ESRF | The European Synchrotron

X-RAY AT-WAVELENGTH METROLOGY OF MULTI-LAYERED SURFACES

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ISDD Office
Instrumentation beamline BM05

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ESRF UPGRADE

Phase I – 180 M€ (2009-2015)

- 19 new endstations and beamlines
- New 8 000m^{2}experimental hall
- Improvement of overall infrastructure and Accelerator systems

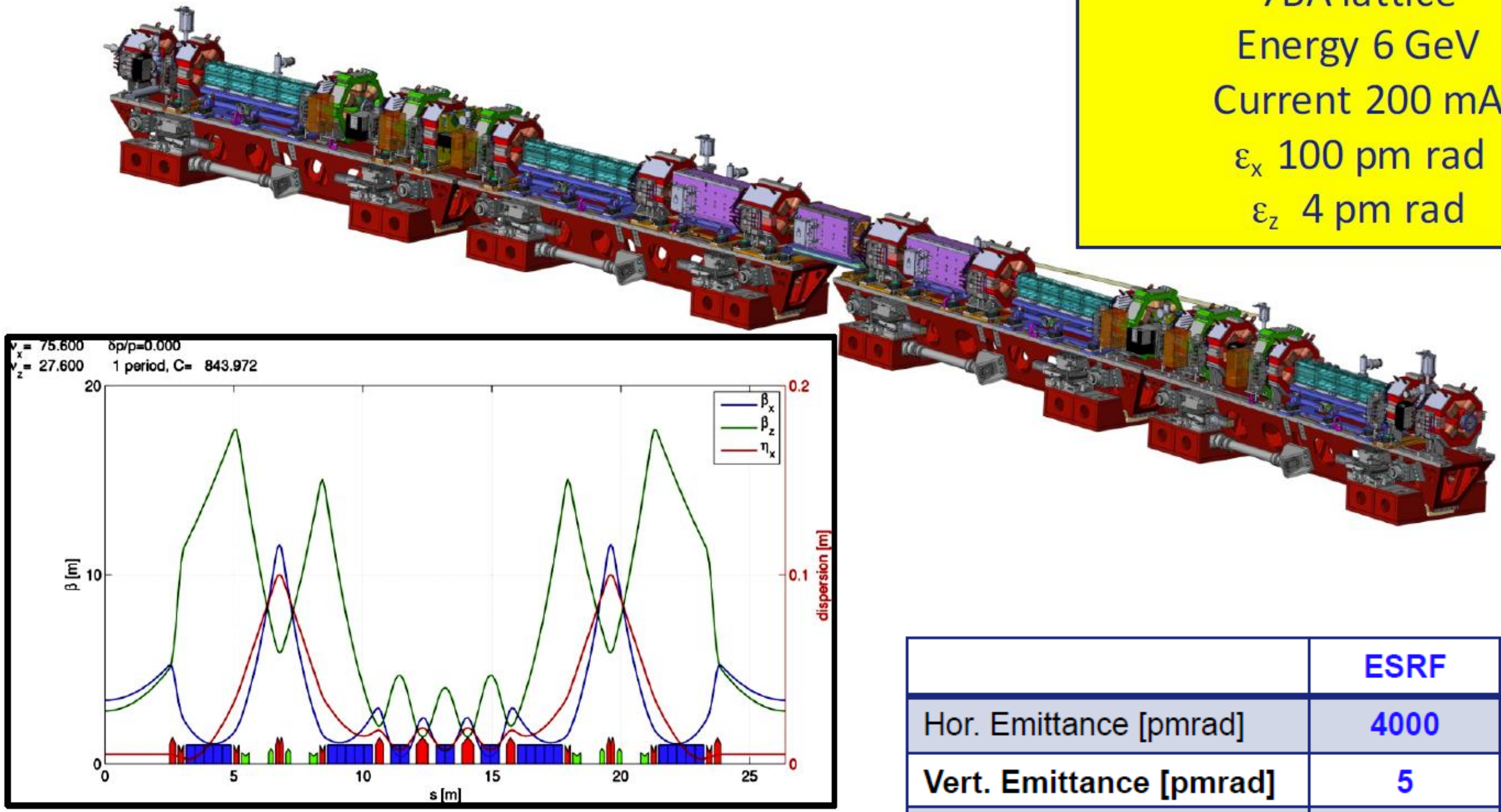
Phase II – 150 M€ (2015-2022)

- a new high energy low-emittance source
- a new portfolio of unique instruments
- an innovative scientific instrumentation programme

THE ESRF-EBS LATTICE

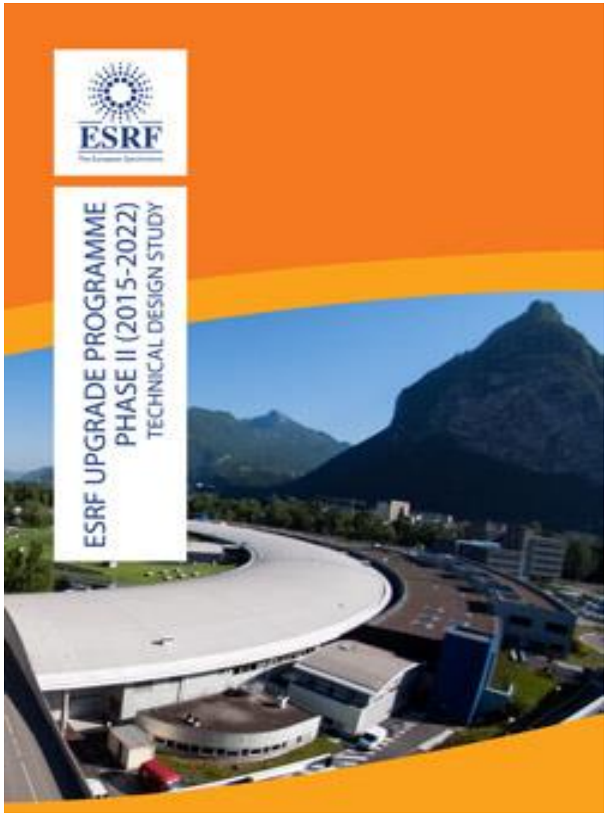
Key Paramaters

7BA lattice
Energy 6 GeV
Current 200 mA
ε_x 100 pm rad
ε_z 4 pm rad



	ESRF	upgrade
Hor. Emittance [pmrad]	4000	135
Vert. Emittance [pmrad]	**5**	**5**
Energy spread [%]	**0.1**	**0.09**
β_x[m]/β_z [m]	**37/3**	**6.9/2.6**

