Wolfgang Kreuzpaintner

2005 – 2010: PhD student

2010 – 2015: postdoc, 2015 – 2018: Akademischer Rat a.Z.

2018 – 2019: research fellow

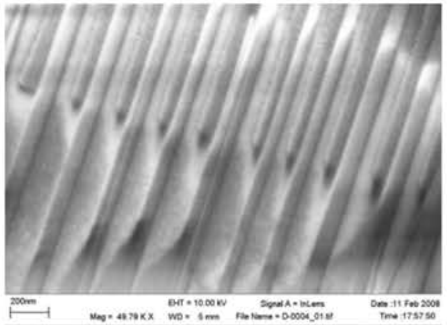
Since 2019: researcher / full professor

Wolfgang.Kreuzpaintner@googlemail.com

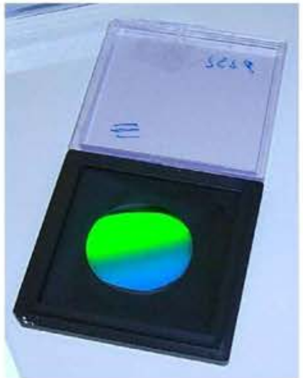
Presenter Background

Scientific background

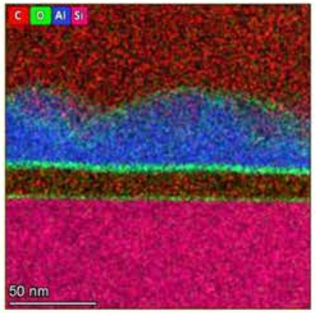
- Thin film, multilayer and interface magnetism, magnetic nanostructures
- energy materials



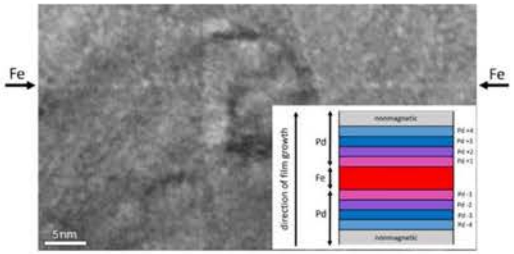
Gd nanowires



Ni nanodots



PTB-7



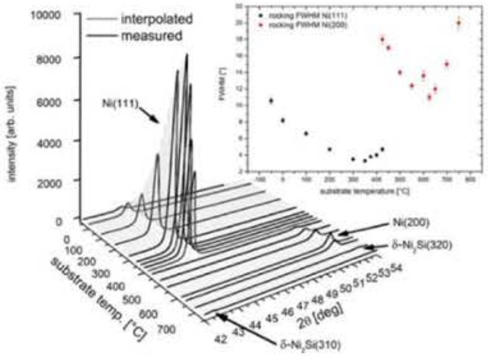
Pd/Fe(1ML)/Pd

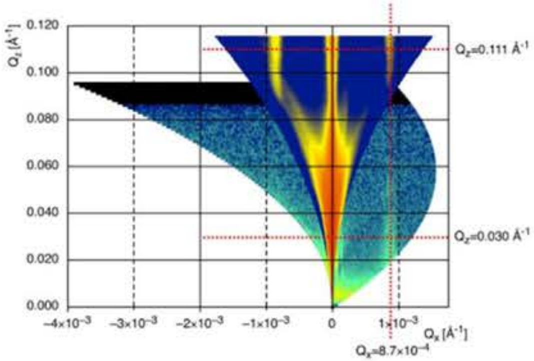
Specialised thin films of high quality

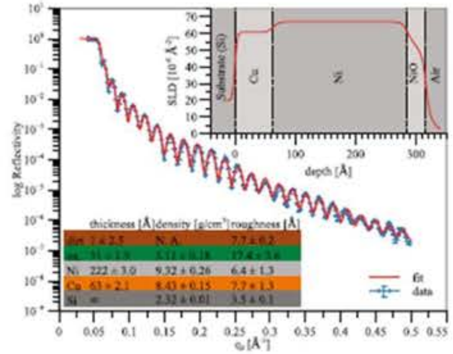
nature

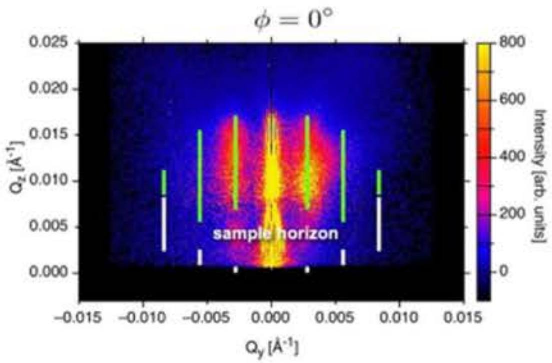
S. Tauchert, M. Volkov, D. Ehberger, D. Kazenwadel, M. Evers, H. Lange, A. Donges, A. Book, **W. Kreuzpaintner**, U. Nowak, and P. Baum, *Polarized phonons carry angular momentum in ultrafast demagnetization*, Nature 602, 73 – 77 (2022). https://doi.org/10.1038/s41586-021-04306-4

X-ray & Neutron scattering









User of various neutron reflectometers (mostly NERO, Amor, Maria, REFSANS)

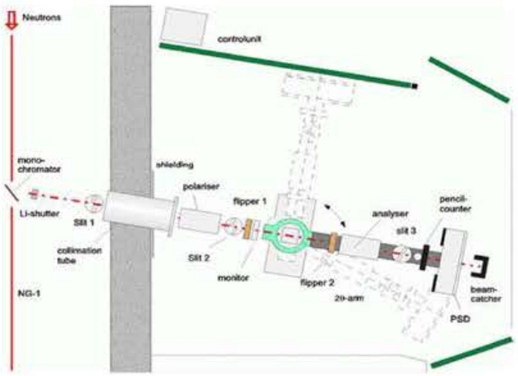
Presenter Background

Beamlines

- NERO: (monochromatic)

internal user and support with user operation
- pre-characterization of user samples (x-ray, PPMS, etc.)
- lab introduction
- support with data processing and analysis (specular/off-specular & polarized/unpolarized)

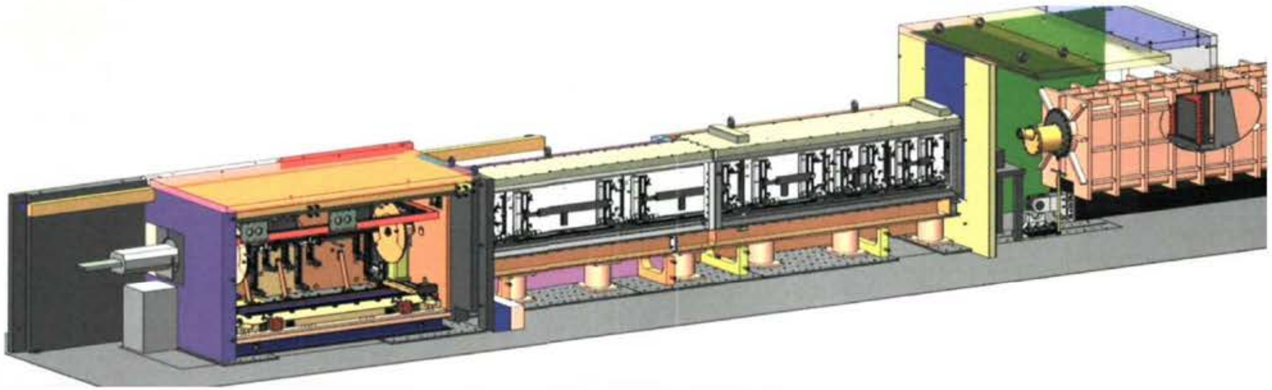
NERO @ GENF:



- REFSANS: (ToF)

internal and friendly user (taking part in the commissioning)
- debugging (2D ToF detector → own software to read and process list mode files)
- fixing (drives of neutron optics)
- improving (suggesting chopper upgrade → done in 2009/2010)
- improvise sample environments
- improvised polarizer

etc.



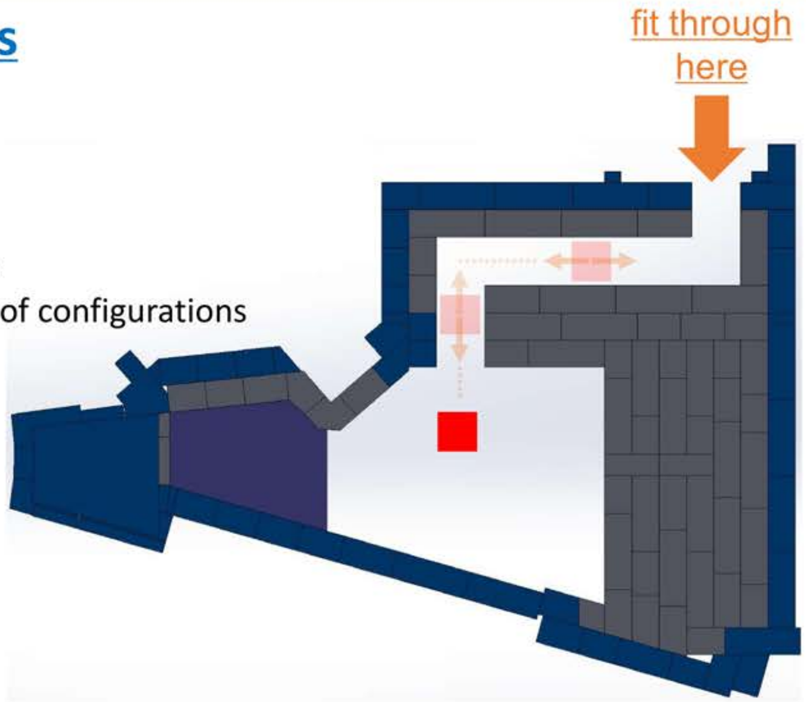
REFSANS @ FRM2

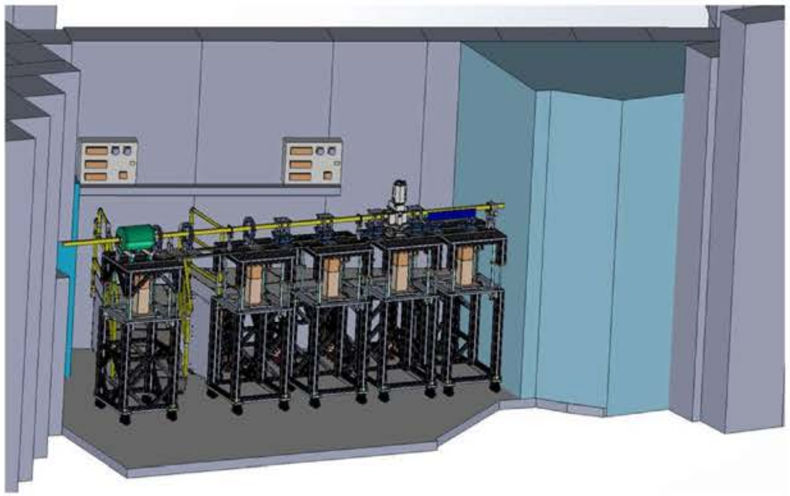
Presenter Background

Beamlines

BL20: - construction work for partially completed beamline
(ToF) - existing hutch, choppers, detectors

Challenges:
- test-beamline for 3 groups (detector, chopper and polarized neutrons
- Groups have very different requirements → simple and quick change of configurations





Presenter Background

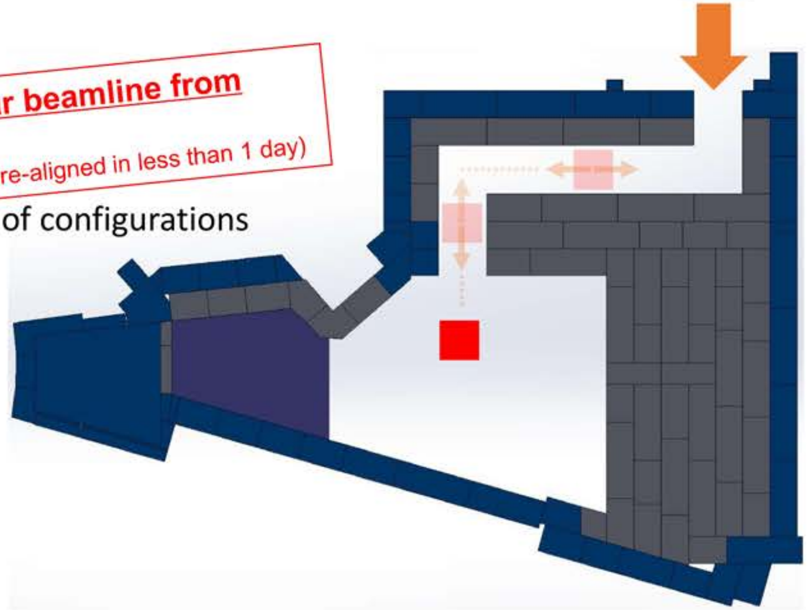
Beamlines

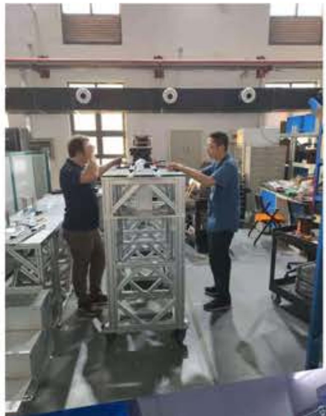
BL20: - construction work for partially completed beamline
(ToF) - existing hutch, choppers, detectors

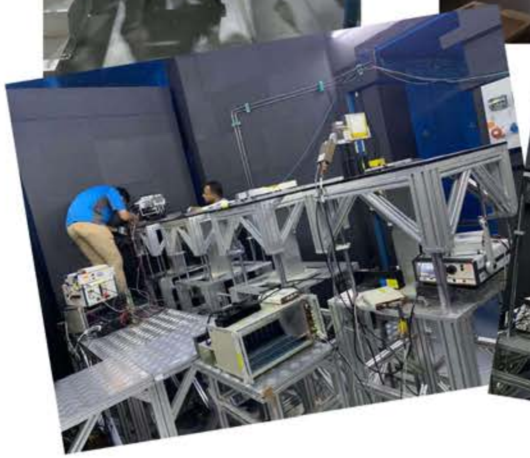
Challenges:
- test-beamline for 3 groups (det
- Groups have very different requ ... quick change of configurations

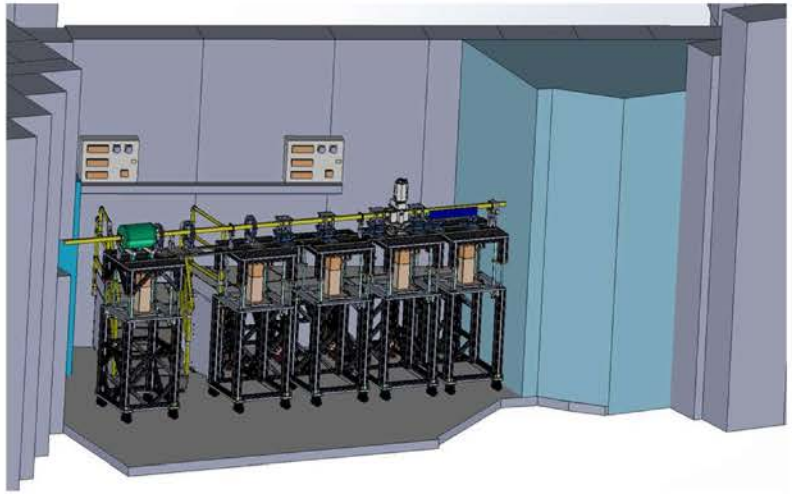
Design and build a fully modular beamline from "what exists"
(A beamline that can fully be (dis-)assembled and re-aligned in less than 1 day)

fit through here



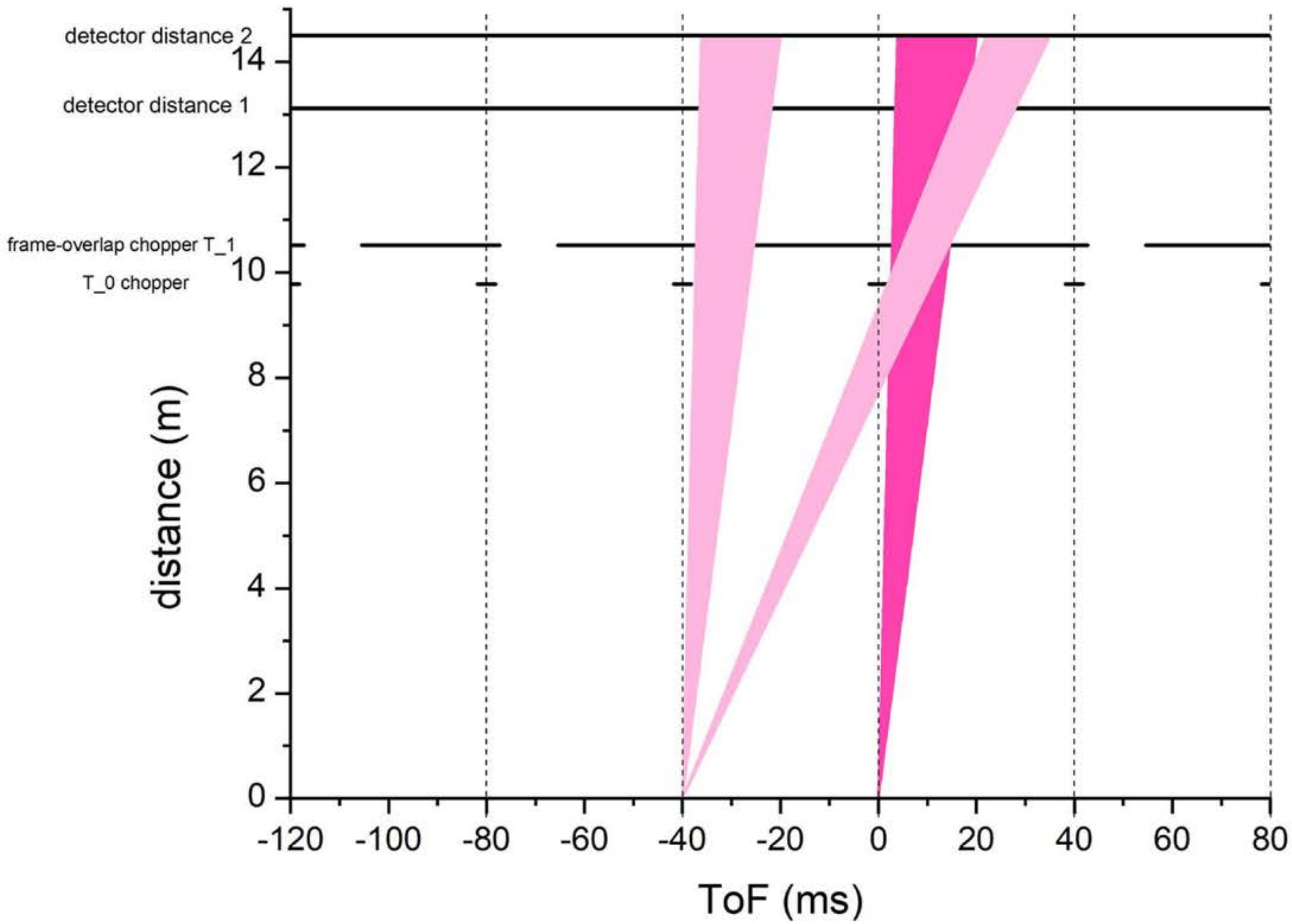






Presenter Background

Neutron Optics



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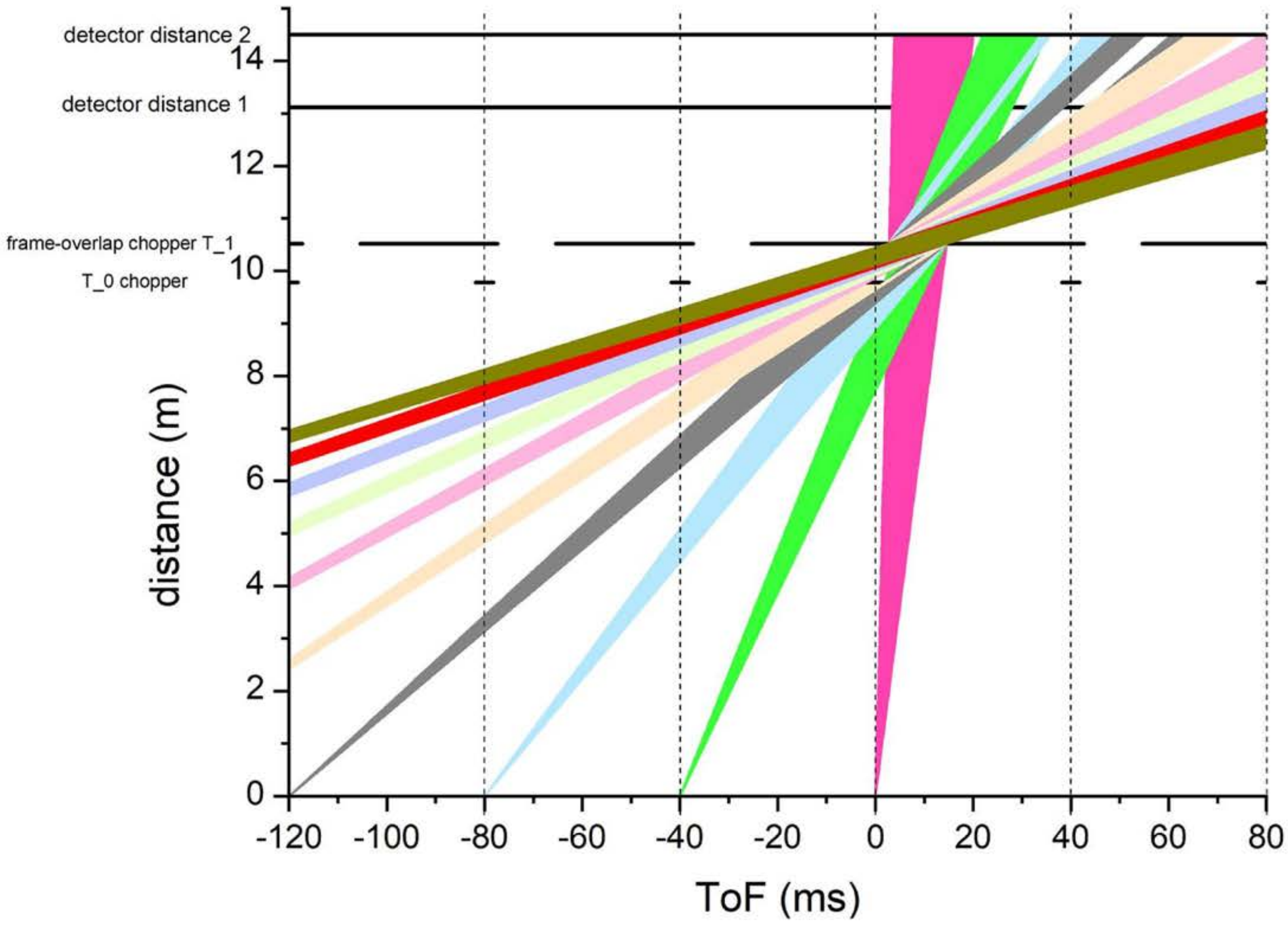
Neutron Optics



