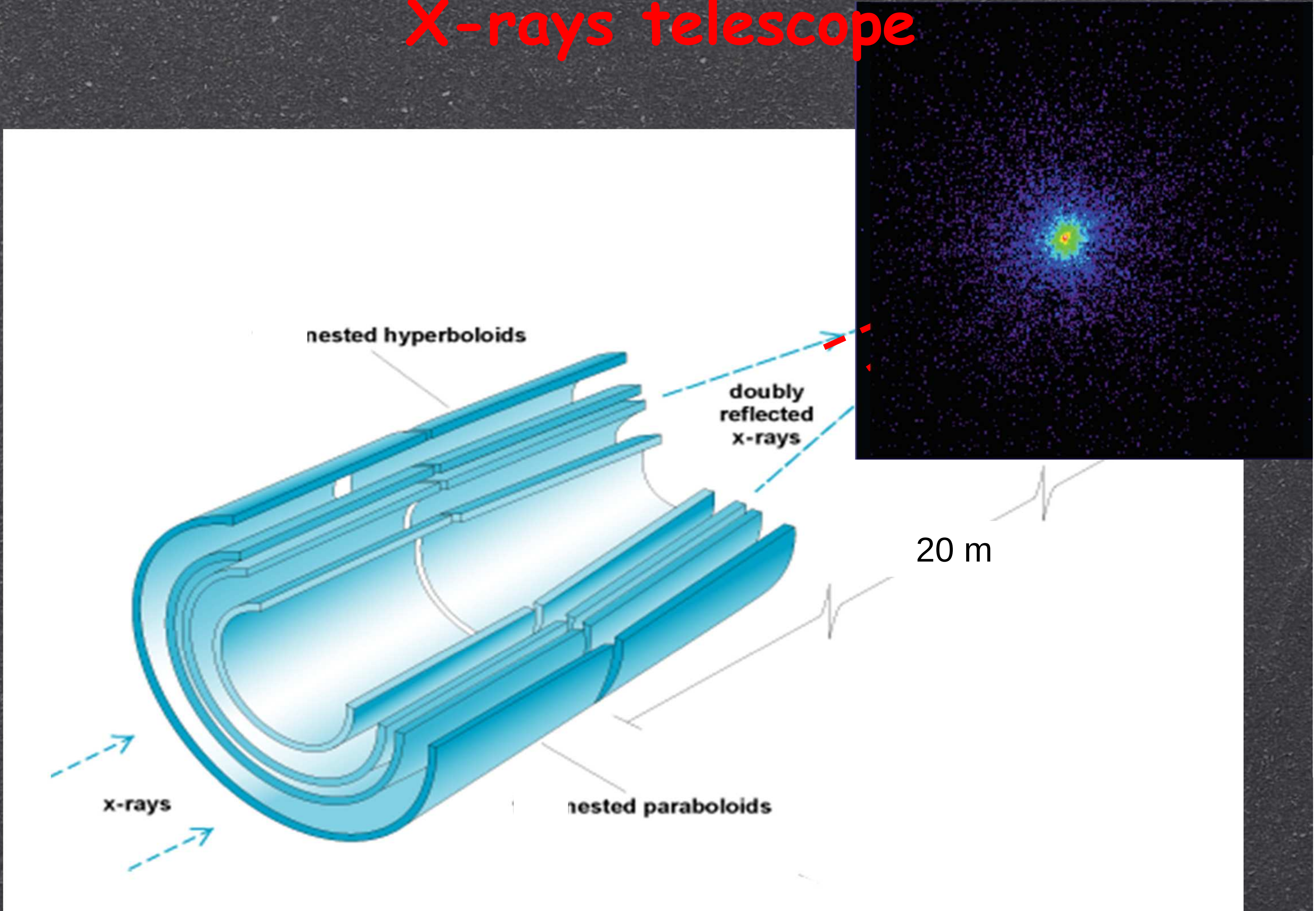
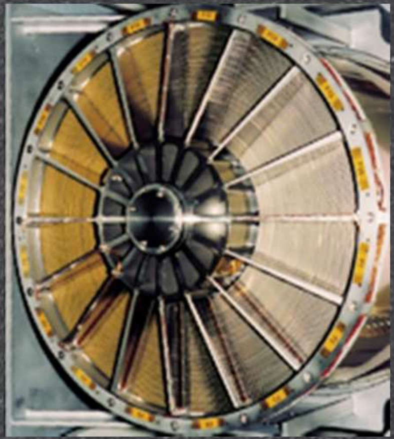
X-ray scattering of periodic and graded multilayer: comparison of experiments to simulation from surface microroughness characterization

X-Ray Optic Group, INAF OAB

X-rays telescope



XMM-Newton



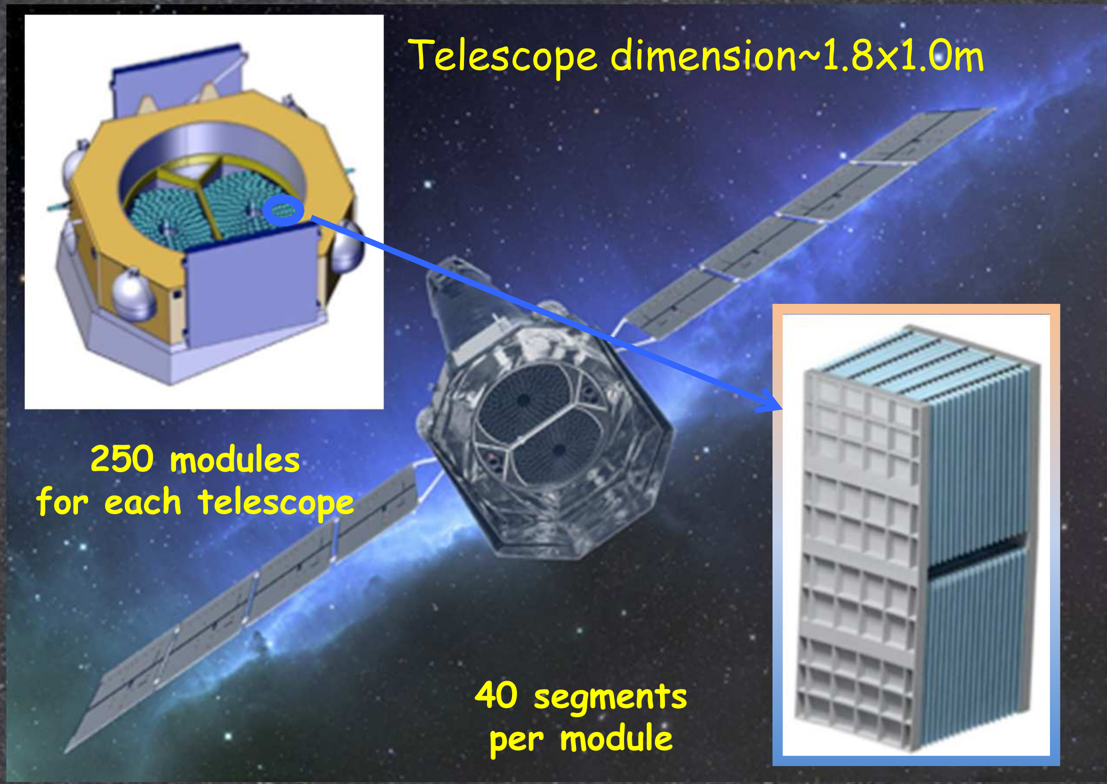
58 mirrors/telescope
Ø outer mirror = 70 cm