X-RAY OPTICS



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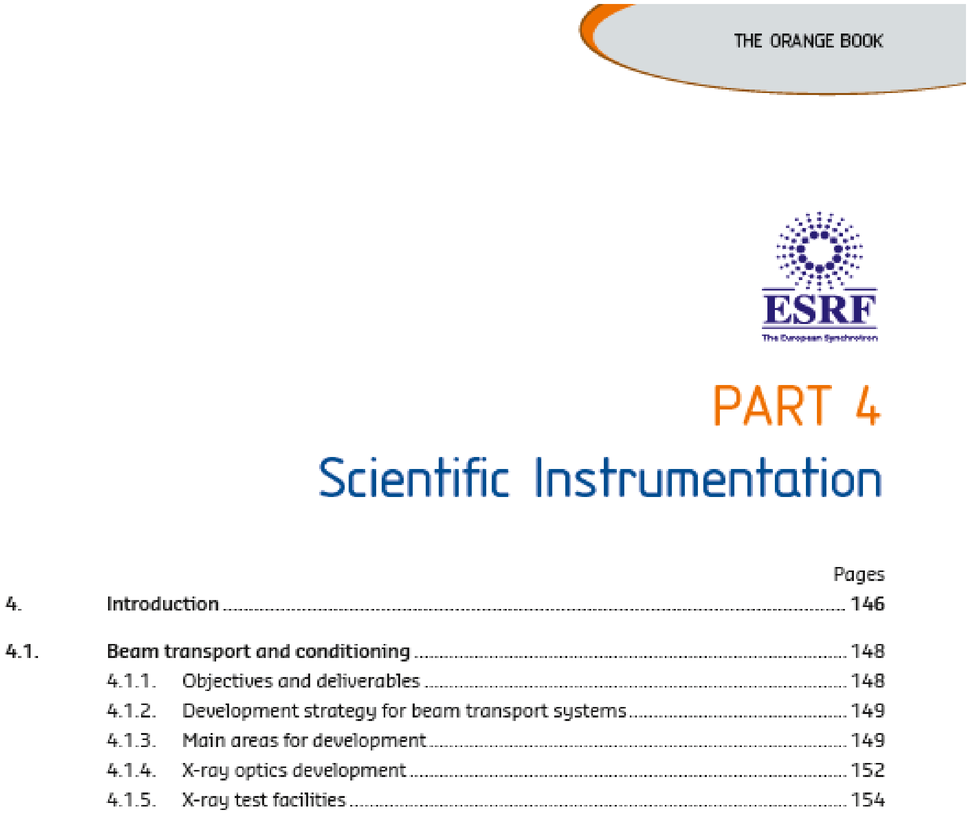
ESRF UPGRADE PROGRAMME
PHASE II (2015-2022)
TECHNICAL DESIGN STUDY



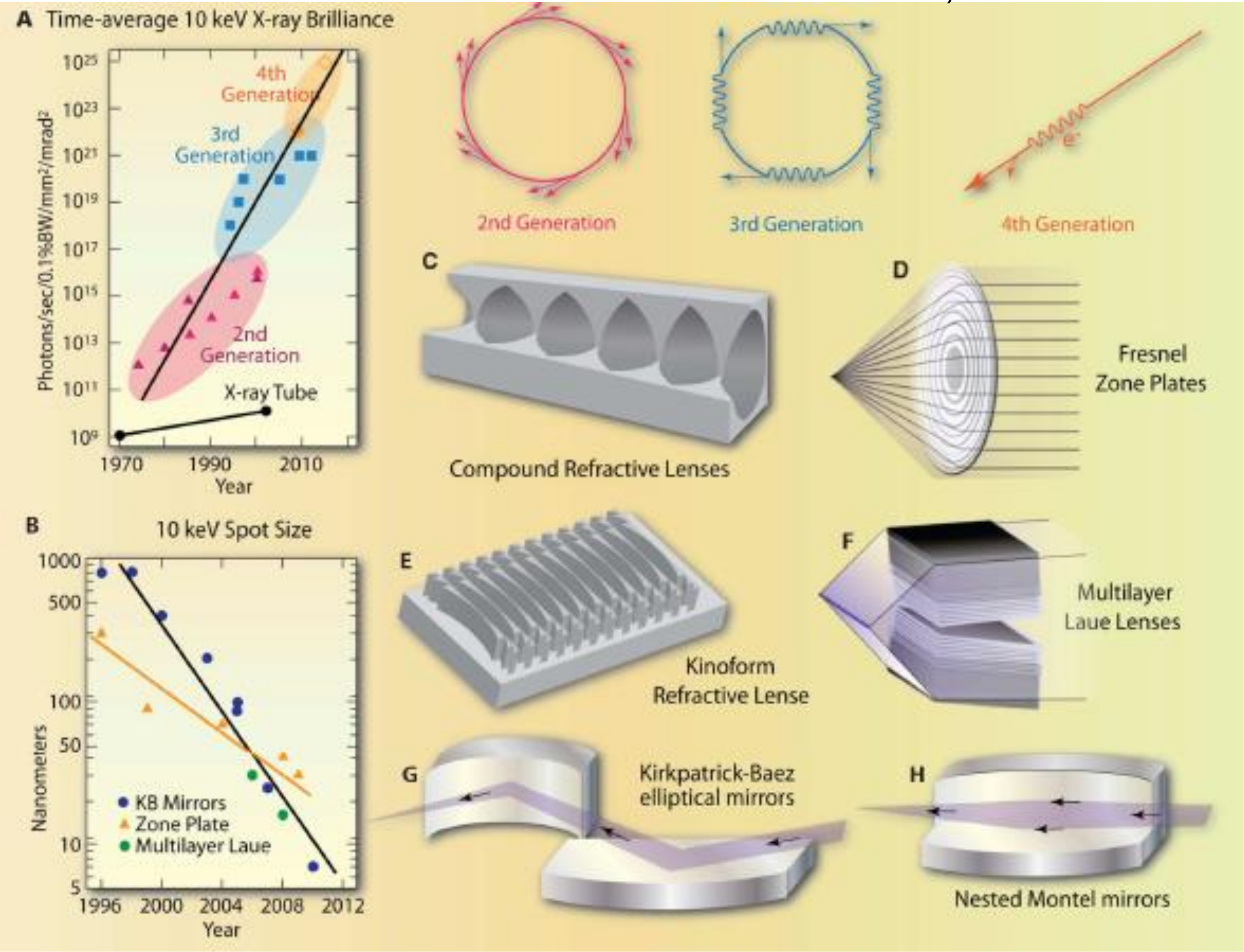
THE ORANGE BOOK

ESRF
The European Synchrotron

PART 4
Scientific Instrumentation

X-RAY MULTILAYER OPTICS

G. E. Ice *et. al.*, Science 2011



Multilayer optics present wide and tunable spectral bandwidths

❖ **Imaging applications** (radiography, tomography, tomosynthesis…)

