

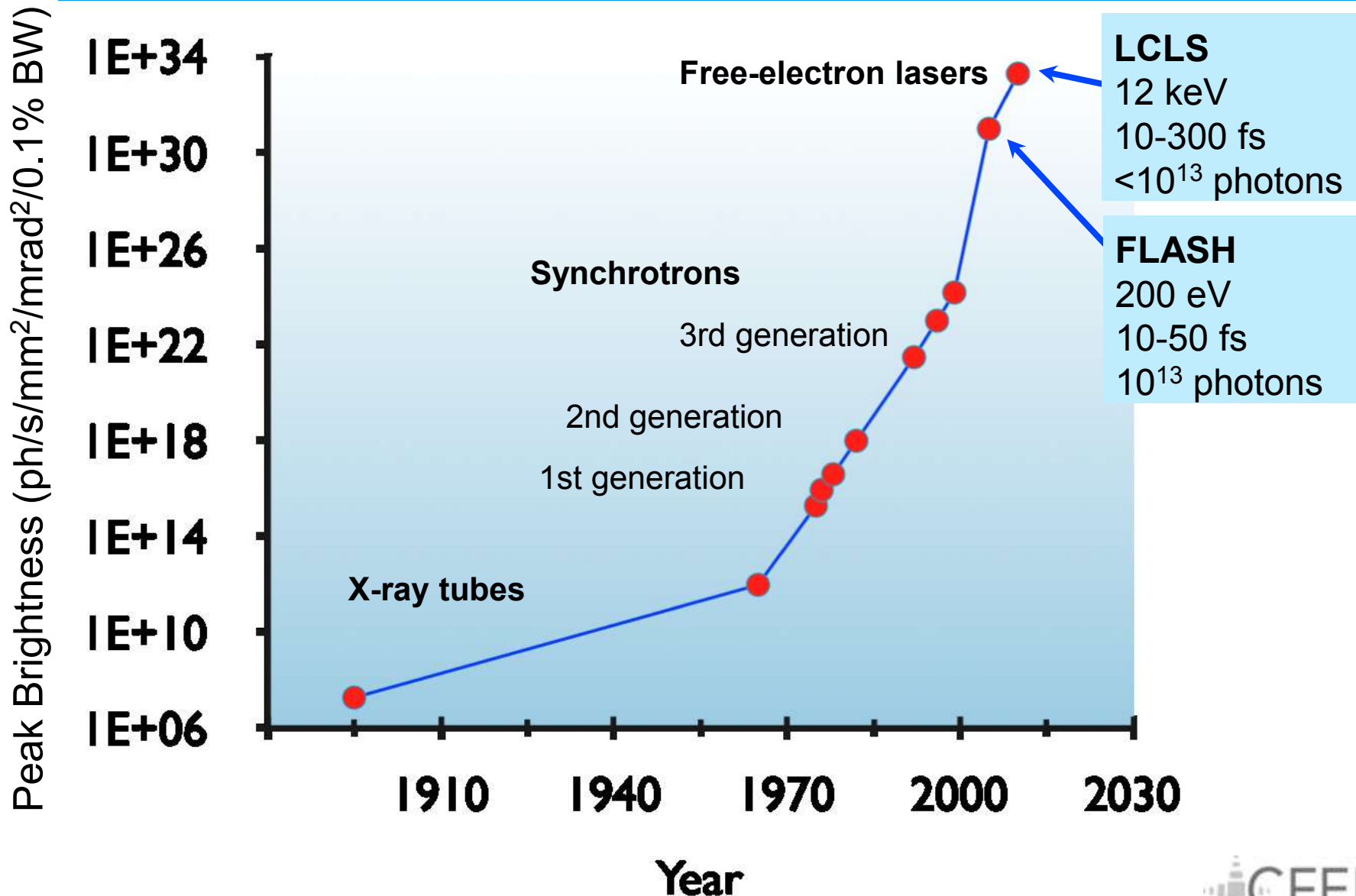
# Development of multilayer-based x-ray optics for FEL and synchrotron applications

**Saša Bajt**

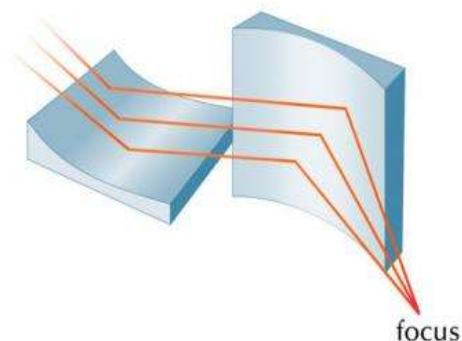
**Photon Sciences, DESY, Hamburg**

*Instrumentation seminar, March 2, 2012*

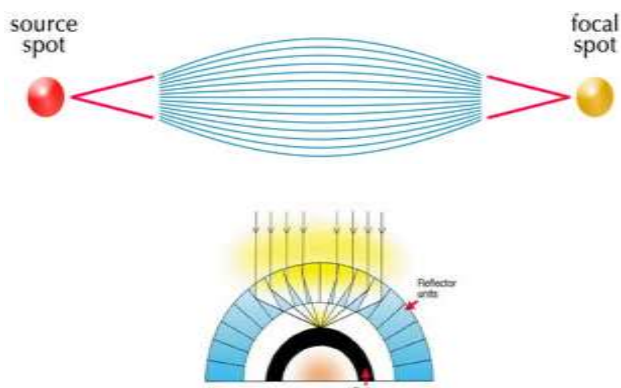
# X-ray sources have developed at a staggering pace since their discovery in 1895



# Different types of X-ray optics



Kirkpatrick-Baez mirrors



Polycapillary optics



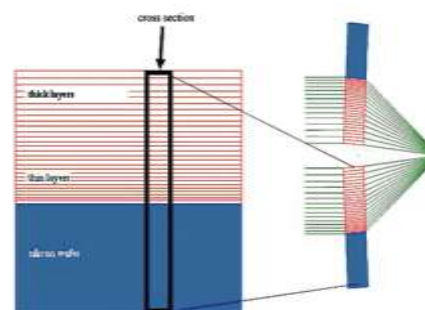
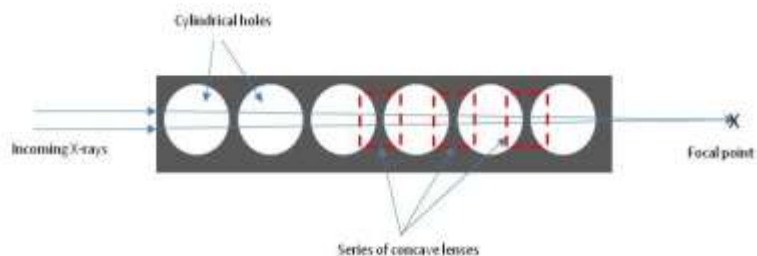
Schwarzschild optics



Wolter optics (nested mirrors)



