同濟大學 精密光學工程技术研究所
INSTITUTE OF PRECISION OPTICAL ENGINEERING TONGJI UNIVERSITY

Nanoscale Multilayers Optics for EUV and X-ray Applications

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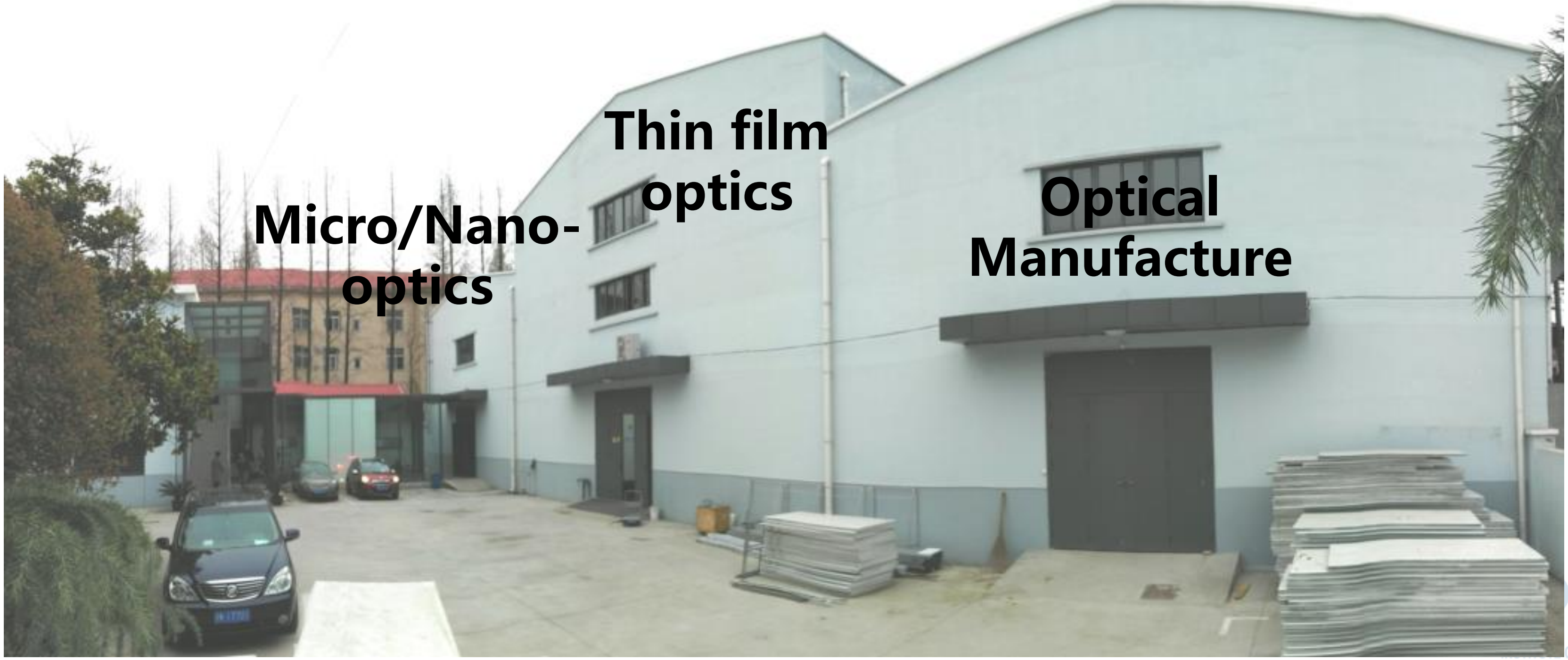
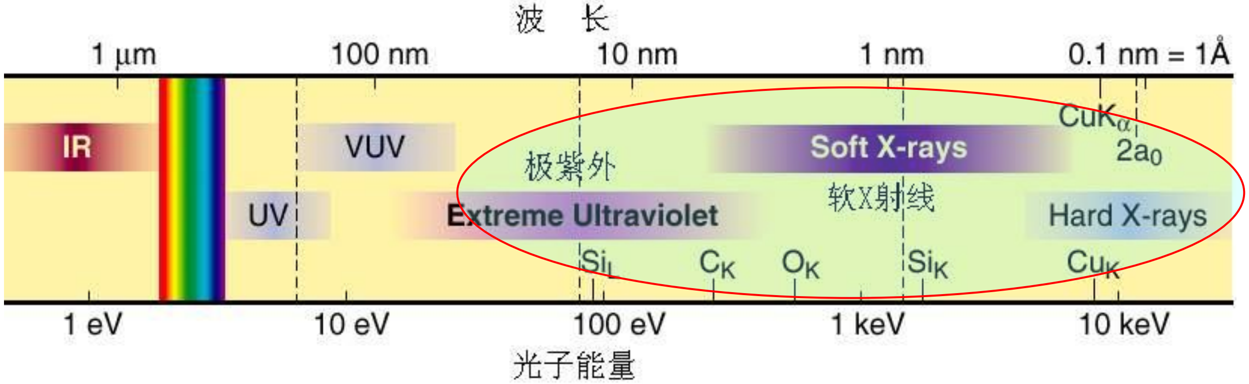
2019.1 New lab in Huxi campus (Shanghai)



EUV & X-ray Science (XUV)

High spatial resolution

High temporal resolution



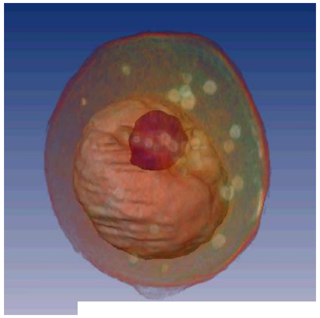
Elemental sensitivity

Large penetration depth

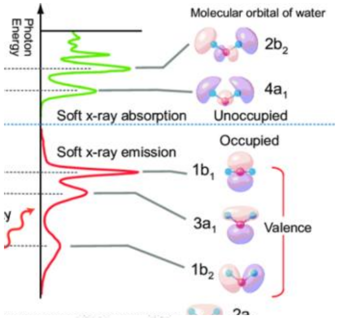
Background

EUV & X-ray Science

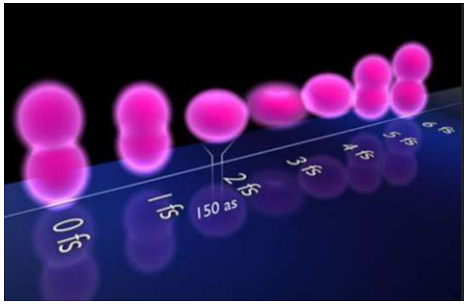
Imaging

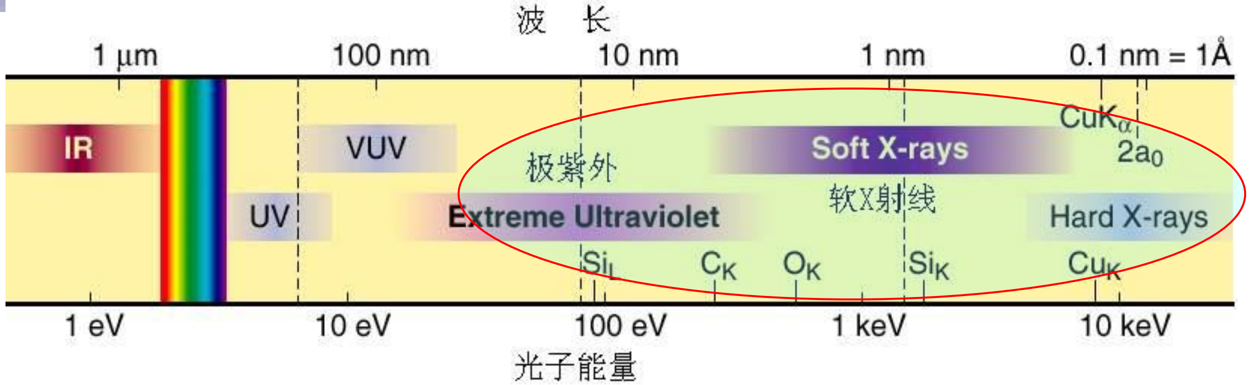


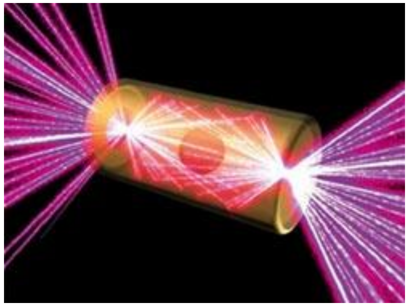
Spectroscopy



Attosecond Science







High energy/strong field observation

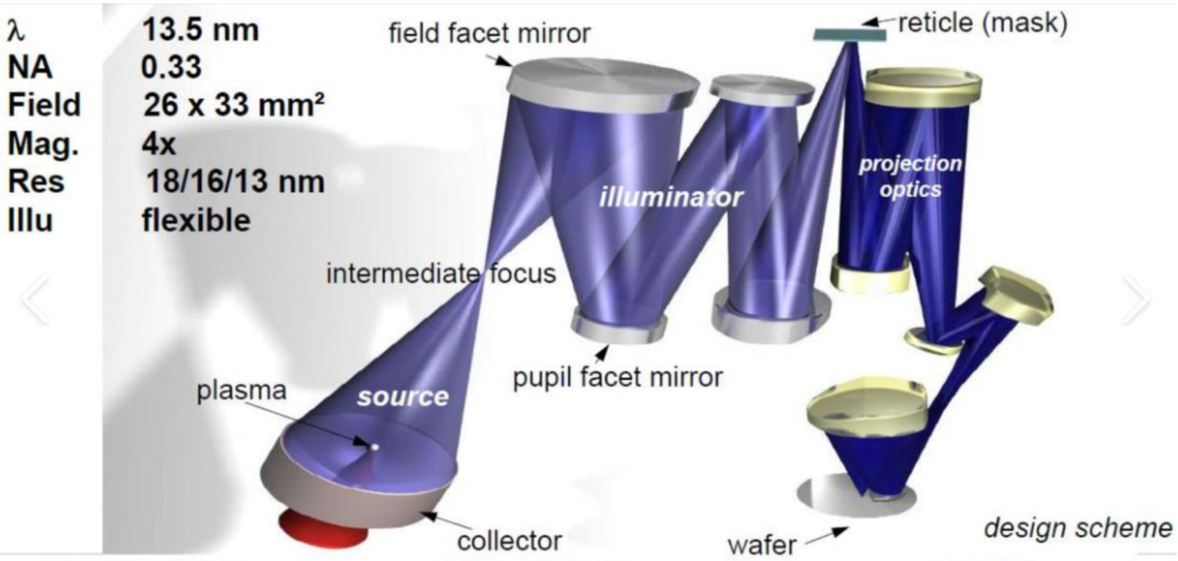
Nanometer Manufacture

Background 〉 EUV & X-ray reflective optics

Short wavelength technology require advanced reflective optics

EUVL scanner

EUV optical system
10~11 mirrors



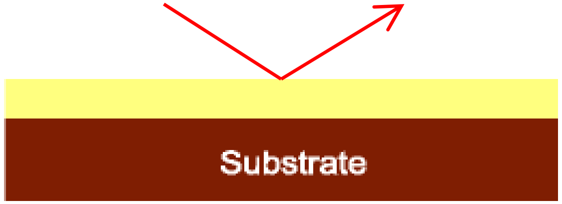
λ	13.5 nm
NA	0.33
Field	26 x 33 mm²
Mag.	4x
Res	18/16/13 nm
Illu	flexible

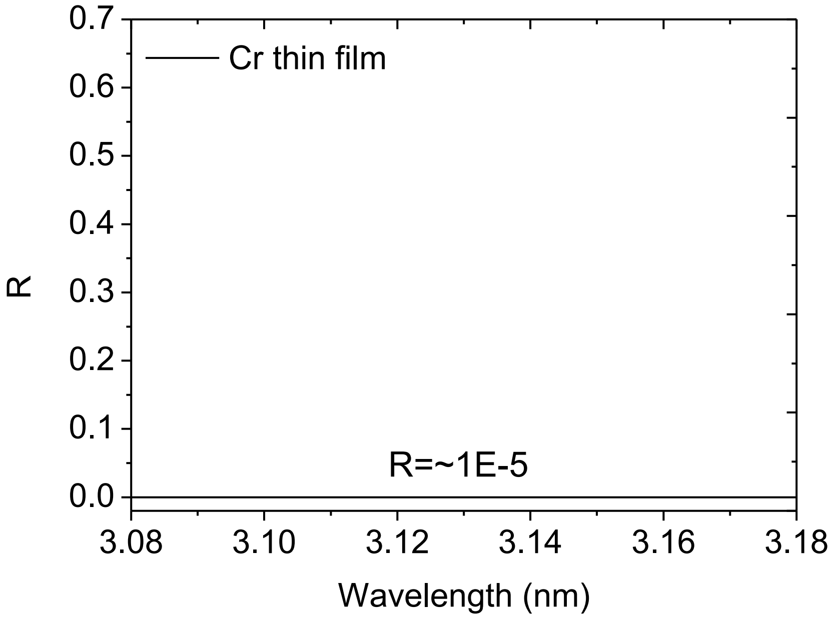
Carl Zeiss SMT GmbH, Winfried Kaiser EUVL Symposium 2015 Maastricht October 7th, 2015

Reflection of short wavelength light

$$n=1-\delta-ik$$

$\delta \rightarrow 0$, k is non-negligible



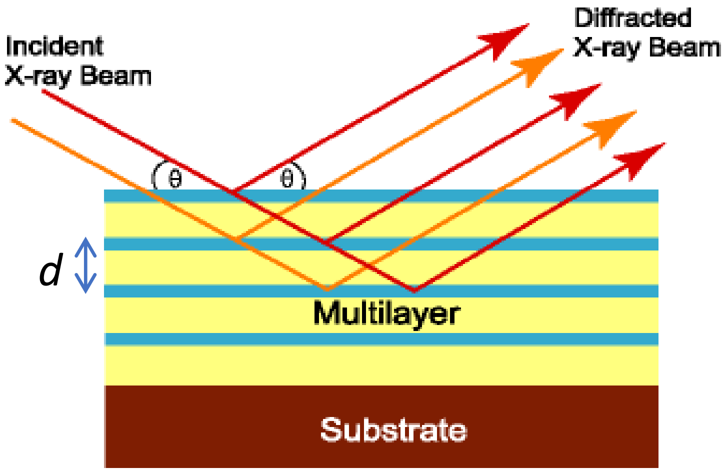


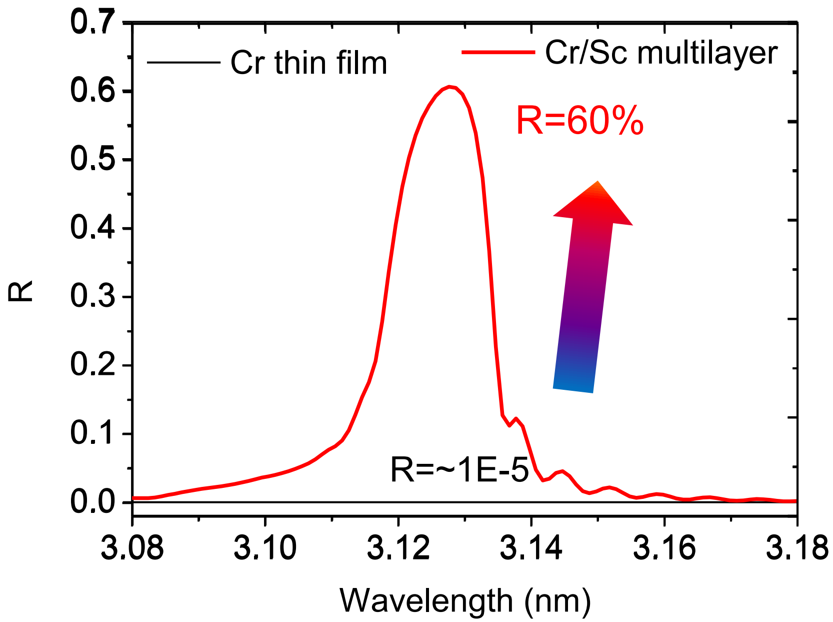
Basics

Reflection of short wavelength light

$$n=1-\delta-ik$$

$\delta \rightarrow 0$, k is non-negligible



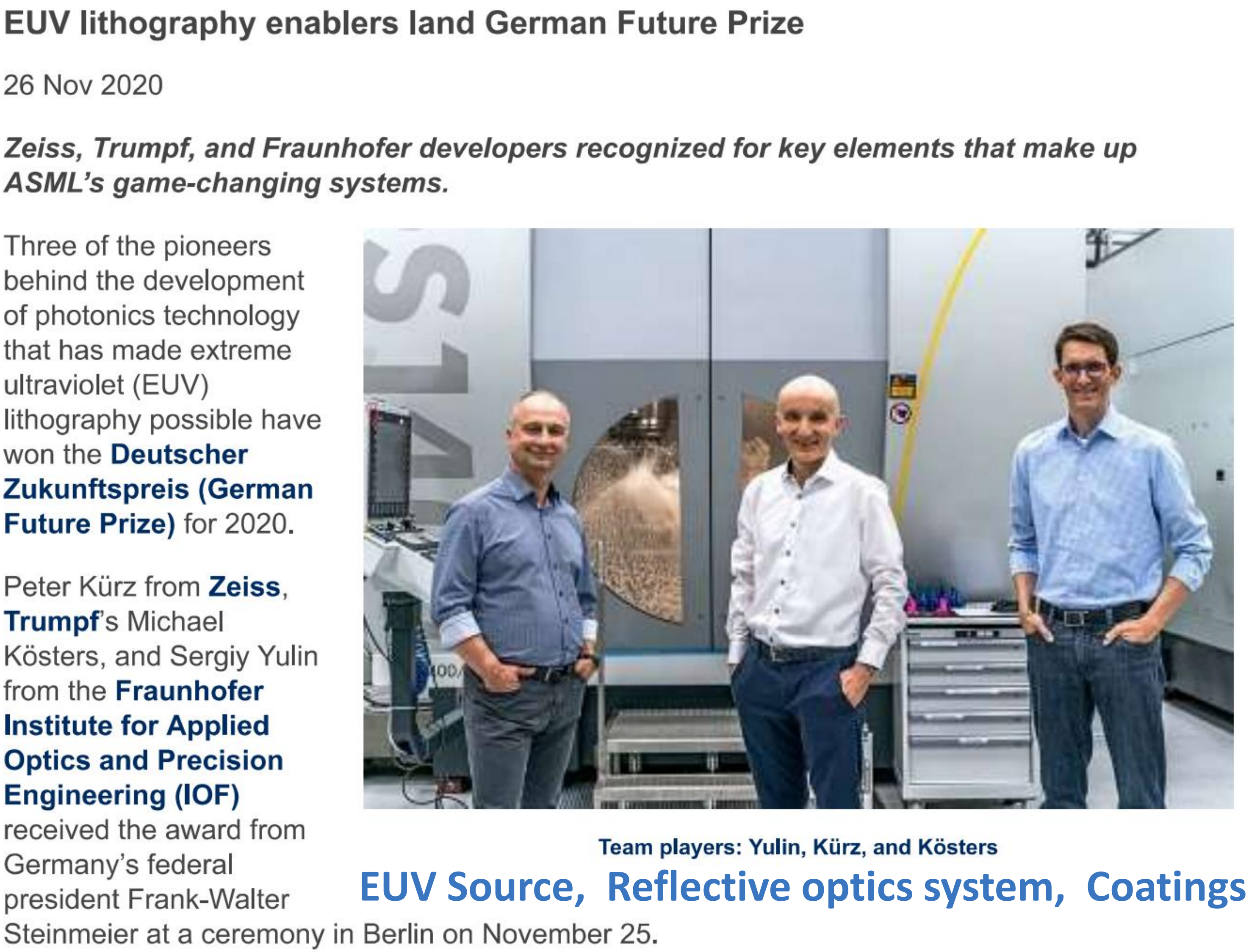


Artifical crystal:

High-z / low-z materials

$2d\sin\theta = k\lambda$ d=1-20nm

Reflection of short wavelength light



EUV lithography enablers land German Future Prize

26 Nov 2020

Zeiss, Trumpf, and Fraunhofer developers recognized for key elements that make up ASML's game-changing systems.

Three of the pioneers behind the development of photonics technology that has made extreme ultraviolet (EUV) lithography possible have won the **Deutscher Zukunftspreis (German Future Prize)** for 2020.

Peter Kürz from **Zeiss**, **Trumpf**'s Michael Kösters, and Sergiy Yulin from the **Fraunhofer Institute for Applied Optics and Precision Engineering (IOF)** received the award from Germany's federal president Frank-Walter Steinmeier at a ceremony in Berlin on November 25.