- High flux (x100 compared to crystal based monochromators)
- Large grazing incidence angle useful for nanofocusing

At the ESRF: ID16NI, ID21, ID19…

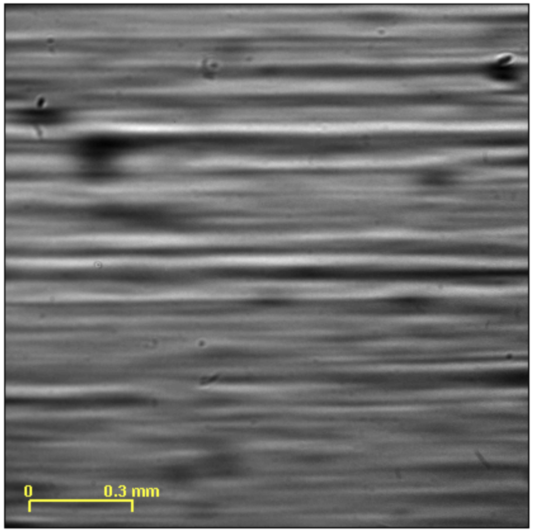
❖ **Spectroscopy applications** with focused beam (µEXAFS, µXANES, µXAS,…):

- Large spectral bandwidth
- Achromatic systems

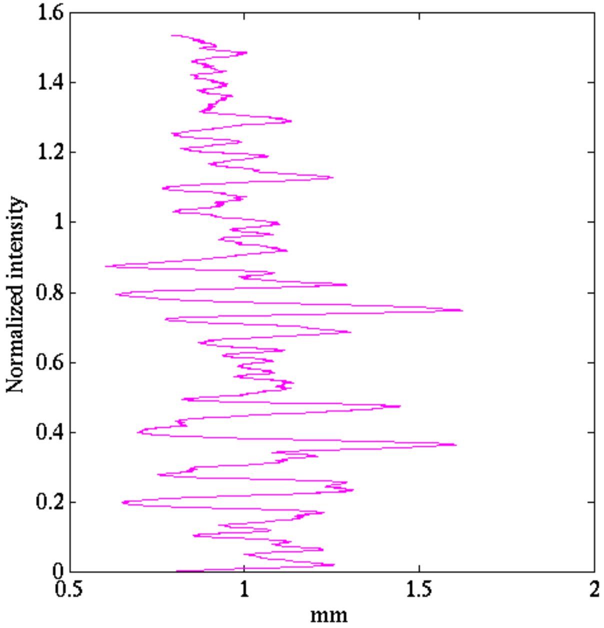
At the ESRF: ID24, BM23, ID16NA…

CURRENT CONCERN

X-ray beam imaged after double reflection on multilayer mirror monochromator



E = 17 keV
Pixel size = 0.75 µm
Distance from monochromator = 15 m



Up to 50 % intensity modulation with 17 x 5 µm transverse coherence at monochromator position.

More detailed characterization in:

A. Rack A *et al.* “Comparative study of multilayers used in monochromators for synchrotron-based coherent hard X-ray imaging”. J Synchrotron Rad. 2010;17(4):496-510.

Such modulation fringes are a huge drawback for:

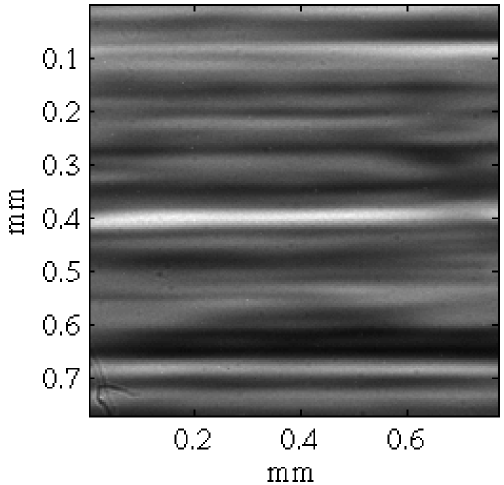
- **Imaging: it does not allow one to take full advantage of the detector dynamic**
- **Spectrometry applications: interference effects generate artefacts in data analysis**

EFFECT OF COHERENCE

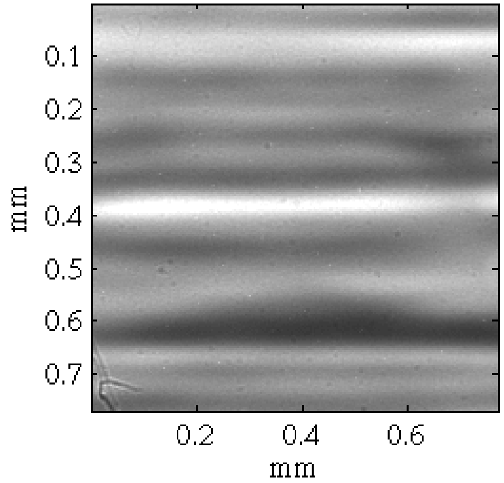
Fringe contrast during storage ring refill (16 bunch mode, 90 mA nominal)

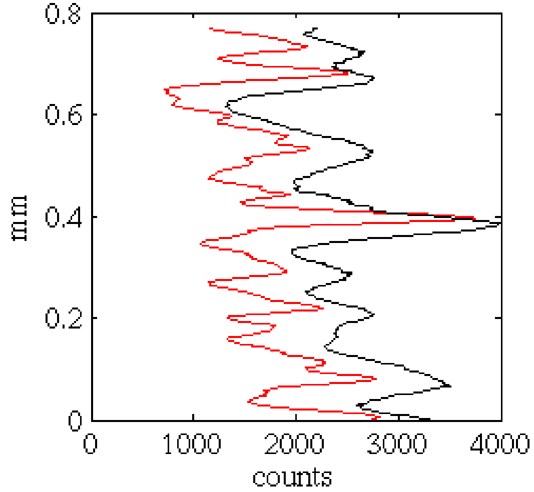
Total refilling perturbation time: ~3 min