Electroformed Nickel
Au coating

HEW = 15 arcsec
Maximum energy = 15 keV

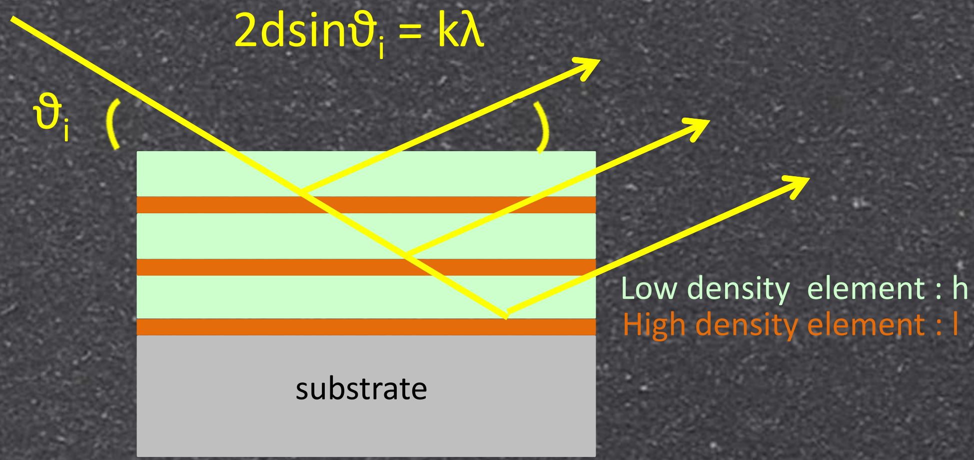
ATHENA

(Advanced Telescope for High ENergy Astrophysics)
formerly IXO (X-Ray Obstervatory)

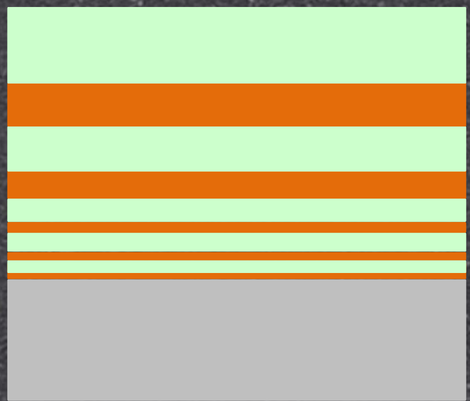


Coated with …

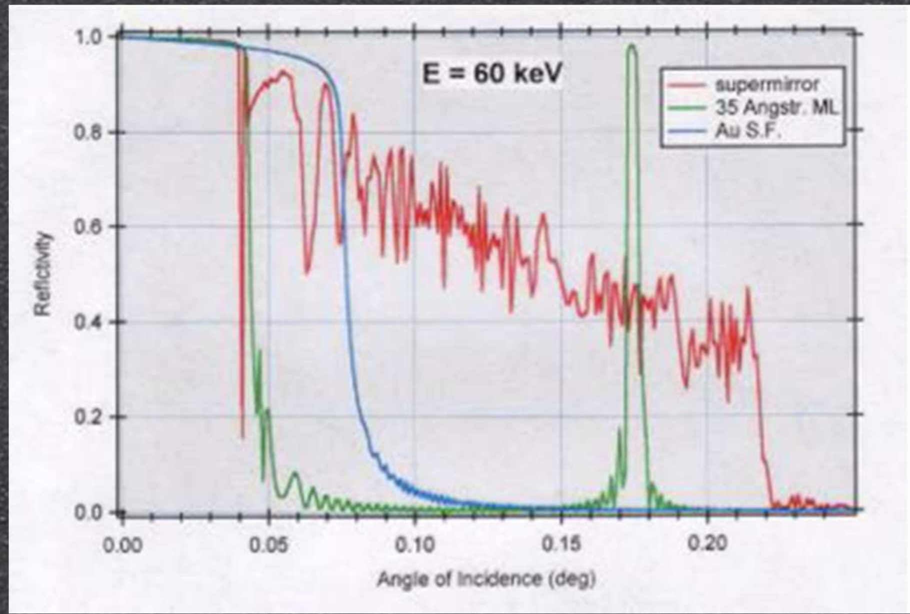
… graded multilayers to reflect hard X-rays



periodic



depth graded

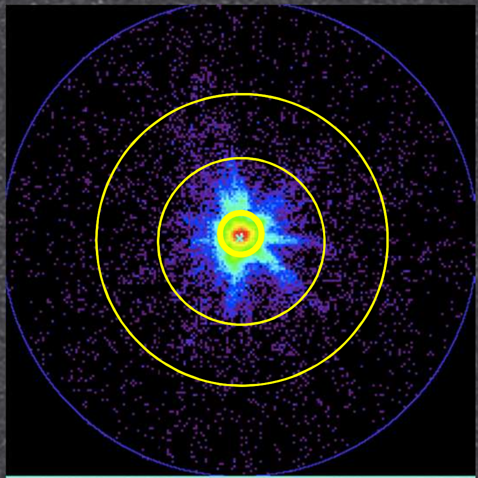


$$d(k) = \frac{a}{(b+k)^c}$$

$$\Gamma = \frac{d_h}{d_h + d_l}$$

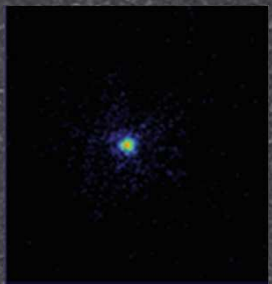
Requirement for Athena : HEW < 10 arcsec (0.1-7 keV) , goal 5 arcsec

HEW : Half Energy Width

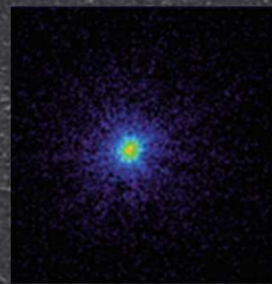


5 arcsec ~ 24 μrad !!

