Grazing incidence PDF for real time studies of thin films

Recent developments at Beamline P07 @ PETRA III

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HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

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colleagues and collaborators

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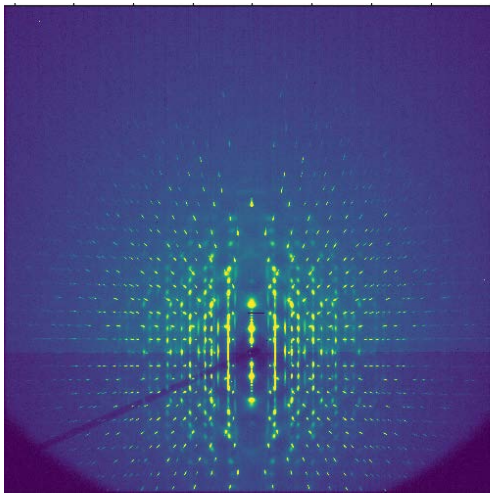
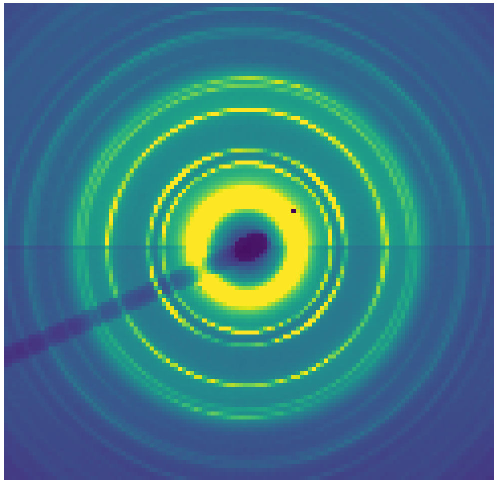
HELMHOLTZ SPITZENFORSCHUNG FÜR GROSSE HERAUSFORDERUNGEN

epitaxy, texture, preferred orientation

effects in thin films due to cinfinement in one dimension

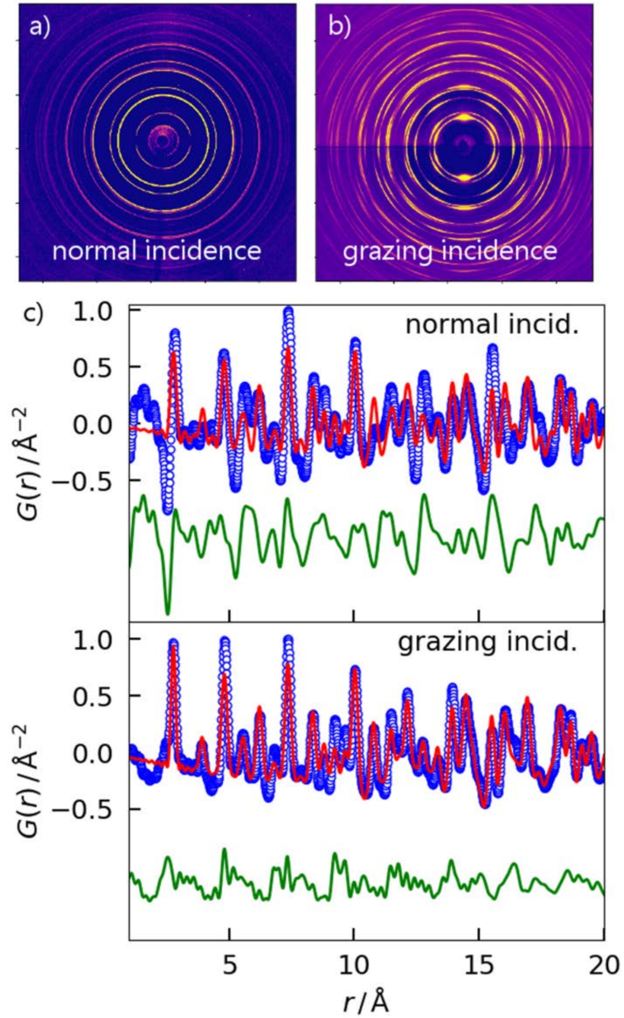


single-crystalline



slight preferred orientation

[111] fiber textured Pt



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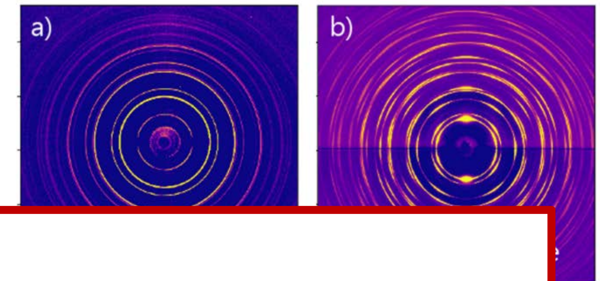
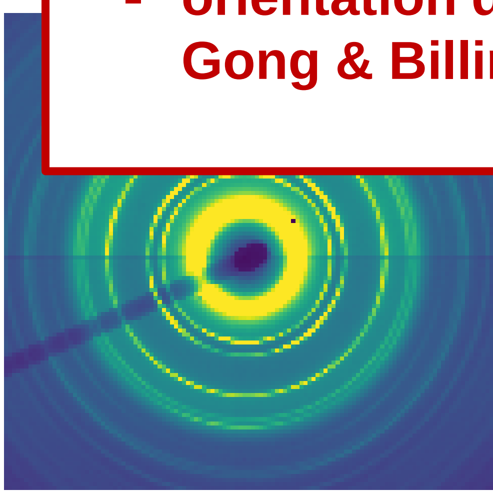
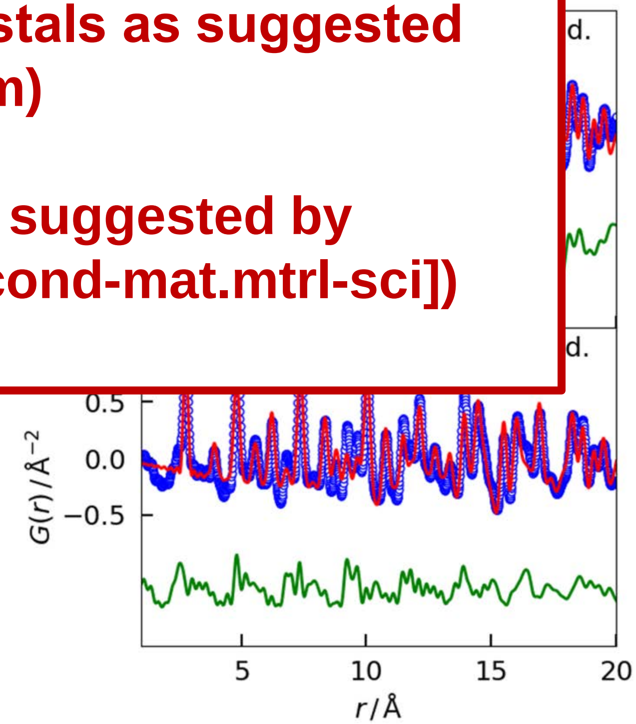