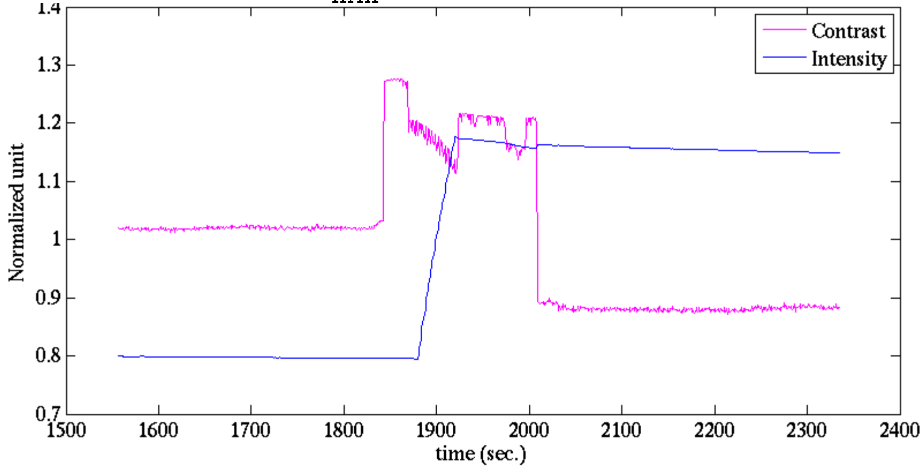
Before refill



After refill





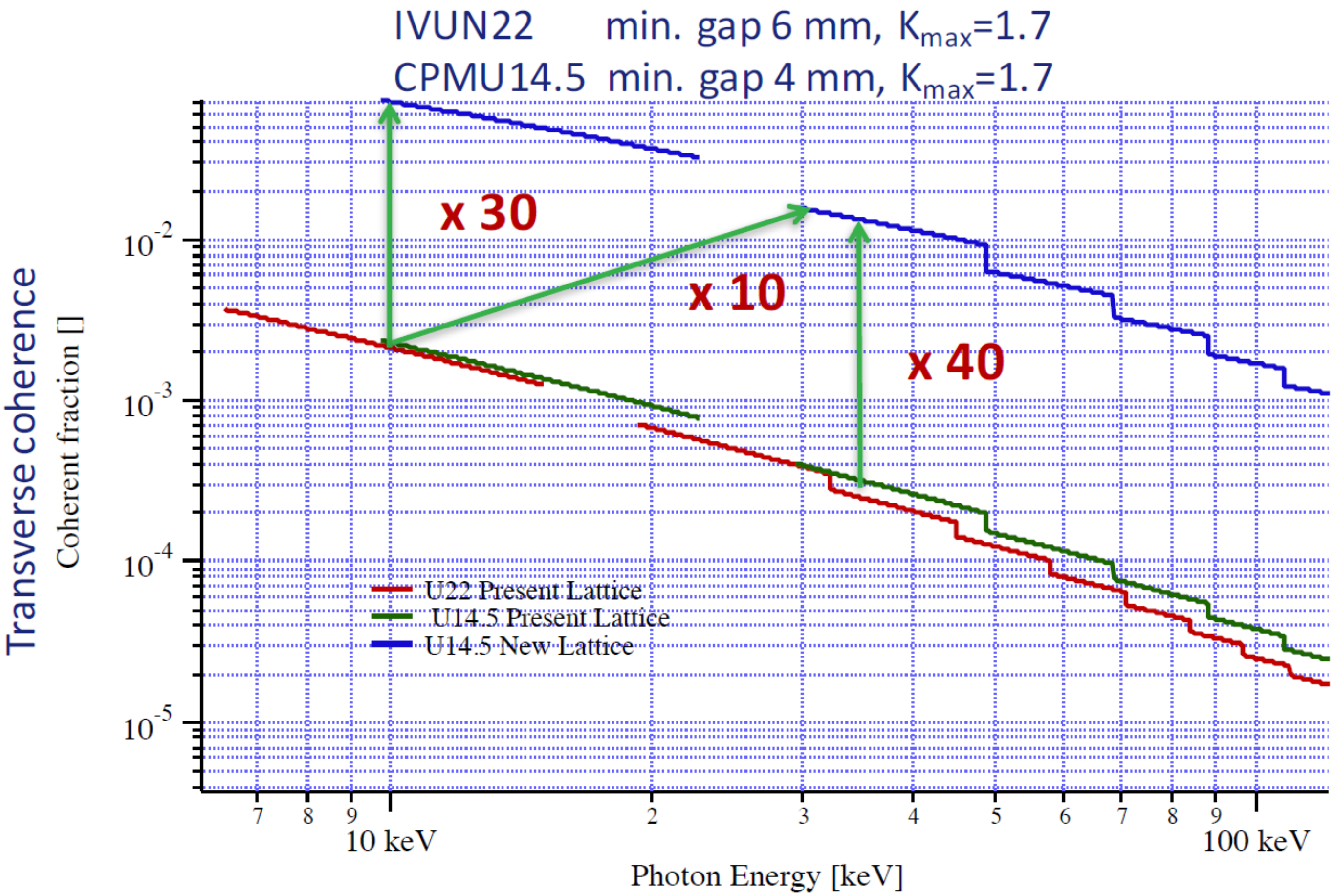


Beamline Double Multilayer Mirror Monochromator:
2 mirrors 300 mm long
Flat substrate from GO
Ru/B4C
40 bilayers, 4 nm period with gradient to match divergence

Clear difference in the visible spatial frequencies

COHERENT FRACTION

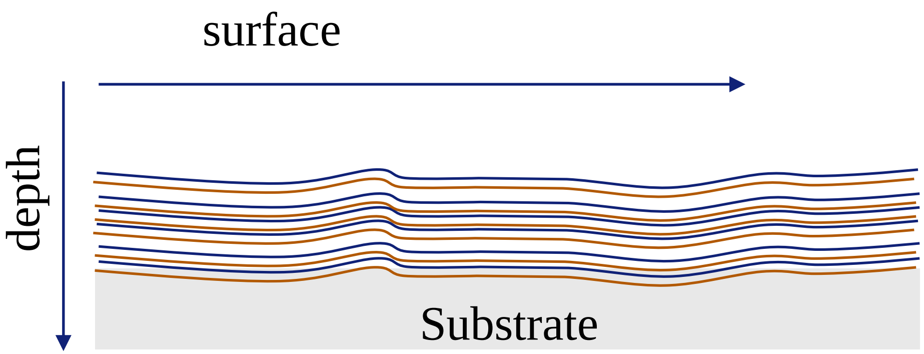
ESRF-EBS: increase of the horizontal coherence by a factor up to 40

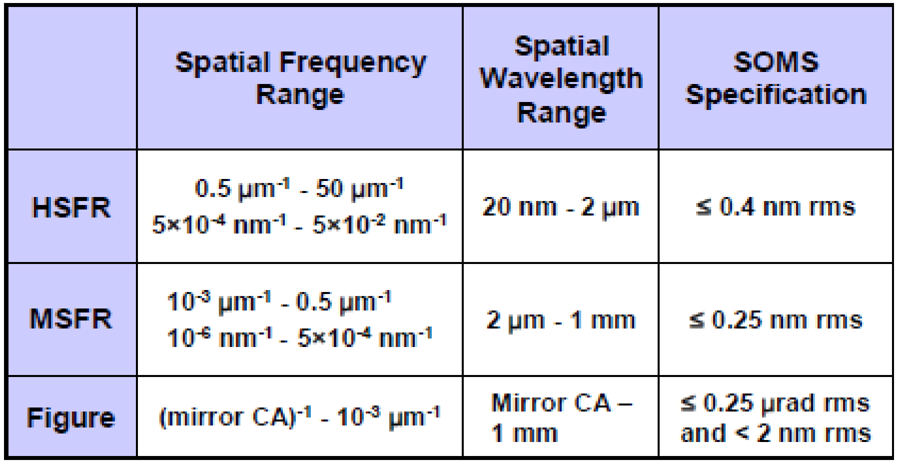


Interference fringes will likely be more pronounced, especially in the horizontal direction.

