wissen schafft nutzen

GKSS FORSCHUNGSZENTRUM in der HELMHOLTZ GEMEINSCHAFT

Single layer mirrors and multilayer mirrors for current and next-generation light sources

Michael Störmer

GKSS-Forschungszentrum Geesthacht GmbH
Max-Planck-Strasse 1
21502 Geesthacht

HELMHOLTZ | ASSOCIATION

GKSS research centre

Member of the Helmholtz Foundation (HGF)

700 employees: 4 research fields
150 people from the Institute of Materials Research

Neutron Research Reactor
FRG-1

HGF Research Fields:	Core Programme:
Key Technologies	**Advanced Engineering Materials**
Structure of Matter	**Research with Photons, Neutrons and Ions (PNI)**
Health	Regenerative Medicine
Earth and Environment	Coastal Dynamics and Causes of Changes

ASSOCIATION

Organigram:

Program PNI-GKSS

Speaker: Prof. Dr. A. Schreyer

Institute of Materials Research
Materials Physics (WP)

Department "Structural Research on New Materials" (WPN)
Prof. Dr. M. Müller

Department "Structural Research on Macromolecules" (WPS)
PD Dr. R. Willumeit

TU Clausthal (TUC), TEXMAT
Prof. Dr. H.-G. Brokmeier

Subtopic "Neutron and Photon Research on Engineering Materials and Components"

Subtopic "Neutron and Photon Research on Nanostructures"

Nanotechnology (WTN)
Dr. M. Störmer

Outstation DESY, Hamburg

- **HARWI II@DORIS** (**WPN**, Dr. T. Lippmann)
- **BW2 (Tom.)@DORIS** (DESY, **WPN**, Dr. F. Beckmann)
- **HEMS@Petra III** (**WPN**, Dr. N. Schell)
- **Imaging Beamline@Petra III** (**WPN**, Dr. A. Haibel)
- **Biosaxs Beamline@Petra III** (EMBL, **WPS**, Dr. V. Haramus)

Geesthacht Neutron Facility GeNF

- **SANS-1** (**WPS,** Dr. R. Willumeit, Dr. V. Haramus)
- **SANS-2** (**WPN**, Dr. M. Sharp, H. Eckerlebe)
- **DCD** (**WPN**, Dr. M. Sharp)
- **ARES-2** (**WPN**, Dr. P. Staron)
- **NeRo** (**WPN**, Dr. D. Lott)
- **PNR** (**WPN**, Dr. D. Solina)
- **Genra-3** (**WPN**, Dr. P.K. Pranzas)
- **POLDI** (**WPN**, Dr. D. Lott, J. Fenske)
- **TEX-2** (**TUC**, Prof. Dr. H.-G. Brokmeier)
- **ICI** (**RB**, Dr. W. Knop)

Outstation FRM II, Garching (near Munich)

- **REFSANS** (**WPN**, J.-F. Moulin)
- **SANS-1** (FRM II, **WPN**, N.N.)
- **STRESS-SPEC** (FRM II, HMI, TUC, **WPN**, Dr. W. Gan)

Research Reactor (R)
Dr. P. Schreiner

Reactor Operations (RB)
Dr. W. Knop

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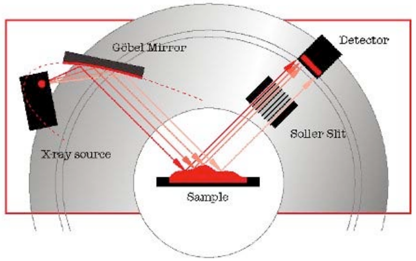
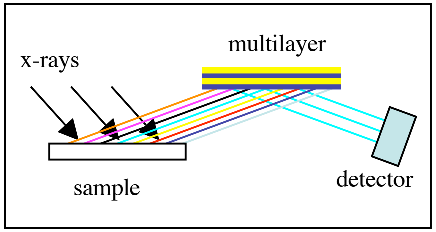
Department Nanotechnology
Group: Thin film technology

GKSS FORSCHUNGSZENTRUM in der HELMHOLTZ GEMEINSCHAFT

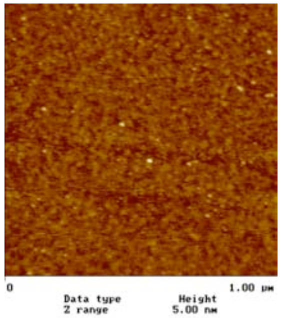
XRR



XRF



AFM



Carbon Coating on Silicon Substrate

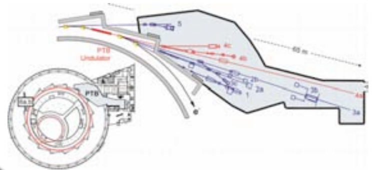
Multilayer preparation: Magnetron-Sputtering

HASYLAB

Inst. of Physics
Academy of Sciences
Prag

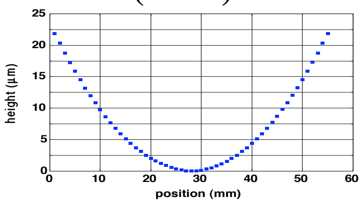
BESSY

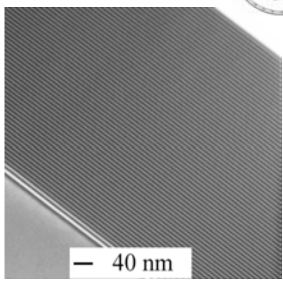
Soft X-ray beamline



PTB

Profilometry (AKF)





TEM (Uni Kiel)

La/C

- **Research in the field of X-ray optics for different wavelengths**
- **Large number of experimental methods**
- **Characterization of thin films: single layer mirrors and multilayer mirrors**
- **Previous focus: mirrors for lab equipment => 2002: foundation of a company: Incoatec**
- **Now: development of new X-ray optics, for instance large FEL optics**

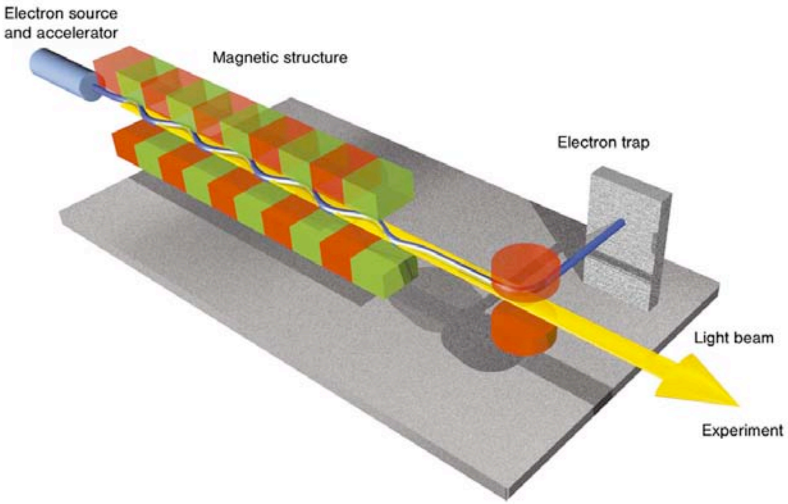
HELMHOLTZ ASSOCIATION

History of the

“GKSS carbon coatings“

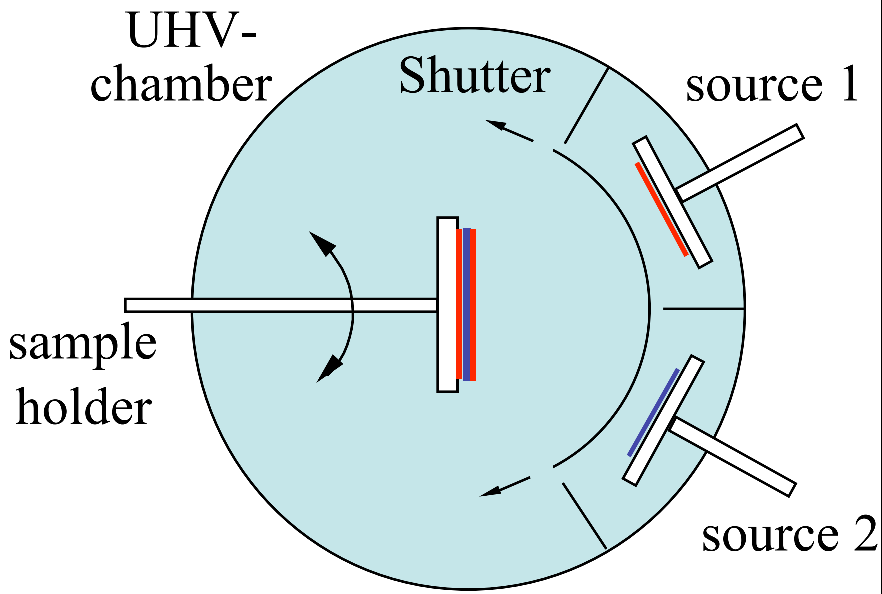
FEL and FLASH Optics

Free-Electron-Laser (FLASH)



Tasks of the Mirrors:
- Beam Guidance
- Beam Alignment
- Monochromatisation

Magnetron sputtering system



working pressure: 0.1 - 0.5 Pa

Ar gas (purity: 99.9999%)