- **3D ΔPDF approach as for single crystals as suggested by e.g. Simonov *et al.* (YELL program)**
- **orientation distribution approach as suggested by Gong & Billinge (arXiv:1805.10342 [cond-mat.mtrl-sci])**

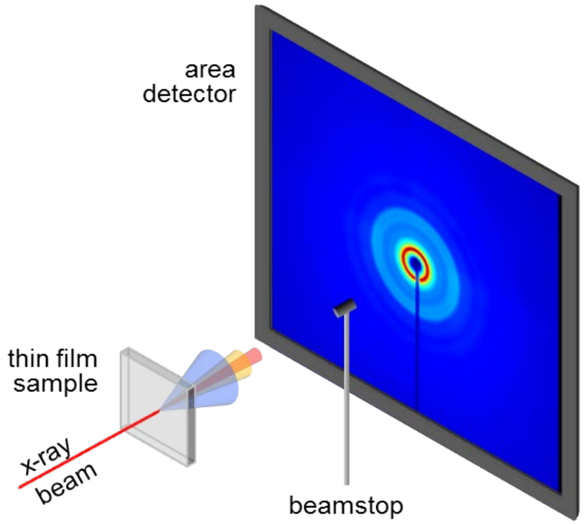


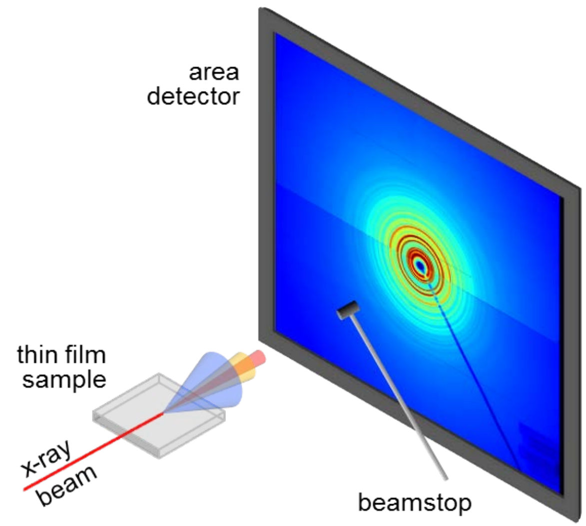
orientation



thin film PDF – comparison of geometries

transmission vs grazing incidence





transmission geometry

- simple experimental setup
- thickness ratio film to substrate ~ 1:1000
- signal to background ratio limits minimum film thickness and time resolution

K. M. Ø. Jensen *et al.*, *IUCrJ* 2 (2015) 481.

grazing incidence geometry

- requires surface diffractometer and focused x-ray beam
- enhanced surface sensitivity
- high time resolution
- *in situ* capailities limited by need to stay in exact alignment

thin film PDF in grazing incidence geometry

a brief history up to the present development

- first measurements at lab source reported more than 30 years ago, PDFs of poor quality

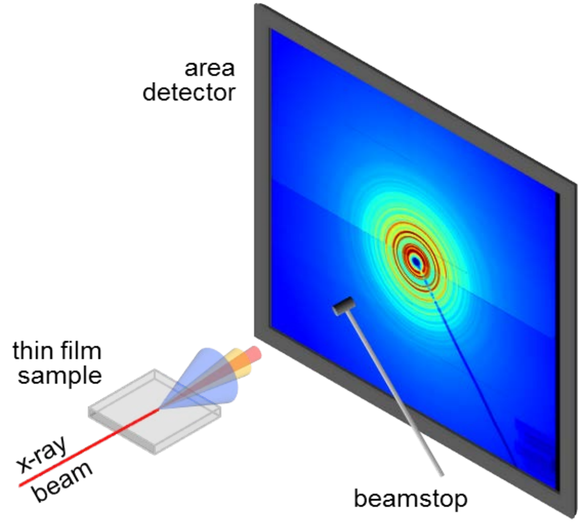
  A. Fischer-Colbrie *et al.*, Phys. Rev. B 38 (1988) 12388.

- more recent synchrotron measurements with hard x-rays and scanning 1D detector, long exposure times

  K. H. Stone *et al.*, APL Mater 4 (2016) 076103.

- now rapid acquisition PDF measurements with high-energy x-rays and 2D detector for sub-second time-resolutiony

  A.-C. Dippel et al., IUCrJ 6 (2019) 290.

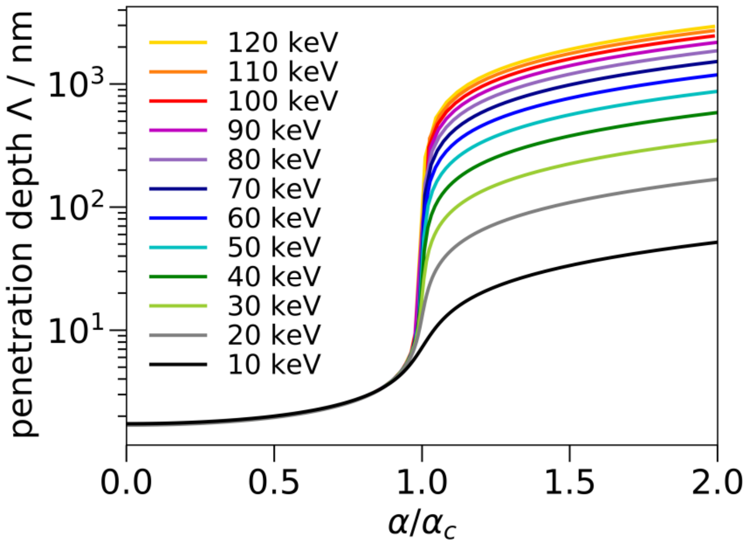


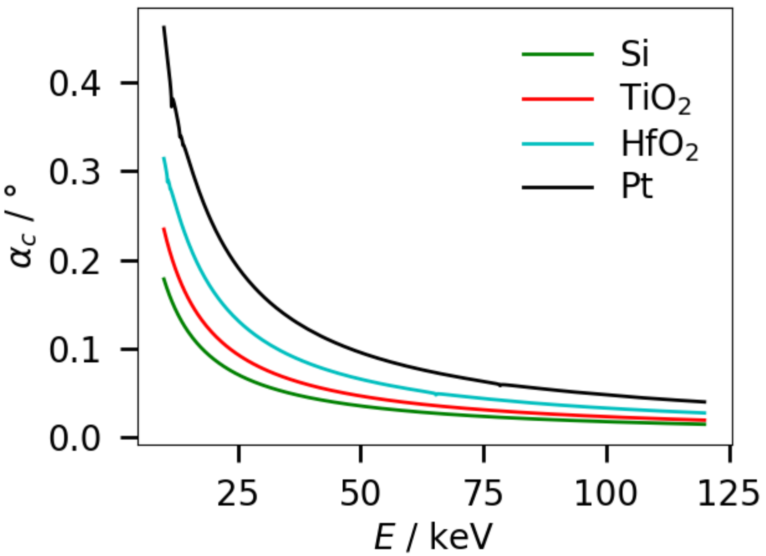
grazing incidence geometry

- requires surface diffractometer and focused x-ray beam
- enhanced surface sensitivity
- high time resolution
- *in situ* capbilities limited by need to stay in exact alignment

surface diffraction under grazing incidence

enhanced surface sensitivity at and below the critical angle





- critical angle $\alpha_c \propto E^{-1}$
- $\alpha_c < 0.1°$ for high energies
- incidence angle $\alpha < \alpha_c$