Team players: Yulin, Kürz, and Kösters

EUV Source, Reflective optics system, Coatings

1 Design of XUV multilayers

2 Interface engineering of nanolayers

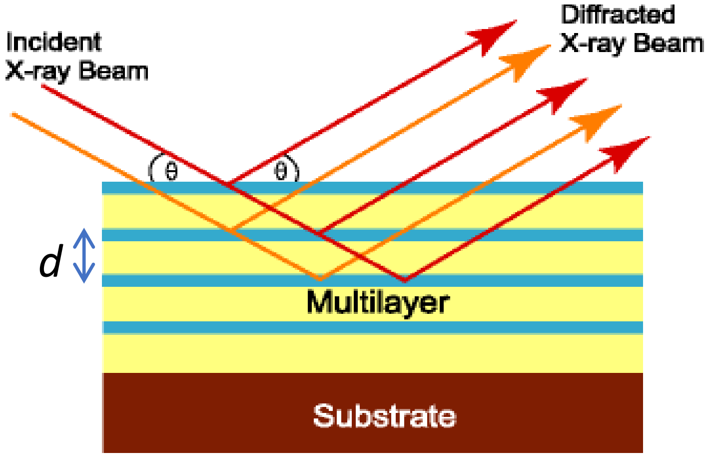
3 Stability of nanoscale ML

4 Deposition of large size mirrors

5 Micro/Nano structured ML

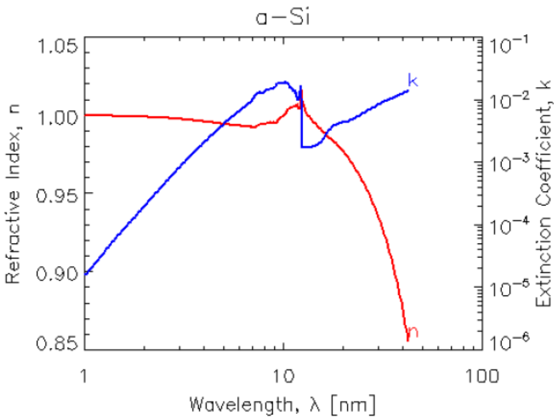
Materials Selection

$n=1-\delta-ik$

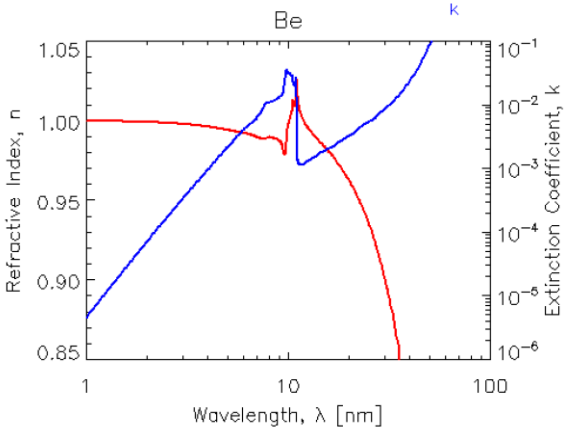


High z – absorber, low z - spacer

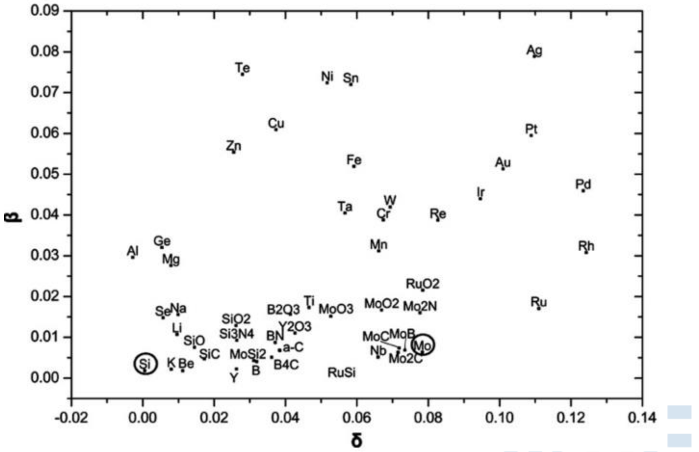
- ✓ Large difference in δ
- ✓ Small absorption, especially for spacer
- ✓ Sharp interface between two materials
- ✓ Stability over time



Si-L edge @ 12.4nm

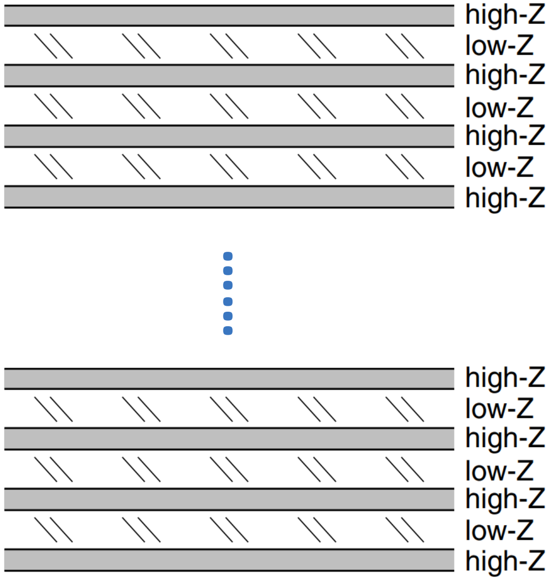


Be-K edge @ 11.0nm



Louis et al. Progress in Surface Science 86, 255 (2011)

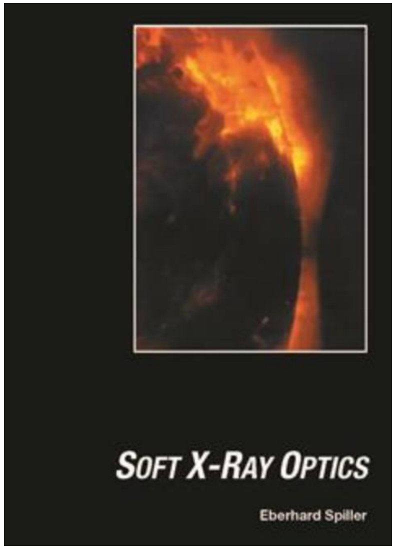
Layer structure



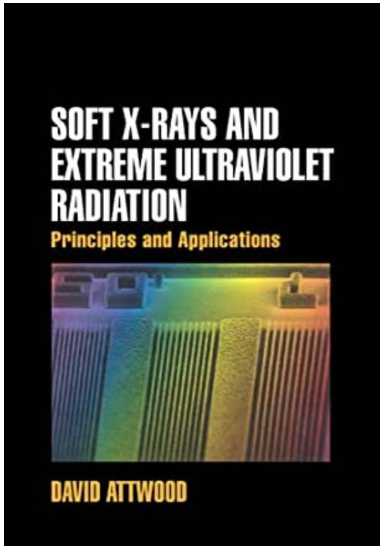
d-spacing $2d\sin\theta = k\lambda$

Thickness ratio $\Gamma = \frac{\Delta t_{\mathrm{H}}}{\Delta t_{\mathrm{H}} + \Delta t_{\mathrm{L}}} = \frac{\Delta t_{\mathrm{H}}}{d}$

Number of bilayers N


SOFT X-RAY OPTICS

Eberhard Spiller

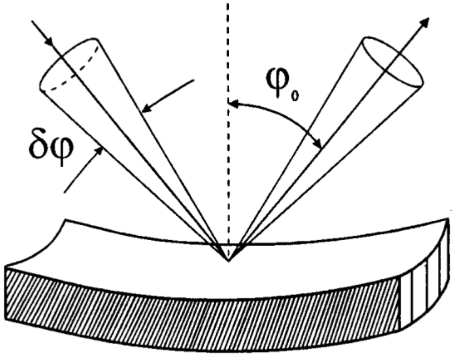

SOFT X-RAYS AND EXTREME ULTRAVIOLET RADIATION

Principles and Applications

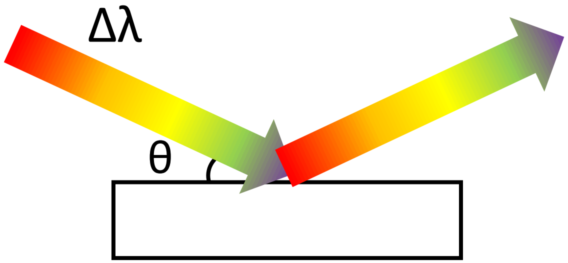
DAVID ATTWOOD

Aperiodic multilayer

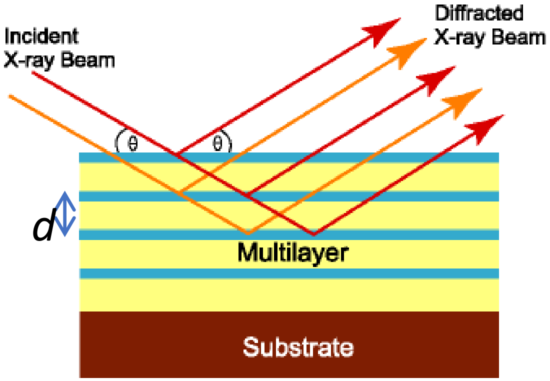
Broadband multilayer



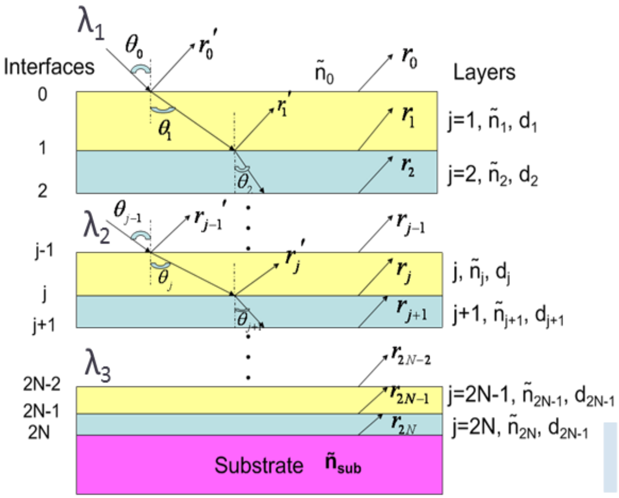
Yakshin et al, Opt. Express, 18, 6957 (2010)



Periodic



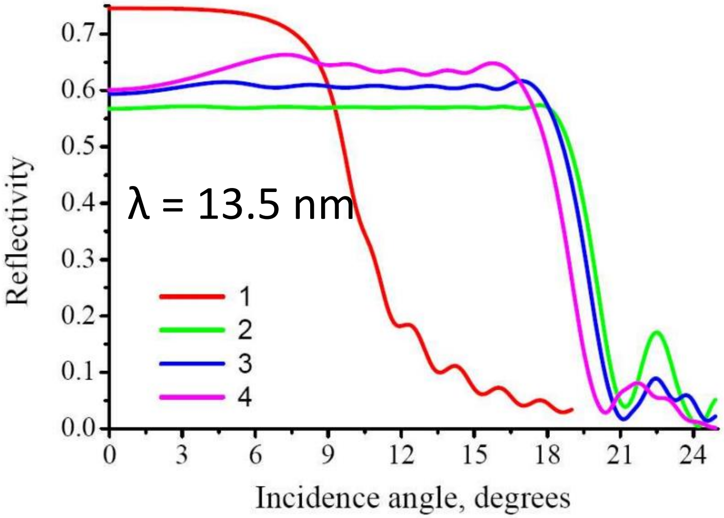
Aperiodic



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

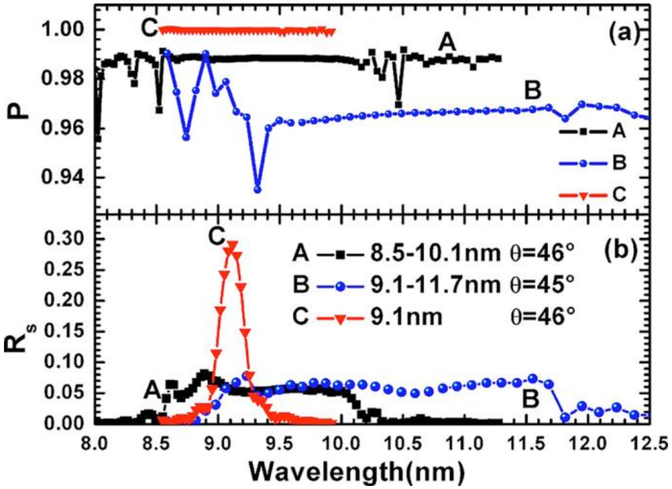
Aperiodic multilayer

Aperiodic Mo/Si mirror for EUVL



Yakshin et al, Opt. Express, 18, 6957 (2010)

Aperiodic Mo/Y polarizer @8.5-10.1nm

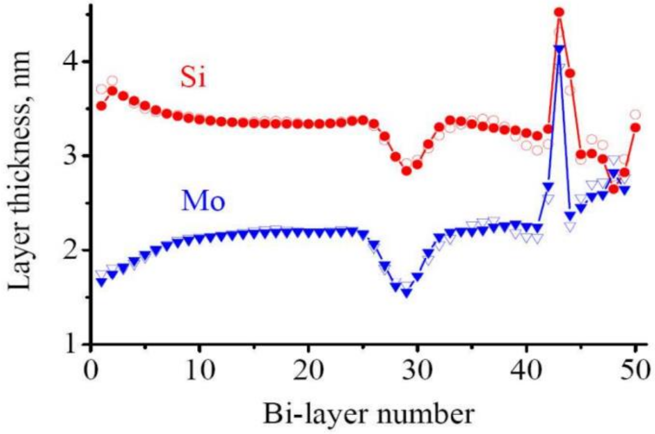


Wang et al, Appl. Phys. Lett. 89, 241120 (2006)

Experimental concern:

Smooth variation of layer thickness

- reduce fabrication difficulty



Yakshin et al, Opt. Express, 18, 6957 (2010)

1 Design of XUV multilayers

2 Interface engineering of nanolayers

3 Stability of nanoscale ML

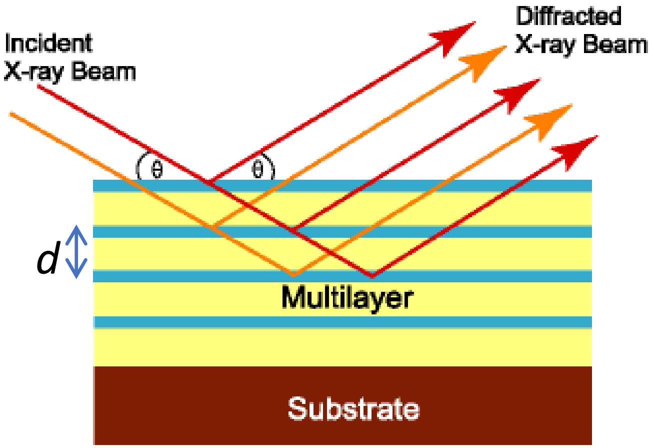
4 Deposition of large size mirrors

5 Micro/Nano structured ML

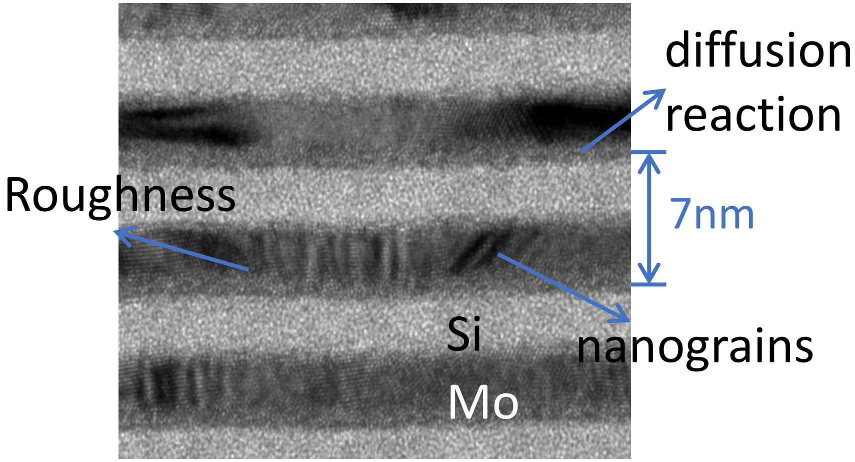
Interface is Imperfect

Short wavelength -> d_{layer}=1~10nm

Ideal



Real

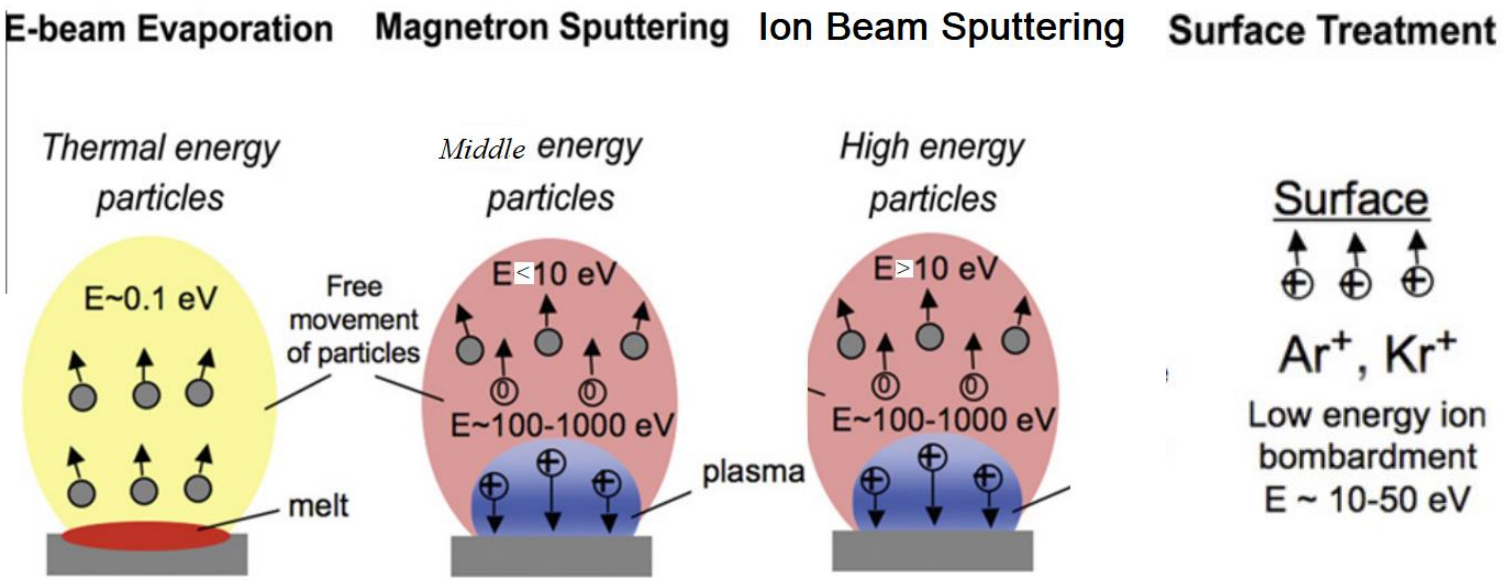


Interface imperfections significantly affect performance!