Development of Mirrors for the “Light of the Future“ successful

Contract with Incoatec: collaboration in the field of nanostructures

Carbon Coatings: Energy-Dependent Reflectivity

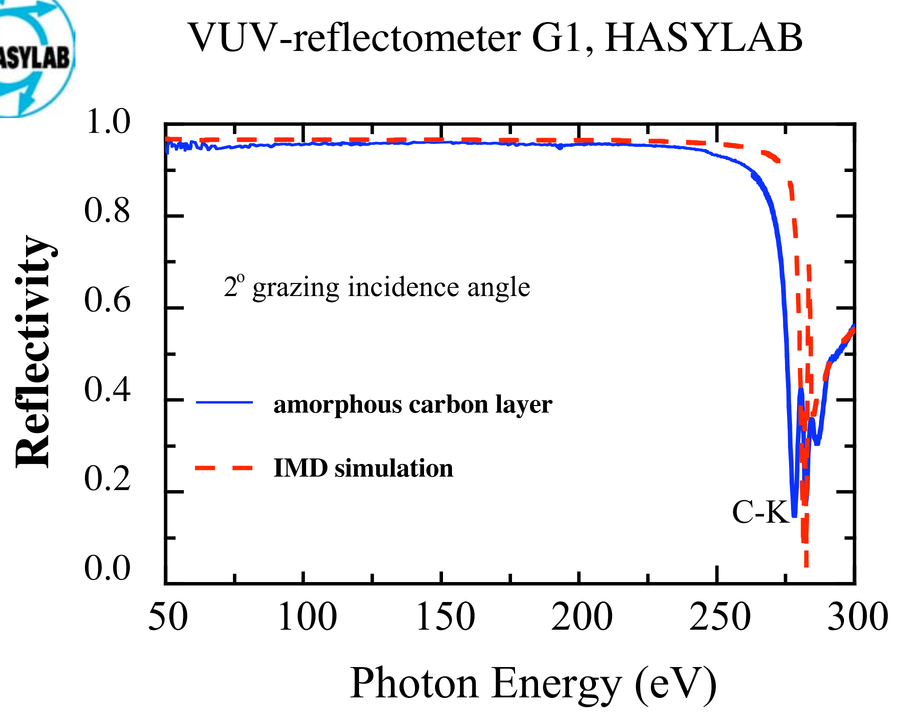
low energy range of 50-200eV (FLASH)
=> elements with a low atomic number

Carbon: absorption edge C_K 284 eV

Measurement: 40 nm C-coating

IMD Simulation:
carbon layer, density of diamond, zero roughness

(IMD -Software for modeling the optical properties of multilayer films, Windt, Computers in Physics, 12 (4) 1998)



Between 50 - 250 eV at 2°: R(E) > 95 %

(Jacobi et al. SPIE Proc. 4700, 2001)

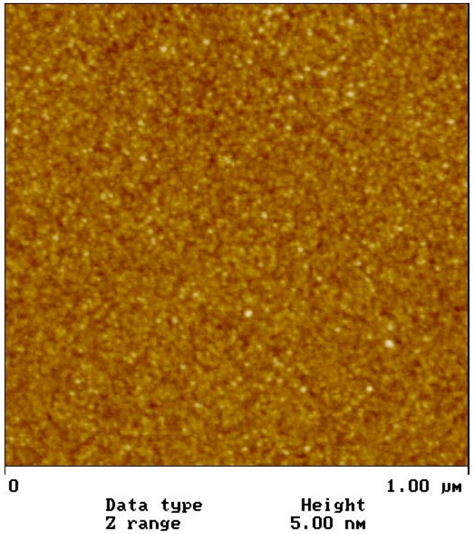
→ Reflectivity is near the reachable theoretical reflectivity of 96.5%
→ Differences in the region of the absorption edge

a-C Films: Atomic Force Microscopy (AFM)

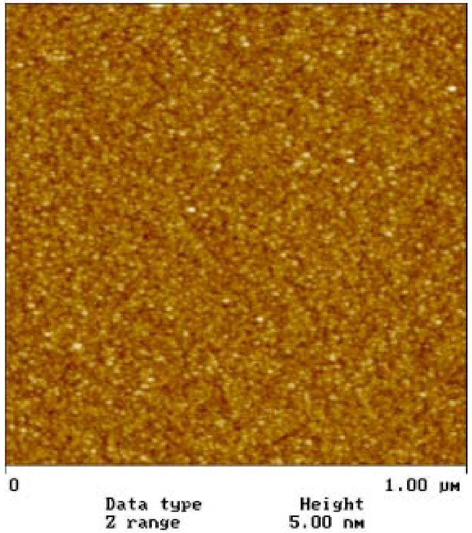
Before deposition:
≈ 0.3 nm

typical of pure, well-polished silicon

in the centre:



close to edge:



area:
1µm x 1µm
(Felten, TU HH)

Noise level: 0.9Å

	Position	RMS roughness (Å)		
5 cm:	Left edge	4.16	3.97	4.10
25 cm:	In the centre	3.73	3.71	3.86
45 cm:	Right edge	3.62	3.72	4.28

After deposition: - mean rms roughness ≈ 0.4 nm
- no difference in roughness over the whole length of the mirror

→ precise deposition process for large X-ray optics

M. Störmer, D. Häußler, W. Jäger, R. Bormann,
Large X-ray Optics: Fabrication and Characterization of Single and Multilayer Mirrors,
2007 Sino-German High Level Expert Symposium on X-ray Optics,
Optics and Precision Engineering 15(12) (2007) 1869-1877. ISSN 1004-924X.

Cylindrical and toroidal substrates

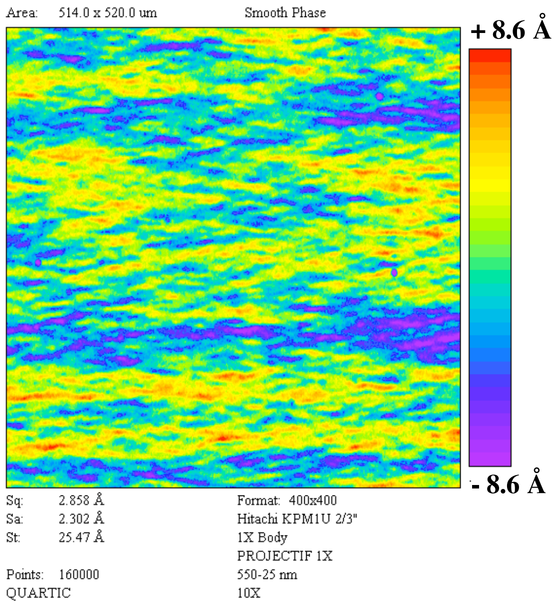
shaped surfaces:

radius of curvature

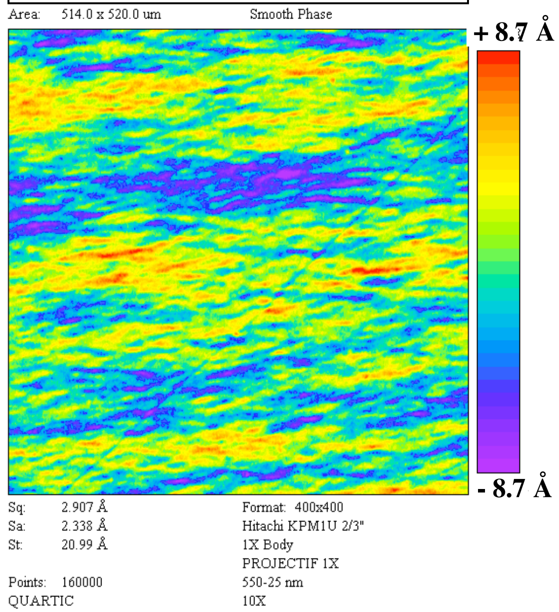
some tens of meters

250 mm long
toroidal silicon substrate
close to its centre:

Before deposition:



After deposition:



Optical profilometry:
area: 514μm * 520μm

A. Liard-Cloup

(Jobin-Yvon)

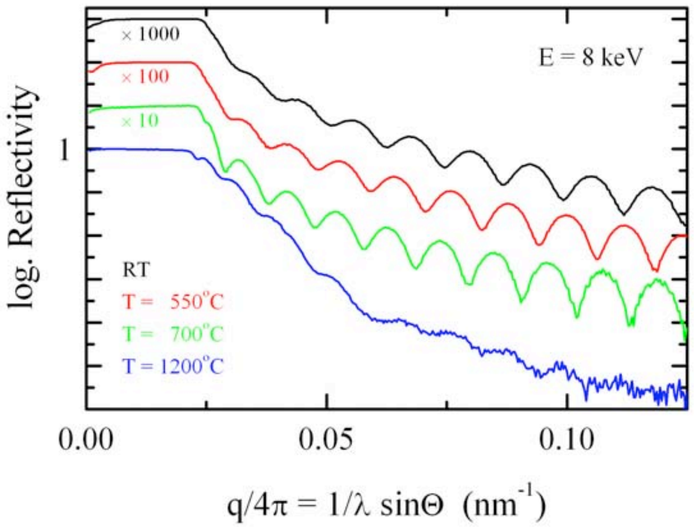
Length	Shape	Observed area	Micro-roughness before deposition	Micro-roughness after deposition
300 mm	cylindrical	centre	3.8 Å	2.7 Å
300 mm	cylindrical	edge	2.3 Å	2.7 Å
250 mm	toroidal	centre	2.9 Å	2.9 Å
250 mm	toroidal	edge	3.7 Å	4.0 Å

Micro-roughness varied from 0.2- 0.4 nm (tolerance: 0.04 nm)

It remained more or less unchanged

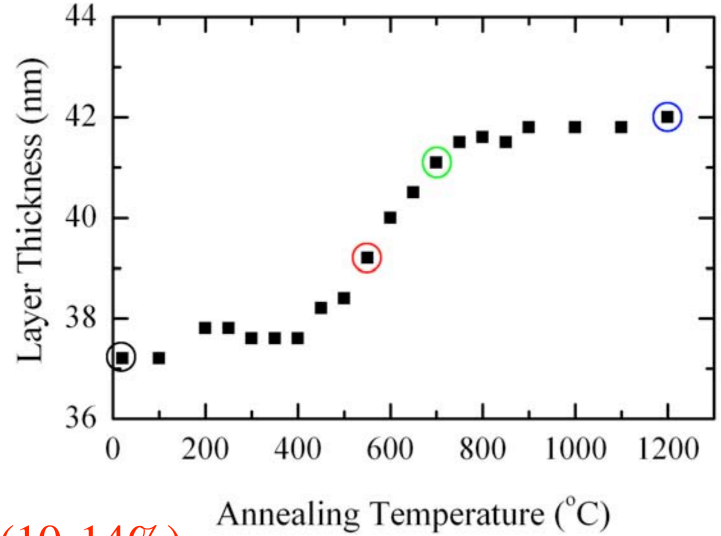
(SPIE Proc. 5533, 2004)