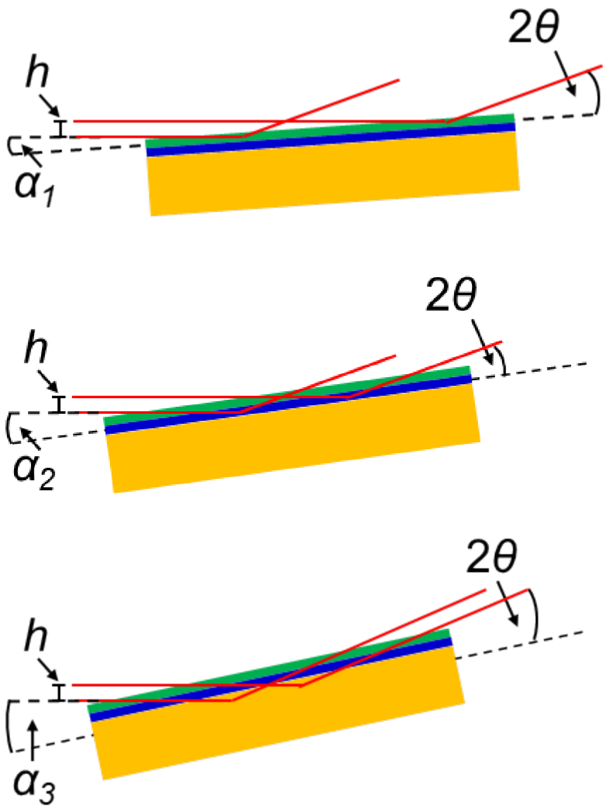
- flat sample
- low roughness
- stay in alignment within 0.001°

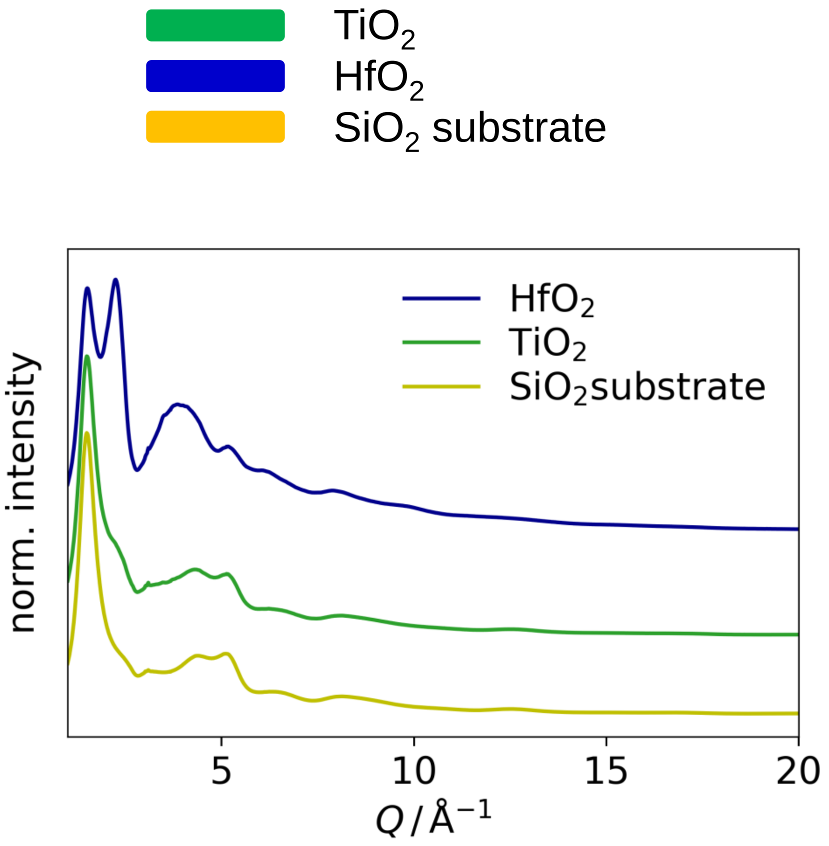
➔ minimized penetration independent of wavelength

depth-profiling of bilayers

variation of incidence angle

incidence angles $\alpha_1 < \alpha_2 < \alpha_3$; beam height h, scattering angle 2θ