Understand the growth behavior of atomic layers → Create interfaces sharper & smoother

Interface engineering

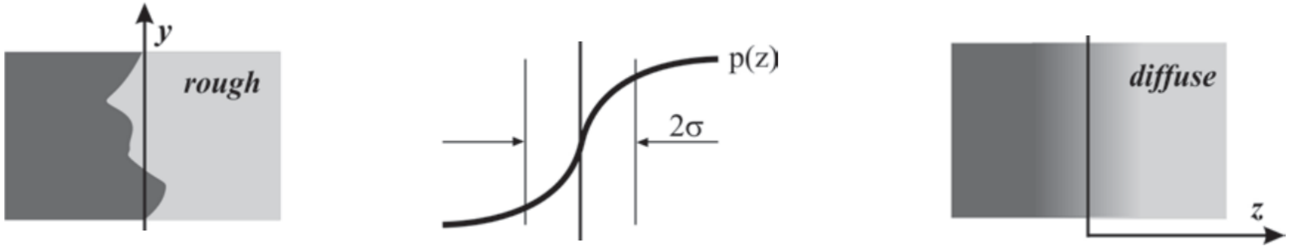
Typical fabrication techniques



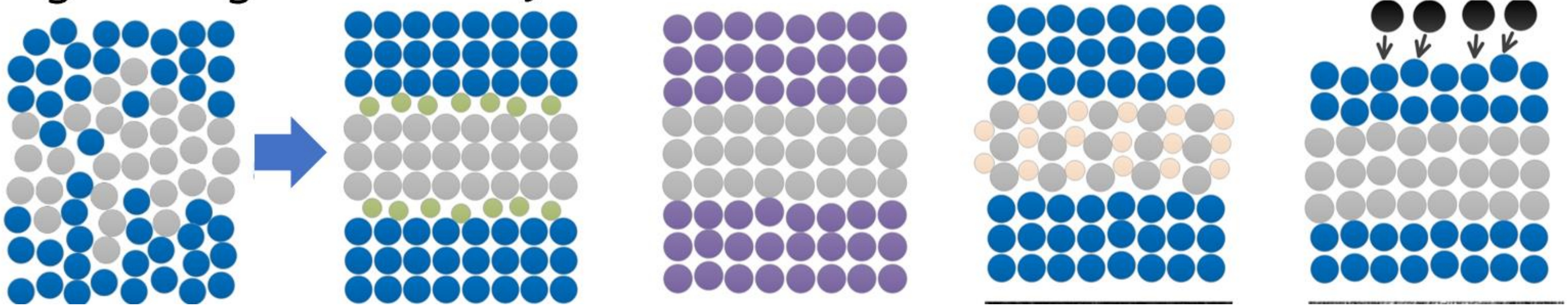
Louis et al. Progress in Surface Science 86, 255 (2011)

Different kinetic energy of deposited atoms will affect the interface roughness/diffusion

Interface Engineering



D. Windt
Imd Software
Installation
Guide & User's
Manual



no interface engineering

a) interface barrier layer

b) immiscible materials

c) reactive sputtering

d) Ions assistance

Barrier layer like B_4C, C … to suppress interdiffusion

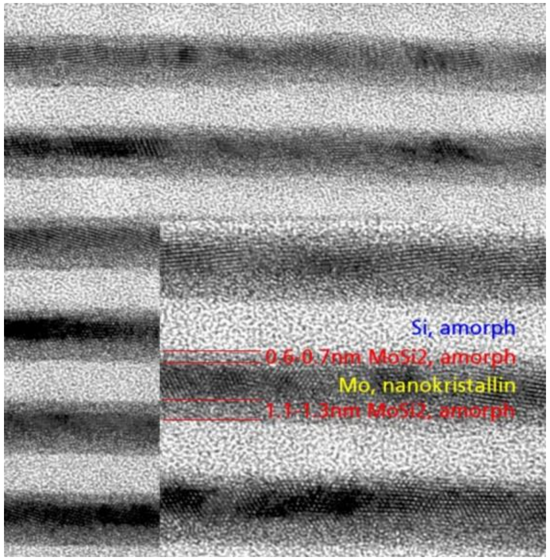
Composing materials have high enthalpy of formation

Mixture gas of Ar + N_2/air… to passivate interfaces

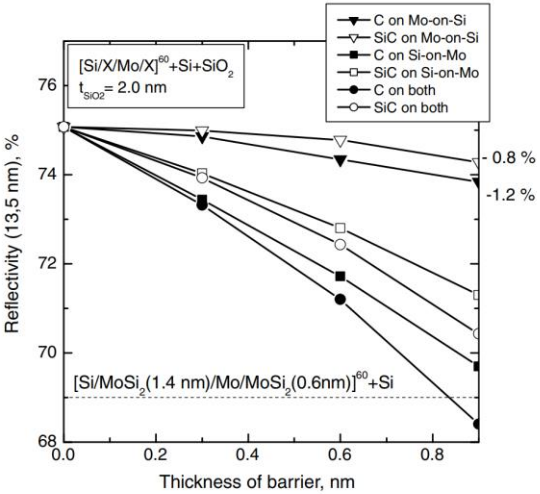
Low energy ions reduce interface roughness

Barrier layer for Mo/Si

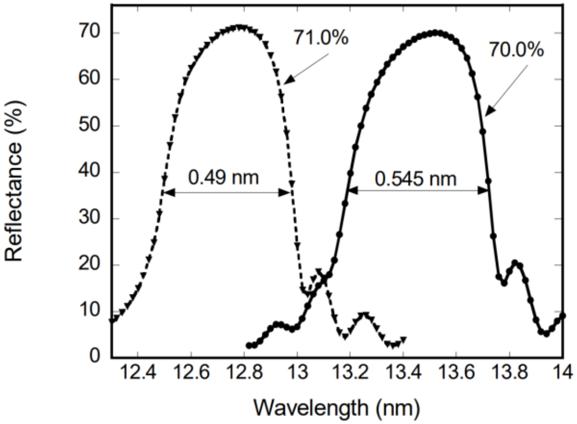
Formation of asymmetric Mo-silicide at two interfaces



Braun et al. SPIE, 4782 (2002)

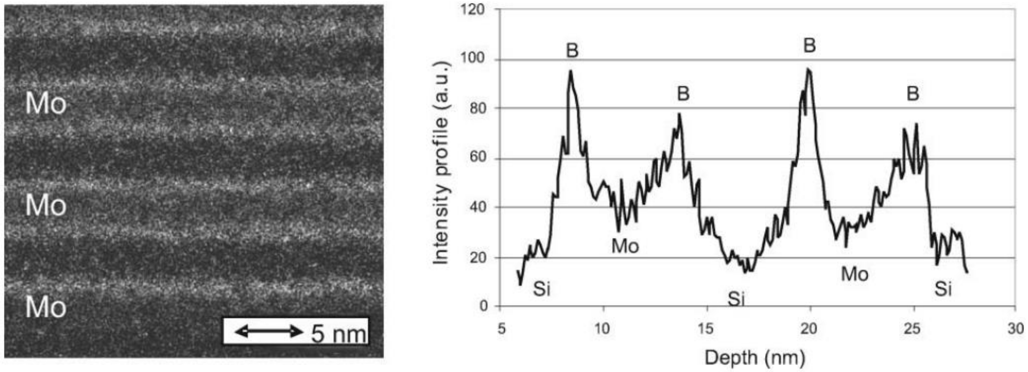


Yulin et al, Microelectronic Engineering 83, 692 (2006)



Bajt et al. SPIE, 4506, 65 (2001)

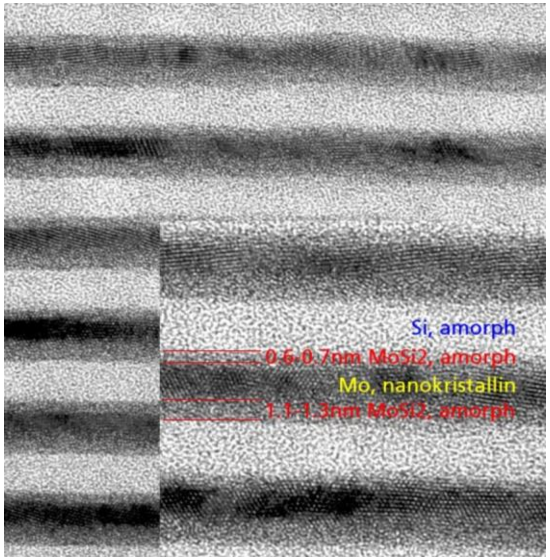
0.4nm B_4C Mo-on-Si
0.25nm B_4C Si-on-Mo



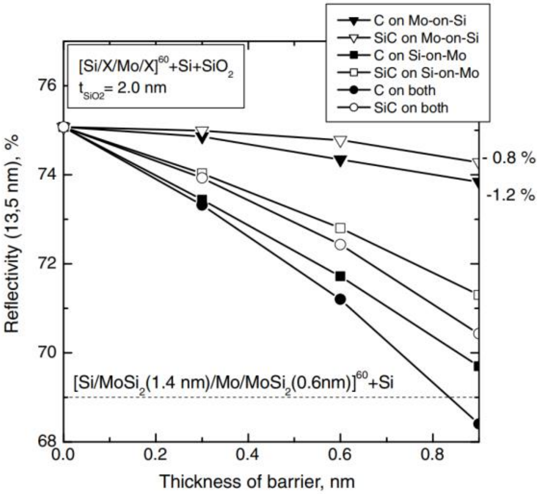
Nedelcu et al, Appl. Opt. 48(2), 155 (2009).

Barrier layer for Mo/Si

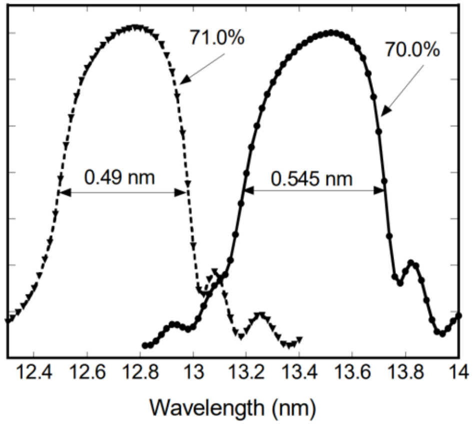
Formation of asymmetric Mo-silicide at two interfaces



Braun et al. SPIE, 4782 (2002)

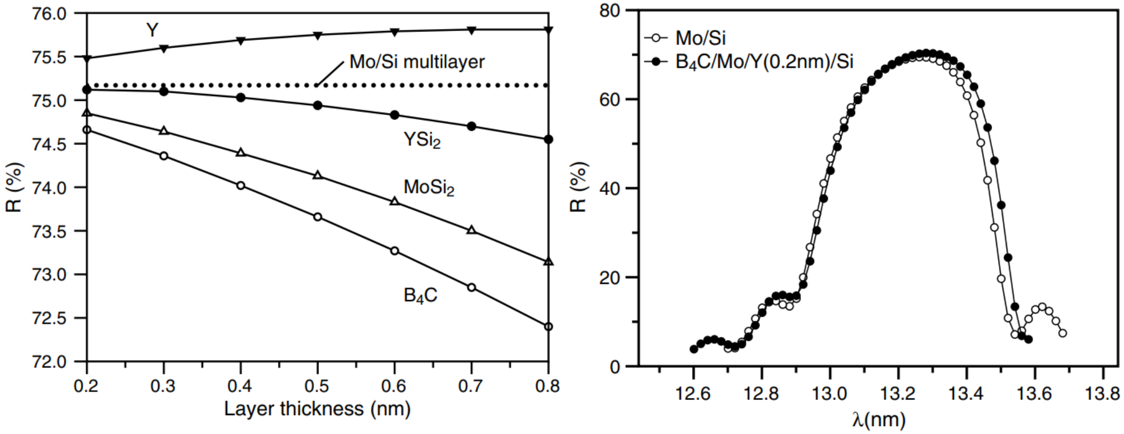


Yulin et al, Microelectronic Engineering 83, 692 (2006)



Bajt et al. SPIE, 4506, 65 (2001)

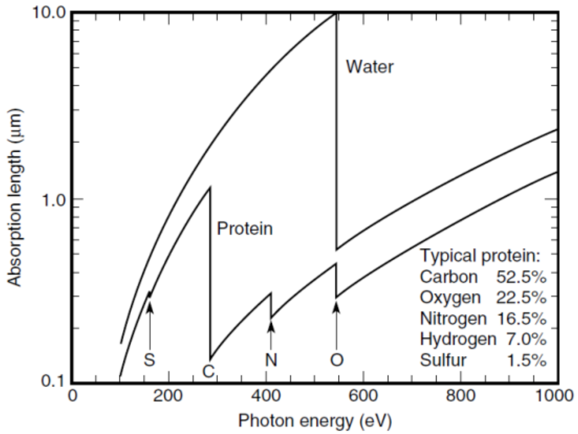
$B_4C/Mo/Y/Si$



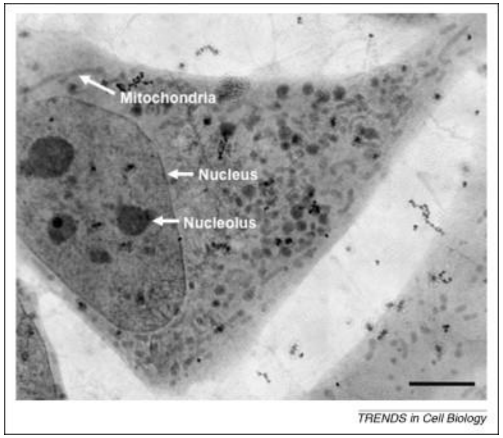
Bosgra et al. Appl. Opt. 51, 8541 (2012)

Barrier layer for Cr/V

Soft X-ray “water window ” imaging (λ=2.2–4.4 nm)



D.T. Attwood, SXR and EUV radiation.



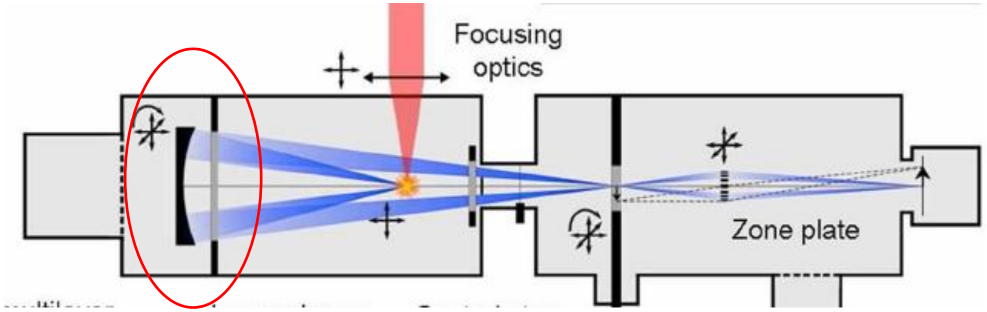
Hydrated biological sample

G. McDermott, et al, Trends Cell Biol. 19(11), 587 (2009)

Cr/V ML collector

d=1.2~1.6 nm

N>300

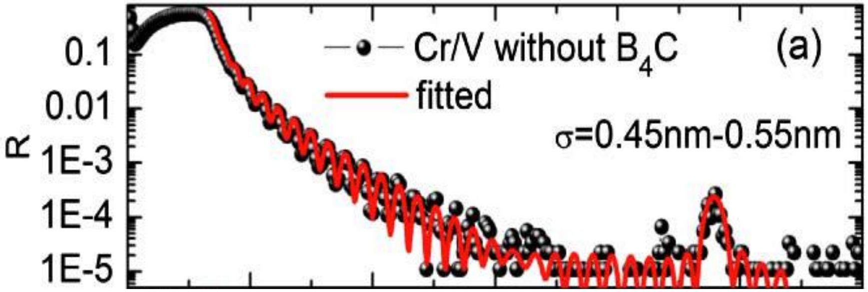


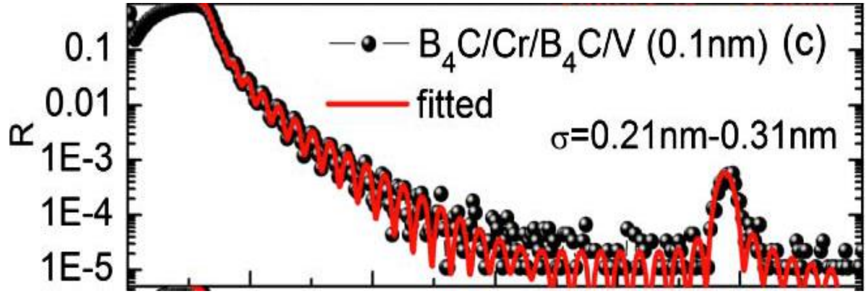
Legall, et al, J. Phys. Conf. Ser. 463, 012013 (2013).

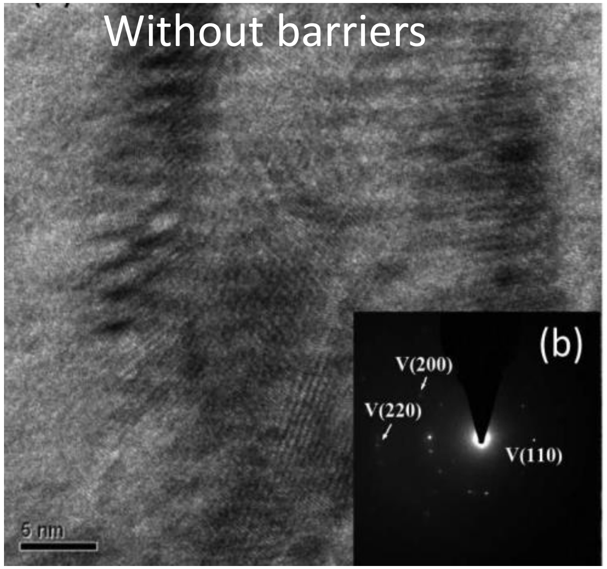
Barrier layer for Cr/V

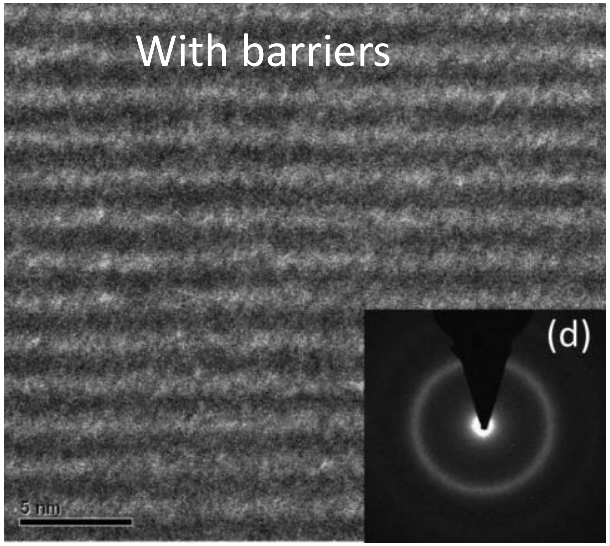
Cr/V ML

$d_{Cr}=d_V=0.9$ nm





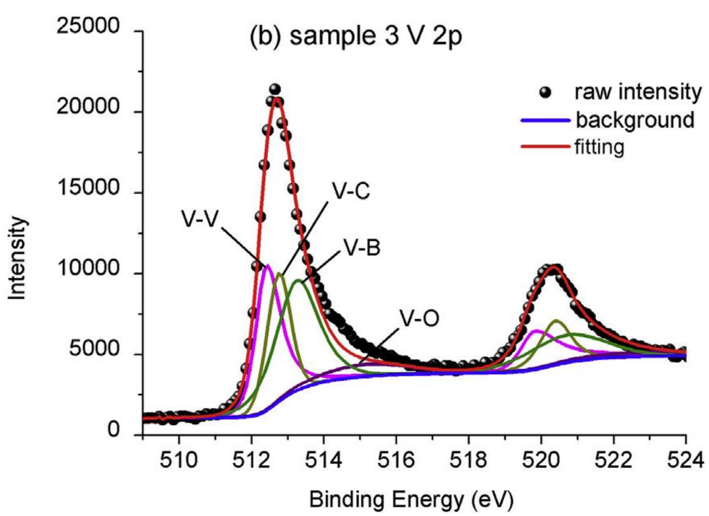


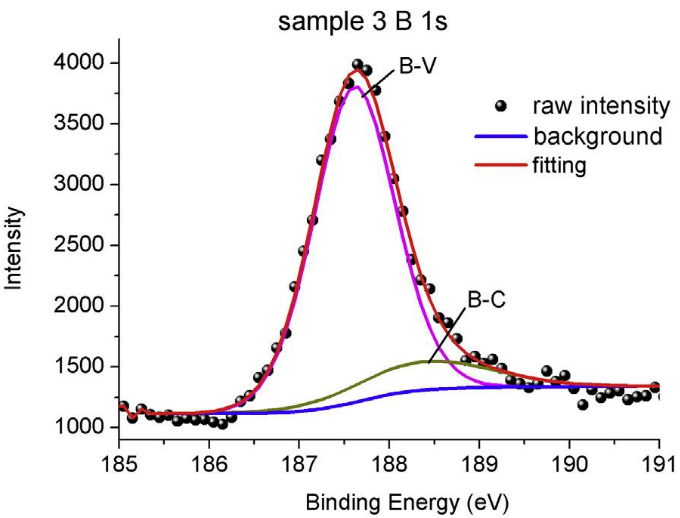


0.1nm B_4C barrier suppress the polycrystalline layer growth

Huang et al, Opt. Lett. 41(4), 701 (2016).

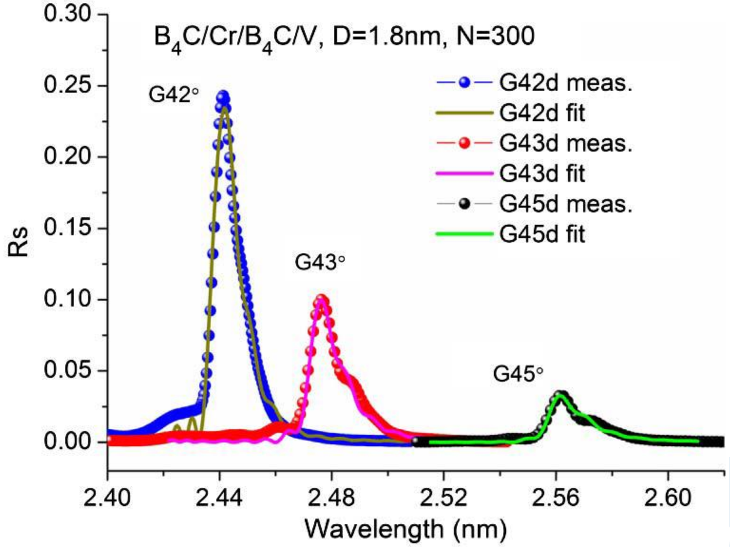
Barrier layer for Cr/V





Compound formation of VB_2 , VC suppress the crystallization and diffusion.