10.000 glasses shaped
with replication technique !!



We have to control the scattering because it affects the HEW



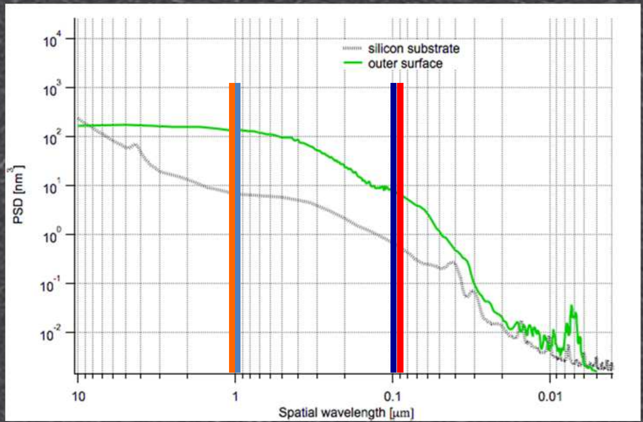
$$HEW(\lambda) = f\left(H_{figure-errors}\right) + f(H_{XRS}(\lambda))$$

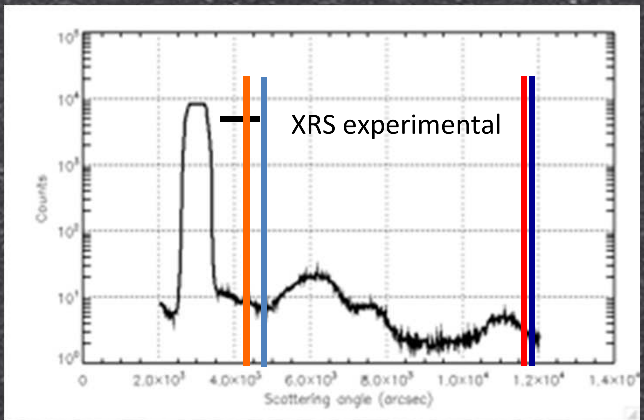
$$H_{XRS}(\lambda) = f(roughness)$$

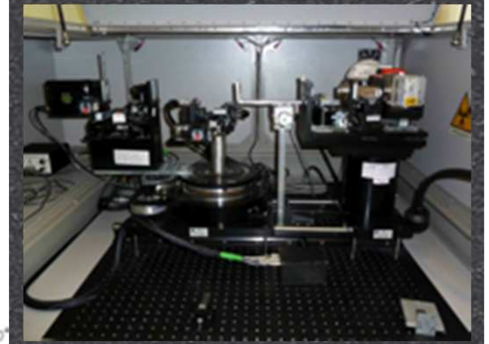
We measure roughness with AFM and XRS with diffractometer. WHY AFM ?

Where do AFM spatial frequencies do scatter ?

AFM







X-ray diffractometer

For E=8.045 keV (1.54 Å)

$$l = \frac{1}{f} = \frac{\lambda}{\left|\cos\vartheta_s - \cos\vartheta_i\right|}$$

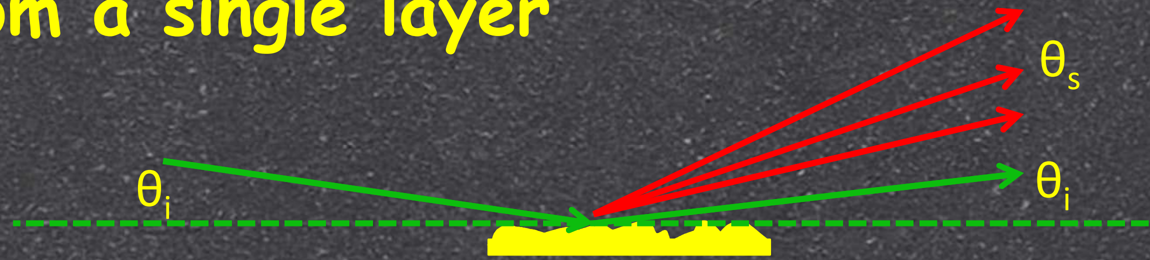
ϑ_i [arcsec]	l [µm]	ϑ_s [arcsec]
3000	10	3211
"	1	4701
"	0.1	11835
"	0.01	36368
2200	10	2480
"	1	4236
"	0.1	11657
"	0.01	36310

How we compute the XRS from roughness data ?

- X-ray scattering is computed within the first order perturbation theory for multilayers

- The roughness evolution of layers other then the substrate and the outer one are computed with the Stearns model

➢ X-ray scattering
from a single layer

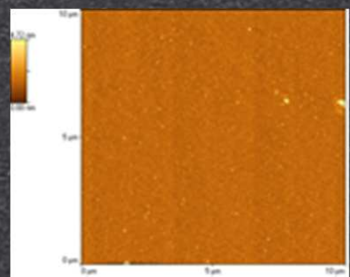


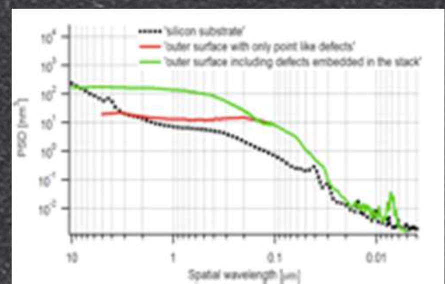
First order perturbation theory
- ✧ Smooth ($2\sigma \sin\vartheta_i \ll \lambda$)
- ✧ and isotropic surface

$$\frac{1}{I_0}\frac{dI_s}{d\theta_s} = \frac{16\pi^2}{\lambda^3} Q_{is} \sin^2\theta_s \sin\theta_i P(f)$$

$$Q_{is} = [r_F(\theta_s)\, r_F(\theta_i)]^{1/2}$$

Q_{is} : polarization factor
r_F : Fresnel reflectivity





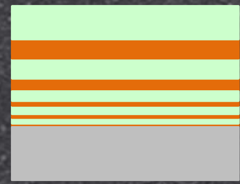
AFM　　PSD (P)