Thermal Stability I



XRR: Cu-K_α (8048 eV)

ex-situ annealing RT - 1200°C
for 80 min (30 min constant)

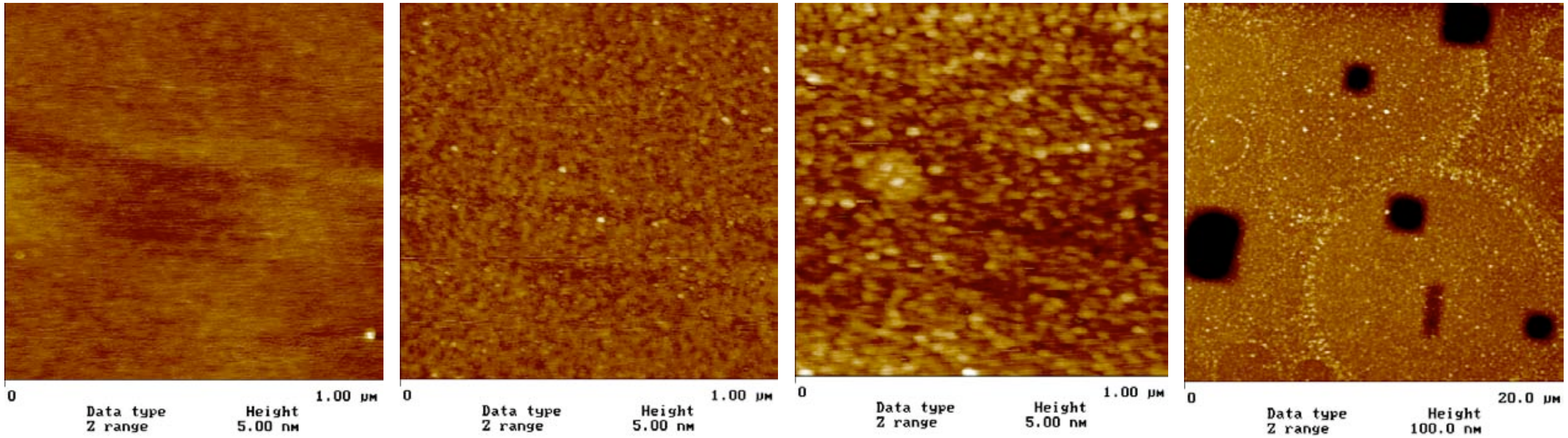


Up to 400°C: film remains constant
400 - 750°C: increase in film thickness (10-14%)
Above 750°C: constant thickness
At 1200°C: layer structure is destroyed

(Jacobi et al. SPIE Proc. 4782, p. 113, 2002)

Mirrors are cooled in the FEL
→ **Good thermal stability under application conditions**

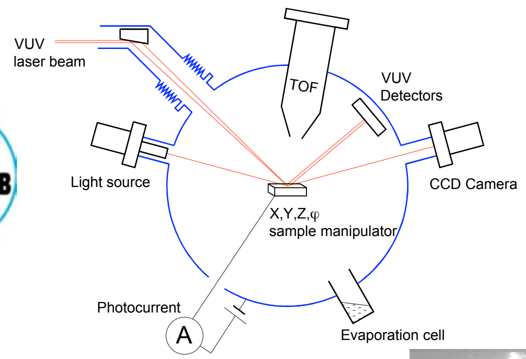
Thermal stability II: AFM



Silicon-Substrate (RT)	C-Film on Silicon (RT)	C-Film on Silicon (1000°C)	C-Film on Silicon (1200°C)
$R_{q,rms}$ = 0.23±0.04 nm	0.24±0.02 nm	0.43±0.18 nm	5.66 ±0.73 nm

- **Roughness unchanged after film deposition**
- **Roughness increases during heating**
- **After 1000°C: surface roughness is still smaller than 0.5 nm** → **R(E) ≈ 87%**
- **After 1200°C: holes in the film** → **layer structure destroyed**

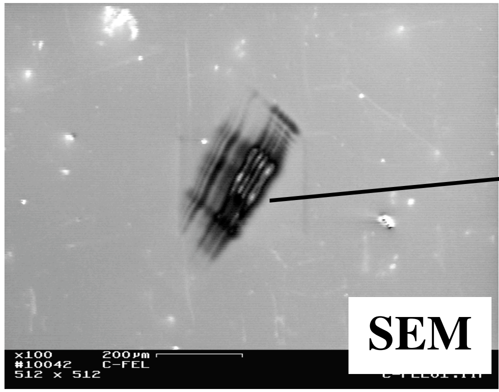
Radiation Stability

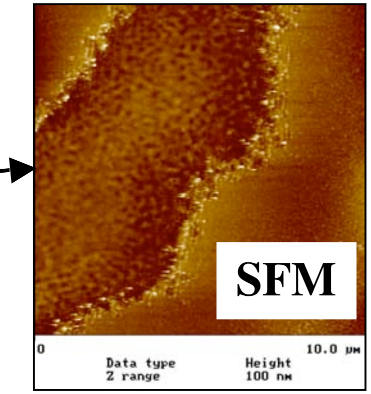


FELIS: Free-Electron-Laser-Interaction with Solids
(R. Sobierajski, J. Krzywinski, HASYLAB)
wavelength: 98 nm (E=12.7 eV)
Pulse length: 100 fs
Pulse energy: 40 μJ

damage threshold: 0.06 J/cm^2

irradiated perpendicular



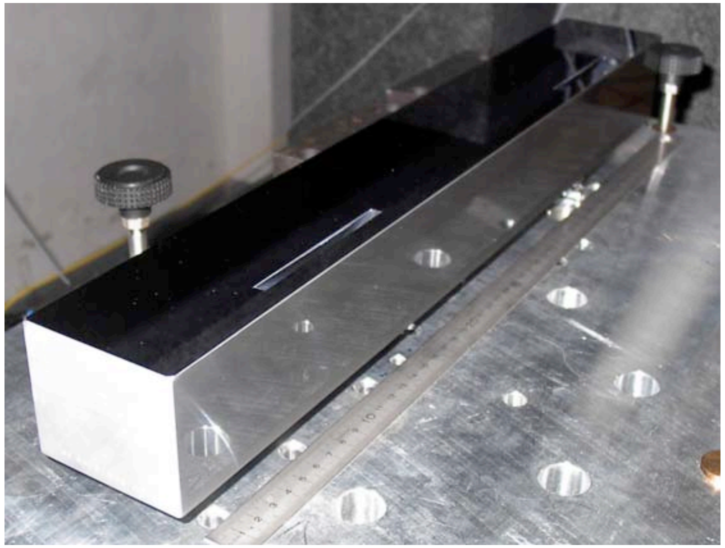


(B. Steeg et al. APL 84, 2002, 657)

(L.Juha et al. SPIE Proc. 5917, 59170F, 2005
L. Juha et al. SPIE Proc. 6586 65860D, 2007)

Mirrors: stable under FLASH radiation:
1. incidence angle: 2° => irradiation under grazing incidence!
2. photon energy: 200 eV => absorption is by two orders of magnitude lower!

510mm plane mirror for FEL-beamline at FLASH / DESY



Measurement result for center line:

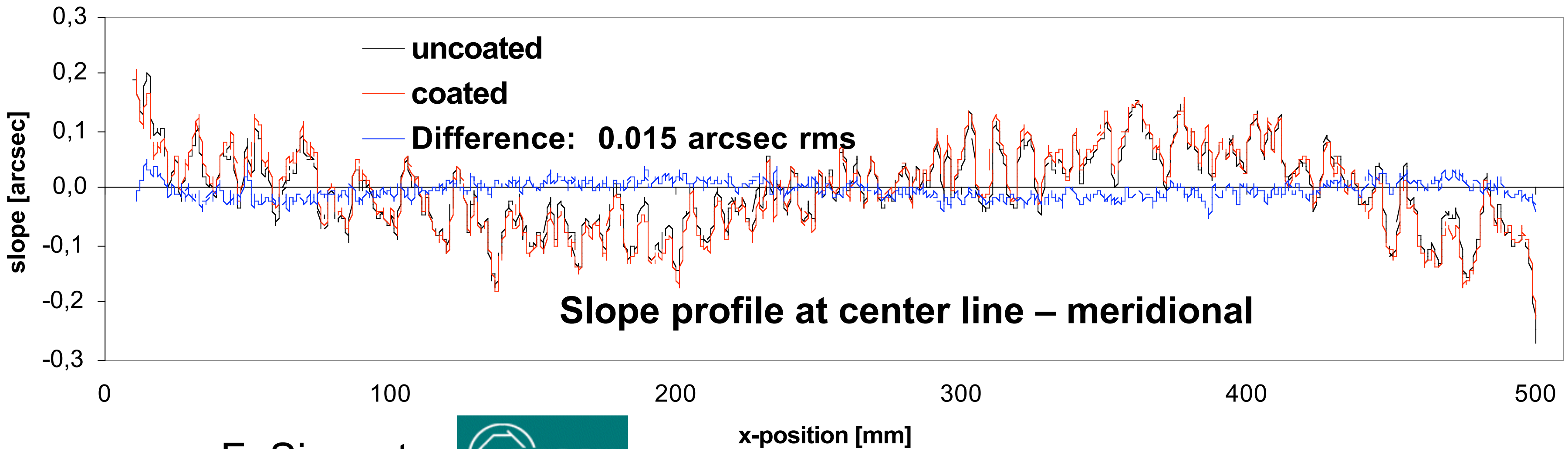
Residual slope - uncoated: 0.33 µrad rms

- coated: 0.33 µrad rms

Radius - uncoated: > 450 km

- coated: > 800 km

Excellent stability of shape !!