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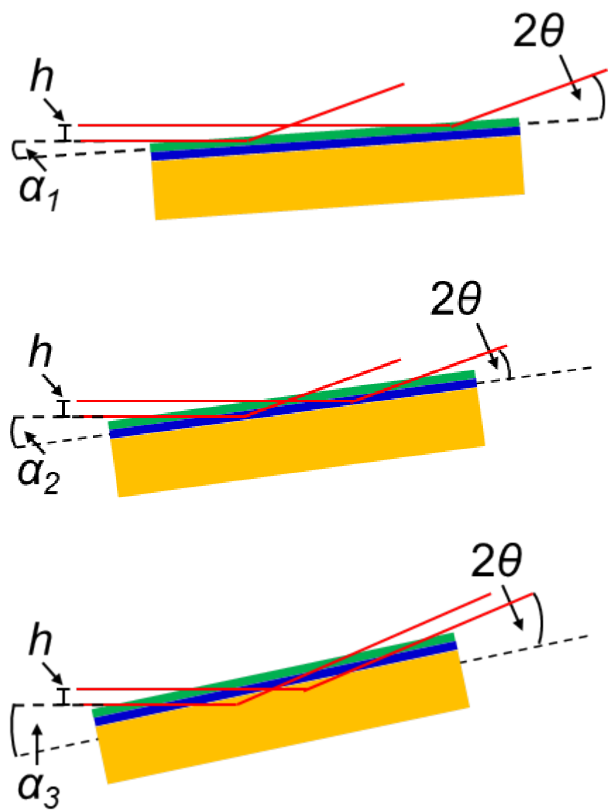
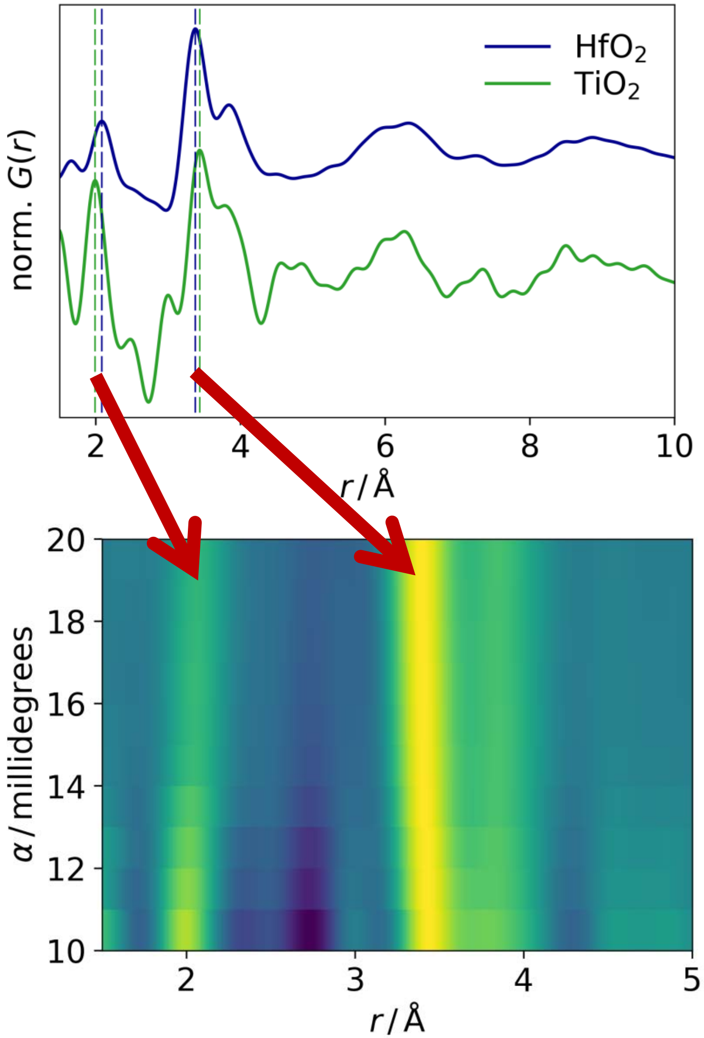
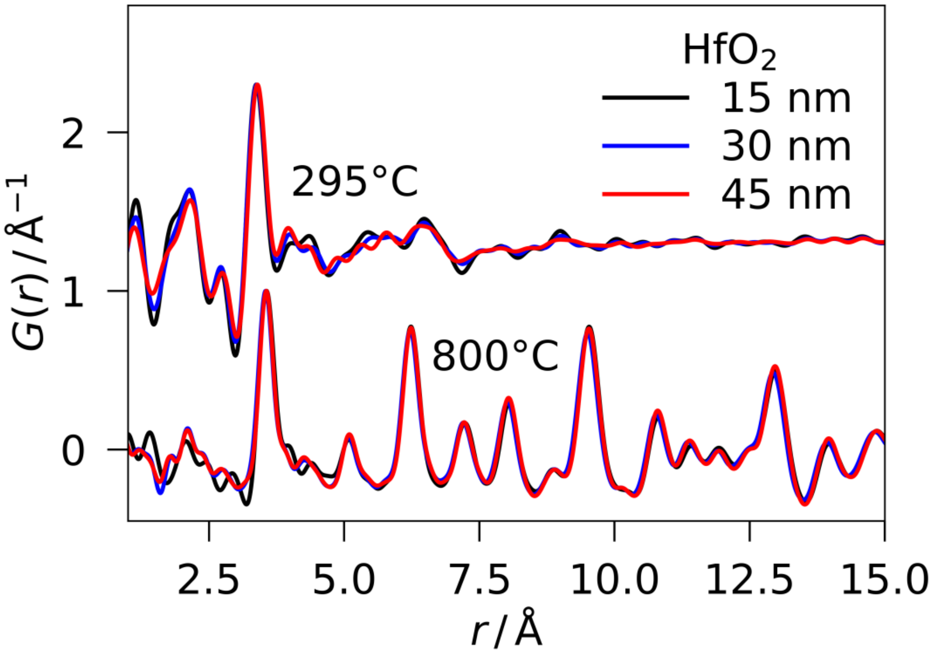


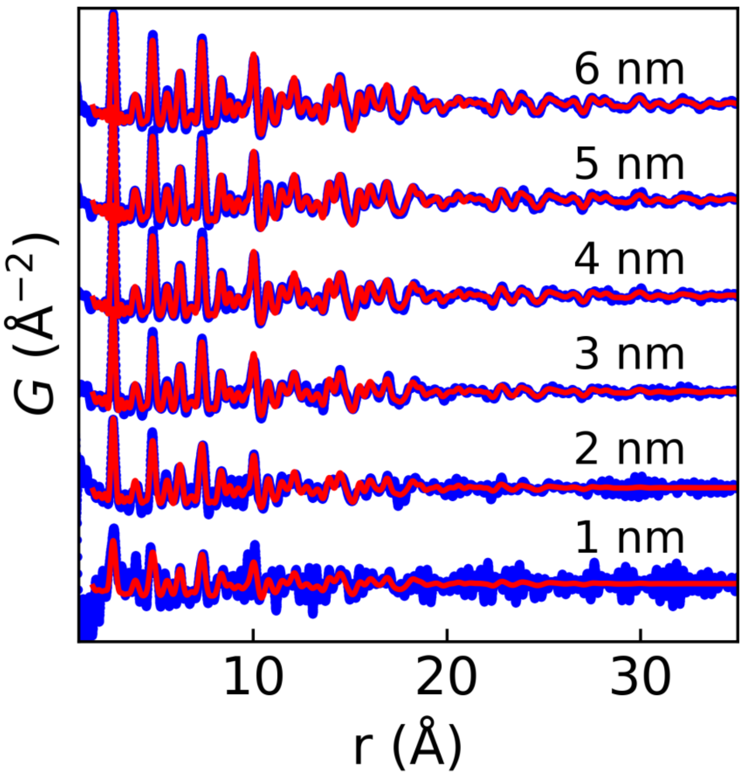


detection limit

comparison of results for different materials and degrees of ordering

- detection limit rather independent of degree of ordering but highly dependent on x-ray scattering power of the sample

