Stress optimization of multilayer Laue lens coatings

Stefan Braun, Adam Kubec, Peter Gawlitza, Maik Menzel, and Andreas Leson

IWS Dresden, Fraunhofer Institute for Material and Beam Technology, Germany

Outline

- Introduction, motivation
- MLL fabrication
- Current challenges and limitations
- Potential solutions for low-stress MLL coatings
- Summary/conclusions

http://www.iws.fraunhofer.de/

Acknowledgements

AXO DRESDEN GmbH: Sven Niese (see also poster session 2)

Fraunhofer IKTS: Jürgen Gluch, Peter Krüger, Ehrenfried Zschech

ESRF, ID13: Manfred Burghammer

DESY, PETRA III: Christian Schroer, Gerald Falkenberg, Ulrike Bösenberg

TU Dresden: Jens Patommel

Funding:
This work is partly funded by the European Union (ERDF) and the Free State of Saxony via the ESF project 100087859 (ENano) and the European Regional Development Fund (EFRE) and the Free State of Saxony.
The project Cool Analytics is part of the Leading-Edge Cluster „Cool Silicon“, which is sponsored by the Federal Ministry of Education and Research (BMBF) within the scope of its Leading-Edge Cluster Competition.

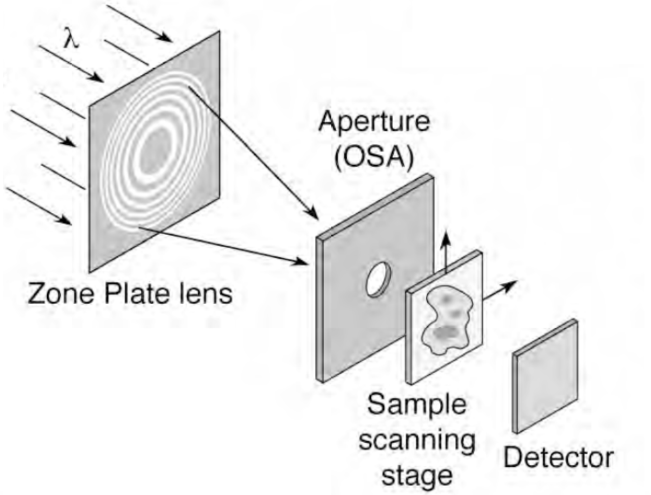
Principles of X-ray microscopy

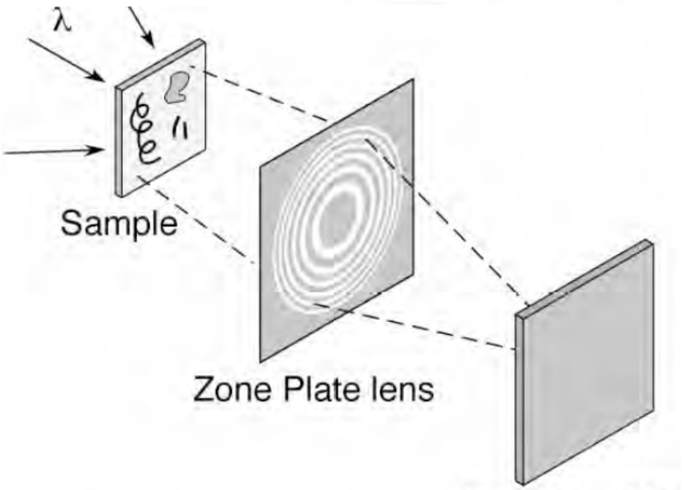
Scanning XRM

- Information about photoemission, fluorescence and transmission
- Spatial resolution in the range of 15 – 50 nm
- Typical exposure time: minutes

Full-field XRM

- Only transmission: Absorption and phase contrast
- Spatial resolution in the range of 10 – 50 nm
- Typical exposure time: seconds





Pictures taken from D. Attwood: *Soft X-rays and Extreme Ultraviolet Radiation: Principles and Applications*, Cambridge University Press, 1999

Current status of X-ray microscopy

Soft X-ray region (E < 2 keV)

- Resolution close to 10 nm using Fresnel zone plates (FZP)
- Relevant almost only at synchrotron sources
- Lab sources only for very few energies (100 eV and water window) available

Hard X-ray region (E > 6 keV)

- Resolution in the range of 25 – 80 nm (mainly limited by the optics)
- Lab sources available => First lab tools (Zeiss/Xradia)
- Efficiency of FZP decreases for higher photon energies