Rs=24% @ λ= 2.44nm
(Grazing 42deg)



P. Li, Q. Huang et al. Vacuum 128, 85 (2016).
Huang et al, Opt. Lett. 41(4), 701 (2016).

Reactive sputtering with N_2

Sputtering with noble gas (Ar)

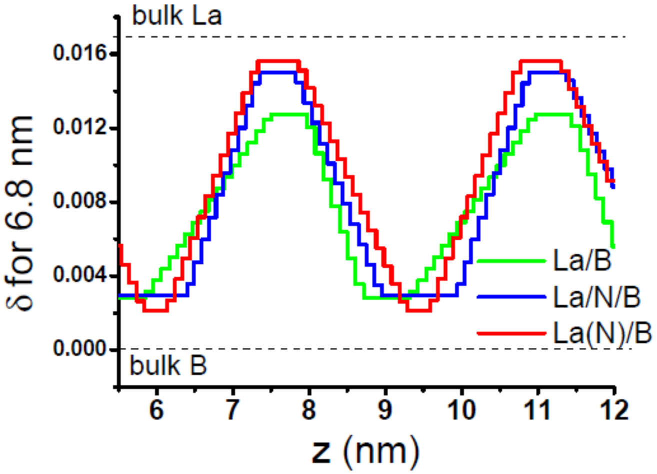
↓

Reactive sputtering with $Ar+N_2$

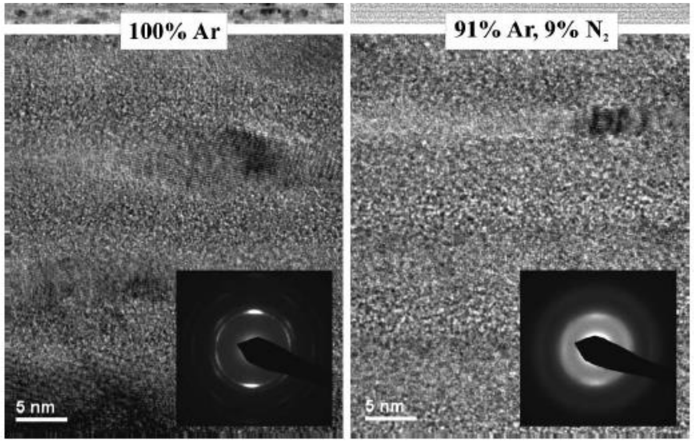
Reduce compound formation

Suppress crystallization

Stress relaxation

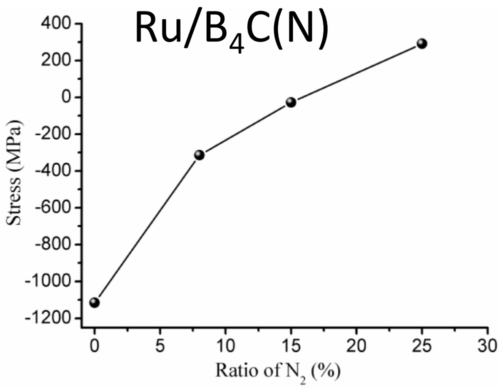


I.A. Makhotkin et al, Opt. Express, 21, 29894 (2013)



Al/SiC

D.L. Windt et al, Appl. Opt., 48, 4932 (2009)



Huang et al, Opt. Express, 26, 21803 (2019)

Reactive sputtering with N_2

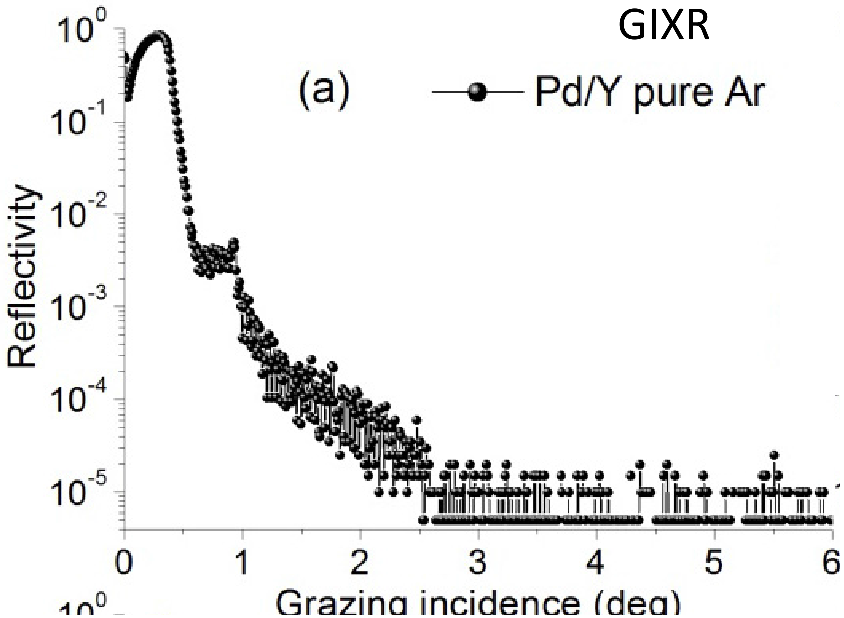
Pd/Y

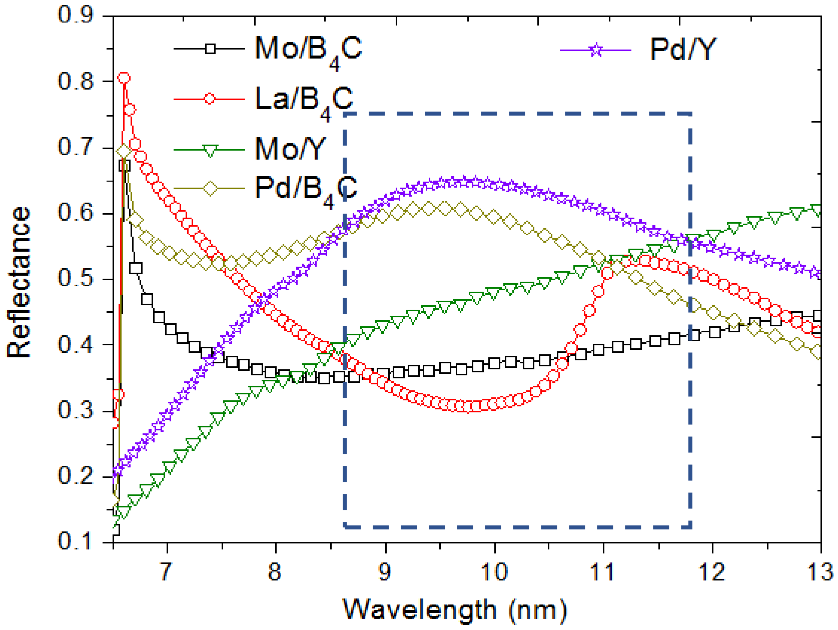
ideal mirror - λ=8.5-12nm

High R/low stress

D=5nm, N=80

Direct current magnetron sputtering





Dramatic interdiffusion between Pd and Y

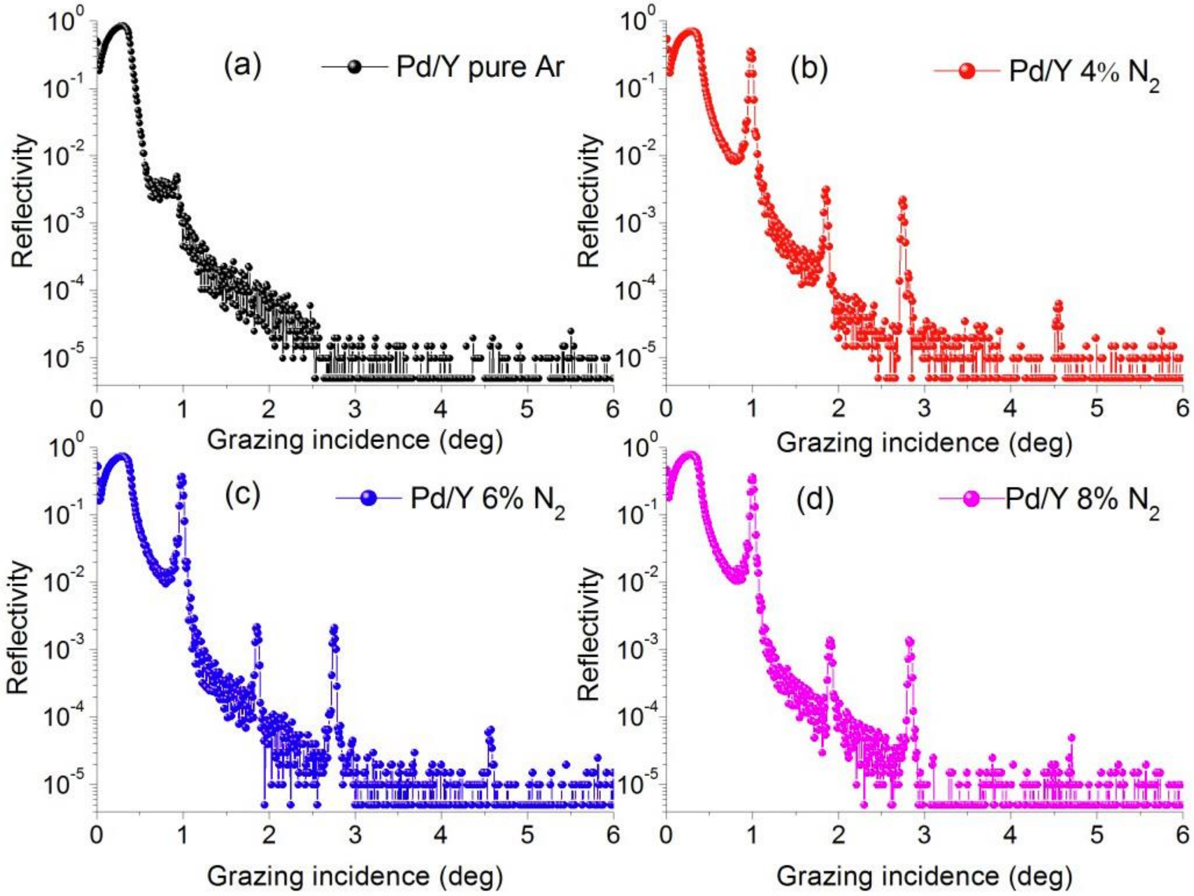
Pd-Y compound: ΔH°= -94 kJ/mol

Interface Engineering

Reactive sputtering with N_2

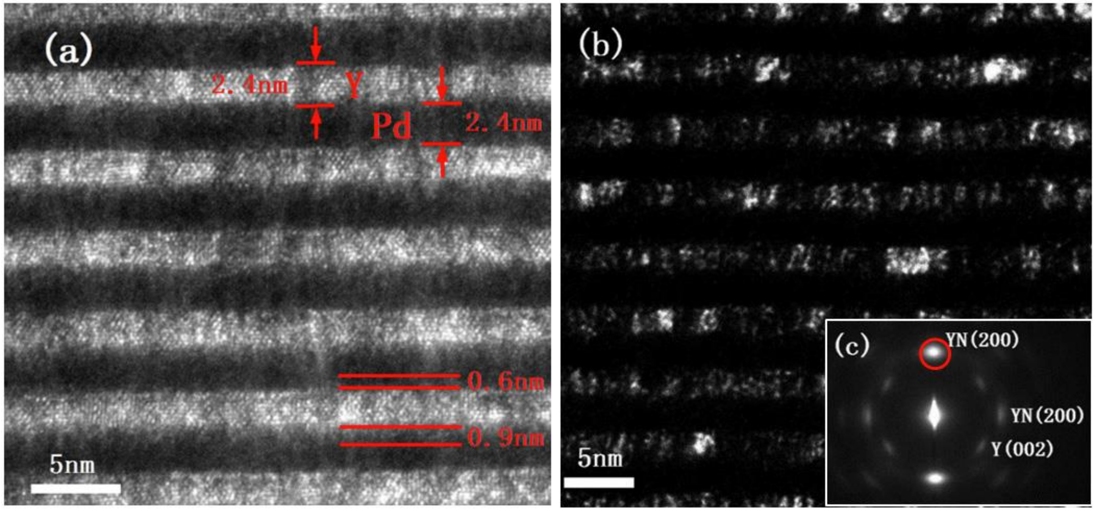
Pd-Y: ΔH°= -94 kJ/mol

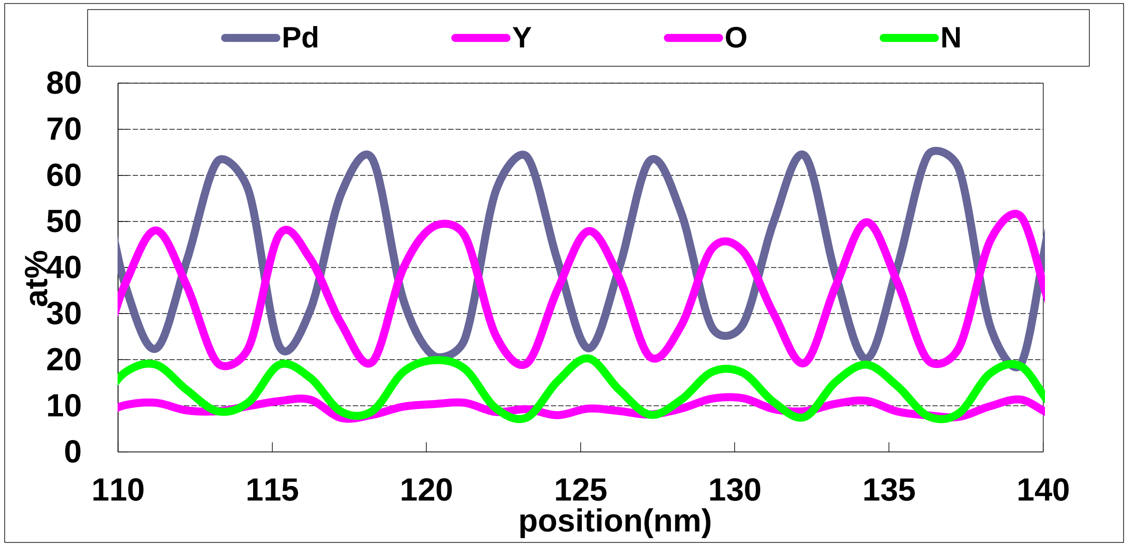
YN: -269 kJ/mol



Huang et al., Opt. Express, 23, 33018 (2015).

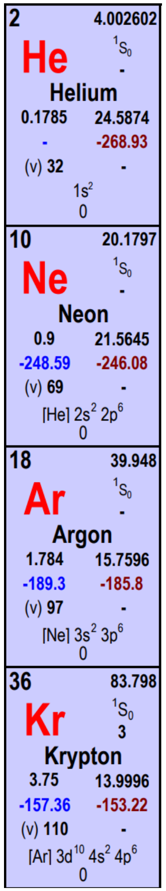
Reactive sputtering with N_2





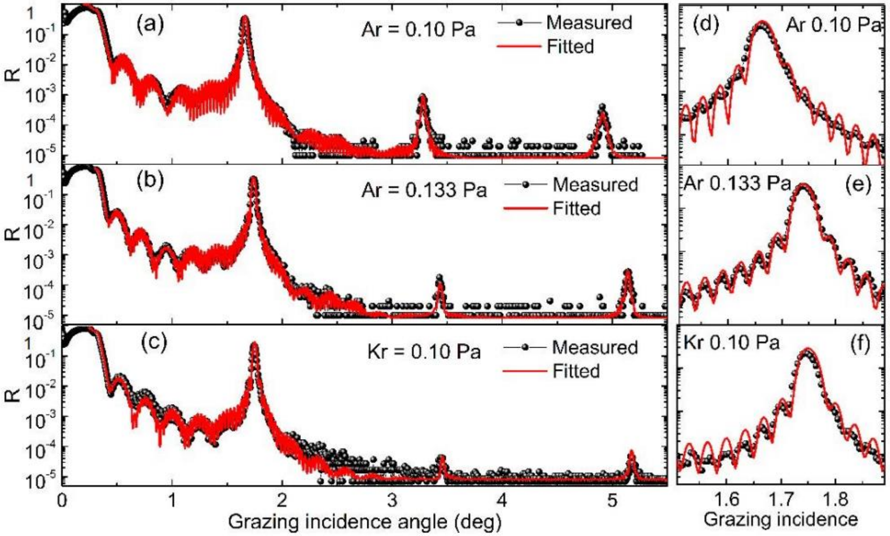
Huang et al, Opt. Express, 23, 33018 (2015).

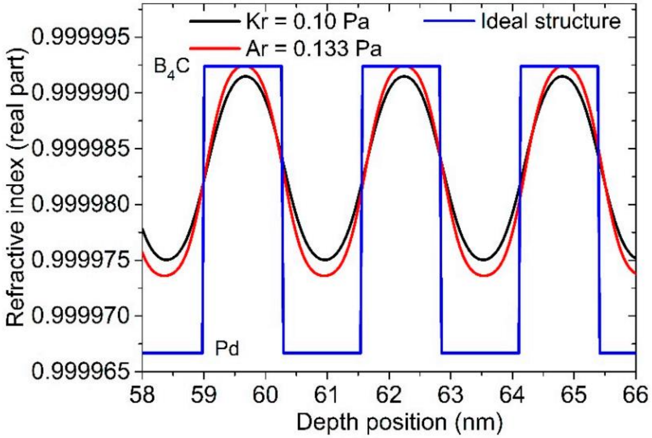
Sputtering with heavier ions



Larger ionization cross section → Lower sputtering pressure

Pd/B_4C, d=2.5nm, N=50~150



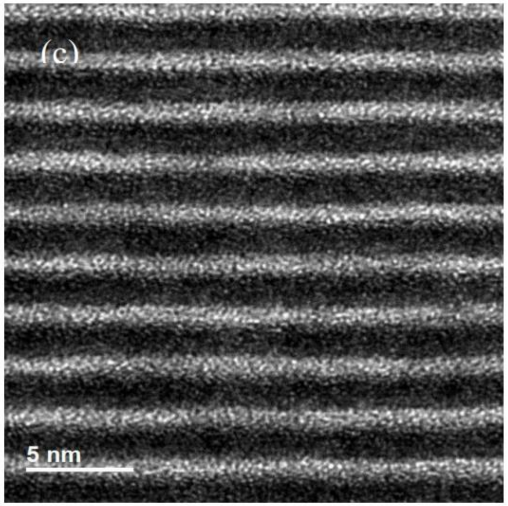


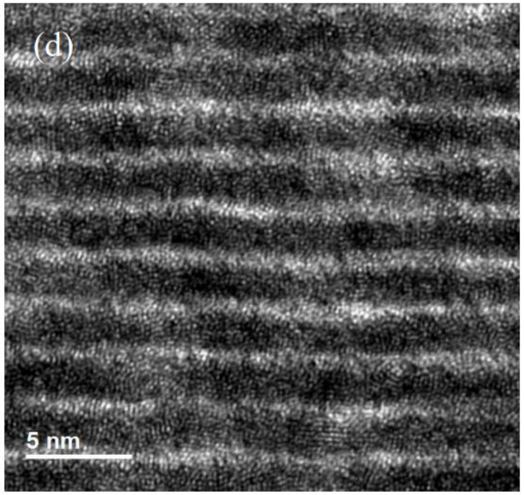
	Materials	Average Layer Thickness (nm)	d-Spacing Drift (pm)	Average Interface Width (nm)
Kr 0.10 Pa	Pd B_4C	1.320 1.247	25	0.30
Ar 0.133 Pa	Pd B_4C	1.337 1.252	30	0.26
Ar 0.10 Pa	Pd B_4C	1.246 1.460	60	0.27

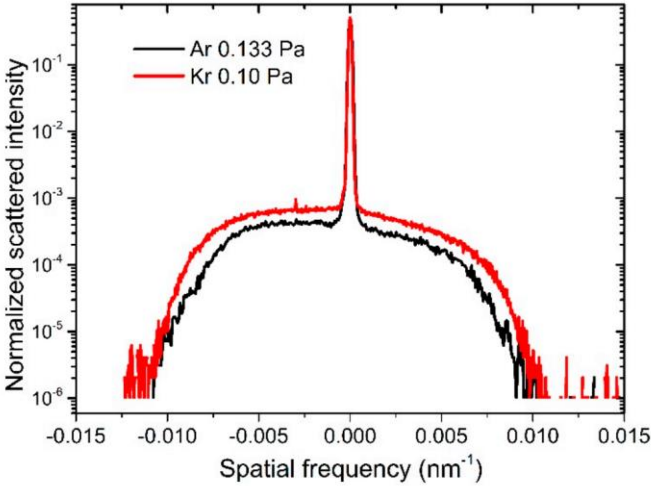
Larger interface width
Lower optical contrast

H. Ni, Q. Huang, et al., Materials, 13, 4504 (2020)

Reactive sputtering with N_2

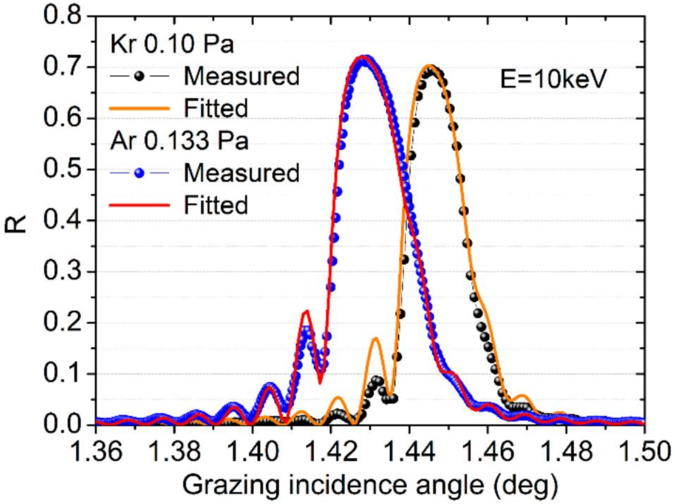






- Increased crystallization -> larger interface roughness
- Less incorporation of sputtering atoms in ML

