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Ultra-short-period WC/SiC multilayer coatings for x-ray applications

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Finn Christensen, Anders Jakobsen (DTU, Denmark)

4th International Workshop on Metrology for X-ray Optics, Mirror Design, and Fabrication, Barcelona, July 5th, 2012

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Presentation overview

- Introduction to LLNL work and facilities
- Motivation
- Multilayer development and substrate surface metrology
- Measurements and fittings at 8 keV (DTU) and 62, 186 keV (NSLS)
- Measurements, fittings and Compton scattering simulations at 378 keV (ESRF)

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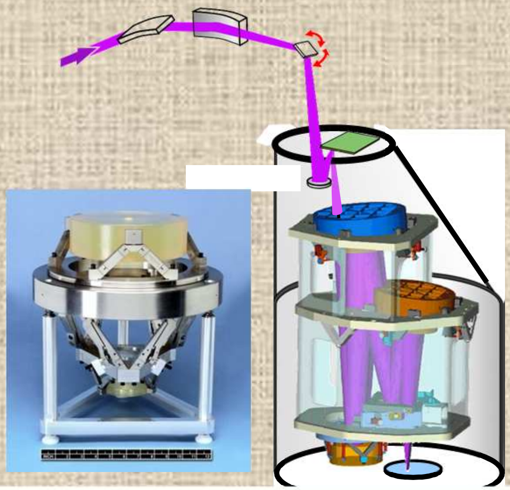
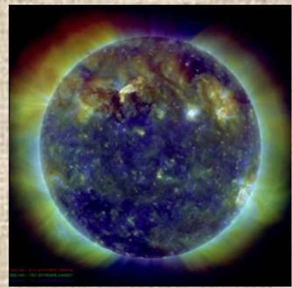
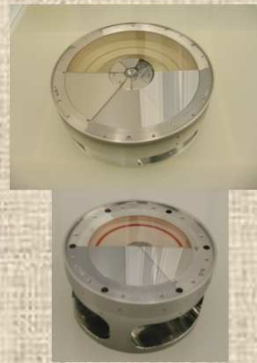
Our group at LLNL has participated in the development of optical elements for a wide range of applications for the EUV, soft and hard x-rays

EUV Lithography



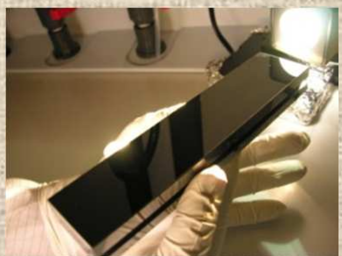
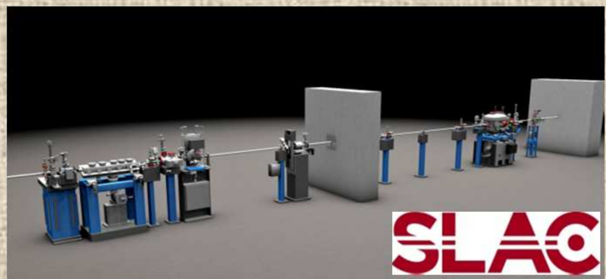
R. Soufli *et al*., *Proc. SPIE* 4343, 51 (2001)
R. Soufli *et al*., *Appl. Opt.* 46, 3736 (2007)

EUV space missions (NASA's SDO and NASA/NOAA's GOES-R)

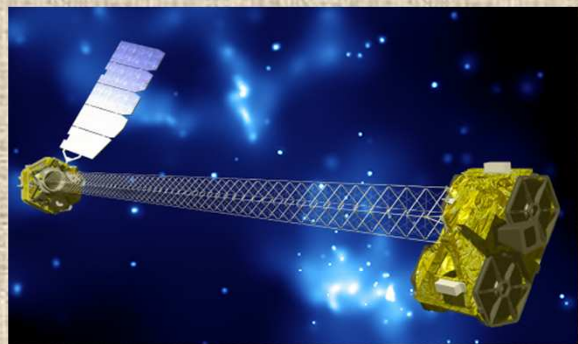
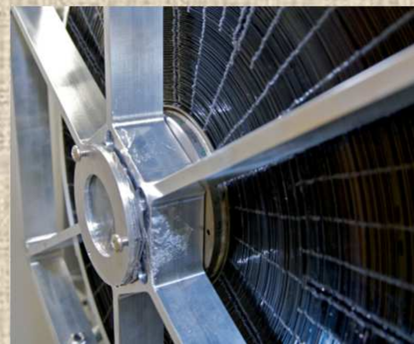

R. Soufli, *et al*., Appl. Opt. 46, 3156-3163 (2007)
R. Soufli, *et al*., Proc. SPIE 5901, 59010M (2005)
P. Boerner *et al*, Solar Physics 275, 41-66 (2012).
J. R. Lemen *et al*, Solar Physics 275, 17-40 (2012).

Soft and hard x-ray facilities and beamlines (LCLS)

R. Soufli, M. Fernandez-Perea, *et al*.,
Appl. Opt. 51, 2118 (2012)

Hard x-ray space missions (NASA's NuSTAR)

C. J. Hailey *et al. Proc. SPIE* 7732, 77320T (2010)
F. A. Harrison *et al. Proc. SPIE* 7732, 77320S (2010)

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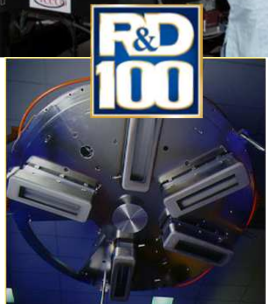
LLNL experimental facilities for coating deposition and surface metrology

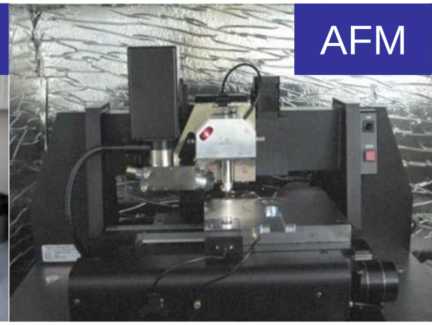
DC- and RF-sputtering multilayer deposition systems



Precision surface metrology

ZYGO

AFM



SEM

Also (not pictured):

- o Contact profilometers
- o Thin film stress measurement apparatus
- o Full-aperture interferometry

Custom cleaning facility for optical substrates

X-Ray Diffractometer

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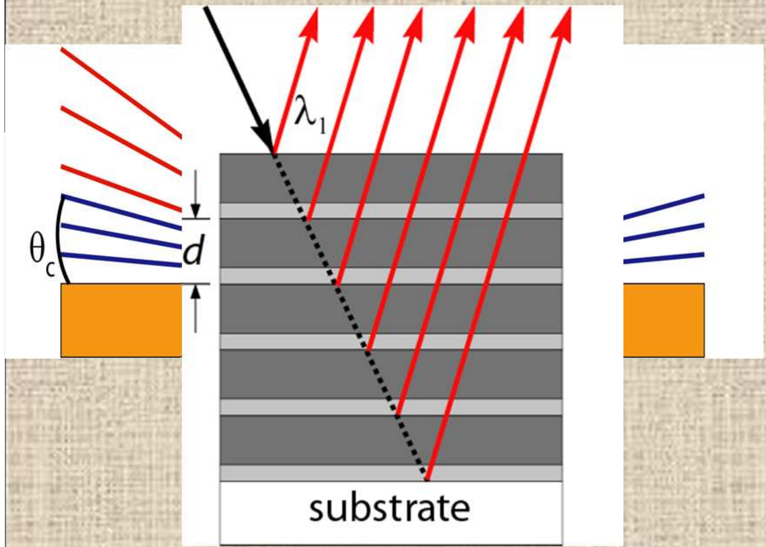
How high in photon energy can we push the use of multilayer coatings?