Compound refractive lenses



Multilayer Laue lens

Image from Argonne National Laboratory

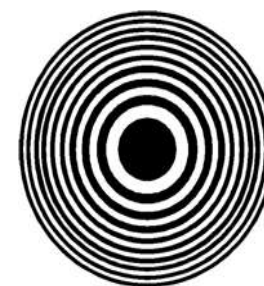


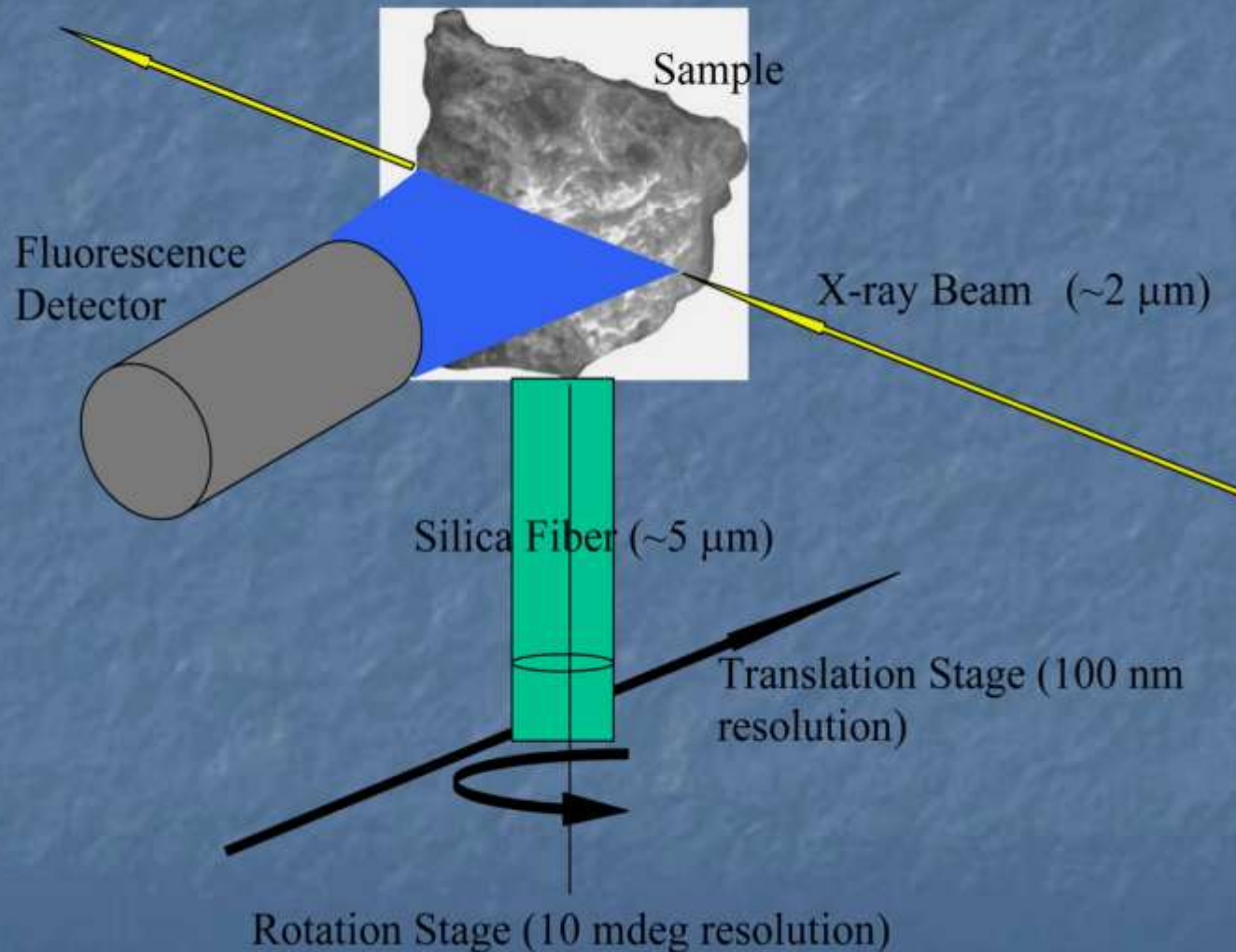
Figure 4: Fresnel zone plate

Fresnel zone plate

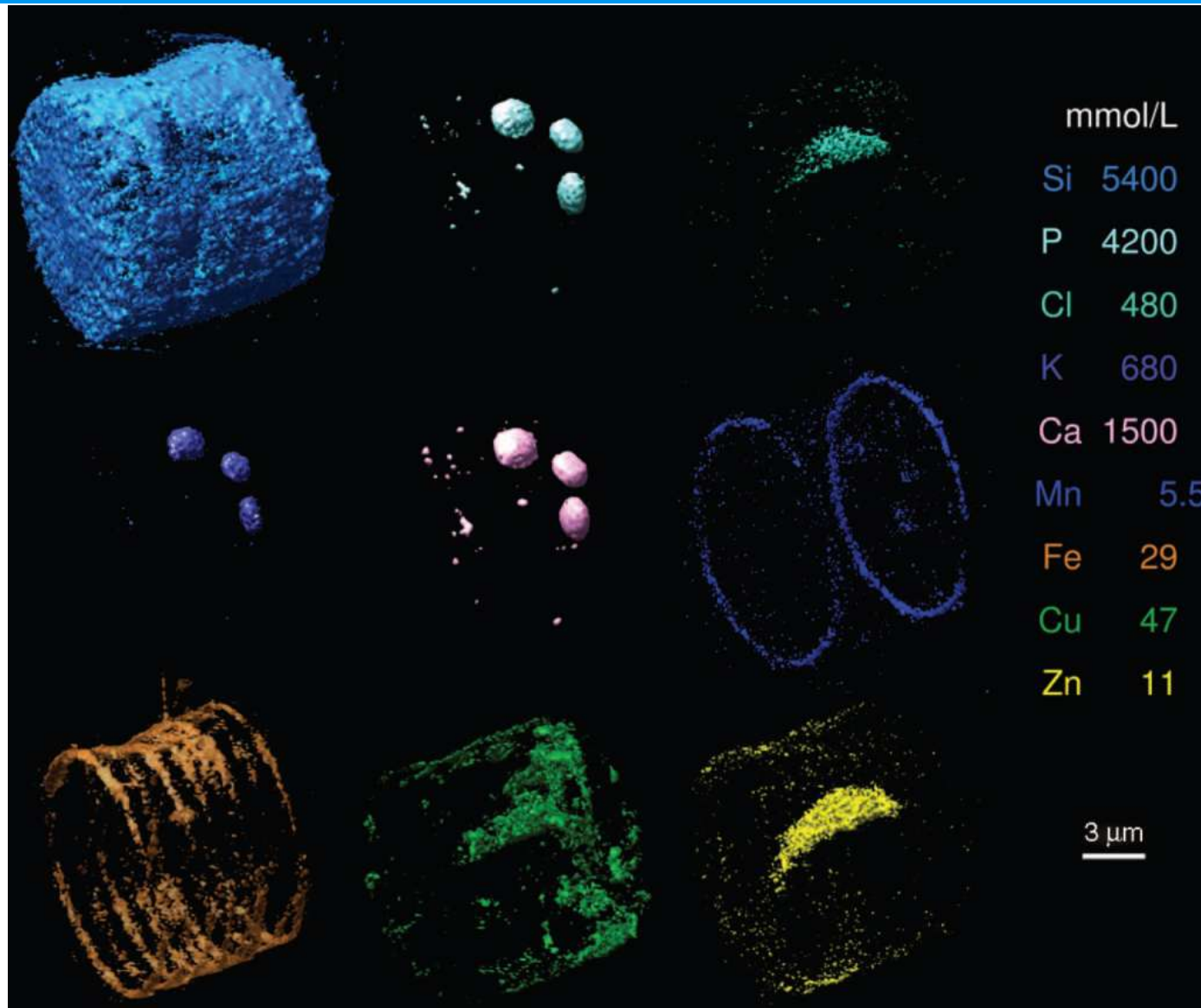
Images from Xradia.com

Images from wikipedia.org

# Fluorescence Microtomography Apparatus

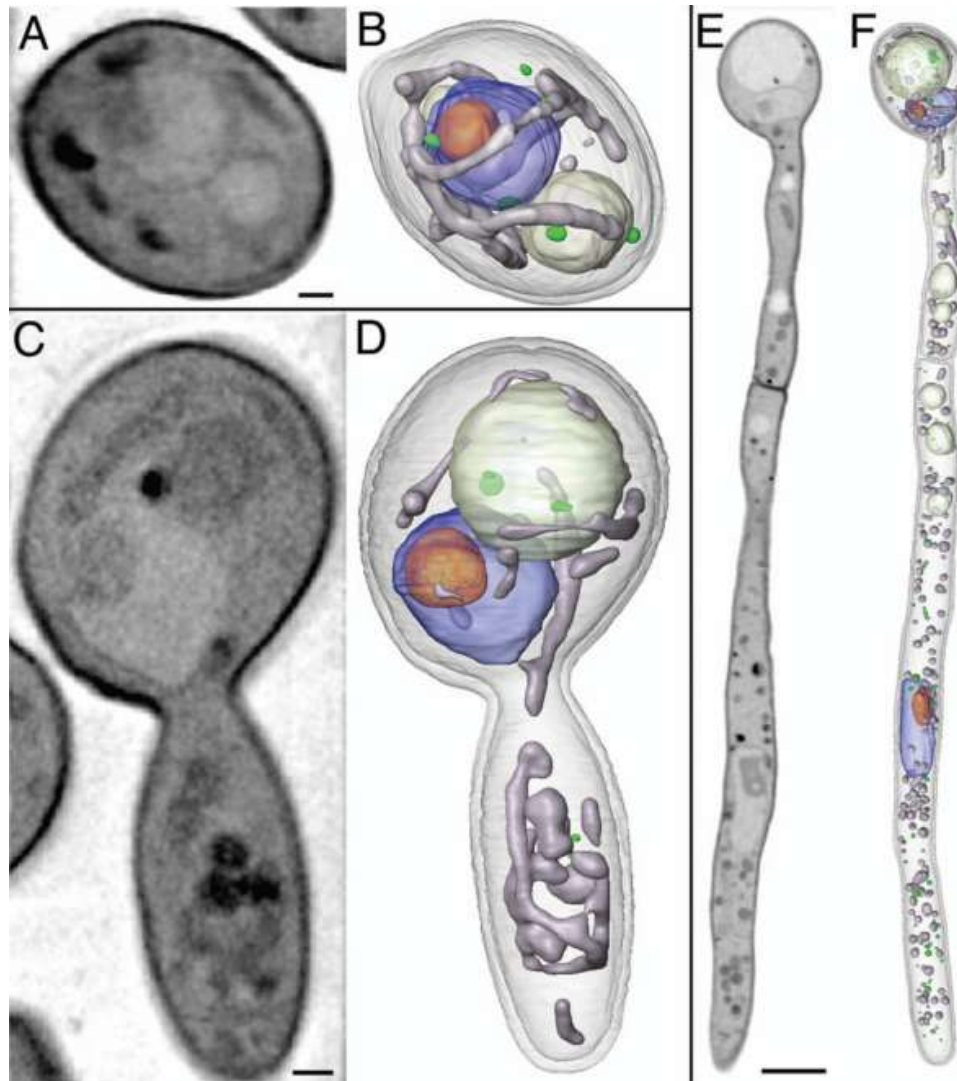


# 3D elemental distributions in the marine protist *Cyclotella meneghiniana* - X-ray fluorescence tomography



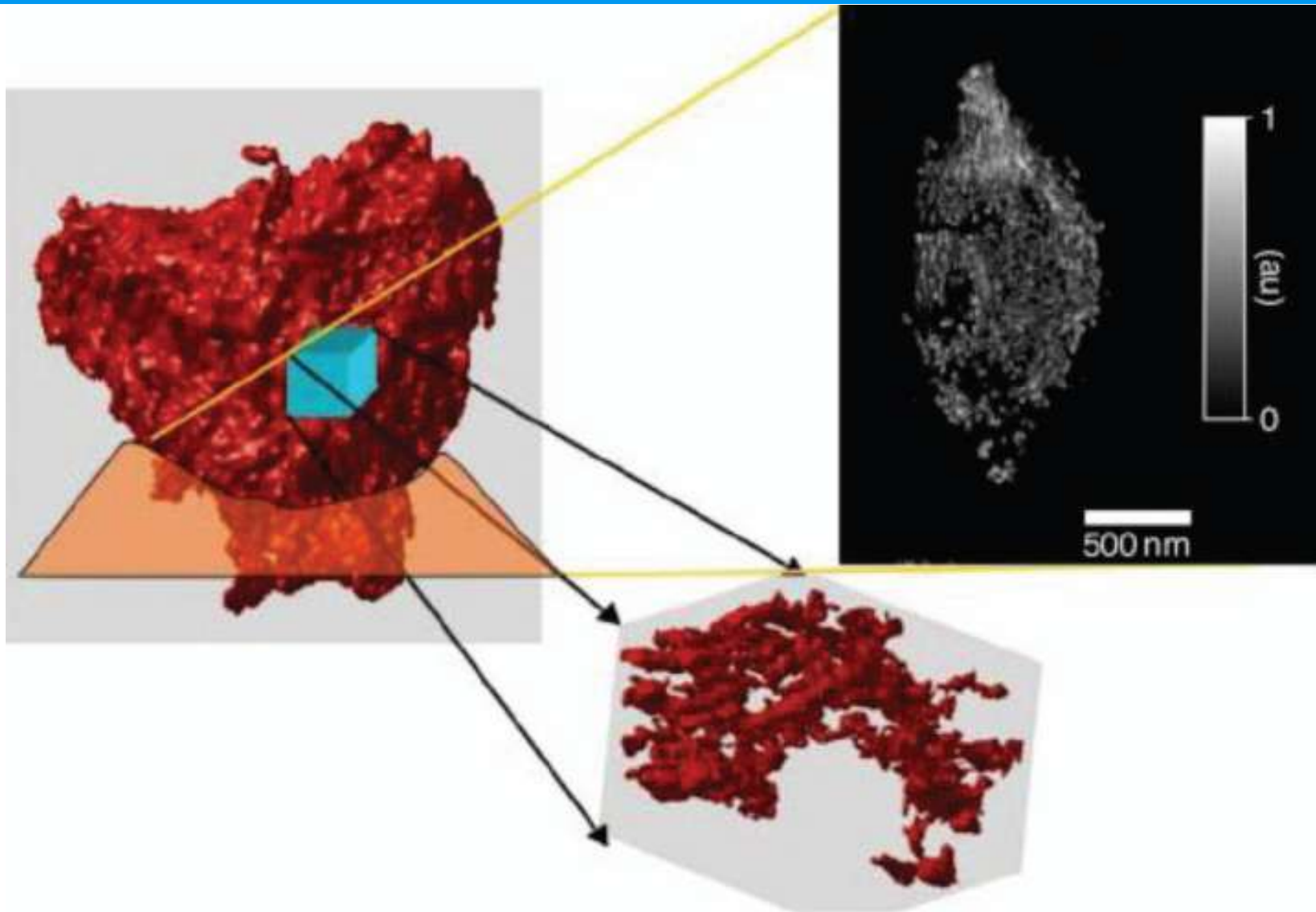
M.D. de Jonge, et al., Proc. Natl. Acad. Sci. USA 107(36) (2010).

# Soft X-ray tomographic reconstruction of phenotypically distinct *C. albicans* cells



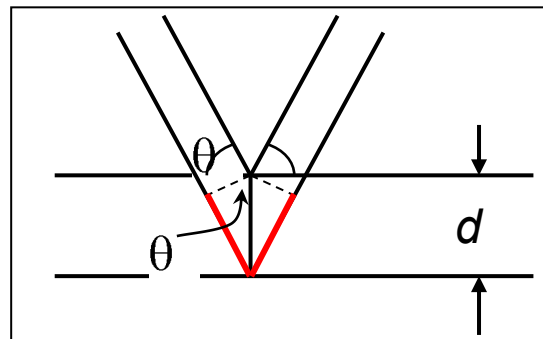
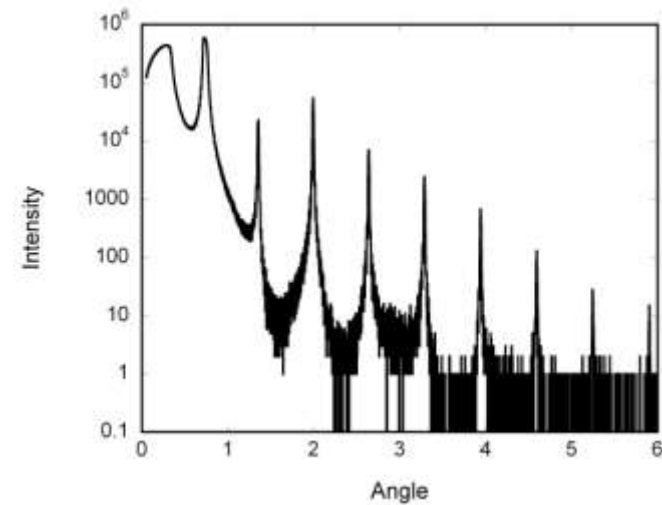
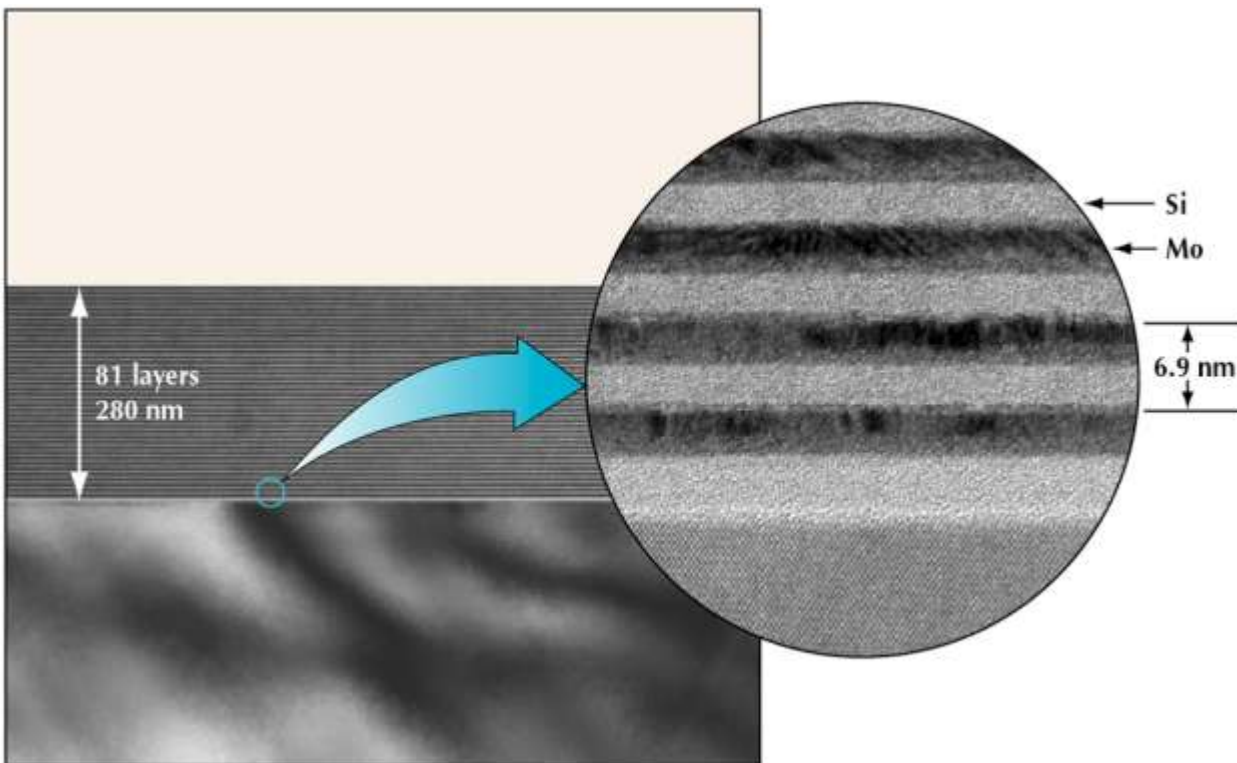
Uchida et al. Proc. Natl. Acad. Sci. USA 106 (2009)

# Diffraction microscope image of a $\text{Ta}_2\text{O}_5$ aerogel foam



Barty et al. Phys. Rev. Lett. 101 (2008)

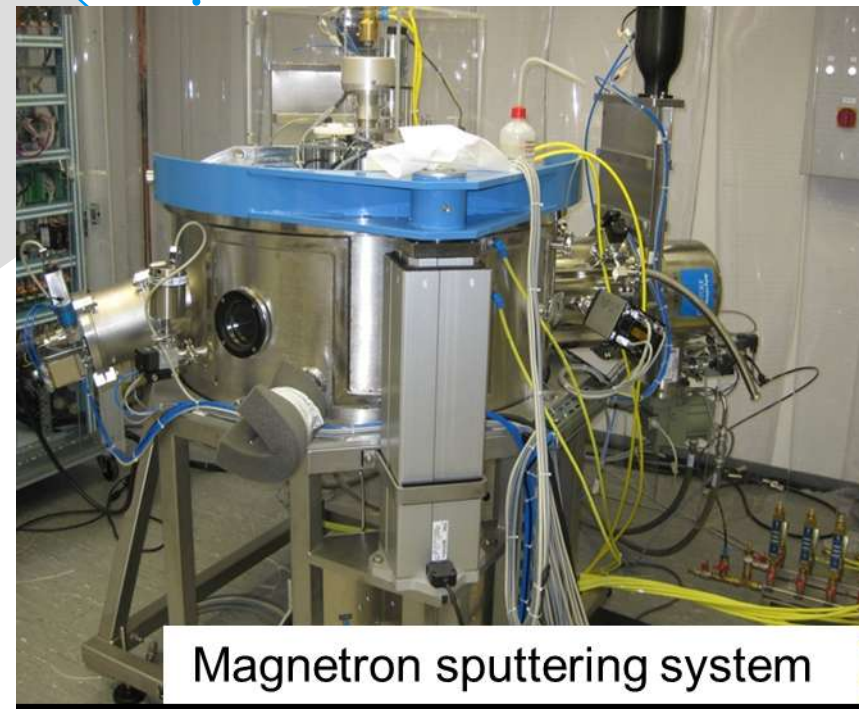
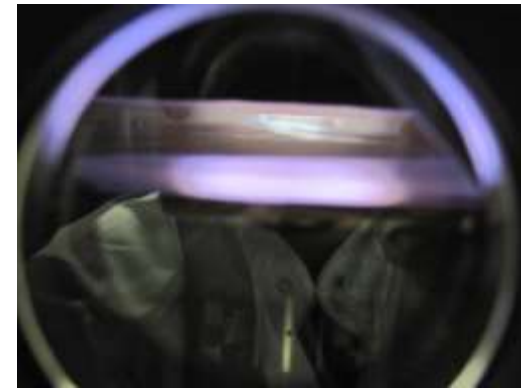
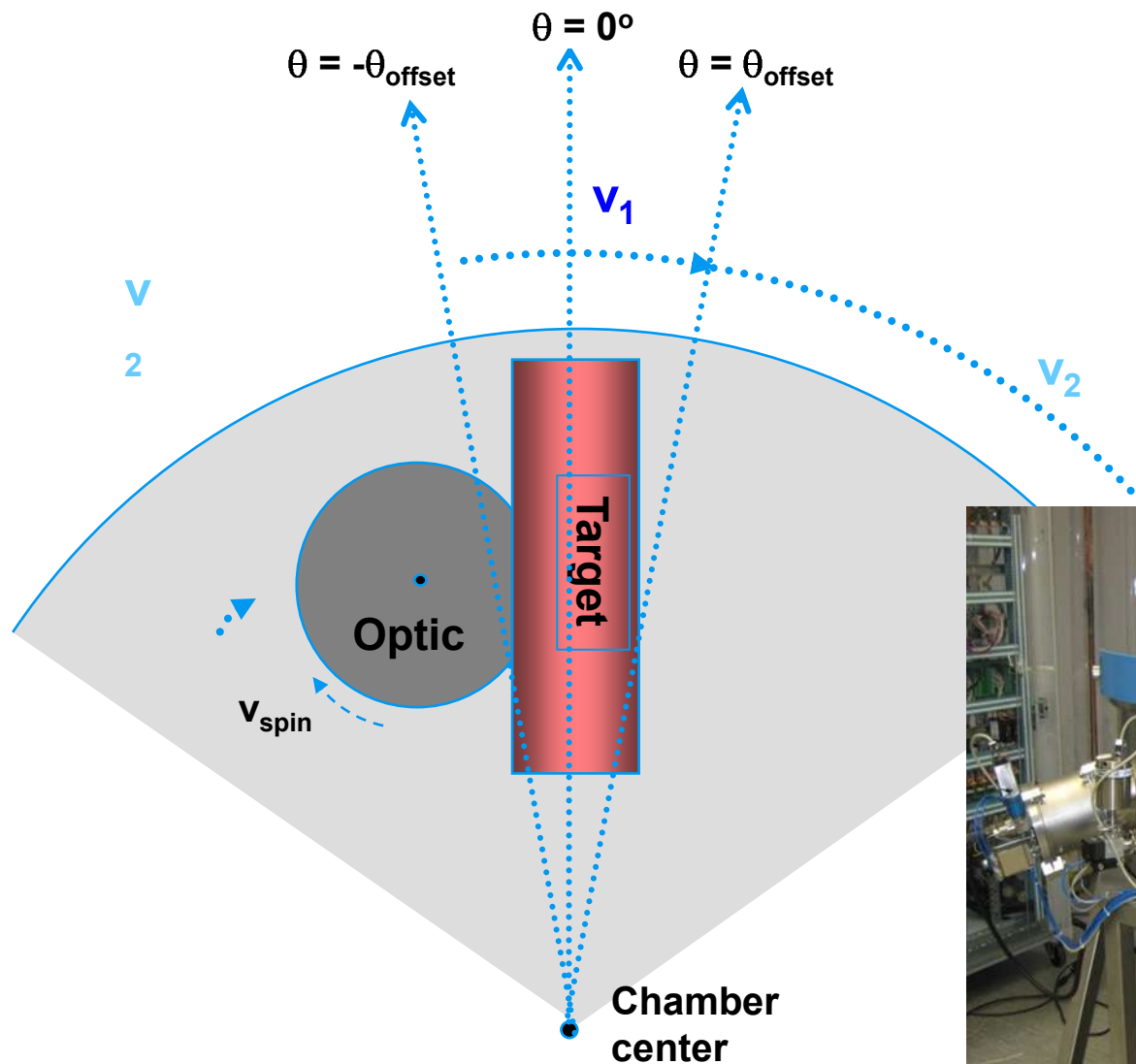
# Multilayer coatings consist of alternating layers of (usually) two materials of differing refractive index