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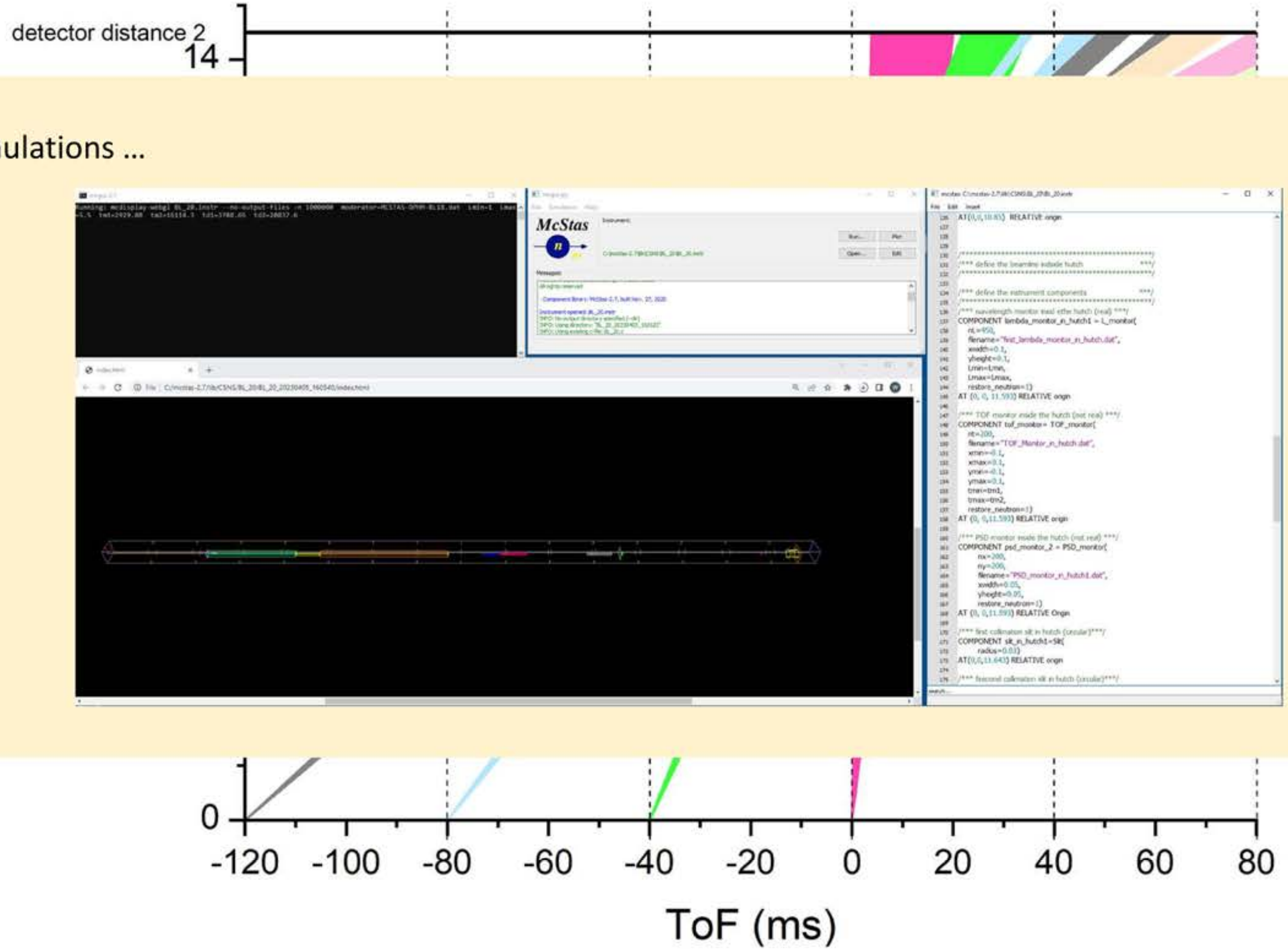
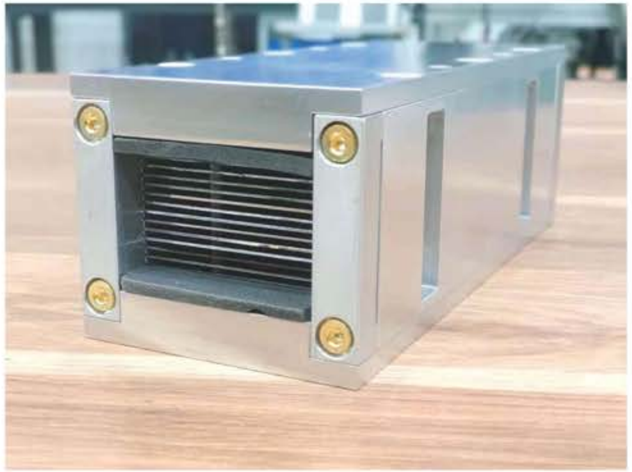
Neutron Optics

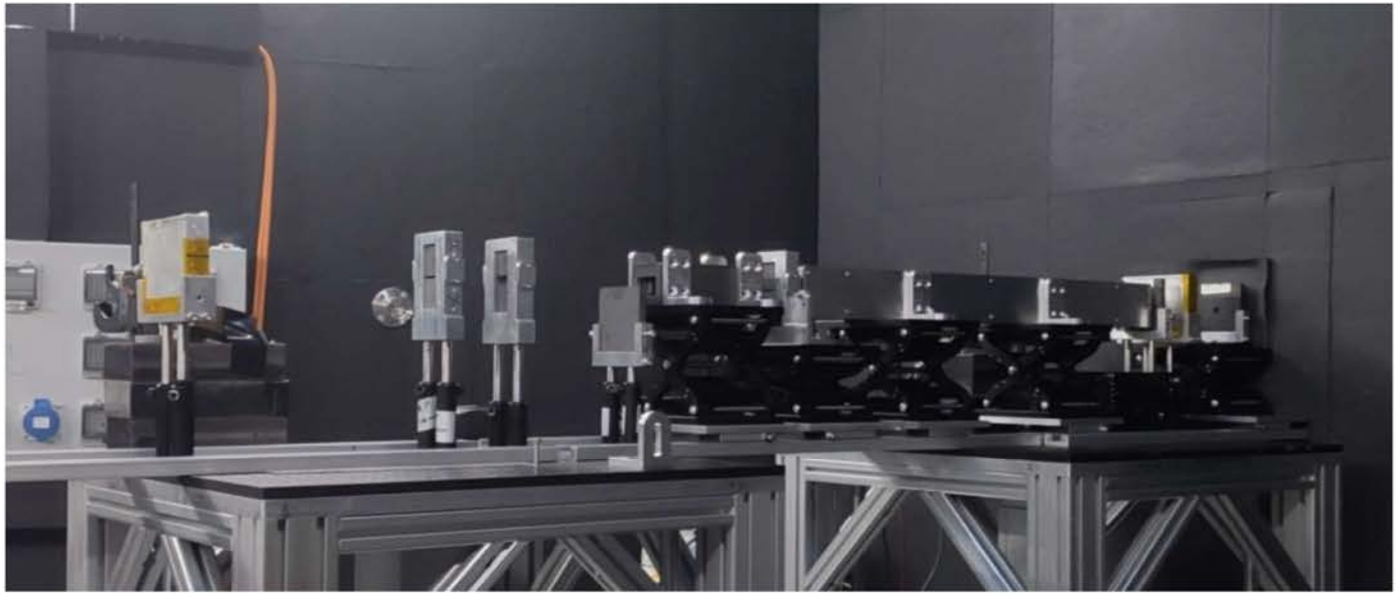


Simulations …

Presenter Background

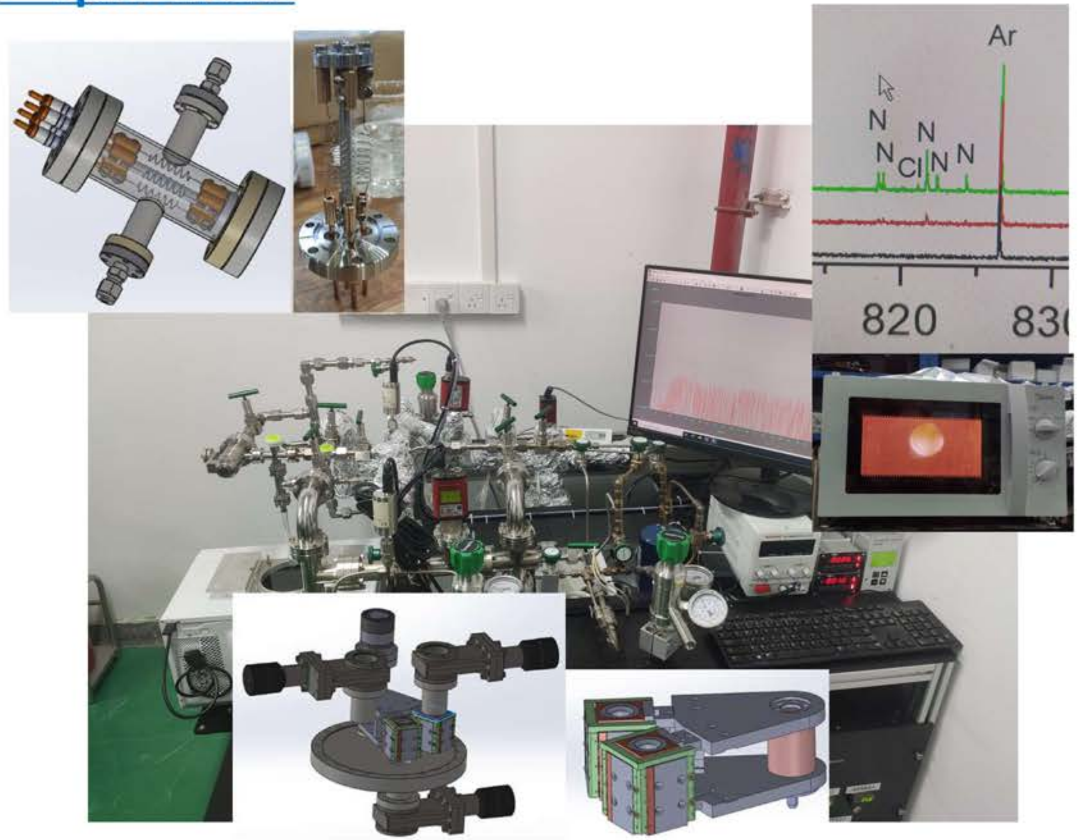
Neutron Optics





Presenter Background

^{3}He – spin filters



1st generation ^{3}He filling station

2nd generation ^{3}He filling station

Workhorse to fill cells:
- Mainly commercial components

Research-centered filling station:
- sophisticated pumping and gas analysis system
- Self-developed 3He gas purifier
- Microwave-discharge-cleaning
- etc.

Presenter Background

^{3}He – spin filters


Chinese National Patent granted

证书号第68

发明专利证书

发明名称：一种灌气站及其应用

发明人：田龙;秦泽聪;谢俊松;叶凡;罗杰羽;王天昊;童欣

专利号：ZL 2022 1 0687189.0

专利申请日：2022年06月16日

专利权人：散裂中子源科学中心;中国科学院高能物理研究所

地址：523808 广东省东莞市松山湖科技产业园区科学园生产力大厦

授权公告日：2024年03月26日　授权公告号：CN 115234829 B

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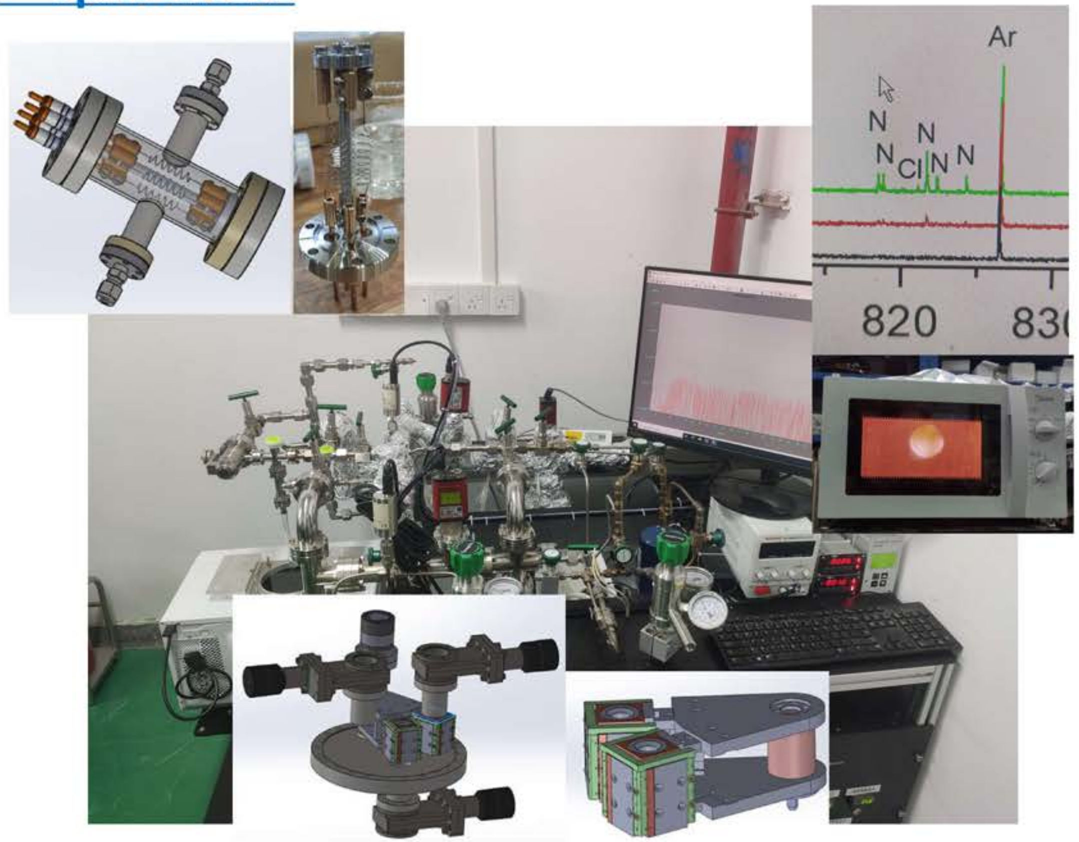
局长
申长雨

2024年03月26日

第1页(共2页)

其他事项参见续页



2nd generation ^{3}He filling station

Research-centered filling station:

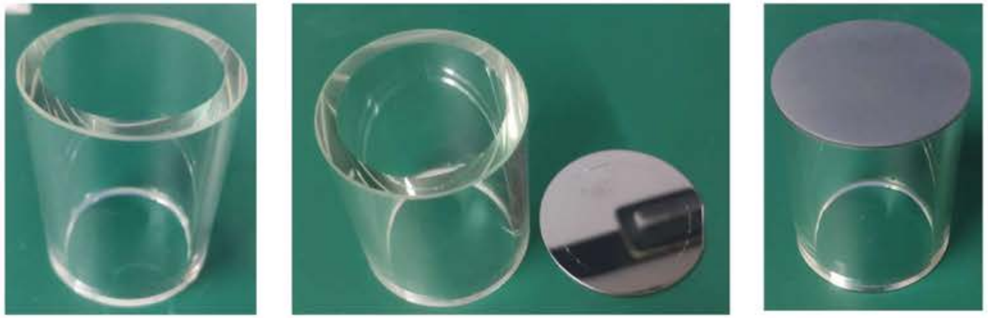
- sophisticated pumping and gas analysis system
- Self-developed 3He gas purifier
- Microwave-discharge-cleaning
- etc.

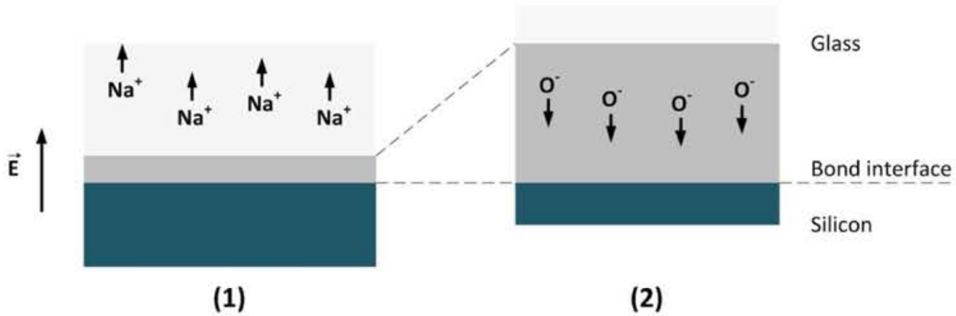
Presenter Background

^{3}He – spin filters

Solving the GE180 glass crisis … in a new way!

Apply a coating to the inside of a glass cell to control the interaction of 3He with the surface?

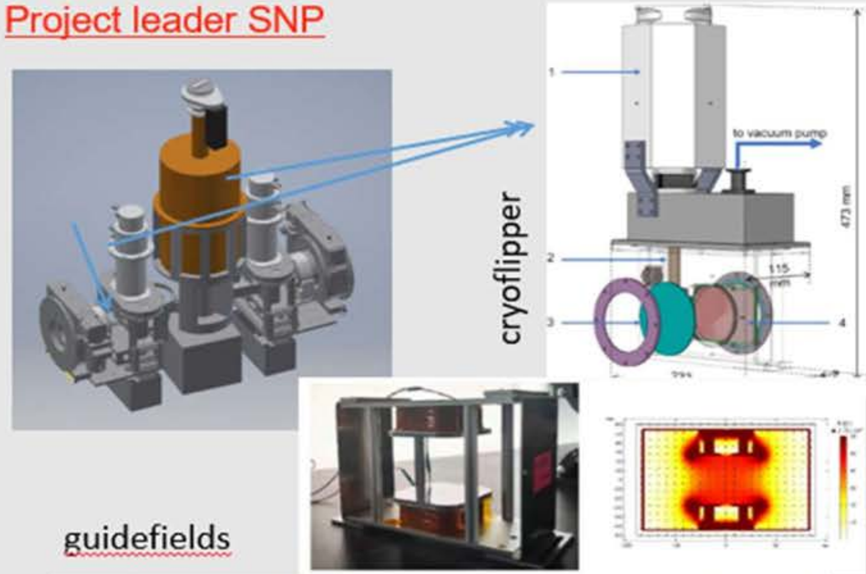




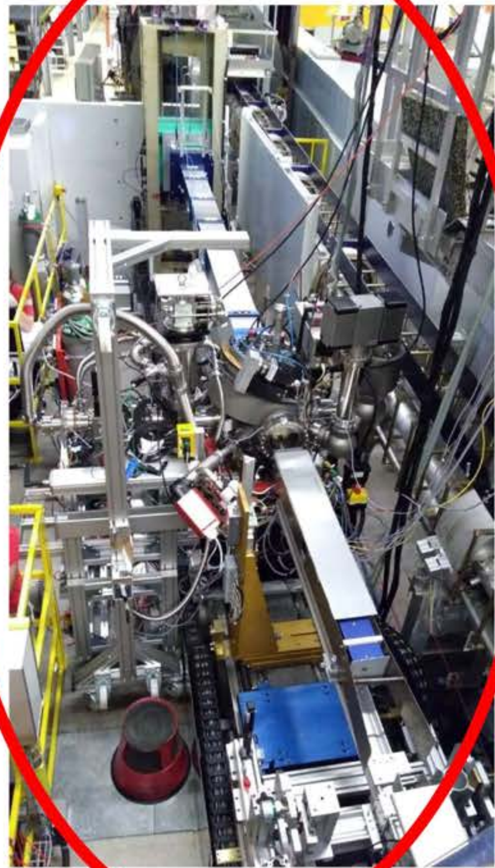
Presenter Background

Sample Environments

Project leader SNP



Y.-C. Dong, T.-H. Wang, **W. Kreuzpaintner**, X.-T. Liu, Z.-H. Li, Y.-D. Kang, J.-P. Zhang, L. Tian, C.-Y. Huang, B. Bai, and X. Tong, *Miniaturized time-of-flight neutron spin flipper using a high-T_C superconductor*, Nucl. Sci. Tech. 33, 145 (2022). https://doi.org/10.1007/s41365-022-01134-7



Patent pending in China

Development of *In Situ* Thin Film Growth Capabilities for Polarized Neutron Reflectometry

Wolfgang Kreuzpaintner

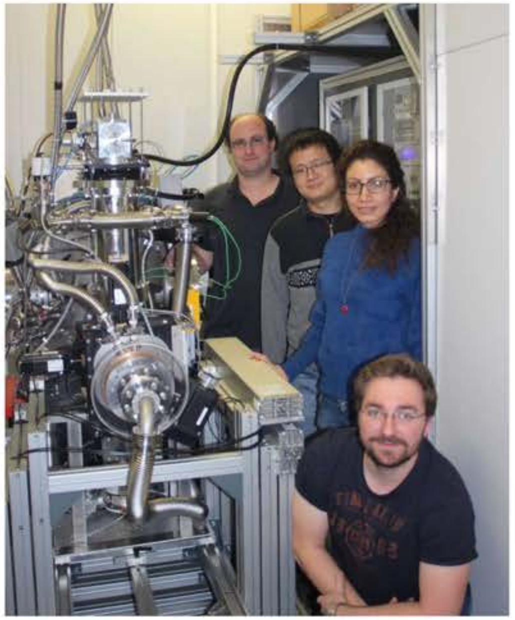
For a review on the topic:

W. Kreuzpaintner, A. Schmehl, A. Book, T. Mairoser, J. Ye, B. Wiedemann, S. Mayr, J.-F. Moulin, J. Stahn, D.A. Gilbert, H. Gabold, Z. Inanloo-Maranloo, M. Heigl, S. Masalovich, R. Georgii, M. Albrecht, J. Mannhart, and P. Böni, *Reflectometry with Polarized Neutrons on In Situ Grown Thin Films*, Phys. Status Solidi B, 2100153 (2021).
https://doi.org/10.1002/pssb.202100153

Outline

Introduction

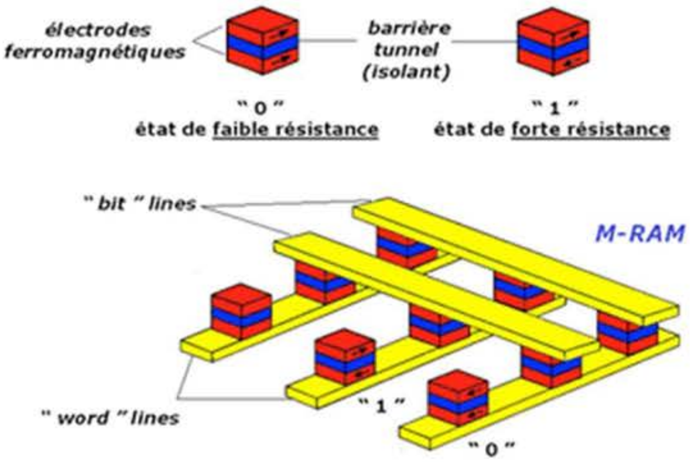
- *In situ* Thin Film Deposition Setup
- Early *in situ* Experiments
- Speeding up the Measurements
- Current Experiments / Possibilities
- Latest Developments

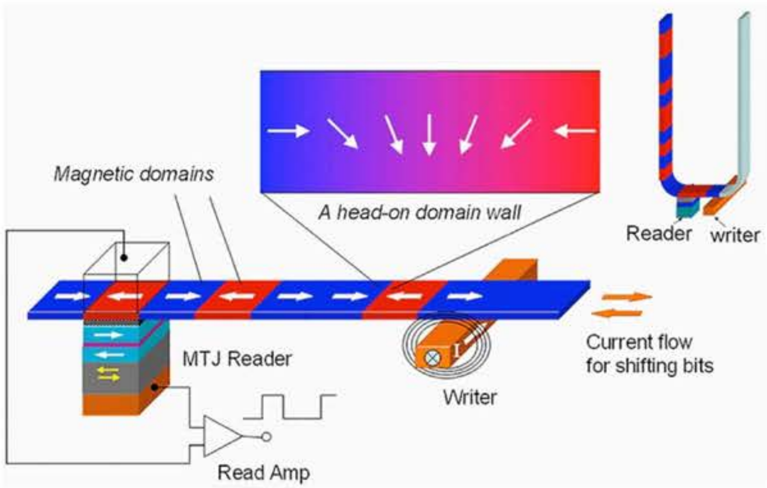


Conclusion and Outlook

Introduction

Magnetic layers and hetero-structures are the basic building blocks of a large number of magneto-electronic devices.