Challenges with FZP

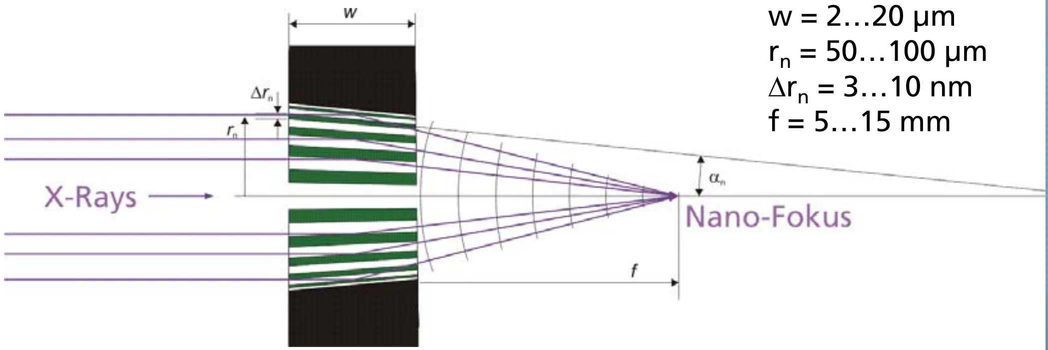
- Significant decrease of the outermost zone widths (determining the resolution) seems to be difficult
- High efficiency for hard X-rays requires high aspect ratios

Multilayer Laue lenses as optics for X-rays

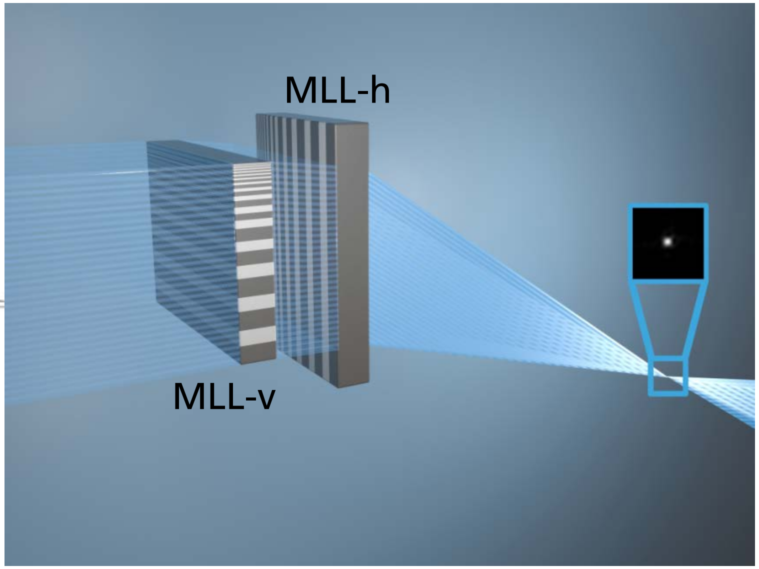
New approach for the fabrication of zone plates with high aspect ratios

- Application of superpolished silicon wafers as substrates
  => extremely smooth layers (= zones) can be deposited
- Coating instead of structuring results in one order lower zone widths
  => higher resolution
- Depth of zones can freely be chosen => high diffraction efficiencies

<u>below:</u> schematic picture of a MLL pair for focusing in one dimension
<u>right:</u> perpendicular arrangement of two MLL pairs for the two-dimensional focusing or imaging



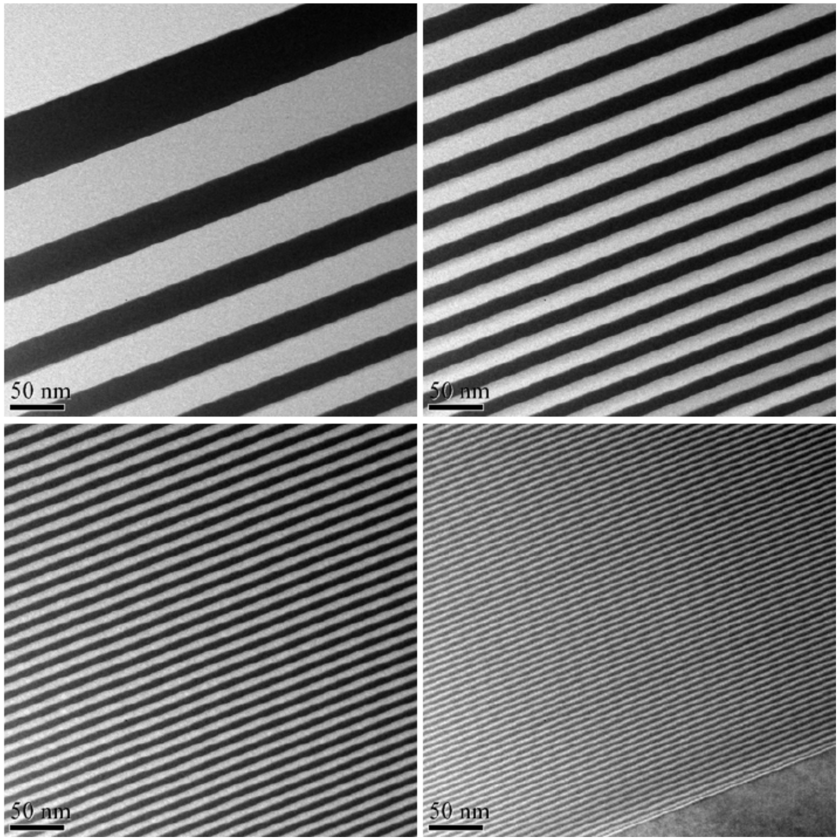
w = 2…20 µm
r_n = 50…100 µm
Δr_n = 3…10 nm
f = 5…15 mm



