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Accurate computation of the X-ray diffraction efficiency of a multilayer coated grating based on a non-conformal deposition model

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Multilayer grating development for SOLEIL beamlines

Energy selection depends on the photon energy range

- Soft X-ray beamlines employ gratings

  Standard grating range typically below 1600 eV (small grazing angle)
- Hard X-ray beamline employ crystals (most often Si 111)

  Range typically over 2000 eV (small *d* spacing and large Bragg angles)

- **Multilayer coated grating are one way of bridging the gap**
  - DEIMOS : 2400 l/mm, Mo/B4C ML coated ; in the grating monochromator

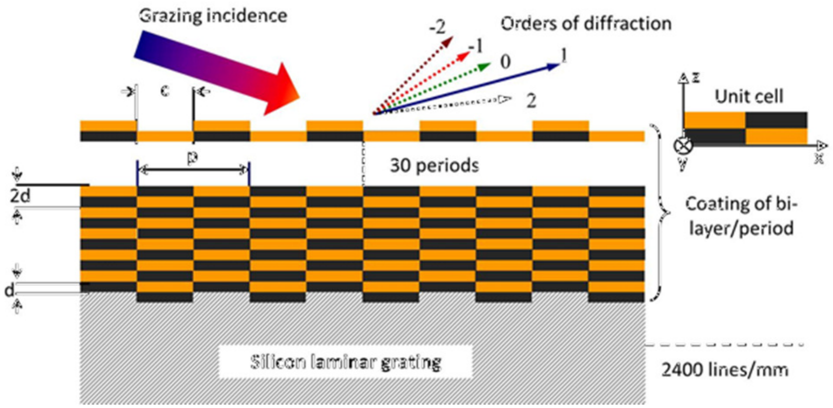
    range 1000 eV – 2500 eV
  - LUCIA : 3000 l/mm, etched in Mo/B4C ML ; in the Crystal monochromator

    range 900eV – 2500 eV
  - SIRIUS: 2400 l/mm, Cr/B4C ML coated ; in a dedicated monochromator

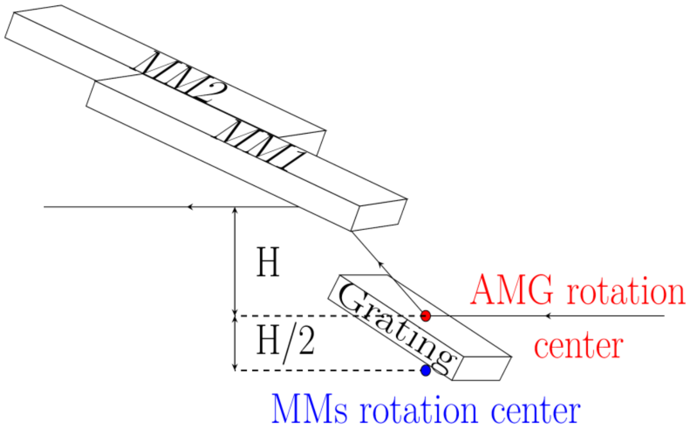
    range 1500 eV – 4500 eV
  - HERMES: *in project* similar to Deimos

The alternate multilayer grating structure

- ML of period *2d* is deposited on a **lamellar** grating of depth *d*
- **Creates a checkerboard like pattern – 2D periodic**
- Horz
  ⟶ *dispersion* ⟶ *resolving power*
- Vert periodicity (~30 periods)
  reflectivity ↗
  grazing angle ↗



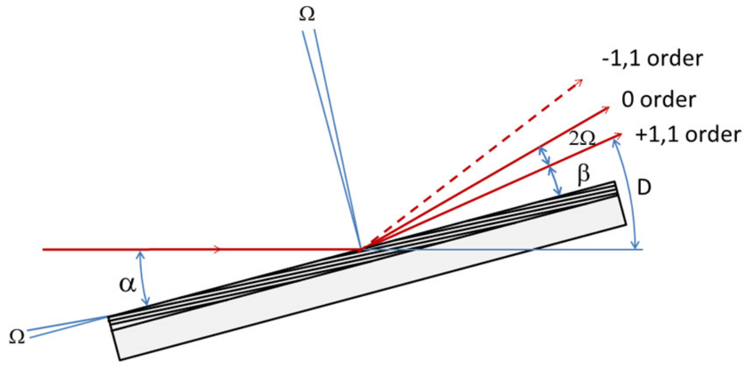
- **Dev. angle > critical angle**
  ⟹ a ML mirror of similar period needed to compensate the grating deviation