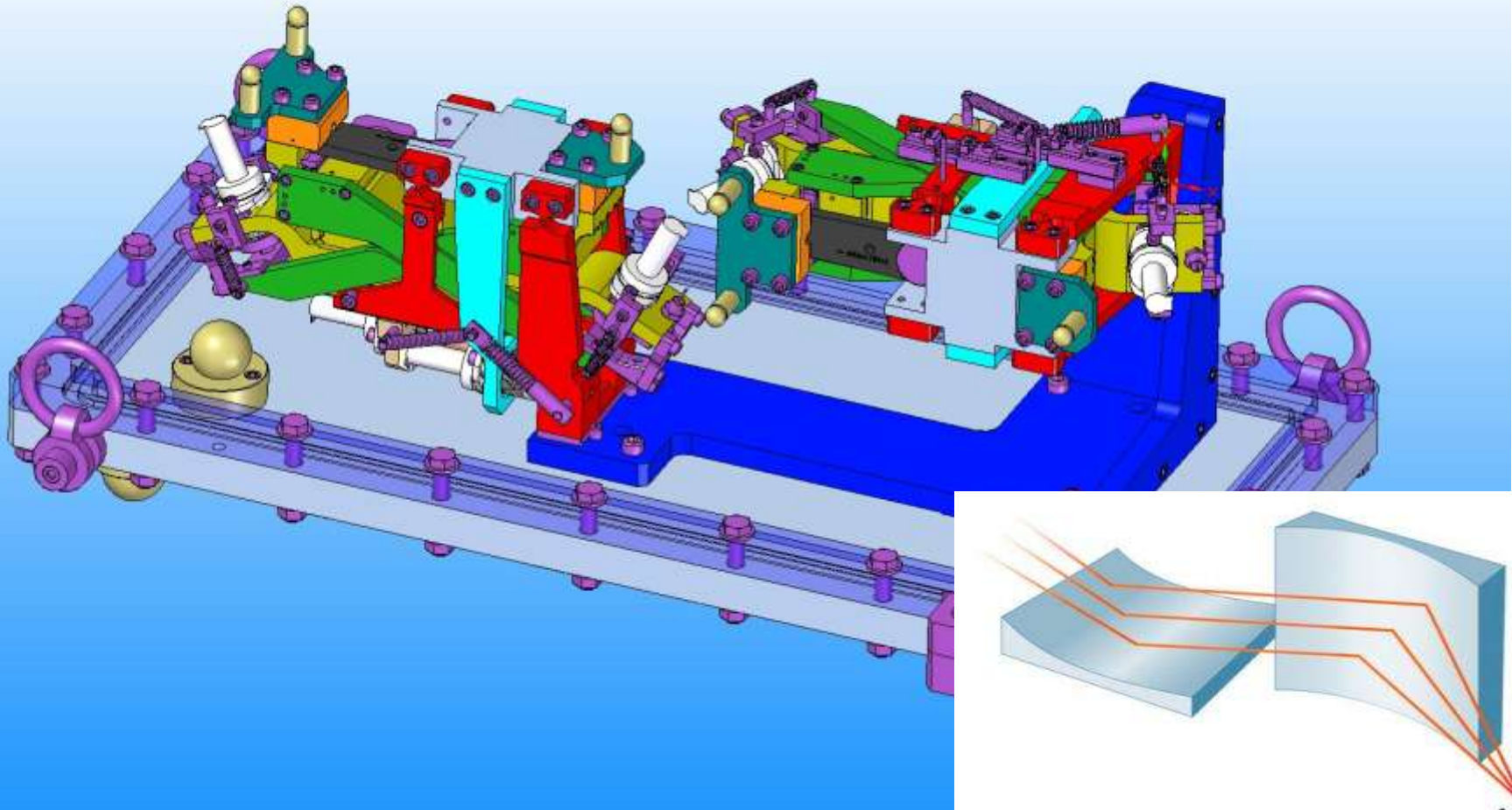
Bragg's Law

$$2d \sin\theta = m\lambda$$

# Velocity modulation and masking are rapidly converging methods used for multilayer thickness control

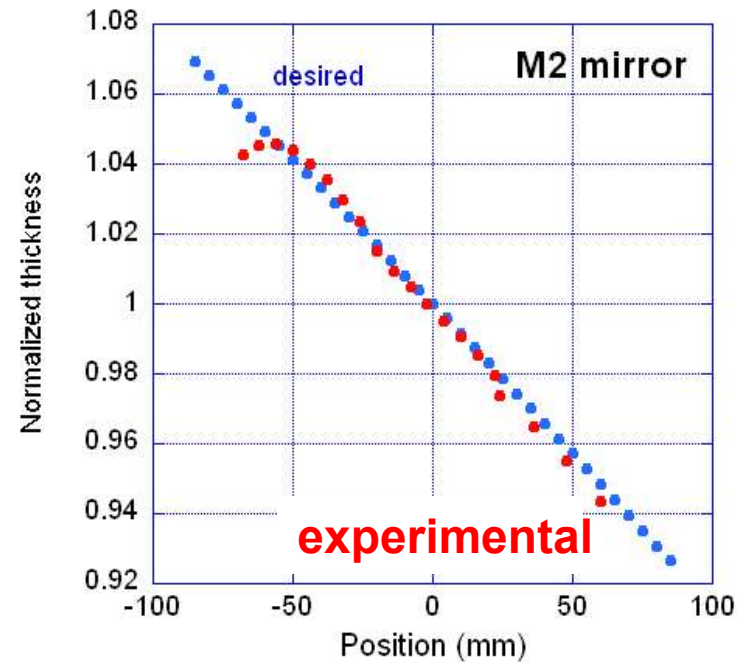
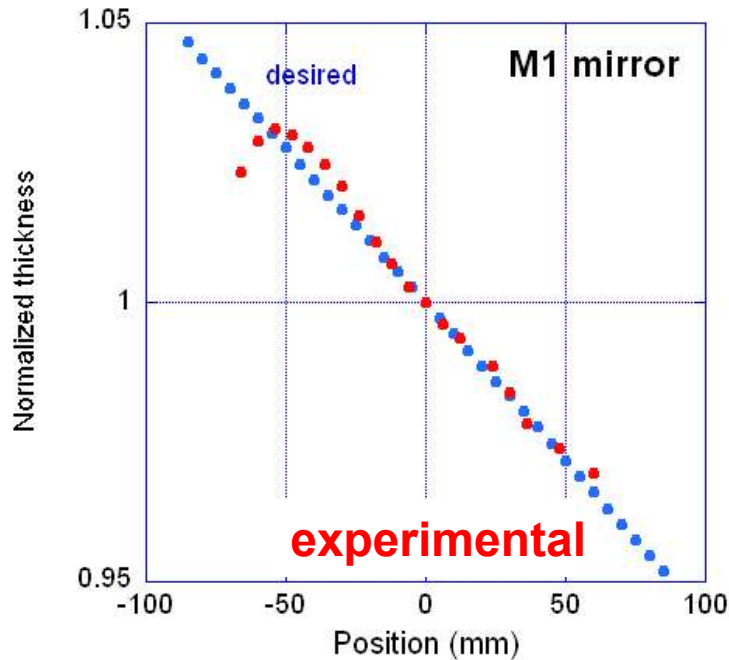


# Multilayer coatings for Kirkpatrick-Baez mirrors

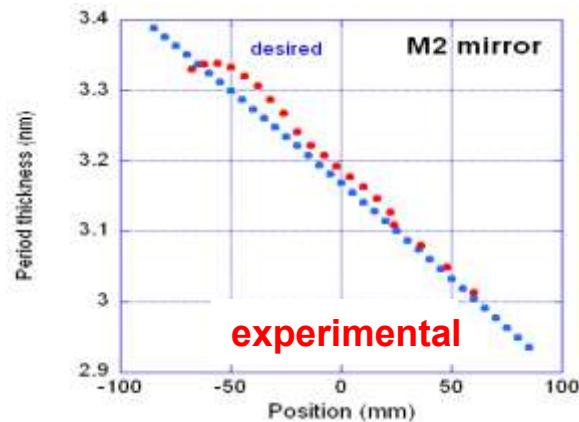
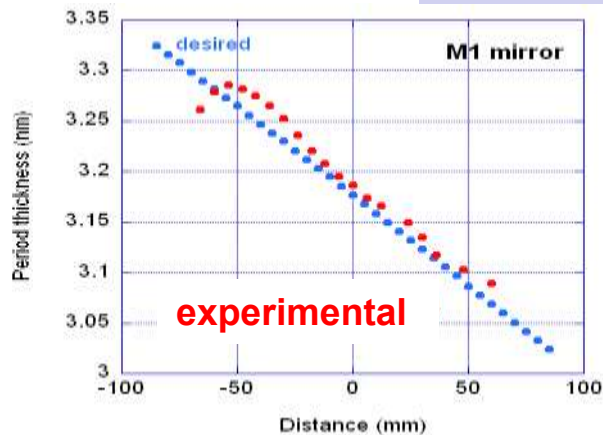


**P01 beamline at PETRA III (Collaboration with Rolf Rohlsberger)**

# Achieved lateral d-spacing gradients match desired gradients within specifications

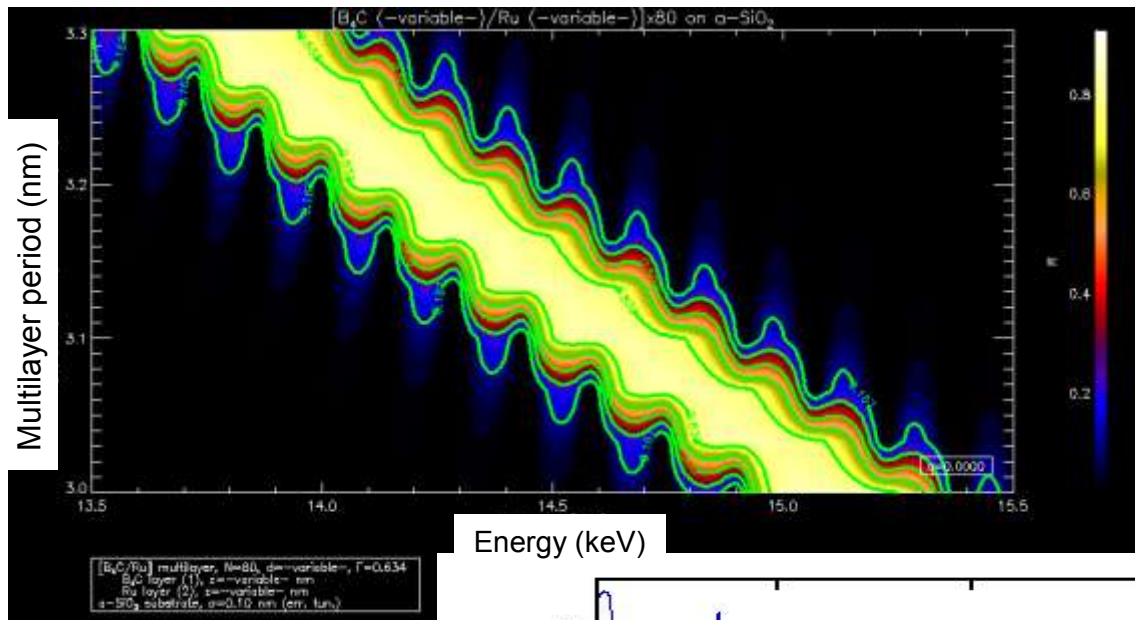


Individual layer thickness  $\sim 1.2\text{-}1.7$  nm

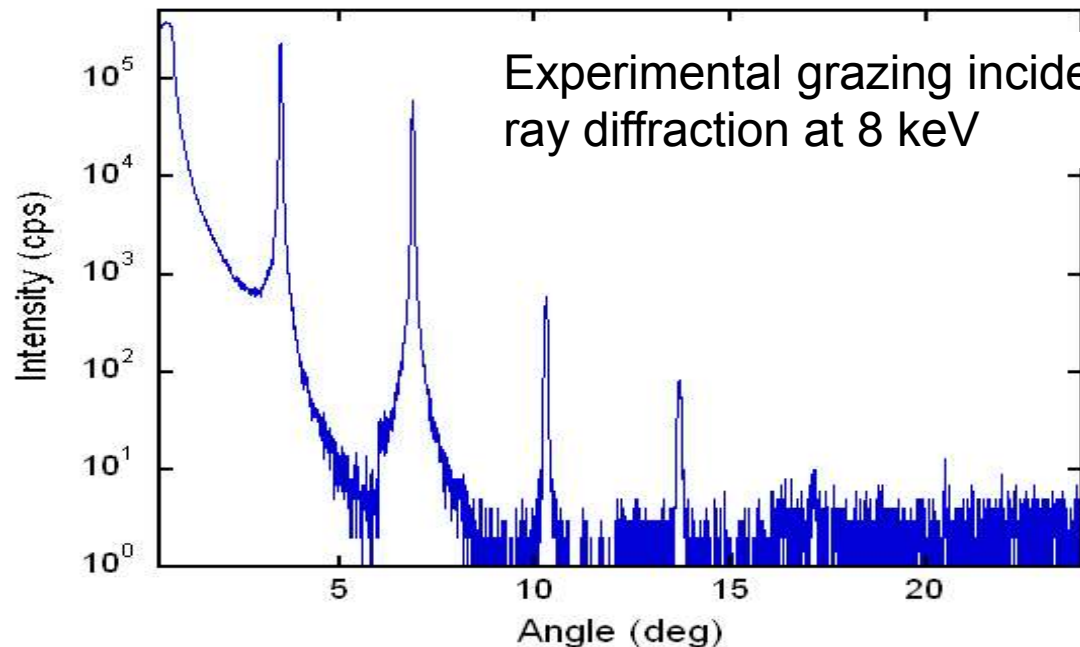


Velocity modulation  
calculated with software  
developed by A. Aquila

# Absolute reflectivity depends on substrate surface roughness, interface roughness and interdiffusion



Simulated reflectivity as a function of energy and multilayer period of Ru/B<sub>4</sub>C multilayer coated mirror