F. Siewert

510mm plane mirror at FLASH after 3 month in use

Micromap measurements:

On Silicon:

Sq = 0.20 nm rms (20x)

0,28 nm rms (50x)

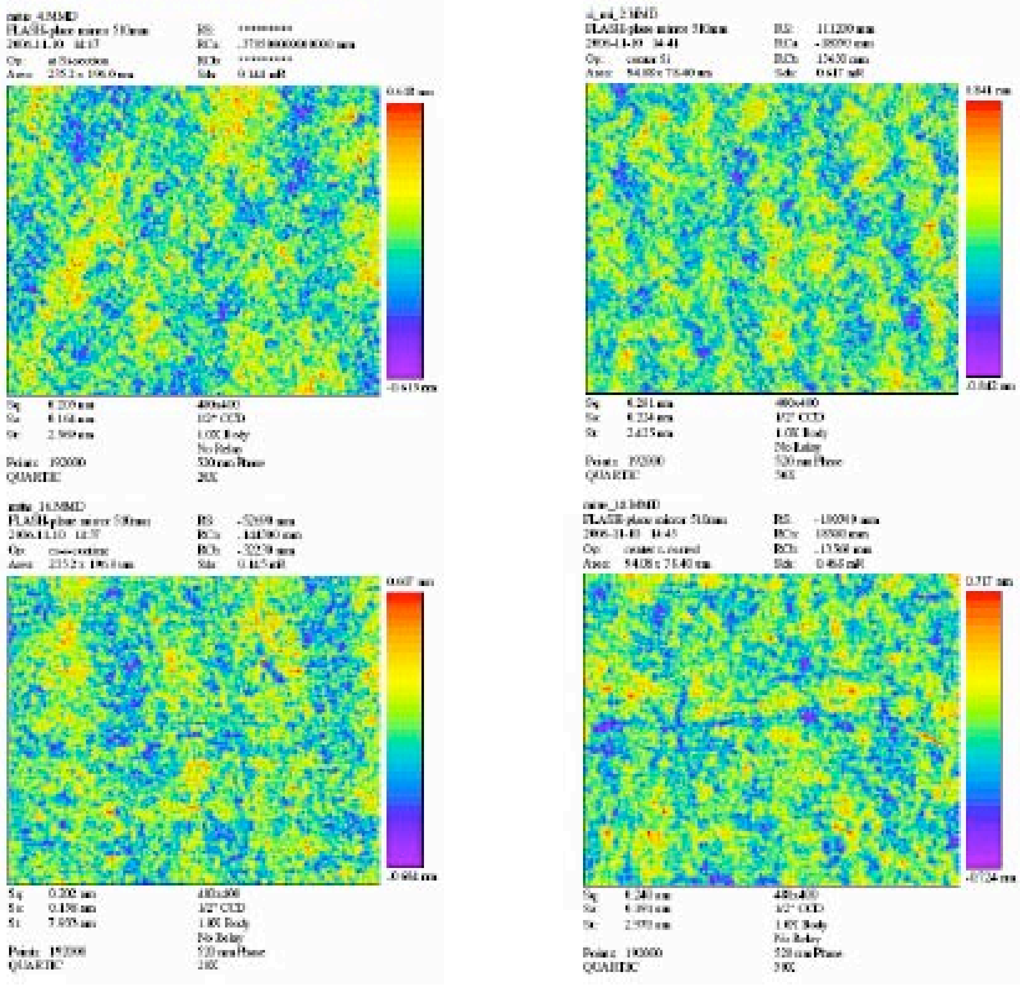
At C-Coating:

Sq = 0.19–0.25 nm rms (20x)

0.18–0.27 nm rms (50x)

No significant damage is found !!!

Magnification: 20x | 50x



F. Siewert

The latest developments:

Single Layer Mirrors and Multilayer Mirrors

+

Deposition length of 1500mm

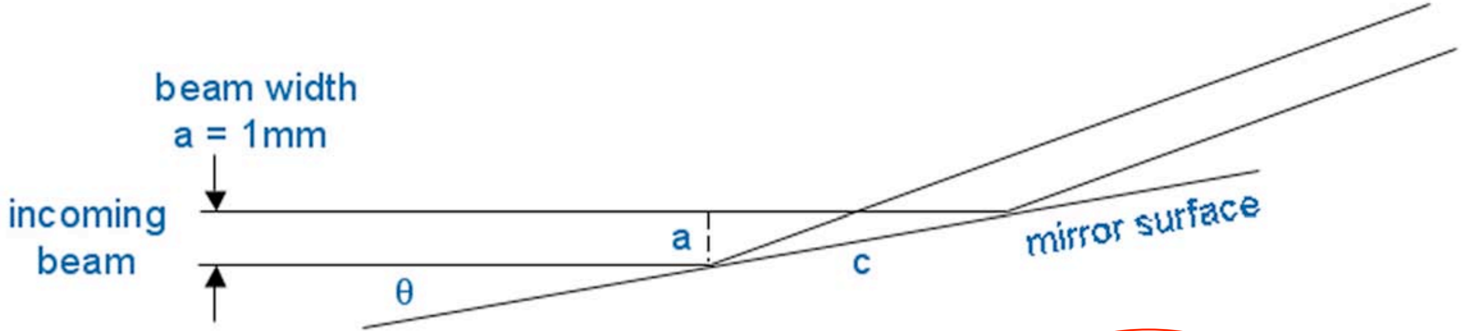
Why is a deposition length of 1.5m needed?

1. X-FEL:

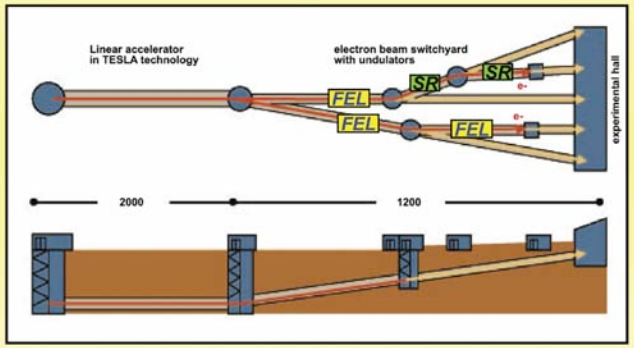
total reflection of a single layer => at 12.4 keV radiation

critical angle of C and W: θ_C = 2.4 and 6.3 mrad

=> incidence angle ≈ 0.05°



$\Rightarrow$ $L > c = a / \sin\theta = 1\text{mm} / \sin 0.05° \approx 1200\text{mm}$



3 FEL and 2 beamlines for spontaneous synchrotron radiation with 10 independent experimental stations

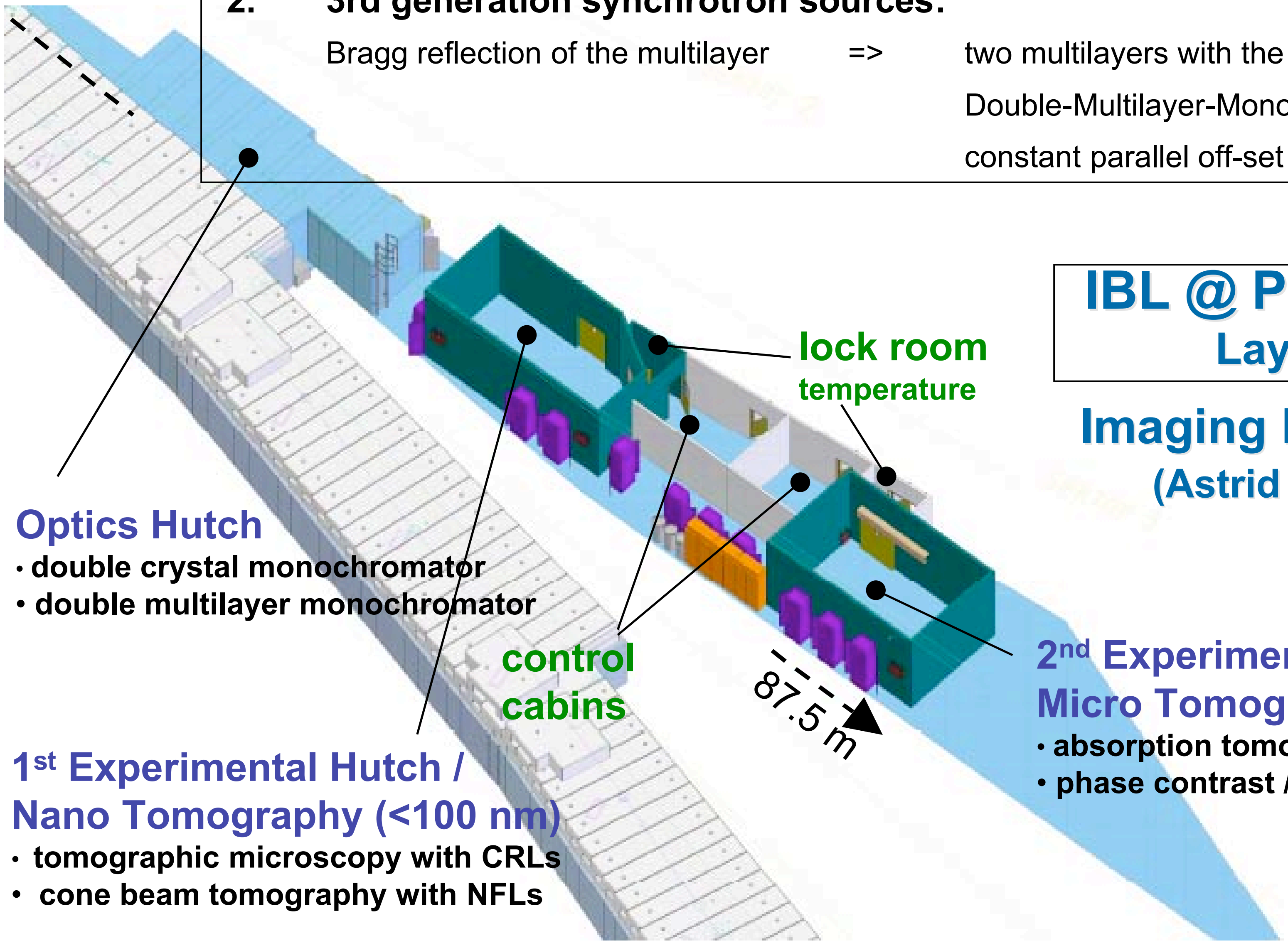
Parallel deposition => 2 identical mirrors