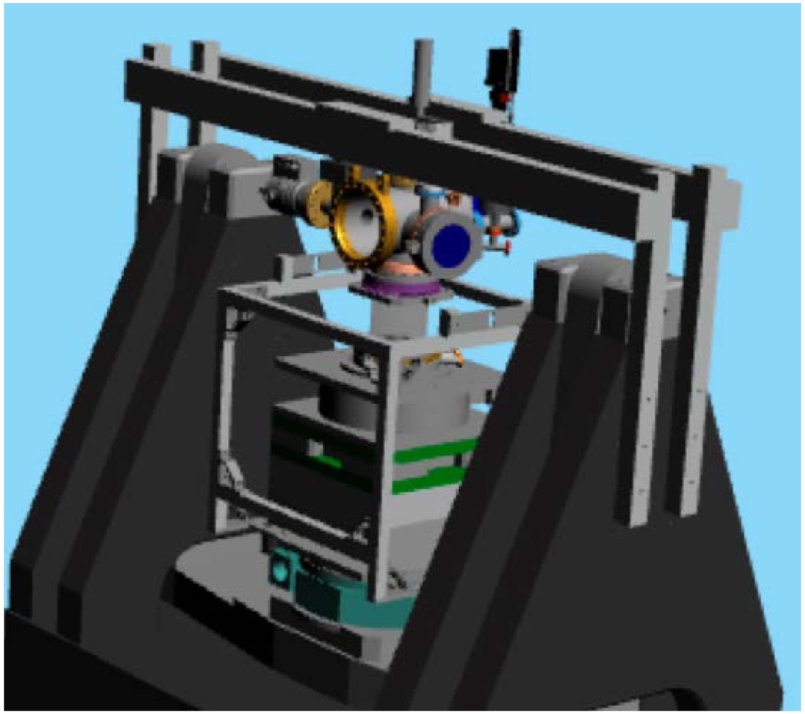


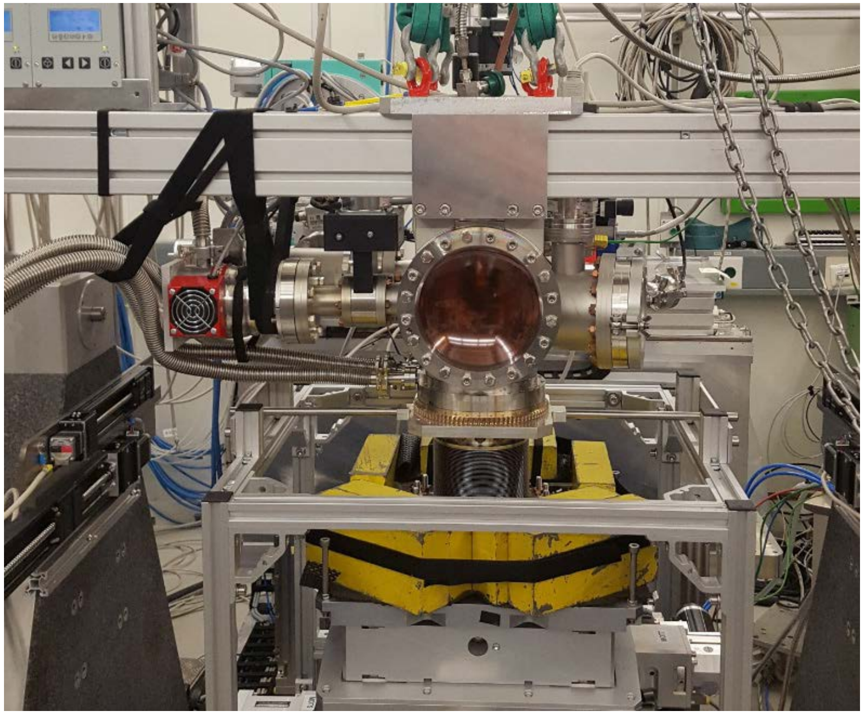


time-resolved PDFs of Pt sputter deposition

evolution of short and long range order

- dedicated rf magnetron sputtering chamber for grazing incidence diffraction



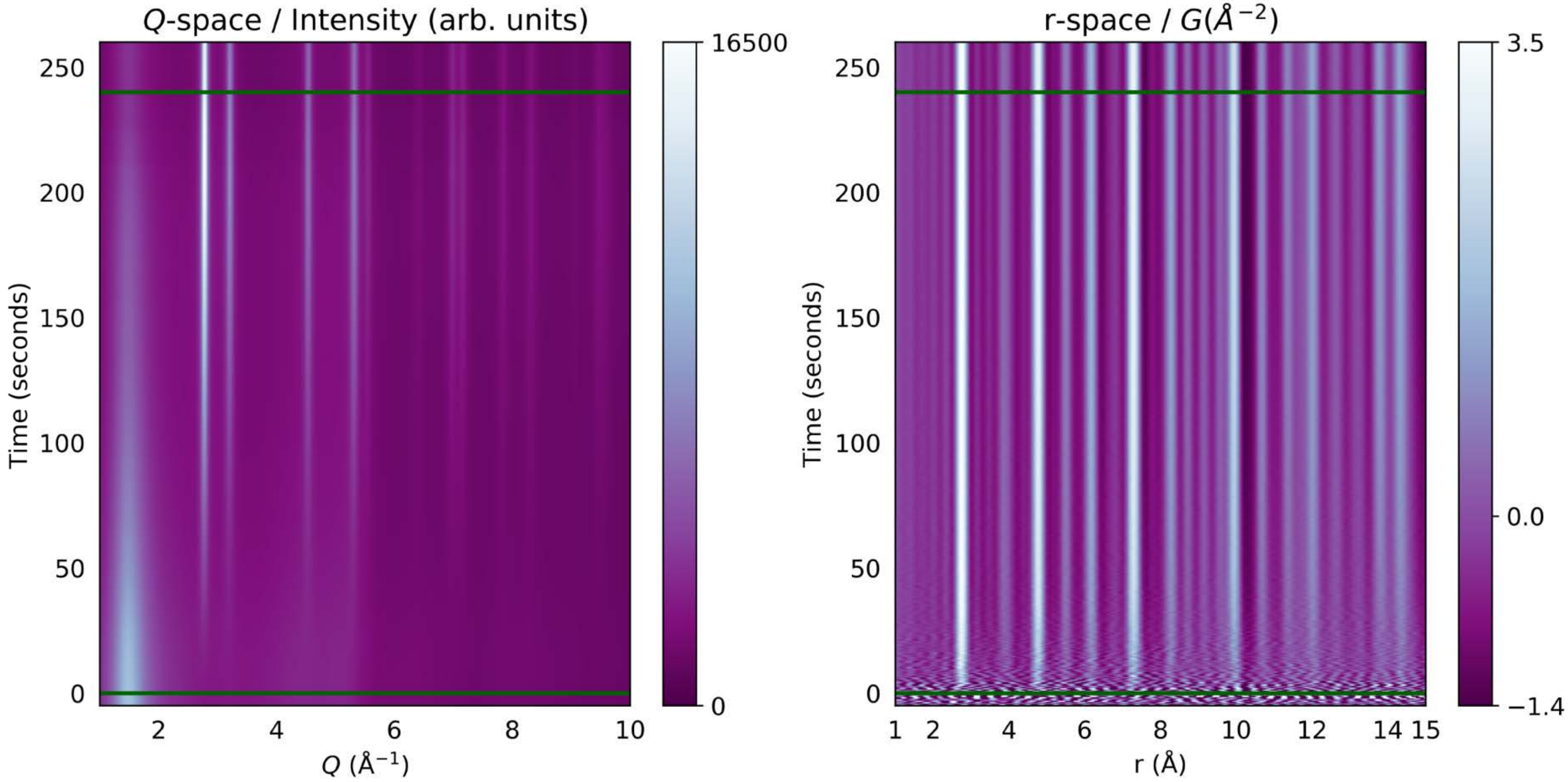


M. Roelsgaard *et al.*, IUCrJ 6 (2019) 299.

time-resolved PDFs of Pt sputter deposition

evolution of short and long range order

- *in situ* PDF study of strain in Pt thin films during thin film growth
- extrapolated deposition rate 1 Å s^{-1}, exposure time 0.5 s