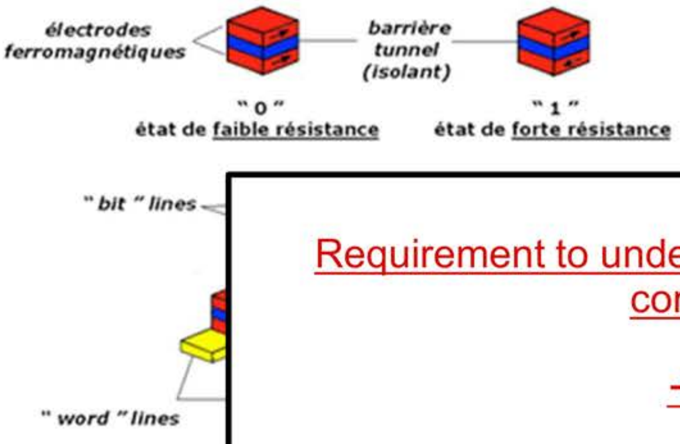
http://www2.cnrs.fr/sites/en/image/fig4albertfert.gif

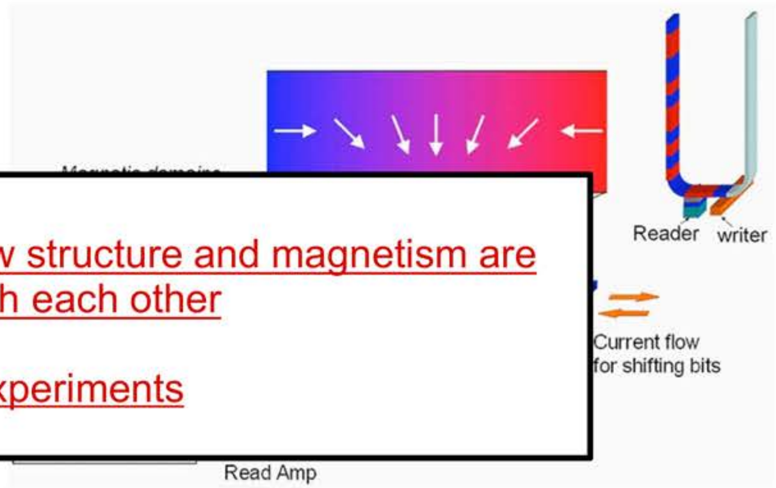
Performance of all devices:

- strongly relies on the magnetic properties of the layers
- is a function of the morphology and microstructure of the layers (functions of growth conditions).

Introduction

Magnetic layers and hetero-structures are the basic building blocks of a large number of magneto-electronic devices.





Requirement to understand how structure and magnetism are correlated with each other

→ *In situ* experiments

http://www2.cnrs.fr/sites/en/image/fig4albertfert.gif

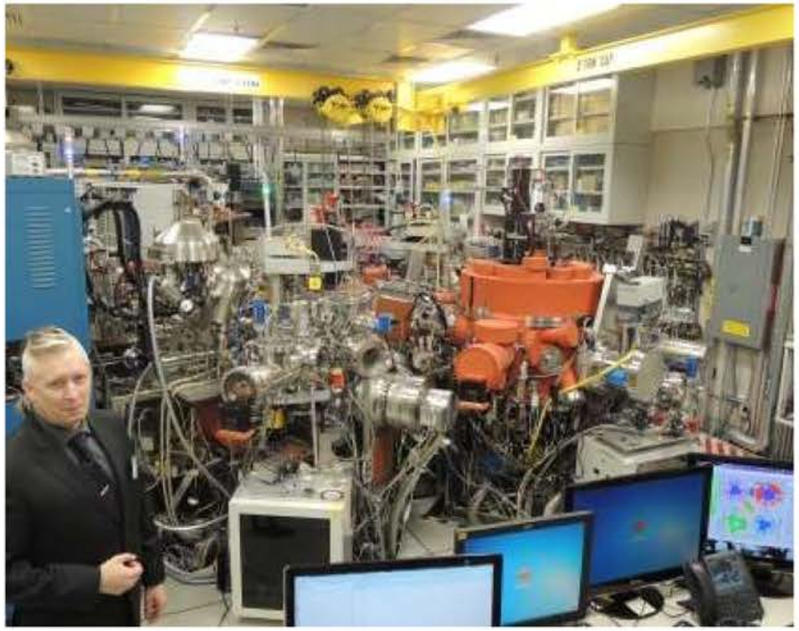
Performance of all devices:

- strongly relies on the magnetic properties of the layers
- is a function of the morphology and microstructure of the layers (functions of growth conditions).

Introduction

Common Practice:

- Structural *in situ* characterization of thin films is common practice (LEED/RHEED, STM, x-ray reflectivity, GISAXS, etc.)
- Magnetic x-ray scattering (XMCD)



http://m.eet.com/media/1172820/1a%20overal%20lab%20x%20420.jpg

http://www.esrf.eu/files/live/sites/www/files/about/synchrotron-science/ESRF-02-386.jpg

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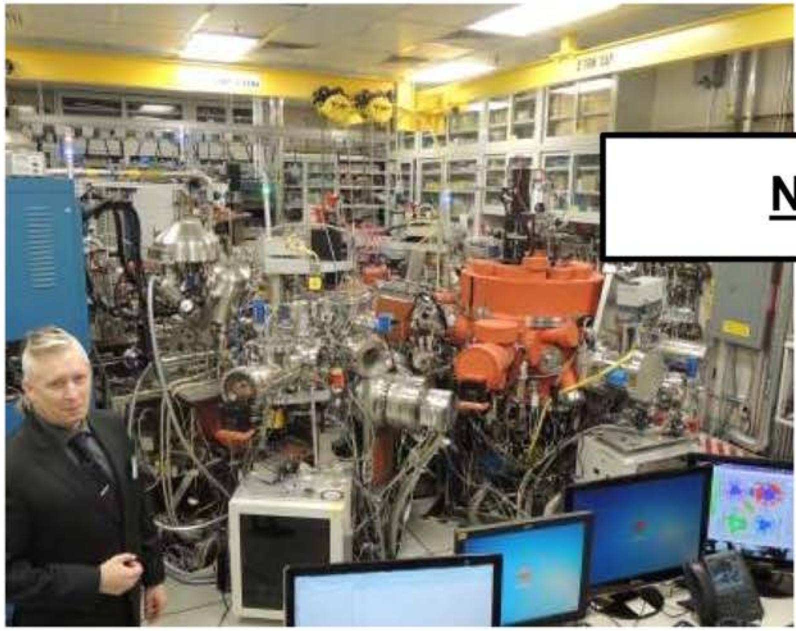


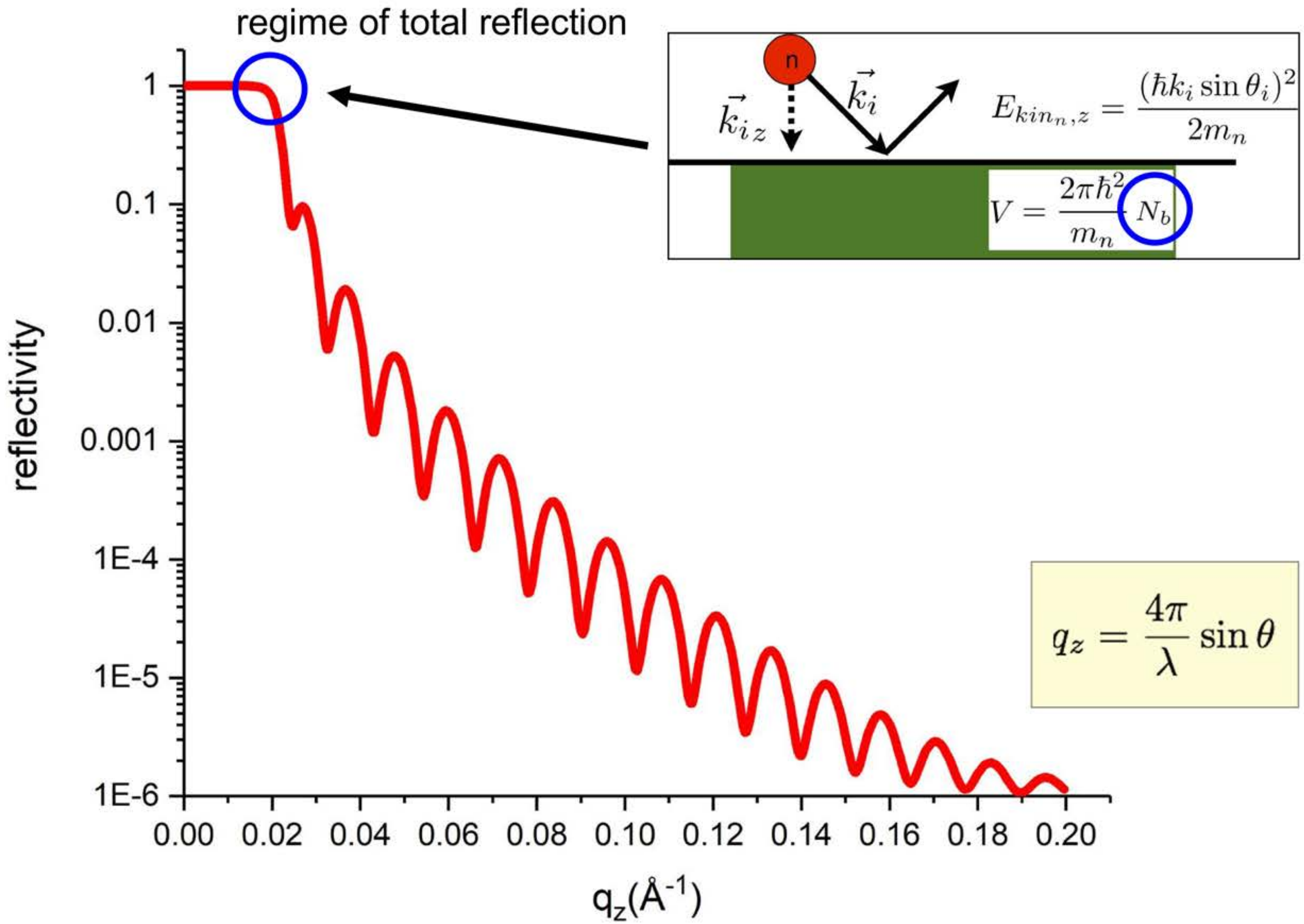
Neutrons ?

http://m.eet.com/media/1172820/1a%20overal%20lab%20x%20420.jpg

http://www.esrf.eu/files/live/sites/www/files/about/synchrotron-science/ESRF-02-386.jpg

Introduction

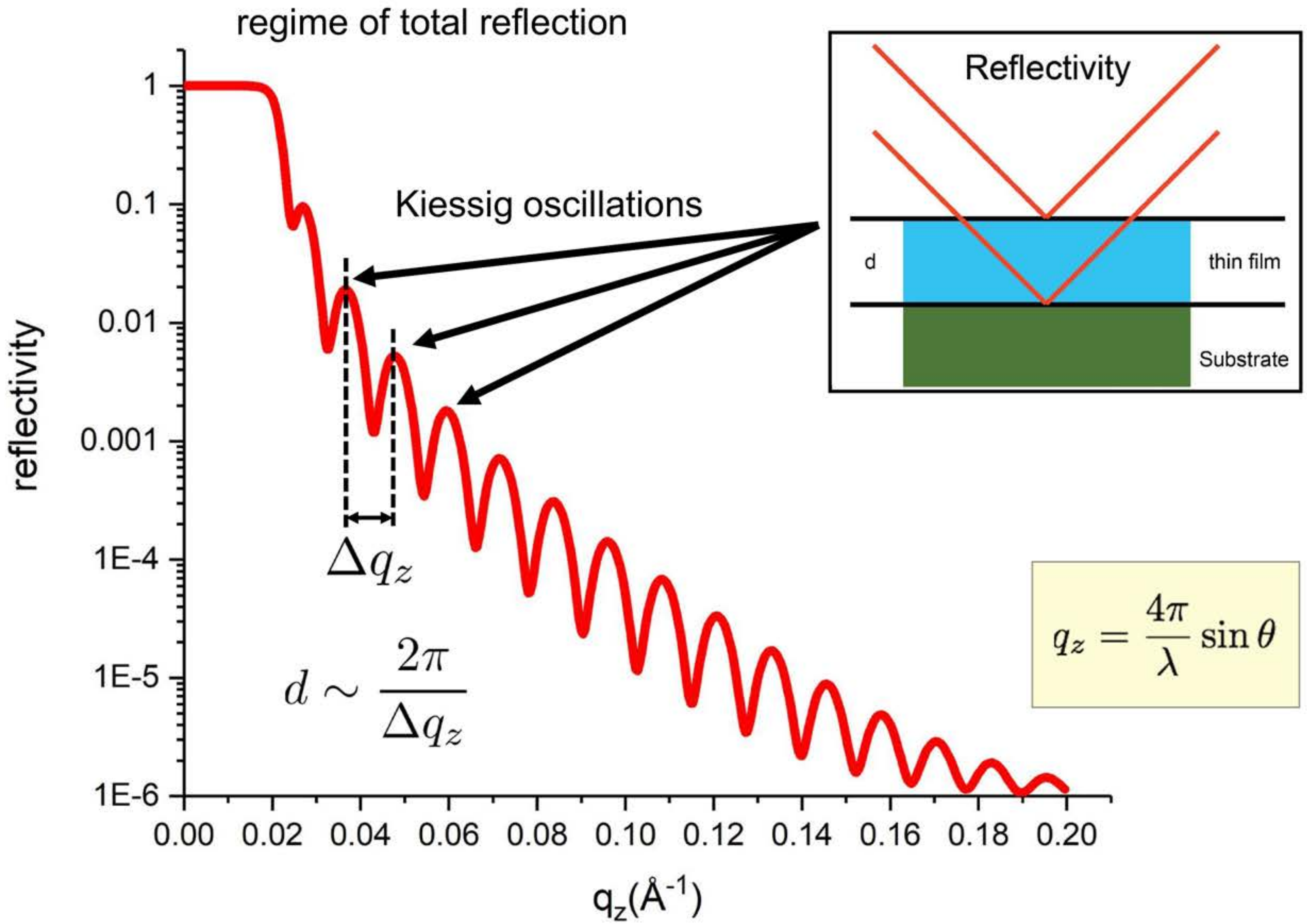
Polarized Neutron Reflectometry



$$q_z = \frac{4\pi}{\lambda} \sin\theta$$

Introduction

Polarized Neutron Reflectometry



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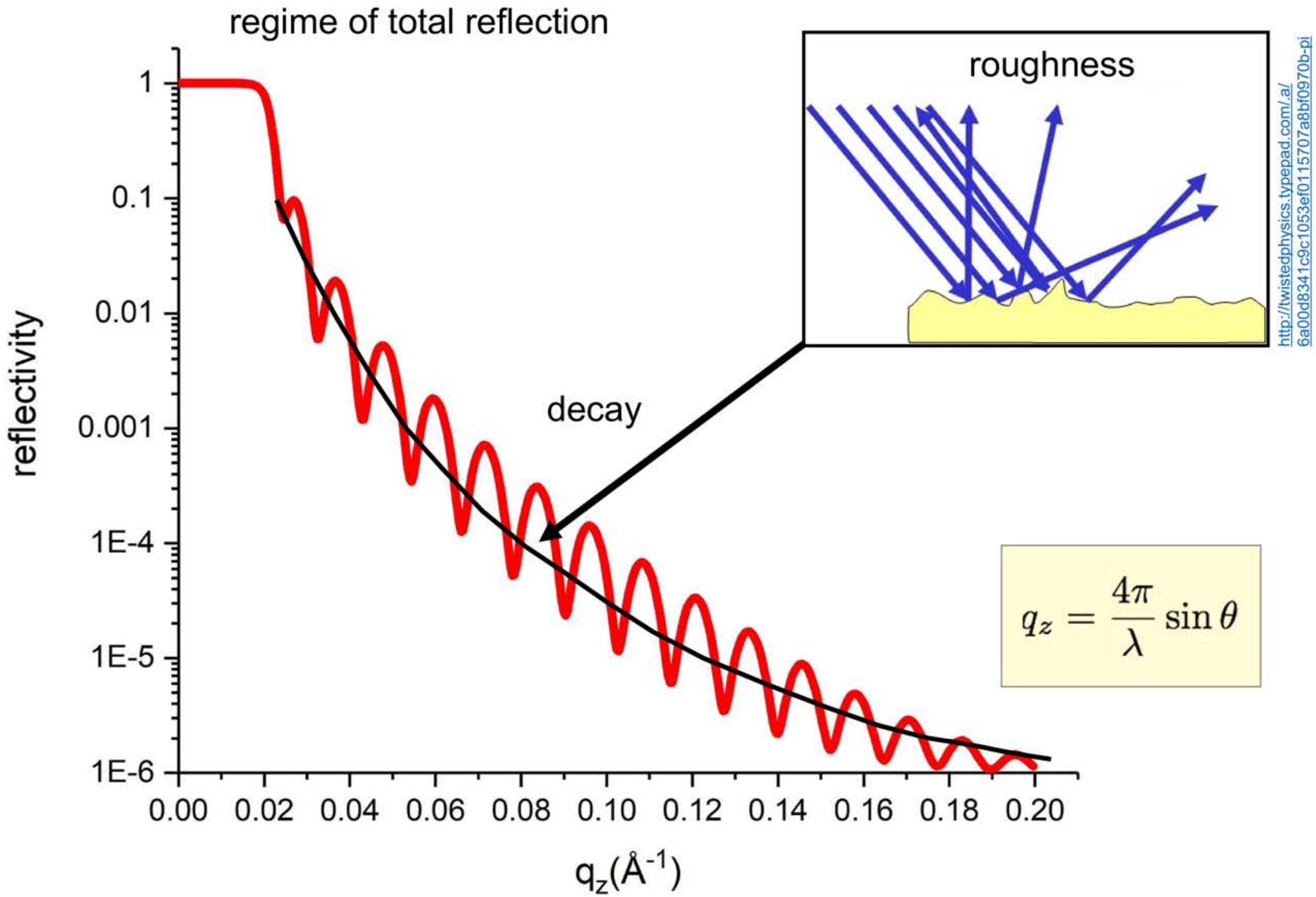
Polarized Neutron Reflectometry



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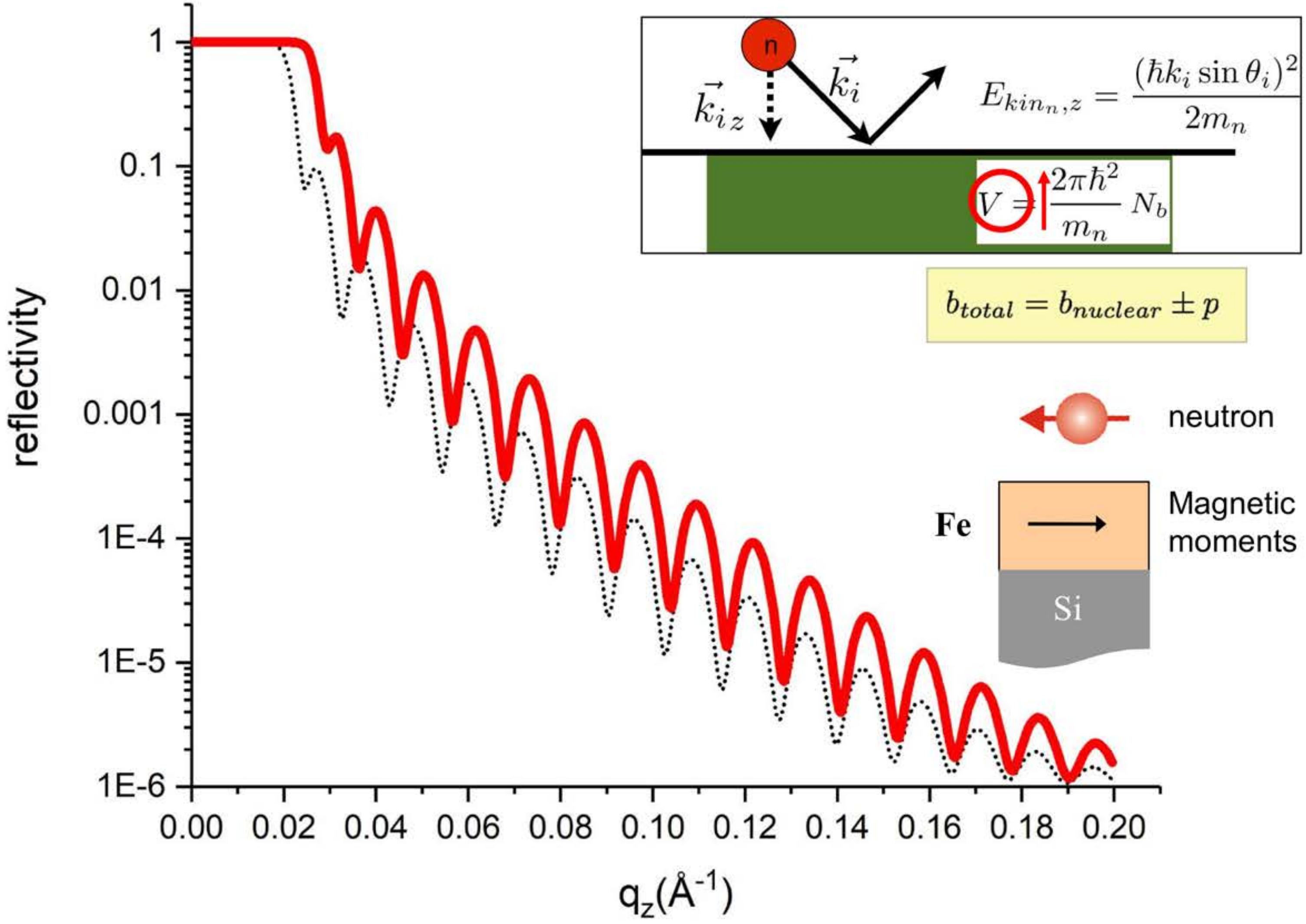
Introduction