2. 3rd generation synchrotron sources:

Bragg reflection of the multilayer => two multilayers with the same properties!
Double-Multilayer-Monochromator (DMM)
constant parallel off-set

IBL @ PETRA III
Layout

Imaging Beamline
(Astrid Haibel)



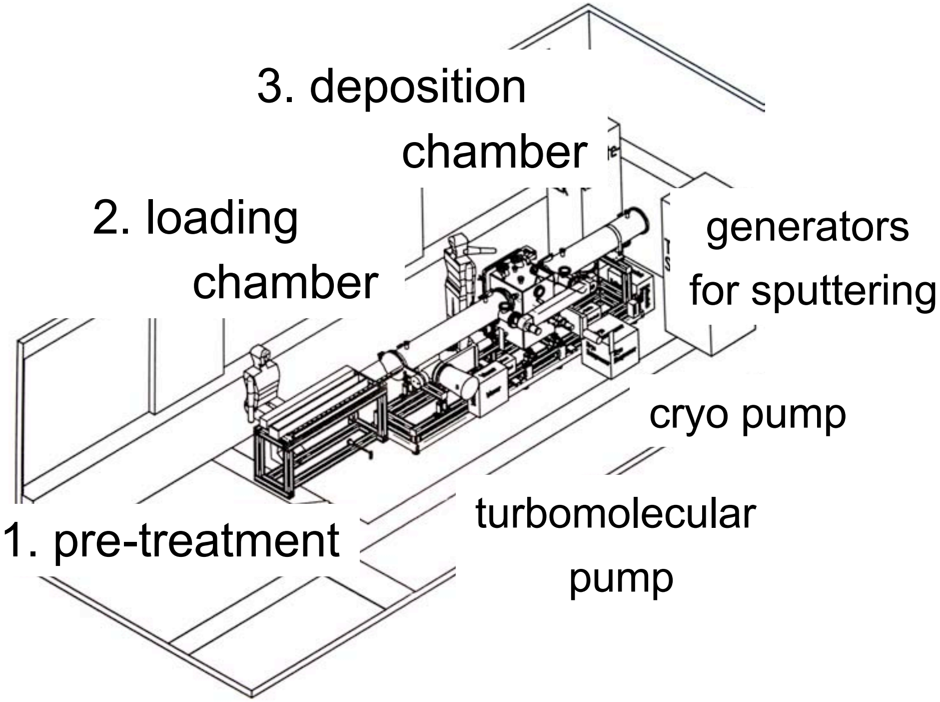
2nd Experimental Hutch / Micro Tomography (< 1µm)
- absorption tomography
- phase contrast / holo tomography

New Sputtering System

Challenge:

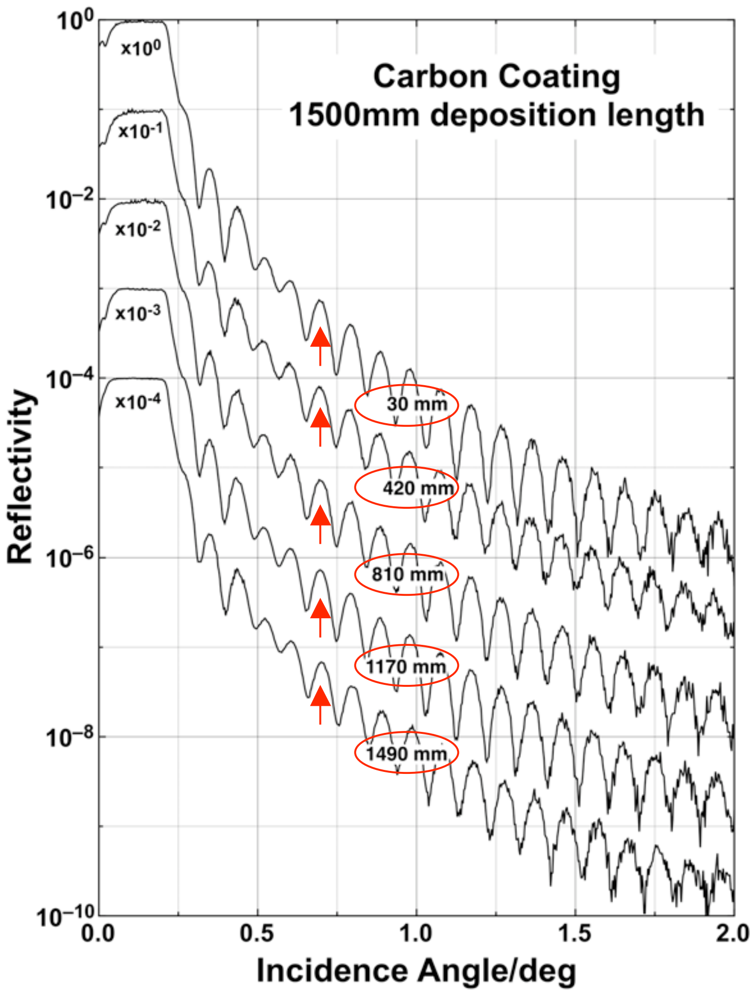
Enlargement of the deposition length
to manufacture

- Single layers and
  Mirror length of 1.5 meters !
- Multilayers:
  Simultaneous deposition of mirror pairs
  to achieve the same properties



M. Störmer, C. Horstmann, D. Häussler, E. Spiecker, F. Siewert, F. Scholze, F. Hertlein, W. Jäger, R. Bormann,
Single-layer and multilayer mirrors for current and next-generation light sources,
Proc. SPIE 7077 (2008) 707705.
http://dx.doi.org/10.1117/12.798895

1. Single layer Mirrors: a-C coatings



- XRR measurements with Cu radiation (lab)
- investigations in tangential direction
- thickness oscillations
- fringe orders at the same angles