➢ X-ray scattering

formula for a multilayer

both periodic and graded

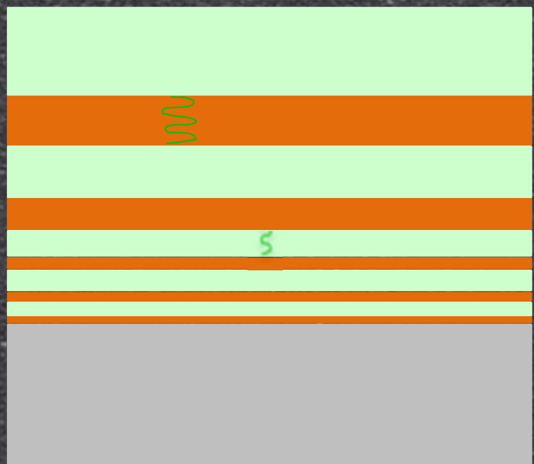
periodic



depth graded

$$\frac{1}{I_0}\frac{dI_s}{d\theta_s} = \frac{16\pi^2}{\lambda^3} Q_{is} \sin^2\theta_s \sin\theta_i \left[P_{unc}(f)+P_{corr}(f)\right] =$$

$$= \frac{16\pi^2}{\lambda^3} Q_{is}\sin^2\theta_s\sin\theta_i\left[\sum_i T_i^2 P_i(f) + 2\sum_{i>j}(-1)^{i+j} C_{ij}(f)\, T_i T_j \cos(\alpha\Delta_{ij})\right]$$



electric fields

depth separation

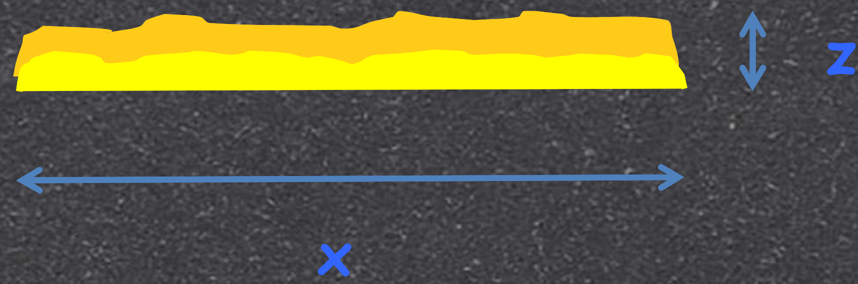
$\alpha = 2\pi/\lambda(\sin\theta_s + \sin\theta_i)$

➢ Roughness growth model (to compute internal PSD)

for a single layer

$$\frac{\partial z(x)}{\partial \tau} = -\nu \left| \nabla^n z(x) \right| + \frac{\partial \eta}{\partial \tau}$$

Stearns 1998



SMOOTHING PROCESS

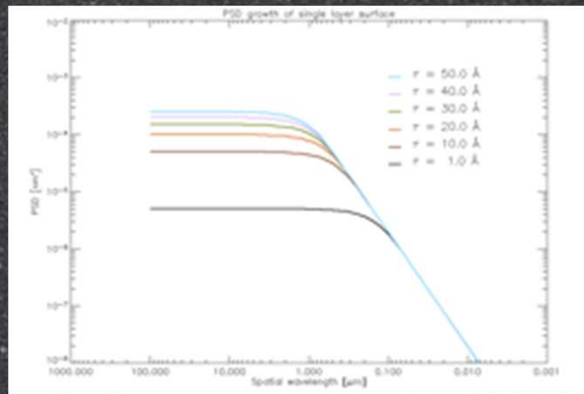
- ν : linked to the growth spatial frequencies
- n : positive integer which depends on the smoothing process

INCREASE IN ROUGHNESS

- η : random shot noise of the deposition process
- τ : layer thickness

$$P^{\mathrm{int}}(f) = \Omega \frac{1 - \exp(-2\nu |2\pi f|^n \tau)}{2\nu |2\pi f|^n}$$

- Ω : volume of the deposited particles
- $l^* = (\nu\tau)^{1/n}$



➢ Roughness growth model (to compute internal PSD)

for a multilayer

$$P_i(f) = P_i^{\text{int}}(f) + P_i^{ext}(f) = P_i^{\text{int}}(f) + a_i(f) P_{i-1}(f)$$

Intrinsic PSD due to the deposition process

Replication on the underlying layer

replication factor

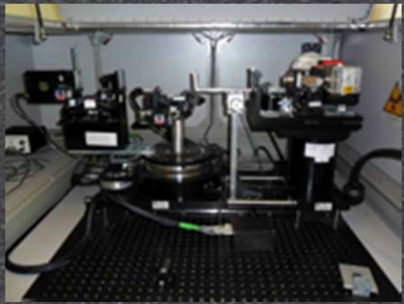
$a_i(f) = \exp(-\nu_i |2\pi f|^n \tau_i)$

… to calculate P_i and C_{ij} in the scattering formula

$$\frac{1}{I_0}\frac{dI_s}{d\theta_s} = \frac{16\pi^2}{\lambda^3} Q_{is} \sin^2\theta_s \sin\theta_i \left[\sum_i T_i P_i(f) + 2\sum_{i>j} (-1)^{i+j} C_{ij}(f) T_i T_j \cos(\alpha \Delta_{ij}) \right]$$

EXPERIMENTAL and SIMULATION

➢ XRR



X-rays diffractometer: BEDE scientific, mod. D1

➢ PPM

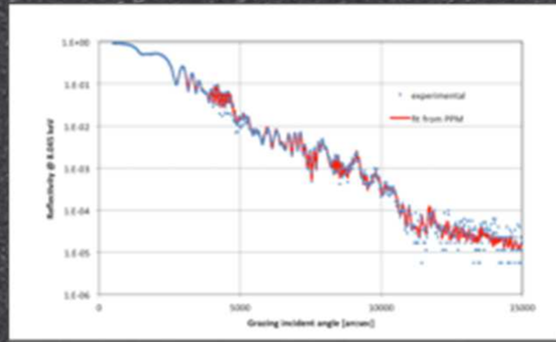
PPM (Pythonic Program for Multilayer), Mirone ESRF