- H.E. Hart et al., *Diffraction characteristics of a linear zone plate,* Journal of the Optical Society of America 56 (1966) 1018
- J. Maser et al., *Multilayer Laue Lenses as high-resolution X-ray optics,* Proc. of SPIE 5539 (2004) 185

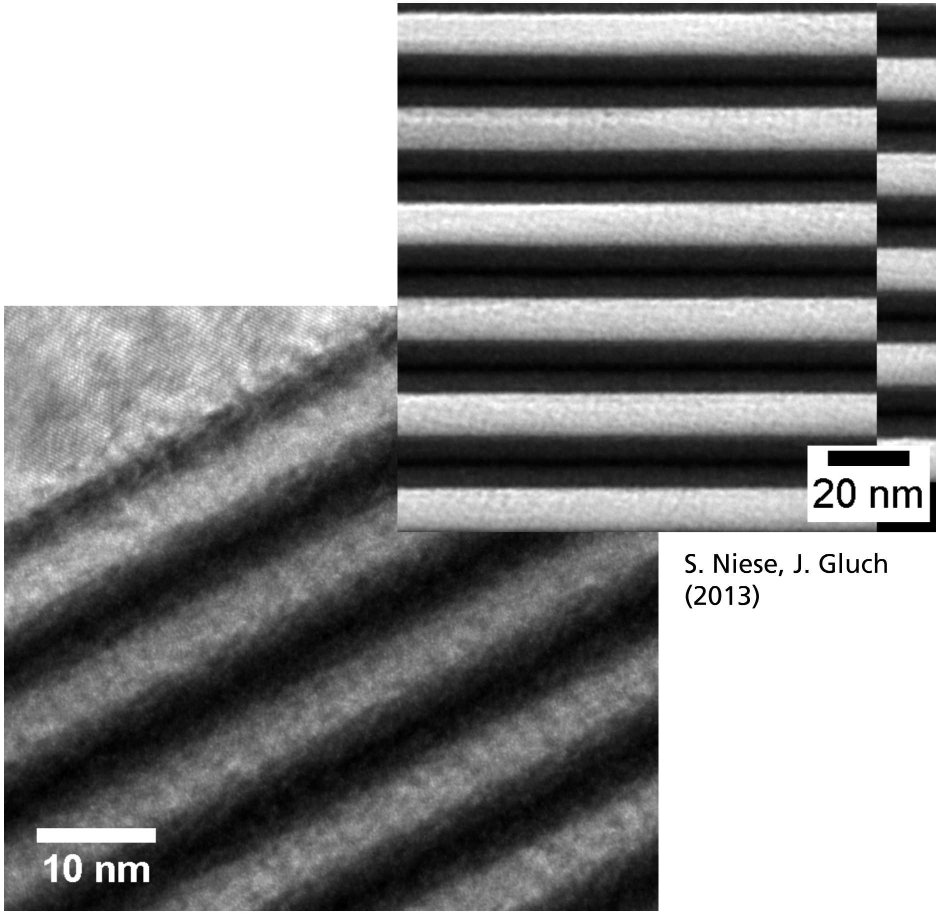
TEM analysis of MLL coatings

Multilayer Laue Lenses ($MoSi_2$/Si)



S. Braun et al, Journal of Physics 425 (2013)

Multilayer Laue Lenses (WSi_2/Si)

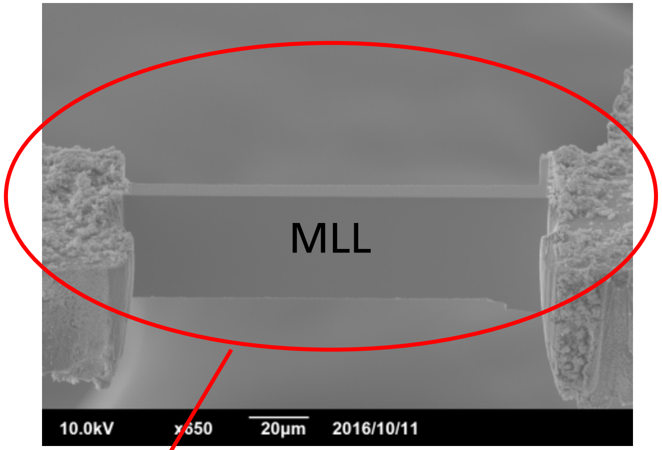


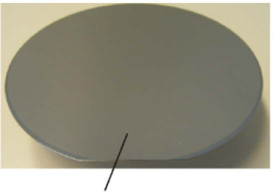
S. Niese, J. Gluch
(2013)

MLL fabrication: Focused ion beam milling

Wafer with multilayer is not enough!