<u>IMD simulations:</u>

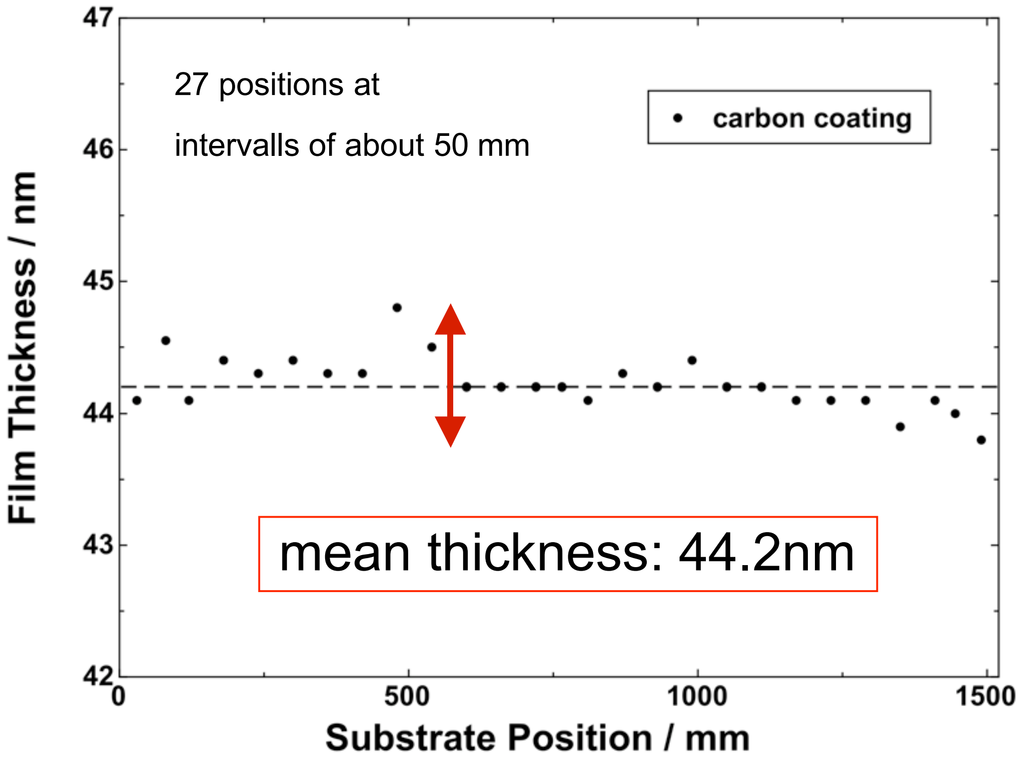
film thickness: 44nm,

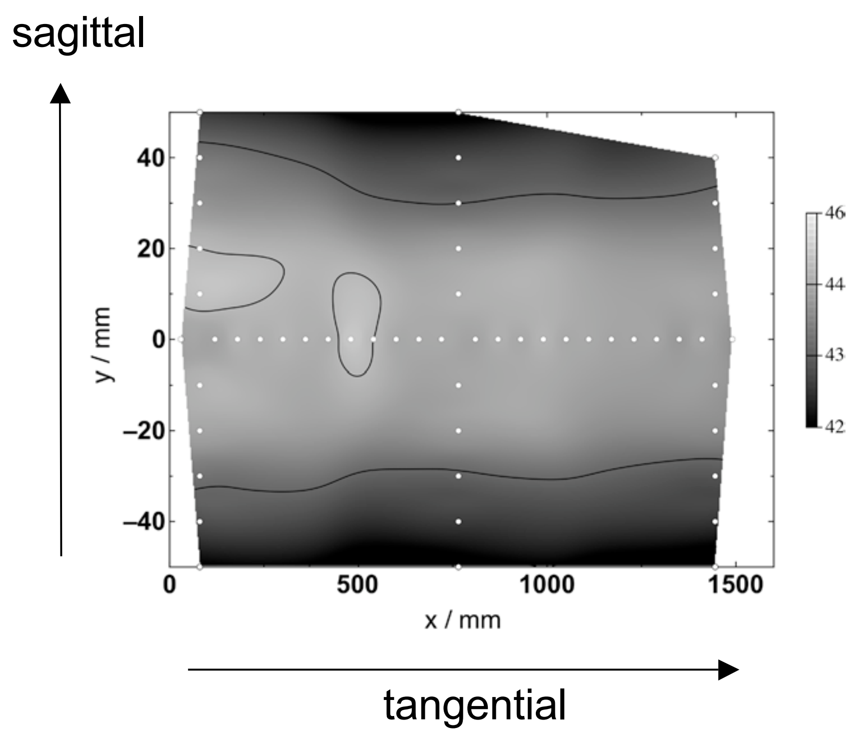
film roughness: 0.5nm,

film density: 2.2 g/cm^3.

High precision of the coating process over a deposition length of 1.5m

Variation in layer thickness



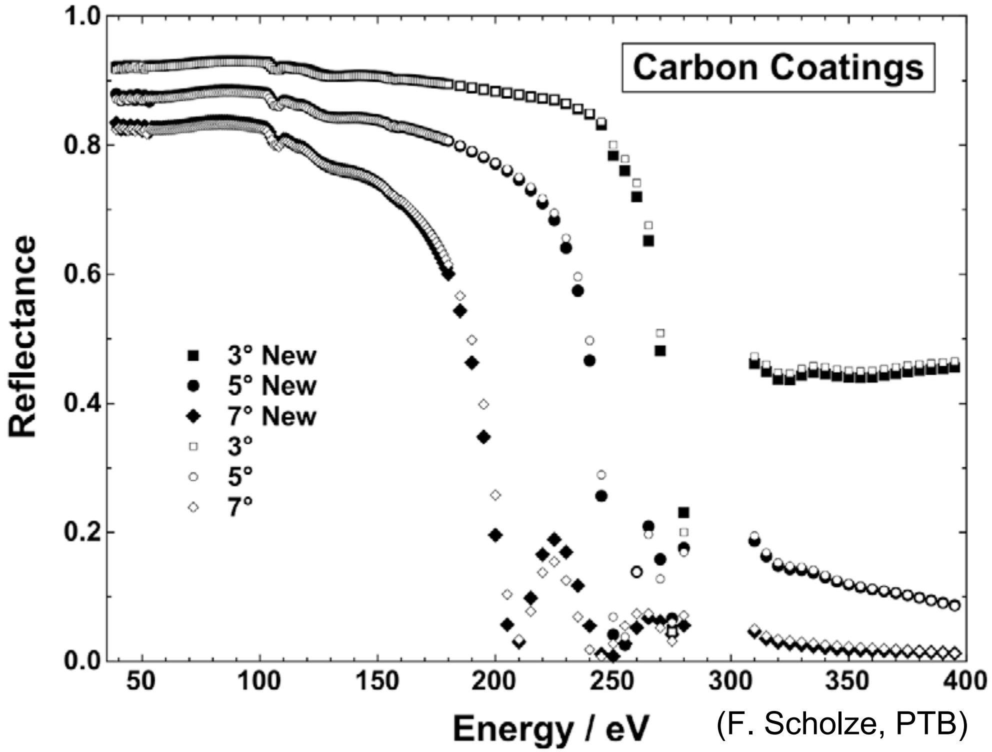


Variation in film thickness:
PV = 1.0nm and σ = 0.2 nm

thickness difference of 3 nm
over a width of 80 mm

Good uniformity in thickness over the whole optical area of the mirror!

Energy-dependent reflectivity



(F. Scholze, PTB)

BESSY II:

PTB soft x-ray radiometry beamline

photon energy range: 35-400eV

various incidence angles: 3°, 5° and 7°

Comparison of two carbon coatings:

- former and new sputtering systems

No difference!

3°: R > 88% 50-200eV

5°: R > 77% 50-200eV

7°: R > 70% E > 162eV

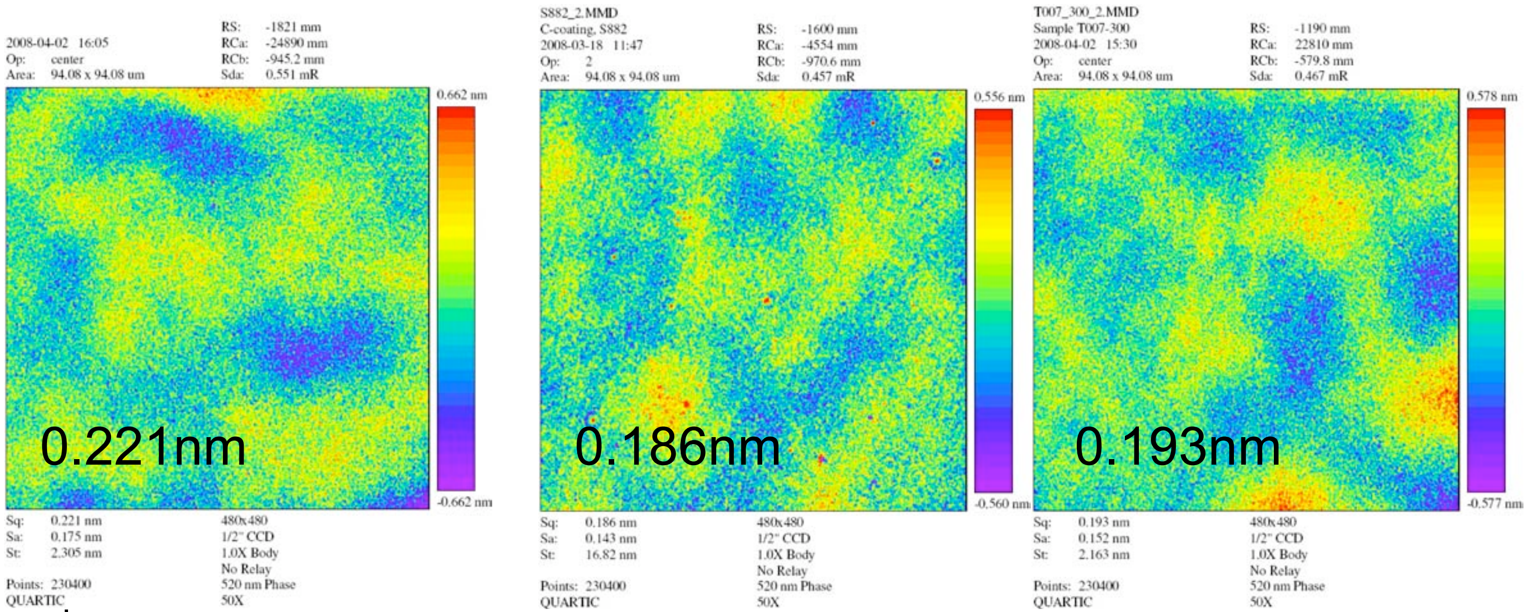
The development of this enlarged system is a success for single layer mirrors !

Micro-Roughness Measurements

Si-Substrate

45nm amorphous C coating on Si substrate

former and new sputtering system



RMS-roughness: 0.221nm 0.186nm 0.193nm

Interference microscopy

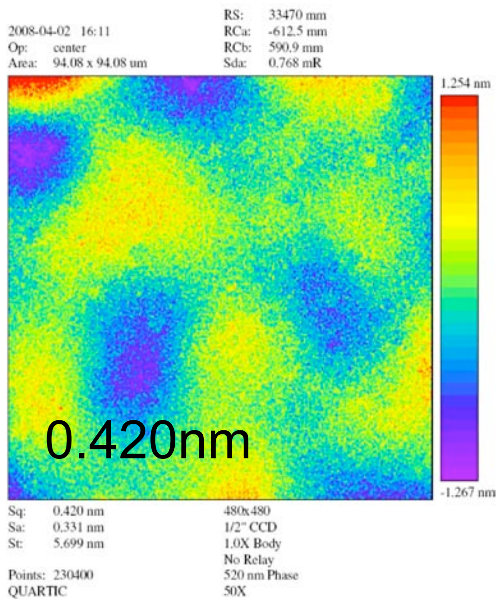
(F. Siewert, BESSY)

measuring area: 94µm x 94µm

- RMS roughness: 0.2nm before and after the deposition
- Magnetron sputtering replicates the substrate roughness

Rougher Si-Substrates

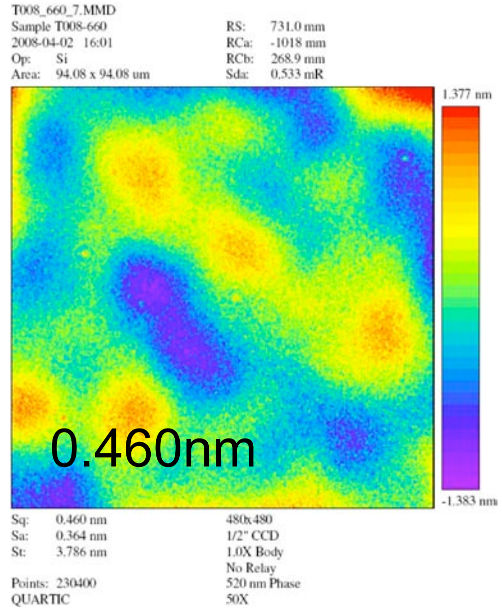
Before deposition:



substrate roughness:

0.4 nm

After carbon deposition:



carbon film roughness:

0.35-0.46 nm

The micro-roughness remains the same over the whole deposition length of 1.5m.