- Difference in HXR reflectance is small



H. Ni, Q. Huang, et al., Materials, 13, 4504 (2020)

1 Design of XUV multilayers

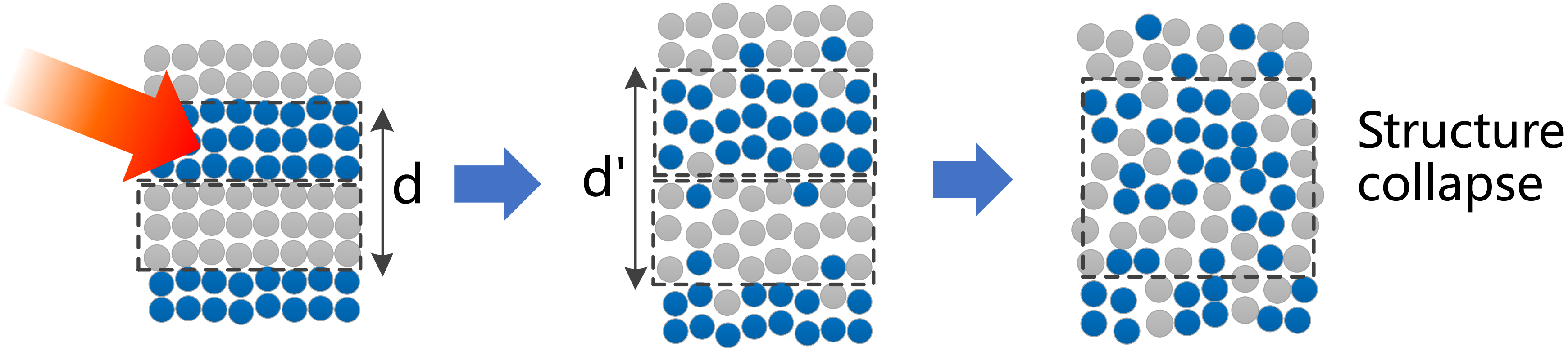
2 Interface engineering of nanolayers

3 Stability of nanoscale ML

4 Deposition of large size mirrors

5 Micro/Nano structured ML

Stability of nanoscale ML



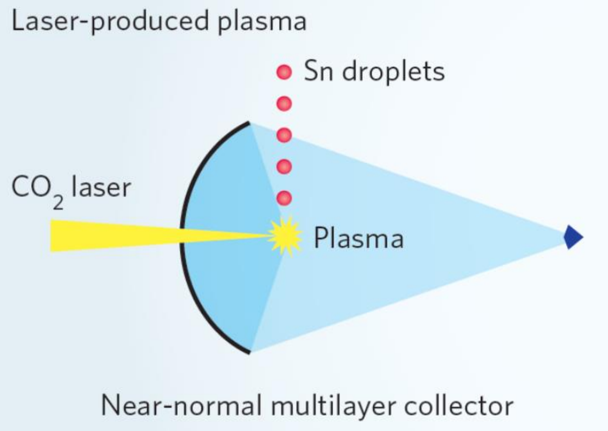
Working conditions

- ☐ Irradiation
- ☐ Surface contamination
- ☐ Thermal load
- ☐ Humidity
- ☐ …….

ML deterioration

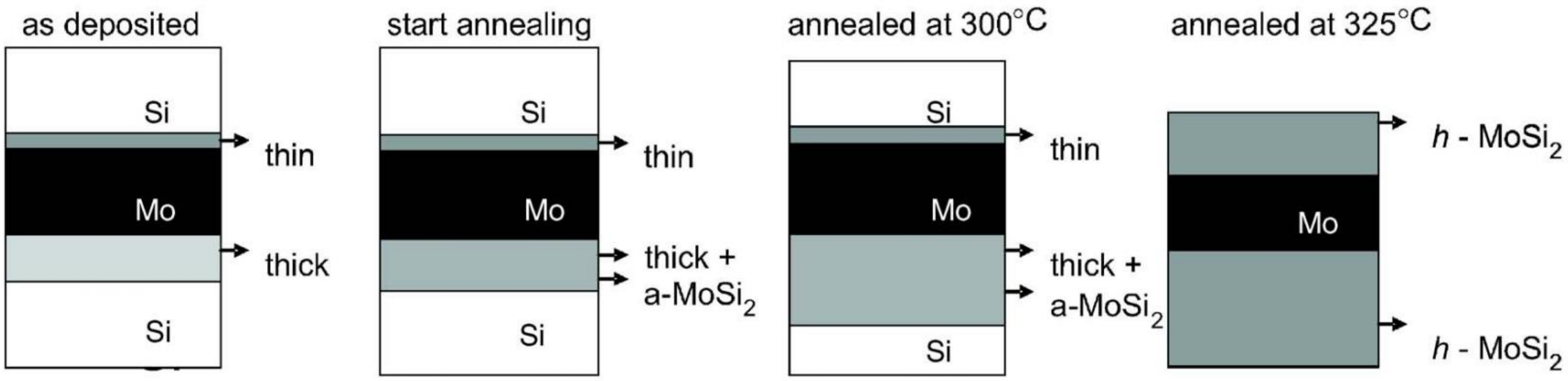
- ☐ recrystallization
- ☐ Increased interdiffusion
- ☐ Compound formation
- ☐ Period expansion/contraction
- ☐ Decrease of reflectance

Thermal stability of Mo/Si



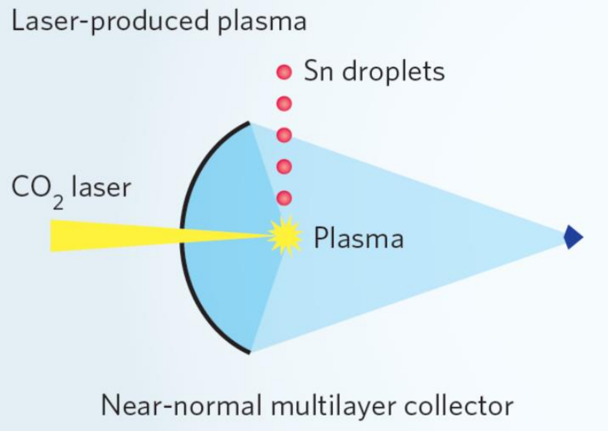
1^{st} collector mirror
– high heat load

Increased compound, nanograins formation of Mo_5Si_3 - $MoSi_2$ and diffusion with temperature



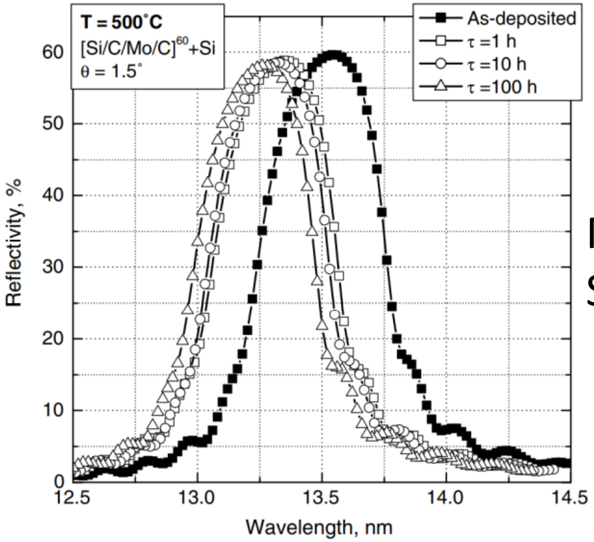
Nedelcu et al., J. Appl. Phys. 103, 083549 (2008); Phys. Rev. B 76, 245404 (2007)

Thermal stability of Mo/Si



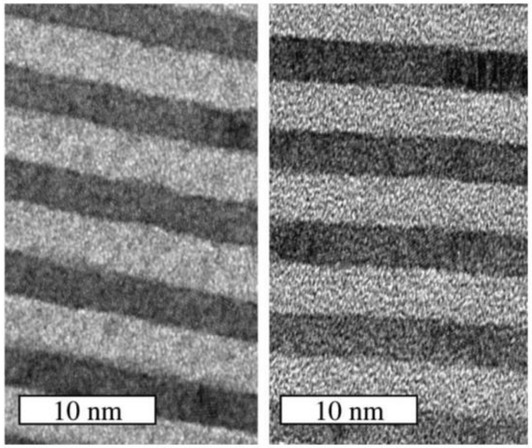
1st collector mirror
– high heat load

C diffusion barriers enhanced thermal stability of Mo/Si



Mo/C (0.8 nm)/
Si/C (0.8 nm)

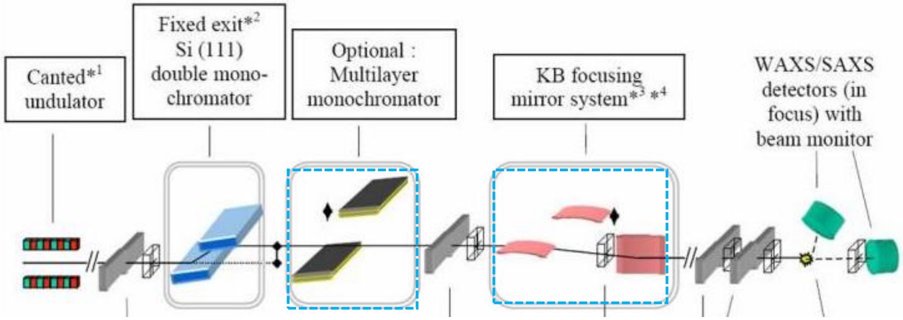
As-deposited 500°C 100h

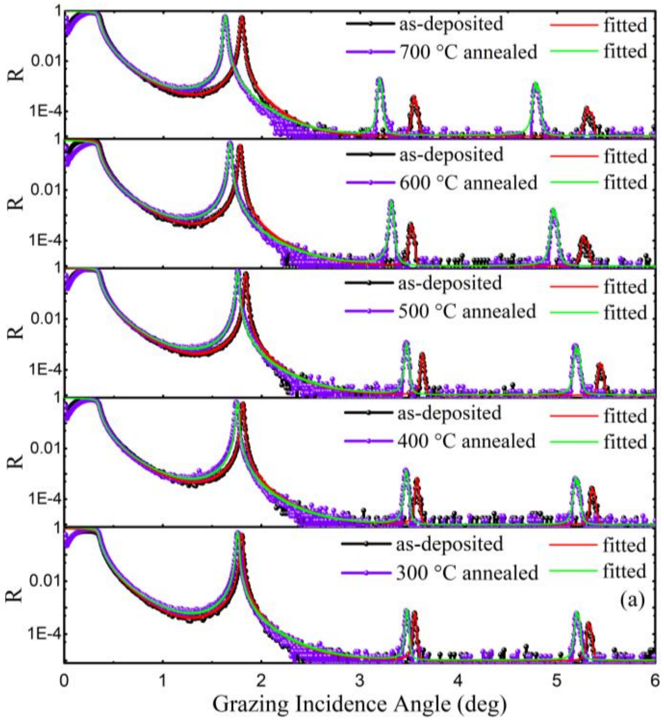


Yulin et al, Microelectronic Engineering 83, 692 (2006)

Thermal stability of Ru/C

Hard X-ray monochromator for synchrotron radiation beamlines

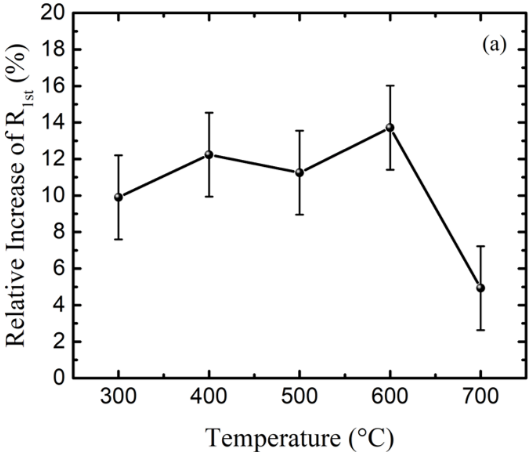


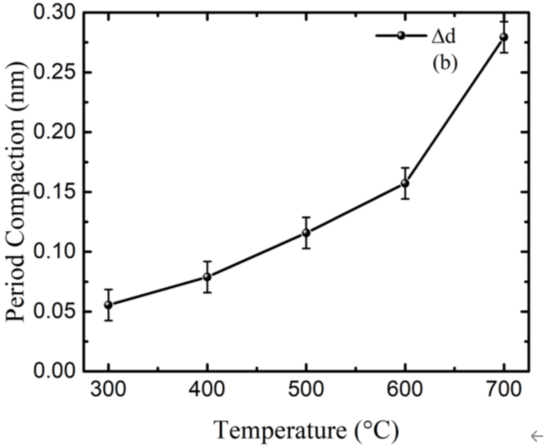


Ru/C ML, d=2.5nm, N=150

T=300-700°C

R increased by 5~10% ; d expands 2%-10%

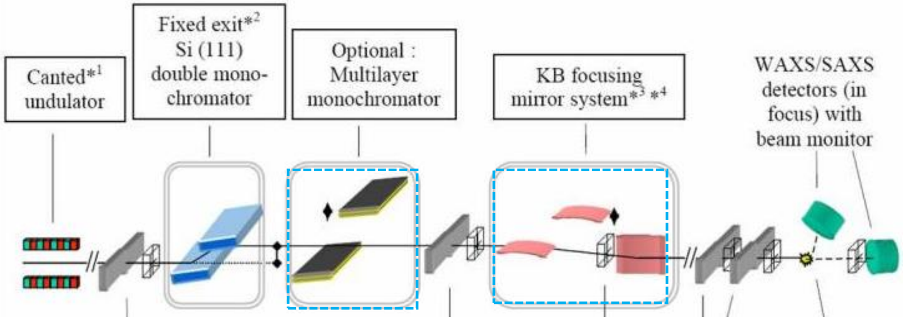




L. Yang, Q. Huang et al, Coatings, accepted (2020)

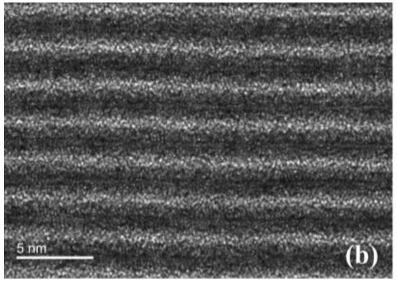
Thermal stability of Ru/C

Hard X-ray monochromator for synchrotron radiation beamlines

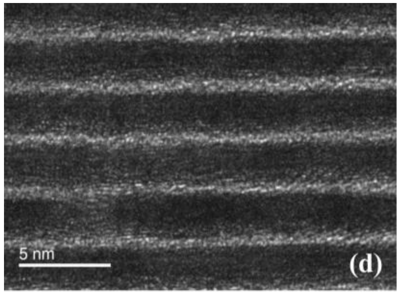


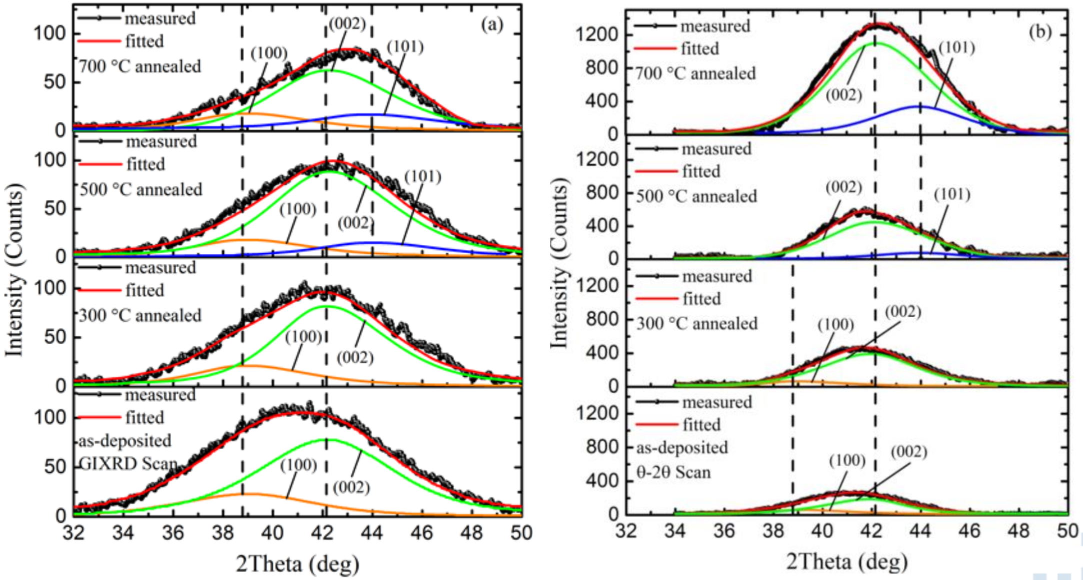
Reduced interdiffusion, improved optical contrast, and larger period, lead to the increased R

As-deposited



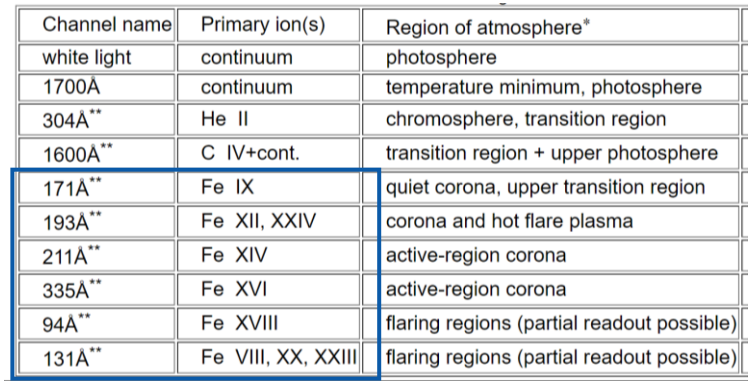
700°C 100h





L. Yang, Q. Huang et al, Coatings, accepted (2020)

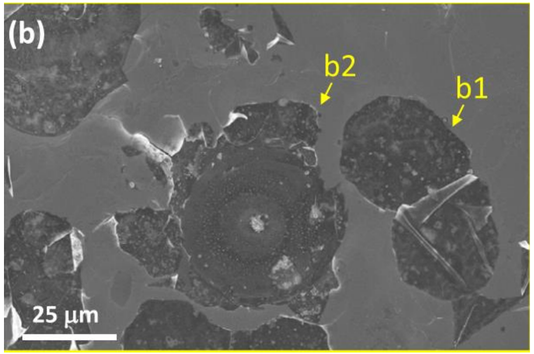
Temporal stability of Mg/SiC

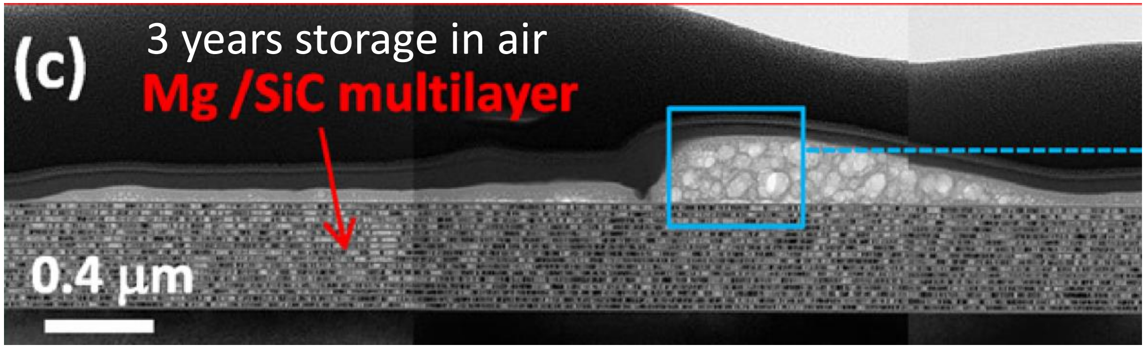


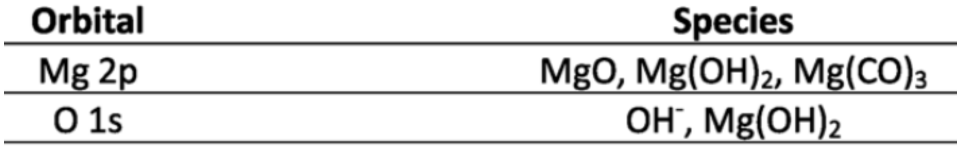
Channel name	Primary ion(s)	Region of atmosphere*
white light	continuum	photosphere
1700Å	continuum	temperature minimum, photosphere
304Å**	He II	chromosphere, transition region
1600Å**	C IV+cont.	transition region + upper photosphere
171Å**	Fe IX	quiet corona, upper transition region
193Å**	Fe XII, XXIV	corona and hot flare plasma
211Å**	Fe XIV	active-region corona
335Å**	Fe XVI	active-region corona
94Å**	Fe XVIII	flaring regions (partial readout possible)
131Å**	Fe VIII, XX, XXIII	flaring regions (partial readout possible)

Mg/SiC provides highest R @ λ > 25nm (d=14nm~25nm)

