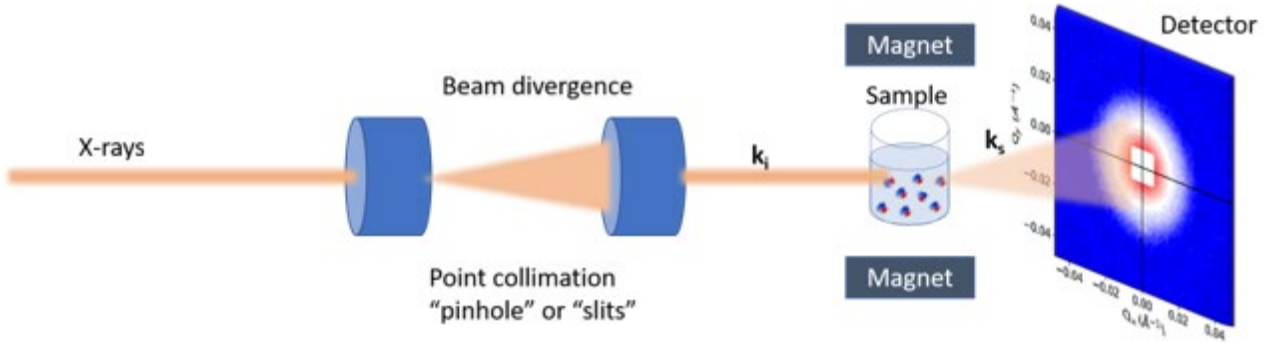
Small-Angle X-Ray Scattering for Studying Nanostructured Systems

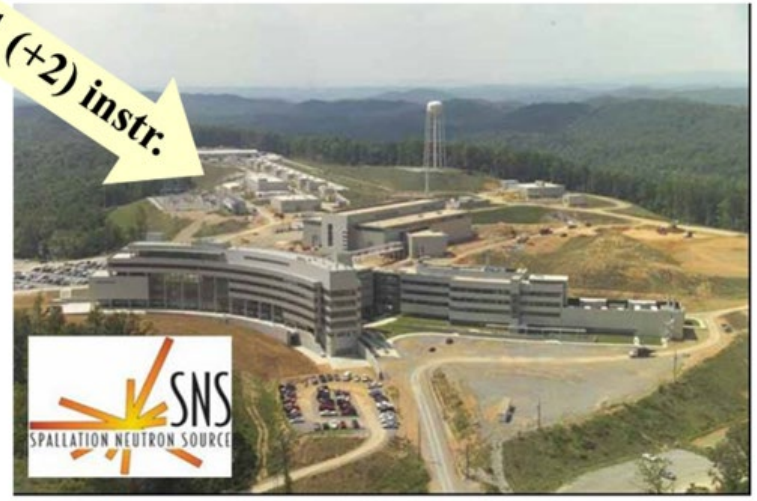
Asmaa Qdemat



Where I work:

Jülich Centre for Neutron Science (JCNS-2)