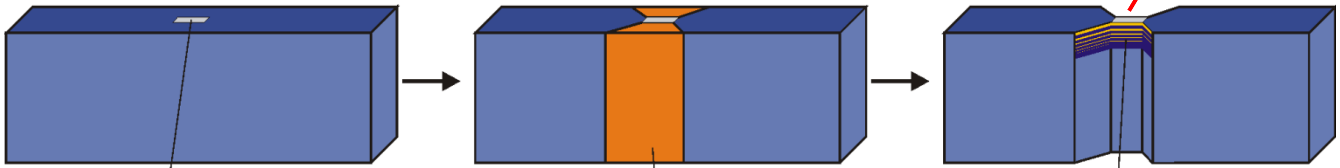
⇒ Cutting and thinning required
⇒ Efficiency can be controlled by the right aspect ratio (lamella-to-layer-thickness)





Silicon wafer with MLL coating

Stripe sawed off from the wafer (about 50 μm x 1 mm)



Coating to protect the MLL structure from ion beam erosion

Area that is removed by focused ion beam milling

Thinned MLL segment = MLL lamella

Requirements for smaller X-ray foci

Current MLL status: **Focal spot size** is not **limited** by the zone width but **by the numerical aperture of the lens**

Restriction from the application: Focal length f > 10 mm (E = 8…30 keV)

⇒ Need to increase the lens aperture
⇒ More and thicker layers!

Challenge: With thicker coatings stress becomes more and more a problem

-> Strong wafer deformation (two times even wafer breaking for $d_{MLL} = 50$ µm)
-> Risk for layer delamination at wafer sawing or FIB milling
-> Risk for cracks in the MLL

⇒ Internal stress of the $MoSi_2$/Si and WSi_2/Si multilayers has to be reduced!

Approaches for MLL stress reduction

1. Change of the MSD process conditions
    - Increase of sputter gas pressure
    - Reduction of magnetron power

⇒ Disadvantages: Increased roughness, increased coating times

2. Change of the thickness ratio WSi_2 <-> Si or $MoSi_2$ <-> Si
    - Reduction of Si thickness
    - Disregarding the strict zone plate law for each zone

⇒ Disadvantages: Decrease of efficiency, limited stress reduction potential

3. Change of the multilayer materials
    - Introduction of layers with tensile stress (metallic materials like Al as spacer and Mo or W as absorber)
    - Introduction of barrier layers between absorber and spacer layers

⇒ Disadvantages: Higher complexity of the multilayer, nano-crystalline layers

Potential solution for MLL stress reduction

Requirements for replacement materials of the spacer layer

- Low Z
- No or tensile stress
- Suitable deposition rate

=> **Aluminum** could be a candidate

<u>However:</u> Strong increase of layer roughness with pure Al has been observed

⇒ Doping of Al as possible solution? (e. g. Al_xSi_{1-x}, Al_xO_{1-x}, …)
⇒ Balancing of stress, roughness and deposition rate possible?

Maybe YES, but currently not the preferred solution at IWS!

Solution for MLL stress reduction

Absorber layer with tensile stress

Absorber layer

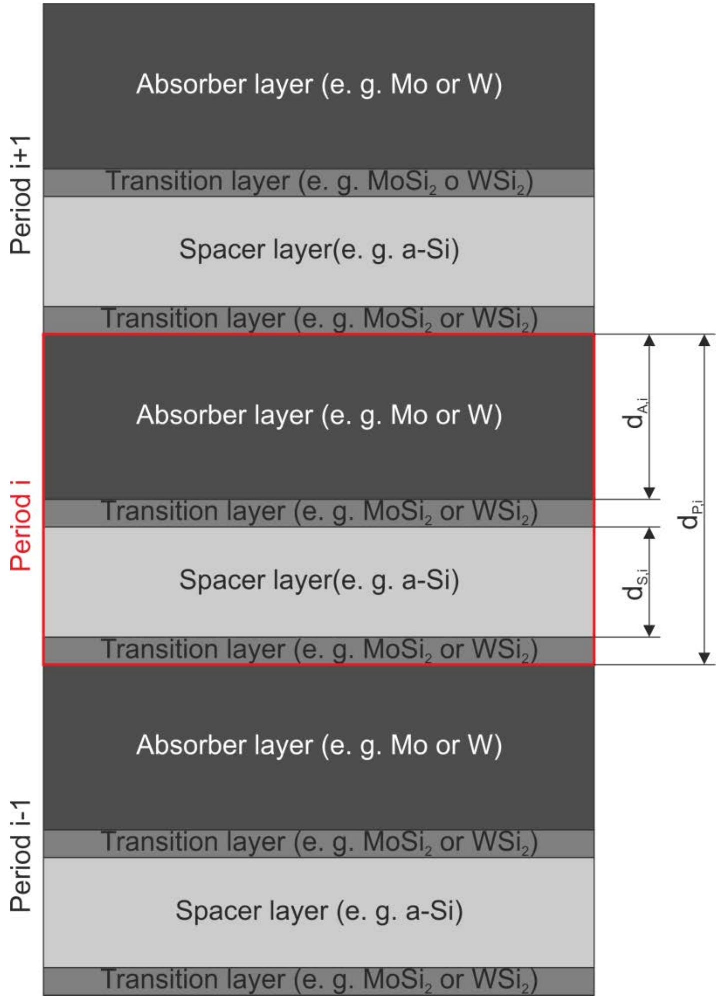
Thickness $d_{A,i}$
Stress $\sigma_{A,i}$