Severe surface corrosion occurred
- reaction of Mg with humidity & reactive ions







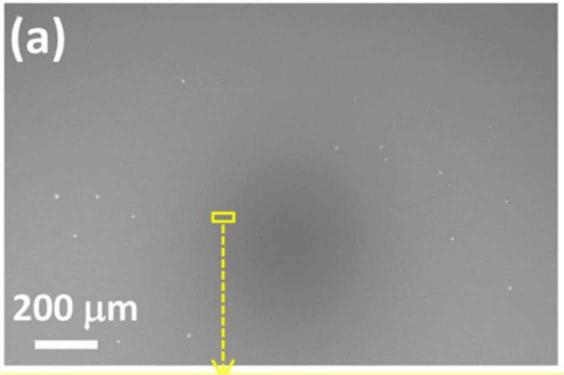
Orbital	Species
Mg 2p	MgO, $Mg(OH)_2$, $Mg(CO)_3$
O 1s	OH^-, $Mg(OH)_2$

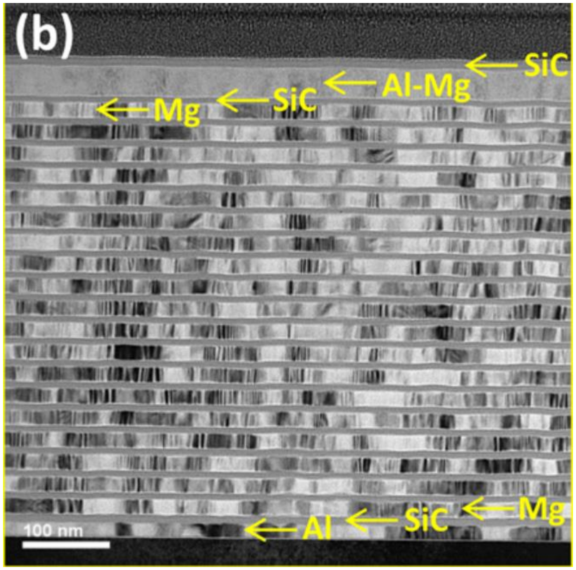
Soufli et al, Appl. Phys. Lett. 101, 043111 (2012)

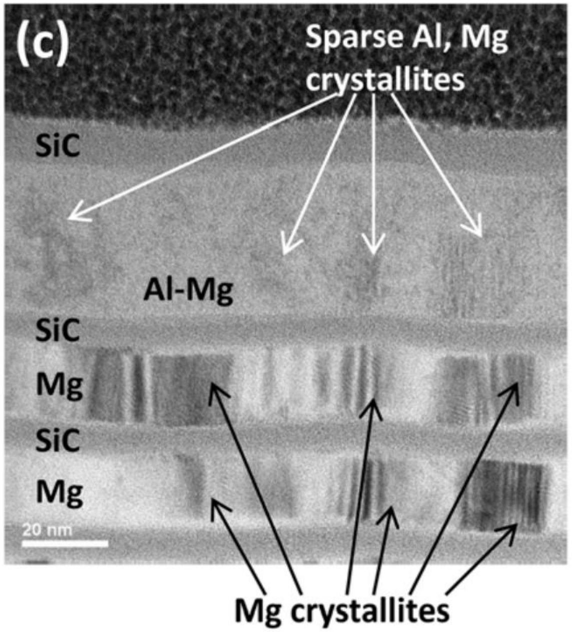
Temporal stability of Mg/SiC

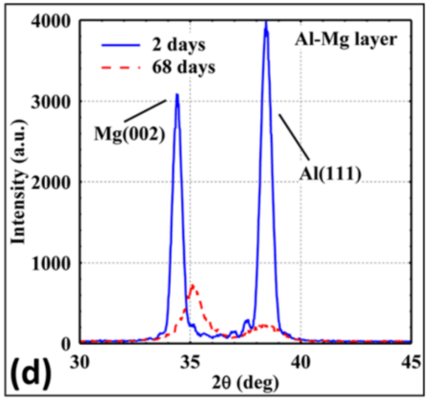
Surface capping layer – protecting ML against degradation

3 years storage in air







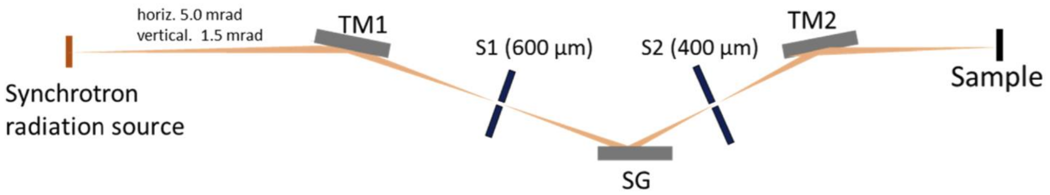


Spontaneous intermixed AlMg amorphous layer act as a dense capping layer against permeability of humidity, oxygen…

Soufli et al, Appl. Phys. Lett. 101, 043111 (2012)

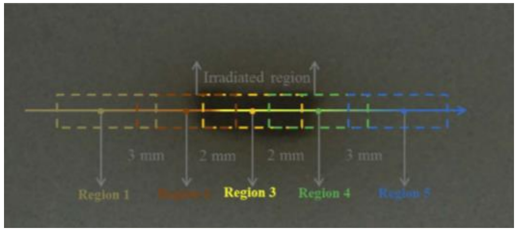
Irradiation stability of Cr/C

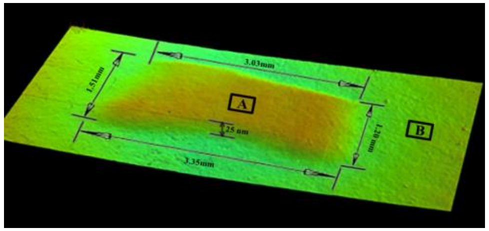
(NSRL)

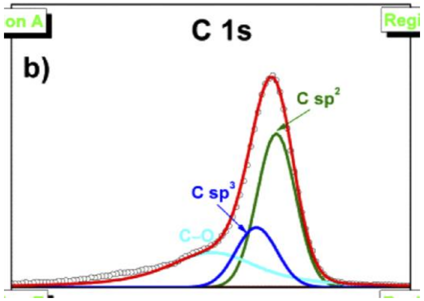


Cr/C ML
d=11.6nm
N=20

White beam irradiation 0.1W/mm^2, 18h , 4.0×10^{-4} Pa

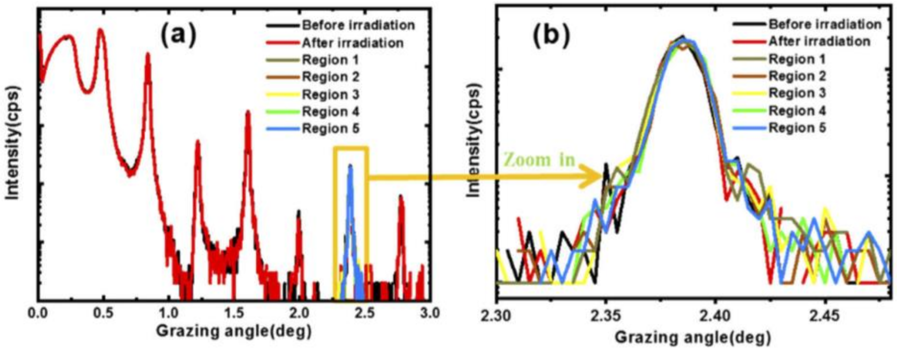


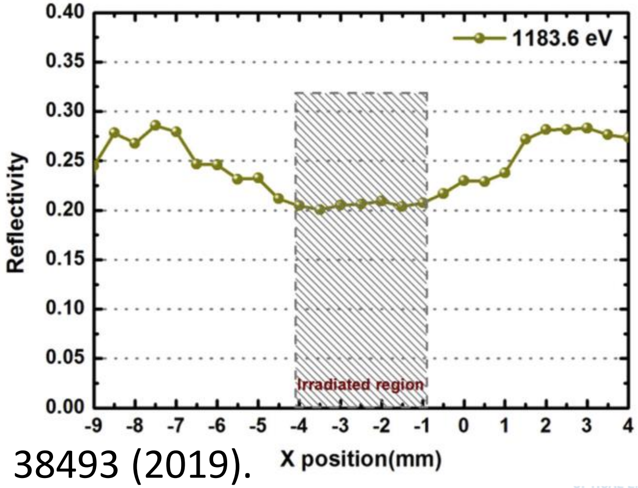




25nm C-based layer generated

Cr/C ML remained intact





J. Feng, Q. Huang, et al., Opt. Express, 27, 38493 (2019).

1 Design of XUV multilayers

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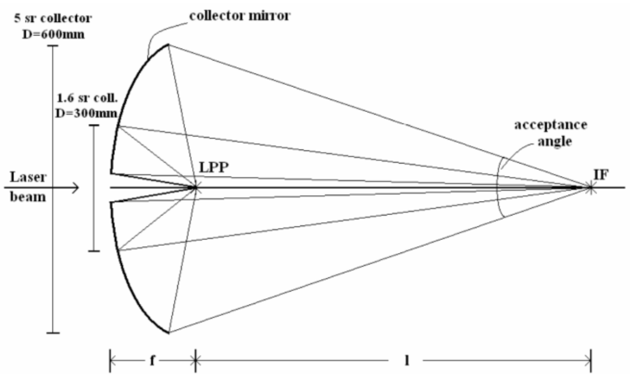
4 Deposition of large size mirrors

5 Micro/Nano structured ML

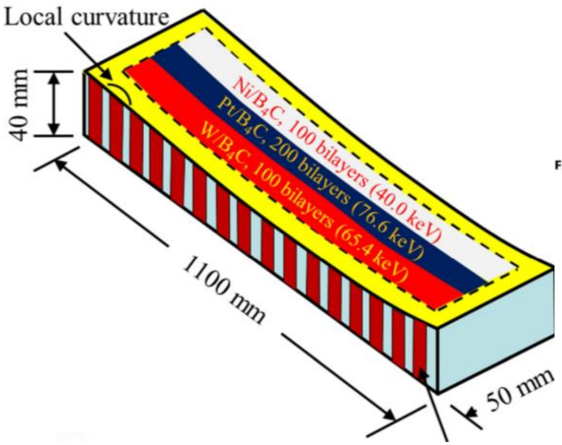
Deposition methods for large mirror

Near normal incidence



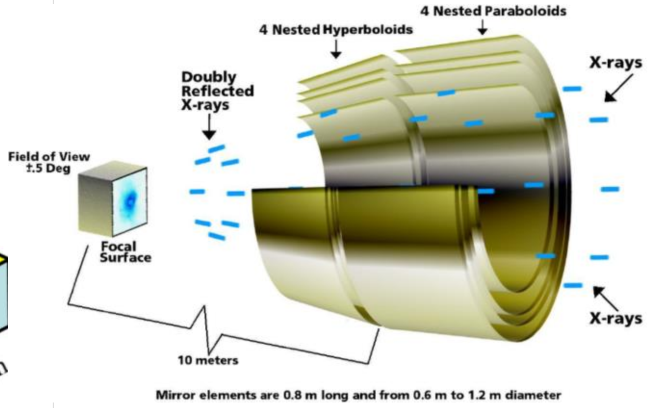
Feigl et al., SPIE, 7077, 70771W, (2008)

Grazing incidence



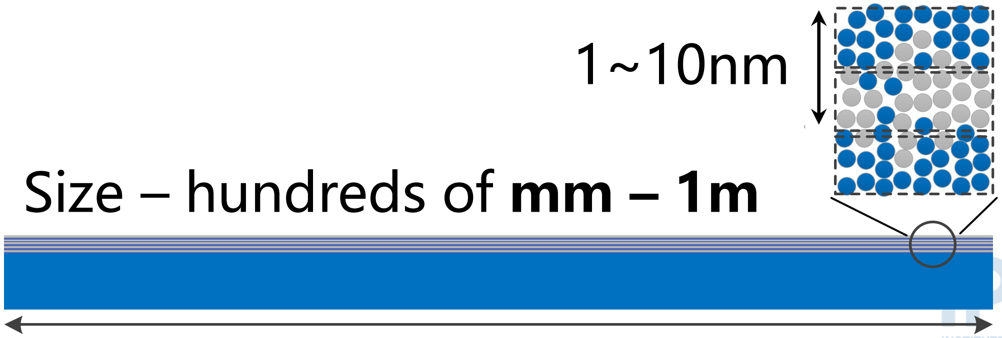
Sutter et al., Opt. Express, 27, 16121 (2019)

Grazing Incidence (nested shell)



O'Dell et al., SPIE, 7803 (2010).

Picometer control over large area of hundreds mm

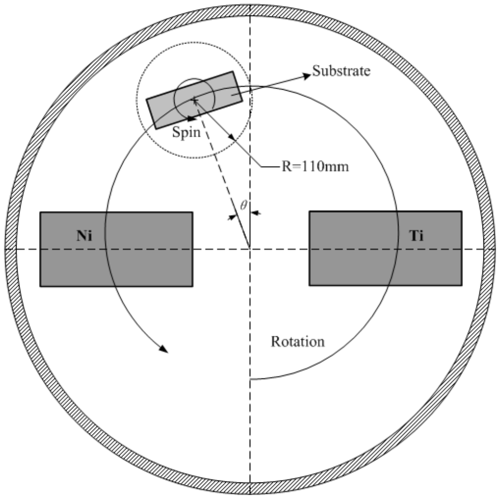


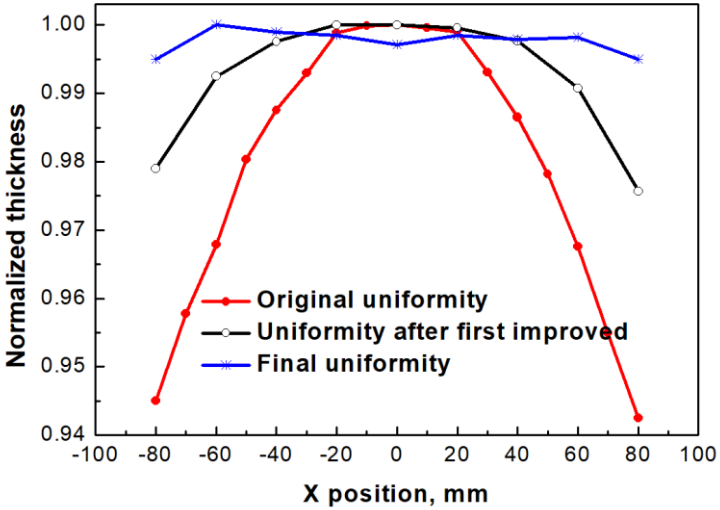
1~10nm

Size – hundreds of **mm – 1m**

Near normal incidence

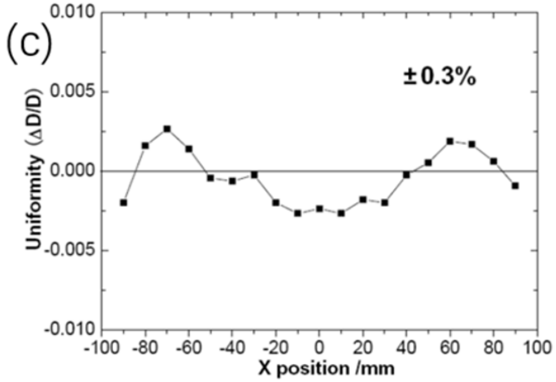
Planetary rotation deposition

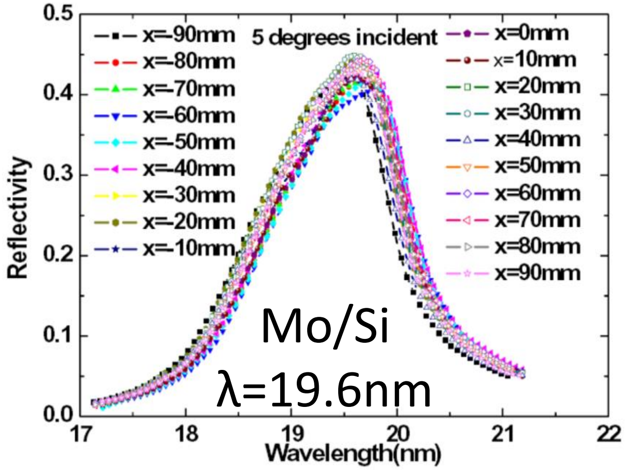




Φ200mm off-axis paraboloid

Uniformity < 0.6% (PV=60pm)





Mo/Si
λ=19.6nm

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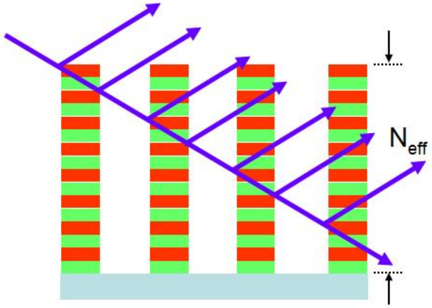
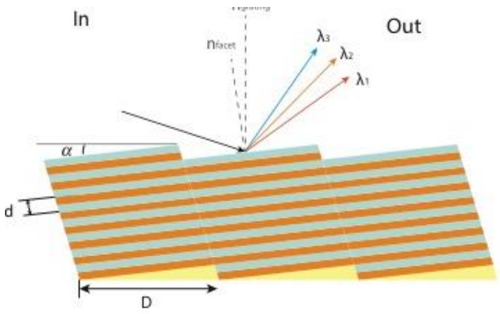
Nano-structured ML

Lamellar Multilayer grating

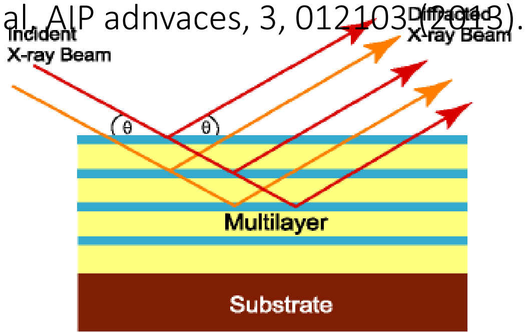


R. Van der Meer et al, AIP adnvaces, 3, 012103 (2013).

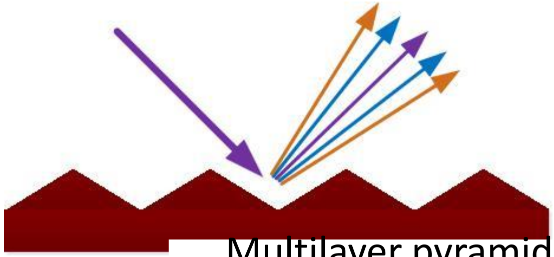
Blazed Multilayer grating (BMG)



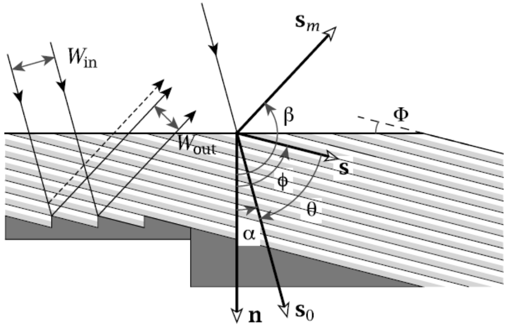
X. Yang et al, J. Opt. Soc. Am. B, 32, 506 (2015).



+

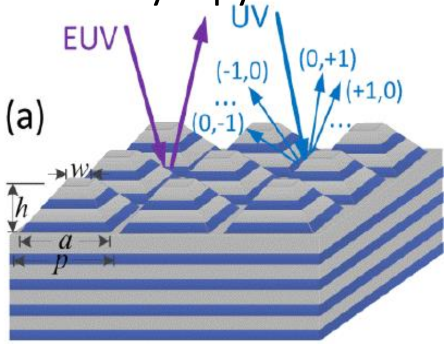


Sliced Multilayer grating



S. Bajt et al, J. Opt. Soc. Am. A, 29, 216 (2012).

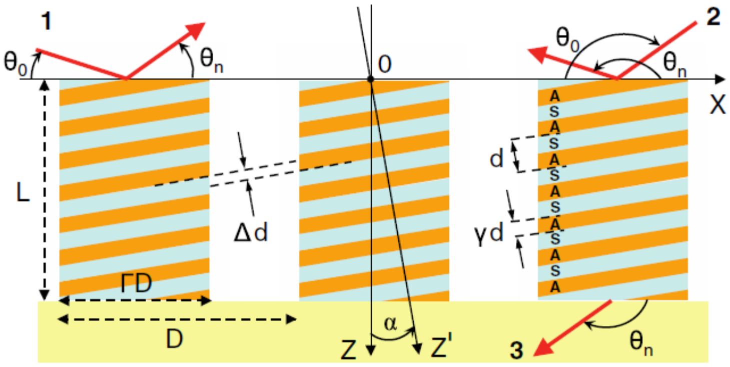
Multilayer pyramids

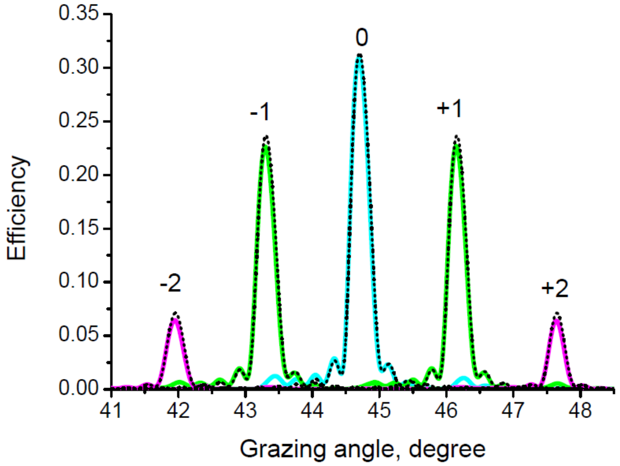


Q. Huang et al, Opt. Express, 3, 19365 (2014).

5.