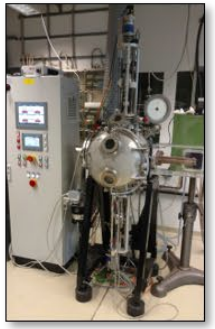
Jülich Centre for Neutron Science



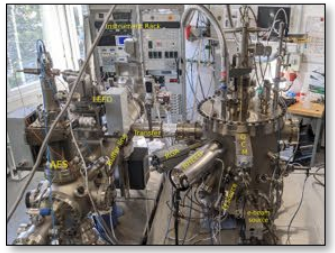
JCNS-2 Facilities

Sample preparation

Single crystal & poly-crystalline



Thin film



Magnetization

MPMS

DynaCool

PPMS

In-house X-ray facilities

4-circle diffractometer

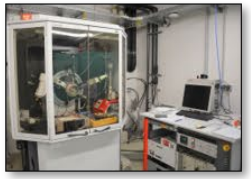
GALAXI

Powder X-ray diffractometer

Supernova single crystal diffractometer

Laue Camera

Bruker AXS reflectometer



Contents

- Introduction
- Instrumentation
- Experimental techniques
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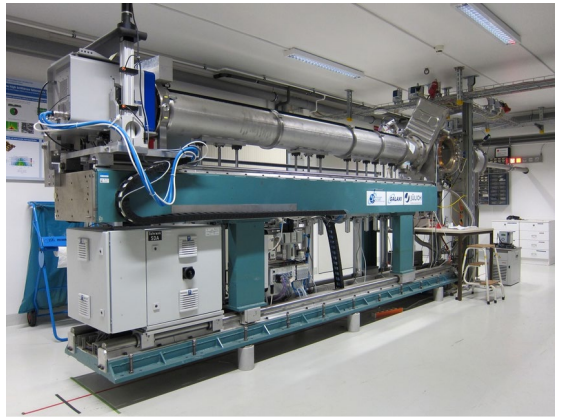
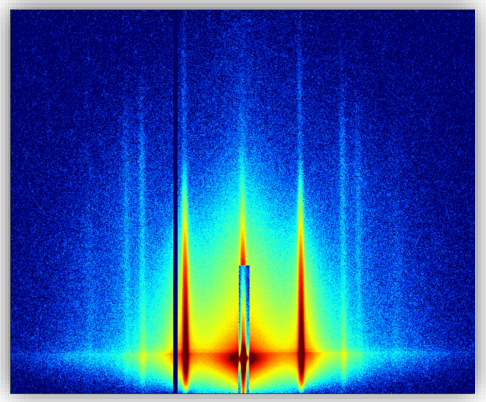
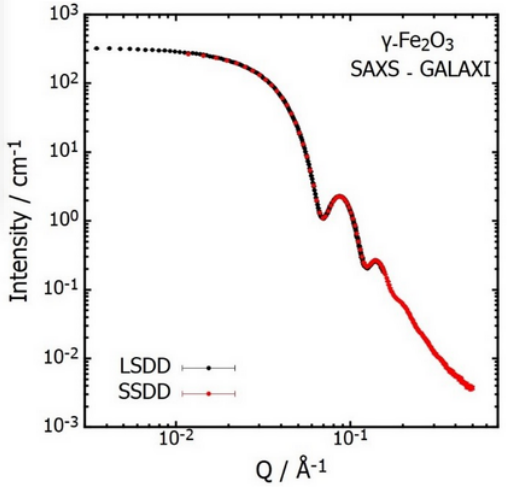
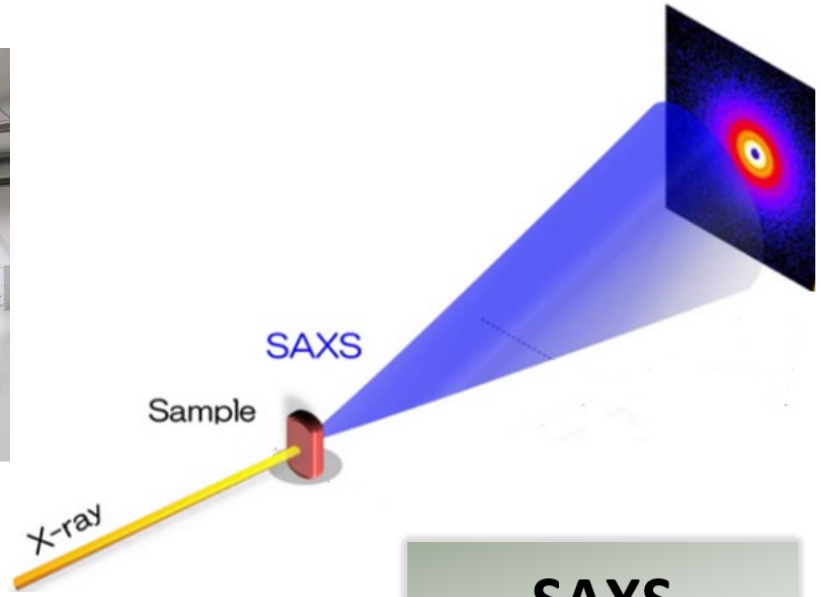
- PDF @ ID09 - SESAME
- Future plans at SESAME