MULTILAYER MIRROR REQUIREMENTS

Multilayer topography





	Spatial Frequency Range	Spatial Wavelength Range	SOMS Specification
HSFR	0.5 µm^{-1} - 50 µm^{-1} 5×10^{-4} nm^{-1} - 5×10^{-2} nm^{-1}	20 nm - 2 µm	≤ 0.4 nm rms
MSFR	10^{-3} µm^{-1} - 0.5 µm^{-1} 10^{-6} nm^{-1} - 5×10^{-4} nm^{-1}	2 µm - 1 mm	≤ 0.25 nm rms
Figure	(mirror CA)$^{-1}$ - 10^{-3} µm^{-1}	Mirror CA – 1 mm	≤ 0.25 µrad rms and < 2 nm rms

Soufli et al., SPIE 2008

X-ray metrology:

- **Depth:**
  - **- X-ray reflectivity**
- **Surface and interfaces:**
  - **- X-ray diffuse scattering**
  - **- Wavefront characterization**

ASSUMPTIONS

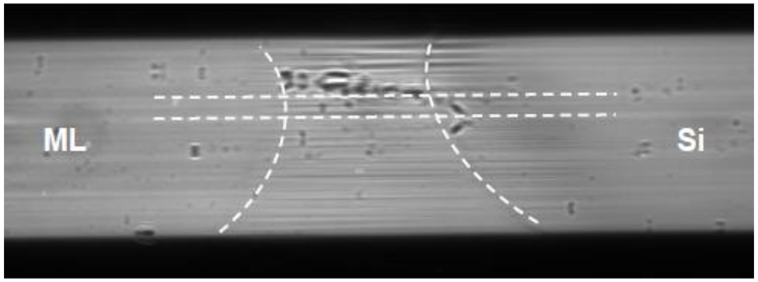
- **Intensity modulations due to transverse and longitudinal coherence of the synchrotron source: interference process**

Coherence lengths:

$$L_L = \frac{\lambda^2}{2 \cdot \Delta\lambda}, \; L_T = \frac{\lambda \cdot p}{2 \cdot S}$$

- **Transmission of the substrate imperfections printed through the ML stack.**

Morawe Ch., Barrett R, Friedrich K, Klünder R, Vivo A. Coherence preservation of synchrotron beams by multilayers. J Phys Conf Ser. 2013;425(5):052027.



Substrate with one side coated and the other not

Total reflection at E = 15 keV

ONLINE METROLOGY APPROACHES

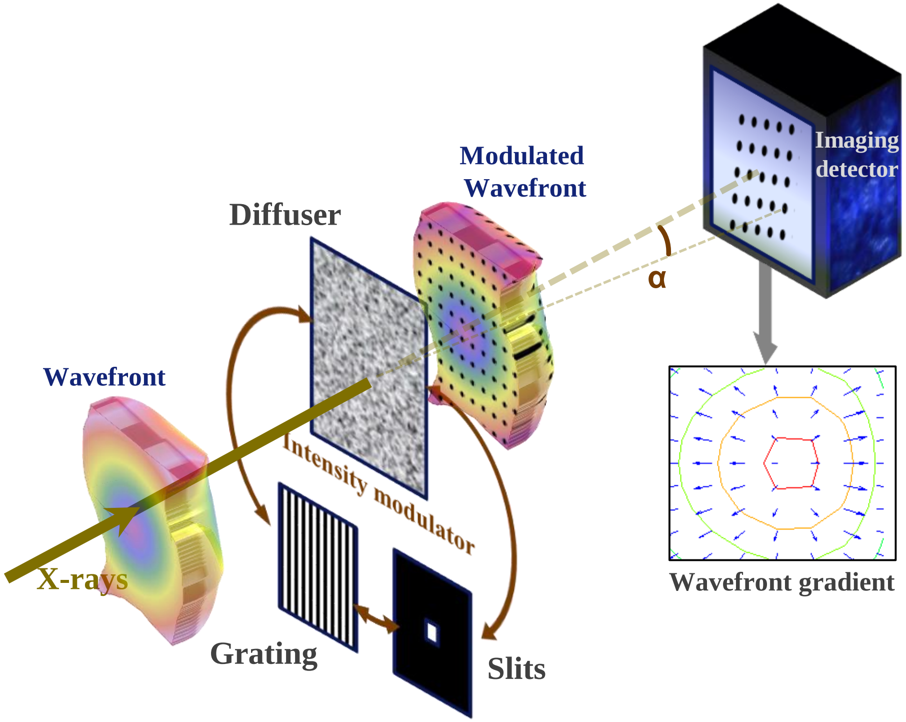
❖ Wavefront propagation based methods:

- Transfer of intensity equation
- Iterative calculation of Fresnel propagation
- Ptychography
- …

❖ Wavefront modulation based methods:

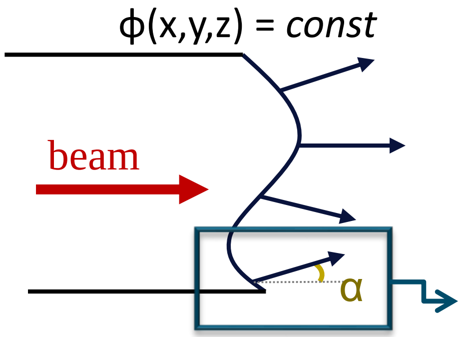
- Pencil beam method
- Grating based methods (mainly interferometry)
- Hartmann like sensors
- Coded apertures based methods
- Speckle based methods
- …

WAVEFRONT SENSING USING MODULATION



Recovery of the deflection angle through:

- Centroid method
- Fourier processing
- Normalized cross-correlation



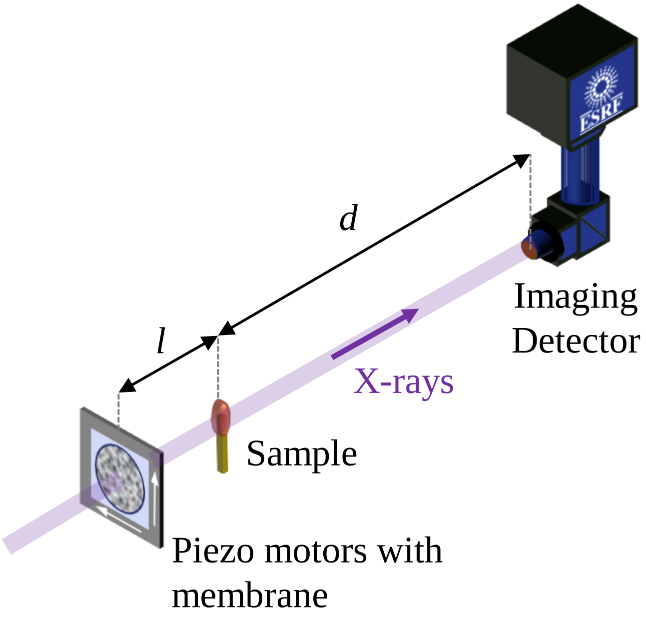
What we sense using (near field) deflection based technique

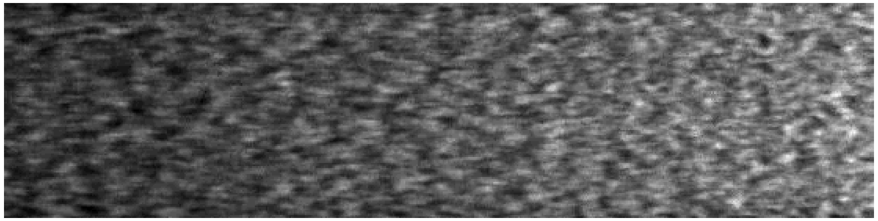
Sensitive to ∇W or $\nabla^2 W$

$$\boxed{\alpha = -\frac{\lambda}{2\pi}\frac{\partial \varphi}{\partial x} = -\frac{\partial W}{\partial x}}$$

SPECKLE BASED APPROACH

Access to the tangential and sagittal slopes with 2D scans of the membrane

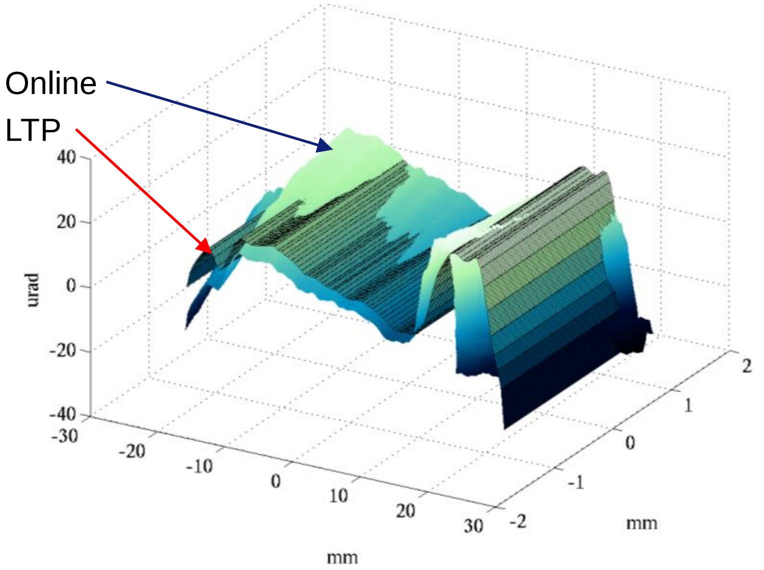




Berujon *et al.* 2014

ESRF BM05 @ 9 keV

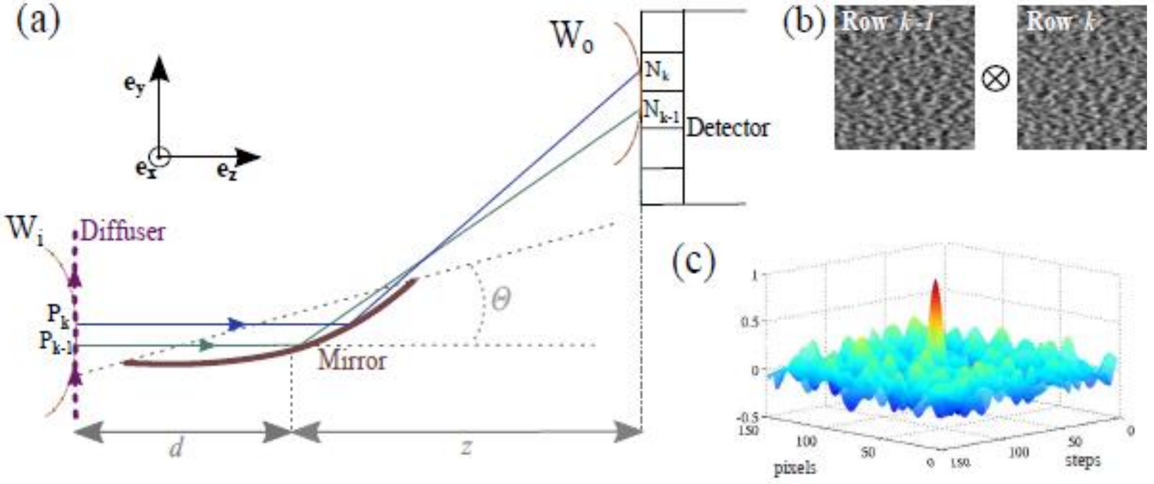
p = 61 m, q = 59 mm , Θ = 6.31 mrad



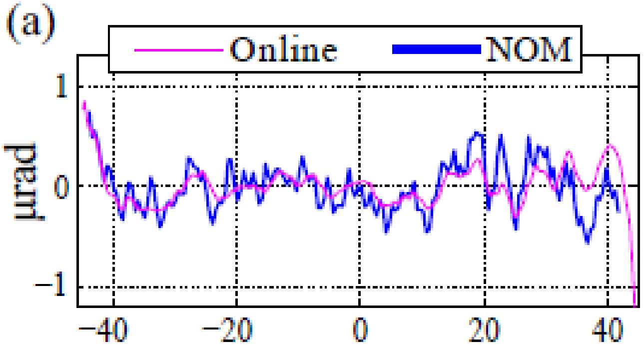
2D tangential slope

Measuring reflective optics: mapping in 2D

SPECKLE BASED METROLOGY

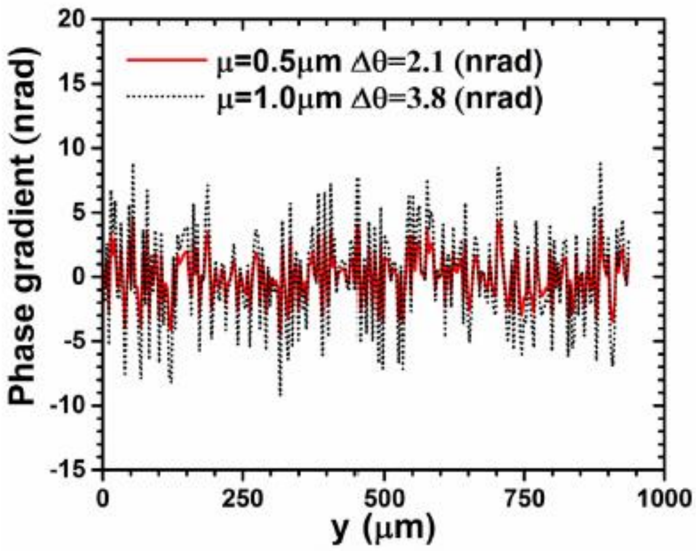


Berujon et al., Optics Express (2014)