INPUT

✧ Nominal power law and densities

OUTPUT

✧ Actual layers thicknesses and densities



➢ AFM

AFM: Bruker (formely Veeco) mod. EXPLORER standalone

Measure substrate and outer layer

➢ Roughness growth

INPUT

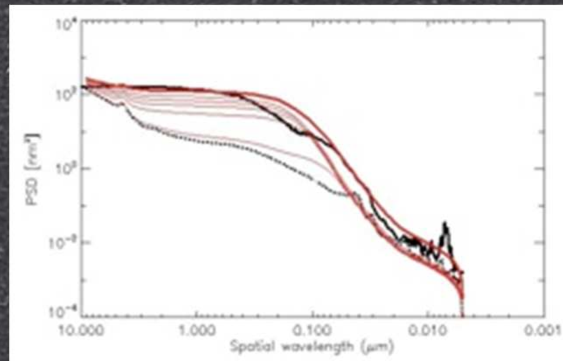
✧ PSD for substrate and outer layer

✧ Stack parameters from PPM

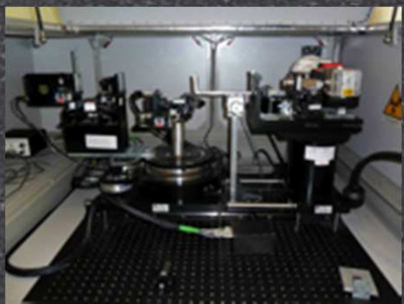
✧ Ω_l l_l n_l and Ω_h l_h n_h

OUTPUT

✧ P_i , C_{ij}



➢ XRS



X-rays diffractometer: BEDE scientific, mod. D1

➢ XRS diagram

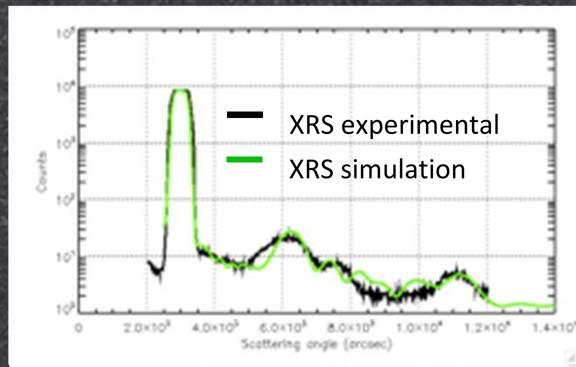
INPUT

✧ Stack parameters from XRR/PPM

✧ P_i , C_{ij} from AFM/Stearns model

OUTPUT

✧ XRS diagram



Samples

Sample 1 Substrate : silicon
Periodic multilayer: N=40 bi-layers, W/Si, d=4.6Å, Γ=0.42,
deposited by e-beam evaporation

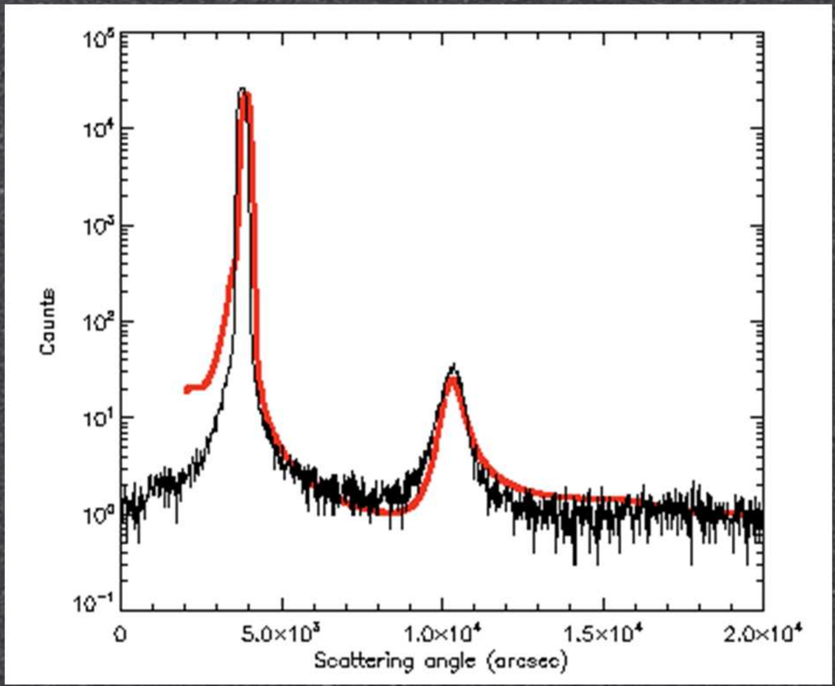
Sample 2 Substrate : silicon
Graded multilayer: N=100 bi-layers, Pt/C,
deposited by magnetron sputtering

Program upgrading validation

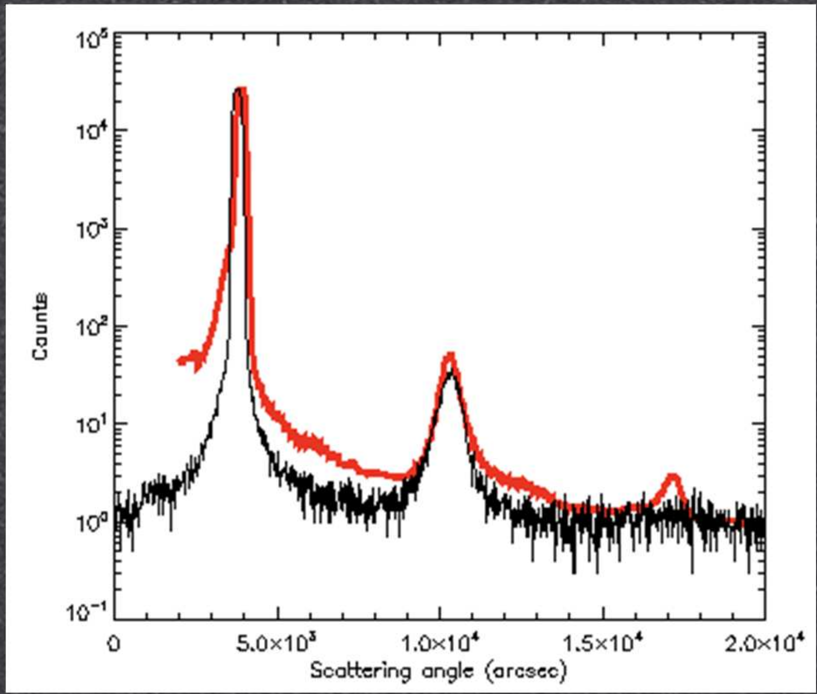
created for periodic multilayers (Canestrari,2006), upgraded to graded multilayers (Salmaso,2011/12)