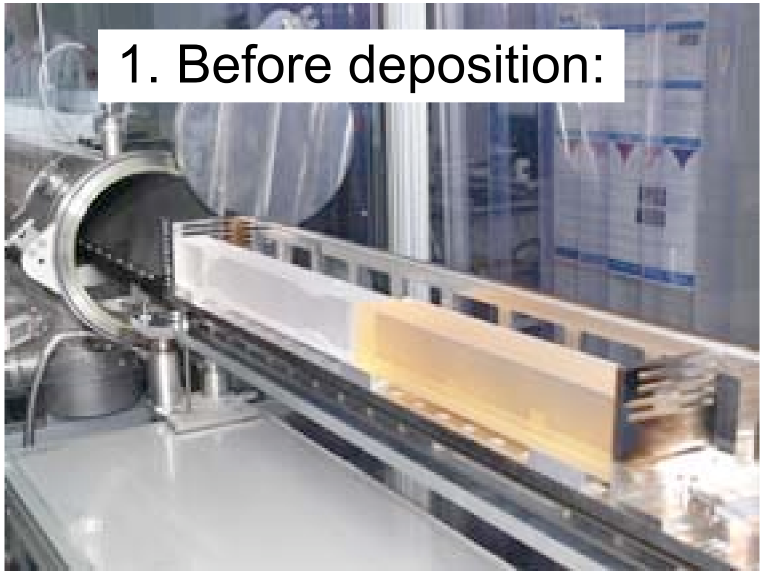
First Application: Single layer mirrors

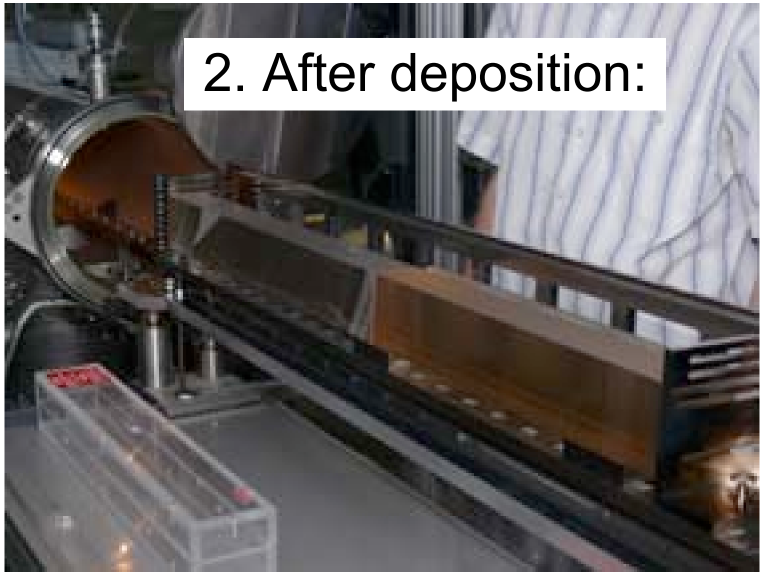
Two flat substrates:

1. 630mm Quartz
2. 460mm Zerodur

After deposition:

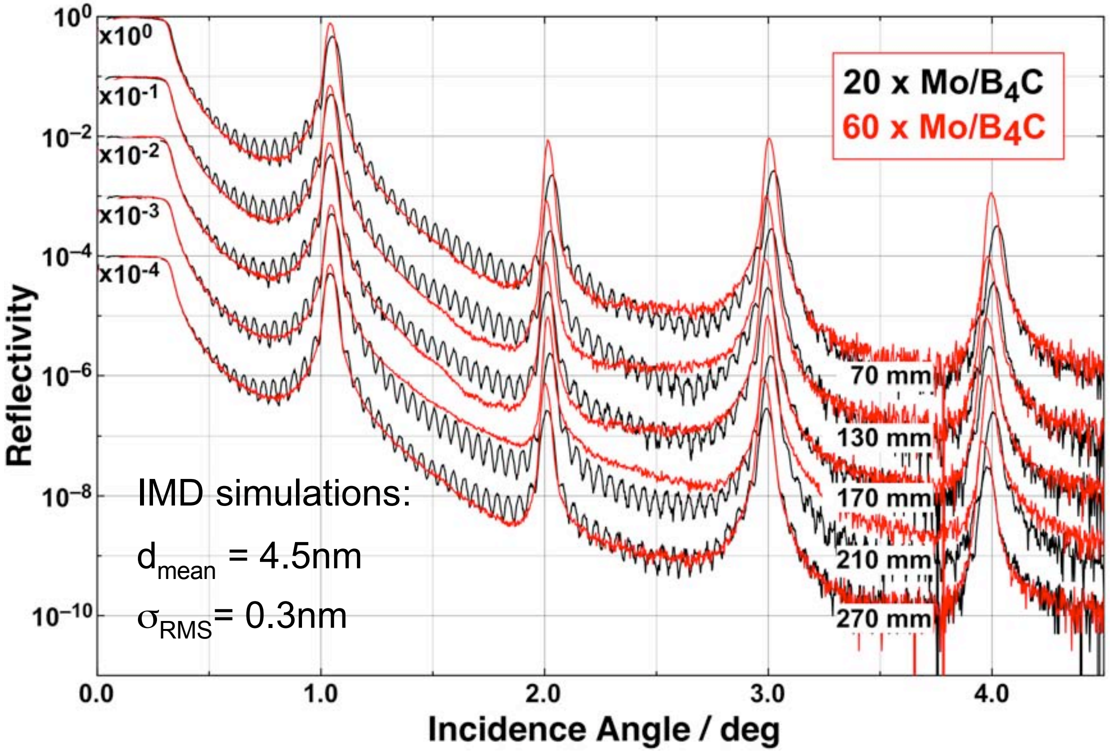
Both substrates are coated with a 45nm thick, amorphous carbon layer





(F. Hertlein, Incoatec)

2. Multilayer mirrors



Mo/B_4C: 10keV

substrate length of 300mm

20 Pairs: 18 Kiessig fringes

dominant Bragg-Peaks:

R > 50% (theor. 60%) 20 pairs

R > 70% (theor. 85%) 60 pairs

Percentage variation of the d-spacing over the whole mirror length: ≈ 3%

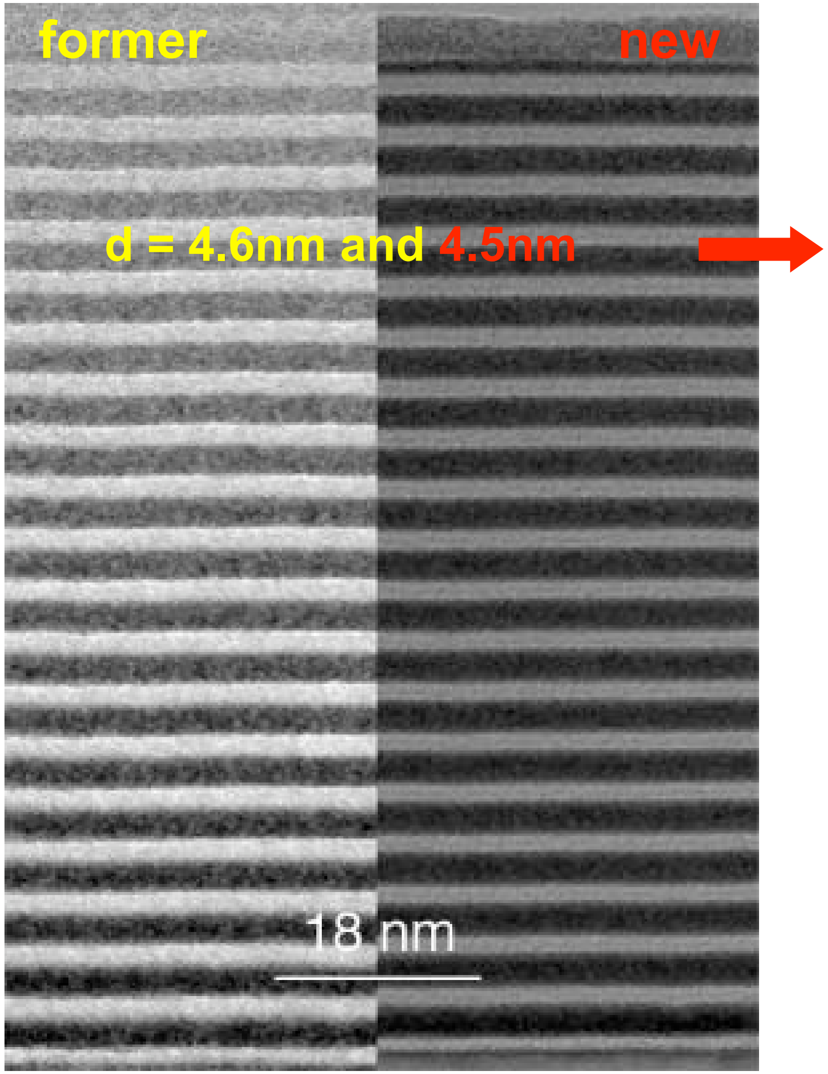
This demonstrates => Good uniformity of the multilayer

=> High precision

=> Excellent run-to-run stability

Deposition length of 1500 mm => coat two mirrors simultaneously (with identical properties!)