- Total reflection on simple interfaces can be used to efficiently reflect x-rays
- At large energies, θ_c is very small (e.g. θ_c (WC) ~ 2 mdeg @ 40 keV)
- Since the early 1970s (E. Spiller) multilayer use has been extended well into the hard x-ray regime

$$m\lambda \cong 2d \sin\theta \quad \text{(Bragg's law)}$$

Limitations as photon energy (E) increases ($\lambda\downarrow$):

1) Smaller *d* is needed for reflection at the same $\theta \rightarrow$ d approaches limit of continuous layer formation (~ 1 nm). Diffusion and roughness at interfaces become crucial.

2) For even higher energies, θ also has to be reduced $\rightarrow$ dramatic reduction of collecting area and tighter figure requirements

3) Additional interactions take place (e.g., elastic and inelastic scattering)

Our goal: demonstrate multilayer operation up to 400 keV

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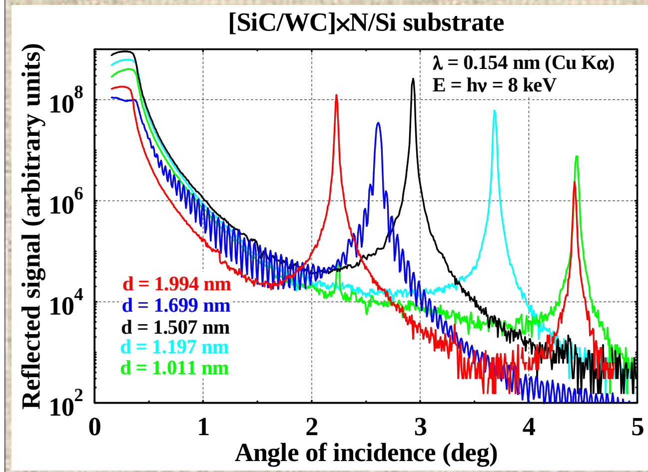
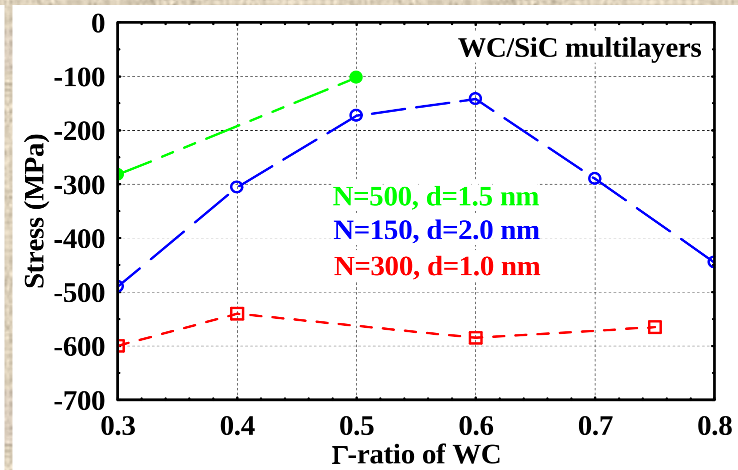
WC/SiC multilayer development on Si wafer substrates