Spacer layer with compressive stress

Spacer layer

Thickness $d_{S,i}$
Stress $\sigma_{S,i}$

Stress compensation: $d_{A,i}\sigma_{A,i} + d_{S,i}\sigma_{S,i} \sim 0$
(in most cases $\sigma_{A,i} > 0$ und $\sigma_{S,i} < 0$)



<u>Aims:</u>

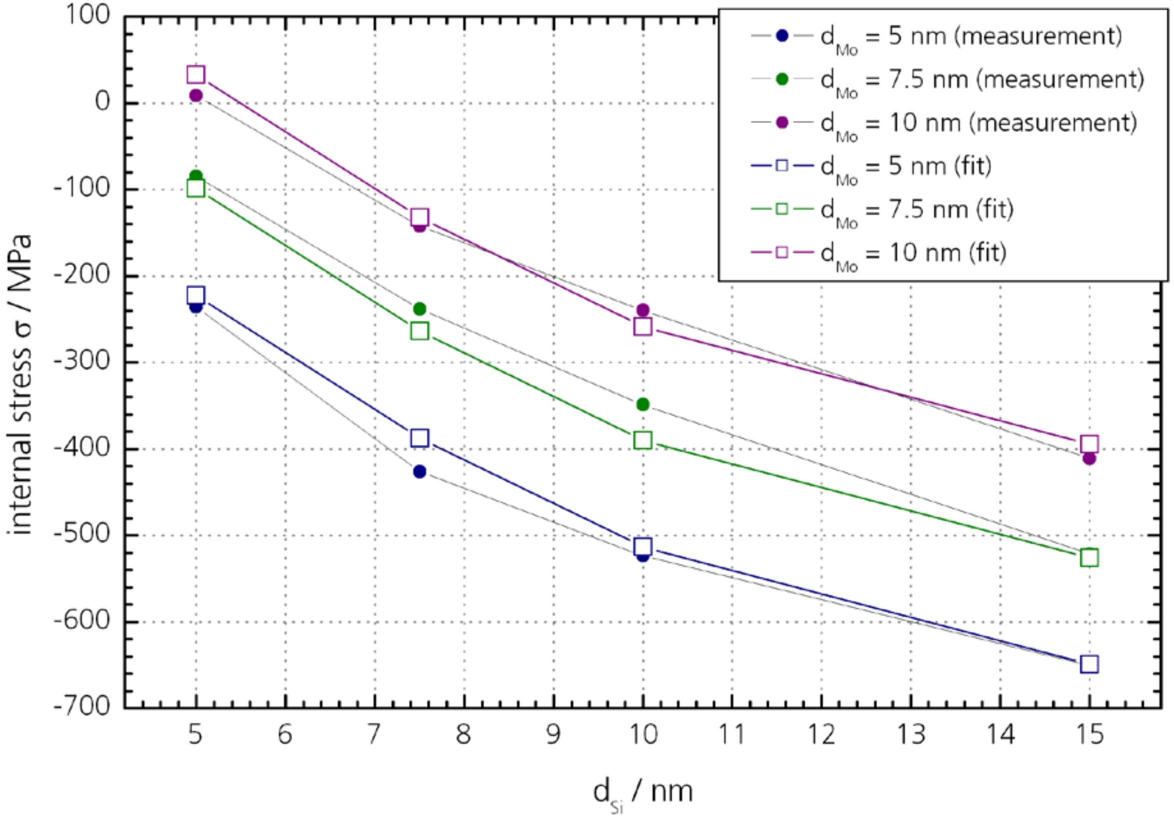
- Compensation of absorber and spacer layer stress
- Filling the remaining thickness by low-stress transition layers (e. g. $MoSi_2$, WSi_2)

=> Only d_p follows the zone plate law
=> Stress-free multilayer periods

Multilayer stress versus single layer thickness

Multilayer: Mo/$MoSi_2$/Si/$MoSi_2$ with $d_{MoSi2,\ total}$ = 2 nm (1 nm at each interface)

Fitting function: $\sigma(d_{Mo},d_{Si}) = a_{Mo}*d_{Mo}^2 + b_{Mo}*d_{Mo} + a_{Si}*d_{Si}^2 + b_{Si}*d_{Si}$