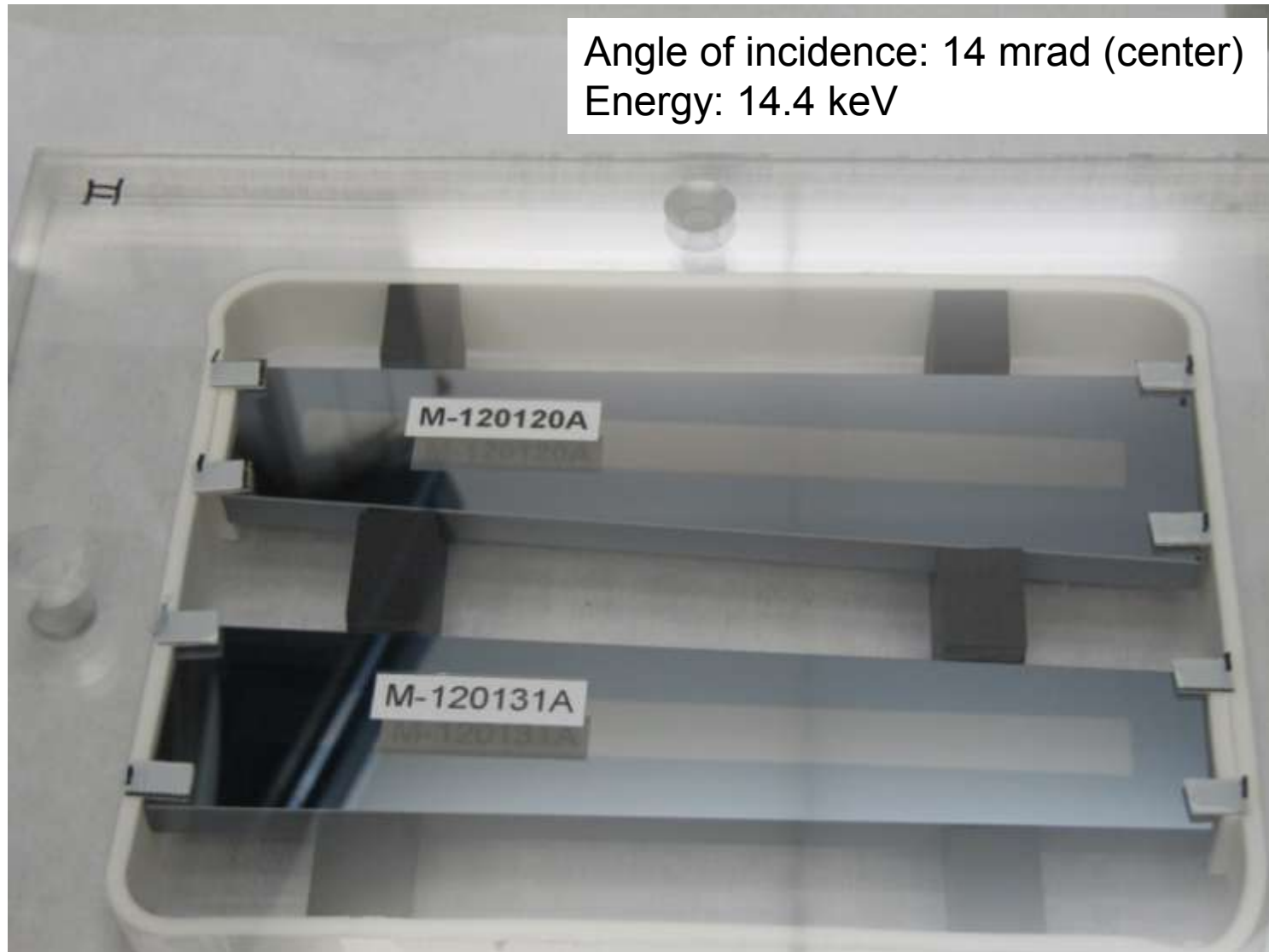


Experimental grazing incidence x-ray diffraction at 8 keV

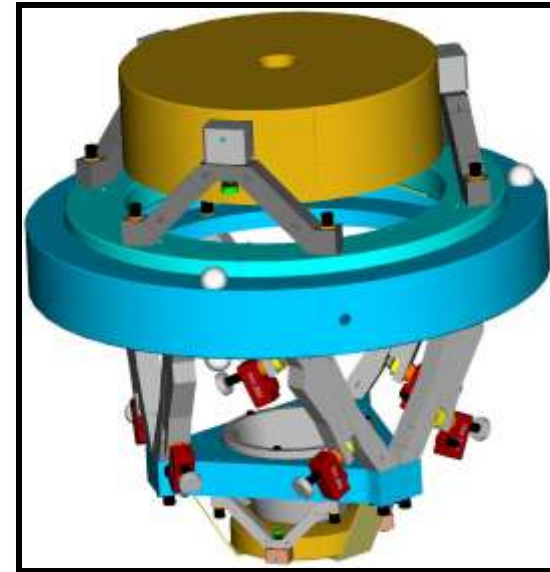
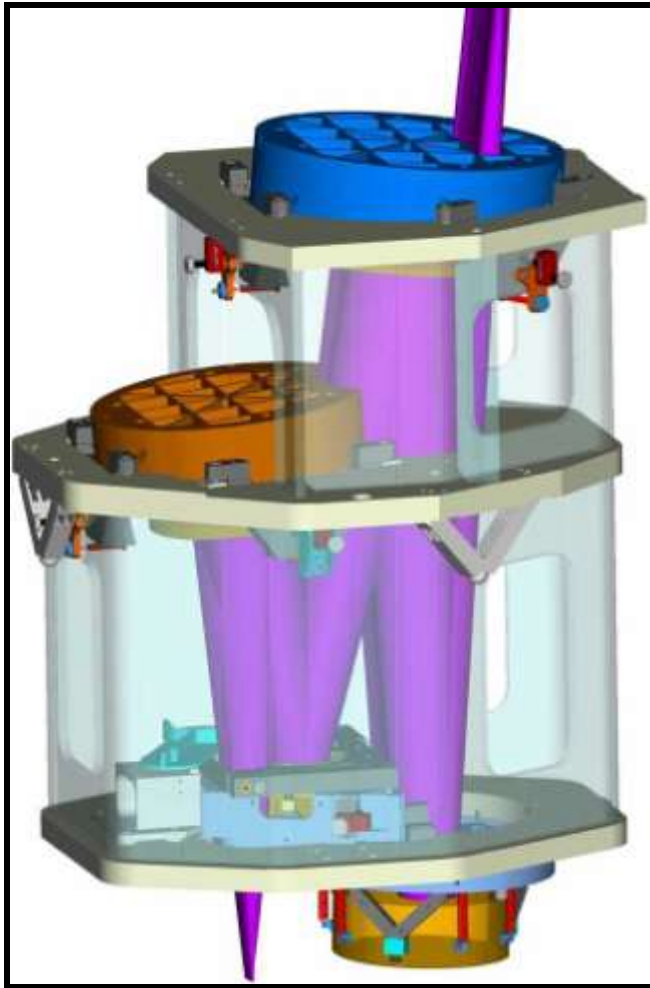
# Two 17 cm long KB mirrors have been successfully coated with high reflectivity Ru/B<sub>4</sub>C multilayer



Angle of incidence: 14 mrad (center)  
Energy: 14.4 keV



# Diffraction-limited performance with normal incidence mirrors was achieved for EUV imaging systems

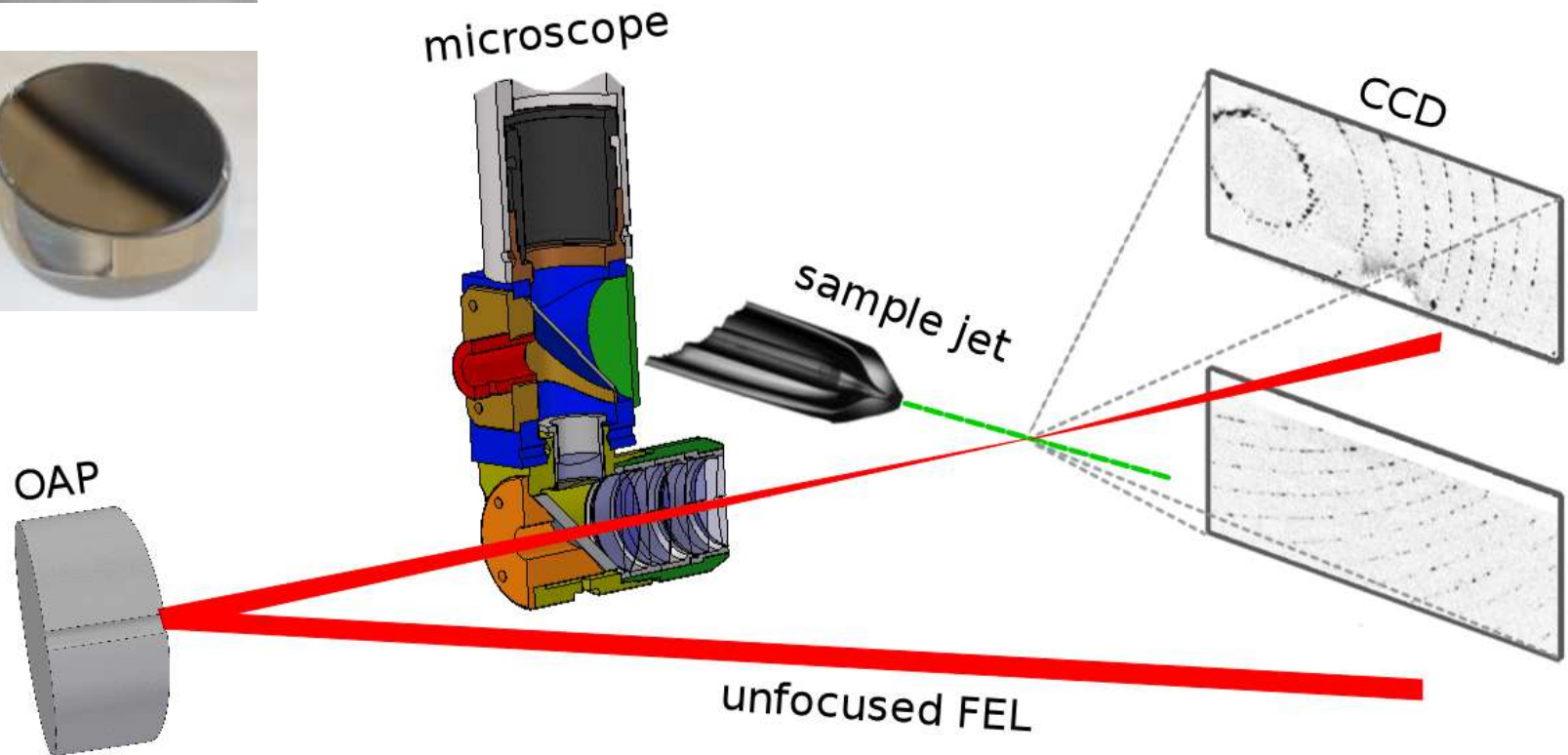


**0.3 NA "MET PO Box 2"**  
**2 Aspheric Mirrors**

**0.1 NA "ETS PO Box 2"**  
**3 Aspheric Mirrors + 1 Sphere**

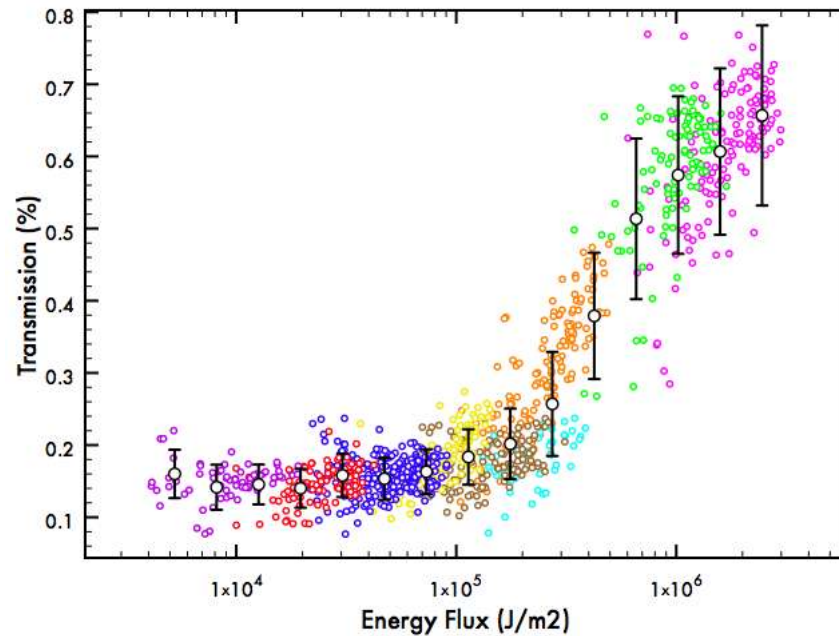
Optics developed, coated and aligned at LLNL as part of EUVL project

# This technology was transferred to normal incidence mirrors for focusing high intensity x-rays from FLASH

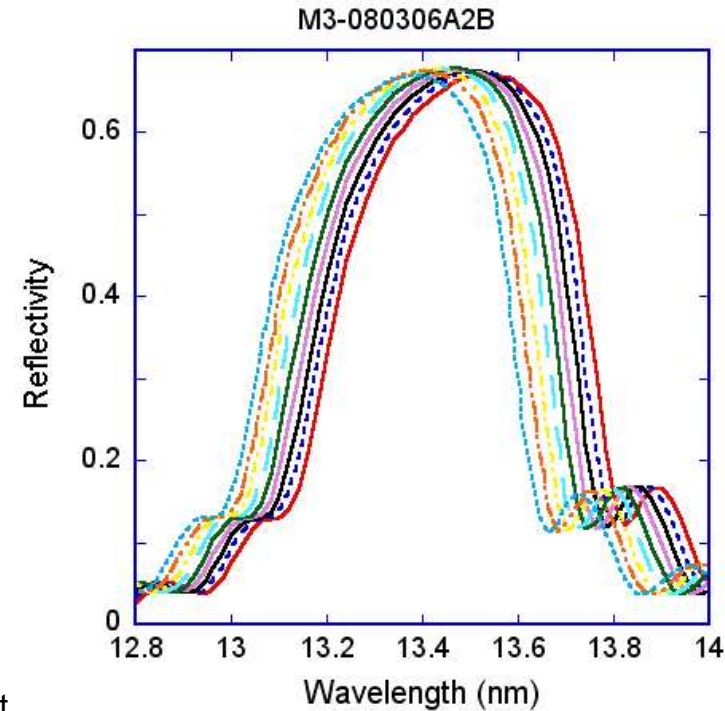
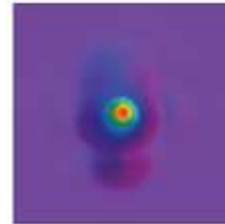


Recent experimental setup in our chamber at FLASH

# Our multilayer coated off-axis parabolic mirror focused 13.5 nm FLASH beam to sub- $\mu\text{m}$ spot



0.35  $\mu\text{m}$  focal spot  
(as predicted by Jacek Krzywinski)



Nagler et al., Nature phys. 5, 693 (2009)  
Vinko et al., PRL 104, 225001 (2010)

A. Nelson et al., Opt. Exp. 17, 18271 (2009)  
S. Bajt et al., SPIE 7361 (2009)

For the first time intensities of 40 mJ / (40 fs (10<sup>-4</sup> cm)<sup>2</sup>), corresponding to power of  $\sim 10^{17}$  W/cm<sup>2</sup>, were achieved and interesting experiments in warm dense matter could be performed.