SAXS

↓

GISAXS / XRR

↓

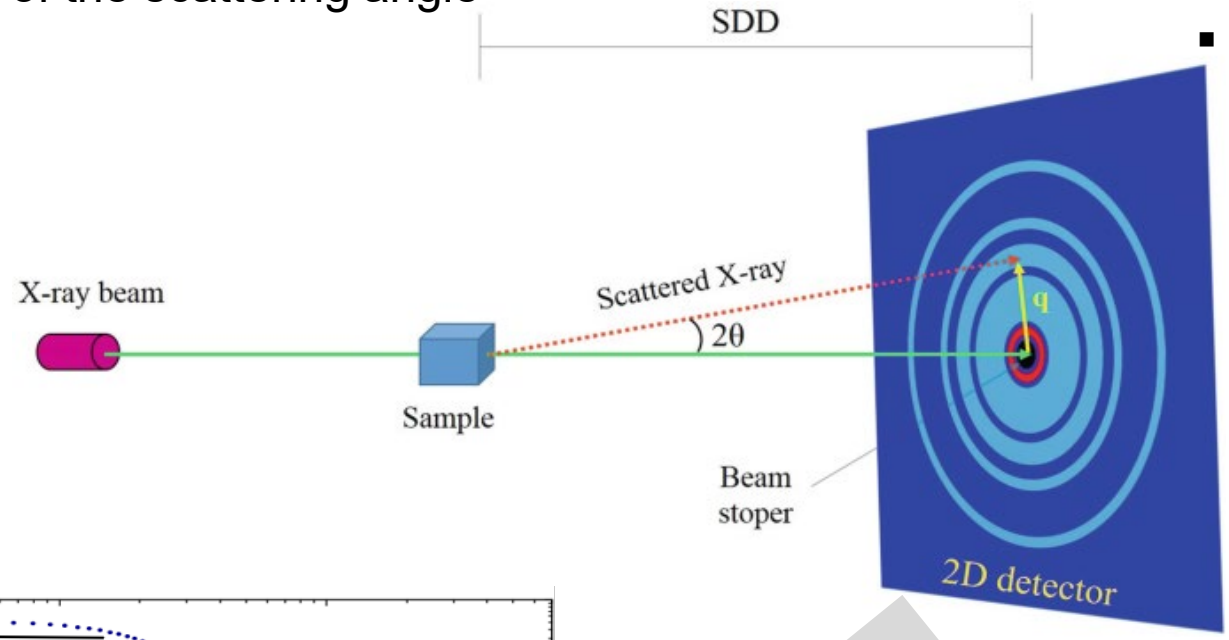
WAXS

"When the scientist have learned how to control the arrangement of matter at very small scale they will see material take an enormously richer variety of properties"

Richard Feynman (1929)

Small-Angle X-Ray Scattering

- Analytical technique, measures the intensities of X-rays scattered by a sample as a function of the scattering angle

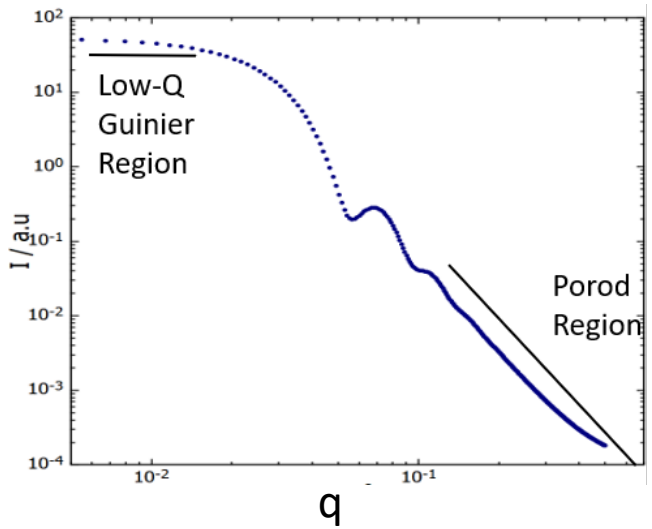


- SAXS → Mesoscopic length scale, 1 to several hundred nm
  - → chemical composition, Particle size, shape and correlations, formation of superstructures
- Transmission mode
- Elastic scattering

$$I(q) = N\left|F(q)\right|^2 S(q)$$

$S(q)$: interactions between particles

$\left|F(q)\right|^2$: Shape, Size



Integration a long azimuthal angle

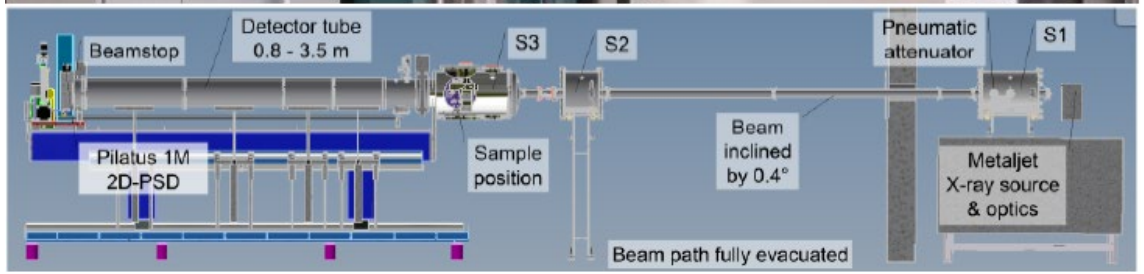
Intensity function (I(q)) related to the scattering vector amplitude (q)