HR-TEM investigations of Mo/B_4C



(D. Häußler, CAU Kiel)

Comparison of two multilayers:

high-Z material Mo is dark (HRTEM image)

In agreement with XRR measurements

Inner structure

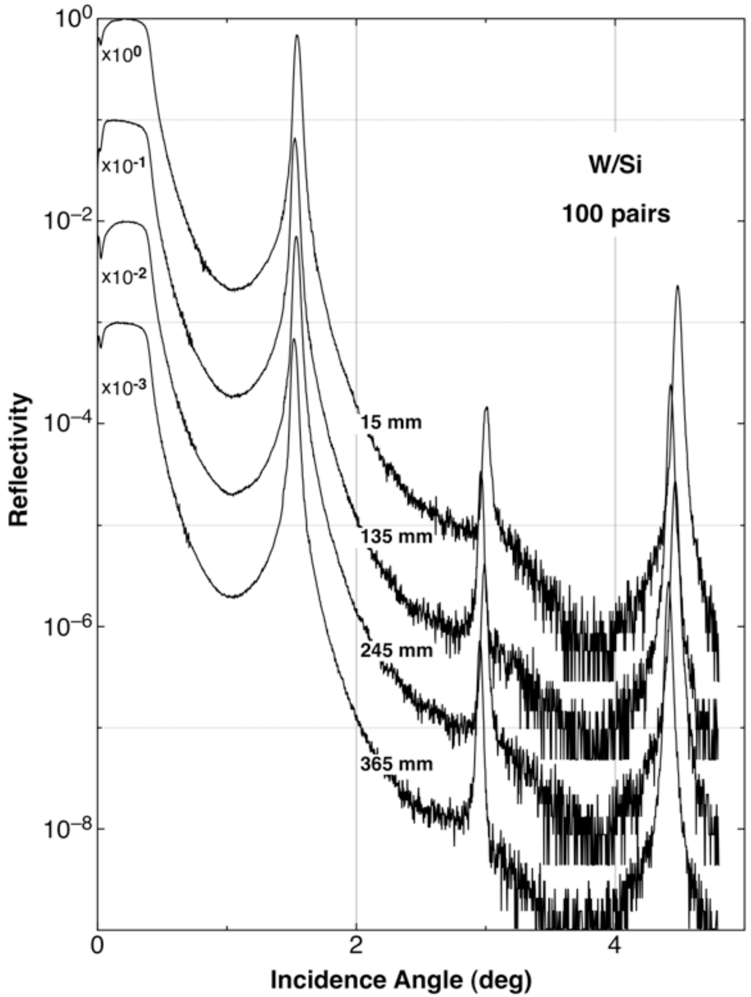
- both layers are amorphous

(absence of crystalline Bragg spots)

Interfaces

- intermetallic phases: no evidence!
- smooth and abrupt

X-Ray Reflectivity measurements (XRR) of a Multilayer Mirror



Former sputtering system (< 550mm)

- **at Cu-radiation (8048eV)**
- **100 pairs W/Si**
- **deposition length: ca. 380mm**
- **3 Bragg-Peaks**
- **Second order suppressed => $d_W = d_{Si}$**

Refsim simulations

reflectivity: $R \approx 66\%$
d-spacing $d \approx 3$Å
$\Gamma \approx 0.5$

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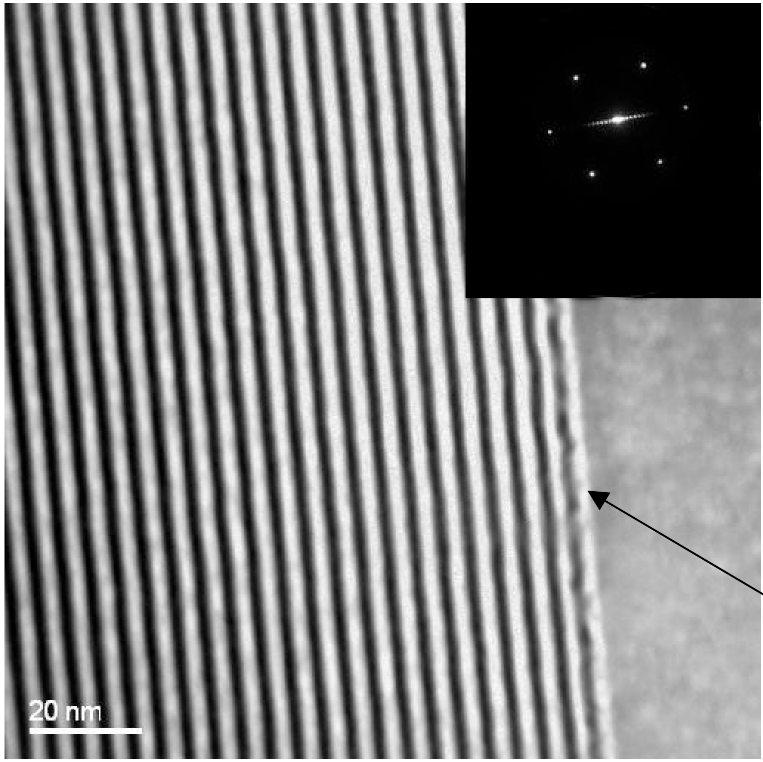
2007 Sino-German High Level Expert Symposium on X-ray Optics,
Optics and Precision Engineering 15(12) (2007) 1869-1877. ISSN 1004-924X.

→ Uniformity of the multilayer coating: d-spacing and reflectivity
→ Precision of the multilayer process over a length of 400 mm

TEM measurements of W/Si



(University Kiel)

multilayer mirror

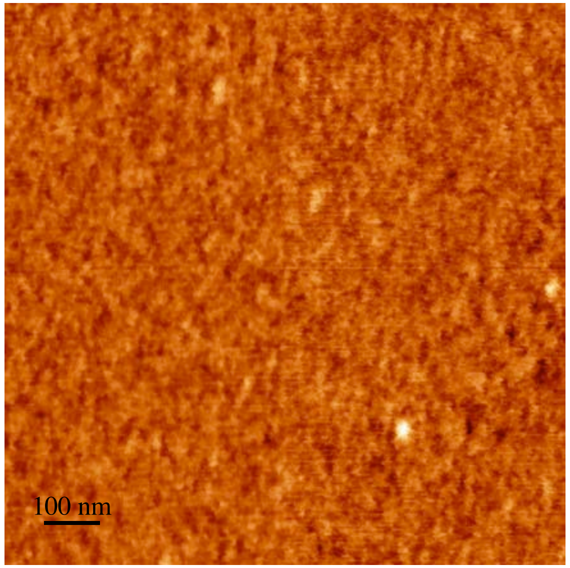
100 pairs

dark layer: metal
bright layer: non-metal

native Si-oxide layer (1-2nm)

- perfect growth of the stack: low layer roughness
- Si-on-W interfaces look sharper than the W-on-Si
- no formation of a compound at the interface

AFM measurement of a multilayer mirror



σ = 0.18 nm RMS

scan area: 1 μm x 1 μm

W/Si multilayer mirror

surface roughness measurements at 6 positions on the whole mirror

before deposition:

RMS roughness: 0.2 - 0.3 nm

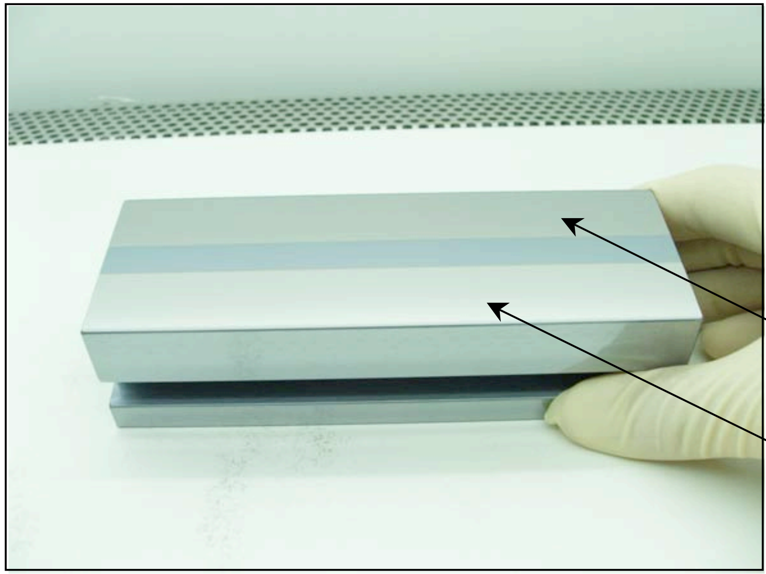
after deposition:

RMS roughness: 0.2 - 0.4 nm

Replication of the substrate roughness: σ < 0.3 nm

Second application: Tomography

Large X-ray multilayer mirror after deposition at GKSS sputtering system



(In collaboration with Incoatec GmbH)

typical multilayer mirror

dimensions 160 x 55 x 40 mm³

plane geometry

upper area: W/Si, 100 pairs
22-45 keV

lower area: Ru/C, 100 pairs
10-22 keV

Synchrotron beamline: monochromatic or white beam

Conclusions

- Single layers and multilayers

- Build up a new sputtering system:
  - Deposition over a length of 1500 mm => XFEL
  - Preparation of pairs simultaneously => IBL: DMM

- Properties of the mirrors:
  - high uniformity in thickness: 3%
  - film roughness of about 0.3 nm

    Magnetron sputtering replicates the substrate roughness!

  - high reflectivity: > 88% at 3° and 50-200 eV (C single layers)

    > 70% at 1° and 8 keV (Mo/B_4C multilayers)
  - good layer quality of the stack:

    layers: amorphous

    interfaces: abrupt and smooth

    intermetallic phases: no evidence

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