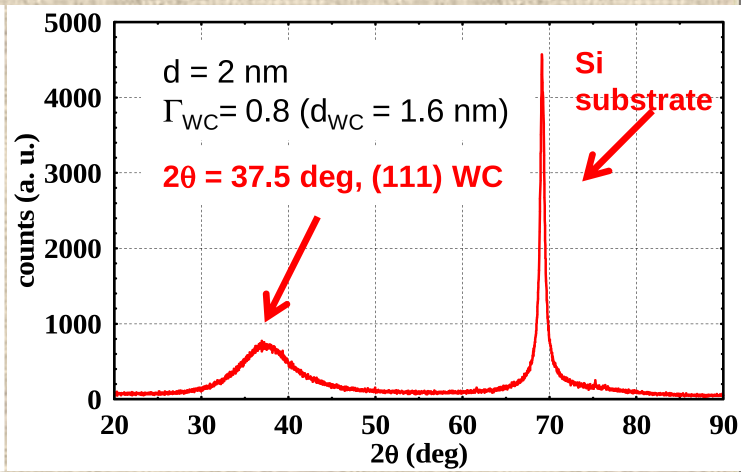
Period calibration via XRD

Stress characterization as a function of period and Γ-ratio = d_{WC} / d

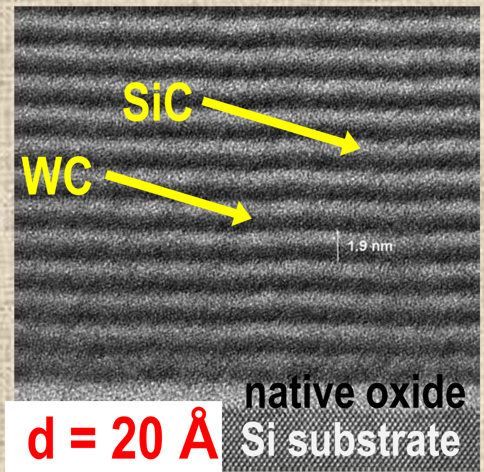
LAXRD

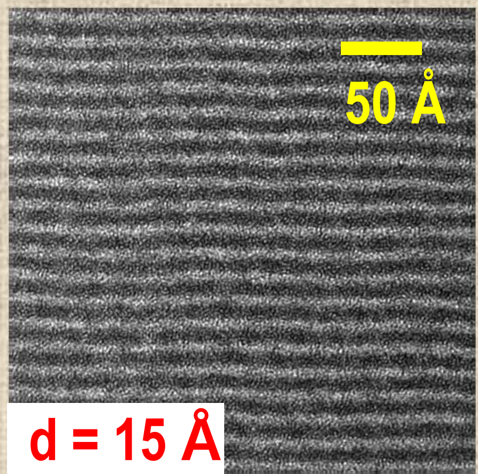


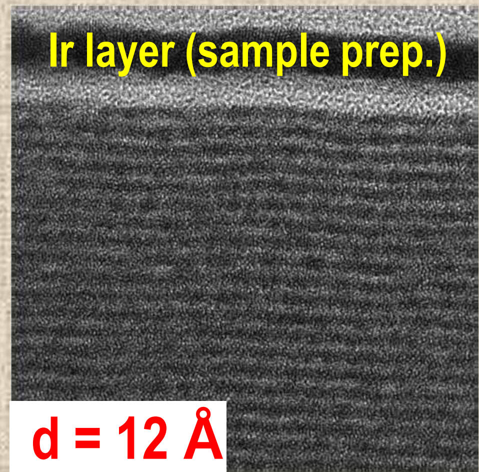


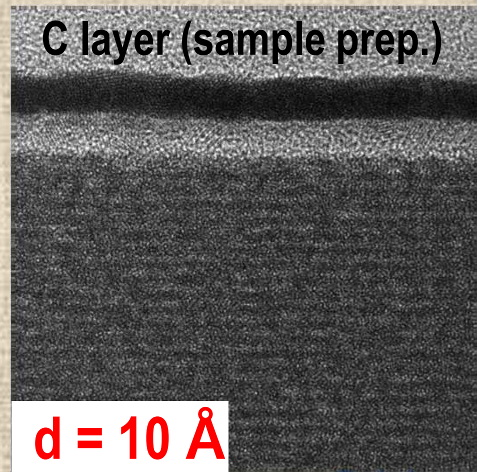


Cross-sectional TEM









* TEM measurements performed at EAG Labs, Sunnyvale (CA)

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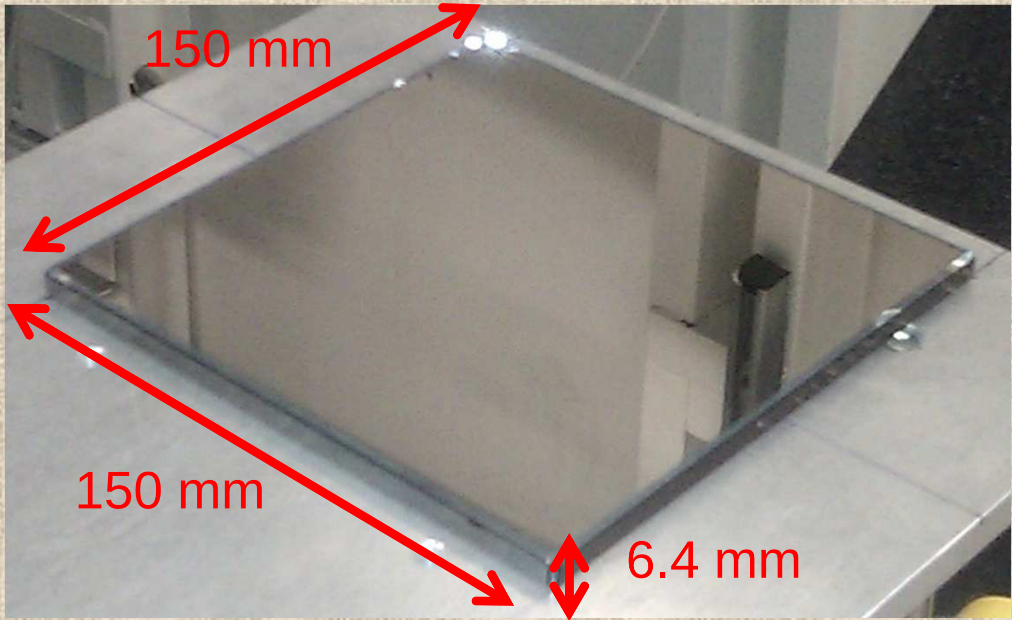
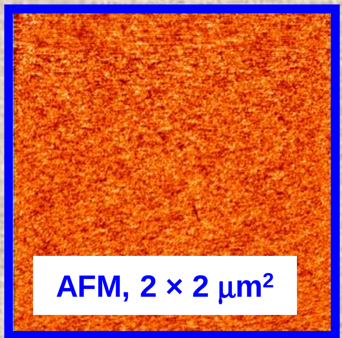
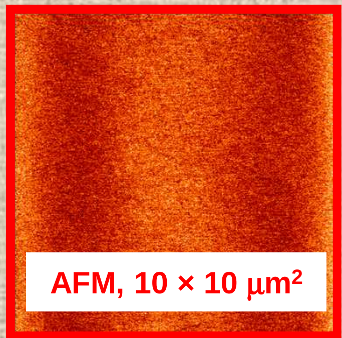
Si wafer substrates are not optimal for high energy/grazing incidence measurements because of large figure errors

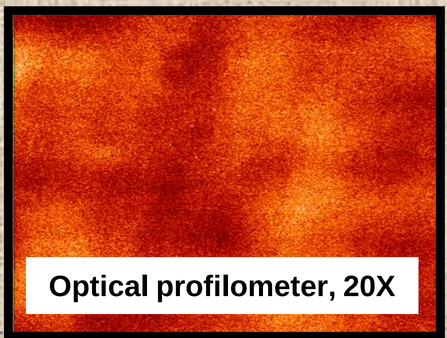


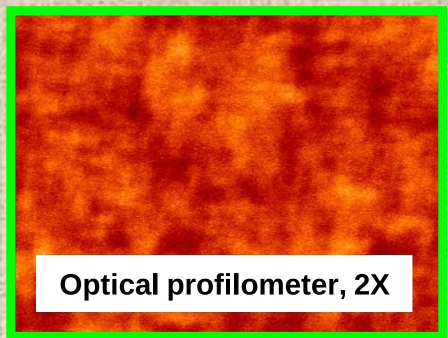
- Super-polished EUV lithography mask blanks made of fused silica were selected as substrates based on their extremely <u>smooth and flat surface</u>.