Fitting results:

a_{Mo} = 0.6281

b_{Mo} = 41.59

a_{Si} = 3.102

b_{Si} = -104.7

Potential solution for MLL stress reduction

Approach: $\sigma = \sigma(d_{Mo}, d_{Si}) = a_{Mo} d_{Mo}^2 + b_{Mo} d_{Mo} + a_{Si} d_{Si}^2 + b_{Si} d_{Si}$

Assumption: $d_{Si} = c^* d_{Mo}$

Aim: $\sigma = \sigma(d_{Mo}, d_{Si}) = a_{Mo} d_{Mo}^2 + b_{Mo} d_{Mo} + c^2 a_{Si} d_{Mo}^2 + c b_{Si} d_{Mo} = d_{Mo}^2 (a_{Mo} + c^2 \cdot a_{Si}) + d_{Mo} (b_{Mo} + c \cdot b_{Si}) = 0$

=> Calculation of $c = d_{Si}/d_{Mo}$ versus $(d_p - d_{MoSi2})$

$d_{Mo}(a_{Mo} + c^2 \cdot a_{Si}) + (b_{Mo} + c \cdot b_{Si}) = 0$ and $d_{Mo} = -(b_{Mo} + c \cdot b_{Si})/(a_{Mo} + c^2 \cdot a_{Si})$

$$d_p - d_{MoSi_2} = d_{Mo} + d_{Si} = -\frac{(1+c)(b_{Mo} + c \cdot b_{Si})}{a_{Mo} + c^2 \cdot a_{Si}}$$

$$c^2 \left[a_{Si}(d_p - d_{MoSi_2}) + b_{Si} \right] + c(b_{Mo} + b_{Si}) + \left[a_{Mo}(d_p - d_{MoSi_2}) + b_{Mo} \right] = 0$$

$p = \dfrac{b_{Mo} + b_{Si}}{a_{Si}(d_p - d_{MoSi_2}) + b_{Si}}$ and $q = \dfrac{a_{Mo}(d_p - d_{MoSi_2}) + b_{Mo}}{a_{Si}(d_p - d_{MoSi_2}) + b_{Si}}$

$\Rightarrow$ $c = -\dfrac{p}{2} + \sqrt{\dfrac{p^2}{4} - q}$ and $d_{Mo} = \dfrac{d_p - d_{MoSi_2}}{1 + c}$ $d_{Si} = c \cdot d_{Mo}$

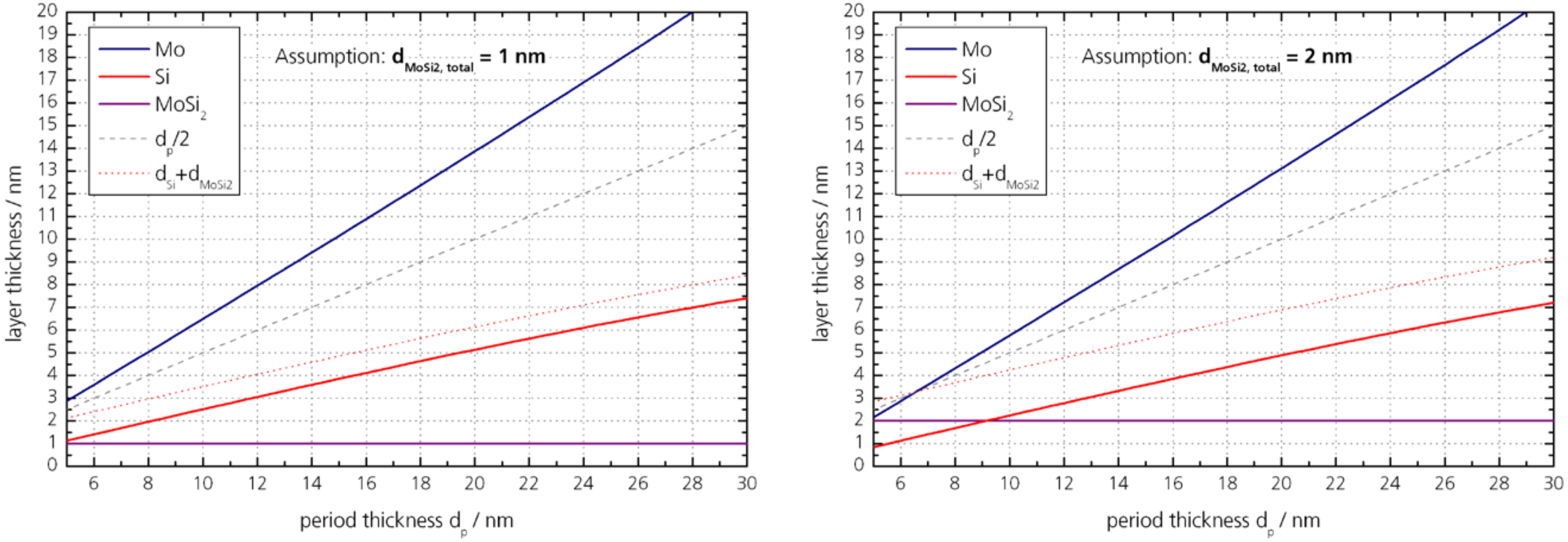
Solution for low-stress MLL coatings

Option 1: Mo/$MoSi_2$/Si/$MoSi_2$ with $d_{MoSi2,\ total}$ = constant

Assumption of two different $MoSi_2$ thicknesses of 1 nm and 2 nm in every period