Diffraction properties of ML gratings

The grating should be considered as a 2D periodic structure

Diffraction properties dictated by



$$\omega = (\alpha - \beta)/2$$
$$D = (\alpha + \beta)$$

- The two periods
  - In-plane period *p*

$$cos(\beta) - cos(\alpha) = 2\, sin(D/2)\, sin(\omega) = k\, \lambda/p$$

  - Out of plane period *d*

$$sin(\beta) + sin(\alpha) = 2\, sin(D/2)\, cos(\omega) = m\, \lambda/d$$

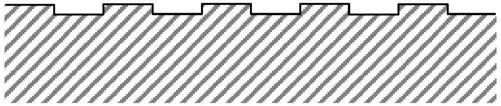
➢ Bragg condition *(neglecting refraction correction)*

$$tan(\omega) = \frac{k}{m}\frac{d}{p} \;\; ; \;\; m > 0$$

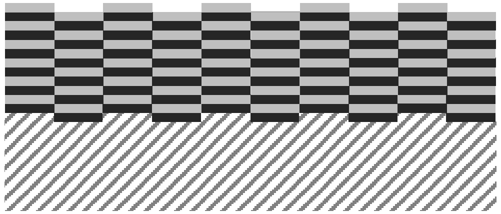
ω is the grating rotation angle from 0 order or Bragg angle

➢ scanning done by keeping ω *fixed* and adjusting the deviation angle "on blaze" scan

The fabrication & control sequence



- Grating masking and etching
- AFM characterization:
  Etched depth, duty cycle , roughness, uniformity
- Multilayer optimization (CARPEM code)
  from measured parameters



- Multilayer deposition (LCFIO)
- Multilayer characterization (LCFIO)
  Cu K_α reflectometry ⟹ *period, gamma, uniformity*
- At wavelength characterization
  diffraction properties

➢ Definition of the matched ML mirror, fabrication and control

The grating of Sirius beamline

Sirius is a hard X-ray beamline

2.2 – 10 keV with a DCM

- ➢1 – 4 keV with a dedicated MLgrating monochromator (PGM type)

The realized ML grating

(80 X30 mm^2, ruled 60x20 mm^2, 40mm thick)

- CrB$_4$C multilayer coating deposited on a 2400 l mm^{-1} laminar grating (Horiba Scientific)
- Groove depth 3.3 nm; ML period 6.3 nm; groove/period = 0.49
- ML : 35 periods; period is 6.3 nm, optimized with CARPEM code
  - target thicknesses: Cr 2.4 nm, B4C 3.9 nm

Characterization Measurements

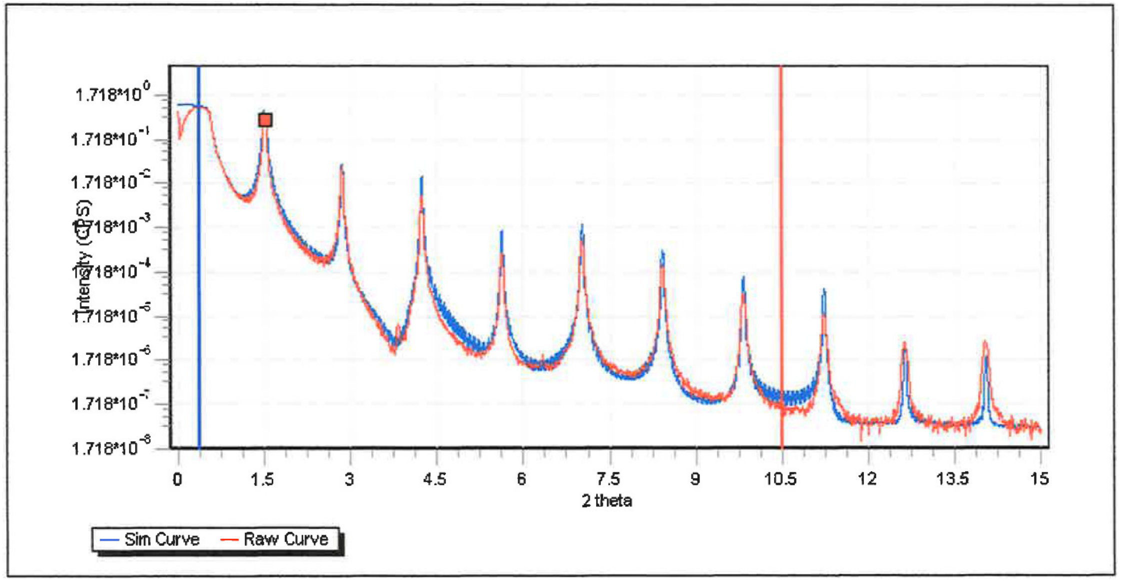
- ML material distribution profile, from Cu K$_\alpha$ angular reflectivity
- Diffraction efficiency