- Slope error ~ 1 μrad

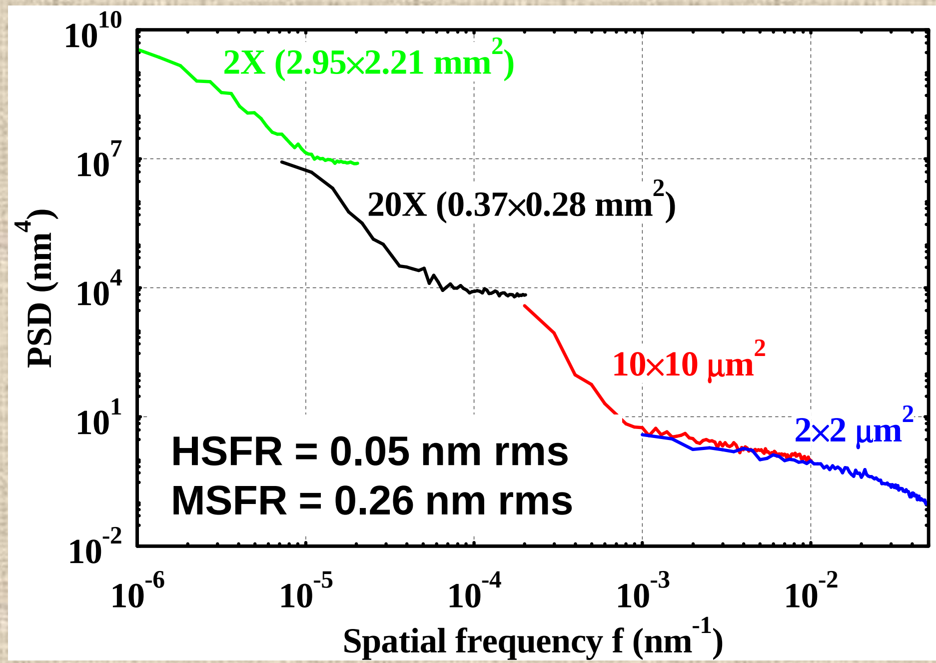








UNCOATED SUBSTRATE



$$\sigma^2 = \int_{f_1}^{f_2} 2\pi f S(f) df$$

where $S(f) \equiv$ 2D PSD (nm^4), $f_1 = 5 \times 10^{-4}$ nm^{-1}, $f_2 = 0.05$ nm^{-1} for HSFR and $f_1 = 10^{-6}$ nm^{-1}, $f_2 = 5 \times 10^{-4}$ nm^{-1} for MSFR

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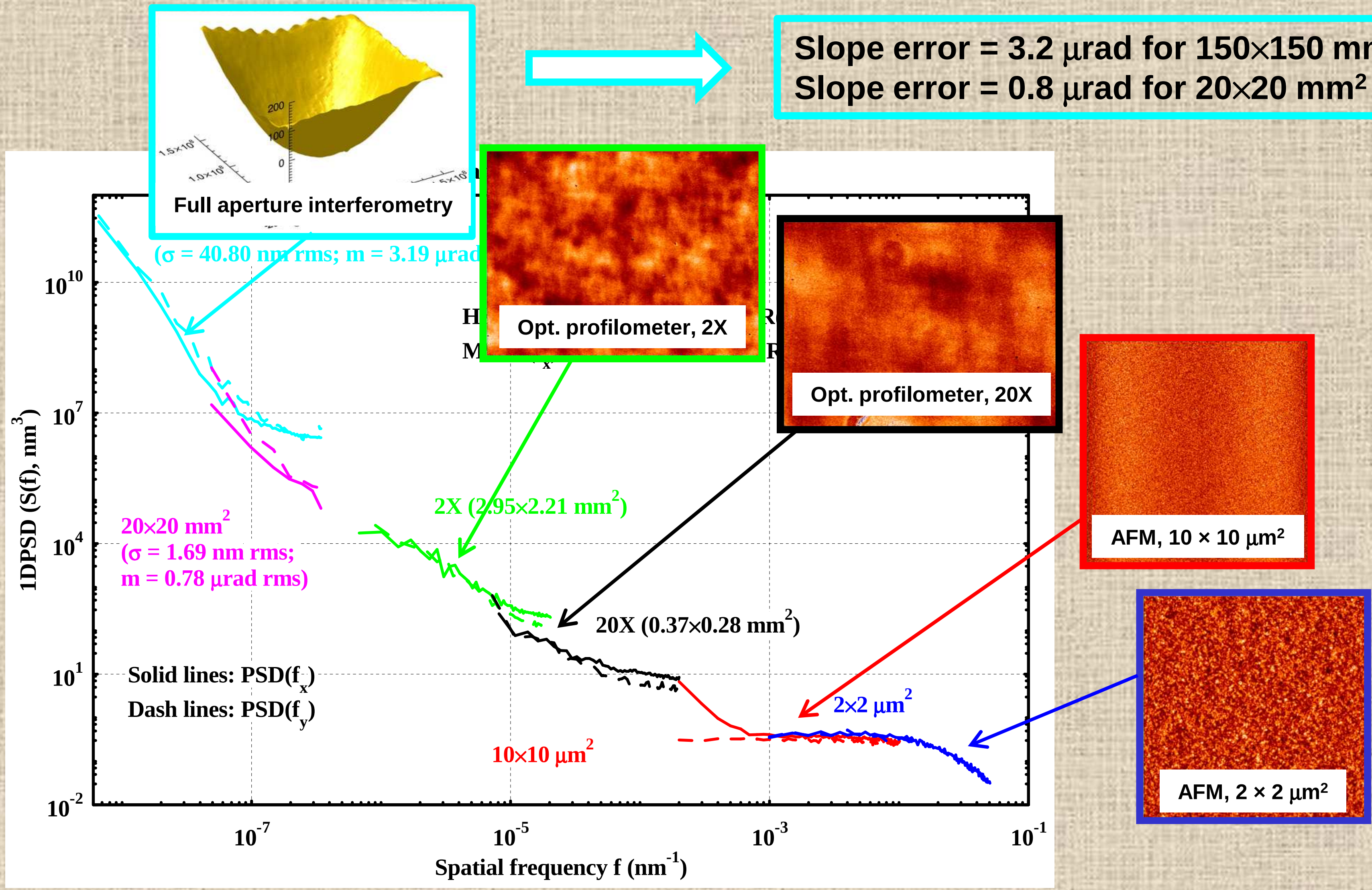
Precision surface metrology on coated mirrors confirms required figure

Slope error = 3.2 µrad for 150×150 mm²
Slope error = 0.8 µrad for 20×20 mm²



$$\sigma^2 = 2\int_{f_1}^{f_2} S_{1D}(f)\,df \qquad m^2 = 2\int_{f_1}^{f_2} (2\pi f)^2 S_{1D}(f)\,df$$

where $S(f) \equiv$ 2D PSD (nm⁴), $f_1 = 5 \times 10^{-4}$ nm⁻¹, $f_2 = 0.05$ nm⁻¹ for HSFR and $f_1 = 10^{-6}$ nm⁻¹, $f_2 = 5 \times 10^{-4}$ nm⁻¹ for MSFR

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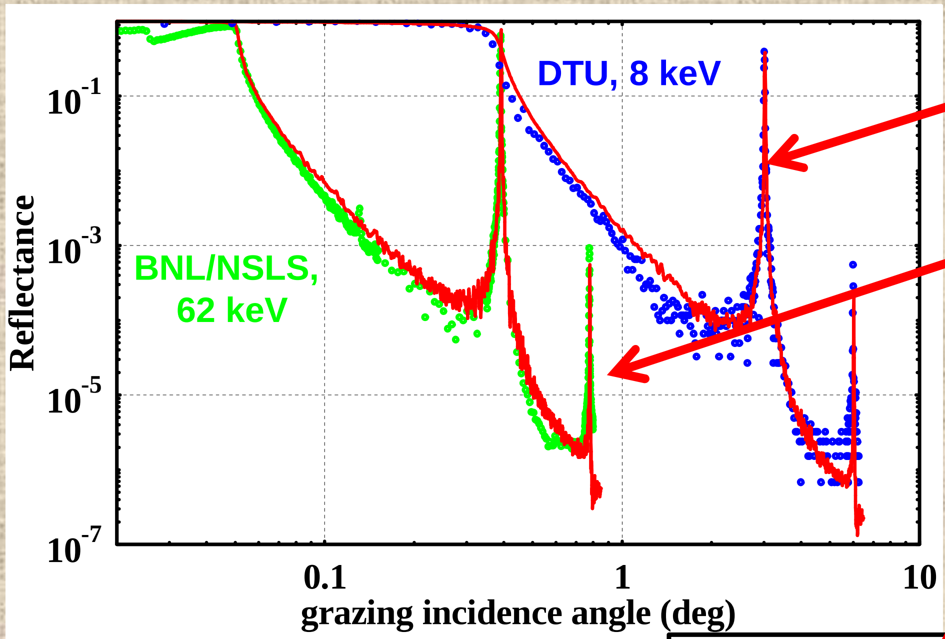
Multilayer model developed by fitting 8 (DTU) and 62 (BNL) keV measurements

