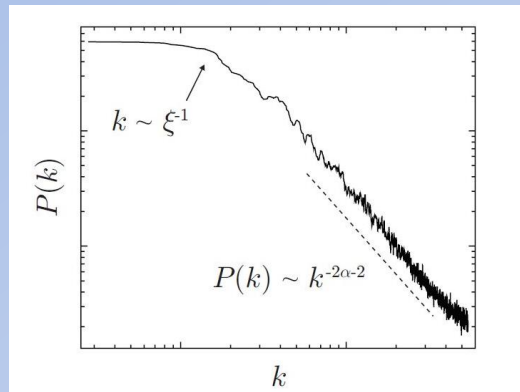
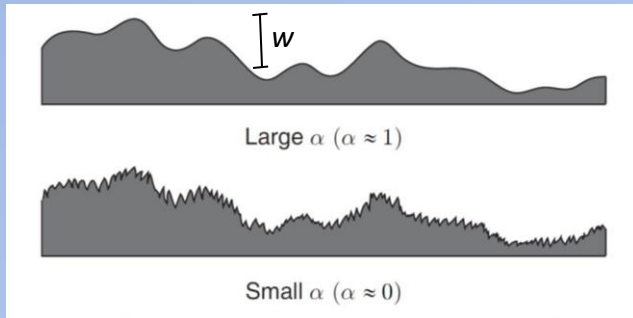
$w$  – interface width (surface rms roughness)

$\xi$  – lateral correlation length

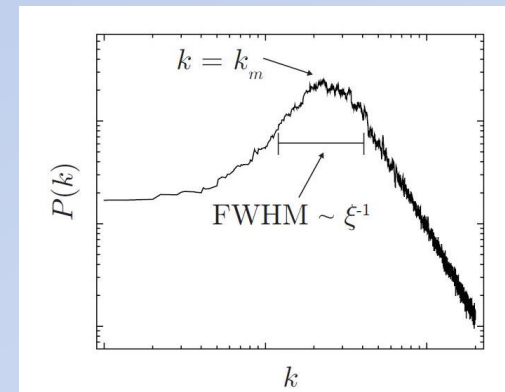
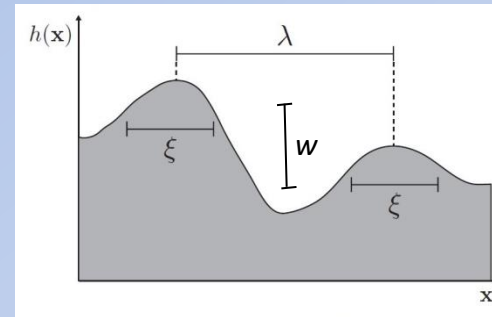
$\lambda$  – mound period (inter-cluster distance)

$J_0$  – Bessel function of zero order

# Self-affine versus mounded surfaces

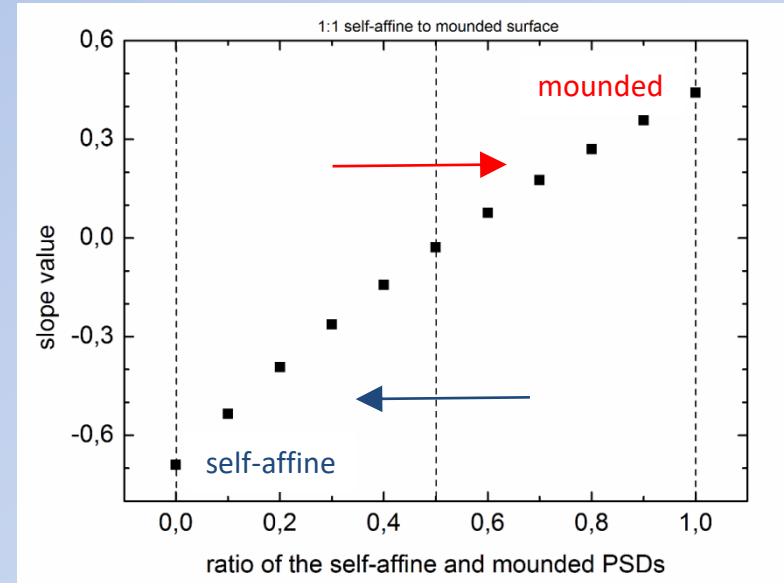
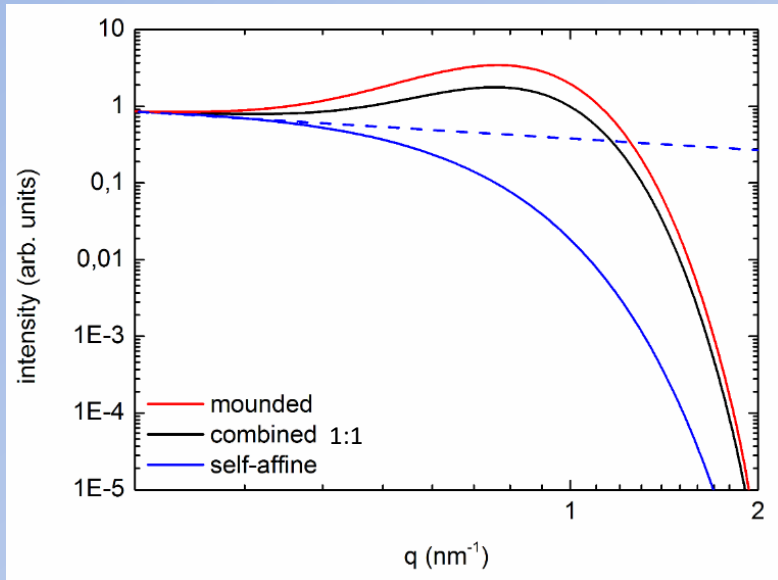