## Considerations:

> Reflectivity, temporal and thermal stability, reactivity, intrinsic stress, availability, price

## Candidates:

Normal incidence: 0 to 5 deg off normal;

Wavelength: 6 to 7 nm (Source: [http://henke.lbl.gov/optical\\_constants](http://henke.lbl.gov/optical_constants))

**Ru/B<sub>4</sub>C (20% at 6.8 nm, N= 150, J. Korthright)**

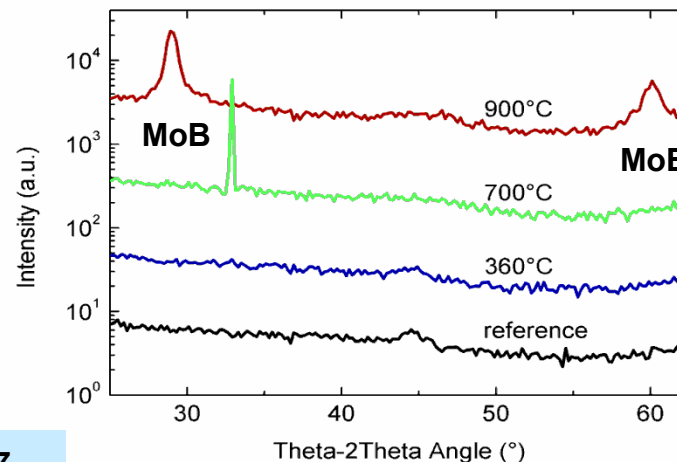
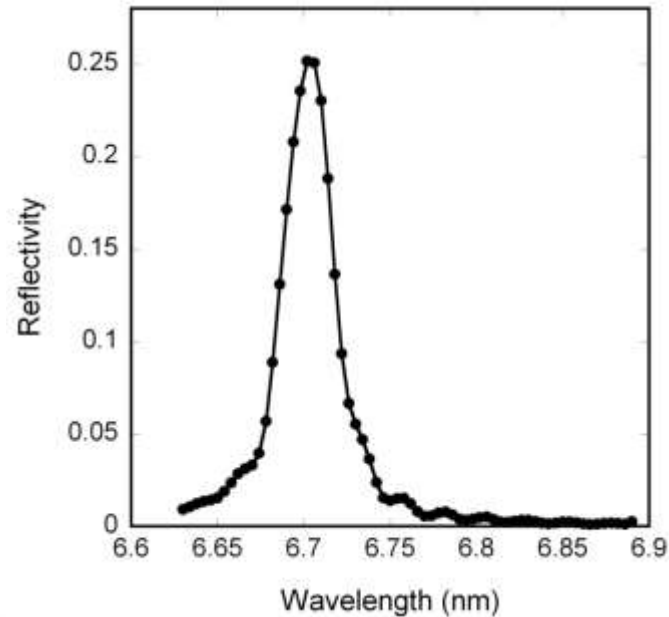
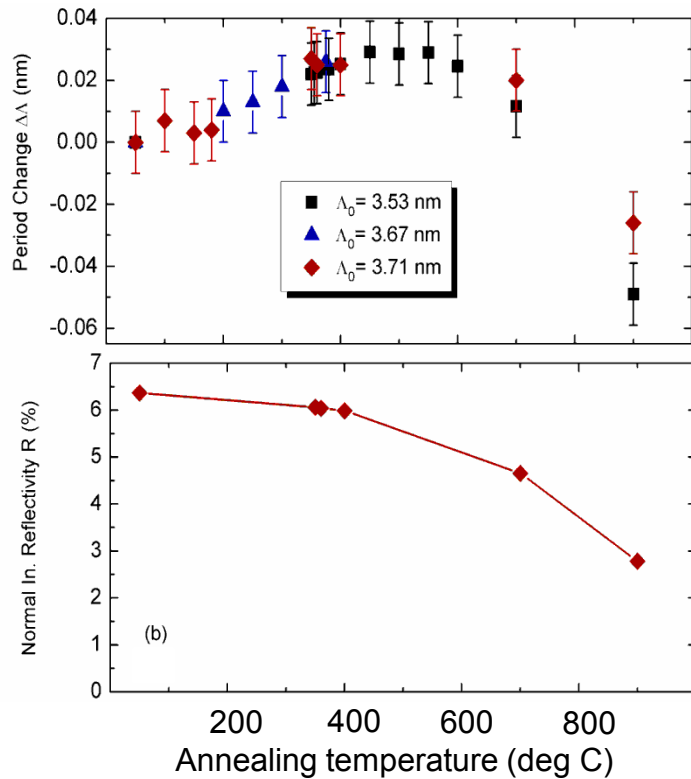
**Cr/C (18.9% at 6.42 nm, N = 150, H. Takenaka)**

**FeCrNi/B<sub>4</sub>C (16% at 6.8 nm, N = 100, D. Stearns and S. Vernon)**

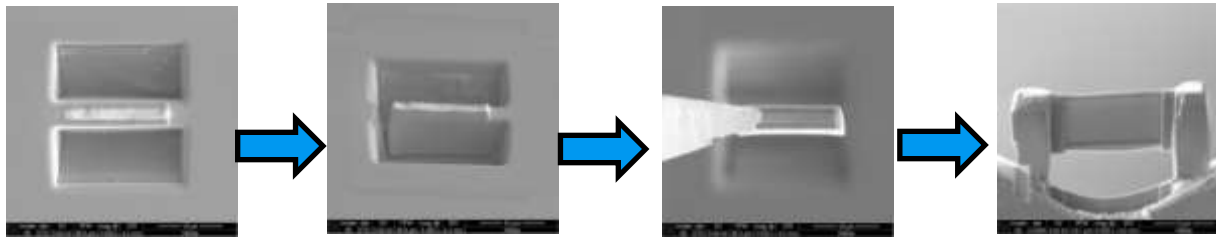
> La/B<sub>4</sub>C, LaN/B<sub>4</sub>C, LaN/B, La<sub>2</sub>O<sub>3</sub>/B<sub>4</sub>C

> Mo/B, Mo/B<sub>4</sub>C

# Mo/B<sub>4</sub>C MLs for 6.8 nm have decent reflectivity and are thermally stable – a good choice for FEL application



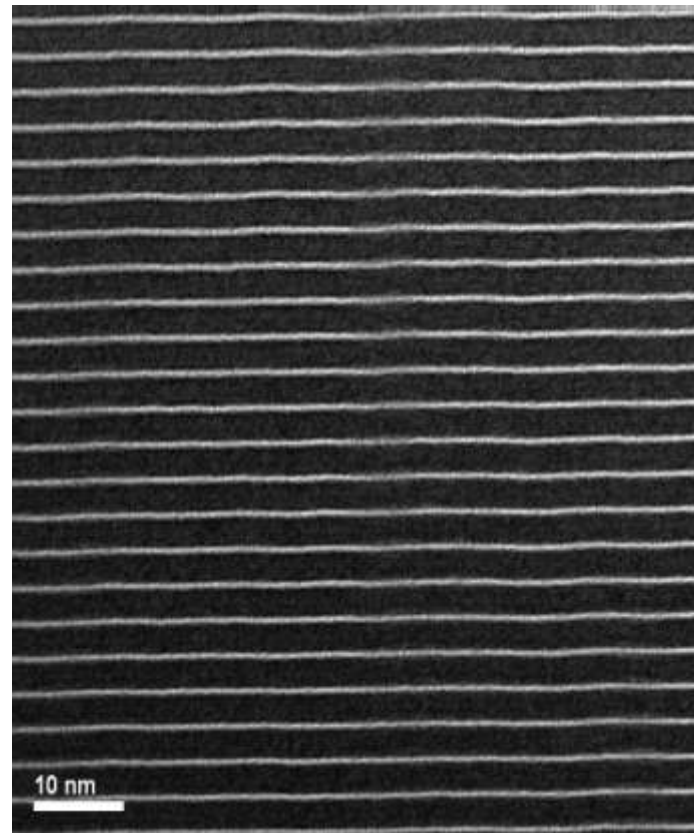
# Mo/B<sub>4</sub>C ML is thermally stable up to 600 deg C



TEM sample prepared by FIB

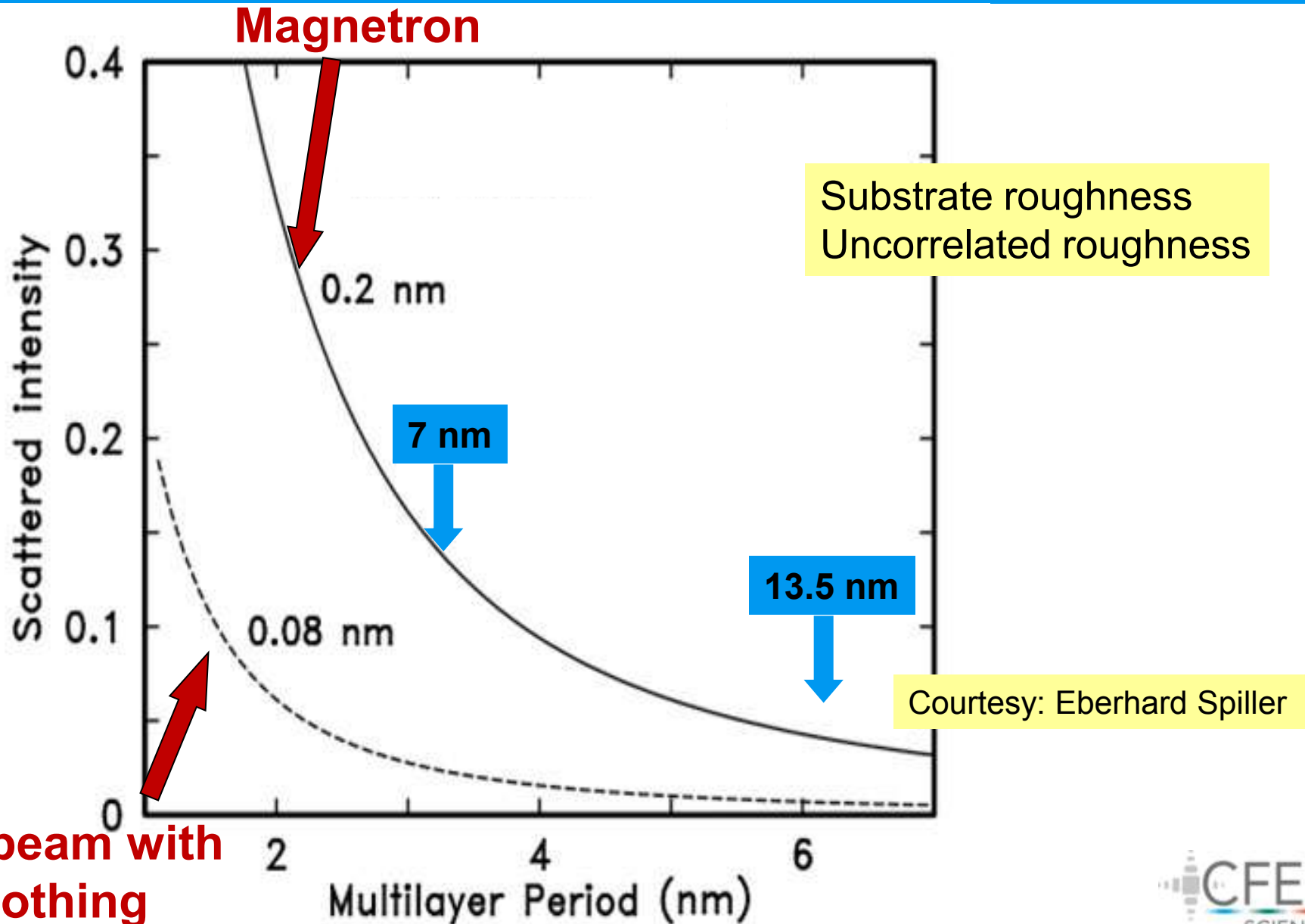


as-deposited state

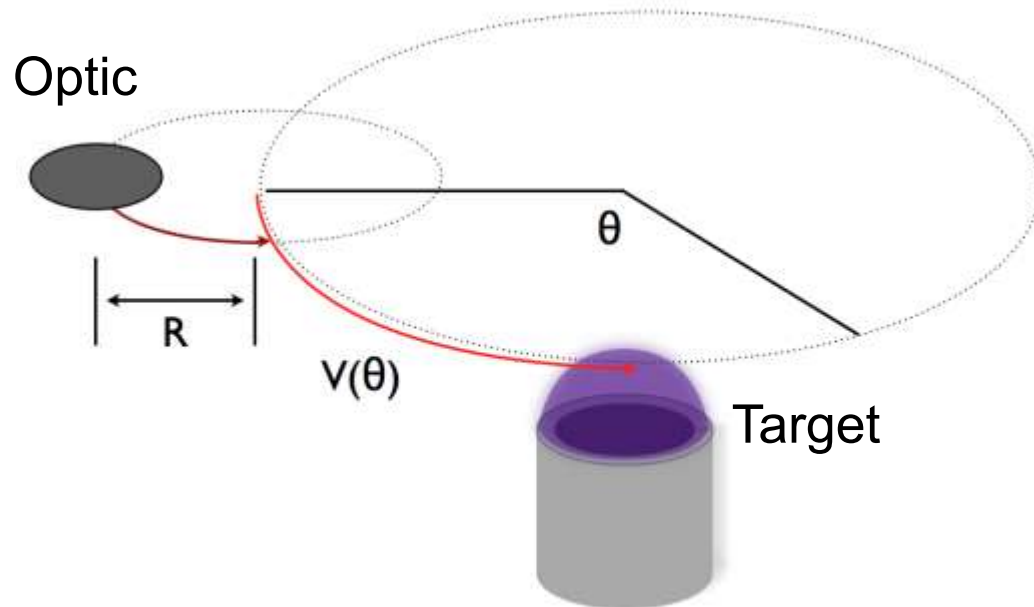


after 1h at 600 deg C

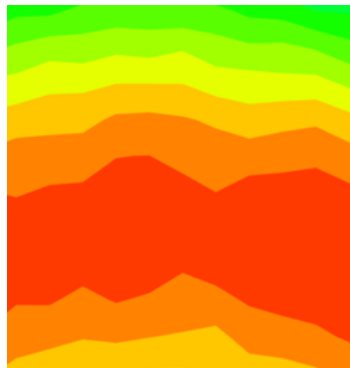
The shorter the wavelength the smaller the period and the higher the impact from interfaces



# Achieved wavelength uniformity of 0.1% across clear aperture

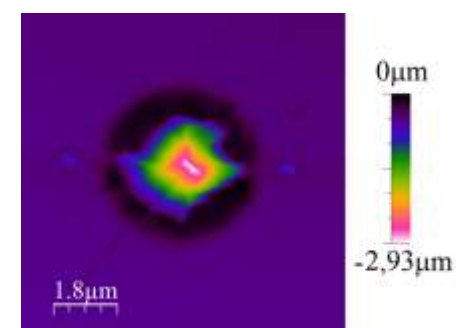
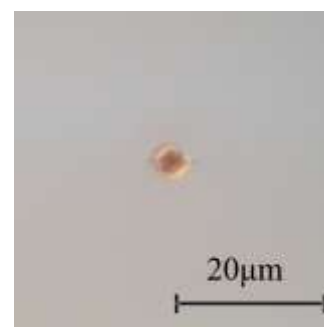
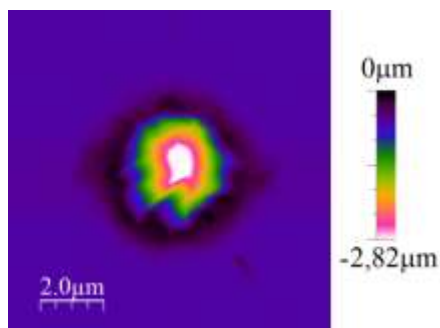
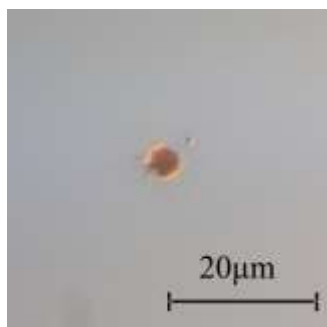
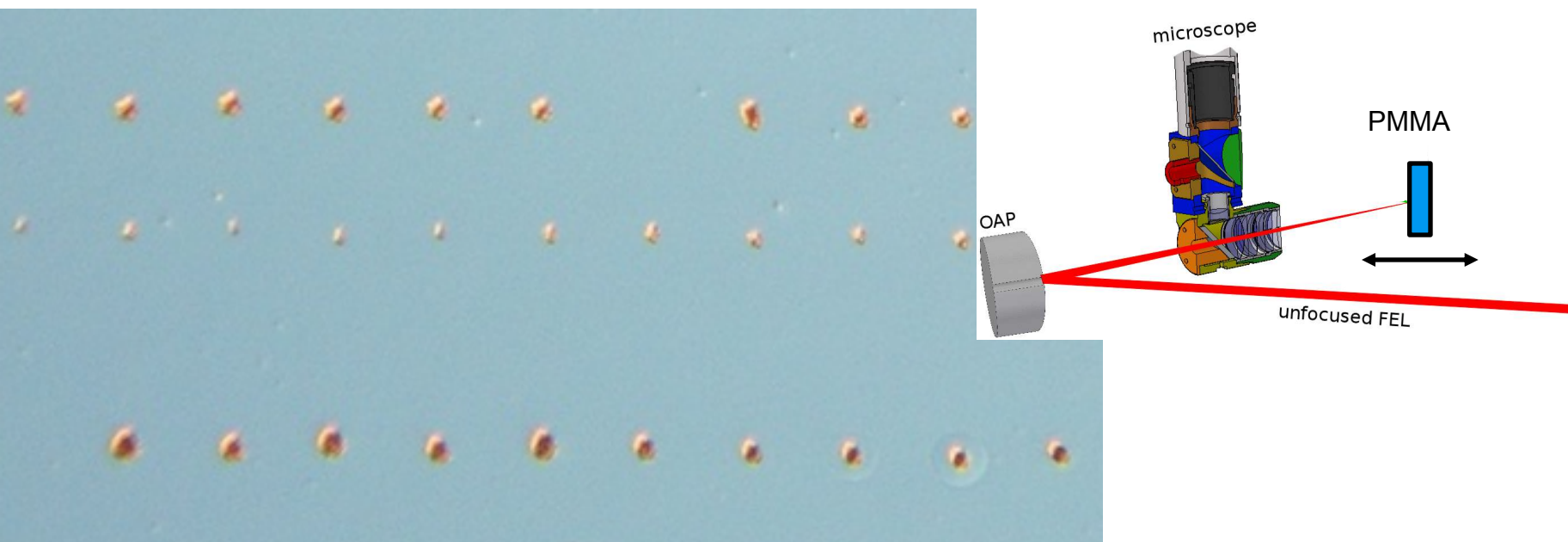