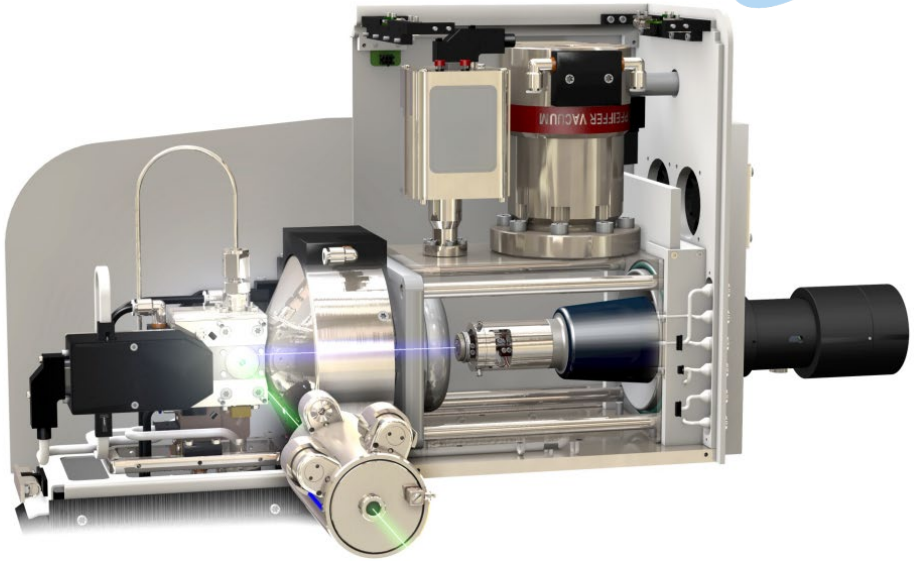
$$q = \frac{4\pi}{\lambda}\sin 2\theta \quad \Rightarrow \quad q = \frac{2\pi}{D}$$

GALAXI @ JCNS

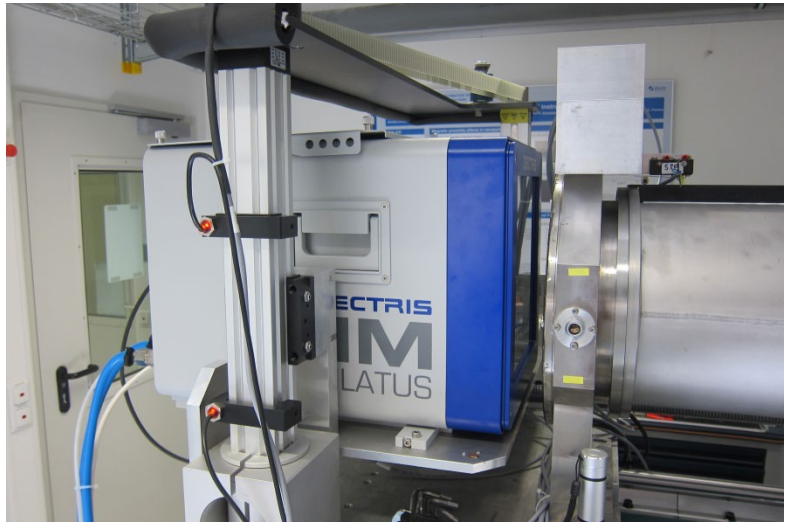
Gallium Anode Low-Angle X-ray Instrument (GALAXI)

- Small angle X-ray diffractometer
- Laboratory X-ray source with the highest brilliance available today