⇒ With increasing period thickness the thickness ratio between d_{Mo} and d_{Si} increases and would result in efficiency losses

⇒ Not a good solution (similar to a simple Γ variation without transition layers)



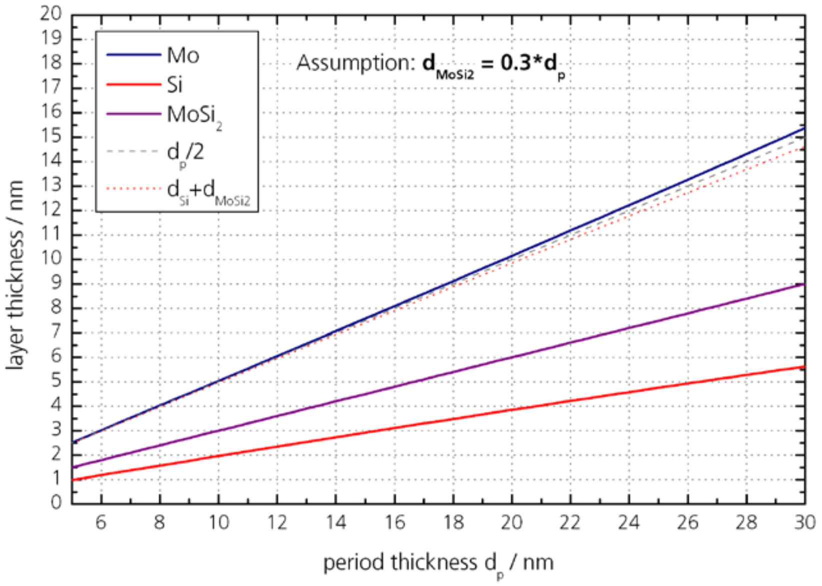
Solution for low-stress MLL coatings

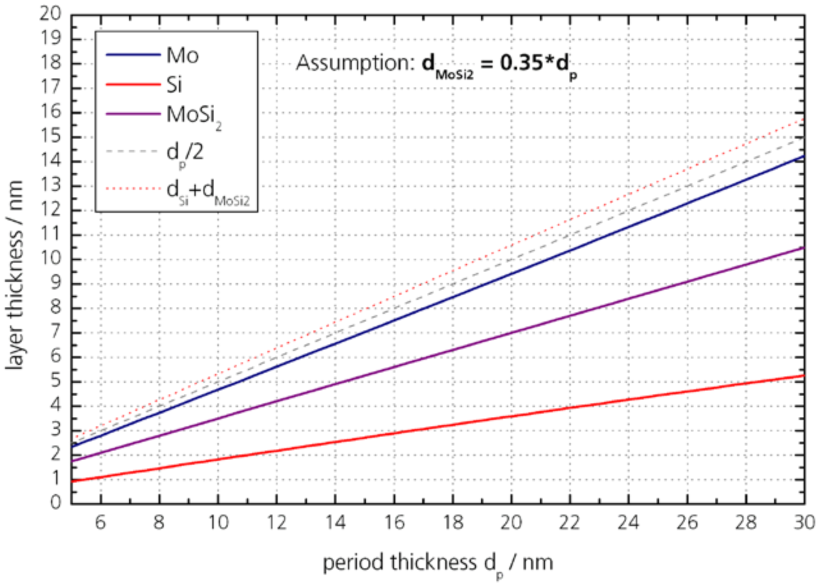
Option 2: Mo/$MoSi_2$/Si/$MoSi_2$ with $d_{MoSi2,\ total} = h*d_p$

Assumption of different $MoSi_2$ thicknesses factors of 0.1, 0.2, 0.3 and 0.35

⇒ Much better behavior of absorber to spacer layer ratio compared to constant transition layer thickness