Polarized Neutron Reflectometry



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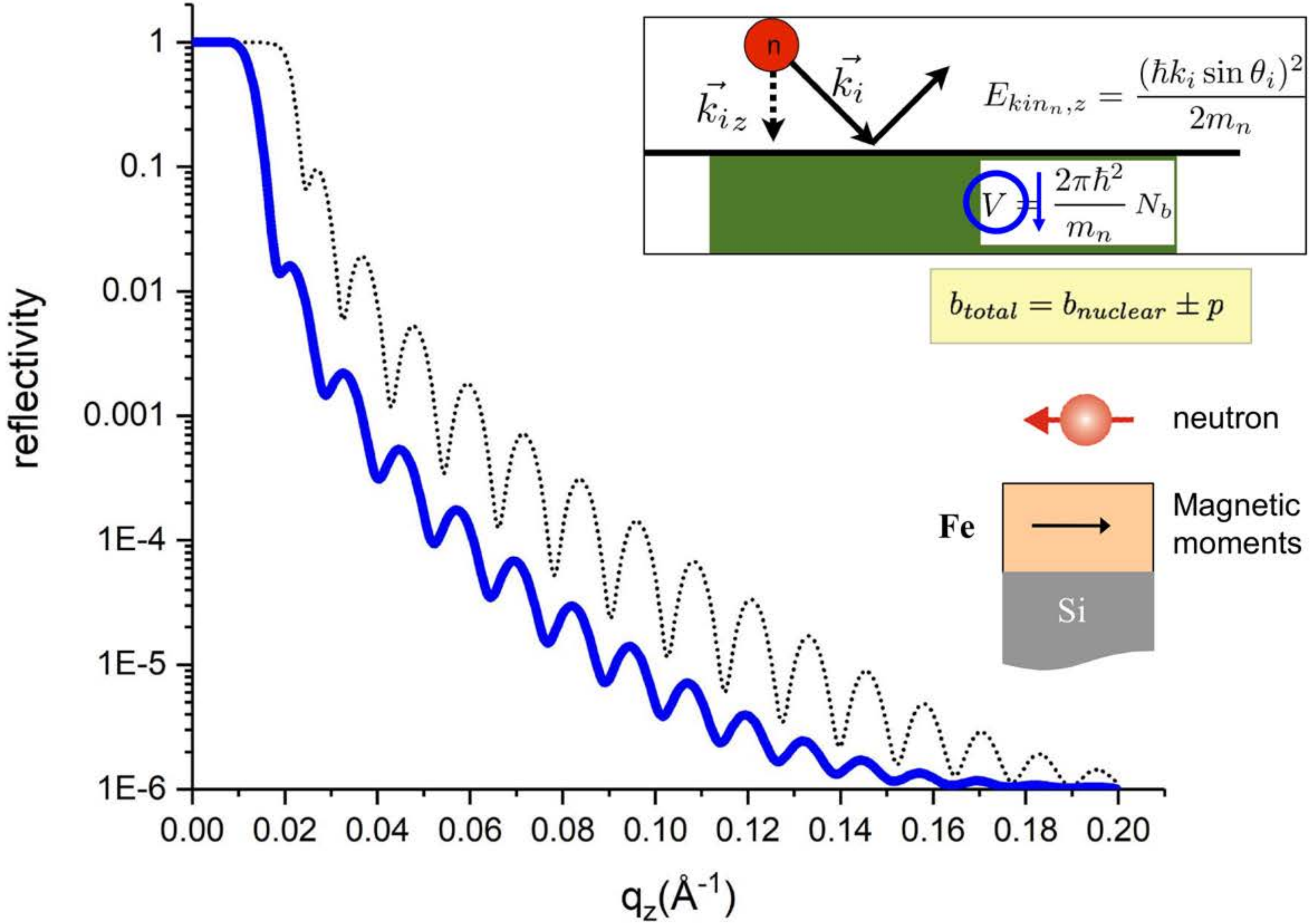
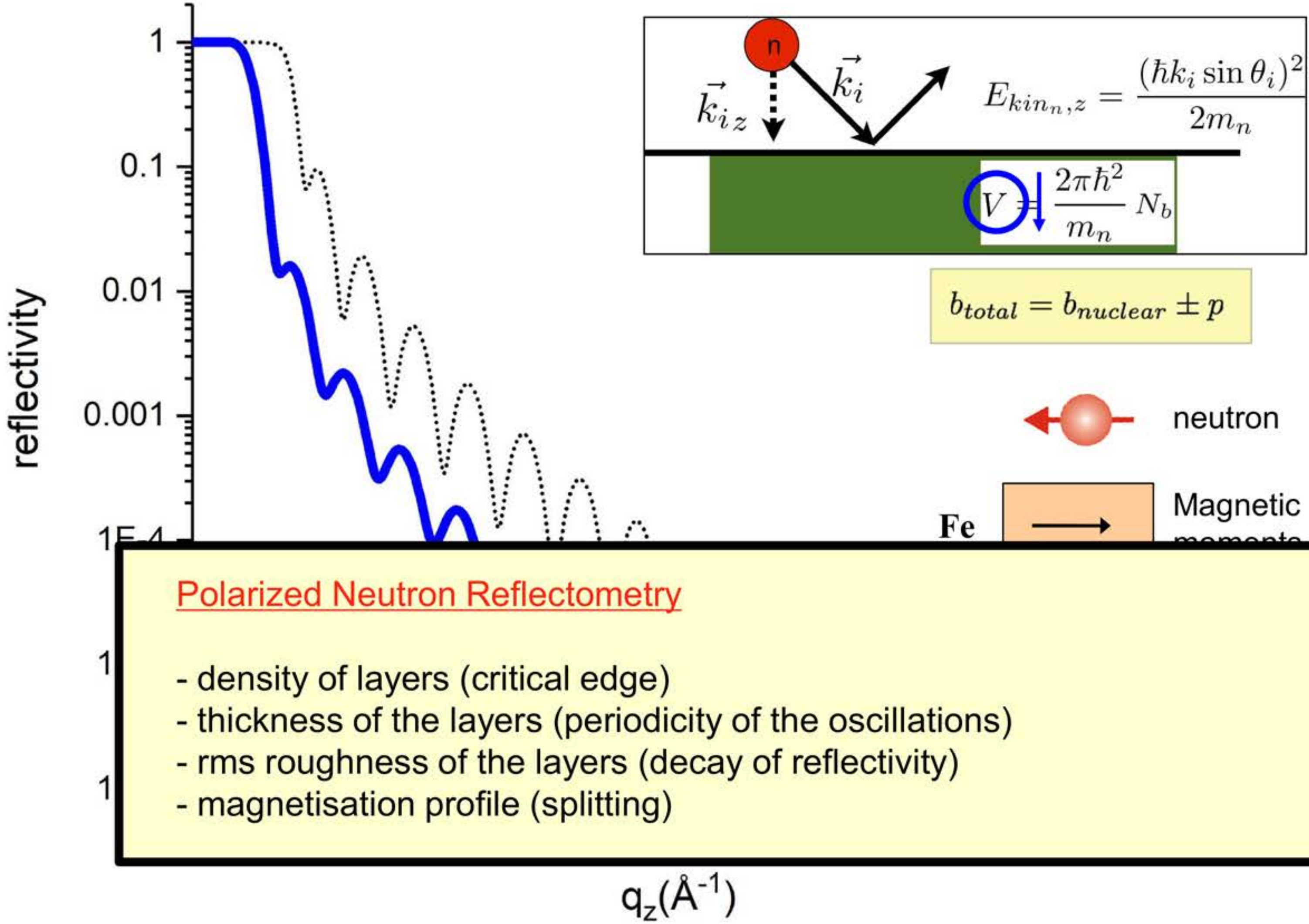
Polarized Neutron Reflectometry



Introduction

Polarized Neutron Reflectometry



Polarized Neutron Reflectometry

- density of layers (critical edge)
- thickness of the layers (periodicity of the oscillations)
- rms roughness of the layers (decay of reflectivity)
- magnetisation profile (splitting)

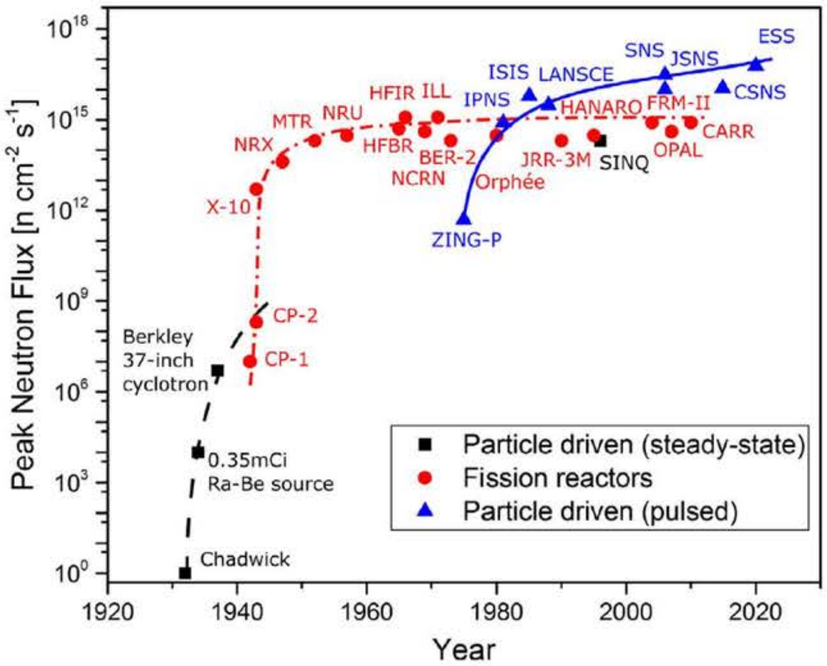
Introduction

Advantages of *in situ*:

- very same sample (not comparing similar samples)
- no capping layer, which could influence the magnetization of the free film
- measuring as function of film thickness flexibility in reacting to results and in adapting research strategy

Why now?

- scientific relevance because of current development in magnetic storage devices
- today new neutron sources being built
- modern neutron optics available



Introduction

Advantages of *in situ*:

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very powerful complementary technique!

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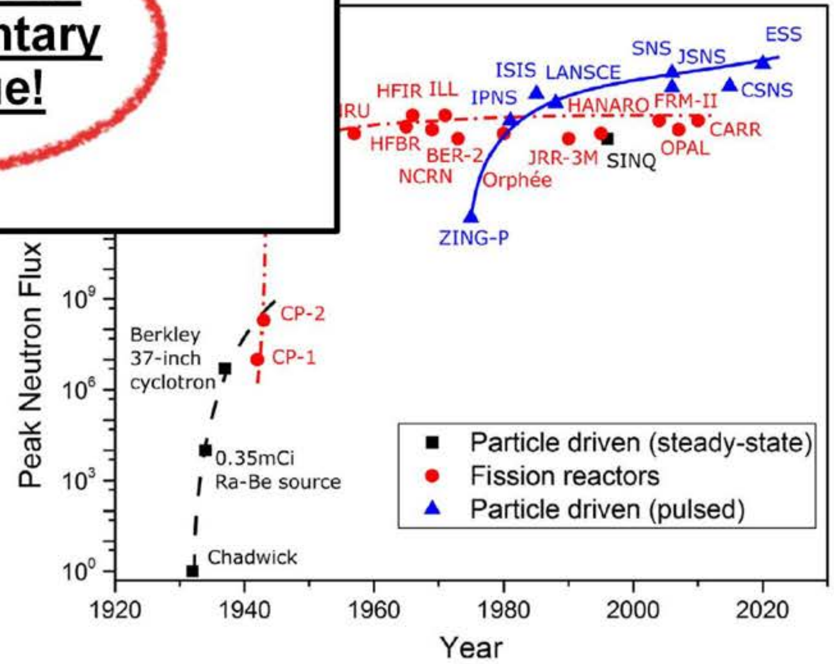

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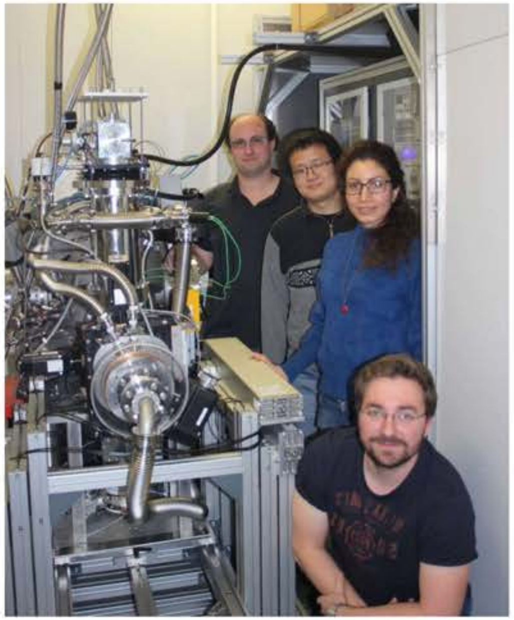


Conclusion and Outlook

In situ Thin Film Deposition Setup

Starting Point

Pre-requisite:

sample must be prepared in the neutron beam under at least HV (or ideally UHV) conditions

TRR 80
„From Electronic Correlations to Functionality“

DFG Deutsche Forschungsgemeinschaft

Initial Construction of in-situ chamber:

Experimentalphysik VI
Institut für Physik
Universität Augsburg

Team:
Andreas Schmehl*
Thomas Mairoser*
Alexander Herrnberger

*Alumni (since 2013)

1st funding period: 01/2010 - 12/2013 (acting PI since Aug. 2013)	→ approx. 450,000 €
2nd funding period: 01/2014 - 12/2017 (PI)	→ approx. 470,000 €
3rd funding period: 01/2018 - 12/2021 (acting PI until End of 2018, than advisor)	→ approx. 440,000 €
2022/2023 TU Munich – Uni Augsburg	→ relocation costs
since 2023 Paul-Scherrer Institut	→ operational costs

In situ Thin Film Deposition Setup

- footprint: less than 1m^2
- total system weight: 700kg
- deposition method: sputtering with three 2“ sources (DC & RF operation)
- Materials: elements, alloys, oxides, nitrides, silicides, etc.

Andreas Schmehl, Thomas Mairoser, Alexander Herrnberger, Cyril Stephanos, Stefan Meir, Benjamin Förg, Birgit Wiedemann, Peter Böni, Jochen Mannhart, and Wolfgang Kreuzpaintner, *Design and realization of a sputter deposition system for the in situ- and in operando-use in polarized neutron reflectometry experiments*, Nucl. Inst. Meth. Phys. Res., A 883, 170-183 (2018).
https://doi.org/10.1016/j.nima.2017.11.086

In situ Thin Film Deposition Setup

Support Infrastructure

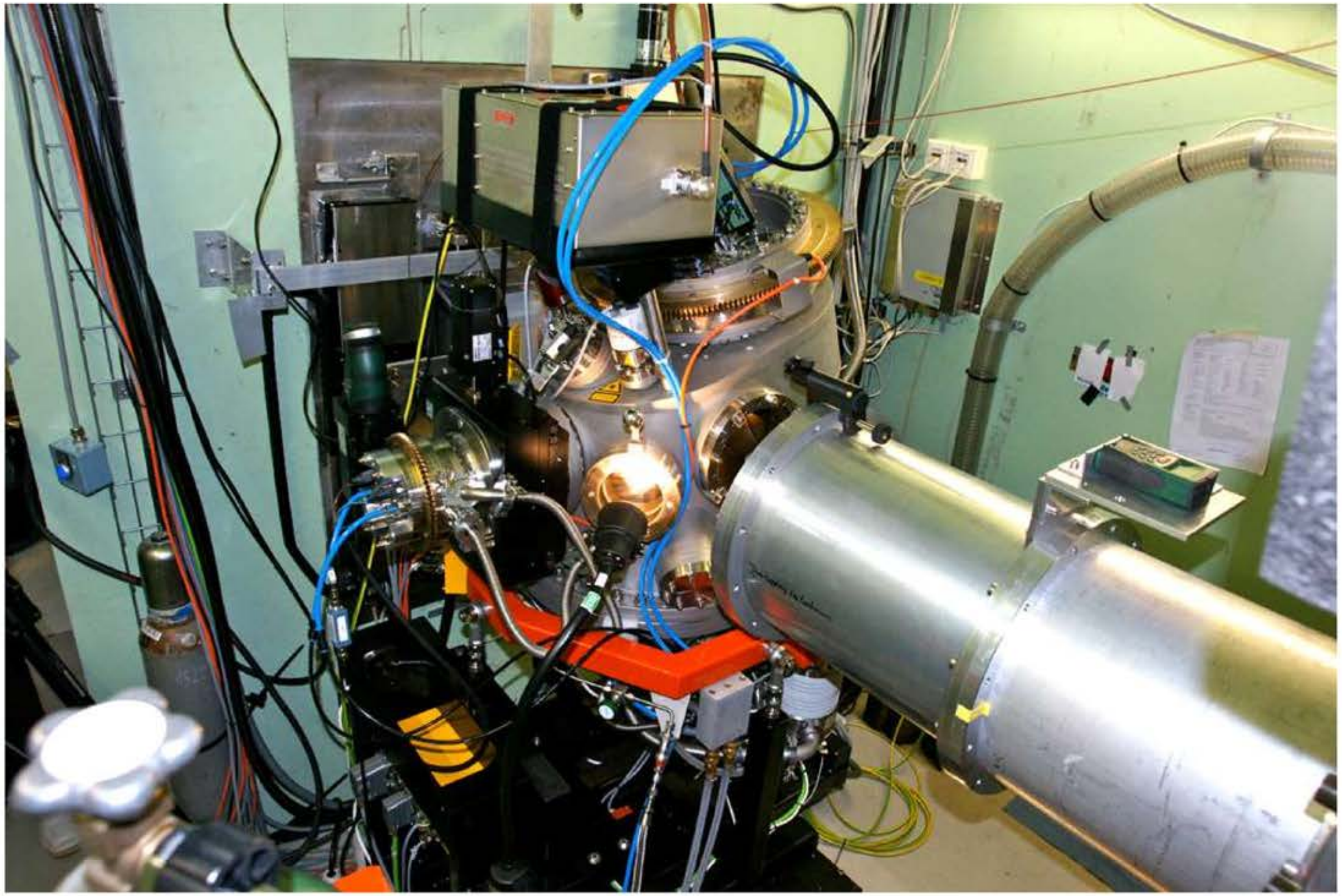
- 2x 2-circle x-ray reflectometer (Siemens D5000, D500-8)
- AFM/MFM
- SEM
- fume hood

- 4-circle diffractometer (Siemens D500)
- UHV chamber for coating tests
- class 100/1000 clean-room environment

S. Mayr, C. Randau, and W. Kreuzpaintner, *Automatic Attenuator Upgrade for a Siemens D500 Diffractometer via a Generic Software Library to Overcome Hardware Limitations,* Nucl. Inst. Meth. Phys. Res., A 855, 61-64 (2017). http://dx.doi.org/10.1016/j.nima.2017.02.088

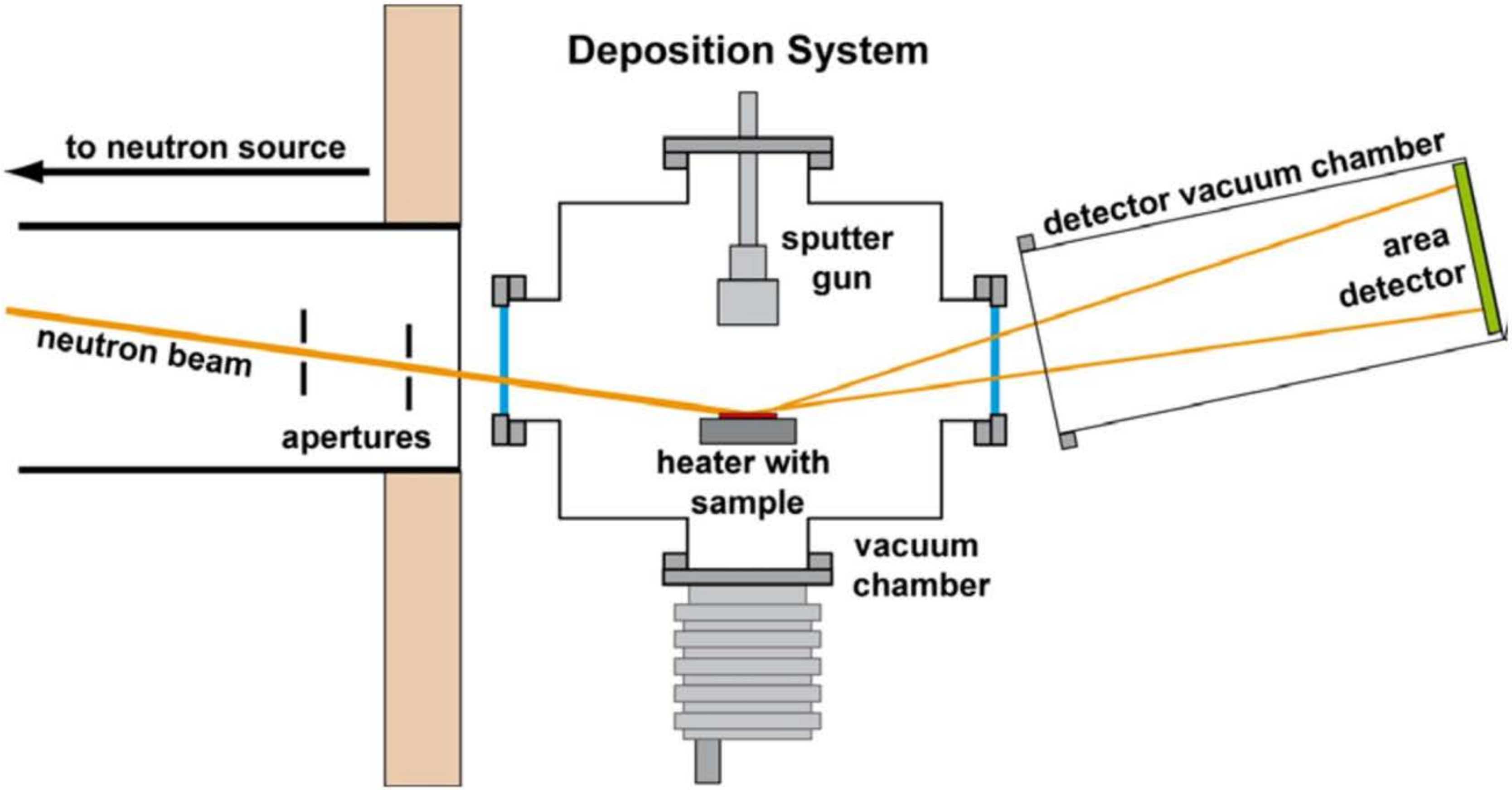
In situ Thin Film Deposition Setup

In situ Thin Film Deposition Setup installed at REFSANS



In situ Thin Film Deposition Setup

Scattering Geometry



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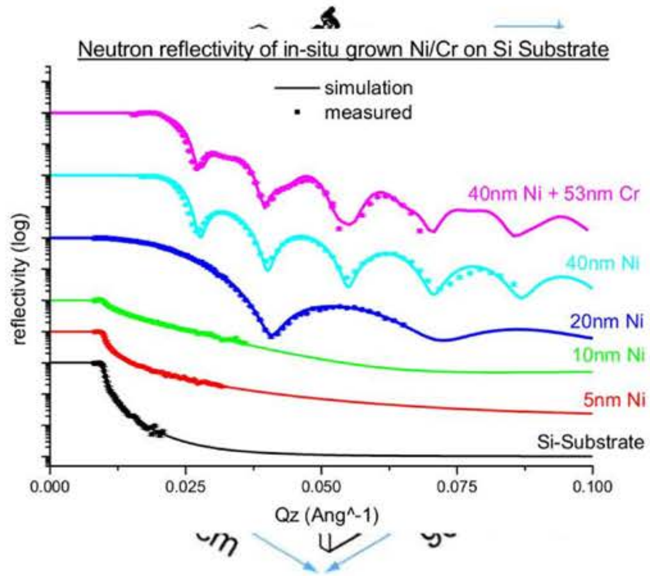
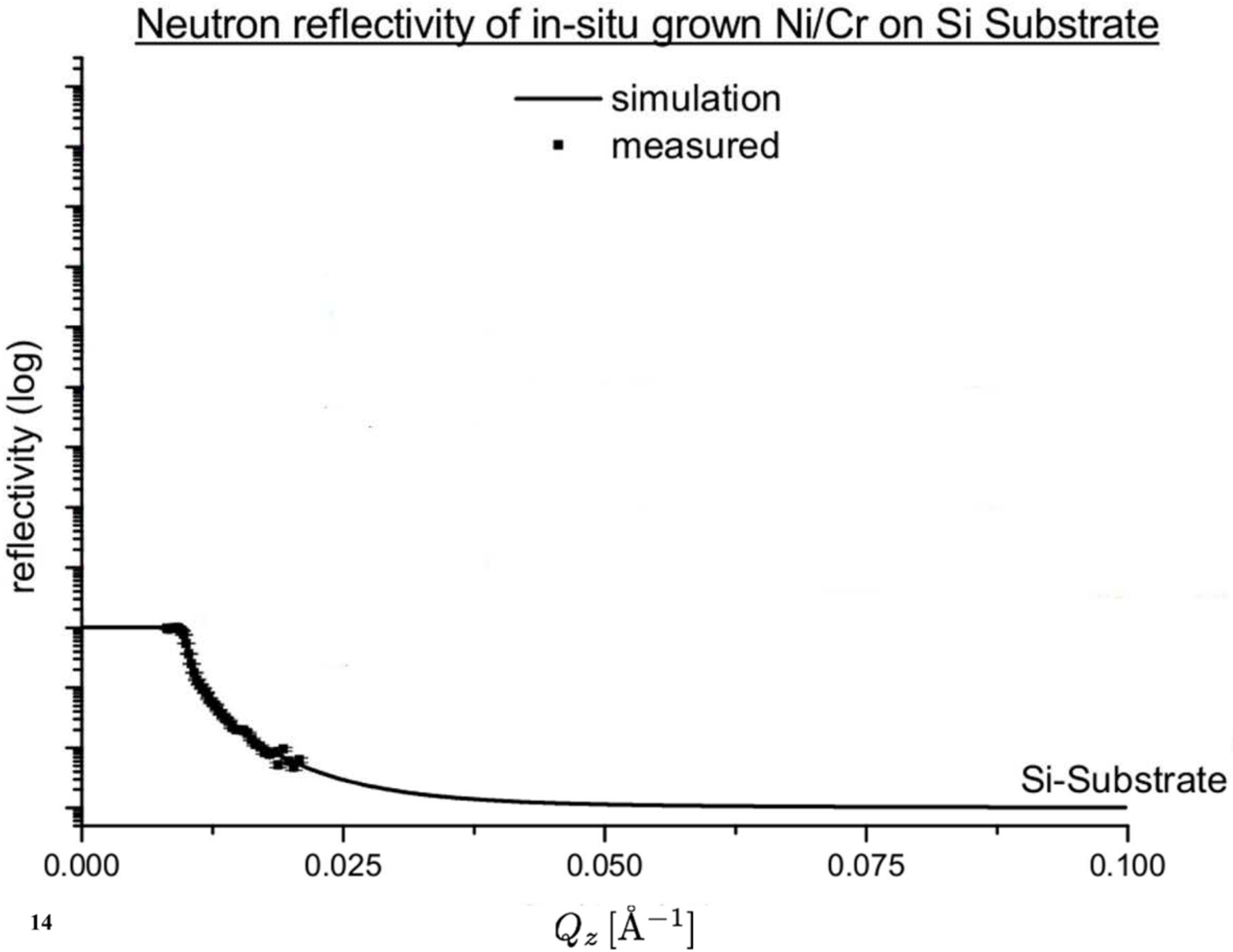
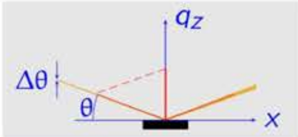
- Latest Developments