2D layer-by-layer growth



3D cluster growth

# From self-affine to mounded surface



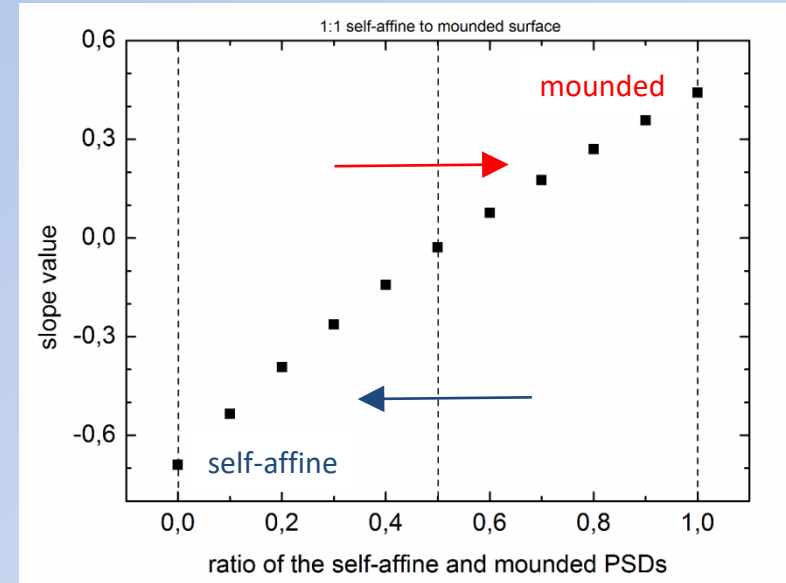
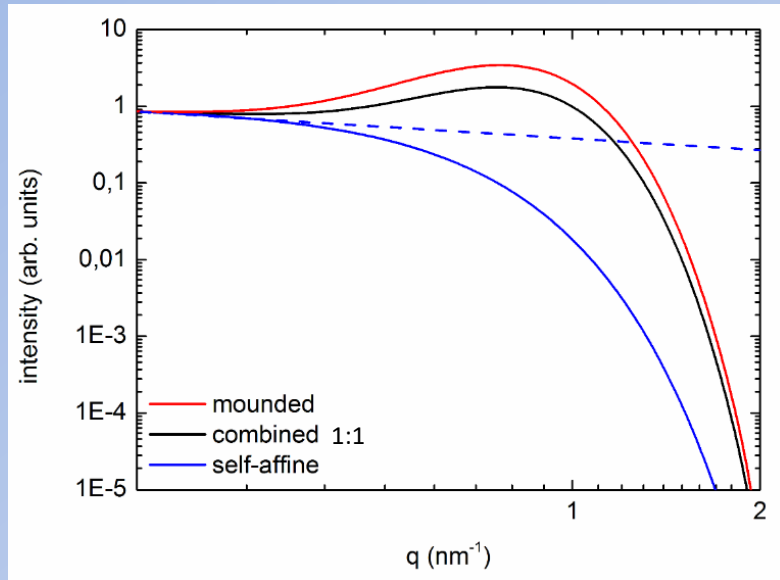
$$m_1 \times \text{PSD}(k)^{\text{self-affine}} + m_2 \times \text{PSD}(k)^{\text{mounded}}$$

( $w = 0.5\text{nm}$ ,  $\xi = 5\text{nm}$ ,  $\lambda = 5\text{nm}$ )

Initial slope values of PSD for different ratios of self-affine and mounded surfaces

slope < 0 => 2D growth  
slope > 0 => 3D growth

# From self-affine to mounded surface



$$m_1 \times \text{PSD}(k)^{\text{self-affine}} + m_2 \times \text{PSD}(k)^{\text{mounded}}$$