⇒ Best agreement for h = 0.3…0.35 => Freedom for efficiency optimization



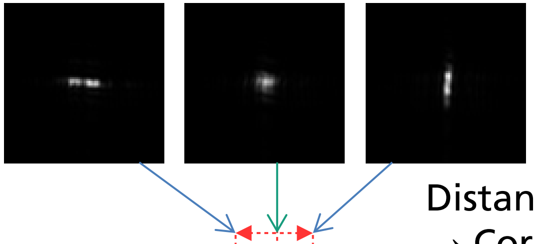


Recent results

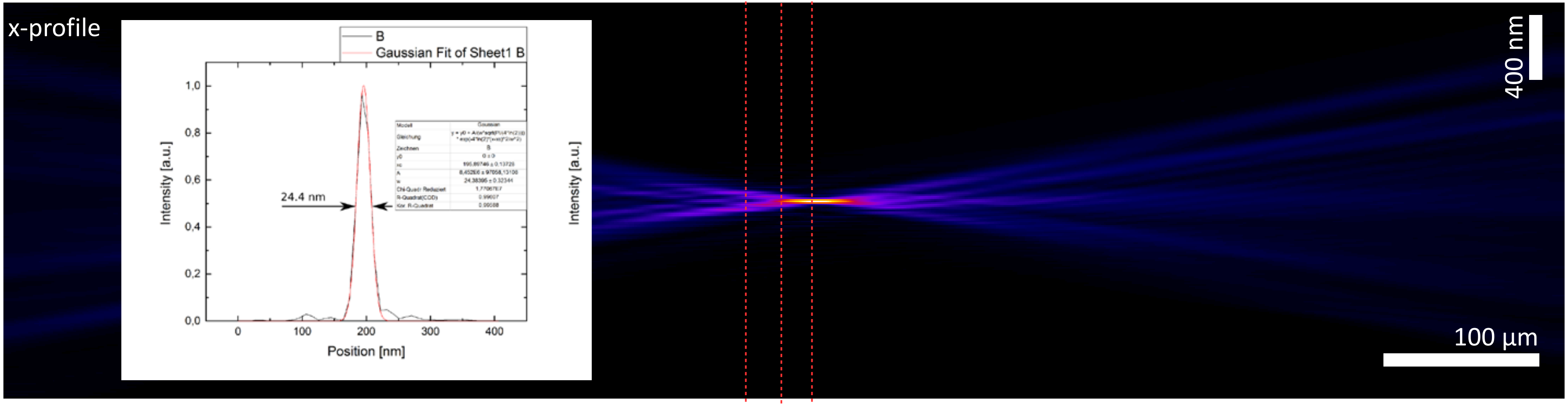
ESRF, October 2016

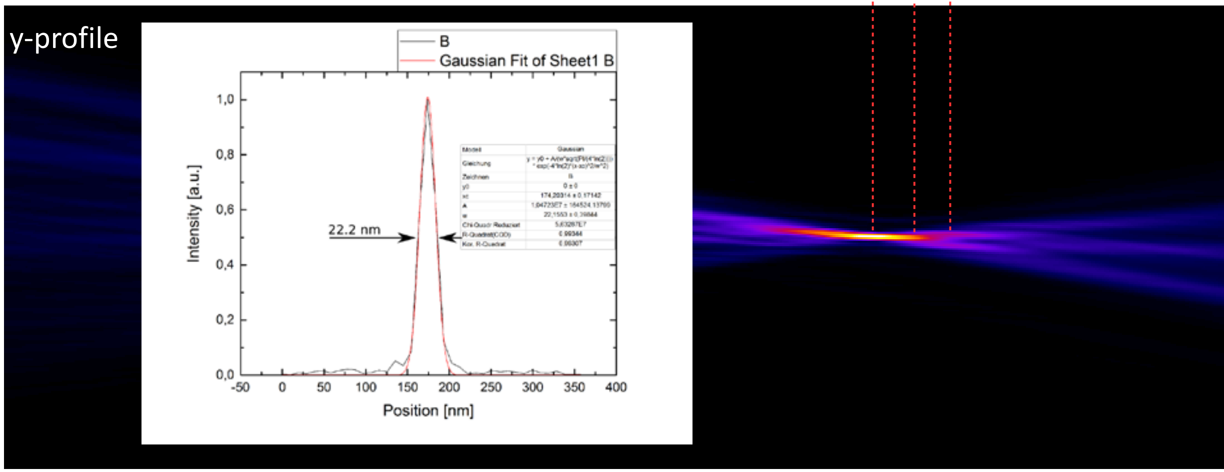
S. Niese, A. Kubec,
M. Burghammer

Beam Propagation Direction



Distance of focal planes is ca. 50 µm
→ Corresponds with lens distance





MLL design:
- Photon energy: 12 keV
- Focal distance: 9 mm
- Working distance: 3.36 mm
- Zones: 970-6970
- Total thickness: 50.3 µm

Summary, conclusions

- Current limitation for X-ray focusing: MLL aperture
- Stress reduction by introducing new material systems: Mo/MoSi$_2$/Si/MoSi$_2$, W/WSi$_2$/Si/WSi$_2$ and Mo/C/Si/C
- Only period thickness follows the zone plate law
- Transition layer thickness should scale with a factor of 0.3-0.35 with the period thickness
- Transition layers act as barrier layers for interdiffusion
  => Thermal stability up to T = 200 °C has been proven
- MLL with total thickness > 100 µm successfully fabricated

Thank you for your attention!

Contact information:

Stefan Braun

IWS Dresden, Fraunhofer Institute for Material and Beam Technology
Department „PVD- and Nanotechnology“
Winterbergstraße 28
D-01277 Dresden

Phone: +49.351.83391-3432
Fax: +49.351.83391-3314

E-mail:
stefan.braun@iws.fraunhofer.de