Sample 1: PERIODIC, W/Si

Incident energy = 8.045 keV (Cu Kα_1 line)
Incidence angle = first Bragg peak



With roughness growth



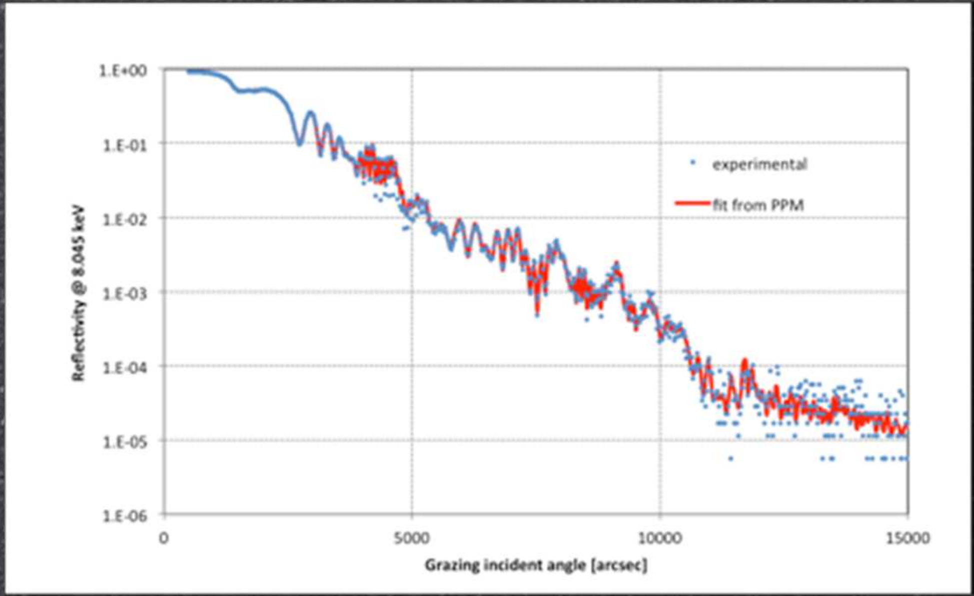
No roughness growth

Application to graded multilayers

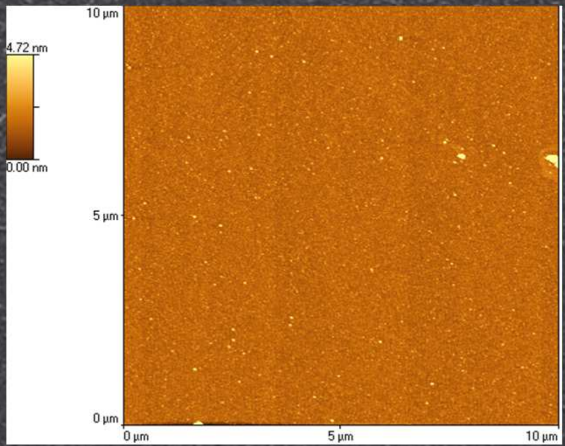
Sample 2, Pt/C

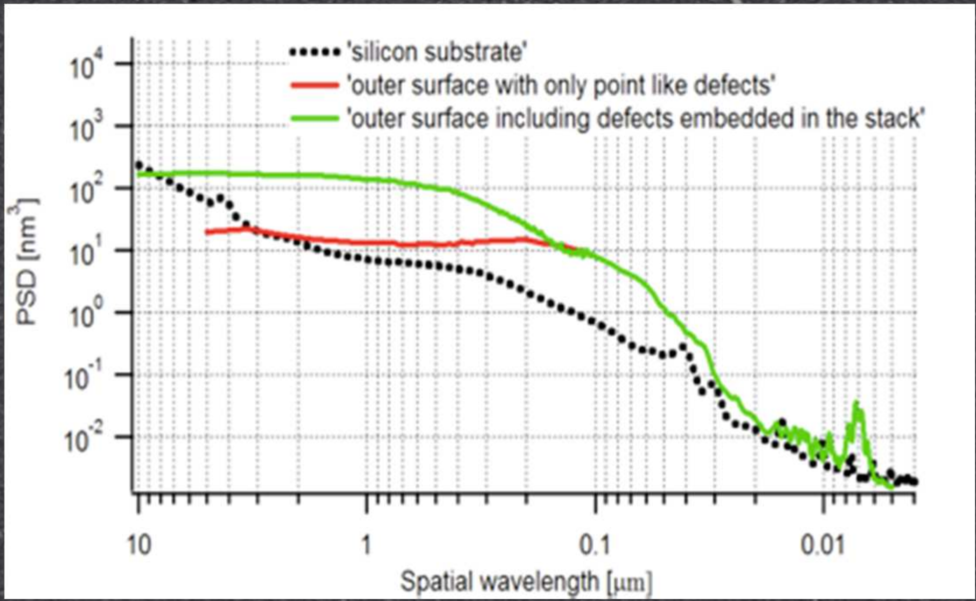
- **XRR measurements**

Incident energy = 8.045 keV (Cu Kα_1 line)