Simultaneous fit at 8 and 62 keV



<u>IMD model*</u>

[SiC/WC]×300/SiO_2 substrate

ML period = 1.47 nm

Γ_{WC} = d(WC)/period = 0.44

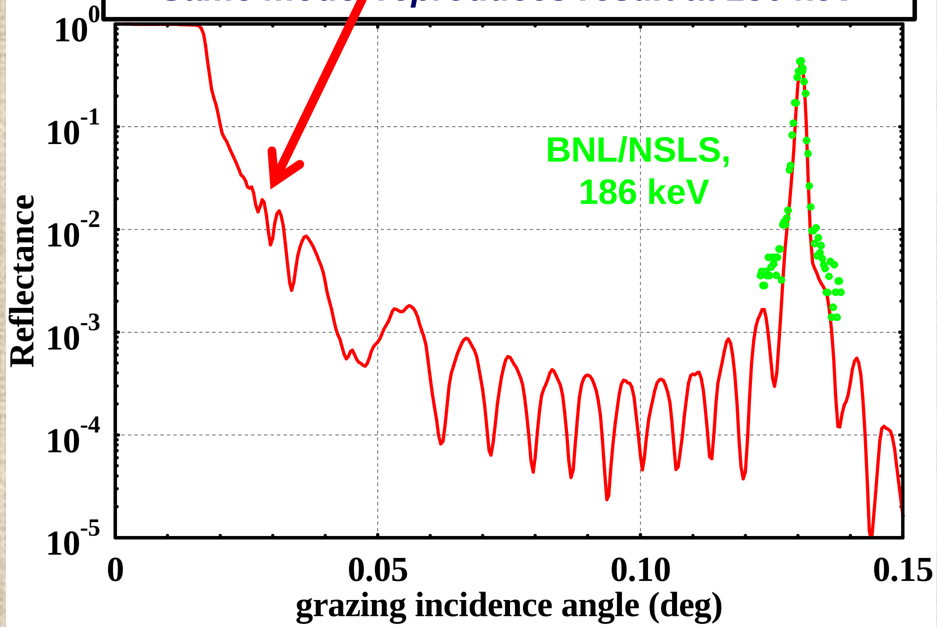
σ= 0.26 nm

SiC and WC optical constants from NIST FFAST database**

* IMD modeling software by D. L. Windt

**http://www.nist.gov/pml/data/ffast/index.cfm

Same model reproduces result at 186 keV



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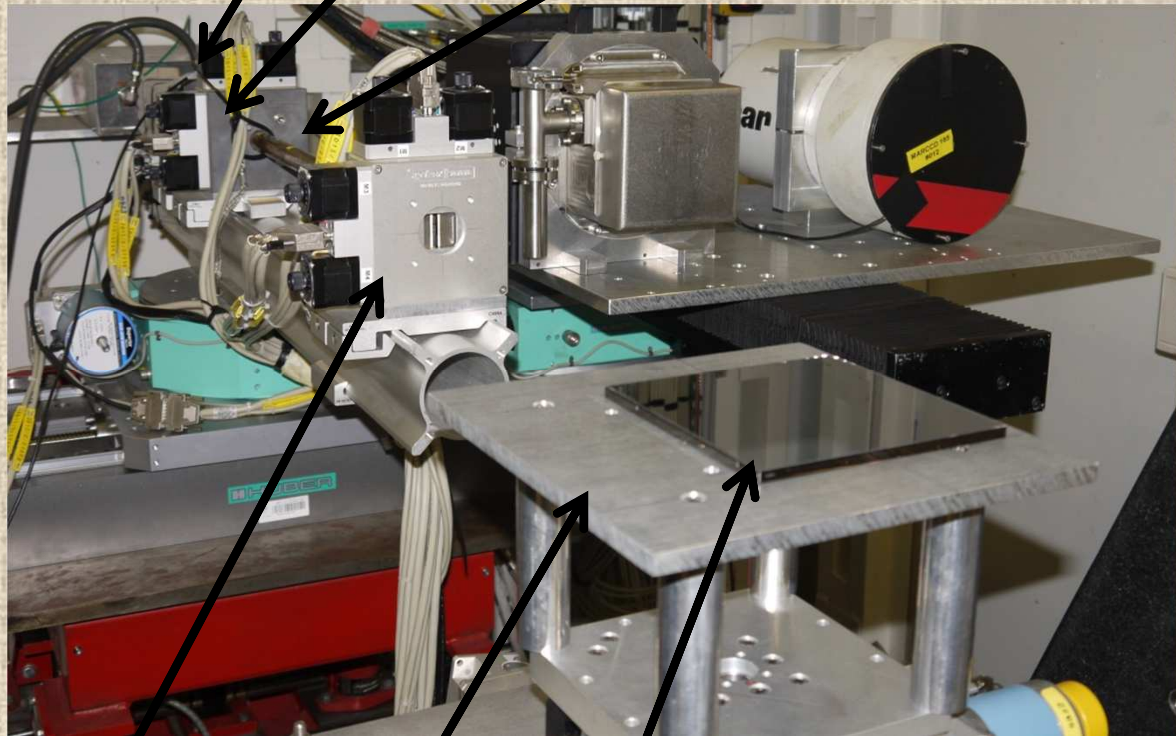
The same samples were measured at ID15A beamline at ESRF (378 keV)

High **E**nergy **M**icro**D**iffraction (HEMD) endstation at ID15A

*tilt monochromator for the measurement of liquid samples was not used in this experiment.