  Precise determination of rocking curves : peak efficiency and Bragg angle

  at Metrology beamline E < 1.7 keV and E > 3 keV

  at Sirius beamline E > 2.2 keV

Cu K_a ML reflectivity



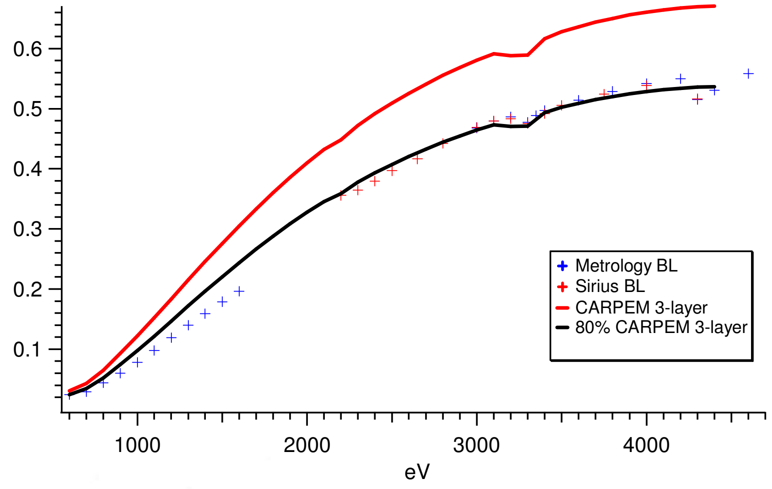
Measured at LCFIO with Bruker Discover D8

Fit with LEPTOS software

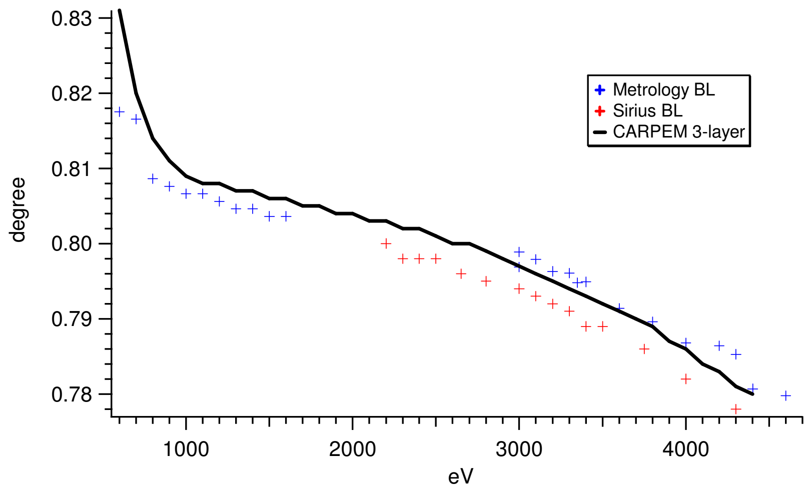
Material	Thickness (nm)	Roughness (nm)	Density	Repetition	Period
Si		0.20	2.33		
Cr	1.95	0.19	7.19	35	6.3 nm
$Cr_{11} (B_4C)_2$	0.93	0.20	5.92		
B_4C	3.42	0.28	2.0 (80%)		
B_2O_3	0.68	0.21	2.8	1	