Single-order diffraction model





Single order condition

$$\boxed{\Delta\theta << \frac{d}{jD\cos\alpha}}$$

$$R_n = \frac{1-V}{1+V} \quad , \quad V = \sqrt{\frac{1-y^2}{1+f^2y^2}}$$

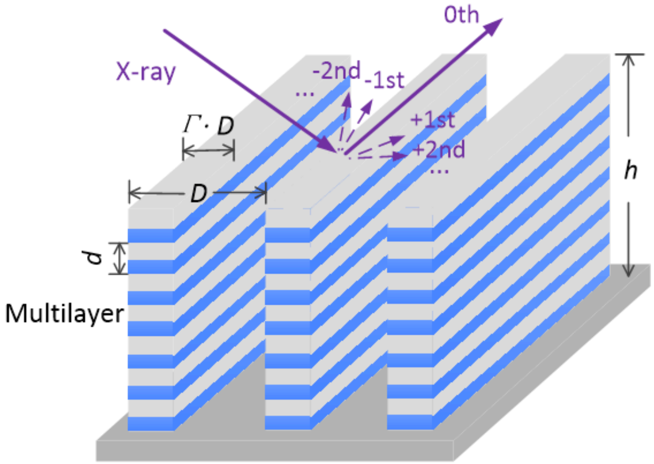
$$y = P_1 \cdot P_2 \cdot P_3$$

$$P_1 = \frac{\sin(\pi j\gamma)}{\pi j(\gamma+g)}; \quad P_2 = \frac{2\sqrt{\sin\theta_0 \sin\theta_n}}{\sin\theta_0 + \sin\theta_n}; \quad P_3 = \left|\frac{\sin[\pi\Gamma(n+jD\sin\alpha/d)]}{\pi\Gamma(n+jD\sin\alpha/d)}\right|$$

Excite single diffraction order with maximum efficiency!

X. Yang, I. Kozhevnikov, Q. Huang, Z. Wang, J. Opt. Soc. Am. B, 32, 506 (2015)

5. Deep etched lamellar ML grating



单级次多层膜层状光栅（工作于0级次）

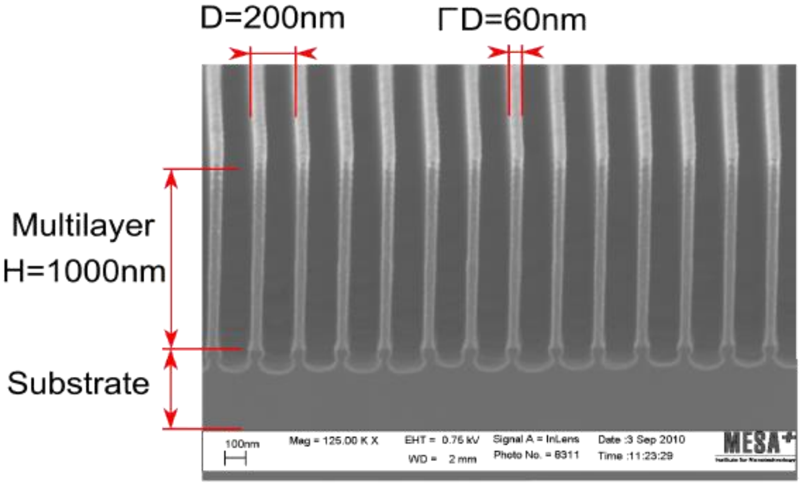
Single order Lamellar Multilayer Grating

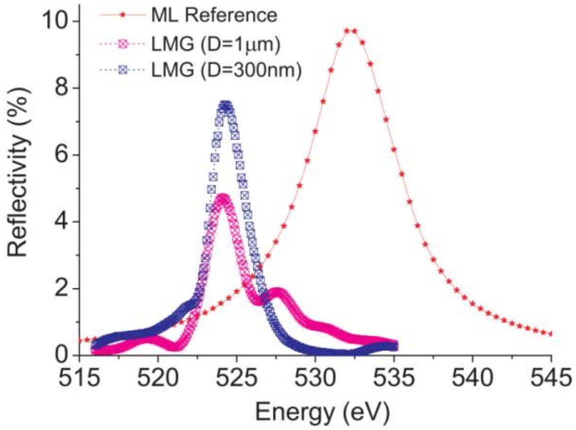
- Reduced X-ray absorption

- Larger penetration depth

✓ Narrow band width, higher resolution

$$\Delta E_{LMG}=\Gamma^{*}\Delta E_{ML}$$



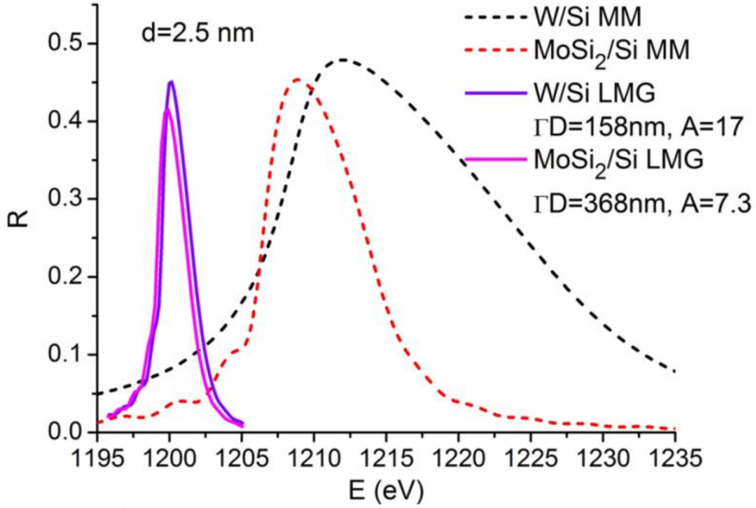


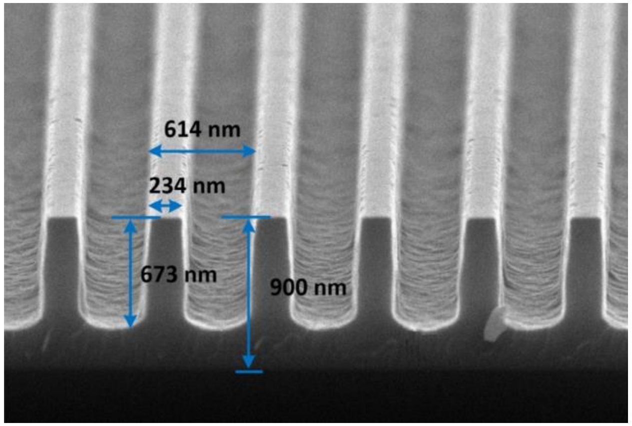
R. Van der Meer, et al., AIP Advances 3, 012103 (2013)

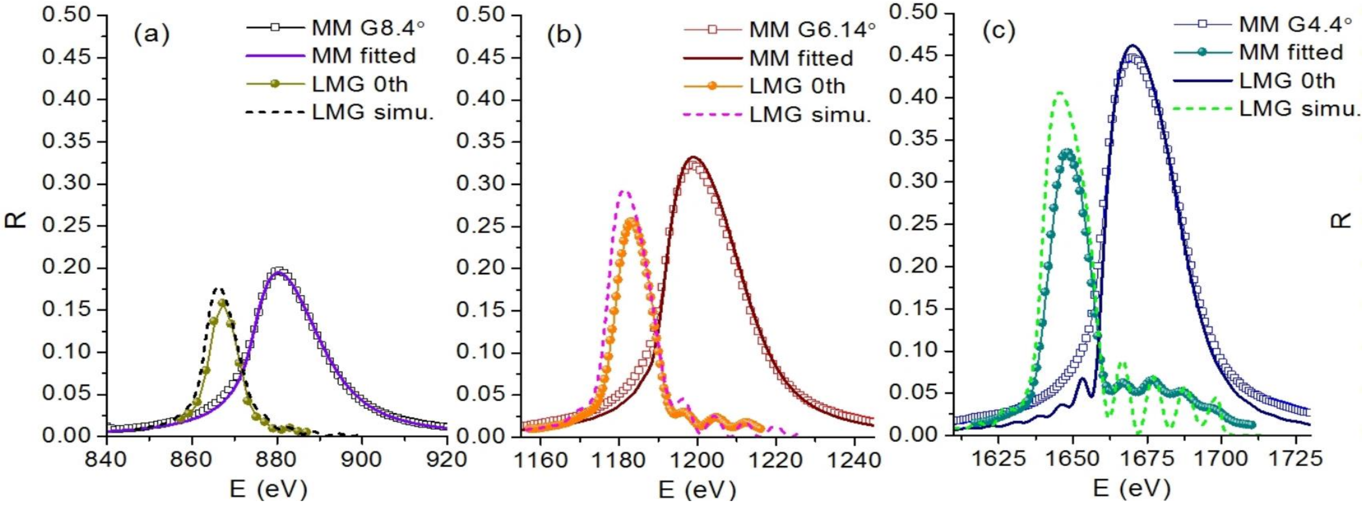
5. Deep etched lamellar ML grating

DLMG using low optical contrast materials

– easier fabrication / higher resolution



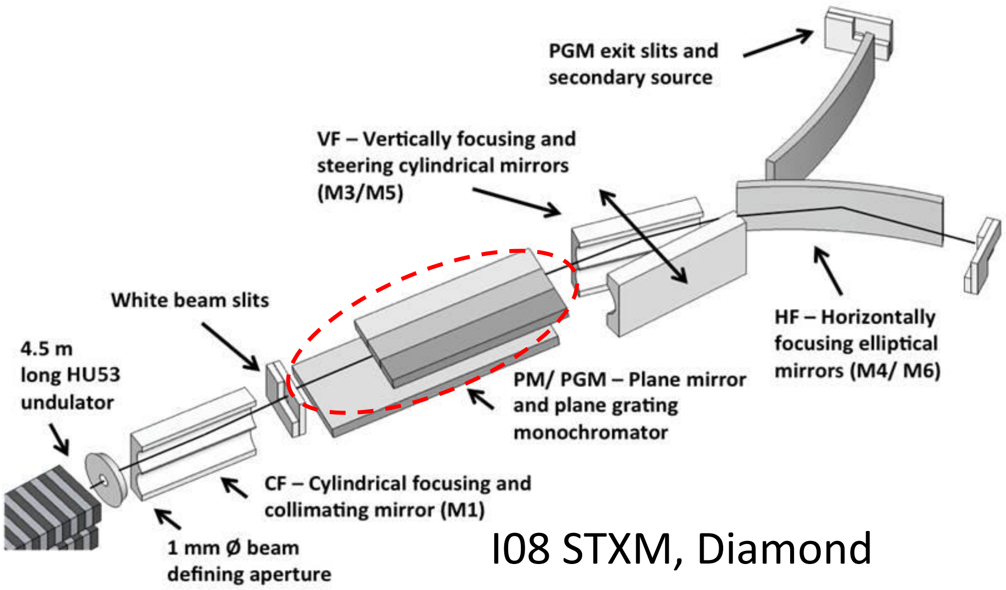




Huang et al., J. Phys. D: Appl. Phys. 52, 195303 (2019)

5. High efficiency tender X-ray grating

Monochromators for SR		
	Cyrstal	grating
Energy range	E>2keV	E<2keV
E=1keV – 4keV ?		



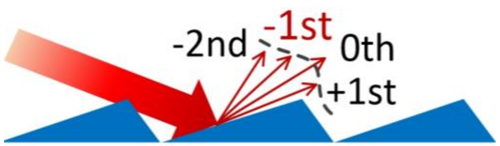
I08 STXM, Diamond

Low efficiency 10%~5%

Flux limited resolution!

5. High efficiency tender X-ray grating

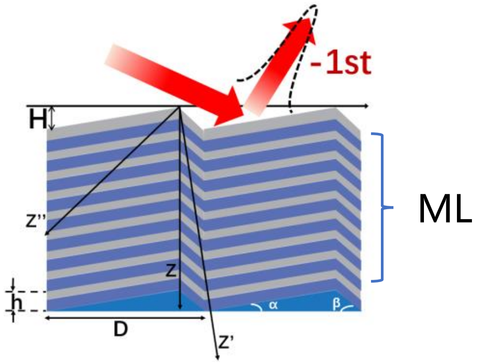
Single layer grating



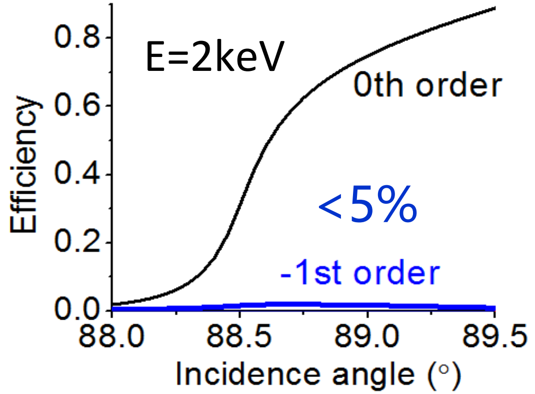
+

ML

ML coated grating

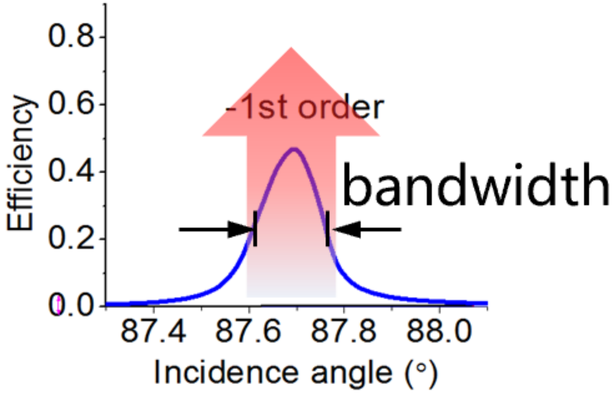


Multi-order low Efficiency



Single order – High efficiency

Resonant efficiency>40%

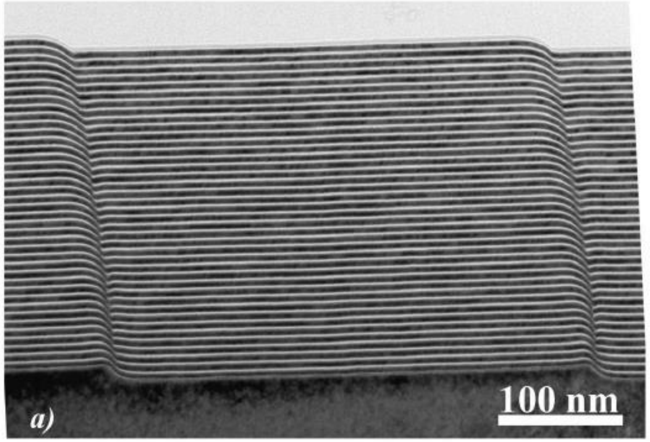


Q. Huang, Igor. Kozhevnikov, et al., Optics Express 28, 821 (2020)

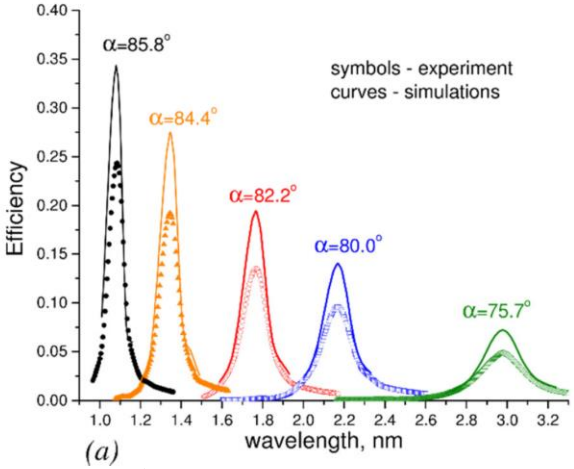
5.

High efficiency tender X-ray grating

-1st efficiency = 24% @ ~1.2keV

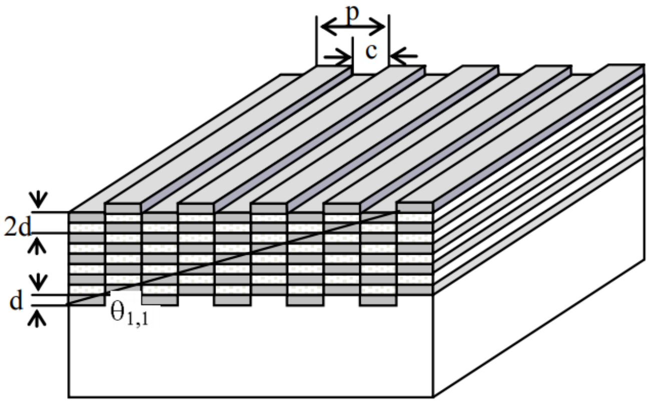


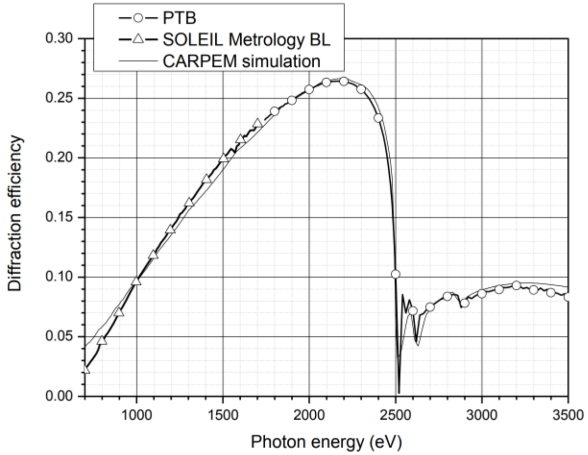
Voronov et al, Opt. Lett. 39, 3157 (2014).



Voronov et al, Opt. Express 24, 11334 (2016).