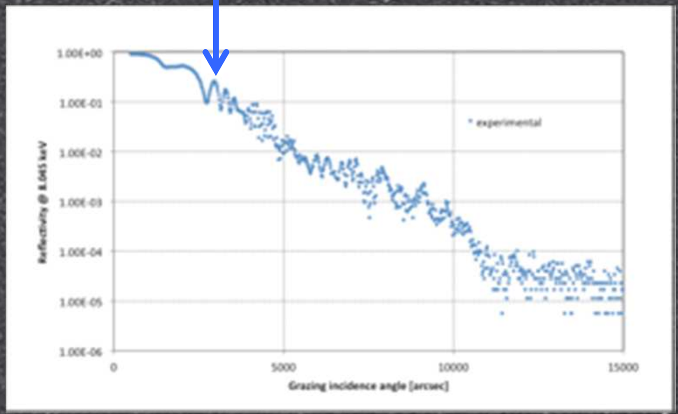
- **AFM measurements**



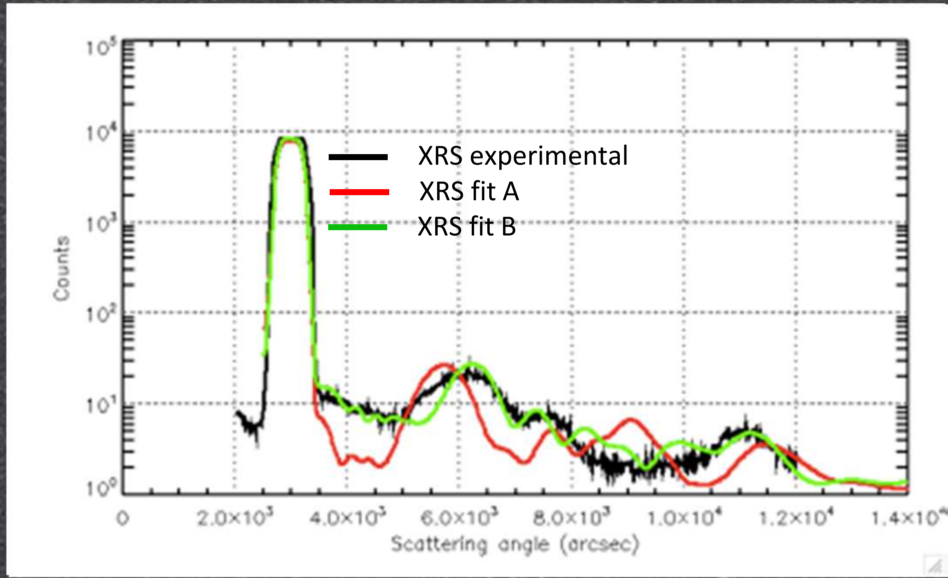


Stack parameters optimization

θ_i=3000 arcsec



➢ XRS measurements 1

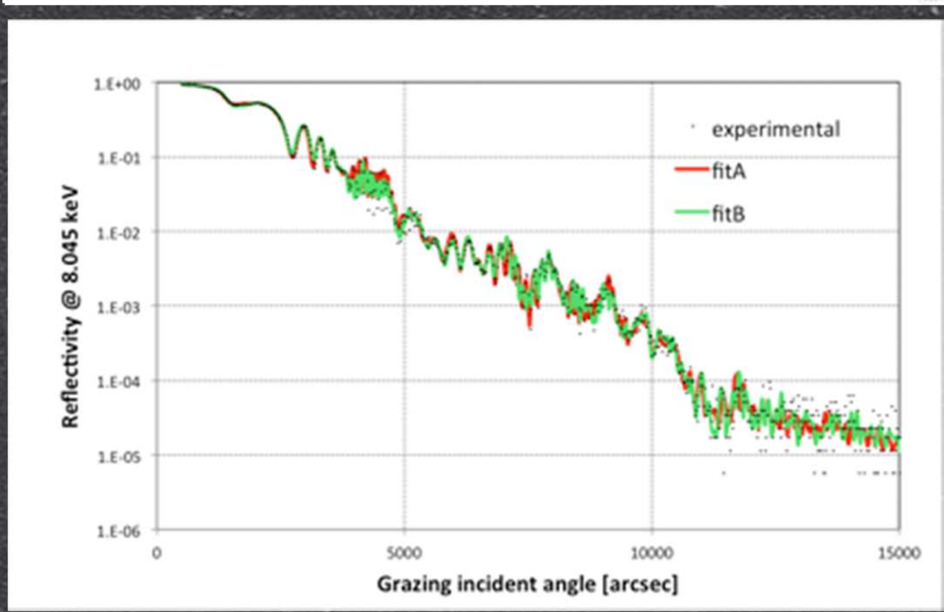


Fit A : stack parameters from PPM

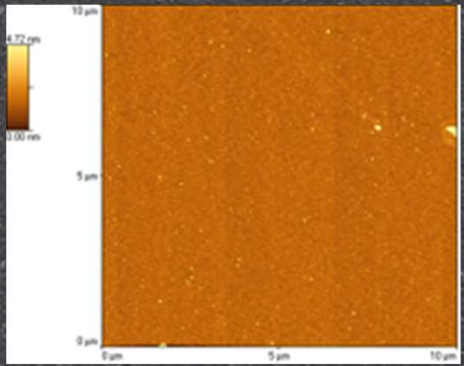
Fit B : optimized stack parameters

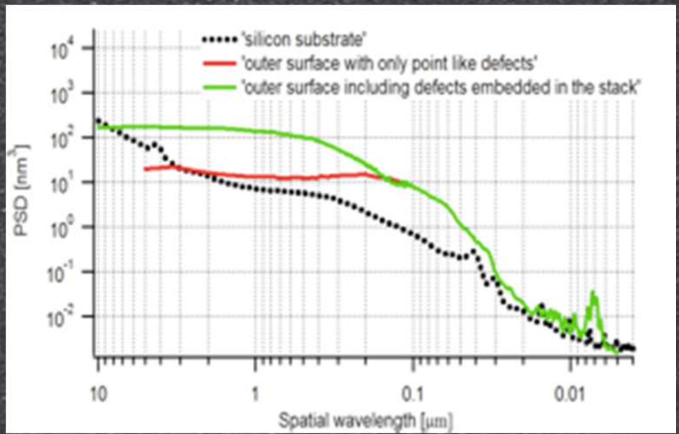
	Pt: *a* [Å], *b*, *c*	C: *a* [Å], *b*, *c*
Fit A	31.08, -0.90, 0.30	53.16, -0.96, 0.19
Fit B	31.00, -0.94, 0.23	53.00, -0.88, 0.21

Layer thickness difference ≤ 3Å for all layers except first C layer where Δ=15.3Å