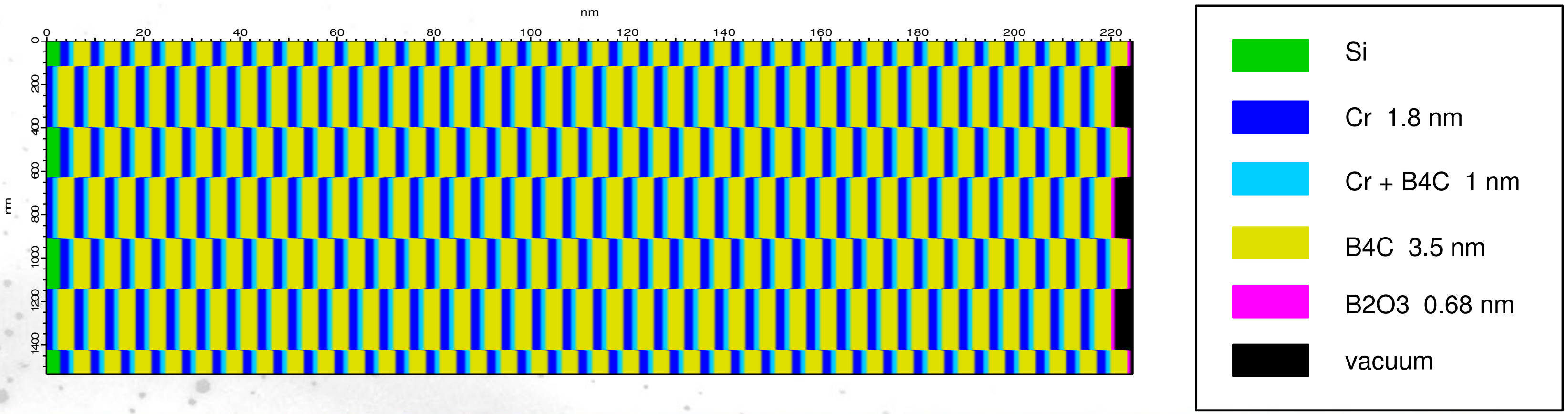
At wavelength measurements vs CARPEM model

Maximum efficiency



Bragg angle ω

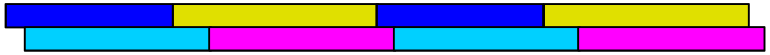




Computation model in the CARPEM code

CARPEM implements a Rigorous Coupled Wave Approximation (RCWA) algorithm coupled with a “R matrix” propagation method[1]

- The structure is divided in layers alternating 2 materials in the period with adjustable duty cycle and centering



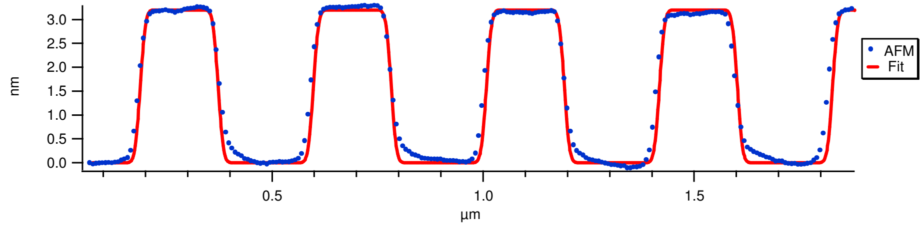
- In the structure the EM field is decomposed in plane wave components
- The components are propagated in z through each layer by Runge-Kutta integration of the Maxwell equations.
- R-K integration is restricted to a slice of limited thickness to avoid large intensity difference in up and down propagating waves. Typically 1 ML period (~6nm)
- The reflectivity matrix of the grating stack is constructed slice by slice, starting the substrate reflectivity from the reflectivity matrix of the underlying stack and the transfer matrix of the added slice

[1] M. Neviere and E. Popov. Light Propagation in Periodic Media : Differential , Theory and Design. Marcel Dekker, 2003

Surface Profile Observation

AFM measured profiles

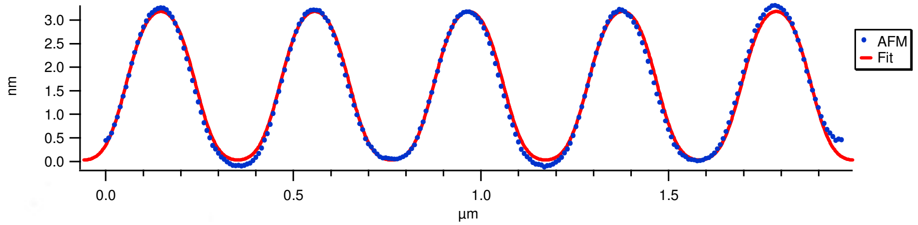
- Before ML deposition



- Fit parameters

  depth : 3.3 nm groove/period : 0.49 (damp parm 0.990)