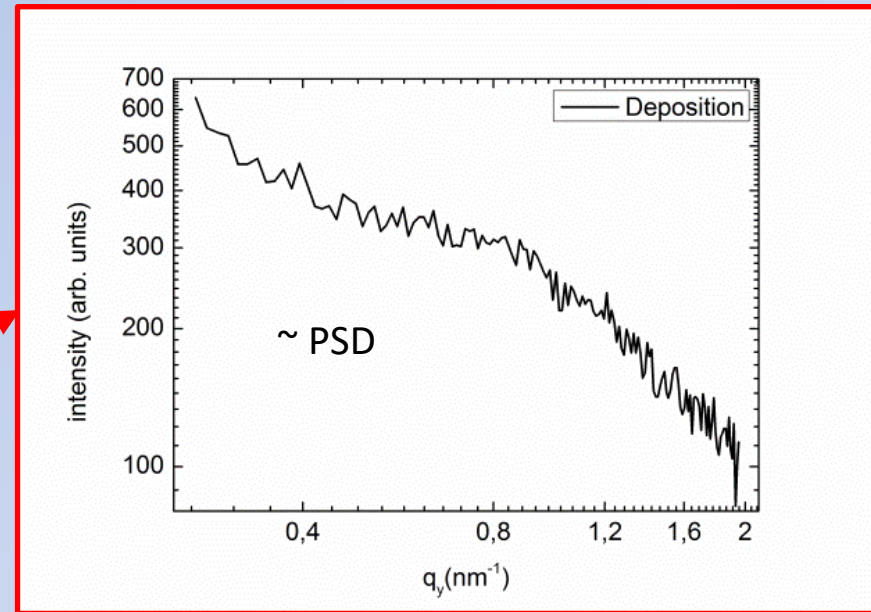
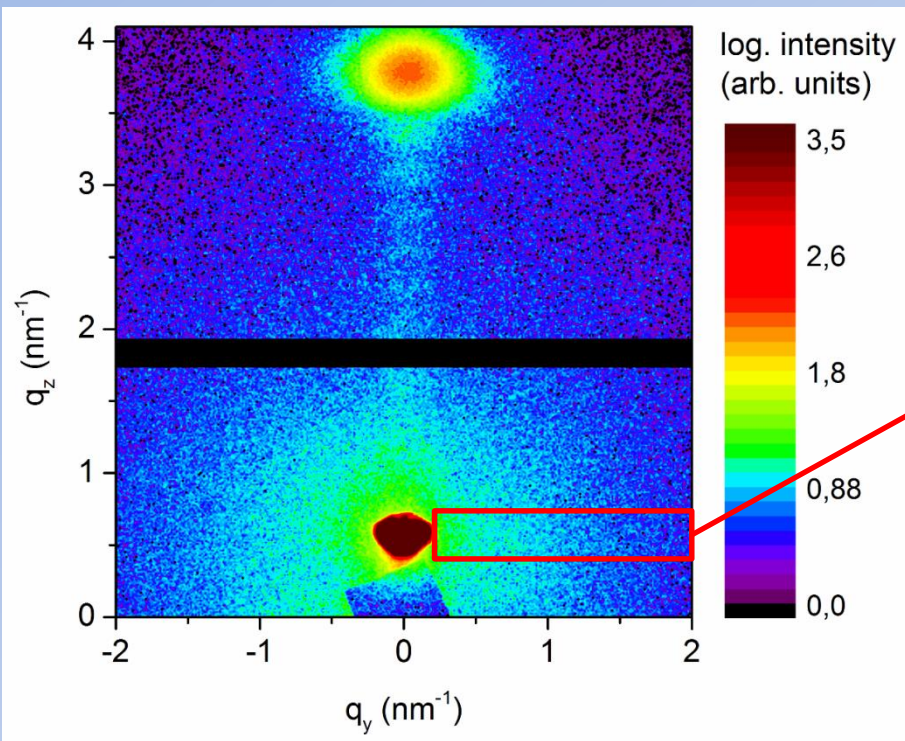
( $w = 0.5\text{nm}$ ,  $\xi = 5\text{nm}$ ,  $\lambda = 5\text{nm}$ )

Initial slope values of PSD for different ratios of self-affine and mounded surfaces