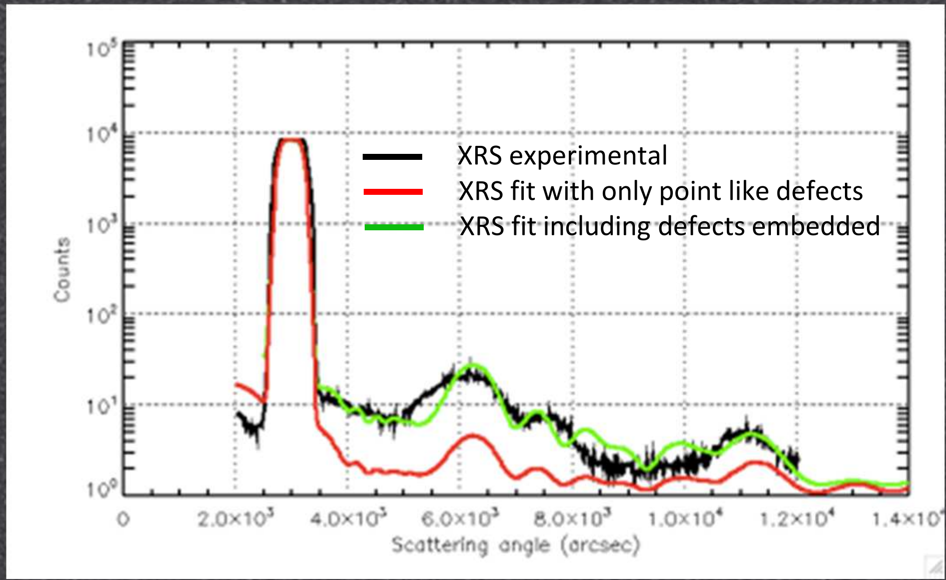
Defects contributing to XRS





- **XRS measurements 1**

θ_i=3000 arcsec

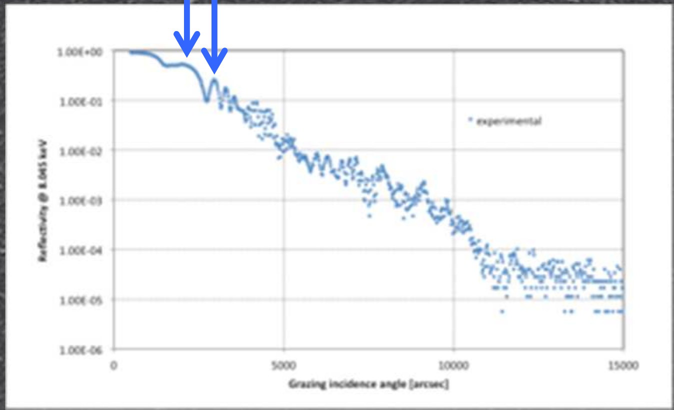


Different incidence angles

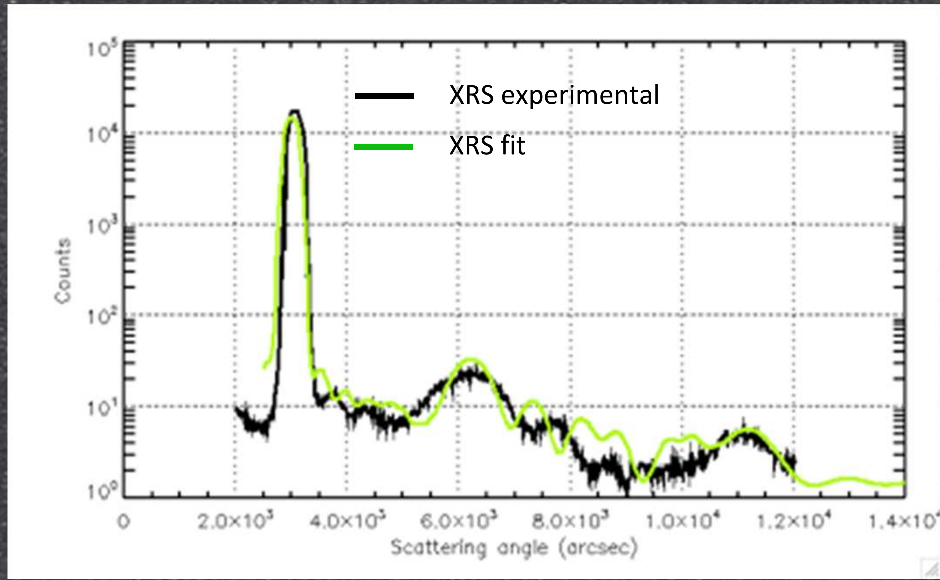
➢ XRS measurements 3

θ_i=2200 arcsec

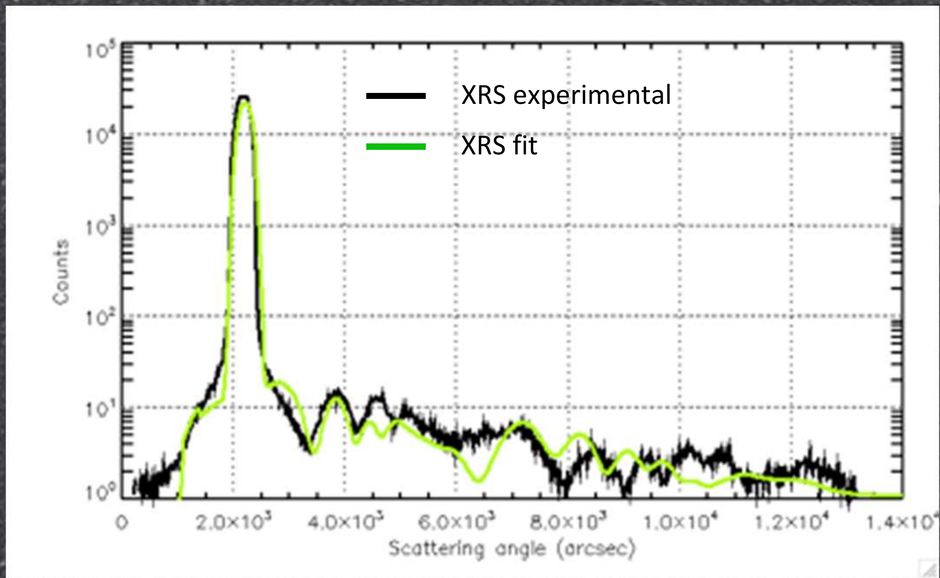
θ_i=3000 arcsec



θ_i=3000 arcsec



θ_i=2200 arcsec



Conclusions

- We have a protocol to predict XRS, and therefore imaging quality, from surface roughness for both periodic and graded multilayers
- The roughening model is validated by the fit of the XRS data
- XRS diagram is even more sensitive than XRR to the actual thickness trend in the stack
- We can separate surface defects by interfacial defects that contribute to XRS of the multilayer

Further development

- Correction for internal refraction with variable Γ
- Automatic search for best stack parameters