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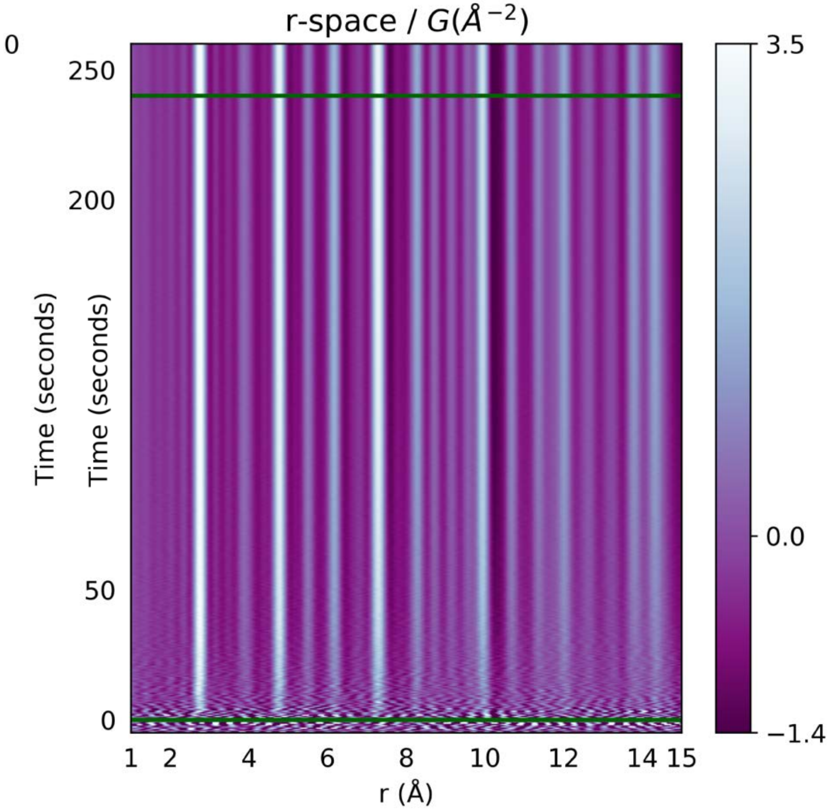
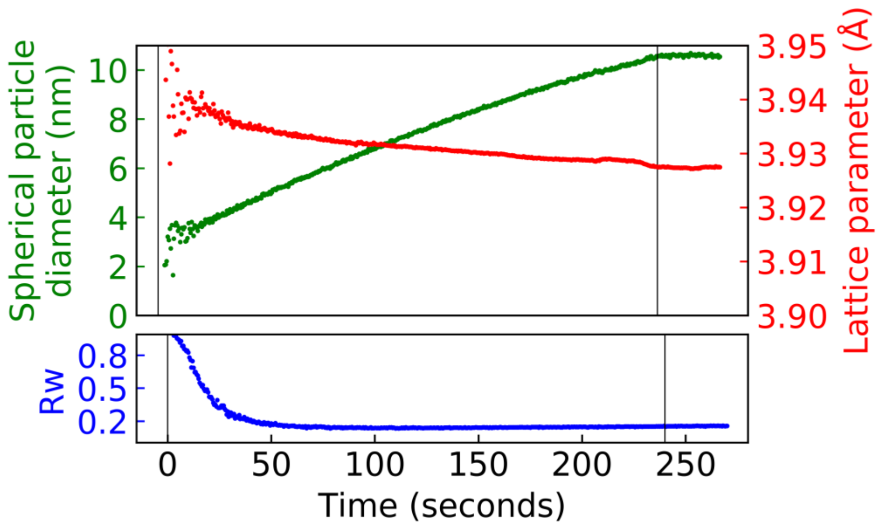
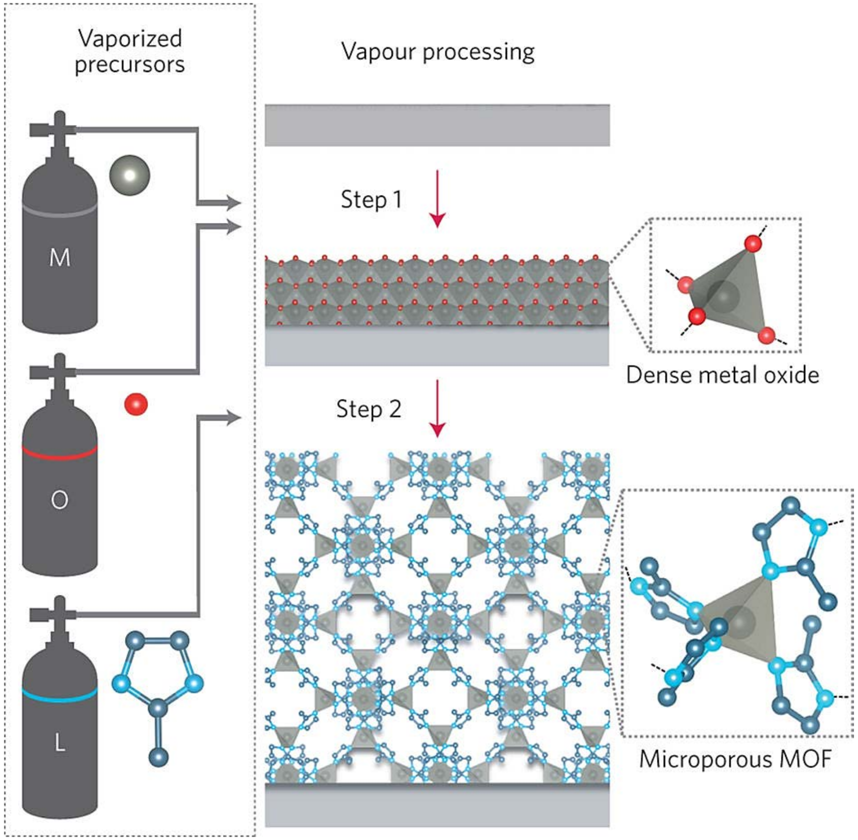




M. Roelsgaard *et al.*, IUCrJ 6 (2019) 299.

MOF film grown by atomic layer deposition

chemical vapor deposition process for ZIF-8 (MOF-CVD)