ultra-short period multilayer  
slope < 0

# Power spectral density from GISAXS

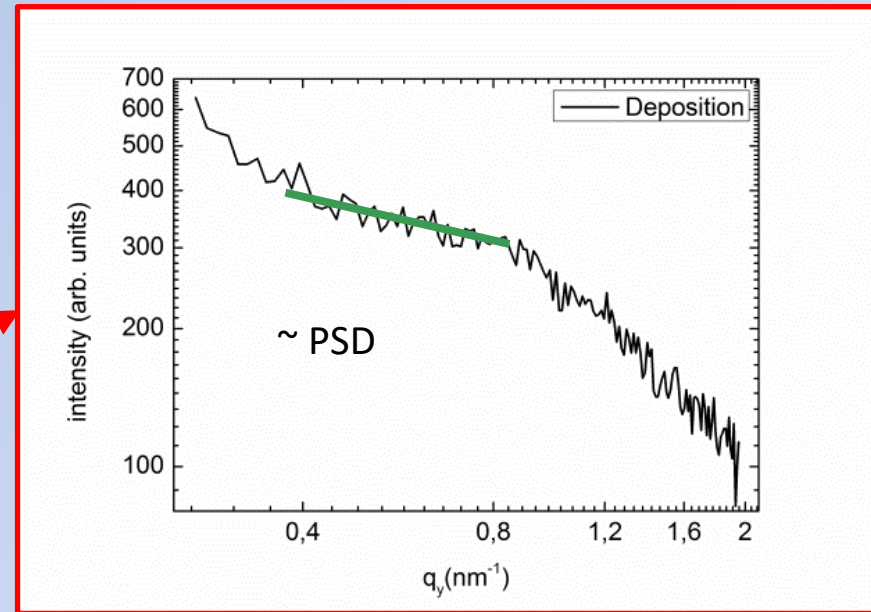
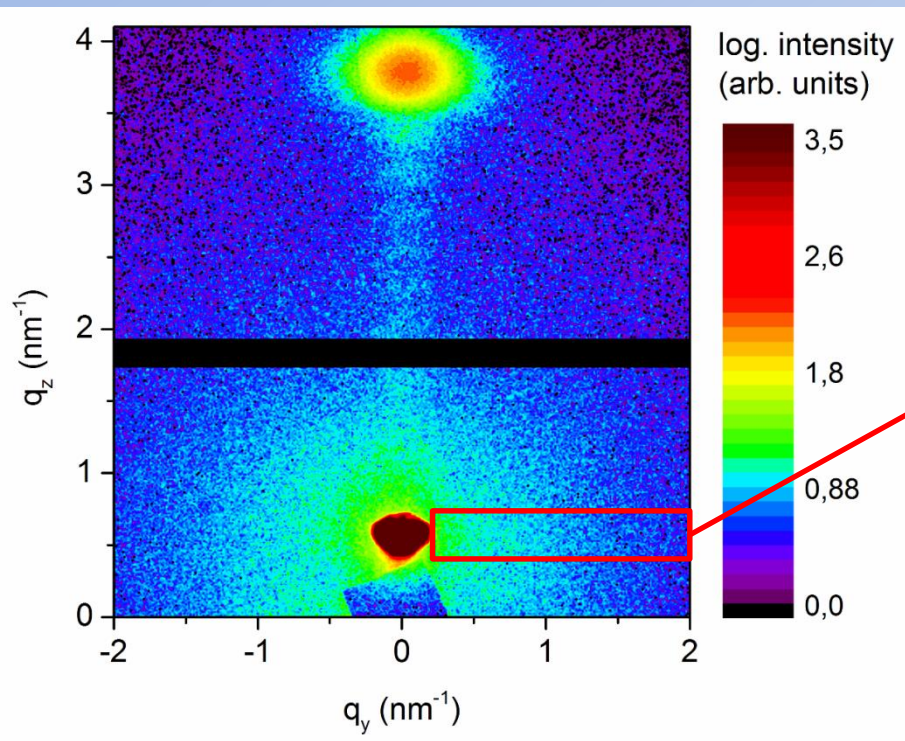
W/B<sub>4</sub>C multilayer on Si/SiO<sub>2</sub> substrate,  $d = 1.5$  nm,  $N = 15$ ,  $\Gamma = 0.33$ ,  $\alpha_i = 0.25^\circ$



Lateral cut of GISAXS pattern at the exit angle close to the critical value for total reflection of the substrate is proportional to PSD function of the growing surface.