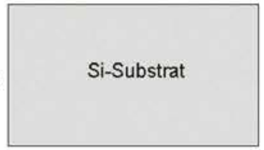
Conclusion and Outlook

Early *in situ* Experiments

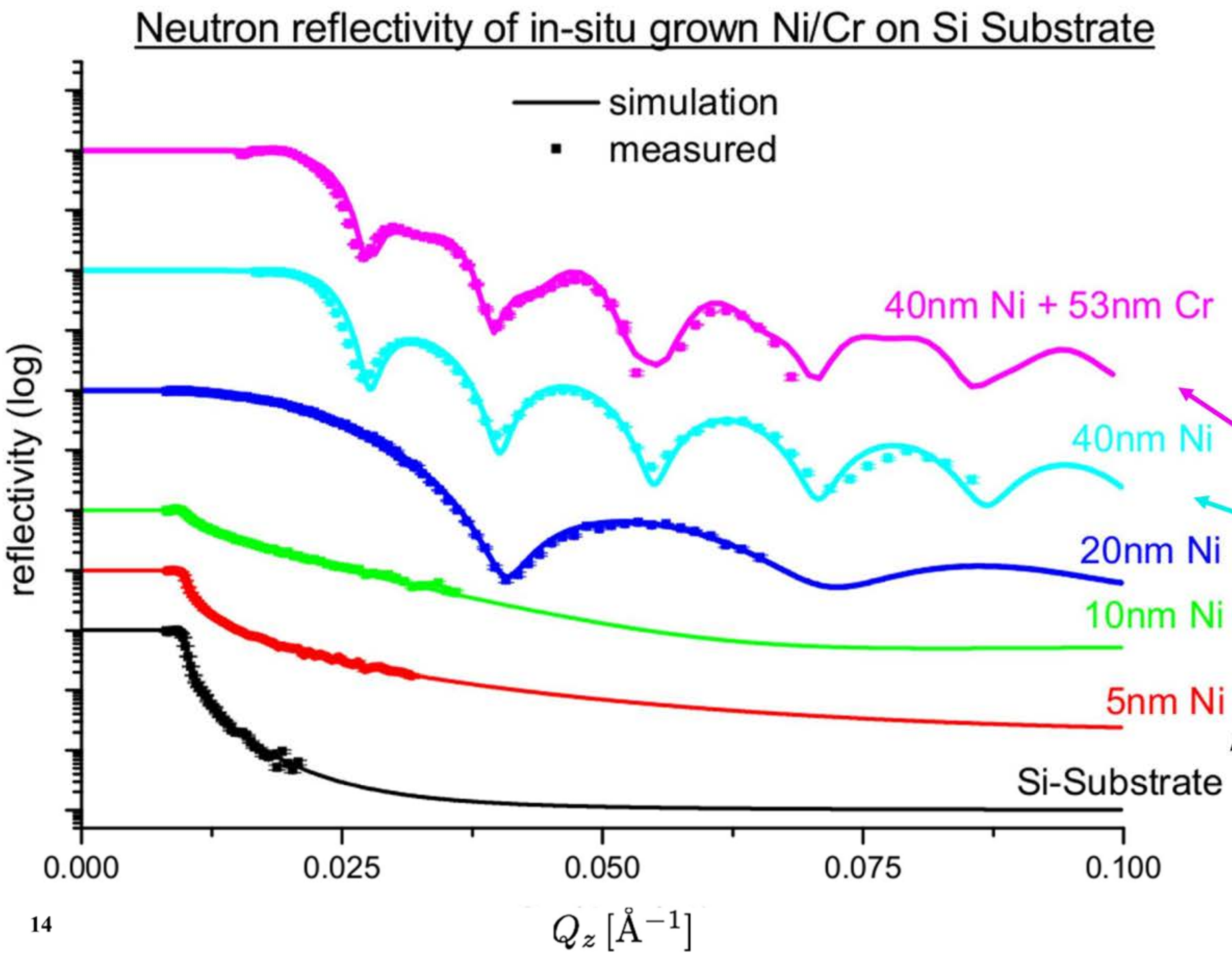


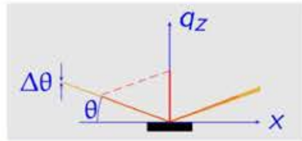


$$q_z = \frac{4\pi}{\lambda} \sin\theta$$

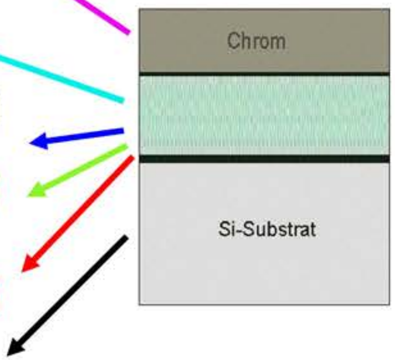


Early *in situ* Experiments

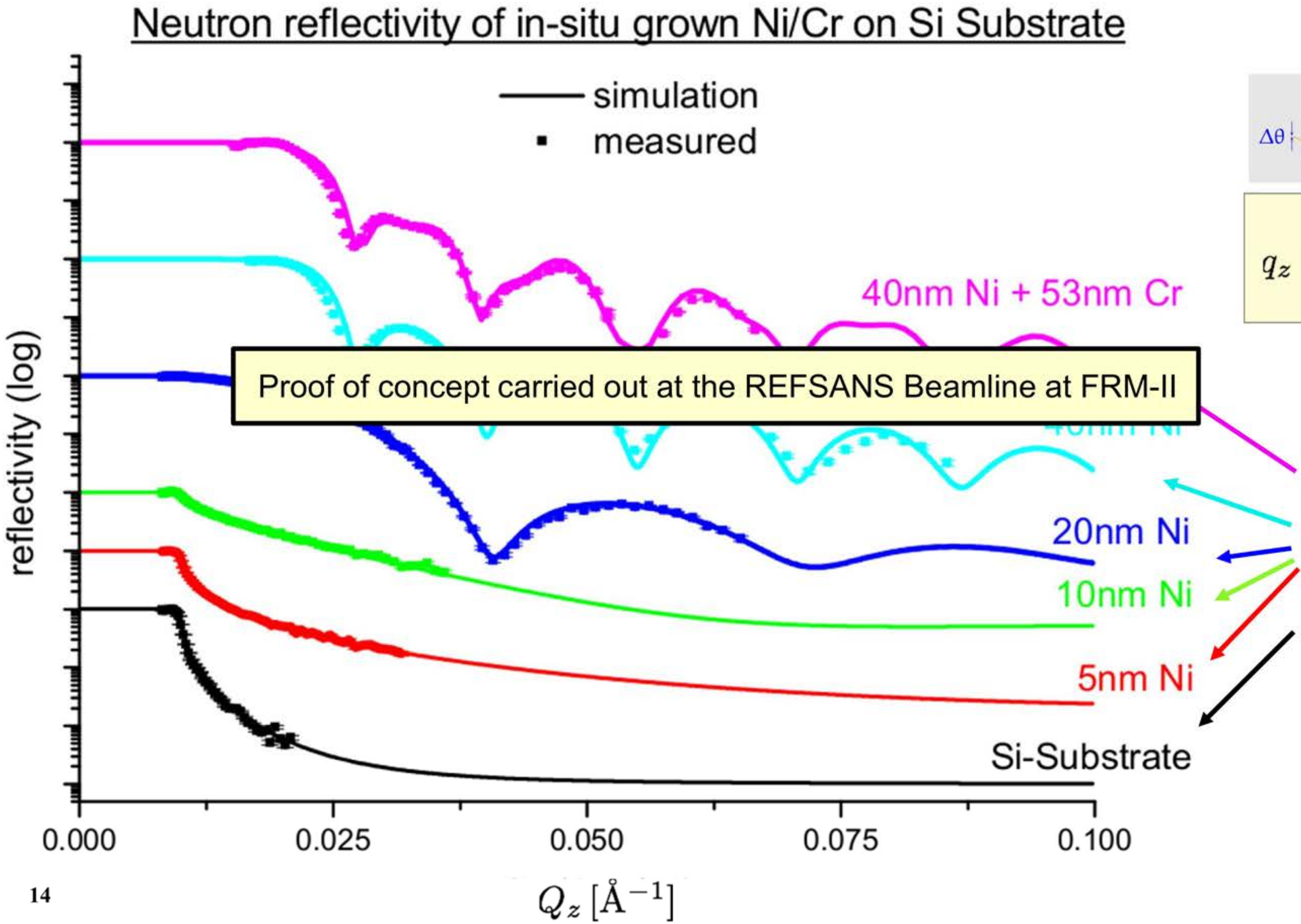




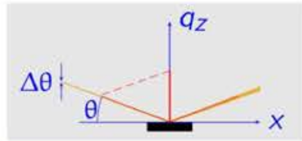
$$q_z = \frac{4\pi}{\lambda} \sin\theta$$



Early *in situ* Experiments

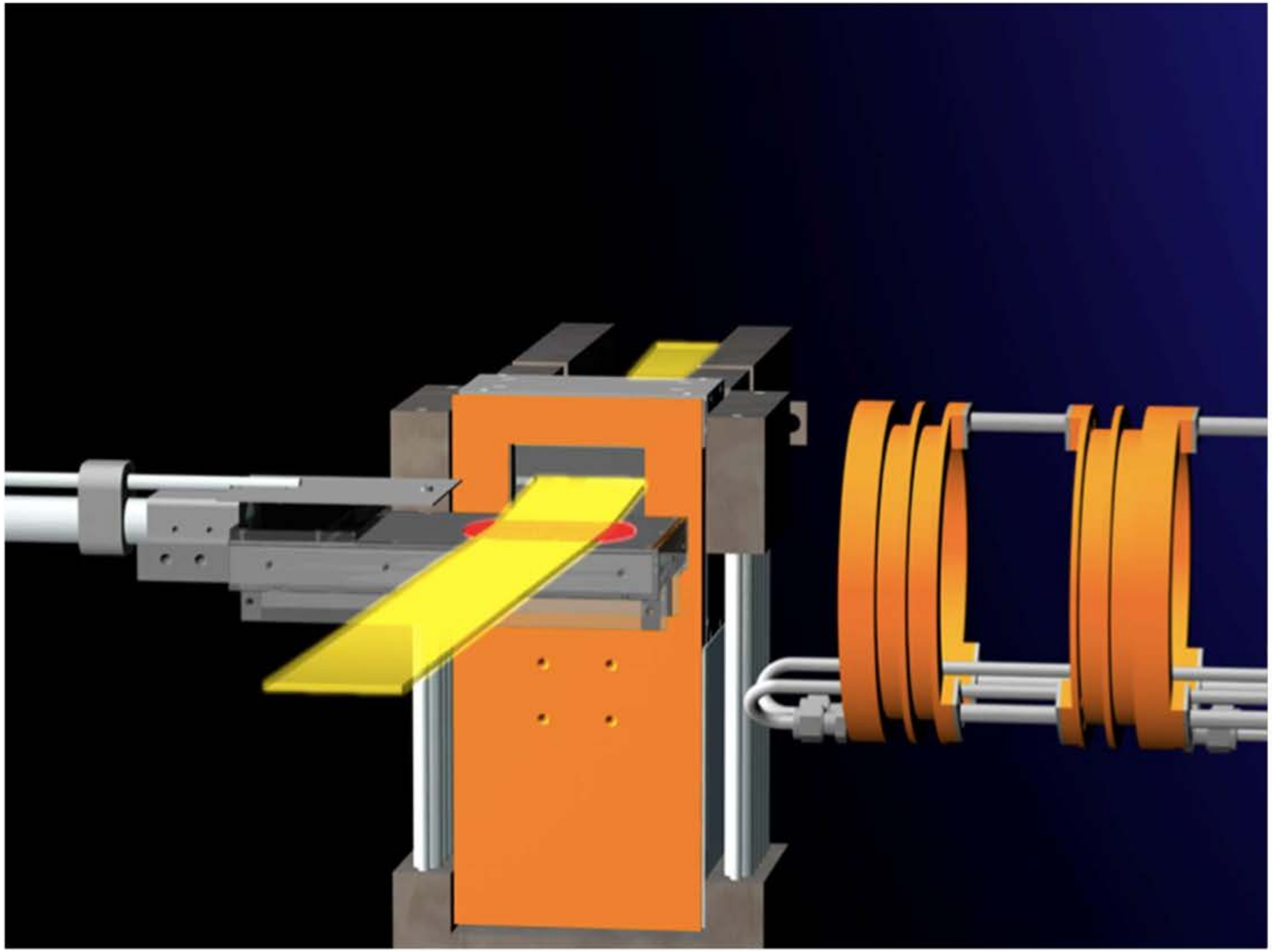


Proof of concept carried out at the REFSANS Beamline at FRM-II

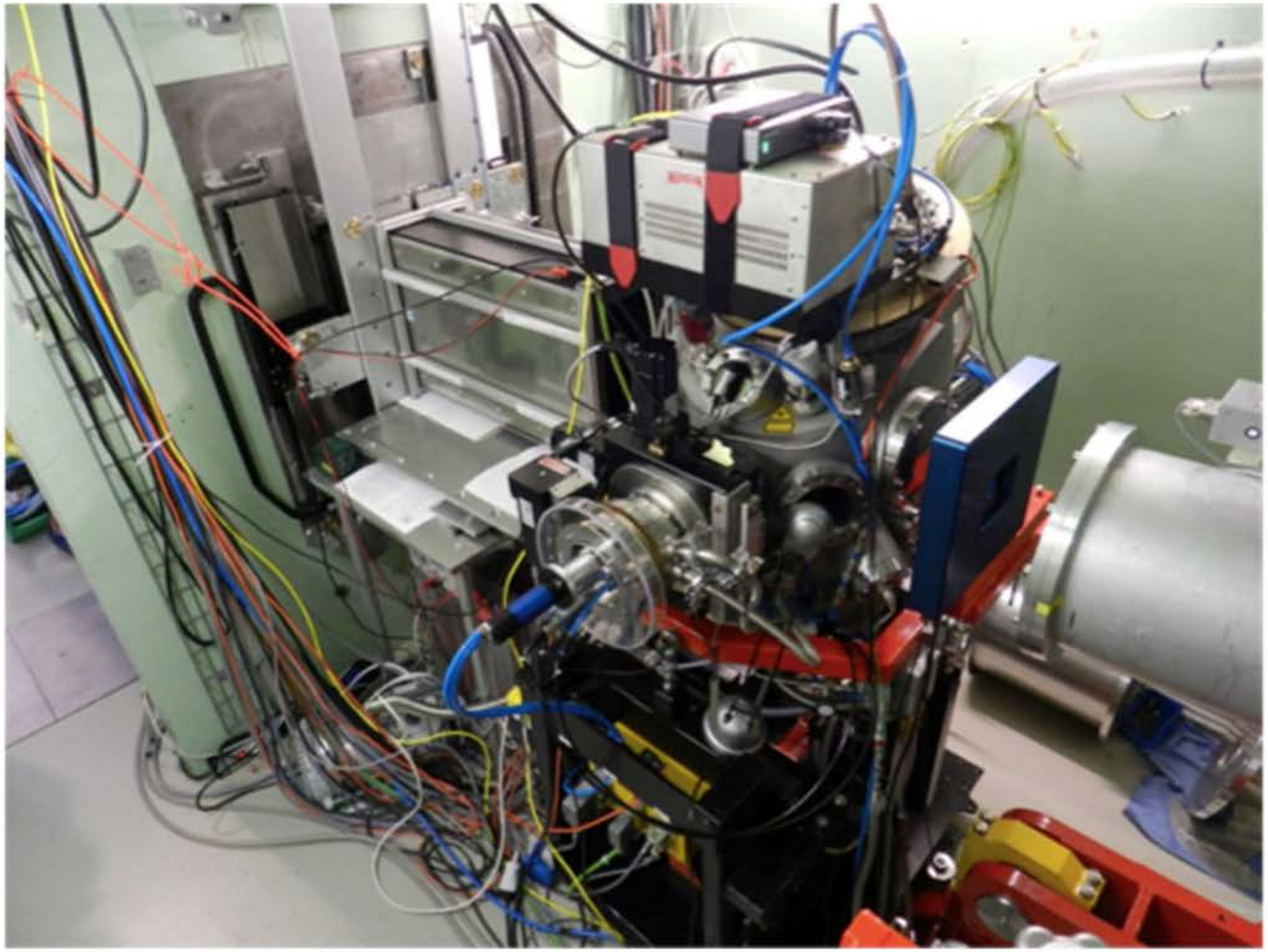


$$q_z = \frac{4\pi}{\lambda} \sin\theta$$

Early *in situ* Experiments

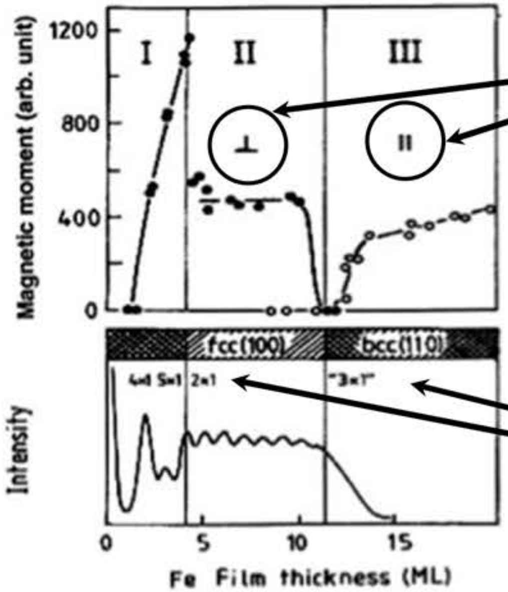


Early *in situ* Experiments



Kreuzpaintner et al., *Application of a portable ^{3}He-based polarization insert at a time-of-flight neutron reflectometer*, *Nuclear Instruments and Methods in Physics Research A 848 (2017) 144–152,* DOI: 10.1016/j.nima.2016.12.017

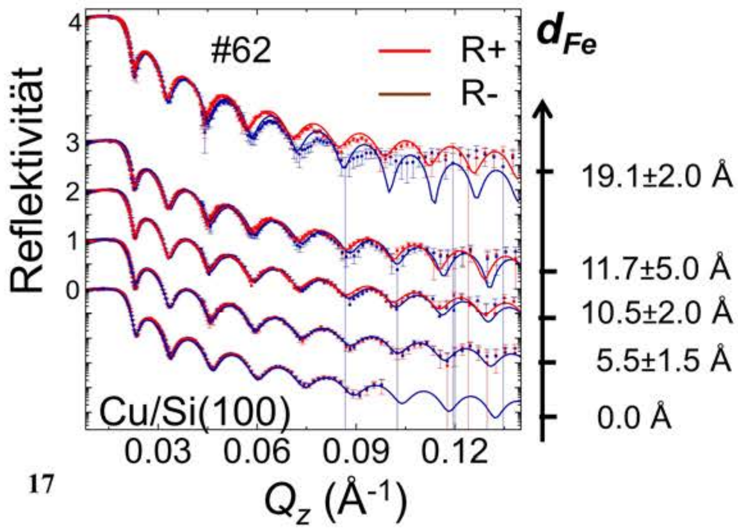
Early *in situ* Experiments

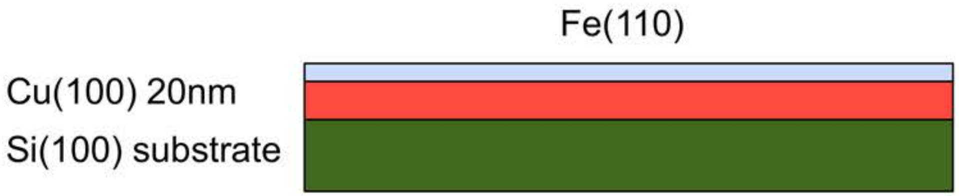


Intrinsic direction of the magnetization is function of thickness

Crystal Structure is function of thickness

M. Wuttig et al., Ultrathin Metal Films: Magnetic and Structural Properties. Springer 2004.





… but: 12 h data accumulation per layer and neutron polarization → total 24 hours!

Early *in situ* Experiments

... too long data collection times, which leads to the big question of ...

„how clean does the surface of the sample really stay during the measurement due to absorption and desorption of „dirt“-adatoms in the vacuum?“

... which leads to the next big question of ...

„how can we make vacuum quality and measurement times compatible?“

- base pressure of vacuum improved to 5E-9 mbar
- use of 99.99999% (7n) pure Ar working gas

→ approx. 30min

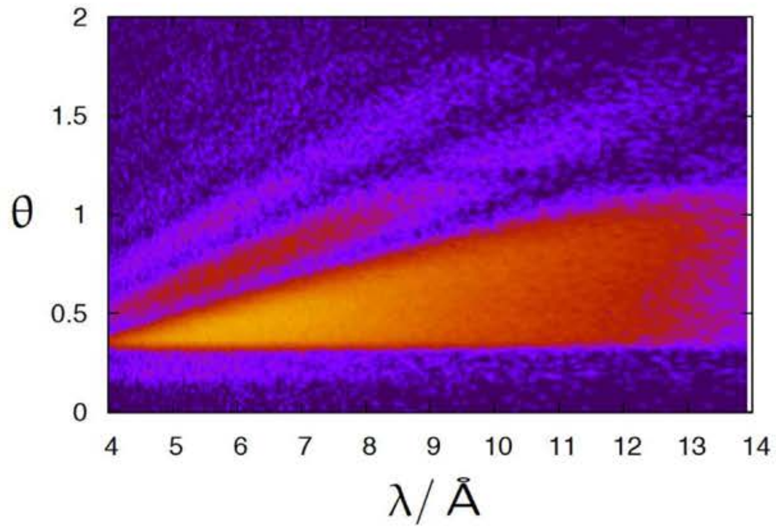
30min for performing polarized neutron reflectivity measurements in a minimum range of:

$$0\,\mathring{A}^{-1} \leq Q_z \leq 0.1\,\mathring{A}^{-1}$$

Outline

Introduction

- *In situ* Thin Film Deposition Setup
- Early *in situ* Experiments
- Speeding up the Measurements
- Current Experiments / Possibilities
- Latest Developments

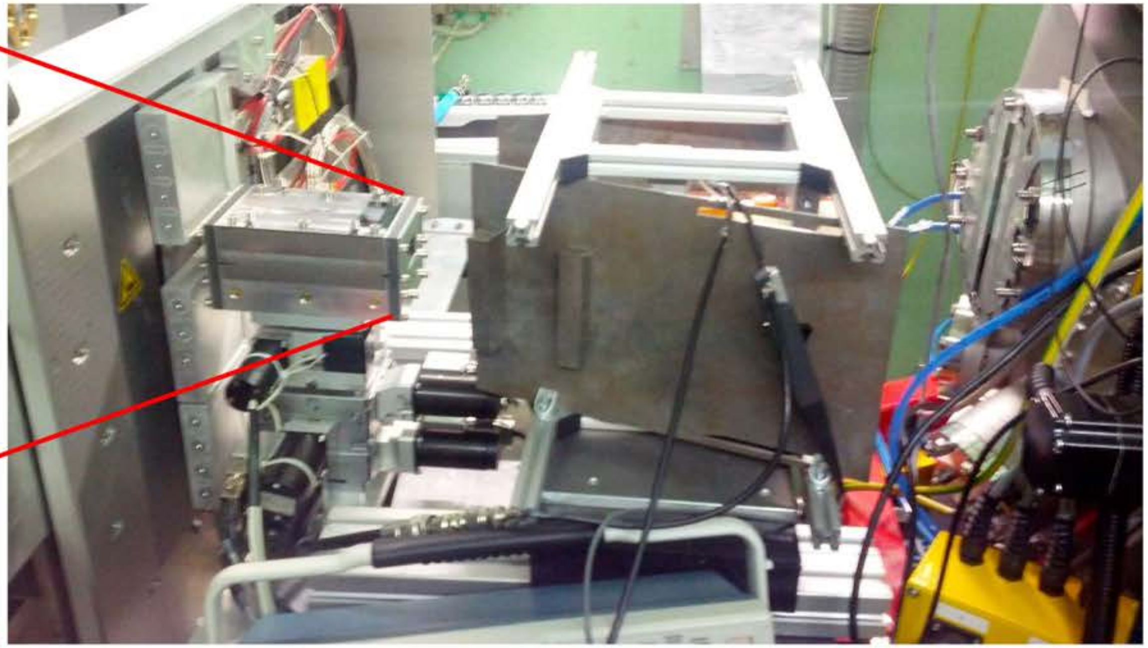


Conclusion and Outlook

Speeding up the measurements

Idea: polarizing supermirror (m=4) and flipper as part of sample environment

Polarizer Design and Construction: W. Kreuzpaintner



- Easier data treatment:
  - No time dependence of polarisation and transmittance
  - Less loss in flux
  - Better signal to noise
  - Real in-situ because sample does not need to be moved