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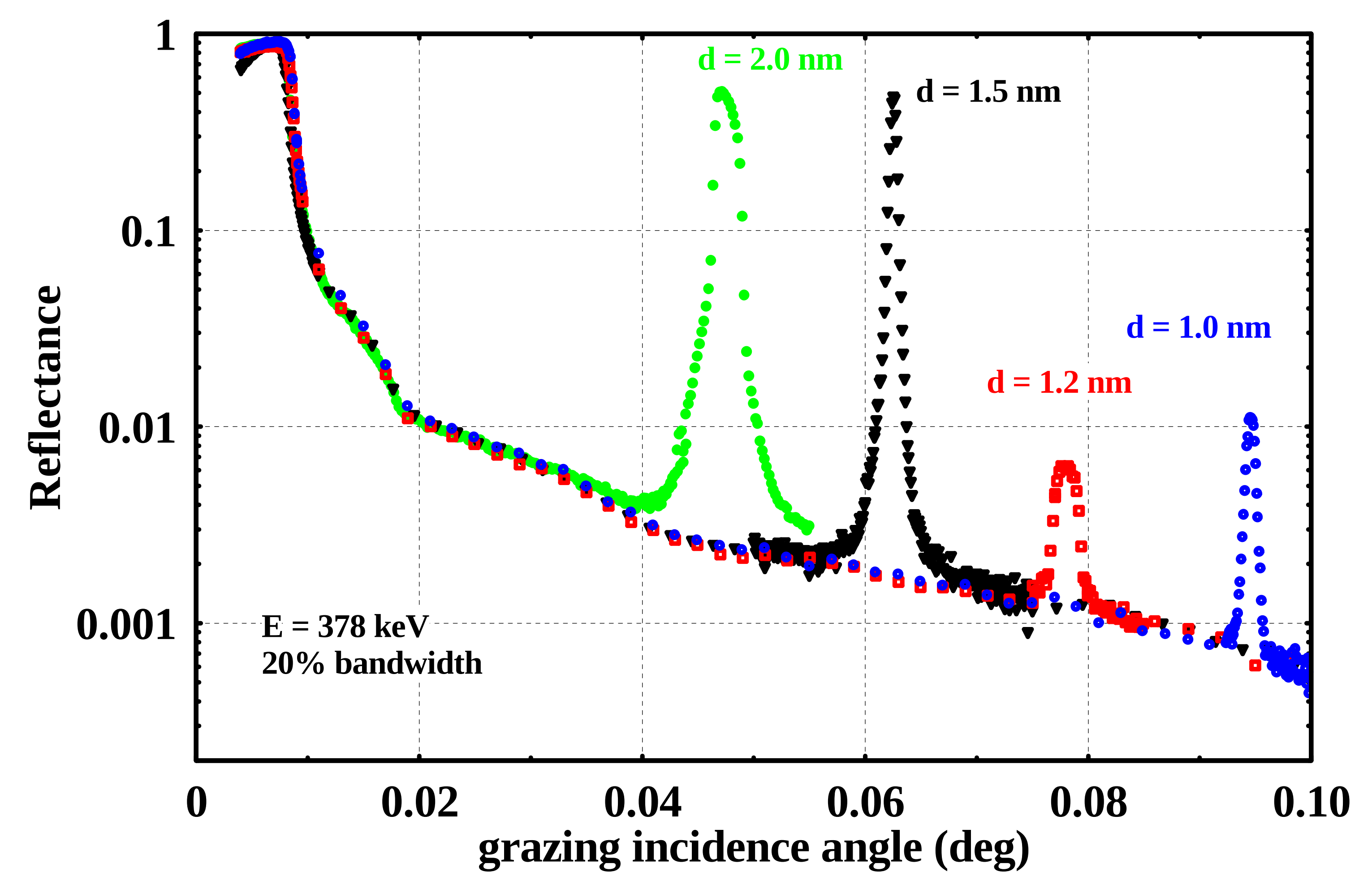
We successfully demonstrated operation of four multilayer mirrors at 378 keV



period (nm)	θ_{Bragg} (mdeg)	θ_{Bragg} (mrad)	R (%)
2.0	48	0.84	50.4
1.5	62	1.08	48.0
1.2	78	1.36	0.6
1.0	95	1.66	1.1

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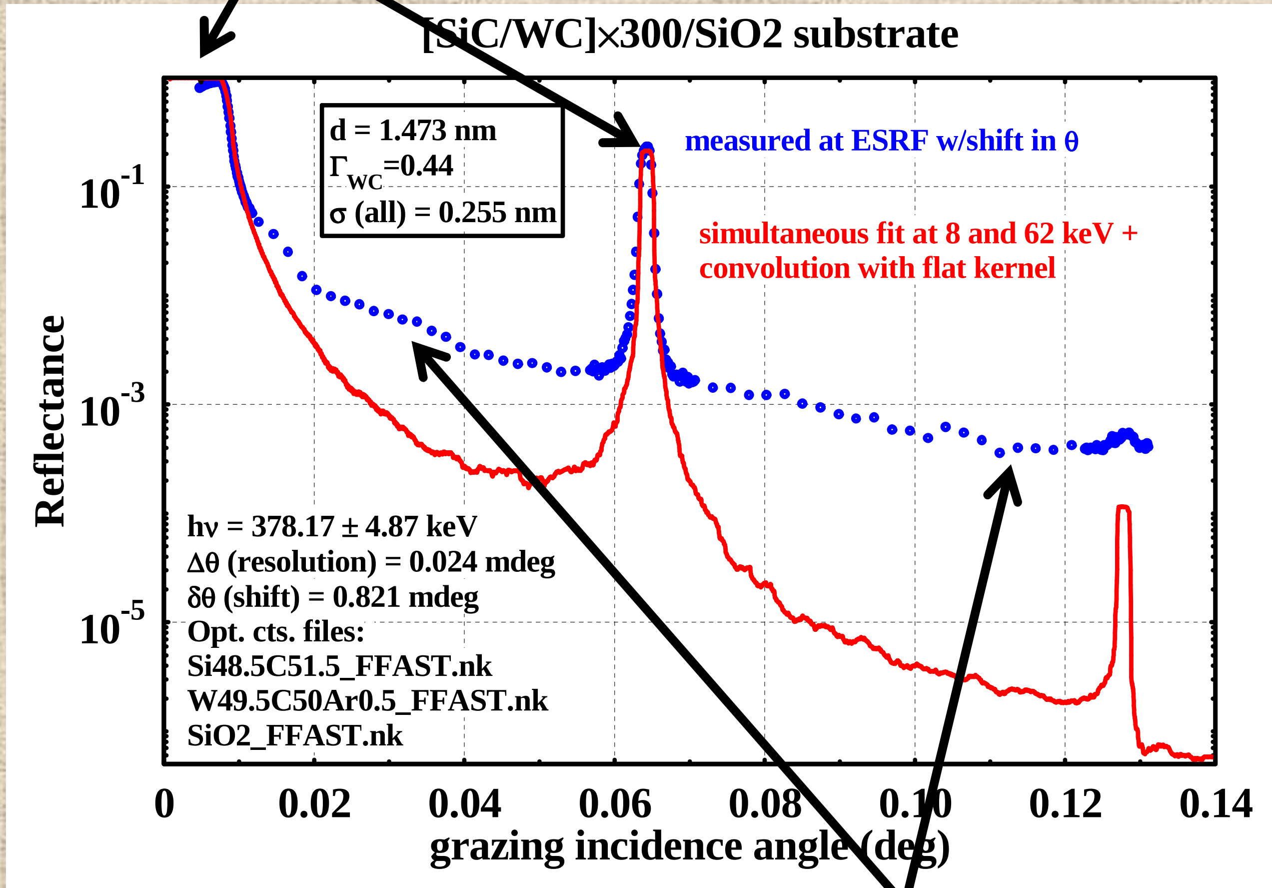
… but the modeled performance did not agree with the measured signal outside of the Bragg peak

IMD model developed for lower energies works well at critical angle and 1st Bragg peak



IMD model does not predict high inter-peak levels. **Hypothesis**: Compton scattering contribution.