- After ML deposition



- The grating profile is damped by the ML coating

- Higher frequencies are damped much faster than lower ones

Layer growth model

- **Main assumption :**

  The deposition rate is modulated in ratio of the local curvature

$$\frac{\partial z}{\partial t} = v_0 + c \frac{\partial^2 z}{\partial x^2}$$

z : position of the growing interface, v_0 average deposition rate

- Assuming the substrate profile is given by its Fourier expansion

$$z(x,t) = \sum_{n=-N}^{N} a_n(t) \exp\left[j \frac{2\pi n x}{p} \right]$$

- Then

$$\frac{\partial}{\partial t} z(x,t) = \sum_{n=-N}^{N} \frac{d\, a_n(t)}{dt} \exp\left[j \frac{2\pi n x}{p} \right] = v_0 - c \frac{4\pi^2}{p^2} \sum_{n=-N}^{N} n^2 a_n(t) \exp\left[j \frac{2\pi n x}{p} \right]$$

$$a_0(t) = a_0(0) + v_0 t \text{ , and if } \mathrm{n} \neq 0 \;\; \frac{da_n}{dt} = -k\, n^2 a_n \;\Rightarrow a_n = a_n(0) \exp\left[-kt\, n^2 \right]$$

- In our model we assume that c (k) does not depend on the deposited material then *t* can be read as the average deposited thickness and the composition at given (x,z) can be easily computed

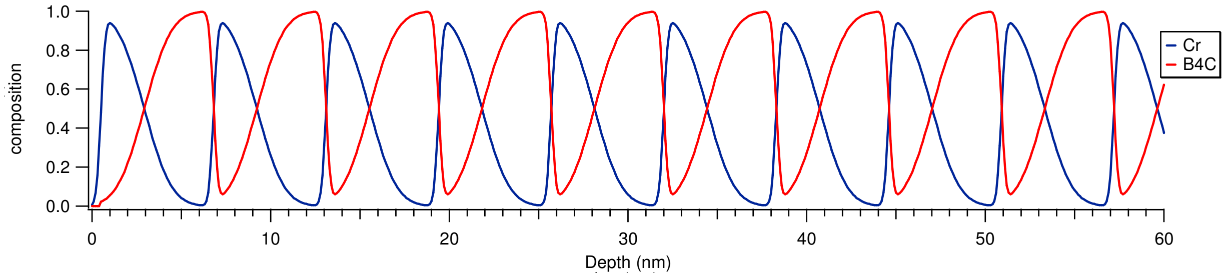
Interface model

- **Grating computations use a limited Fourrier expansion of the dielectric function**
  - High frequency material and density fluctuations contributes only by the average of the susceptibility (ε -1)
  - Rough but sharp interface between A and B with gaussian roughness σ_r the composition can be modeled by error function distributions
  - Inter-diffusion can be modeled by a similar composition profile σ_d then represents the inter-diffusion layer thickness ($erf(0.5) \approx 0.5$)
- In practice, σ_r and σ_d are combined

- **Limit of the model**
  - The low frequencies of the roughness are not accounted for all ML periods are strictly equal -> errors in evaluating high reflection orders