Angular accuracy can be pushed down to a few nanoradians
Resolution of ~1 µm at the detector plane

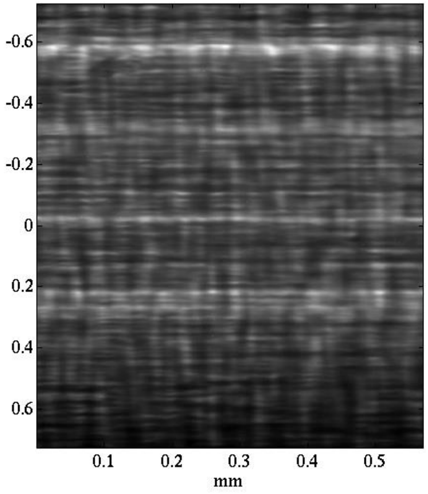
Wavefront sensitivity better than 1 pm

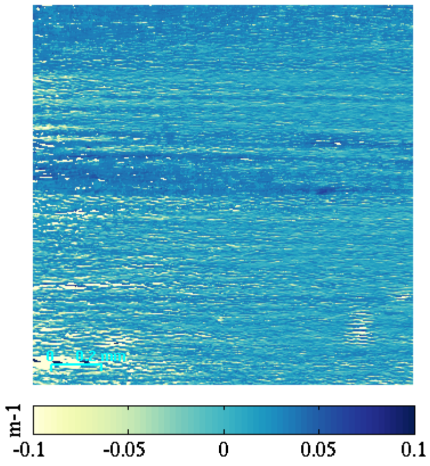


Wang H, Kashyap Y, Sawhney K. Speckle based X-ray wavefront sensing with nanoradian angular sensitivity. Opt Express. (2015)