Reflectivity map at a constant wavelength of 6.7980 nm across clear aperture



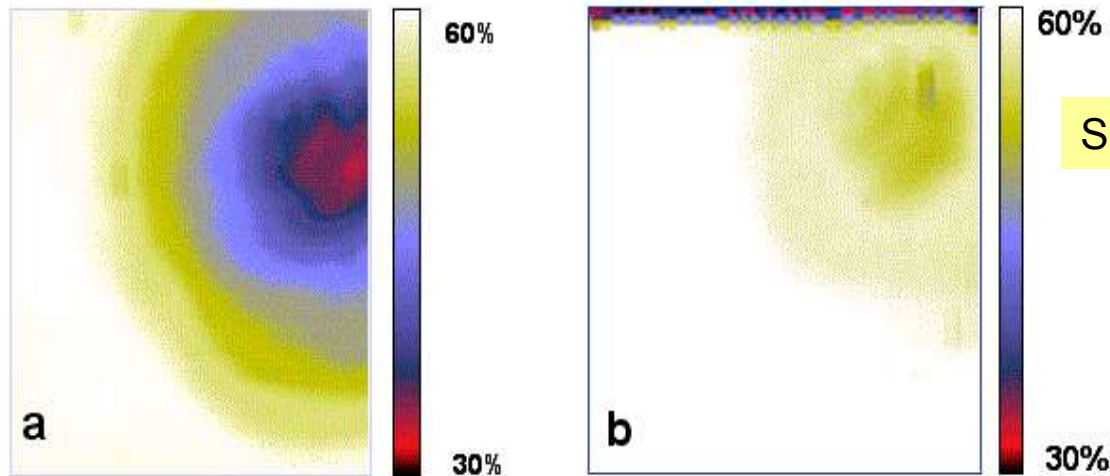
Super-polished substrate with <0.2 nm HSFR and MSFR and figure error of 0.2-0.3 nm

# Finding the best focal spot using PMMA



Collaboration with T. Burian, V. Hájková, J. Chalupský, L. Juha (IOP, Prague)

# Carbon contamination is also a well known problem in synchrotron and EUVL community



S. Bajt et al., SPIE 7361 (2009)

Reflectivity map shows modulations due to the overlayer thickness variation

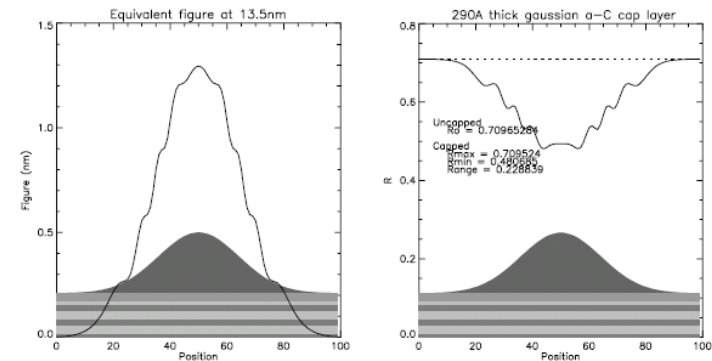
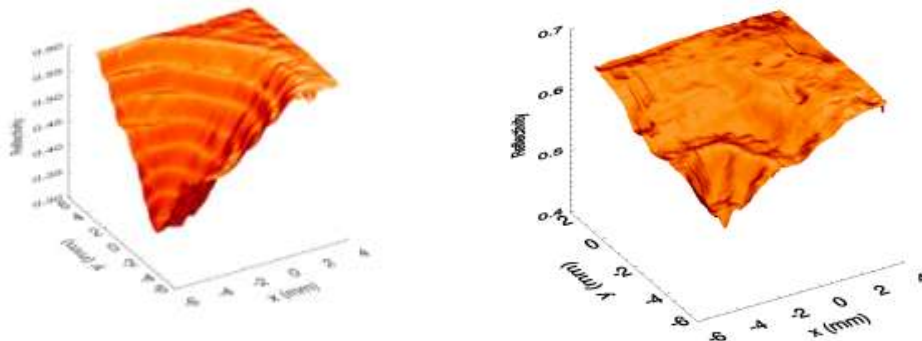
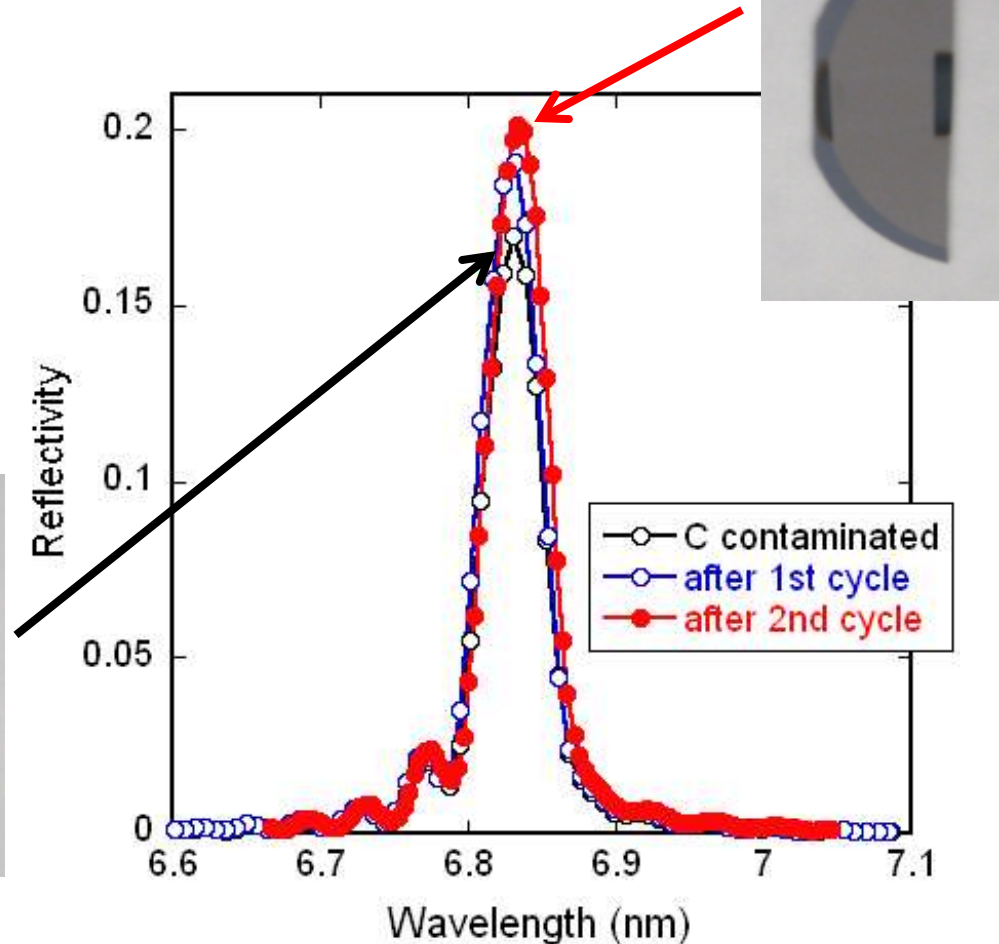


Figure 6

A. Barty and K. Goldberg, SPIE 5037 (2003)

Oxygen plasma cleaning is effective but does not completely recover the initial reflectivity

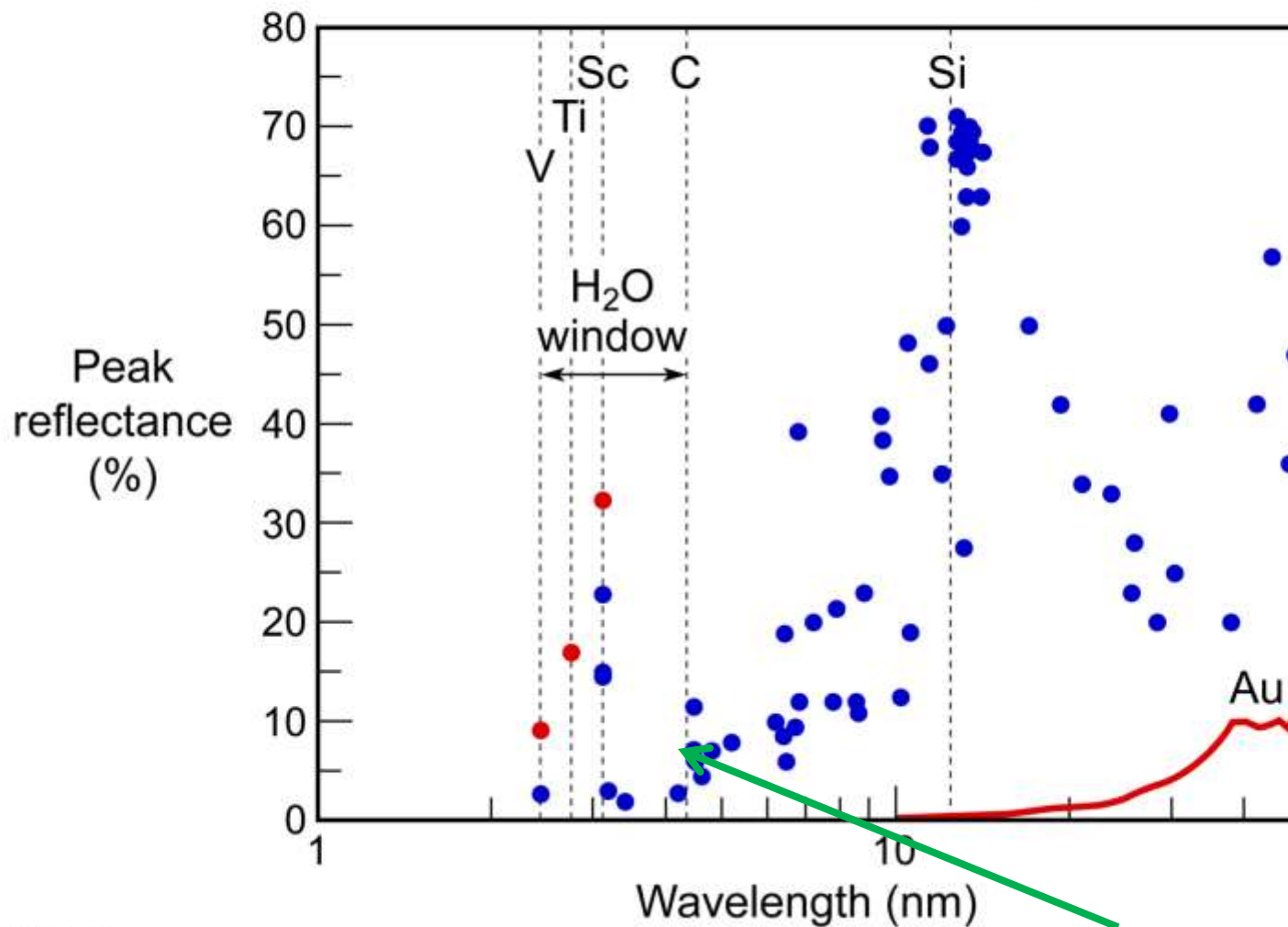
# We are developing processes to remove surface contamination from carbon containing MLs



and preliminary results are very encouraging

Miriam Barthelmeß

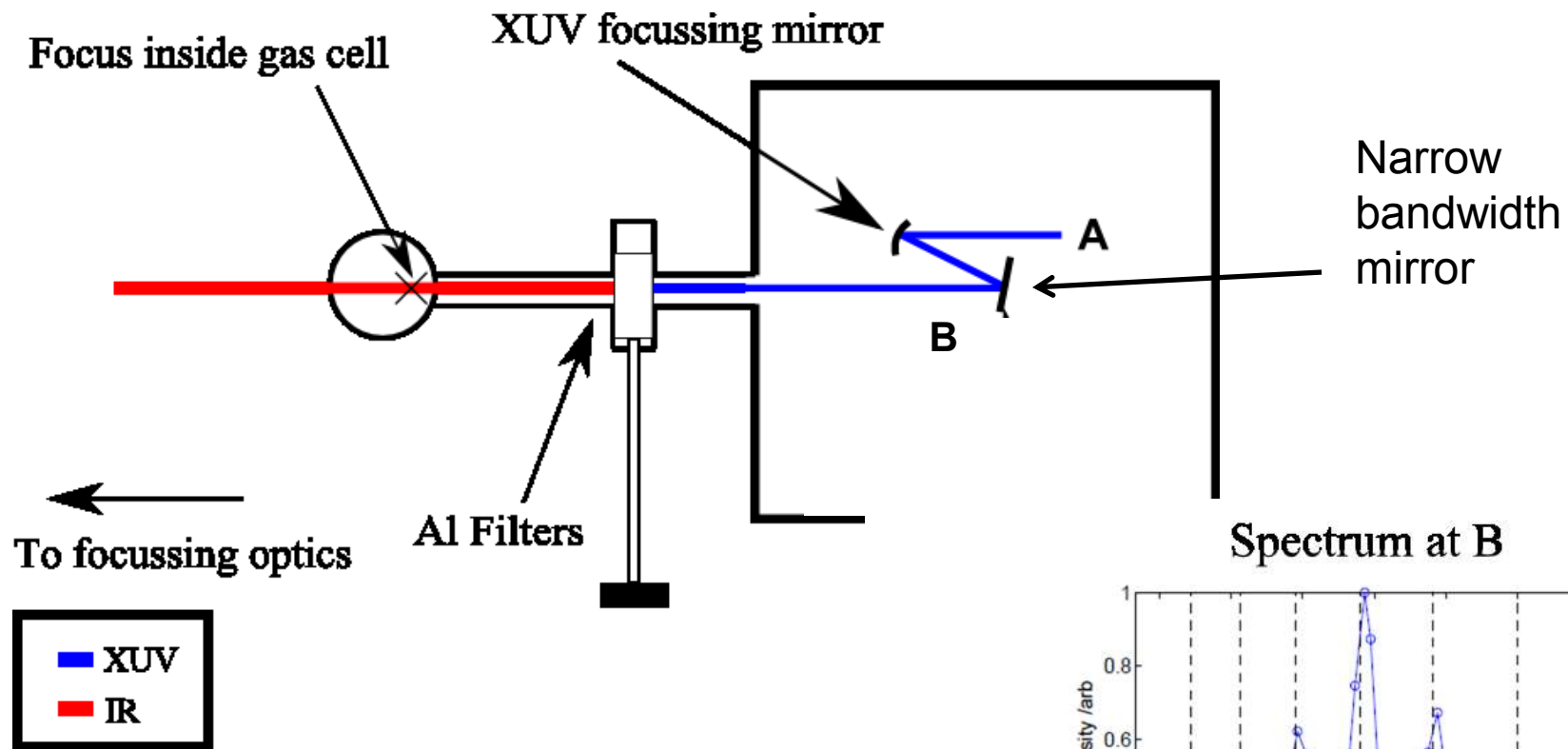
## Near-Normal Incidence Multilayer Mirrors