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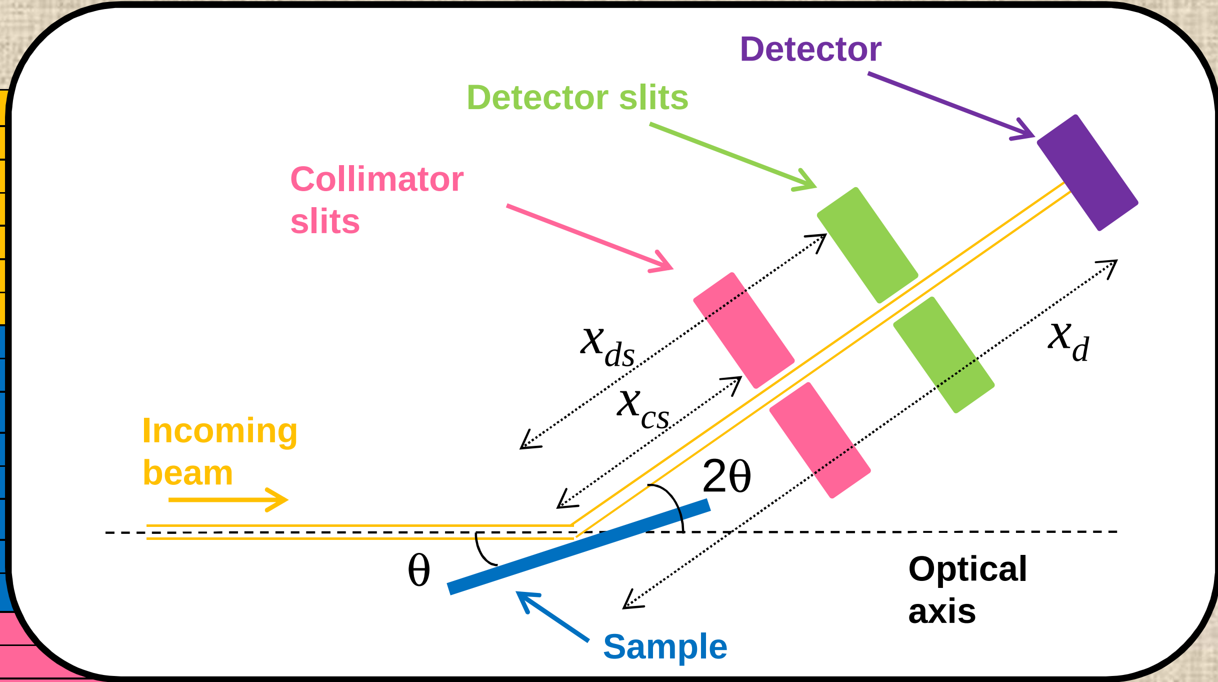
Precise knowledge of beamline components is essential for experiment modeling

Source	Distance to sample	7.3 m
Beam	Width	2.5 mm
	Height (sample out)	11 microns
	Divergence	0.024 mdeg
	Energy	378.17 keV
	Bandwidth	9.74 keV
	Spatial distribution	gaussian
Sample, substrate	Size	150x150x6.4 mm^3
	Composition	SiO2
	Density	2.203 gcm^-3
Sample, multilayer	Composition (RBS)	W99Si97C203Ar
	Thickness	450 nm
	Density	8.108 gcm^-3
Stage	Al plate thickness	15 mm
	Air gap below Al plate	100 mm
Collimator slits	Model	IB-C30-AIR from JJ X-ray
	Blade thickness	1 cm
	Distance to sample	0.59 m
	Vertical gap (csvg)	0.4 mm, asymmetric
	Horizontal gap (cshg)	2.5 mm
	Composition	WF20 tungsten carbide, 86.4-87.2% WC, 11.5-12.5% Co, 1.1-1.3% VC+Cr3C2
	Cross section	16 mm-radius curved edge
Detector slits	Model	IB-C30-AIR from JJ X-ray
	Blade thickness	1 cm
	Distance to sample	1.32 m
	Vertical gap (dsvg)	0.1 mm
	Horizontal gap (dshg)	2.5 mm
	Composition	WF20 tungsten carbide, 86.4-87.2% WC, 11.5-12.5% Co, 1.1-1.3% VC+Cr3C2
	Cross section	0.5 deg knife edge
Detector	Distance to sample	1.36 m
	Thickness	5 mm
	Composition	NaI



Precise knowledge of beamline components (e.g., composition, dimensions, locations) is essential for quantitative assessment of multilayer performance