-1st efficiency = 27% @ 2.2keV





F. Choueikani, Opt. Lett. 39(7), 2141 (2014)

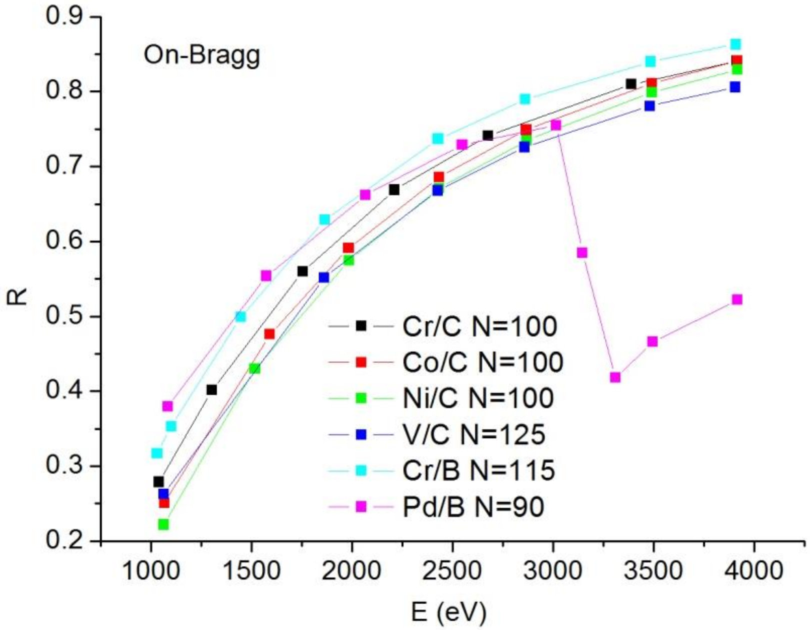
5. High efficiency tender X-ray grating

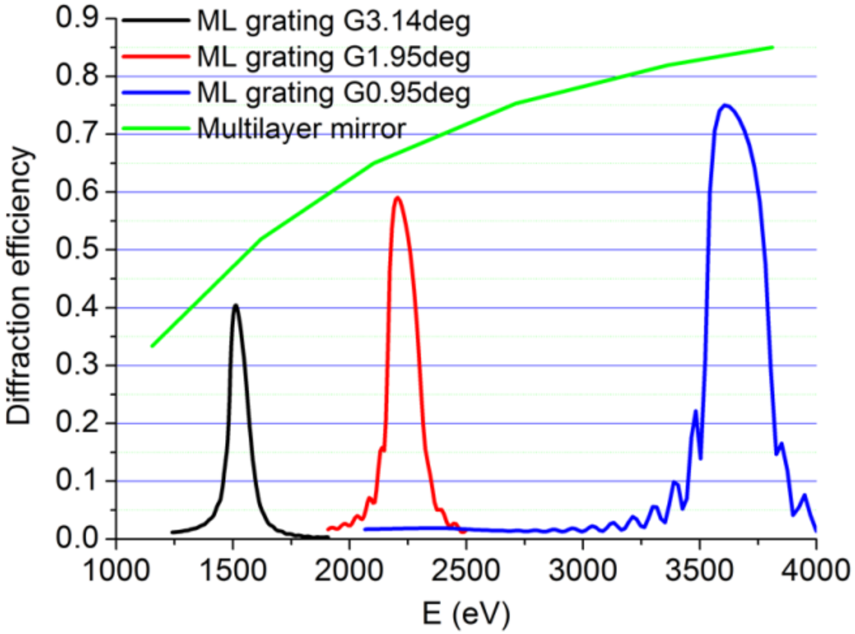
IPOE + HZB Helmholtz Zentrum Berlin + UNIVERSITY OF TWENTE. XUV

Cr/C multilayer

Ideal 1st order efficiency = 40%-70%

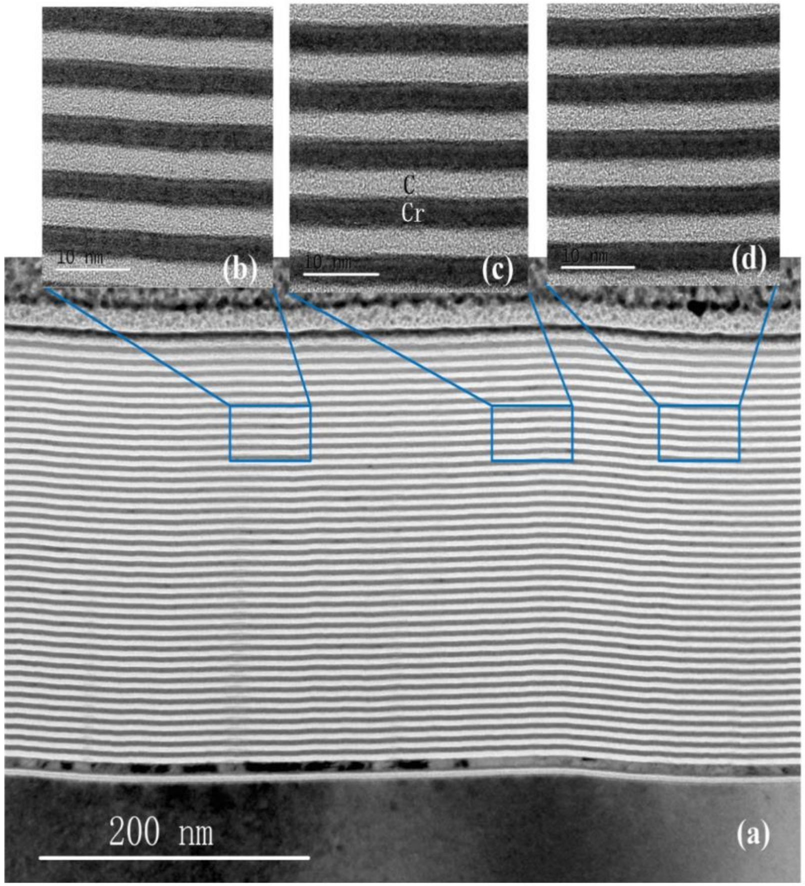
2000l/mm, BA=~0.84deg

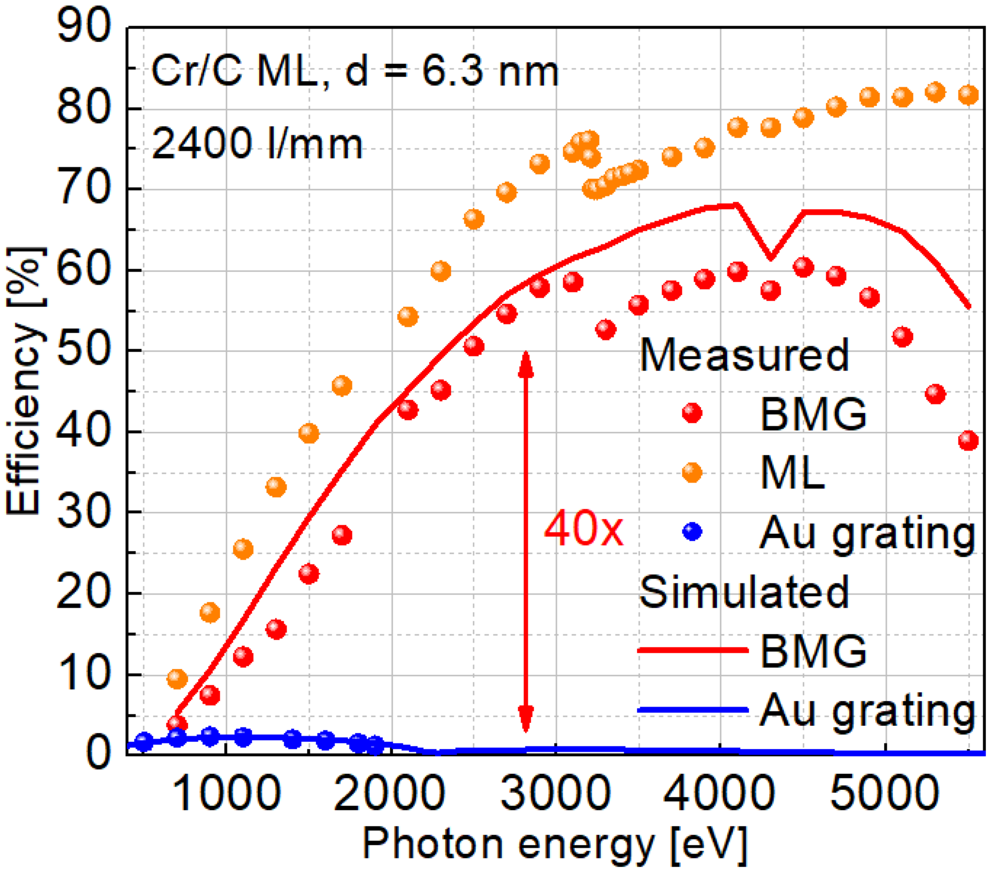




5. High efficiency tender X-ray grating

2400l/mm Cr/C BMG , -1st order efficiency = 60% @ 3keV !

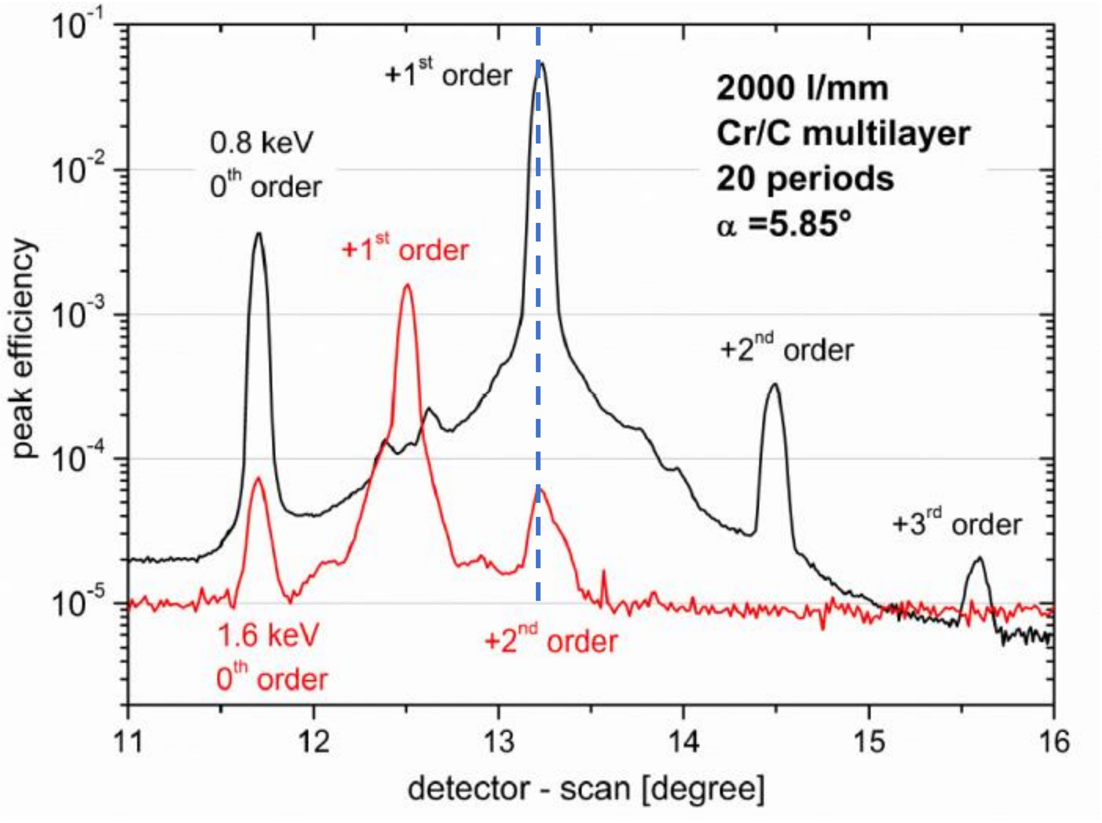




Q. Huang, A. Sokolov, et al., Optics Express 27, 16833 (2019)

High efficiency tender X-ray grating

High harmonics suppression, due to the mismatch of the high order diffraction condition



F. Senf, Q. Huang, et al. Opt. Express, 24, 13220 (2016)

High resolution

Traditional gratings

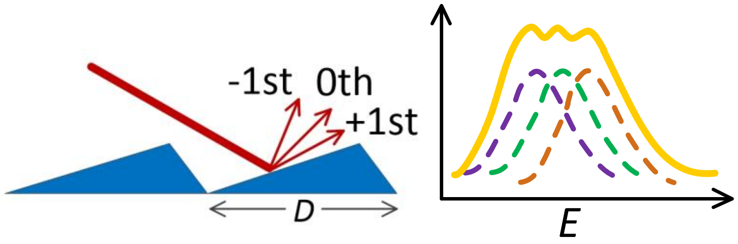
(D>300nm)

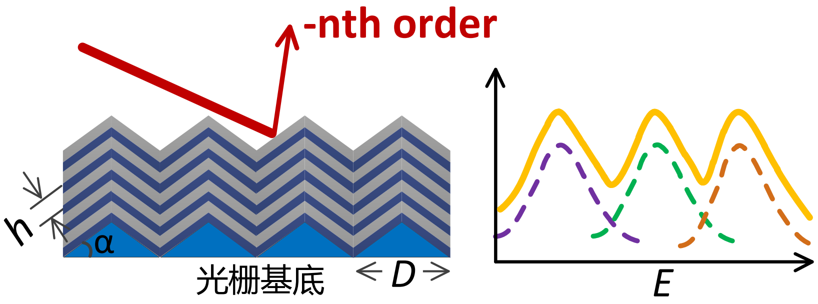
Low dispersion & resolution

ML nanograting

(D=50nm)

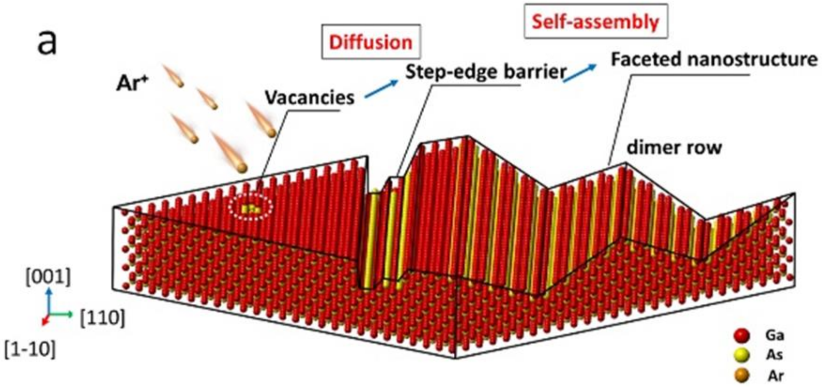
High dispersion & resolution





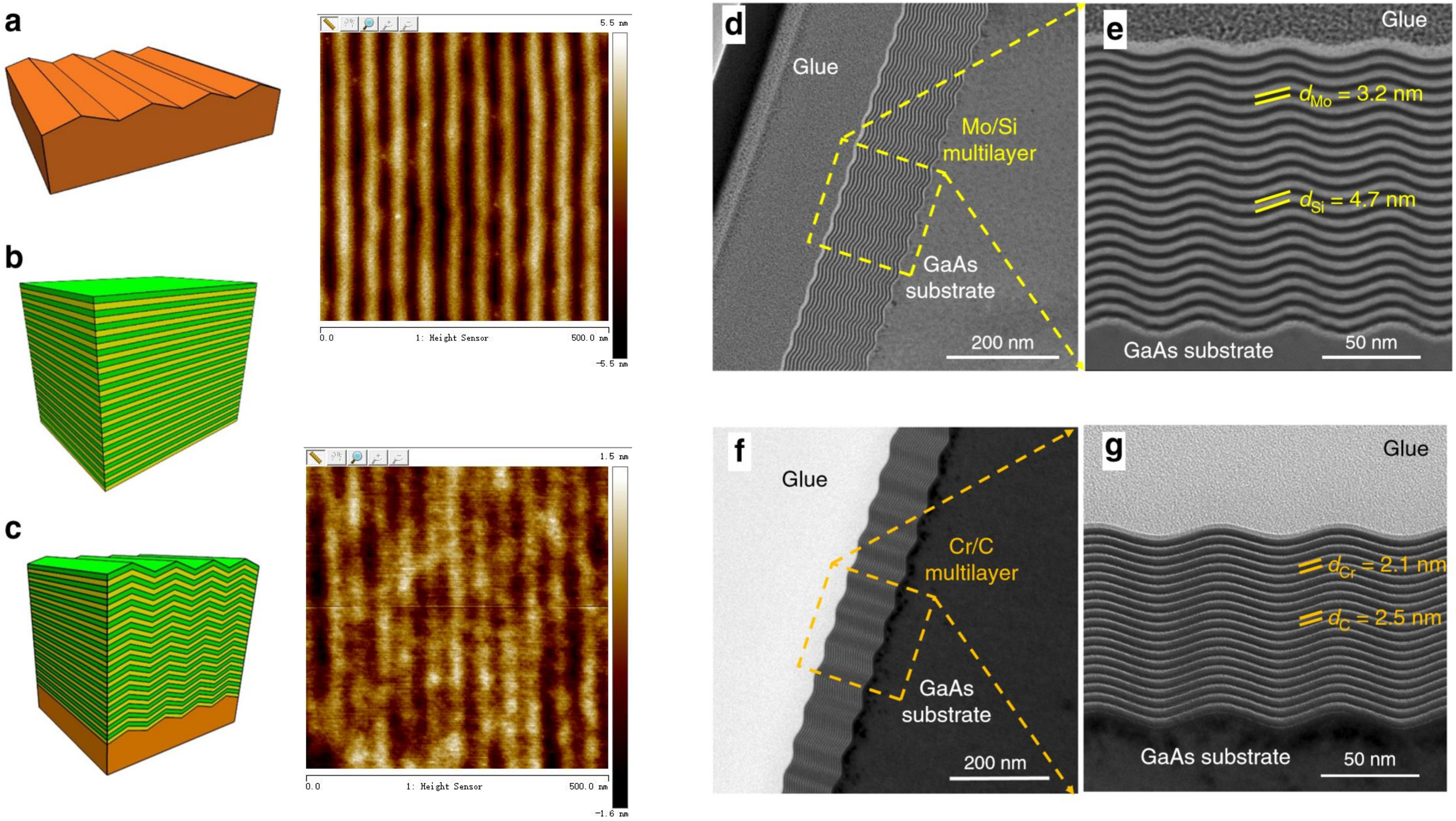
Self-assembly based on ion bombardment

- Ultrashort period grating structures



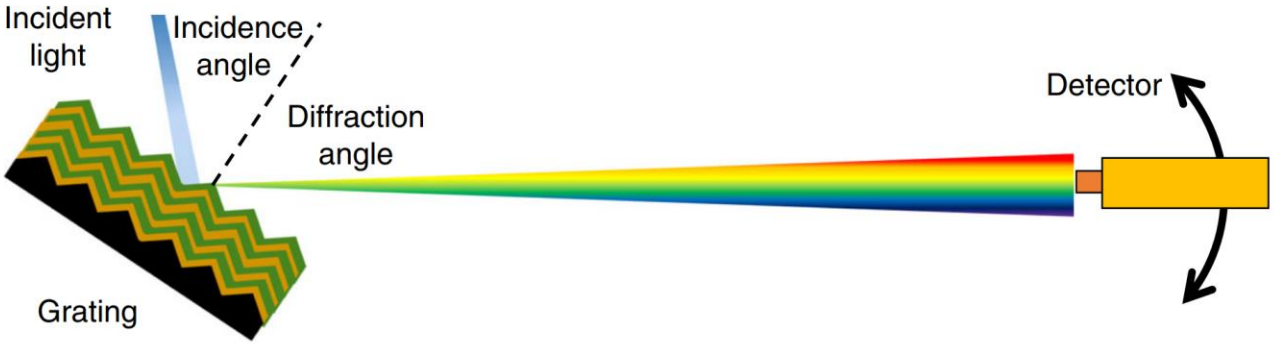
Xin Ou, et al, Phys. Rev. Lett., 111, 016101 (2013)

Conformal growth of ML on nanostructures – forming three dimensional ML gratings



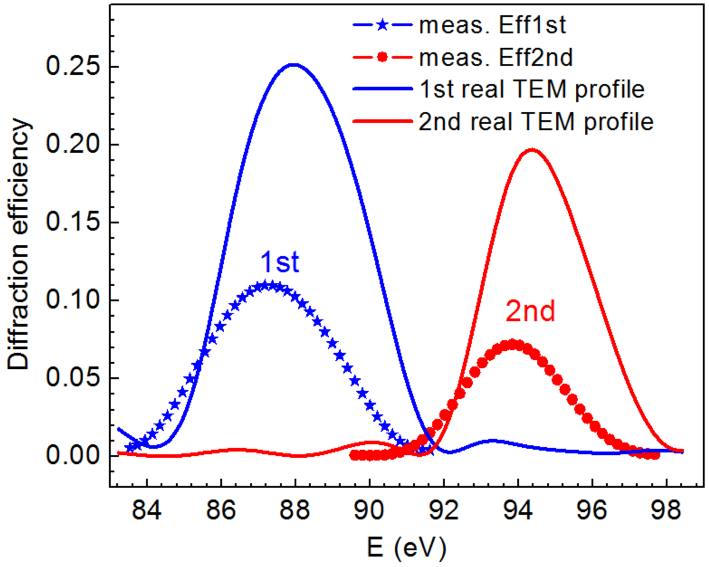
Q. Huang, Xin Ou, et al., Nature Communications, 10, 2437 (2019).

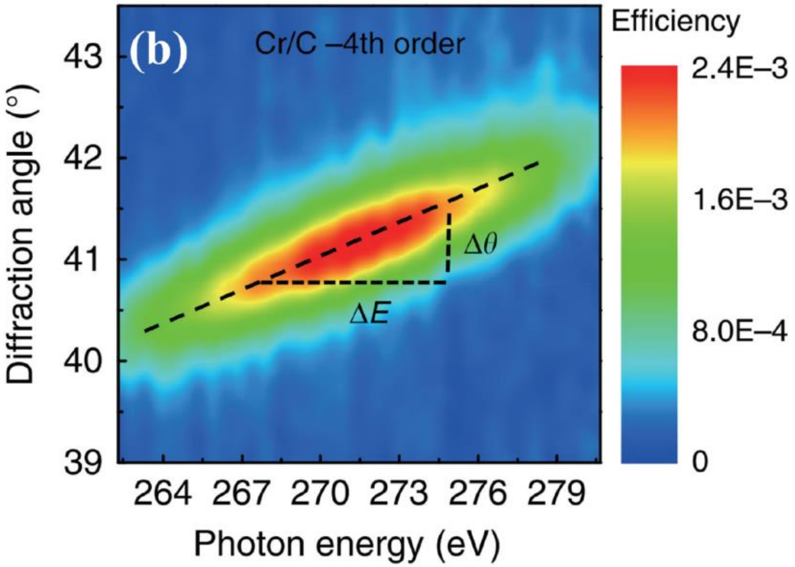
High resolution



1st order Efficiency = 11%

Angular dispersion@270eV =0.093°/eV





Q. Huang, Xin Ou, et al., Nature Communications, 10, 2437 (2019).

XUV multilayers

1. Design of XUV multilayers – periodic / aperiodic structures

2. Fabrication of nanometer ML

3. Stability under working conditions

Understand the layer growth behavior & interaction between materials at atomic level

⬇

interface / surface engineering - improve interface quality & stability

4. Large scale fabrication – real optics – thickness control

5. Nanostructured ML optics – 1D -> 3D

Acknowledgement

SSRF， NSRL, BSRF
China Academy of Engineering Physics

Shubnikov Institute of Crystallography, Russia

HZB Helmholtz Zentrum Berlin
(Institute for Nanometer Optics)
(BESSY-II)

SIMIT
微系统所

diamond
Optics Group

UNIVERSITY OF TWENTE.
XUV

Elettra Sincrotrone Trieste
BEAR beamline

Thank you for your attention!