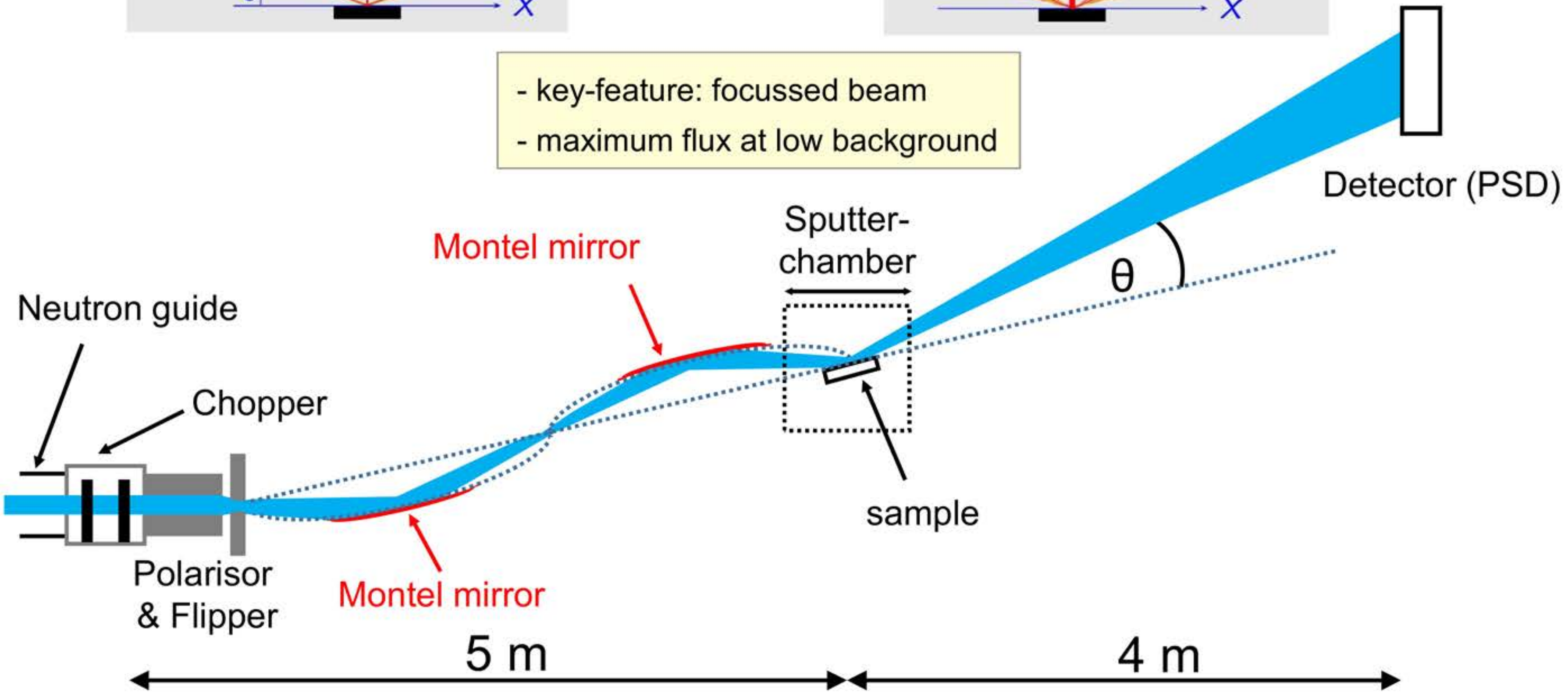
- Investigated system Fe/Cu/Si(100) non epitaxially grown
- Observation: reduction in measurement time from 24h to 16h

→ Need more flux to get to 30min!

Speeding up the measurements

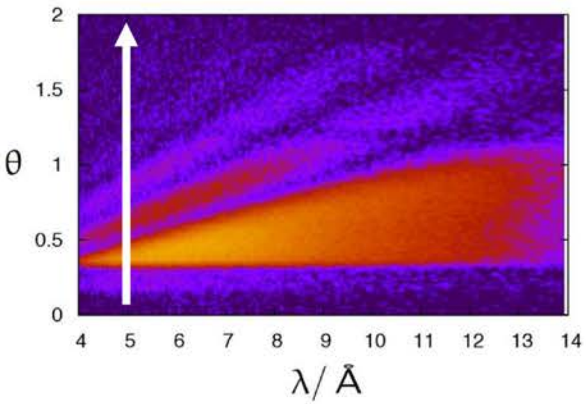
SELENE -- Elliptic (Montel) mirrors -- prototype on AMOR @ SINQ

- key-feature: focussed beam
- maximum flux at low background

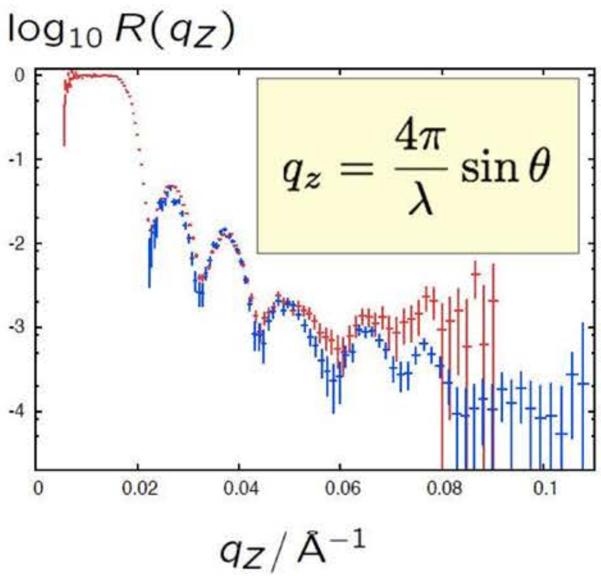


J. Stahn, T. Panzner, U. Filges, C. Marcelot, and P. Böni, Nucl. Instrum. Methods A **634**, S12-S16 (2011)

Speeding up the measurements

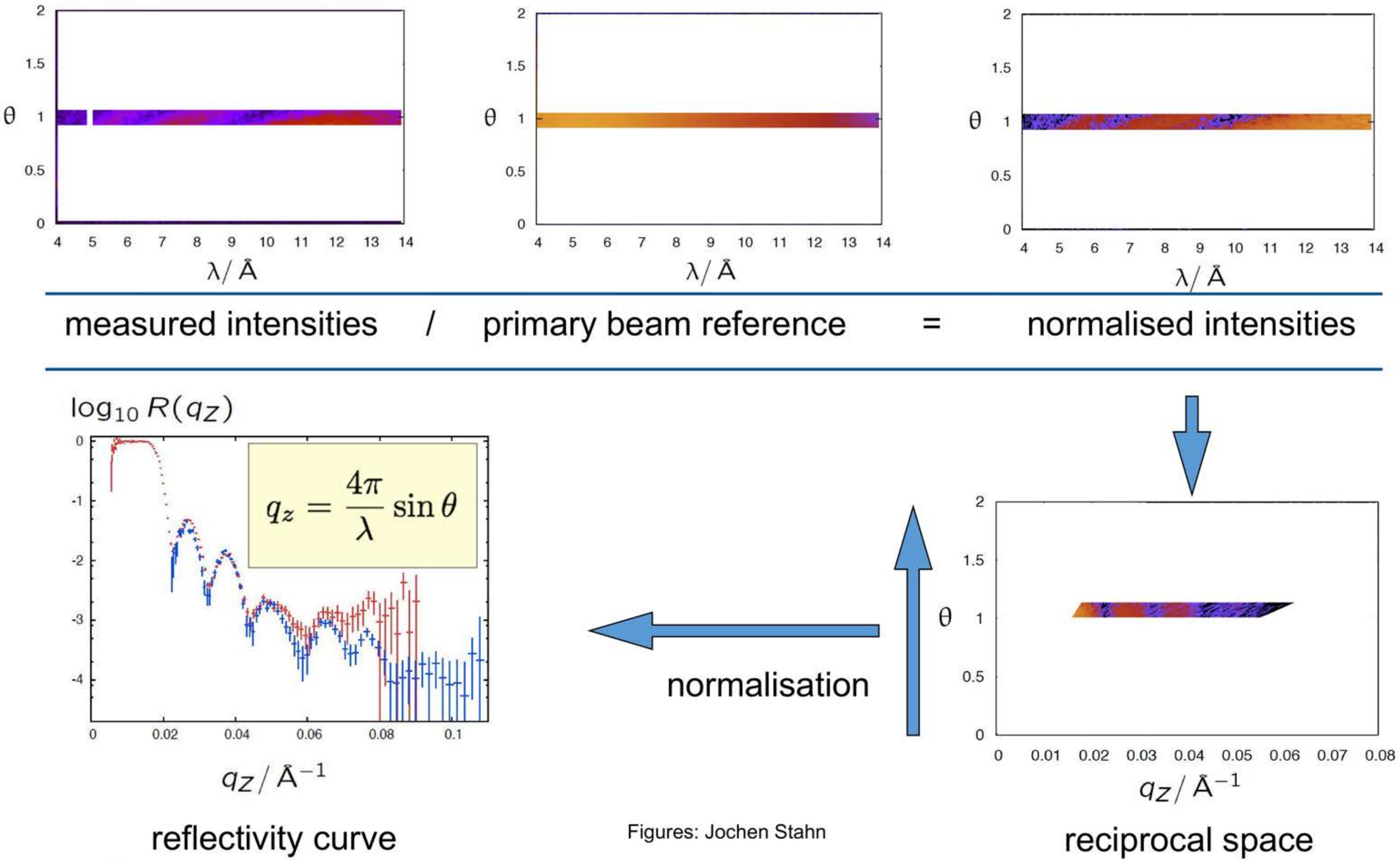


measured intensities



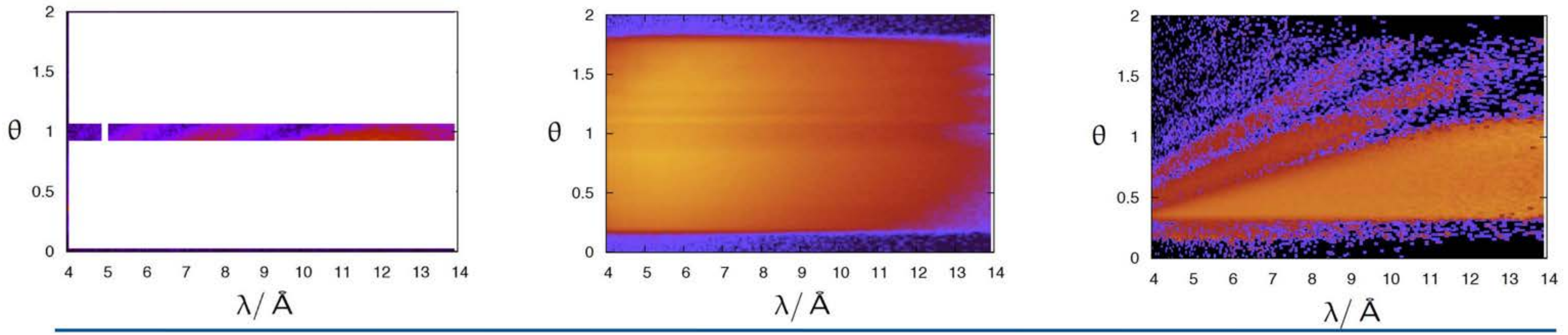
reflectivity curve

Speeding up the measurements

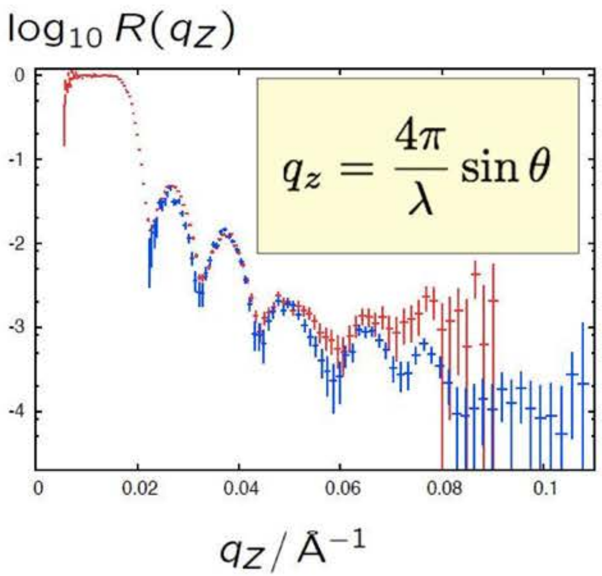


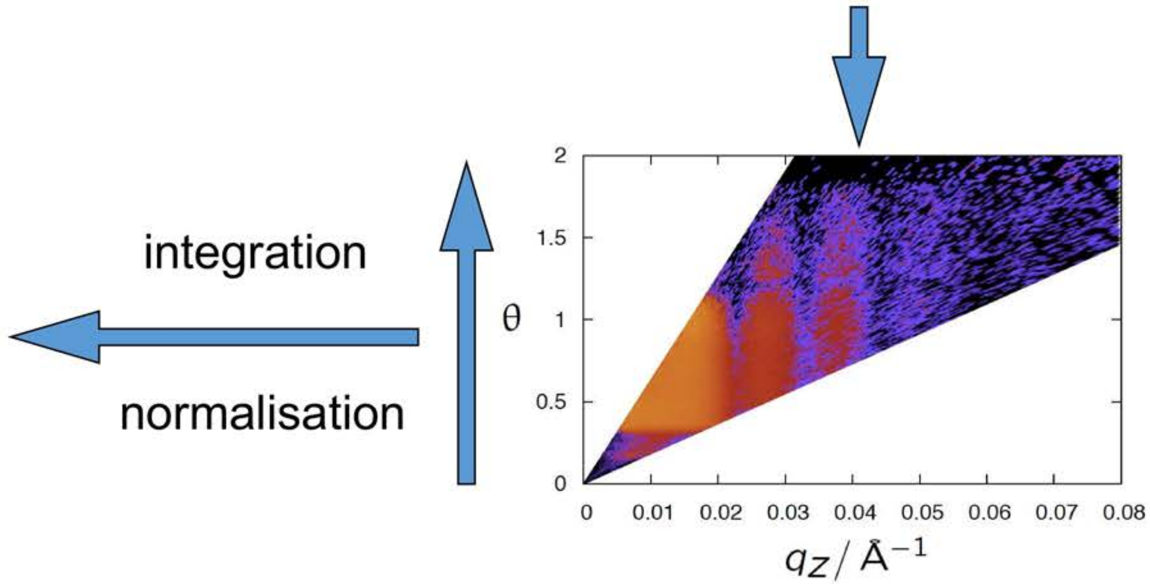
Figures: Jochen Stahn

Speeding up the measurements



measured intensities / primary beam reference = normalised intensities



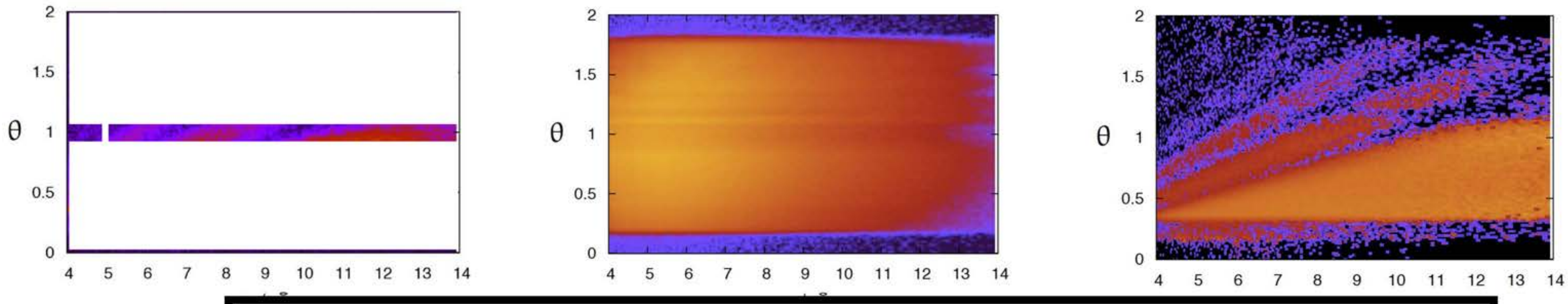


reflectivity curve

Figures: Jochen Stahn

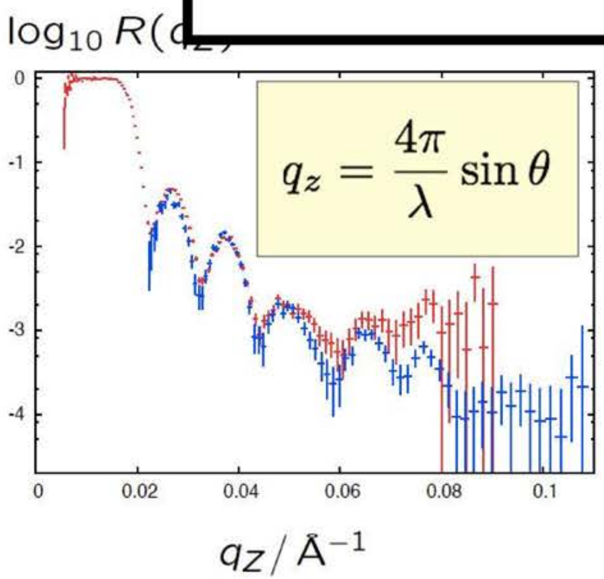
reciprocal space

Speeding up the measurements



measure … es

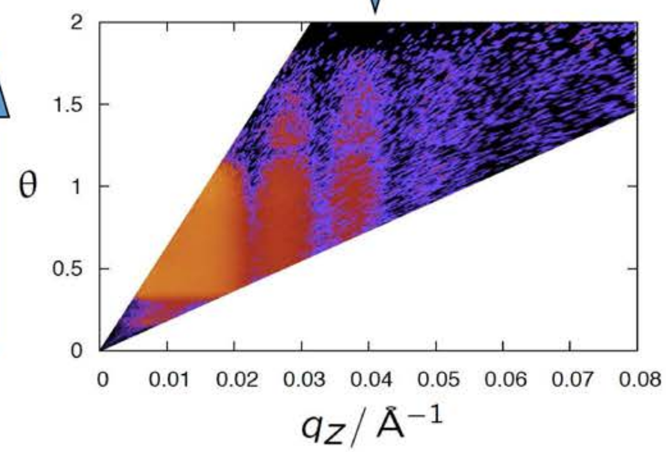
<u>15 min per spin-direction !</u>



reflectivity curve

integration

normalisation



reciprocal space

Figures: Jochen Stahn

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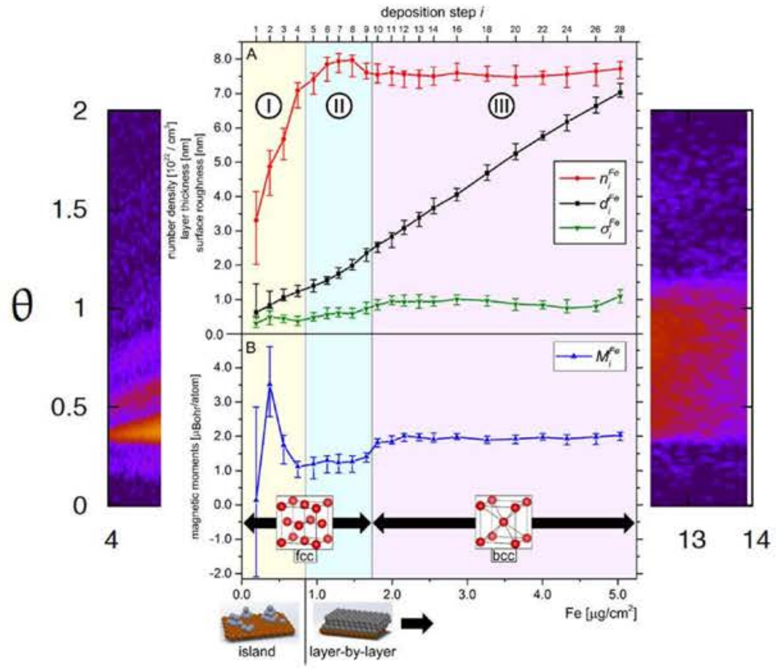
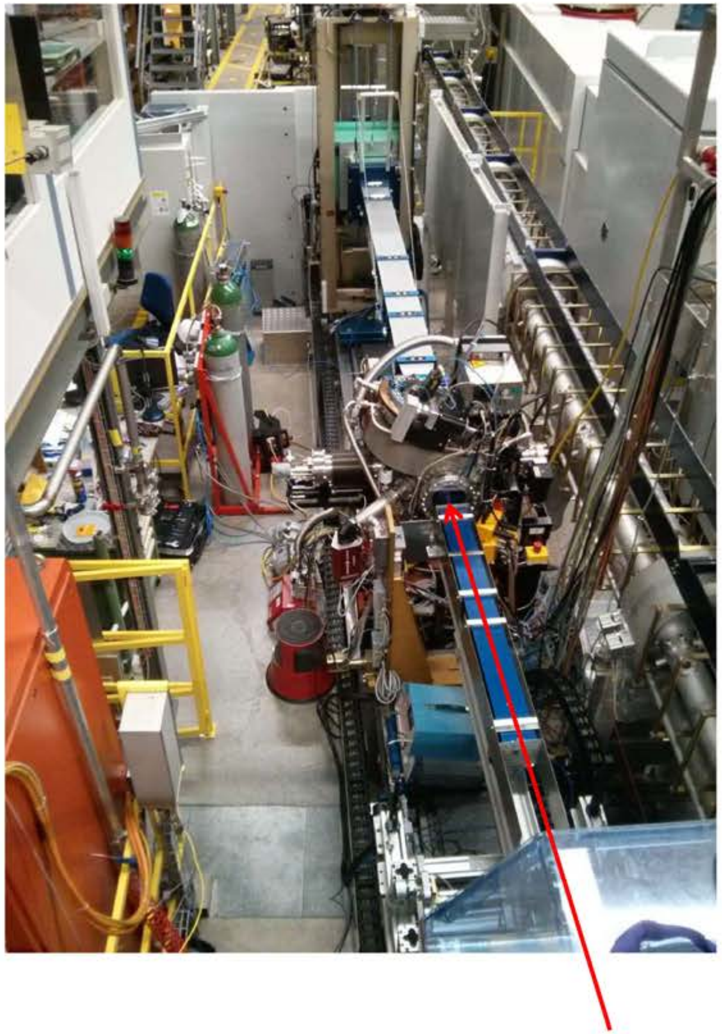


Conclusion and Outlook

Current Experiments / possibilities

Selene:

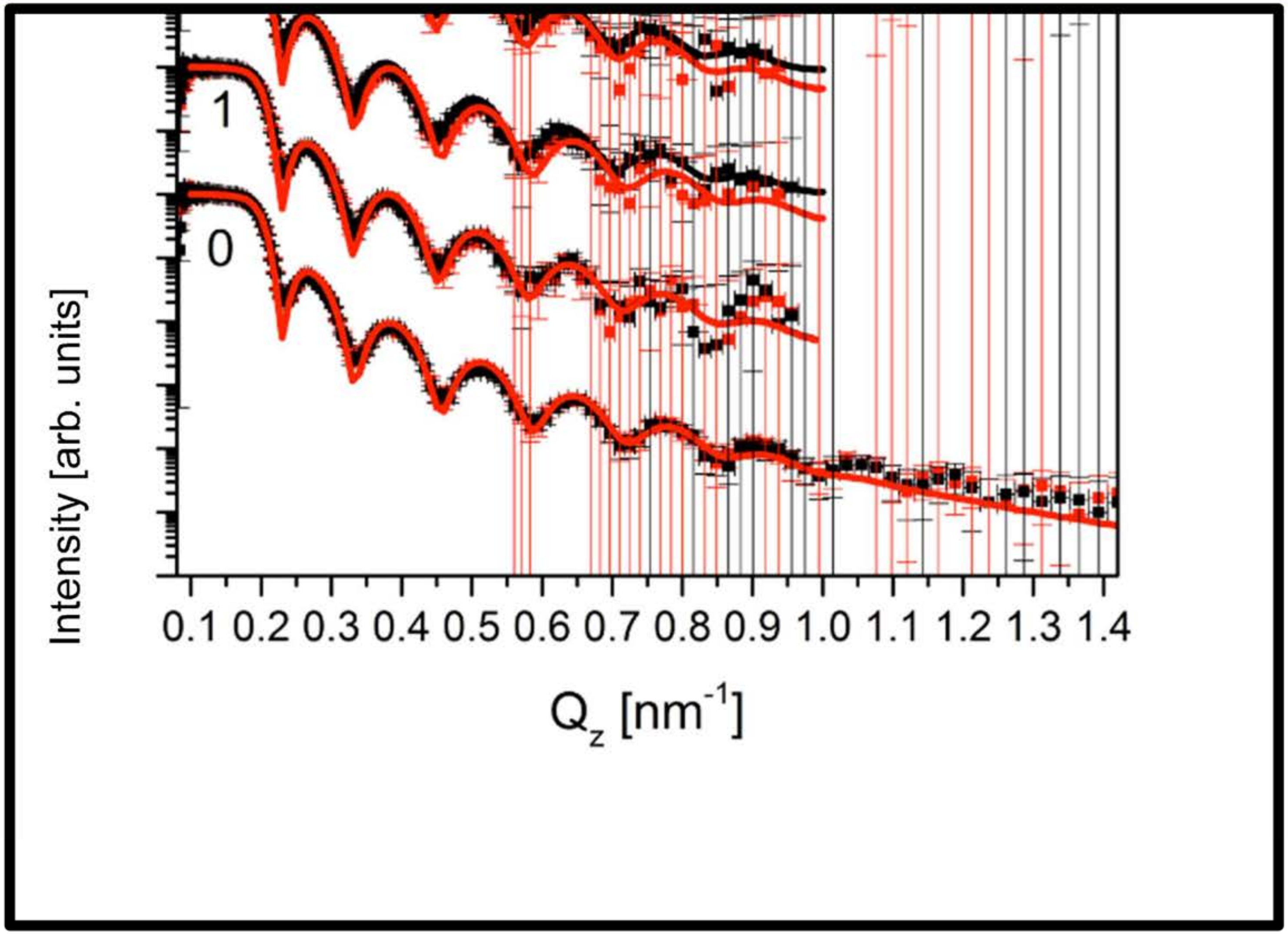


Neutrons

AMOR @ PSI

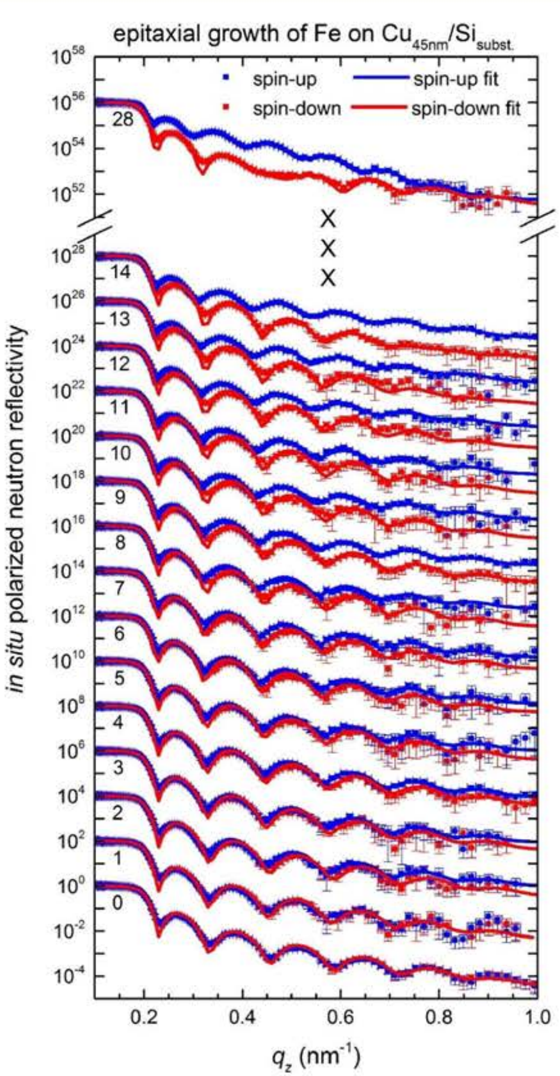
Current Experiments / possibilities

epitaxial growth of Fe on $Cu_{45nm}/Si_{subst.}$

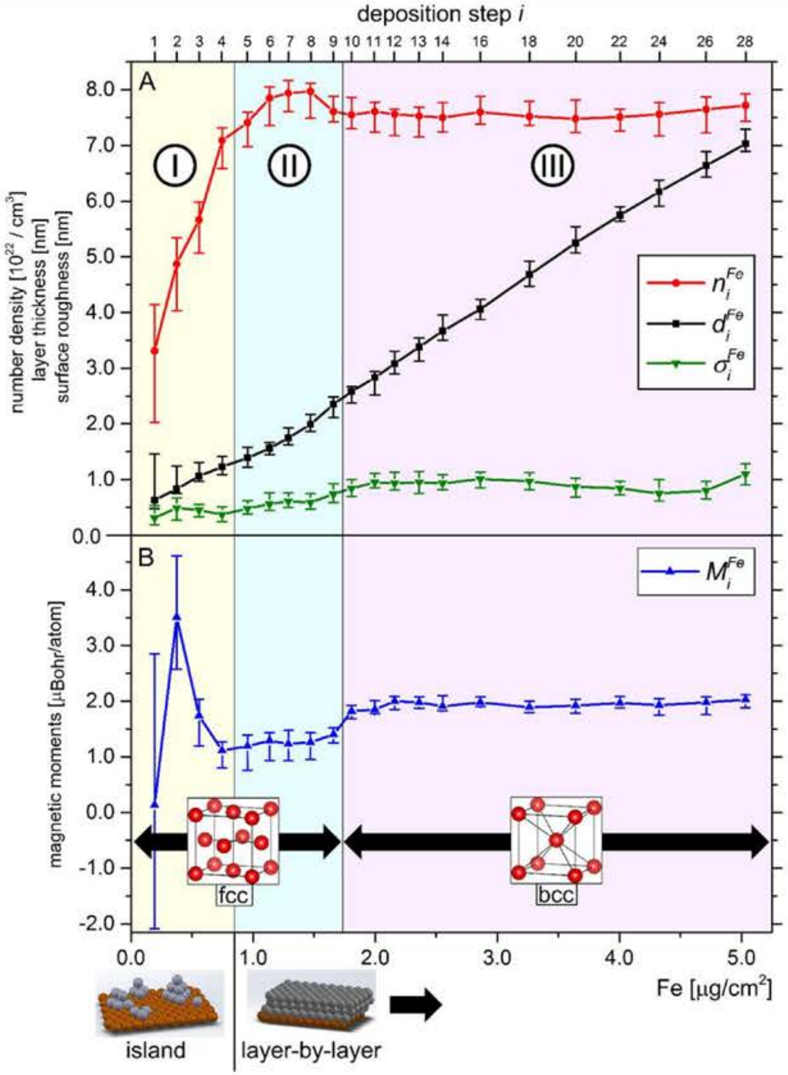


RT, 70mT

Current Experiments / possibilities



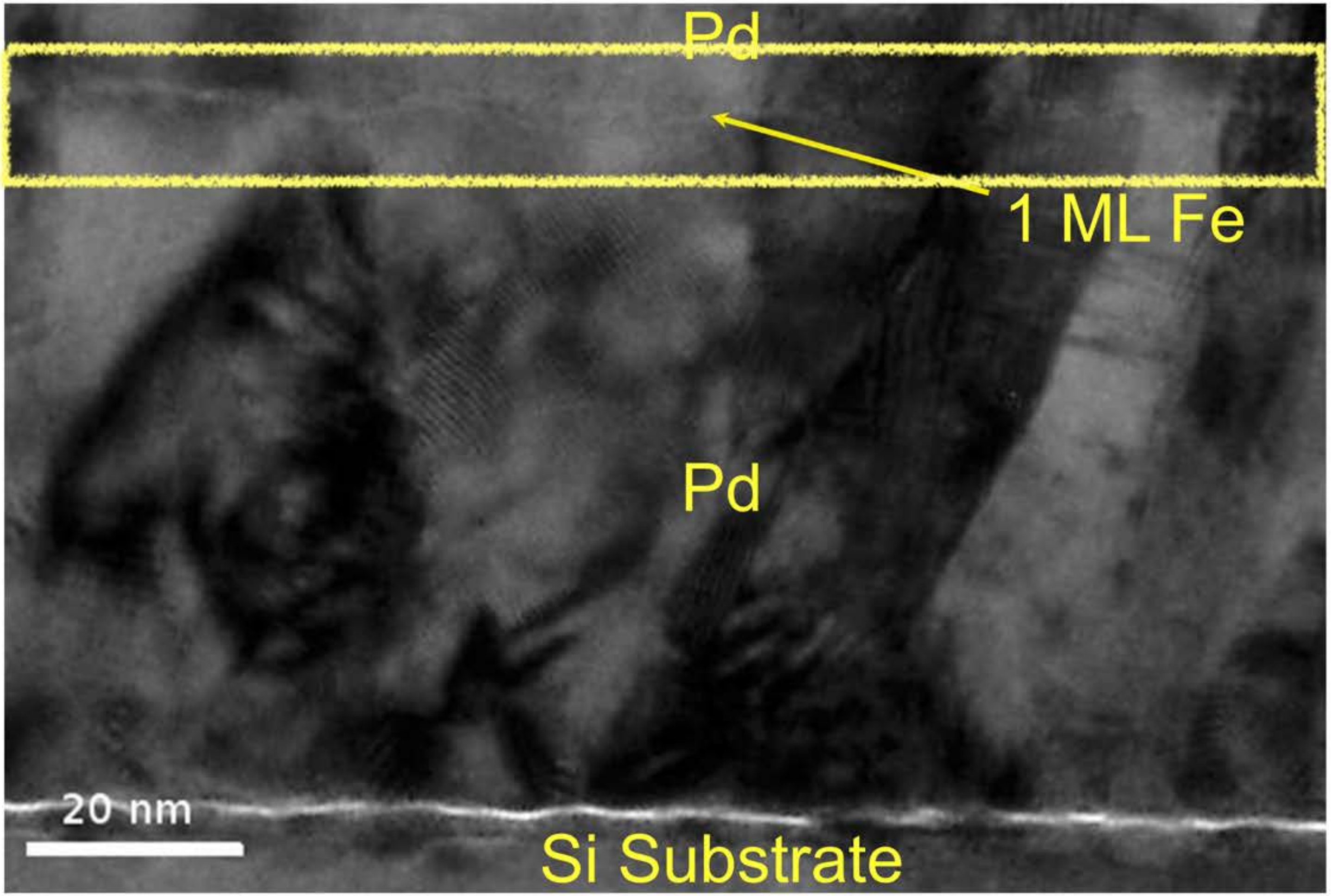
parameters



Wolfgang Kreuzpaintner, Birgit Wiedemann, Jochen Stahn, Jean-François Moulin, Sina Mayr, Thomas Mairoser, Andreas Schmehl, Alexander Herrnberger, Panagiotis Korelis, Martin Haese, Jingfan Ye, Matthias Pomm, Peter Böni, and Jochen Mannhart, *In situ Polarized Neutron Reflectometry: Epitaxial Thin-Film Growth of Fe on Cu(001) by dc Magnetron Sputtering*, PHYSICAL REVIEW APPLIED 7, 054004 (2017)
https://doi.org/10.1103/PhysRevApplied.7.054004

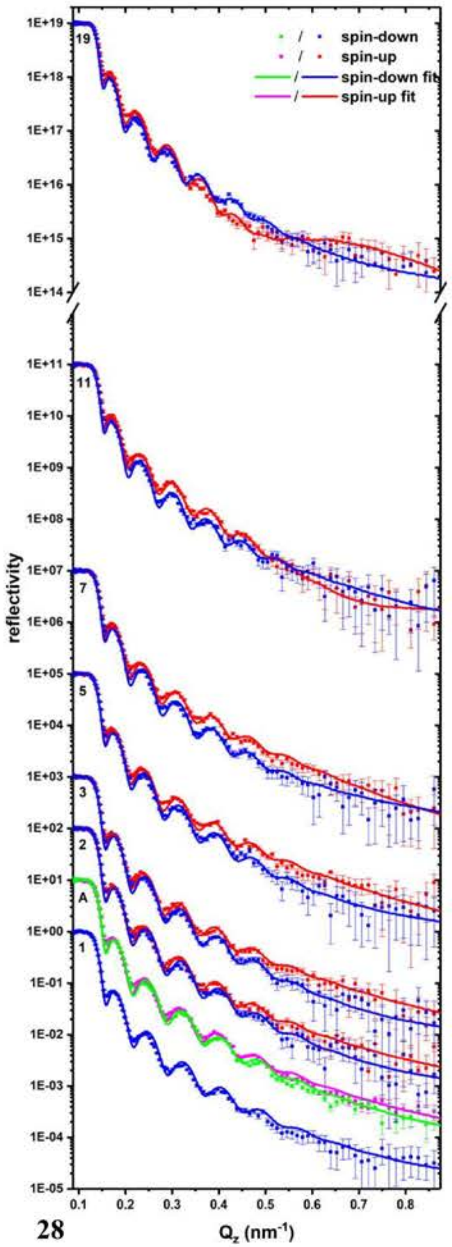
Current Experiments / possibilities

Fe-layer induced ferromagnetism in Pd



J. Guo, University of California, Irvine

Current Experiments / possibilities

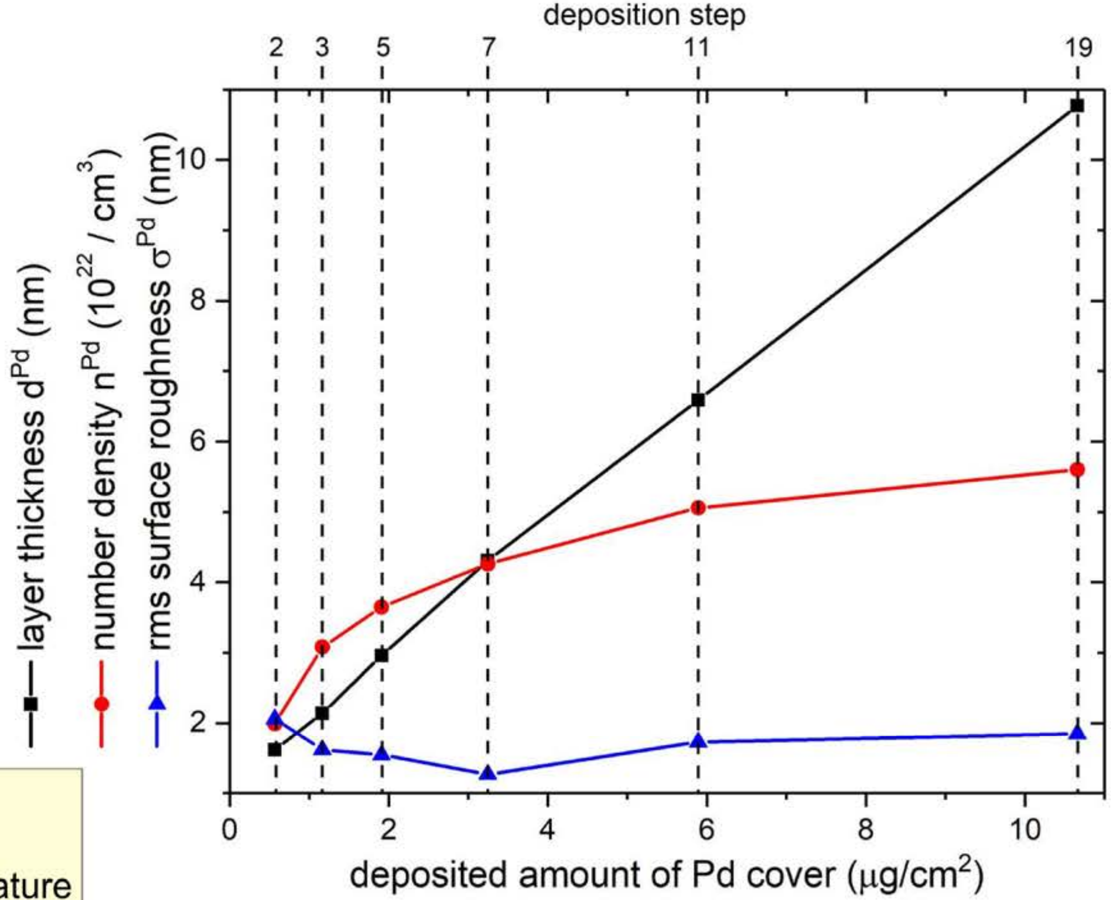


Fe-layer deposition step:

- continuous layer (rms roughness of Pd seed is reproduced by Fe)

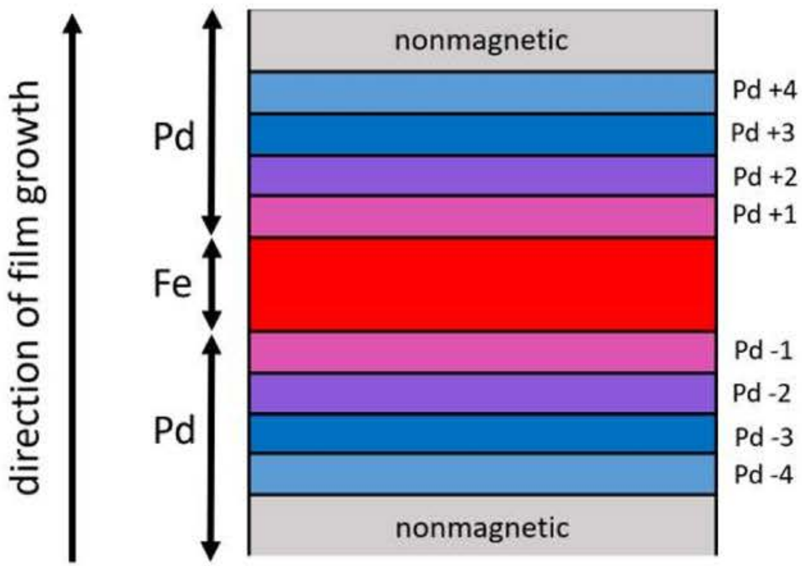
Structural parameters

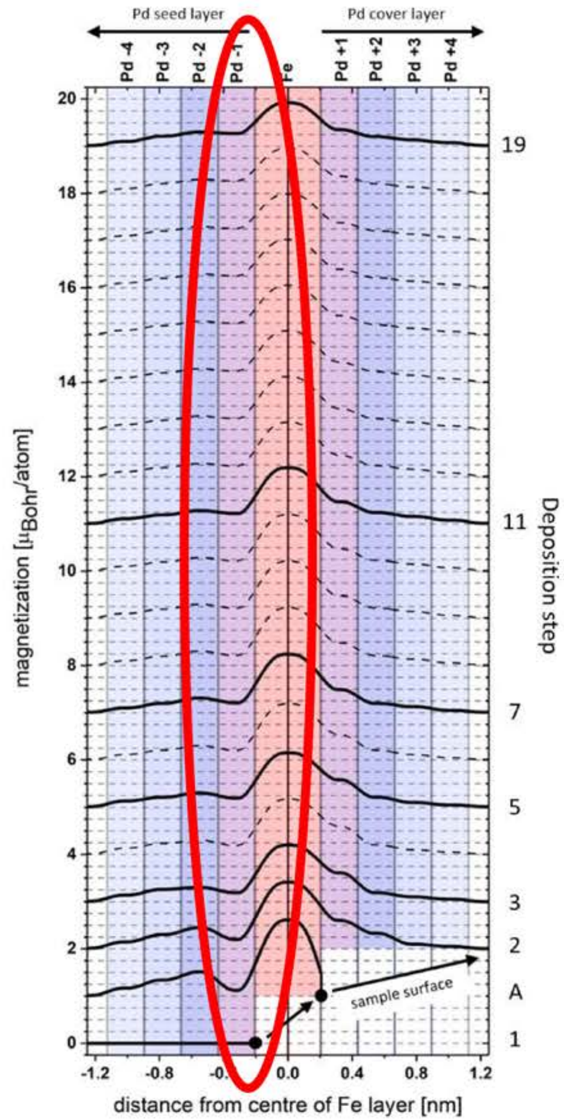
Pd-cover-layer:



Parameters:
- H = 70 mT
- Room temperature

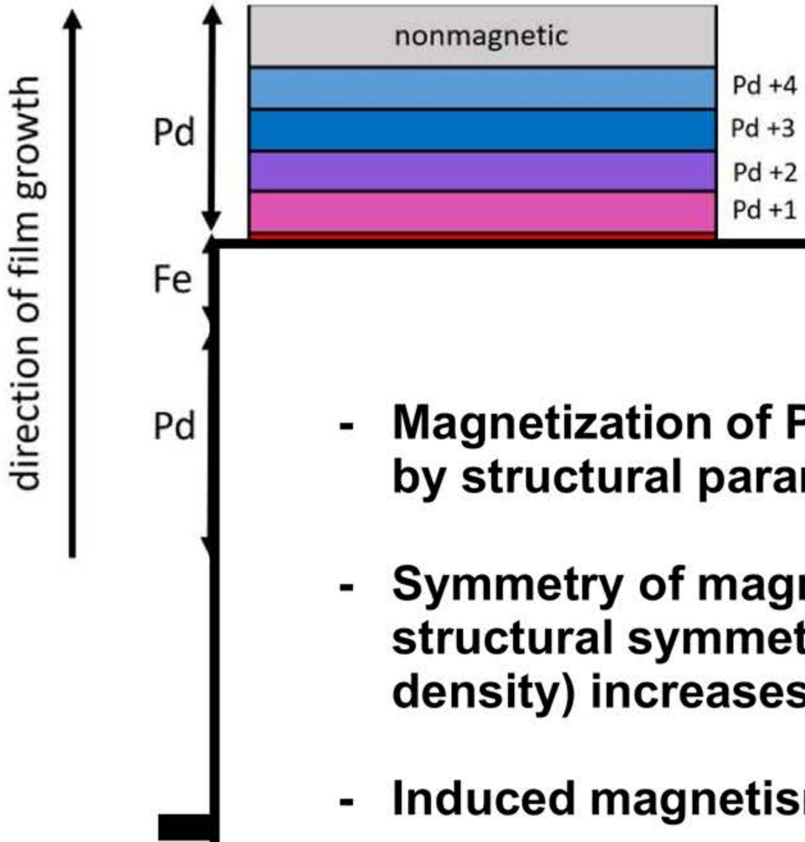
Current Experiments / possibilities

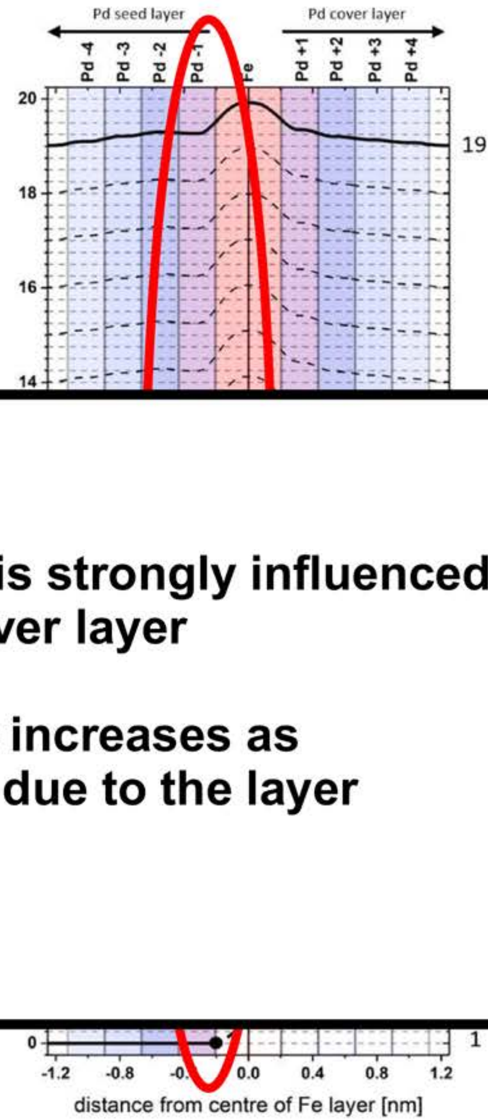




S. Mayr, J. Ye, J. Stahn, B. Knoblich, O. Klein, D.A. Gilbert, M. Albrecht, A. Paul, P. Böni, and W. Kreuzpaintner, *Indications for Interfacial Dzyaloshinskii-Moriya Interaction at the Pd/Fe/Pd Interface, Studied by In Situ Polarized Neutron Reflectometry*, Phys. Rev. B 101, 024404 (2020)
https://doi.org/10.1103/PhysRevB.101.024404