1 **oxide precursor deposition**

ALD **ZnO**: diethylzinc + water

$$Zn(C_2H_5)_2 + H_2O_{(g)} \rightarrow \boldsymbol{ZnO} + 2C_2H_{6\,(g)}$$

2 **vapor-solid reaction**

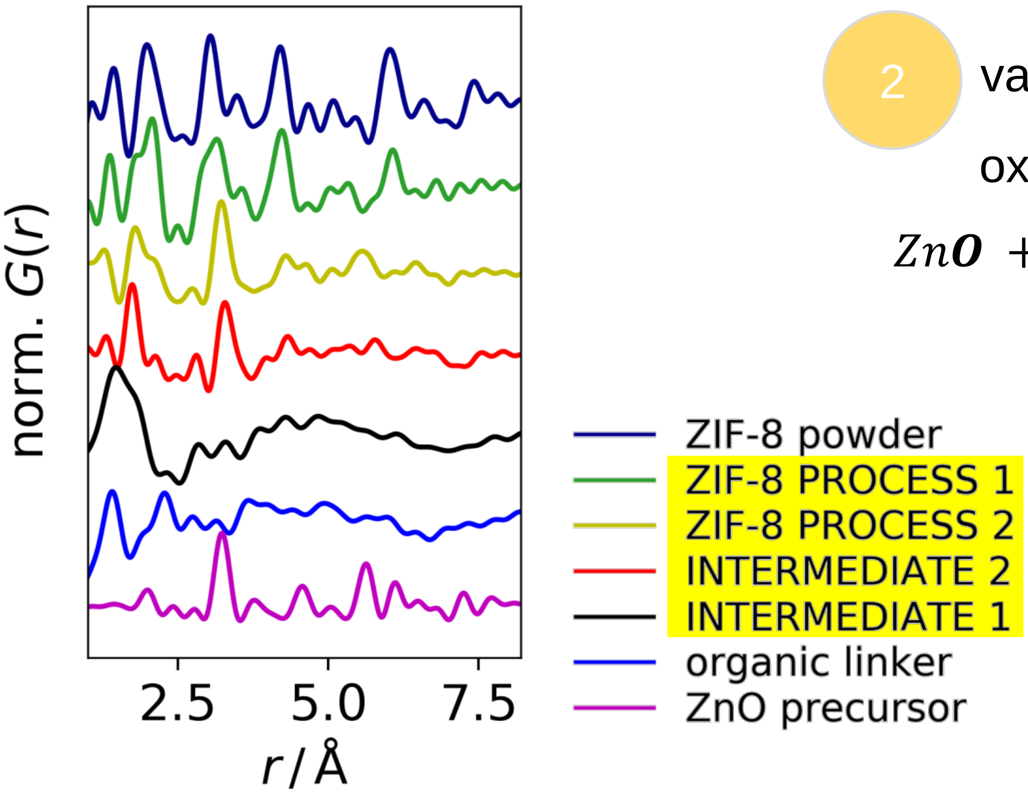
oxide-to-MOF transformation

$$ZnO + 2\,HmIM \rightarrow H_2O + \boldsymbol{ZIF8}$$

I. Stassen *et al.*, Nat Mater, 15 (2016) 304

MOF film grown by atomic layer deposition

metal-organic chemical vapor deposition (MOF-CVD) process for ZIF-8



2 vapor-solid reaction

oxide-to-MOF transformation

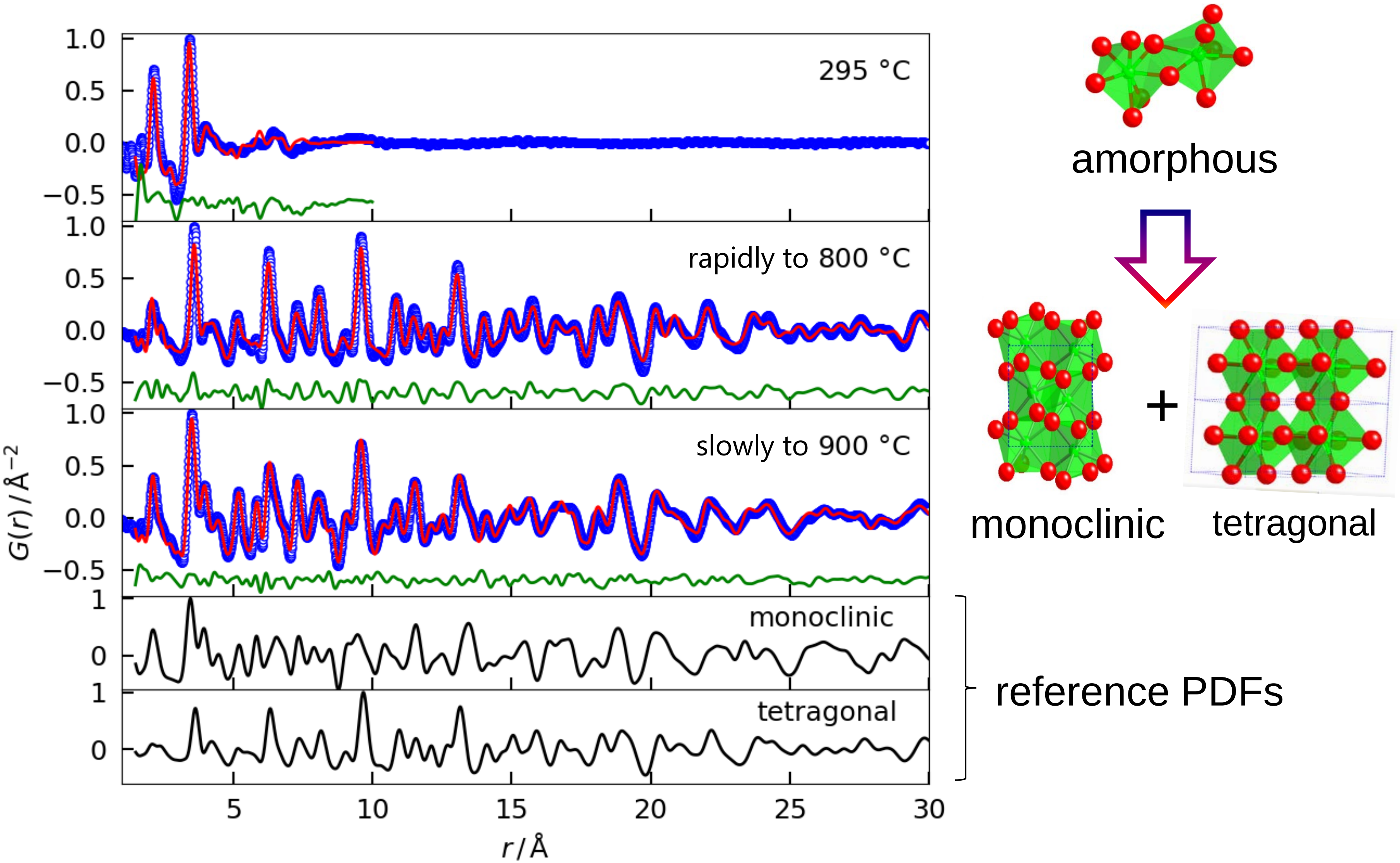
$$Zn\boldsymbol{O} + 2\,\boldsymbol{H}mIM \rightarrow H_2\boldsymbol{O} + \boldsymbol{ZIF8}$$

similar study for wet-chemical synthesis of ZIF-8 by Terban *et al.*
Nanoscale 10 (2018) 4291.