# Power spectral density from GISAXS

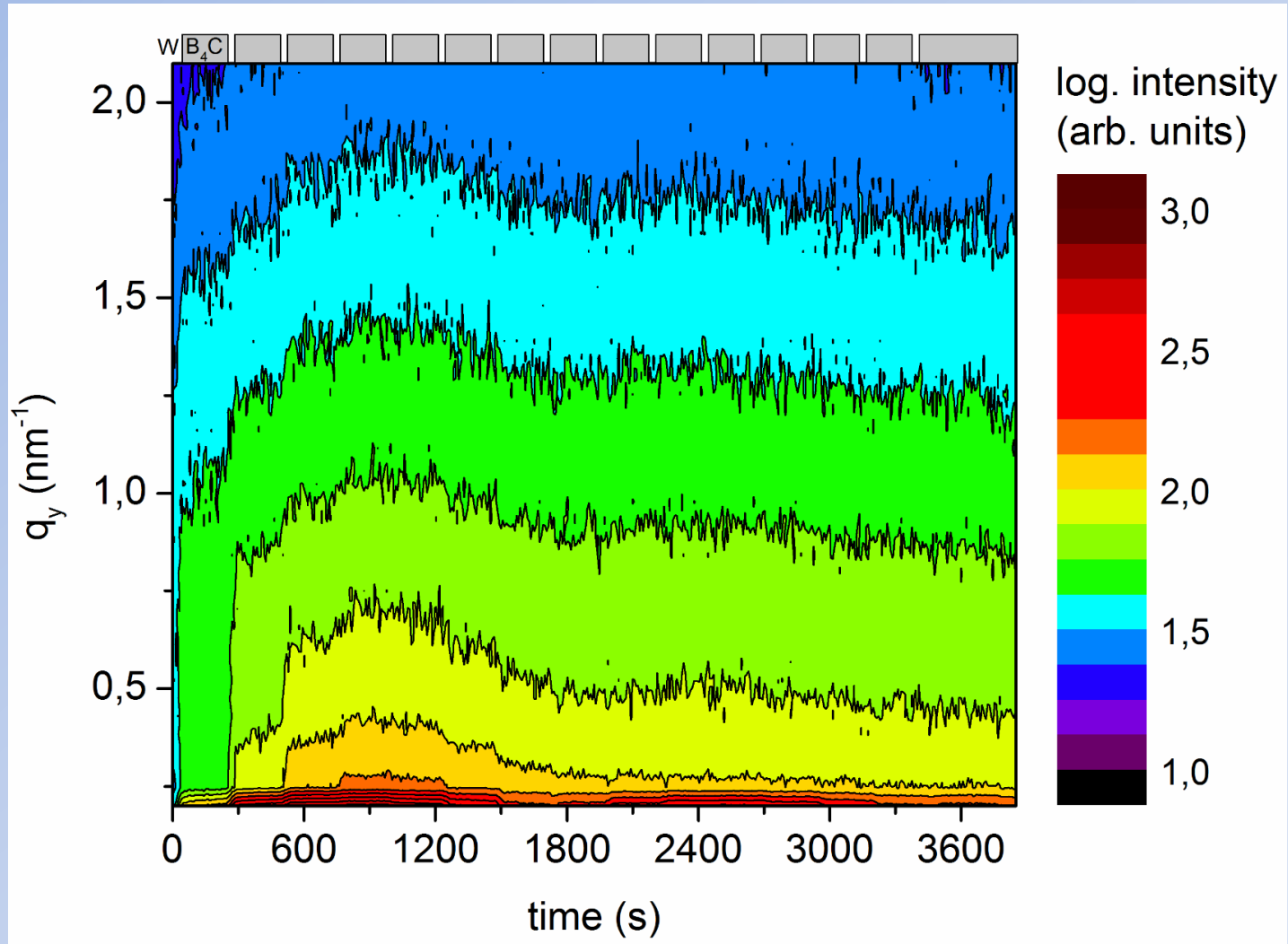
W/B<sub>4</sub>C multilayer on Si/SiO<sub>2</sub> substrate,  $d = 1.5$  nm,  $N = 15$ ,  $\Gamma = 0.33$ ,  $\alpha_i = 0.25^\circ$