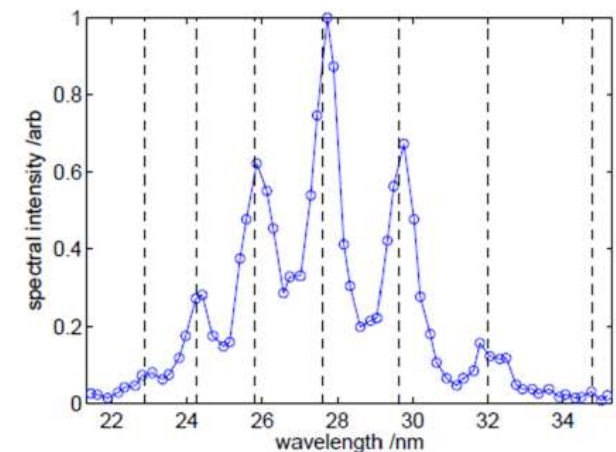
From: Prof. D. Attwood's lecture

4.2 nm < λ < 4.4 nm

# HHG sources produce short and intense pulses

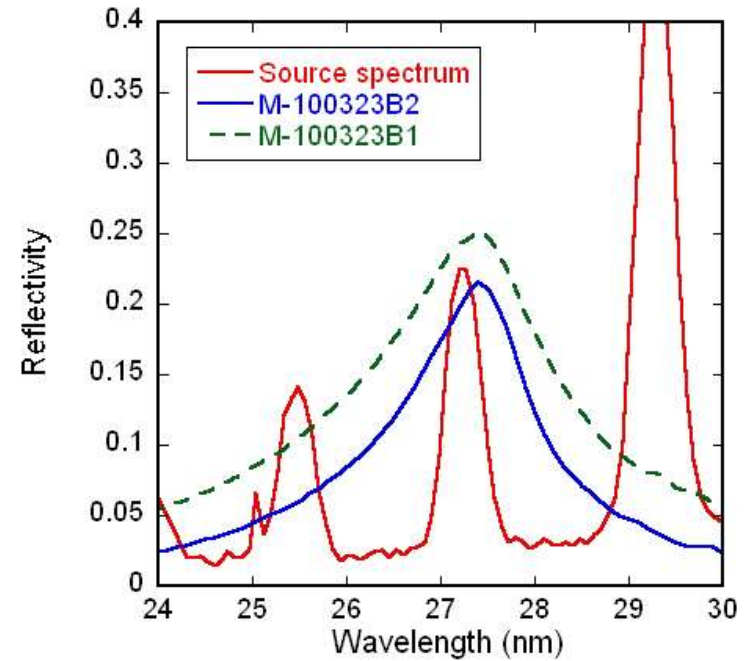
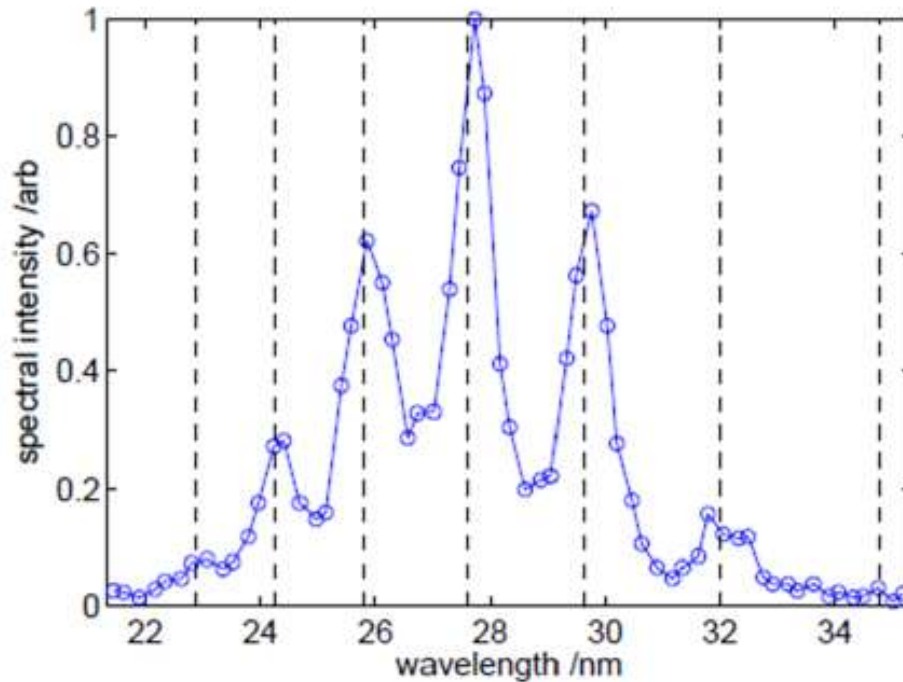


Spectrum at B



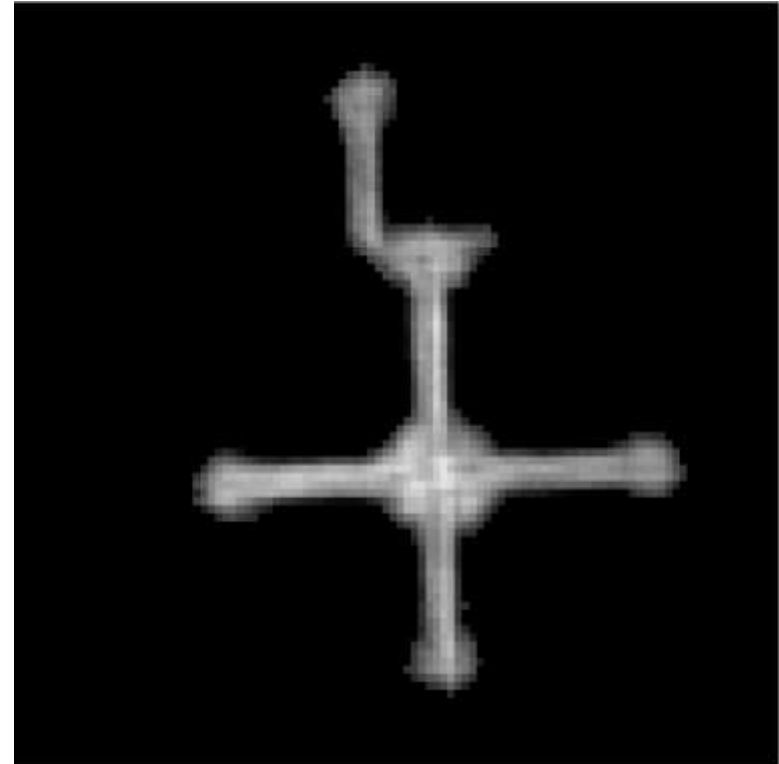
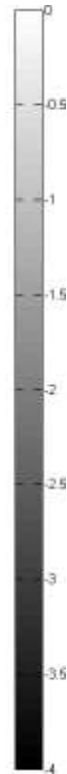
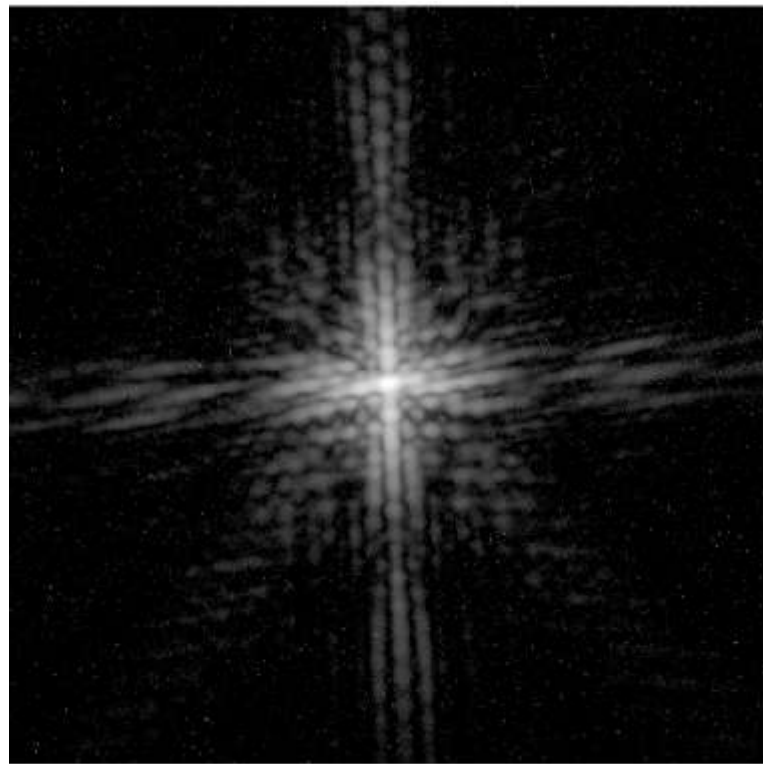
Collaboration with A. Parsons, Univ. of Southampton, UK

## Spectrum at B



Collaboration with A. Parsons, Univ. of Southampton, UK

# Coherent diffractive imaging and reconstruction done using narrow bandwidth multilayers



X-ray diffraction obtained with narrowband multilayer

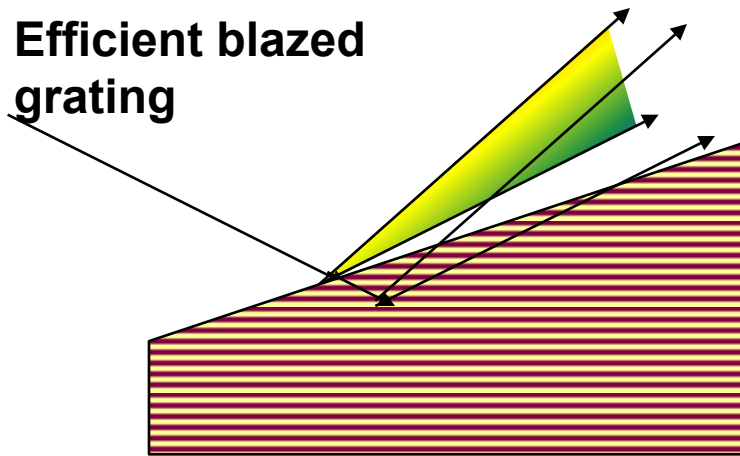
Reconstructed image

Collaboration with A. Parsons, Univ. of Southampton, UK

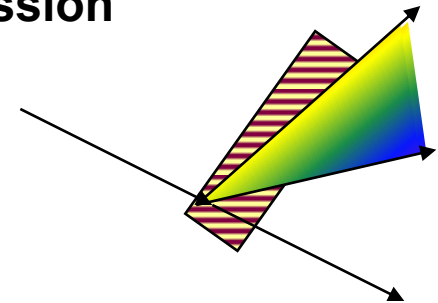
# Novel X-ray optics are based on thick multilayers



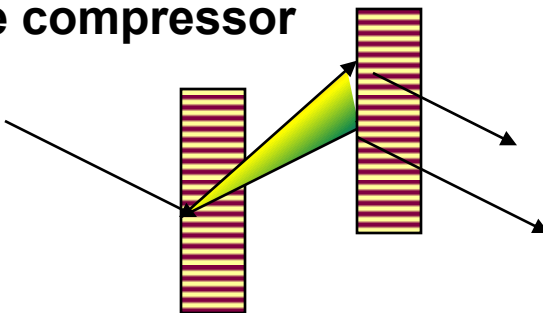
**Efficient blazed grating**



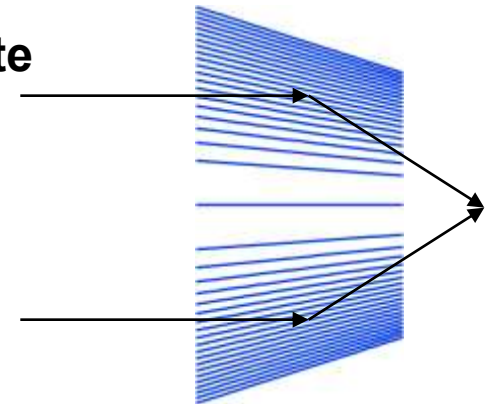
**Highly-dispersive transmission grating**



**Pulse compressor**



**Volume zone plate**



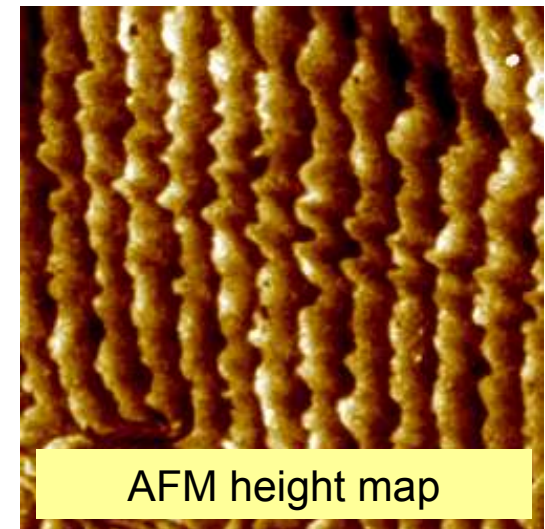
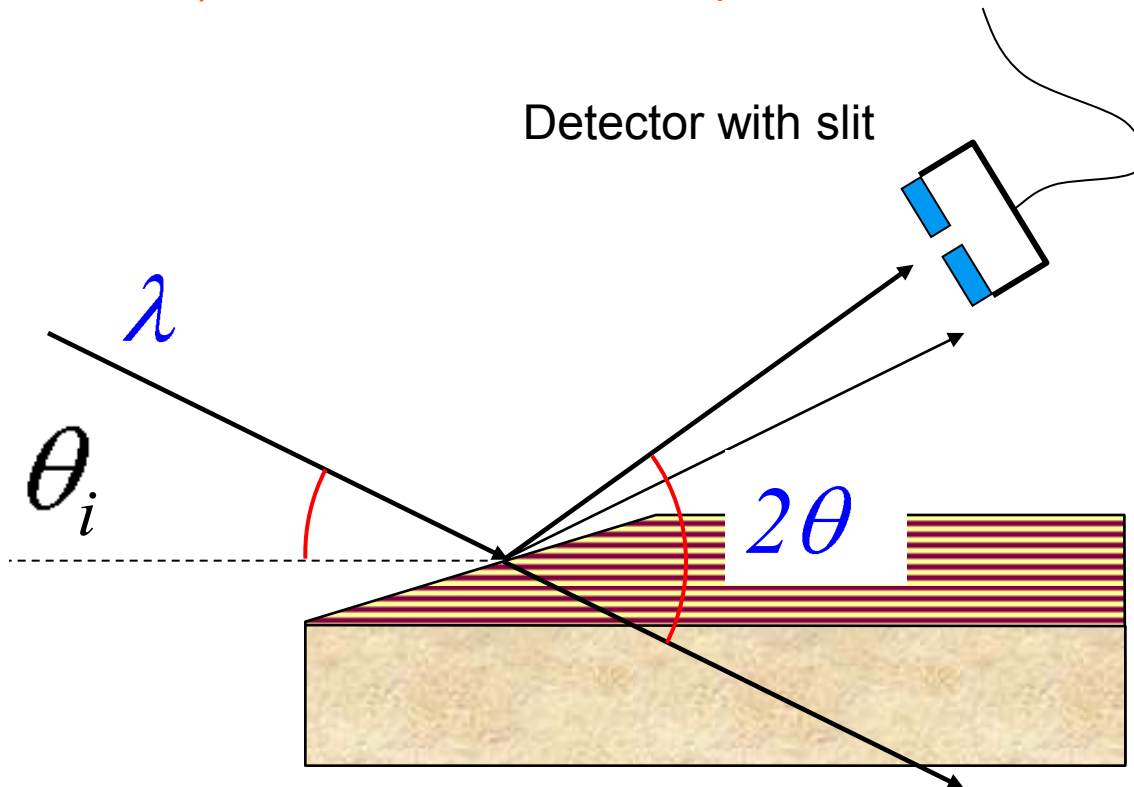
## Challenges:

- Deposition of very thick ( $>50\mu\text{m}$ ) multilayers
- Characterization, sectioning, mounting, alignment
- Theoretical understanding