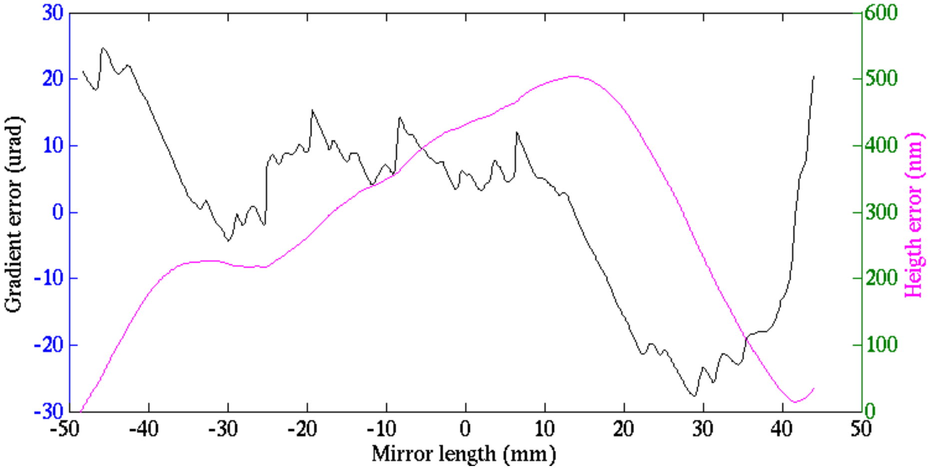
MULTILAYER MIRROR ONLINE METROLOGY

M2: $[W/B_4C]_{20}$ Λ=4nm on flat substrate

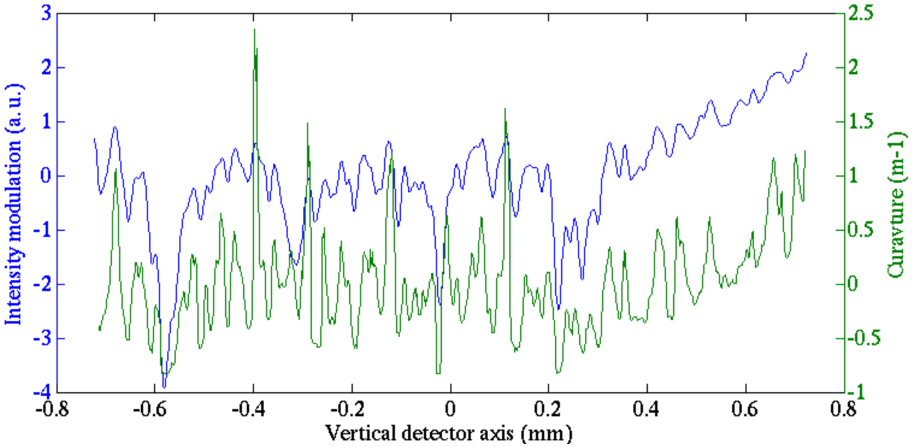



Vertical curvature

Height profile and slope error



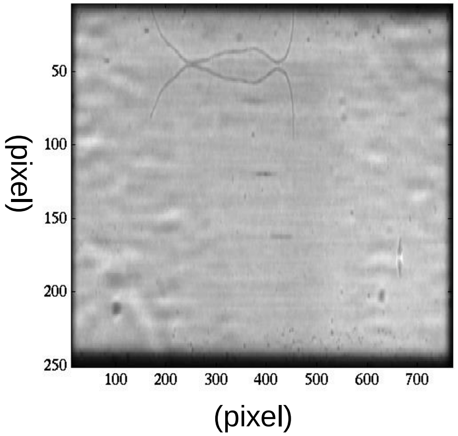
Mirror curvature and modulation fringes



Excellent correlation between the fringes location and the mirror curvature

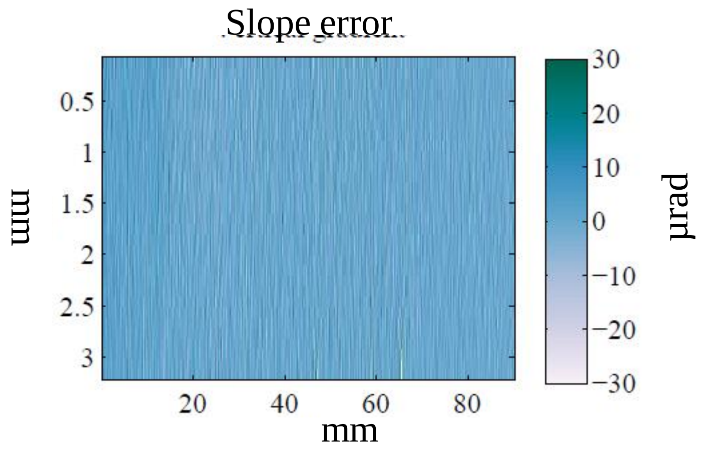
RESULTS ON ML MIRRORS

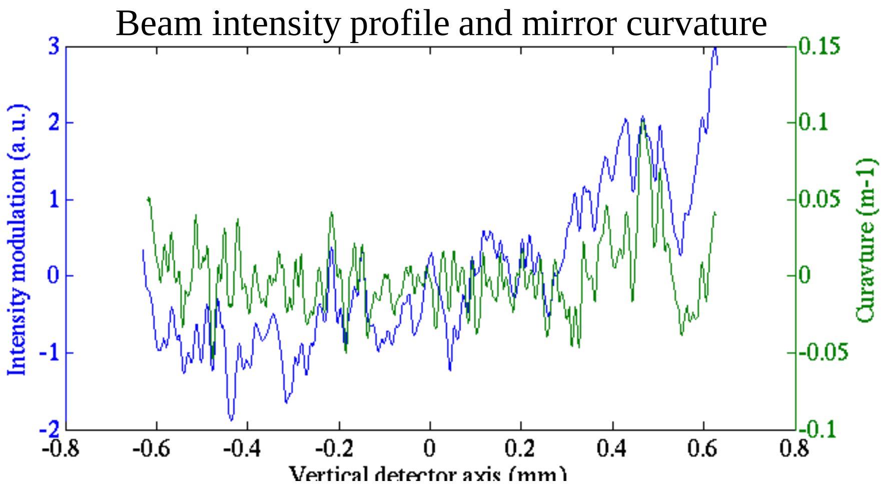
M1: $[W/B_4C]_{60}$ Λ=4nm, 100 mm long substrate

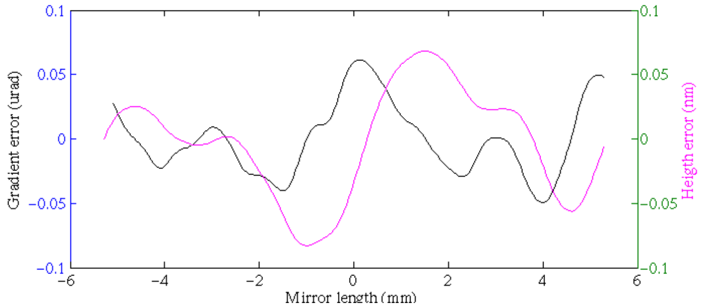


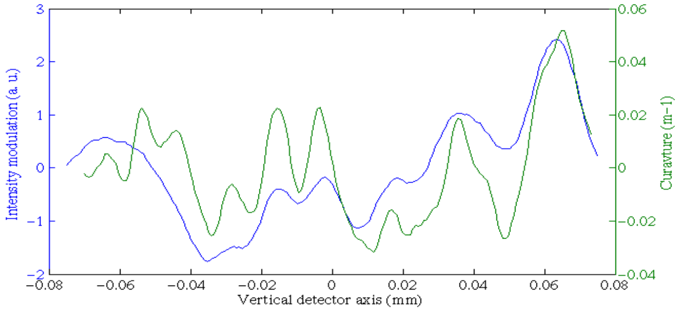
E = 12.4 keV

Very good substrate quality as measured by Fizeau interferometry









Nearly perfect correlation between mirror curvature and modulation fringes

Deviation equivalent to the thickness of a hair on the road from here to Amsterdam

EXPERIMENTAL VALIDATION OF ASSUMPTIONS

Nicolas J, García G “Modulation of intensity in defocused beams” (2013)

Far from focus, the modulated intensities can be approximated by:

$$F_z(x) = \sum_{i=1}^{N} \frac{F_i\left(u(x)\right)}{\left|2z\left(\frac{1}{R_z} + \frac{d\varepsilon}{du}\left(u(x)\right)\right)\right|}.$$

In other words, a smooth background with some modulation proportional to the curvature of the mirror

- ❖ **It is not so much the slope error that matters. For instance, bigger slope errors can generate less interference fringes than a flatter mirror.**
- ❖ **What really matters is the local curvature. One needs to care about the spectral distribution of the slope errors especially in the mm^{-1} spatial frequency range.**

FUTURE

- **Manufacturing of substrate surfaces with better flatness seems difficult. Little improvements observed in the last 10 years.**

- **A possible direction could be differential deposition with high frequencies control: use of a narrow mask to compensate for substrate defects**

- **Control the properties of X-ray propagation in the ML stack by simulation of the X-ray propagation in the multilayer system using adapted Takagi-Taupin equations.**

Cf. P. Piault's Poster

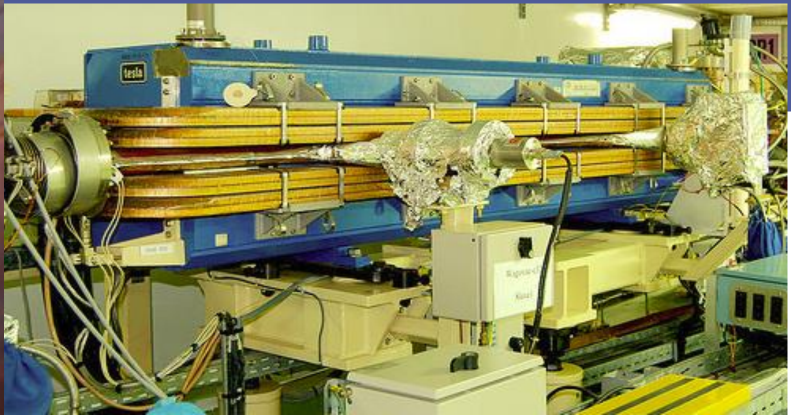
- **Further investigations of the effect of roughness interfaces on the beam coherence properties**

FINAL WORDS

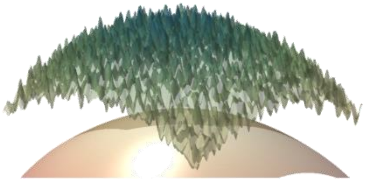
- Substrate shape is still problematic and is likely to remain as such since the manufacturing quality has reached a limit.

- A substrate manufacturing process generating less curvature in the mm^{-1} frequencies range should be favored.

- Finally deposition and material selection strategies must be implemented to suppress the unwanted interference fringes at coherent sources.



Our current source and soon a museum piece



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- Ian Pape, Andy Malandain, Simon Alcock
- Diamond B16 former colleagues

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Thank you