➔ observe ZIF film formation *in situ* in MOCVD reactor

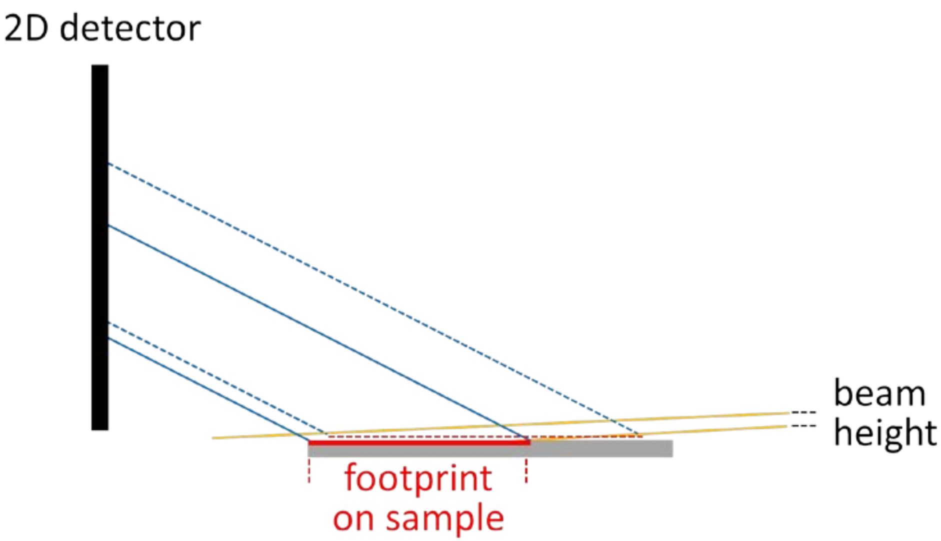
polymorphism of spin-coated ZrO_2 thin films

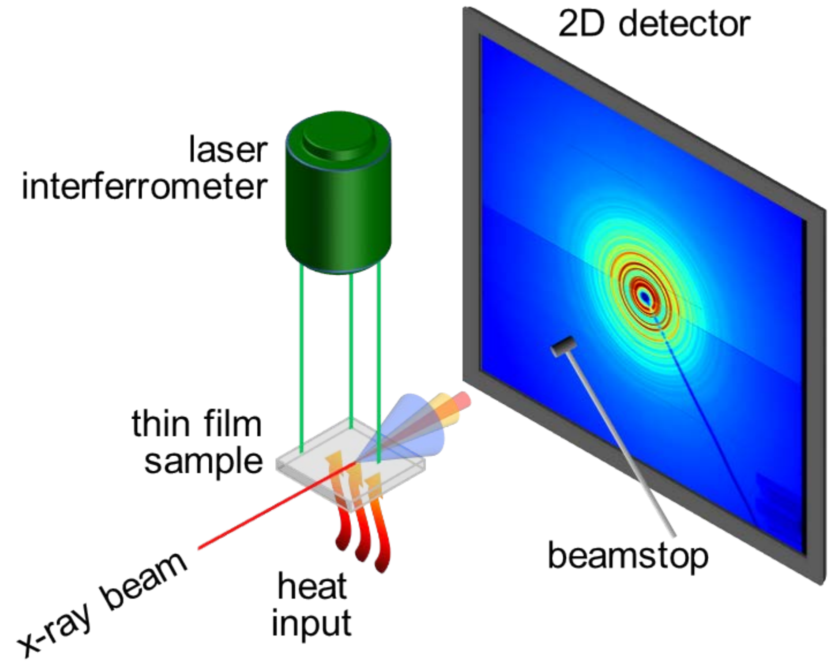
real space Rietveld refinements



variable temperature grazing incidence PDF

sample stabilization concept





keep sample position stable within

➔ < 1 µm in height

➔ ~ 0.001° in incidence angle

PIER

Partnership of
Universität Hamburg and DESY

A.-C. Dippel *et al.*, PIER Seed Project 2018, www.pier-hamburg.de

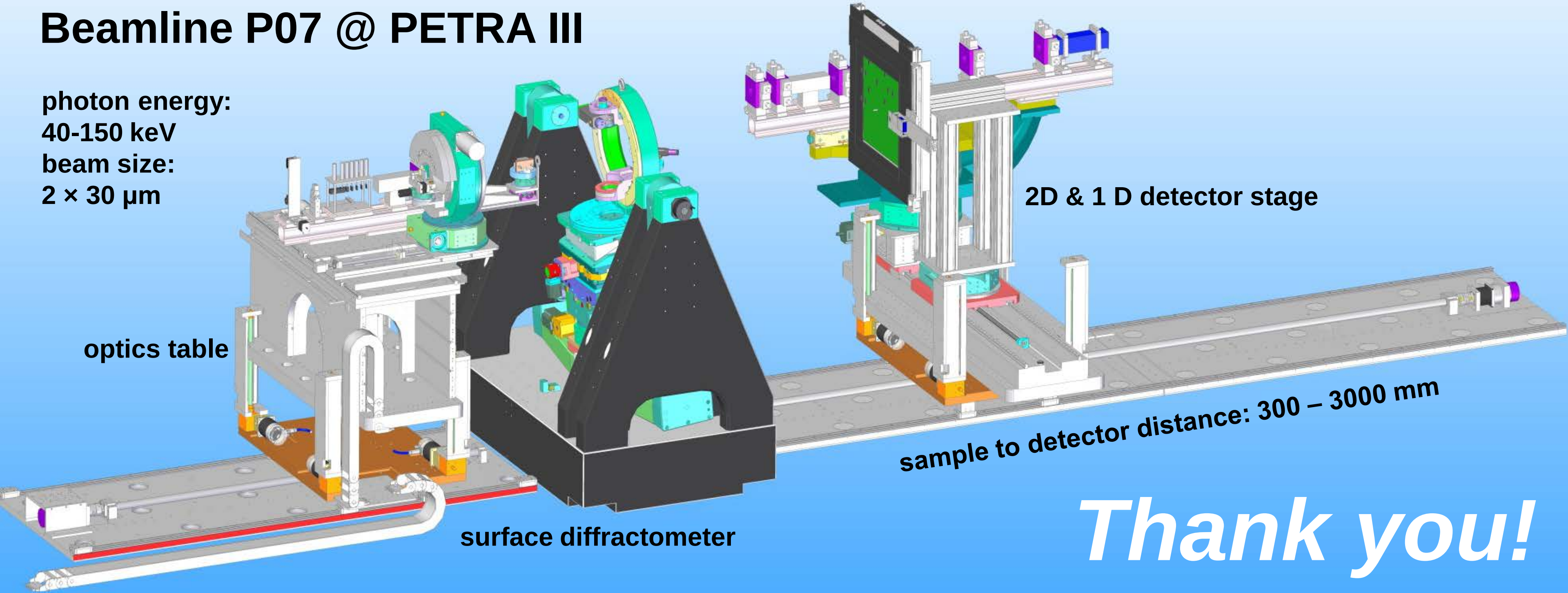
take home message

current and future *in situ* grazing incidence PDF capabilities

- models for texture and preferred orientation are work in progress
- linking properties to atomic structure for real layer systems
- *in situ* thin film growth studies, *e.g.* strain evolution in metallic films
- understanding crystallization kinetics and polymorphism in oxide films

Beamline P07 @ PETRA III

photon energy:
40-150 keV
beam size:
2 × 30 µm



Thank you!