The ML model

Cr and B_4C distribution profiles

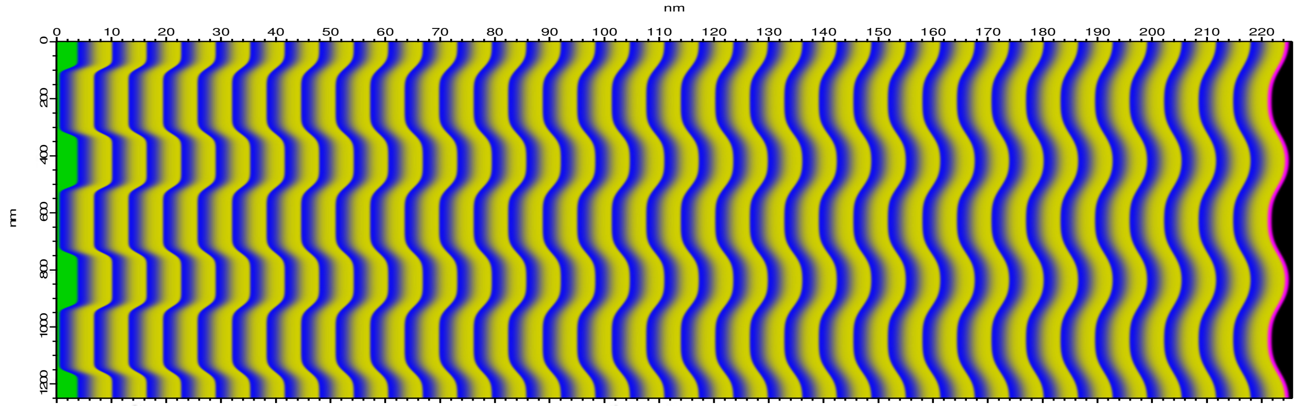


Interfaces

B4C over Cr: $\sigma = 1.2$ nm

other interfaces: $\sigma = 0.2$ nm

Material distribution map



🟩 Si

🟦 Cr 2.4 nm

🟨 B4C 3.9 nm

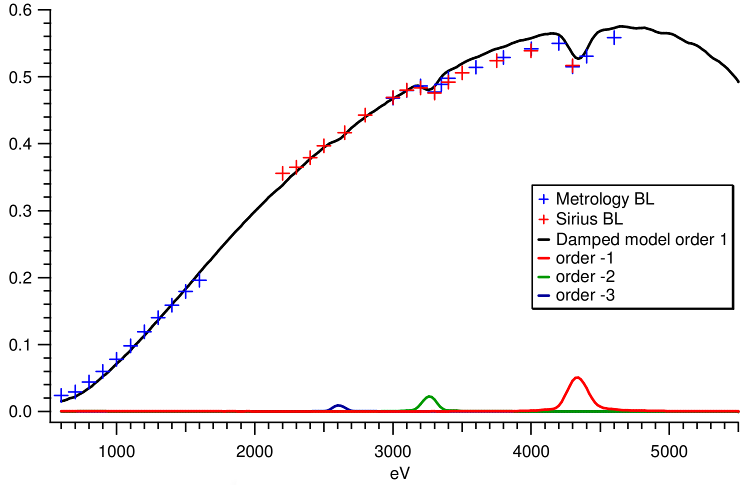
🟪 B2O3 0.68 nm

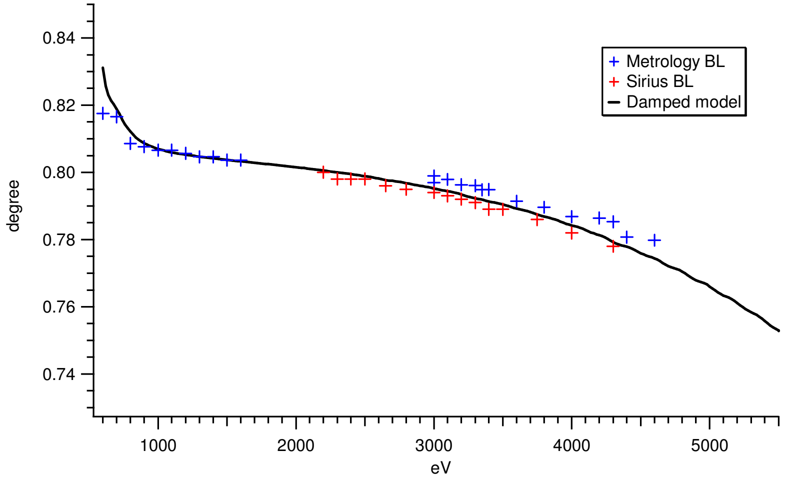
⬛ vacuum

The dielectric constant is tabulated with a step of 0.1 nm

CARPEM has been modified to work with interpolated tabulated data

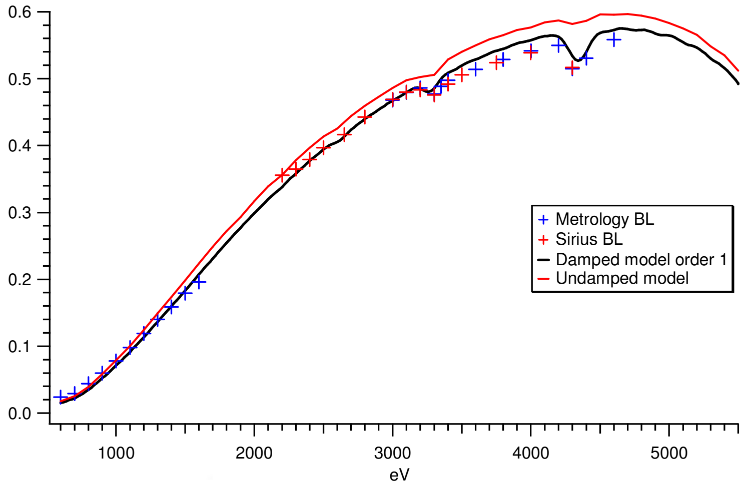
Result of the model

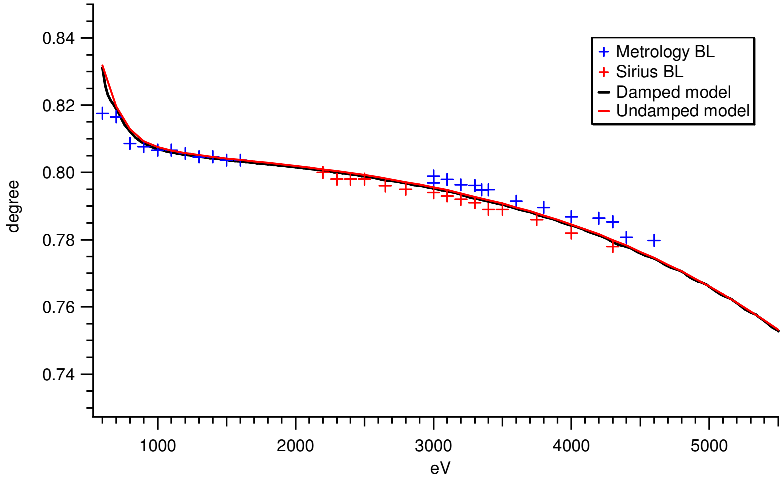


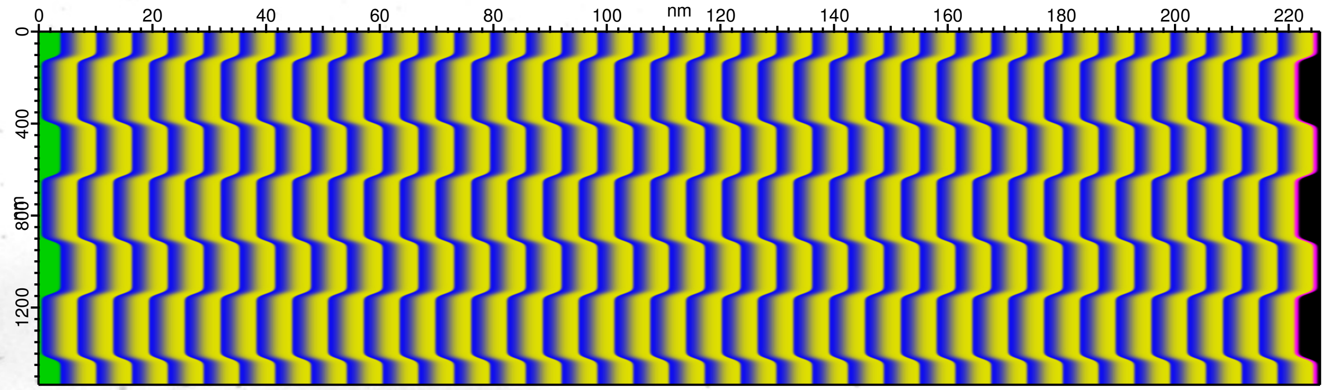


- Bragg angle (maximum of the rocking curve)
  is sensitive to refraction thus average index of refraction
- Efficiency is mostly affected by modulation change with depth
  note that glitches corresponding to angle coincidence with other orders are well predicted by the model

Undamped model







Conclusions

- A non-conformal model of thin film growth has been used to model the Sirius ML grating
- It fits AFM grating surface measurement before and after ML coating
- It explains the general trend of grating efficiency in order 1
- It explains the glitches due to coincidence with authorized Bragg reflection in different grating orders
- It fits the measured dependence of Bragg angle vs energy

- Keeping sharp lamellar layer profile has an significant impact on efficiency
- Non conformal growth must be taken into account in the design studies