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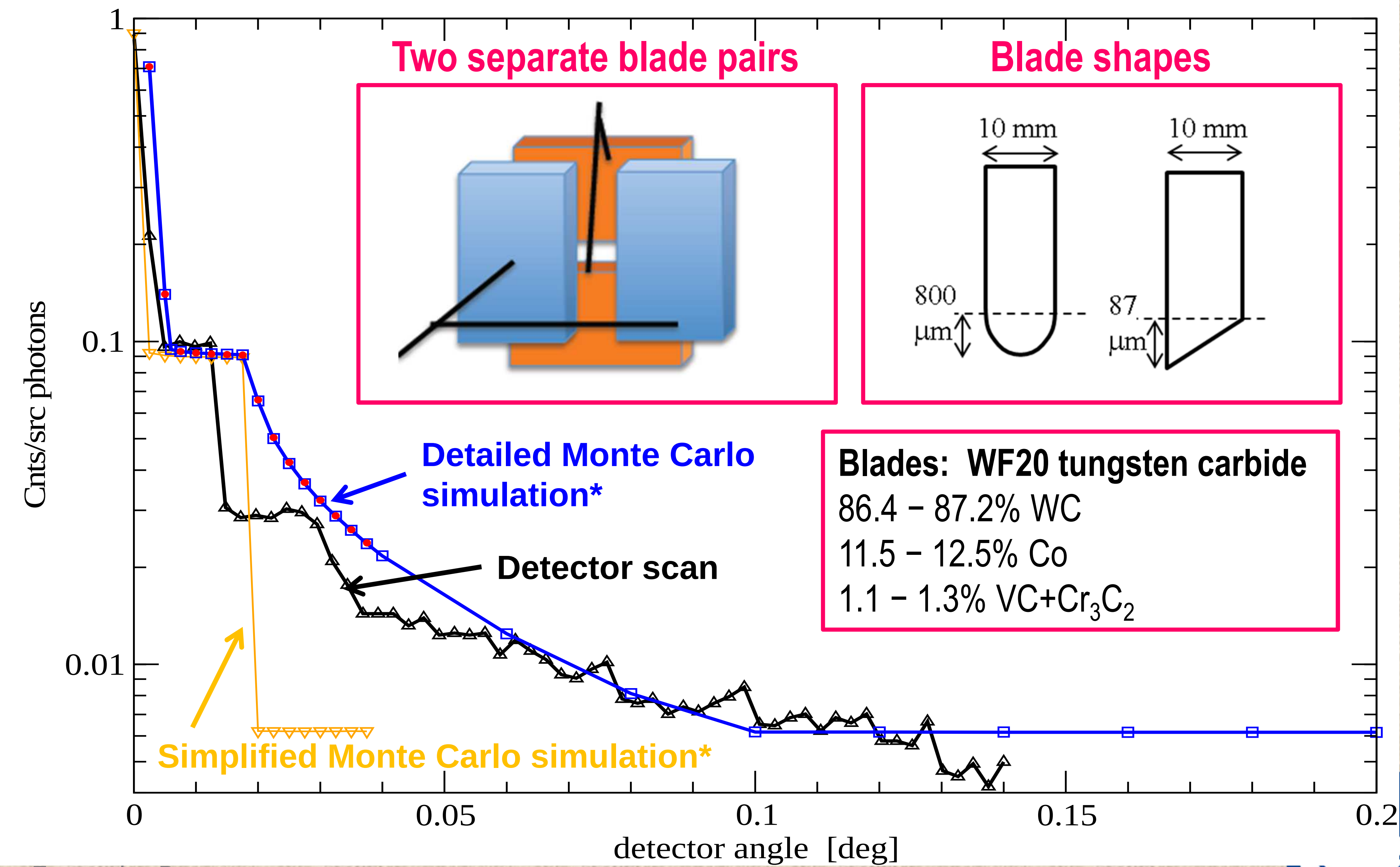
Slit geometry and composition were crucial for accurate Monte Carlo transport simulations of Compton scattering contribution

Background measurements were used to refine experimental setup model



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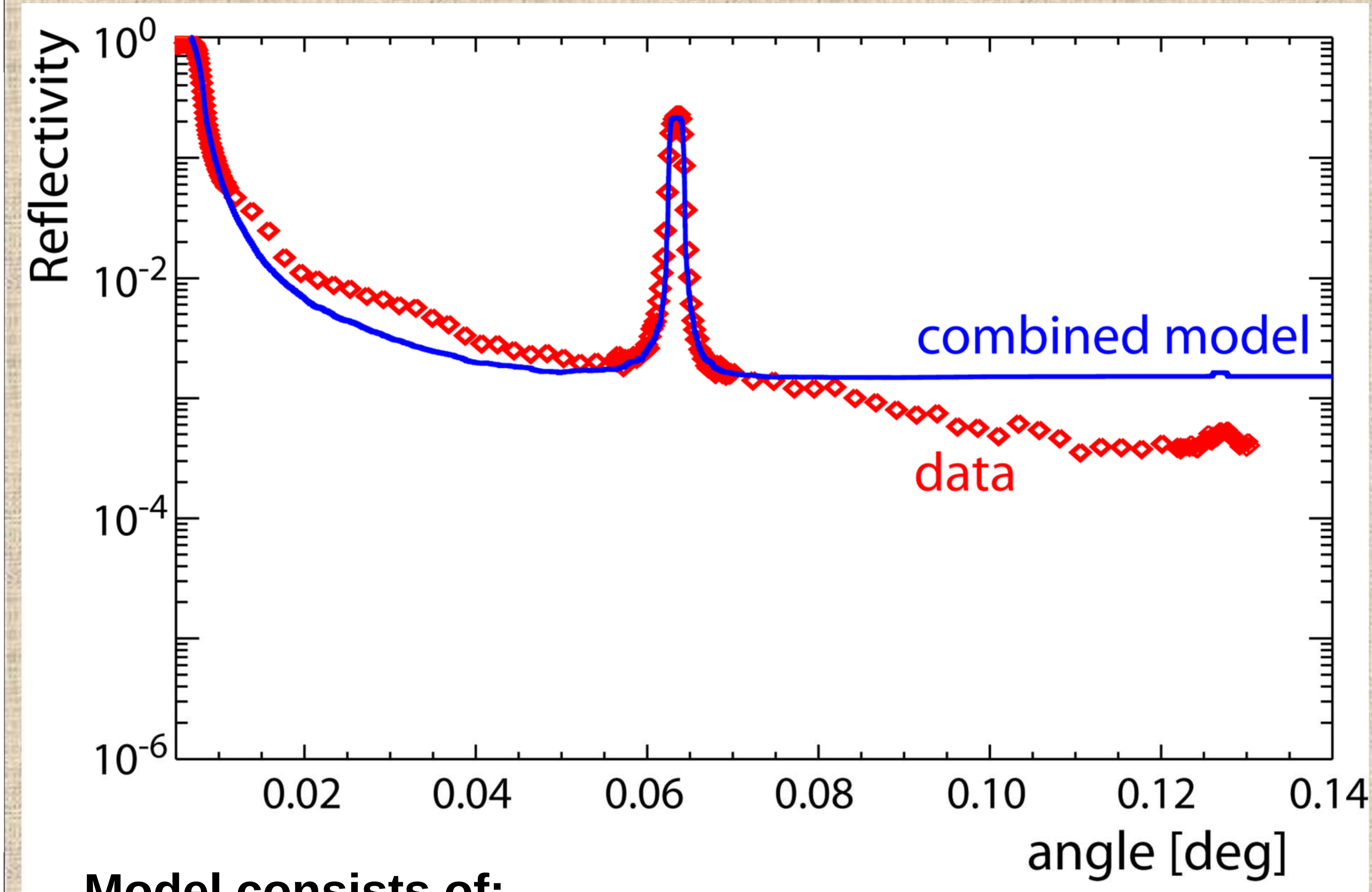
*Calculations performed with MCNP code by Los Alamos National Lab

Incorporation of Compton scattering in our model successfully reproduces measured data



parameters

E_0 = 378 keV
ΔE = 10 keV
N = 300
d = 14.7 Å
Γ = 0.44
σ = 2.6 Å

Model consists of:

1. Wave: constructive-interference reflectivity, derived from fits at 8 & 62 keV
2. Particle: Monte-carlo derived Compton scattering

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Conclusions

- We have demonstrated that multilayer coatings can be used at photon energies as high as 378 keV

- Selection of appropriate material pair with low interface diffusion and smooth and flat substrate

- Reflectance w nt experimental f that reproduced k ical-angle fall-off) d

- A Compton sc keV measuremen

- The Compton scattering contribution was modeled with a Monte Carlo simulation, providing a good match between model and measurement

- Multilayer coatings could be used in beamline components, space telescopes, radioisotope detection systems and other applications at energies of ~400 keV.

Thank you for your attention!!

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