Current Experiments / possibilities





Results

- **Magnetization of Pd below the Fe is strongly influenced by structural parameters of Pd cover layer**
- **Symmetry of magnetizationprofile increases as structural symmetry (in particular due to the layer density) increases.**
- **Induced magnetism and IDM**

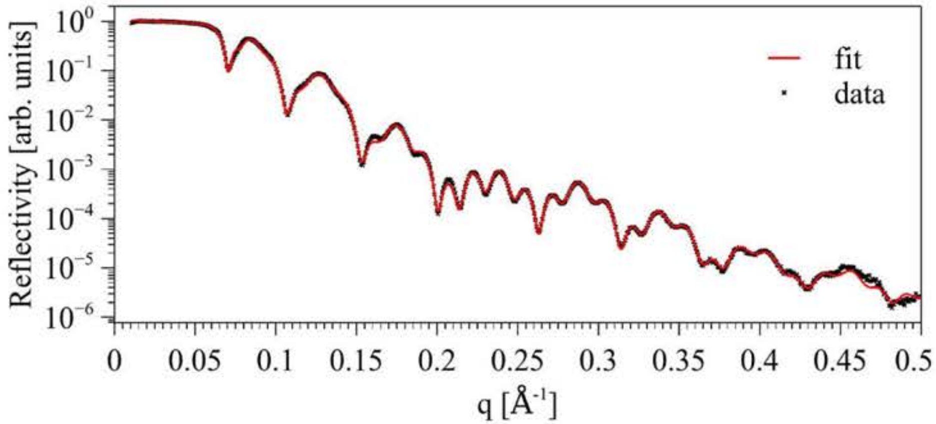
S. Mayr, J. Ye, J. Stahn, B. Knoblich, O. Klein, D.A. Gilbert, M. Albrecht, A. Paul, P. Böni, and W. Kreuzpaintner, *Indications for Interfacial Dzyaloshinskii-Moriya Interaction at the Pd/Fe/Pd Interface, Studied by In Situ Polarized Neutron Reflectometry*, Phys. Rev. B 101, 024404 (2020)
https://doi.org/10.1103/PhysRevB.101.024404

Current Experiments / possibilities

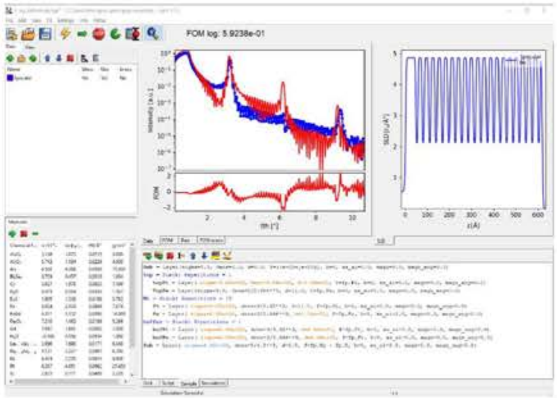
Solving the Phase Problem

Neutron reflectometry:

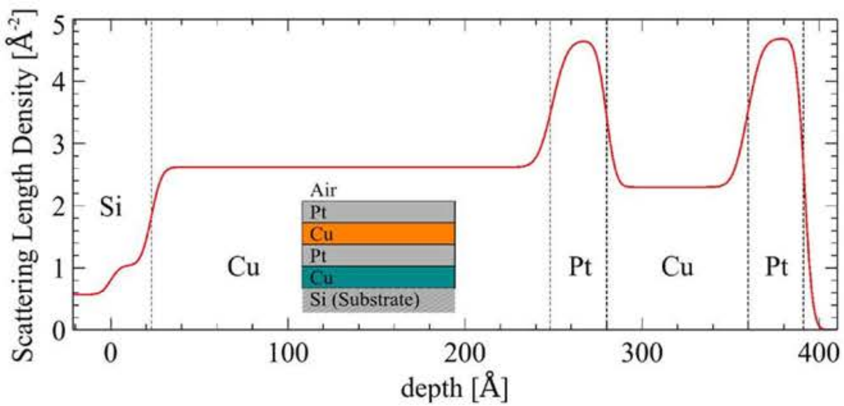
$$I = \frac{\#\text{reflected neutrons}}{\#\text{incident neutrons}}$$



Parameter Fitting:



https://a.fsdn.com/con/app/proj/genx/screenshots/Screenshot%202021-04-28%20204628.png/max/max/1



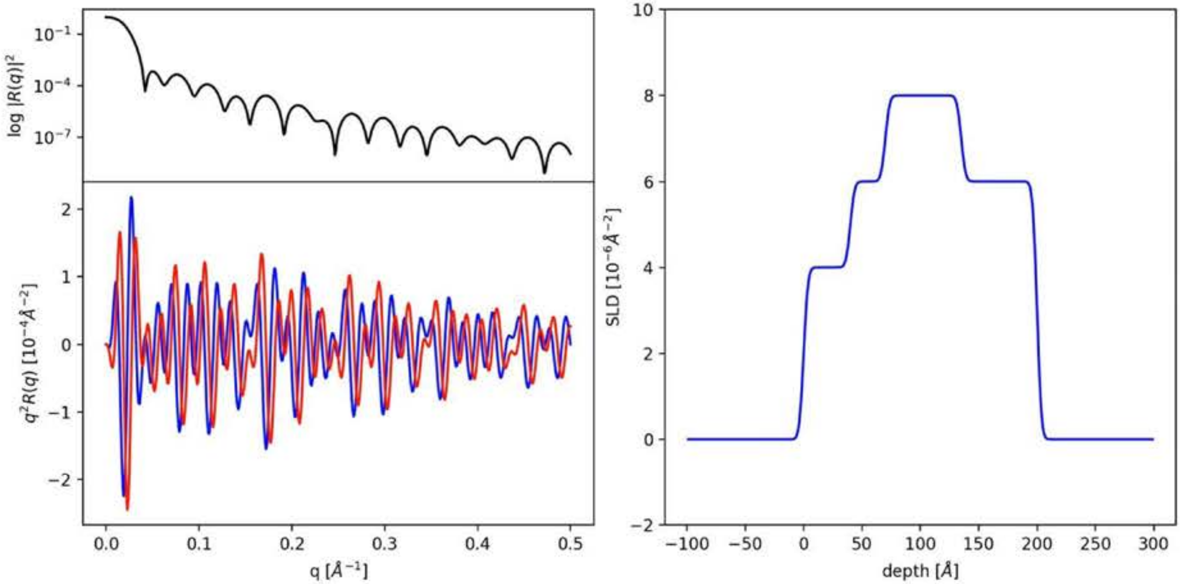
Current Experiments / possibilities

The phase problem:

$$|R(q)|^2 \sim \frac{16\pi^2}{q^4} \left| \int \frac{\mathrm{dSLD}}{\mathrm{d}z}(z) e^{iqz} \mathrm{d}z \right|^2$$

Convolution of the SLD with the inverse fourier transform of any phase yields the same reflectivity

Different SLDs yield the same reflectivity



Current Experiments / possibilities

A long standing Problem:

PHYSICAL REVIEW B
CONDENSED MATTER

THIRD SERIES, VOLUME 48, NUMBER 1 — 1 JULY 1993-I

Phase determination in x-ray and neutron reflectivity using logarithmic dispersion relations

William L. Clinton
Department of Physics, Georgetown University, Washington, D.C. 20057
(Received 23 November 1992)

Logarithmic dispersion relations are shown to be applicable to the determination of the phase of the structure factor of surface layers probed by neutron and x-ray reflectivity. For certain profiles it is shown that the phase $\Phi(q)$ of the structure factor $F(q)$ is determined *entirely* by the observed reflectivity $R(q)$ through the modified Hilbert transform $\Phi(q)=2q/\pi\int_0^\infty \ln[|F(q')|/|F(q)|]/(q^2-q'^2)dq'$, where q is the momentum transfer, and $F(q)$ is related to $R(q)$ and the Fresnel reflectivity via $R(q)=R_F(q)|F(q)|^2$.

Physics Letters A 170 (1992) 347–351
North-Holland
PHYSICS LETTERS A

A proposal for the determination of the phases in specular neutron reflection

H. Fiedeldey
Department of Physics, University of South Afri

PHYSICAL REVIEW B — VOLUME 52, NUMBER 15 — 15 OCTOBER 1995-I

Exact determination of the phase in neutron reflectometry

C. F. Majkrzak and N. F. Berk
Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899-0001
(Received 18 January 1995)

ELSEVIER
Current Opinion in Colloid & Interface Science
Volume 17, Issue 1, February 2012, Pages 44-53

Phase-sensitive specular neutron reflectometry for imaging the nanometer scale composition depth profile of thin-film materials

B.J. Kirby [a], P.A. Kienzle [a], B.B. Maranville [a], N.F. Berk [a], J. Krycka [a], F. Heinrich [b], C.F. Majkrzak [a]

ELSEVIER
Physica B: Condensed Matter
Volume 248, Issues 1–4, 15 June 1998, Pages 338-342

Phase determination and inversion in specular neutron reflectometry

C.F. Majkrzak a, N.F. Berk a, J.A. Dura a, S.K. Satija a, A. Karim b, J. Pedulla b, R.D. Deslattes c

Experimental Approbation of Reference Layer Method in Resonant Neutron Reflectometry

E. S. Nikova, Yu. A. Salamatov, E. A. Kravtsov, M. V. Makarova, V. V. Proglyado, V. V. Ustinov, V. I. Bodnarchuk & A. V. Nagorny

Physics of Metals and Metallography 120, 838–843 (2019) | Cite this article
62 Accesses | 1 Citations | Metrics

Published: 06 December 2016

Use of gadolinium as a reference layer for neutron reflectometry

Yu. A. Salamatov & E. A. Kravtsov

Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques 10, 1169–1172 (2016) | Cite this article
35 Accesses | 7 Citations | Metrics

… and more …

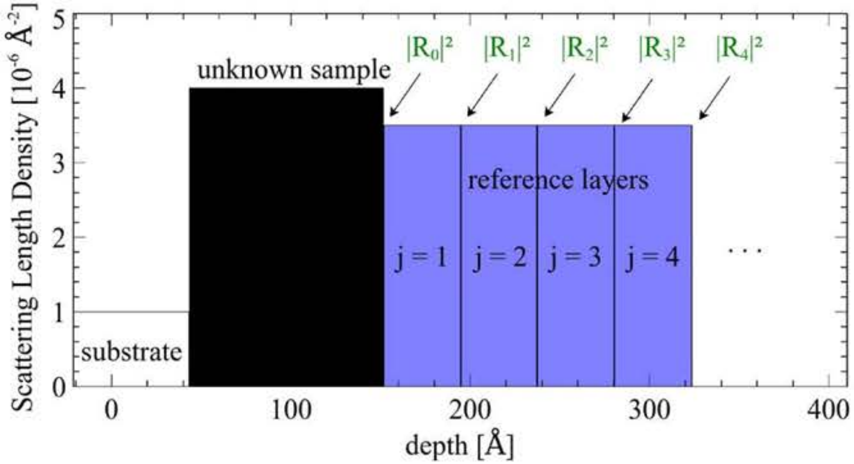
We propose a solution based on the “reference layer method”

Current Experiments / possibilities

Reconstructing the Phase information with the help of *in situ* grown additional layers

Variation of Reference Layers (for *in situ* use)

$$\frac{1+|R_j|^2}{1-|R_j|^2} = c_{\text{known},j} \cdot \begin{pmatrix} \alpha \\ \beta \\ \gamma \end{pmatrix}$$



After measuring at least 3 reflectivities $|R_i|^2$ with known reference layers, one can retrieve the reflectivity of the initially unknown part

$$R_0 = \frac{\beta - \alpha + 2i\gamma}{\alpha + \beta + 2}$$

A. Book, S. Mayr, J. Stahn, P. Böni, and W. Kreuzpaintner, *Phase Reconstruction of a Cu(001) Seed Layer from in situ Polarized Neutron Reflectometry Data using Fe Reference Layers*, Nucl. Inst. Meth. Phys. Res., A 1023, 165970 (2021). https://doi.org/10.1016/j.nima.2021.165970

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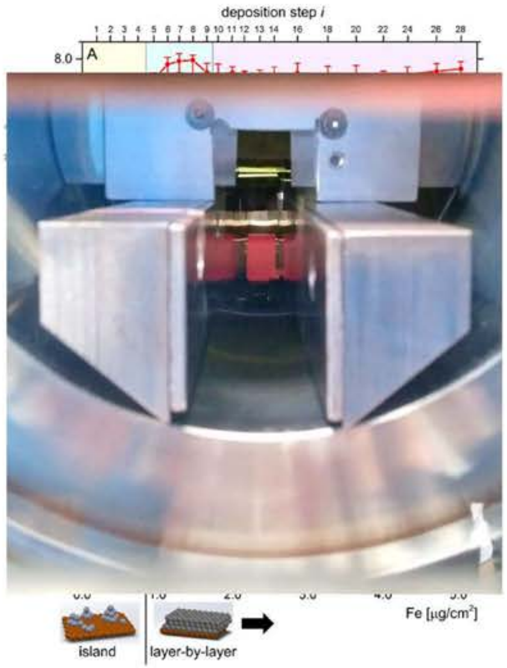
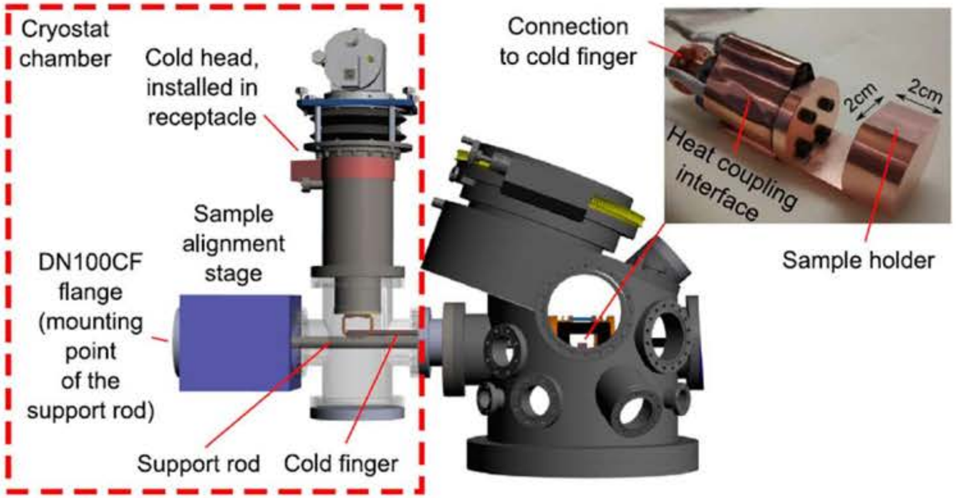
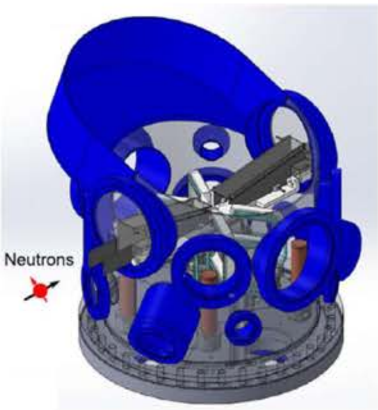


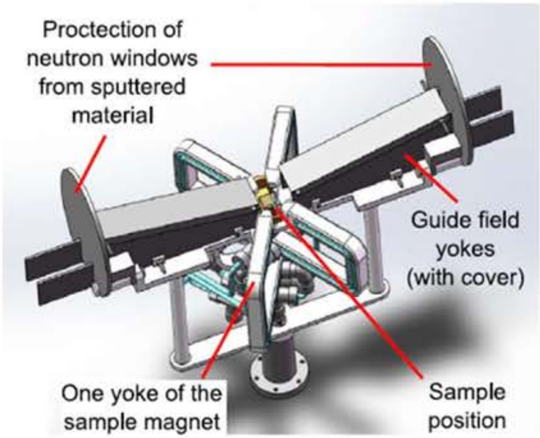
Conclusion and Outlook

Latest Developments

Even more optimized sample environment

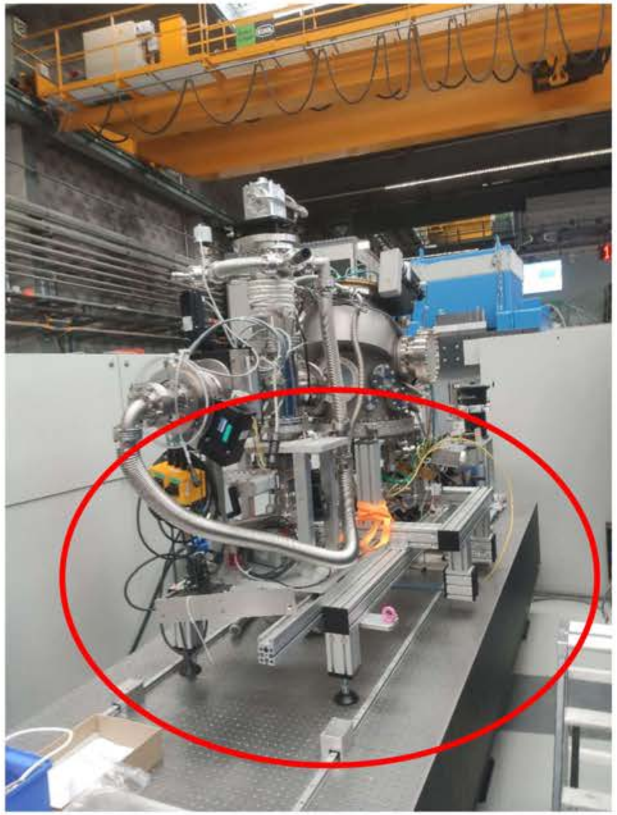






Latest Developments

Transferred and handed over to PSI



New stand to fit the upgrade of the Amor Beamline

https://www.psi.ch/sites/default/files/styles/primer_content_xl/public/2021-09/amor_areal.png?itok=rxOO5f0R

Christine Klauser

Jochen Stahn

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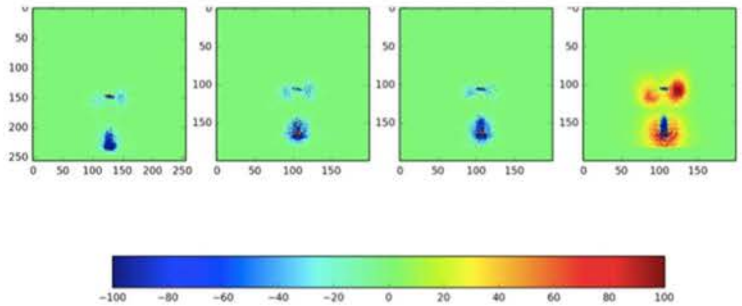
- Current Experiments / Possibilities
- Latest Developments

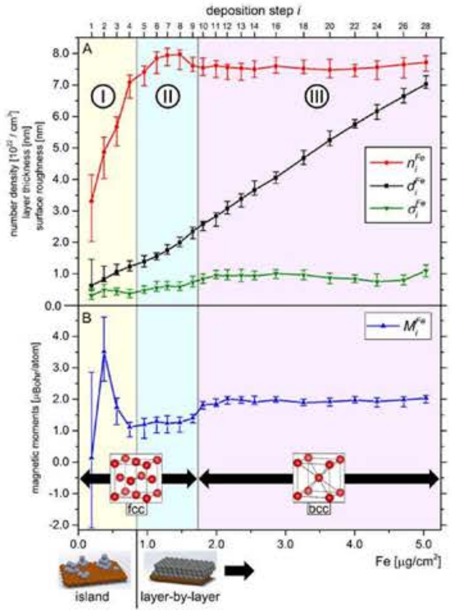


Conclusion and Outlook

Conclusion and Outlook

Development

- sample environment
- methodology of *in situ* PNR
- fully completed
- Available for users on Amor @ PSI



Applications:

- layer-by-layer growth of Fe on Cu
- Pd/Fe/Pd trilayer
- Phase reconstruction

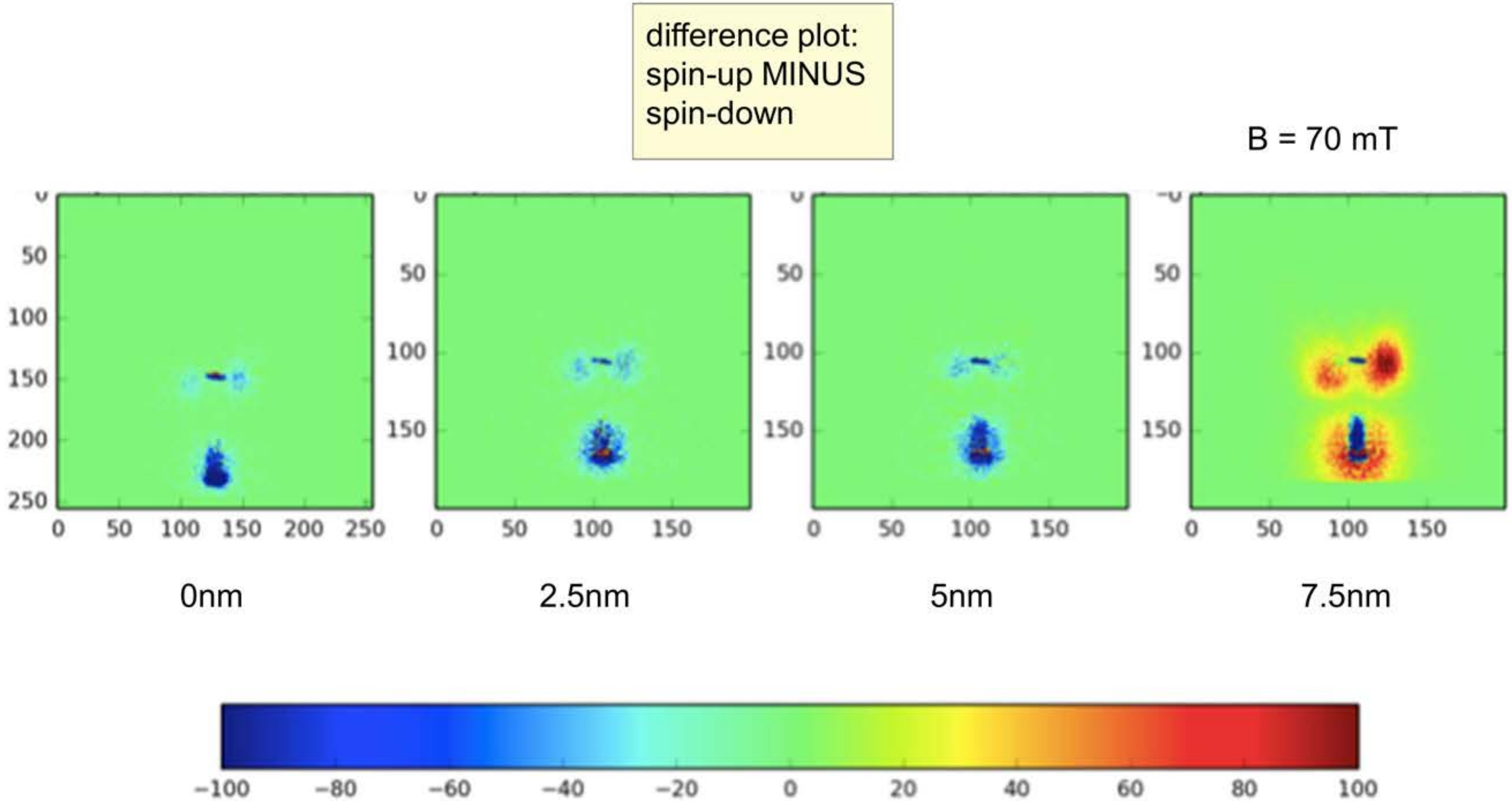
Increase of interest in the technique by external users

- Magnetism
- Soft matter (additionally → GISANS)

Conclusion and Outlook

ToF-GISANS test @ REFSANS:

- onset of magnetism on nanostructured surfaces (Fe on facetted Sapphire)



Conclusion and Outlook

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Thank you for your attention.

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