# Our first thick multilayer consisted of 5000 layers and was tested at ALS



~17.5  $\mu\text{m}$  thick ML with 7 nm period

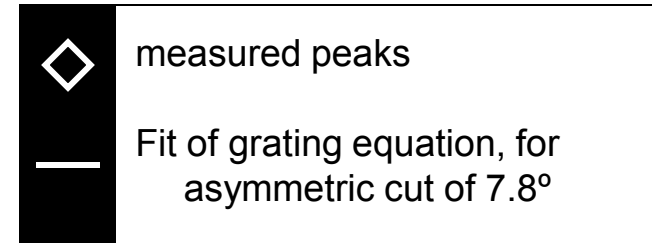
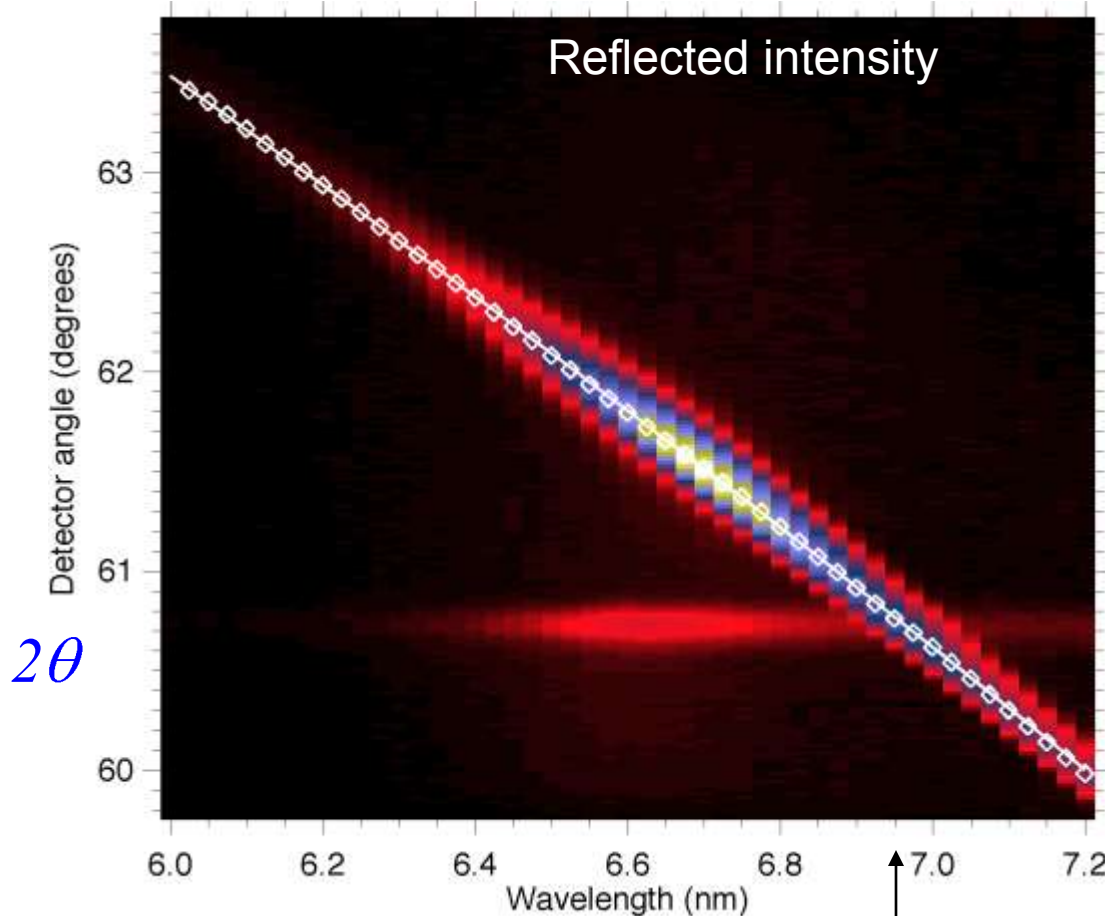


The incidence angle  $\theta_i$  was determined relative to multilayer planes, not the surface normal of the grating.

We measured dependence of intensity on  $\lambda$  and  $\theta$



# The dispersion of highly efficient grating matches theoretical prediction



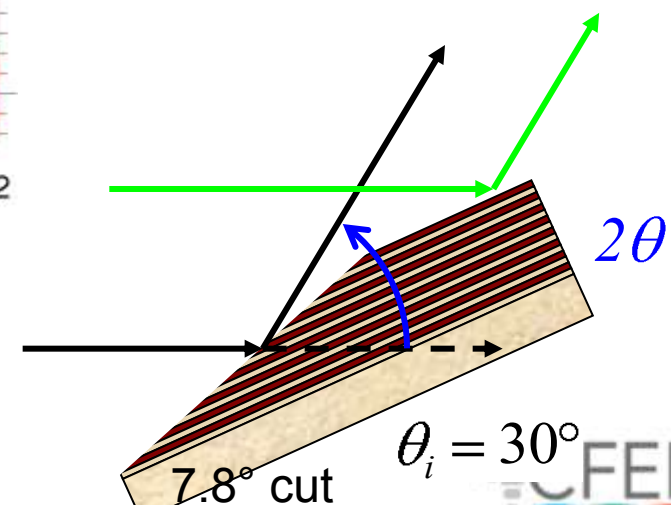
$2\theta$

$d = 6.9 \text{ nm}$   
 Cut angle =  $7.8^\circ$   
 Equivalent to 20,000 lp/mm

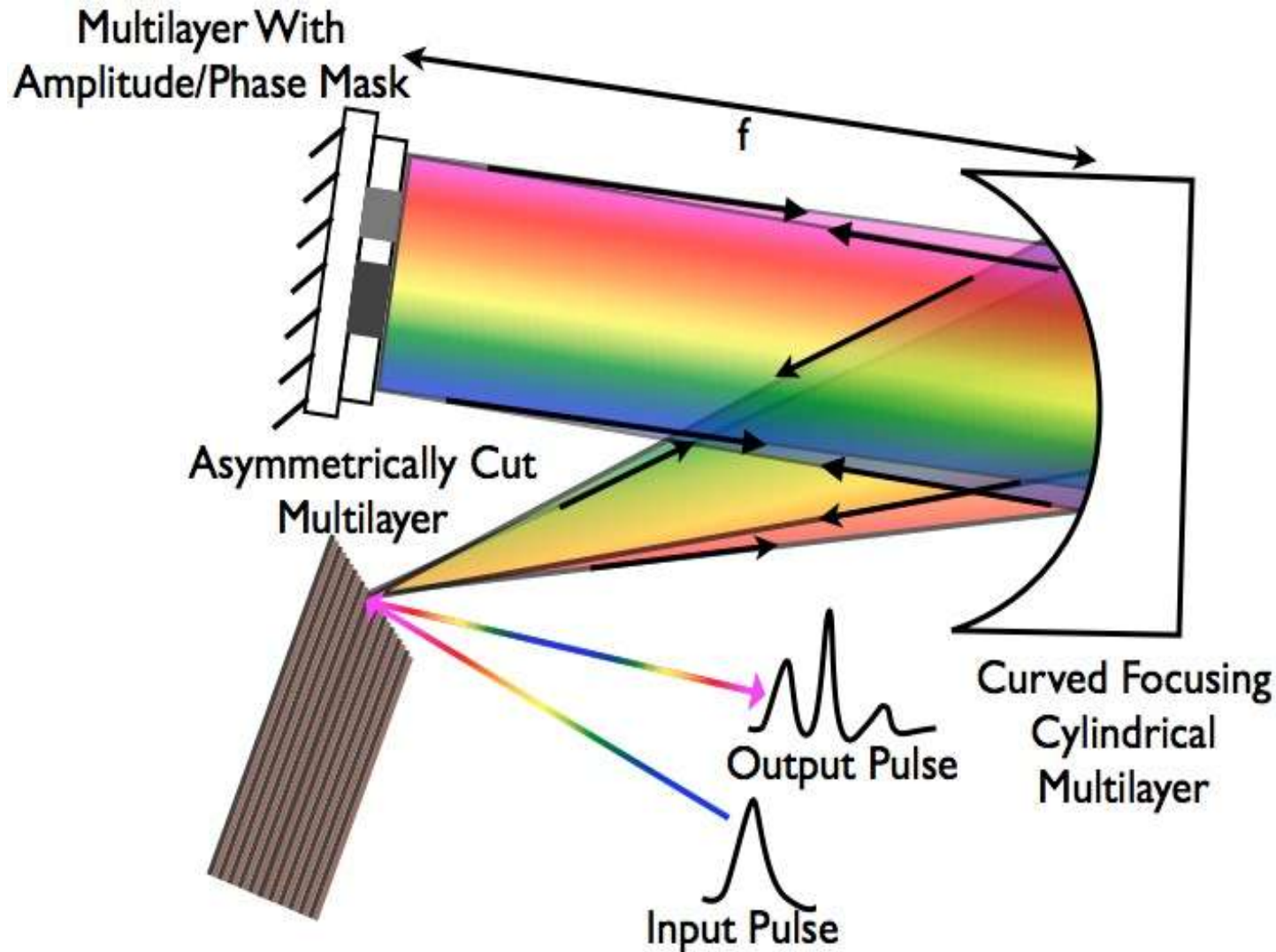
$$\lambda = 2d \sin \theta_B$$

$(\Delta\lambda = 0)$

$(\Delta\theta = 0)$   
 $2\theta = 2\theta_B$

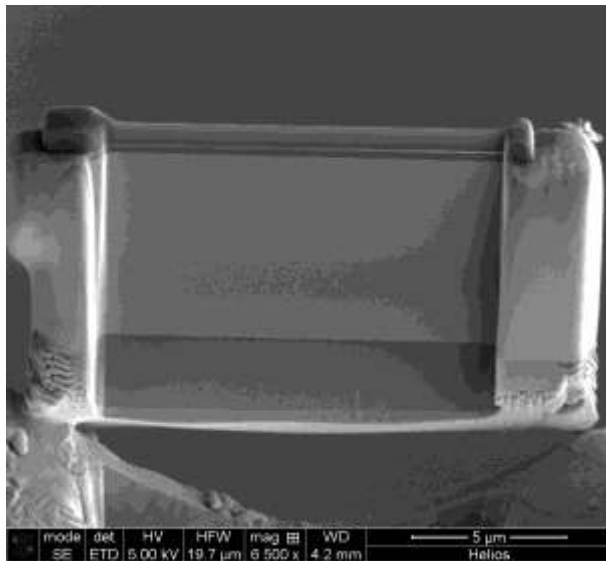
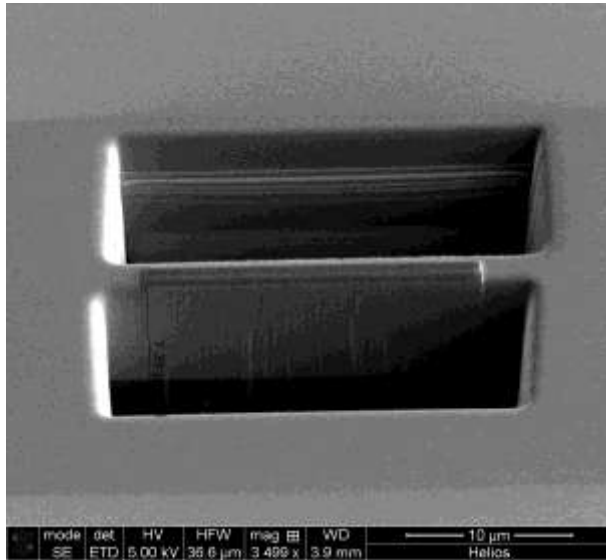


# We will pulse shape FEL (FLASH) beam using multilayer coated optics



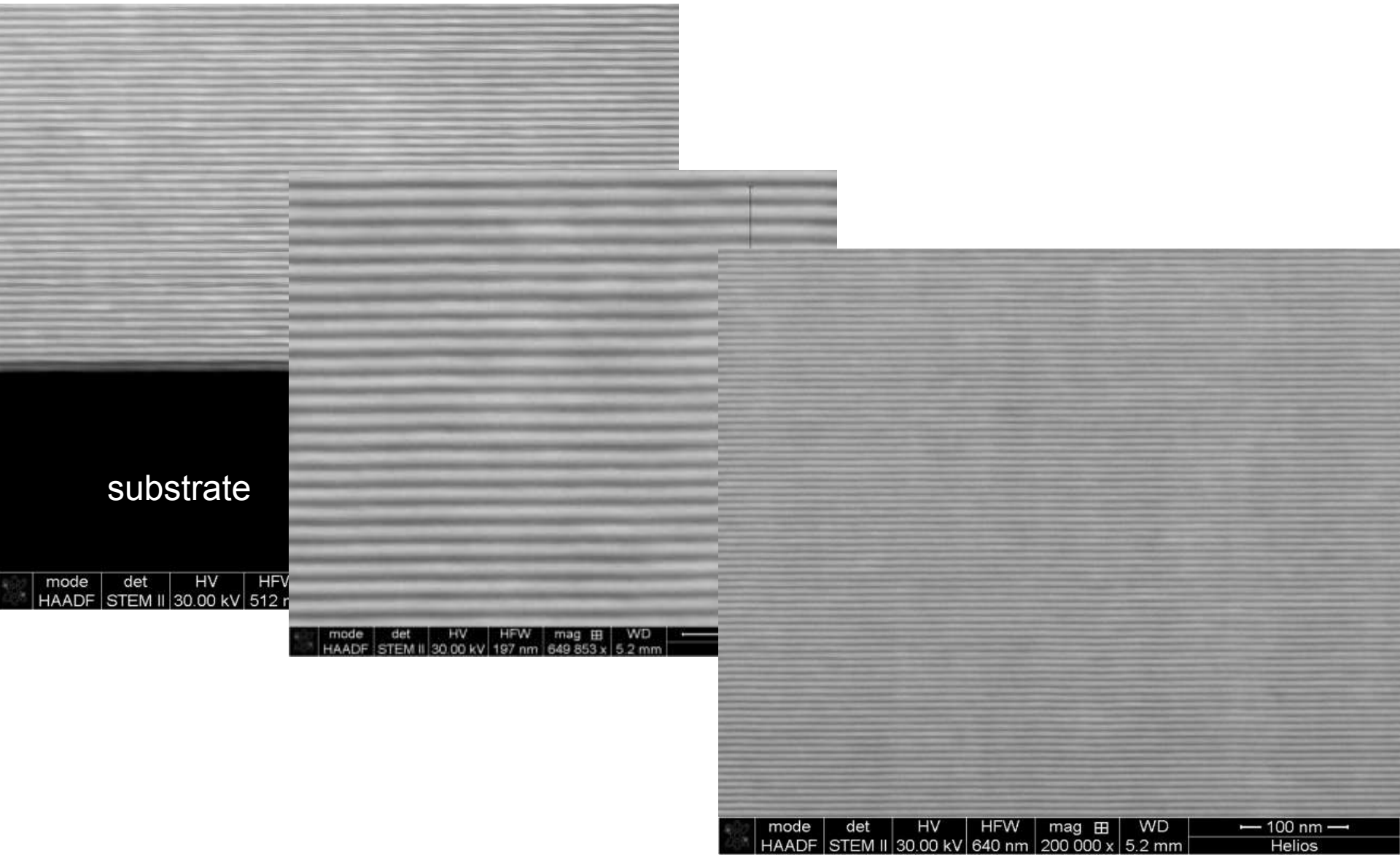
A. Aquila, M. Drescher, T. Laumann, M. Barthelmess, H. N. Chapman, and S. Bajt,  
International Journal of Optics, Vol. 2011, Article ID 417075

# We use dual beam FIB for imaging, patterning and etching nanostructures including multilayers



Lamellas prepared and imaged by Miriam Barthelmeß

# Cross section images of a 2000 layer thick multilayer show uniform thick layers and smooth interfaces



- State-of-the art sputtering deposition tools for “conventional” and thick multilayer optics
- Other equipment for characterizing and manipulating nano-layered and nanostructures
- Normal incidence optic for soft x-ray regime
- Narrow bandwidth optics
- Development of volume optics based on thick multilayers (gratings, pulse compressors, volume zone plates)
- Novel X-ray optics can be used to manipulate the phase space of X-rays, especially for PETRA III and XFEL beams

This type of optics will have impact in:

- Bio-imaging
- Materials and environmental sciences
- X-ray astronomy

# Acknowledgements



A. Aquila\*, M. Barthelmeß, M. Prasciolu (DESY)  
J. Schulz\*, H. Fleckenstein, L. Gumprecht, *H. N. Chapman (CFEL)*  
*B. Nagler (LCLS)*  
*S. Vinko (Oxford Univ.)*  
A. Parsons (Univ. of Southampton)  
*T. Burian, V. Hájková, J. Chalupský, L. Juha (Academy of Sciences of the Czech Republic)*  
*R. Treusch, S. Toleikis and FLASH team (DESY)*  
*C. Laubis, C. Buchholz, F. Scholze (PTB)*  
*E. Gullikson (ALS)*  
*F. Siewert, F. Schäfers (BESSY II)*  
A. Berg, T. Burmester, T. Delmas, H. Mahn (DESY)

*\* Now at European XFEL*