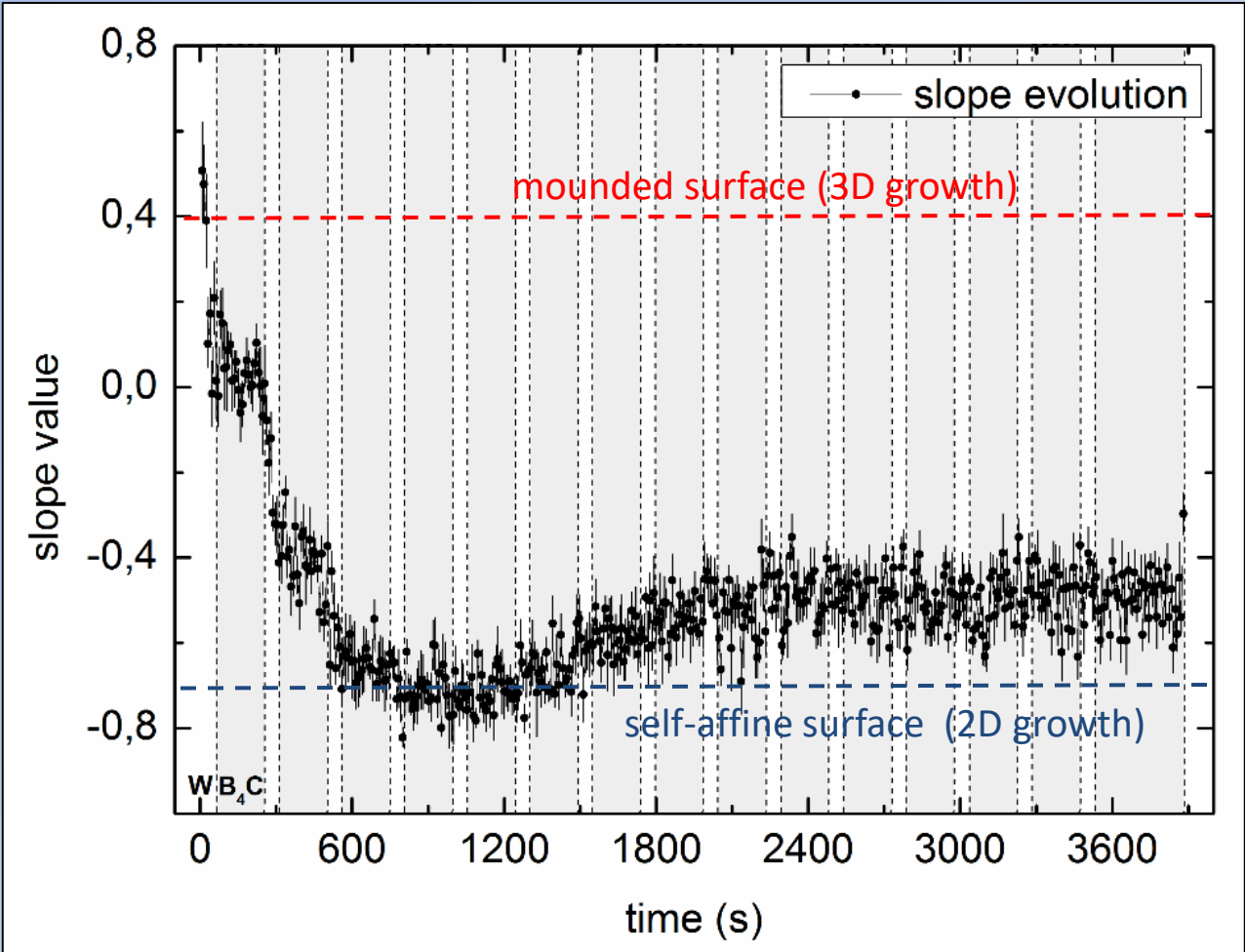
Lateral cut of GISAXS pattern at the exit angle close to the critical value for total reflection of the substrate is proportional to PSD function of the growing surface.



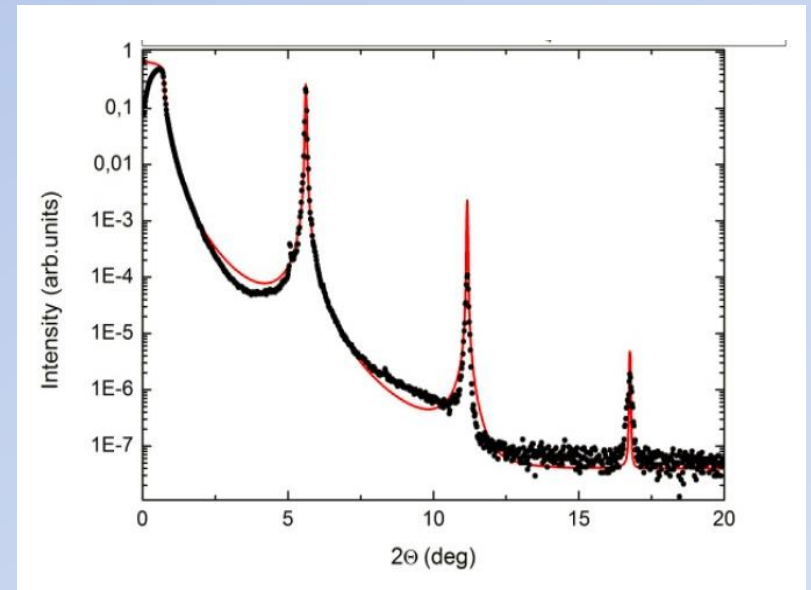
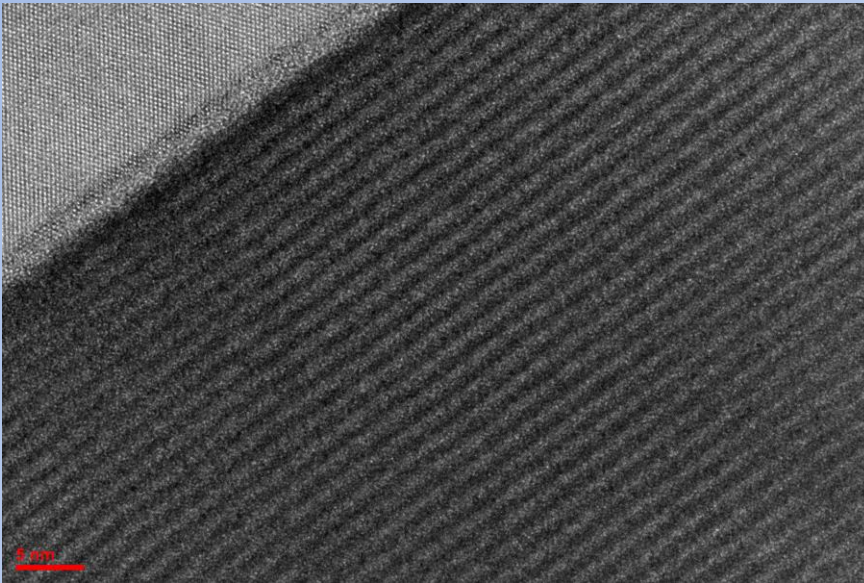
# Temporal evolution of lateral cuts of GISAXS patterns



# Temporal evolution of PSD slope derived from GISAXS

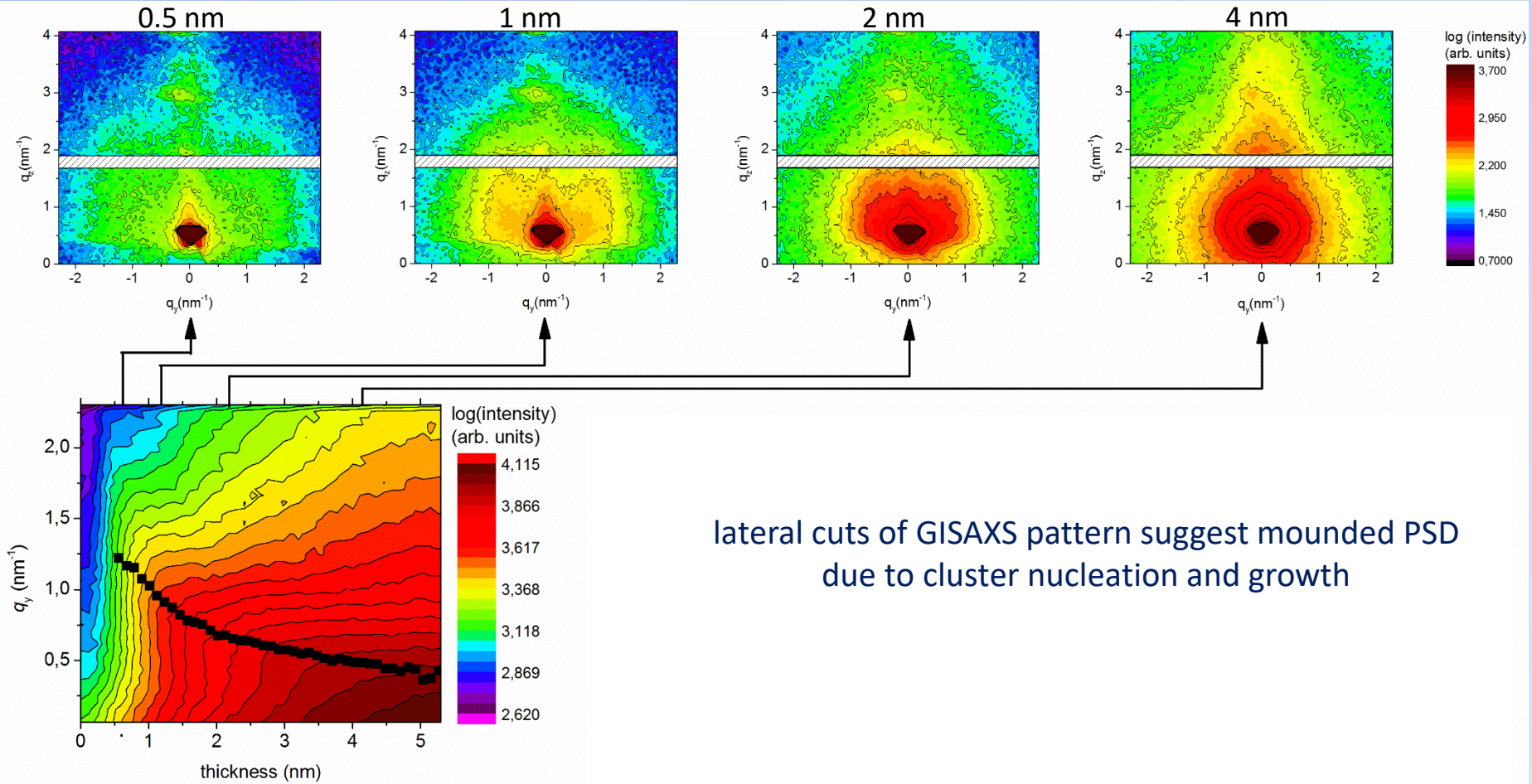


# TEM, XRR

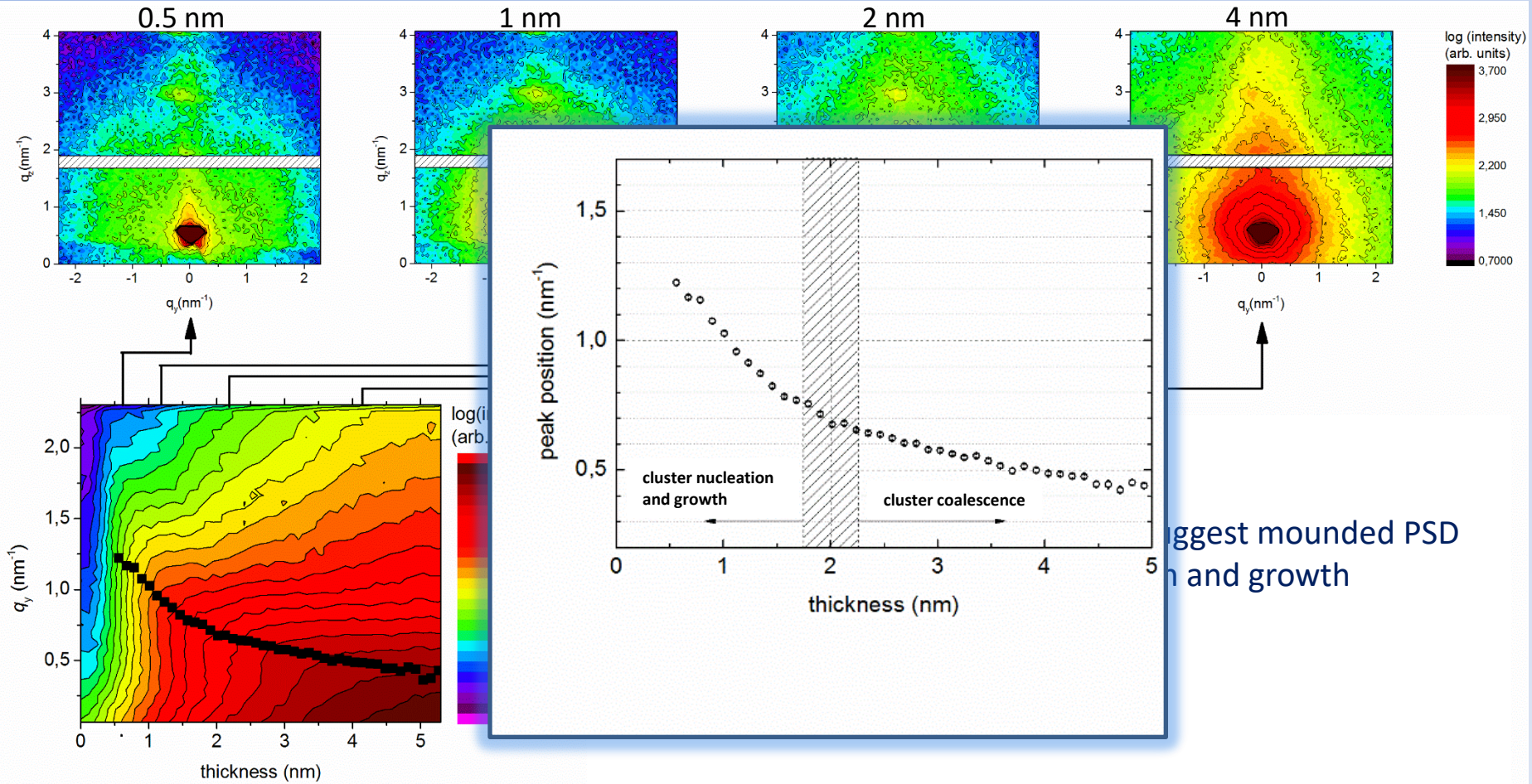


W/B<sub>4</sub>C multilayer on Si/SiO<sub>2</sub> substrate,  $d = 1.5$  nm,  $N = 300$ ,  $\Gamma = 0.33$ ,  $\alpha_i = 0.25^\circ$

# 3D growth of tungsten on SiO<sub>2</sub>



# 3D growth of tungsten on SiO<sub>2</sub>



largest mounded PSD  
n and growth

# Message

- 👉 Buildup of interfaces in W/B<sub>4</sub>C mirrors is governed by an interplay between W agglomeration on SiO<sub>2</sub> and counteracting 2D layer-by-layer growth favored by B<sub>4</sub>C.
- 👉 “Healing” effect of B<sub>4</sub>C on the interface roughness plays a crucial role in the preparation of ultrashort-period multilayer mirrors without a need for additional interface treatment.
- 👉 In-situ GISAXS analysis revealed an additional role of the spacer beyond a merely optical one.
- 👉 Potential of laboratory-based in-situ GISAXS for analyses of multilayer growth was demonstrated using the latest-generation microfocus X-ray sources and fast 2D detectors.

## Co-workers

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## Projects

- ❖ European Science Foundation, COST MP1307, CA15107
- ❖ European Research and Development Agency, ITMS 26220220170
- ❖ M-ERA.Net, XOPTICS
- ❖ Slovak Research and Development Agency, APVV-14-0745
- ❖ Grant Agency VEGA Bratislava, projects 2/0004/15

**Thank you for your attention !**