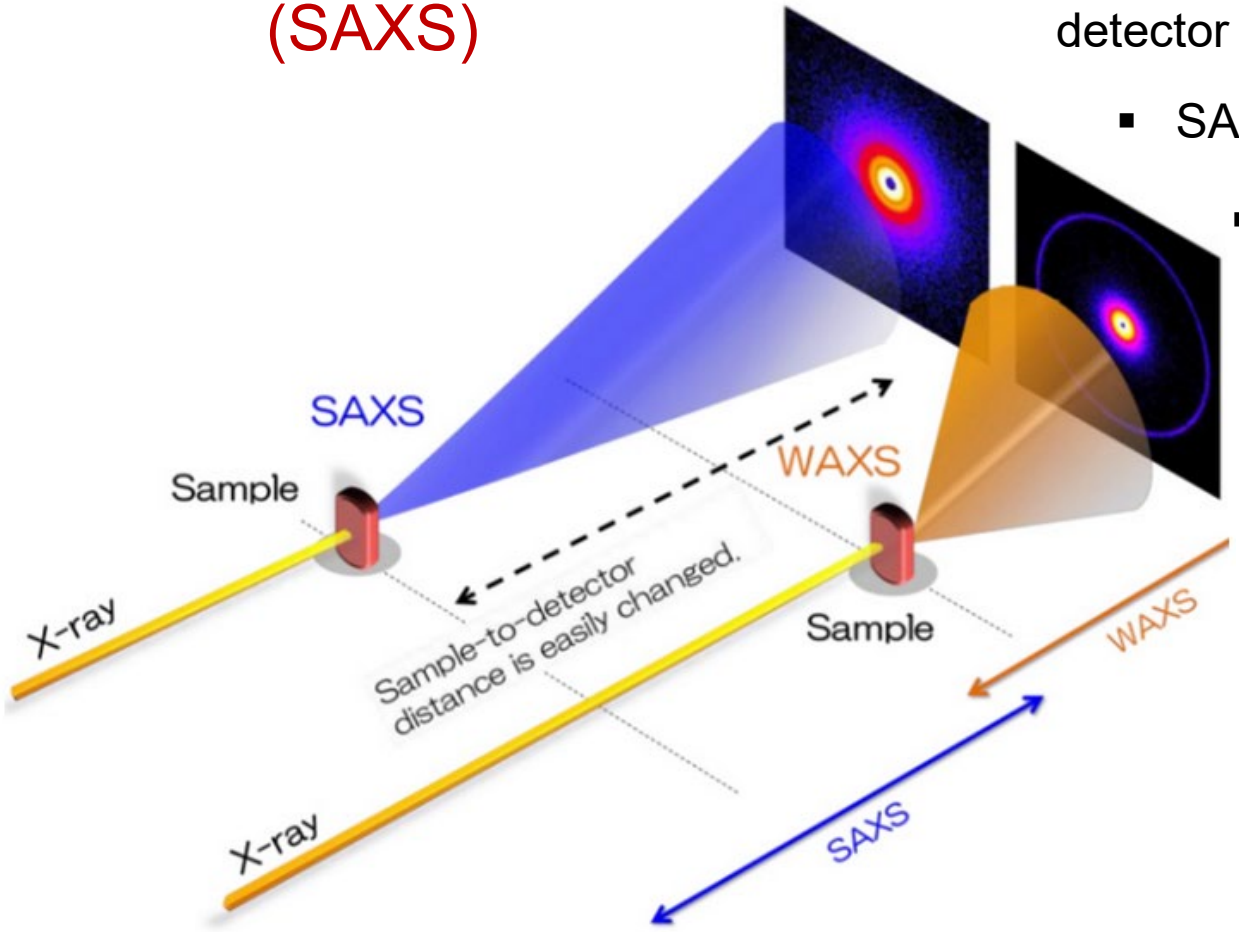
Metaljet from Bruker AXS



1M Pilatus from Dectris

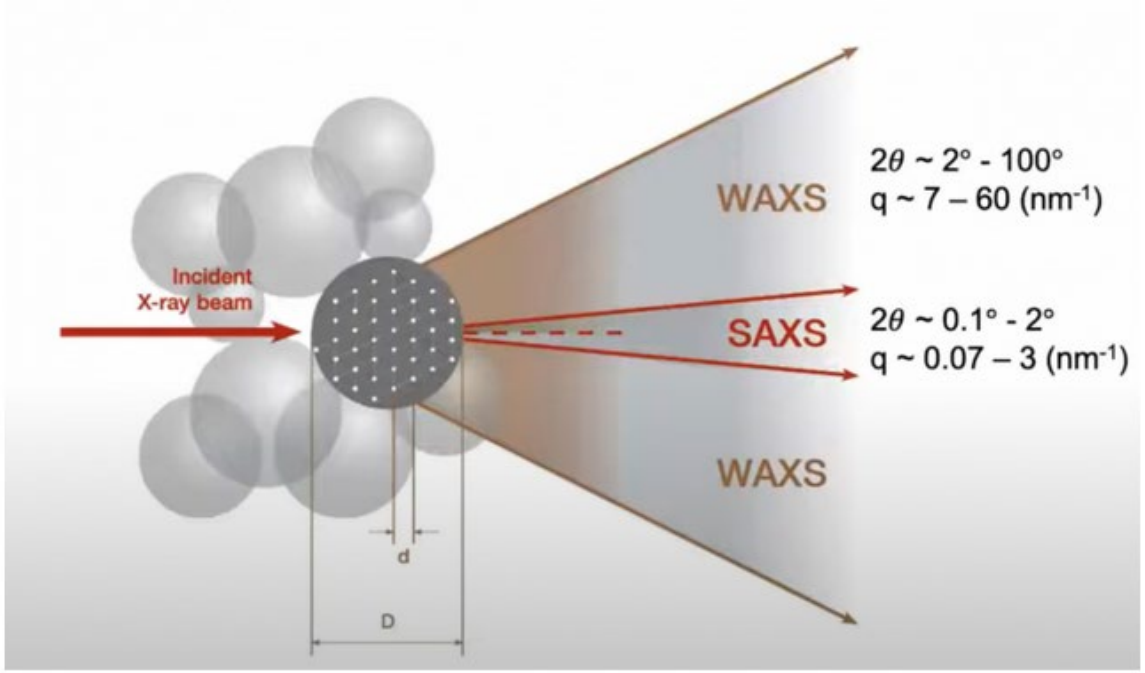
Experiments @ GALAXI

Small Angle X-ray Scattering (SAXS)



Wide Angle X-ray Scattering (WAXS)

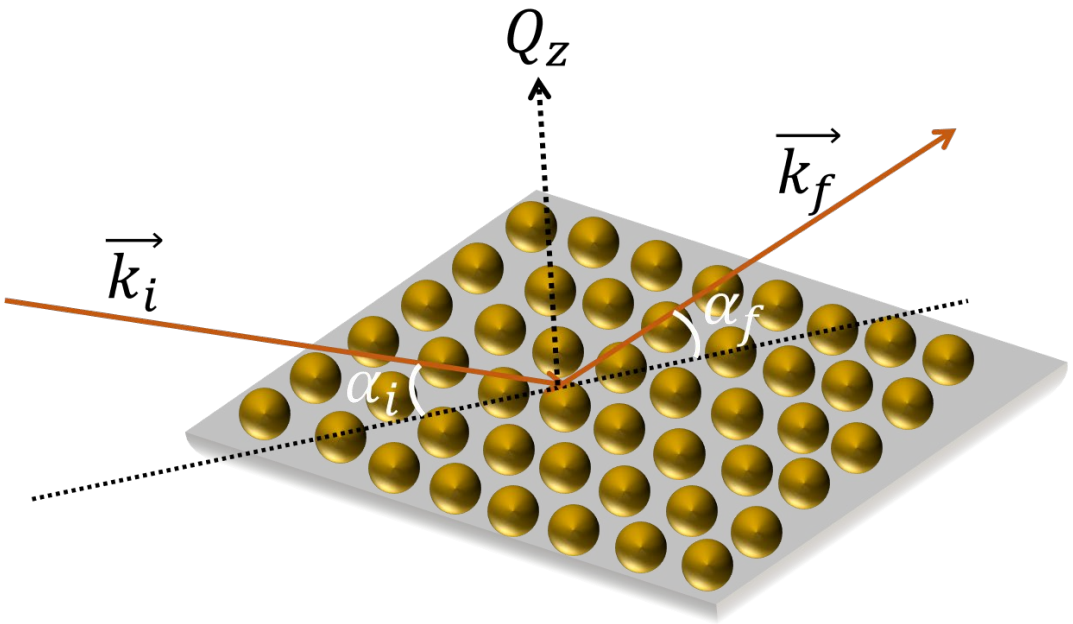
- SAXS and WAXS can be done simultaneously by moving the detector closer or further away from the sample
  - SAXS ⟶ very small angles ⟶ nanoscale resolution
  - WAXS ⟶ wider angles ⟶ atomic resolution



https://www.princetoninstruments.com/learn/x-ray-scattering/intro-to-x-ray-scattering

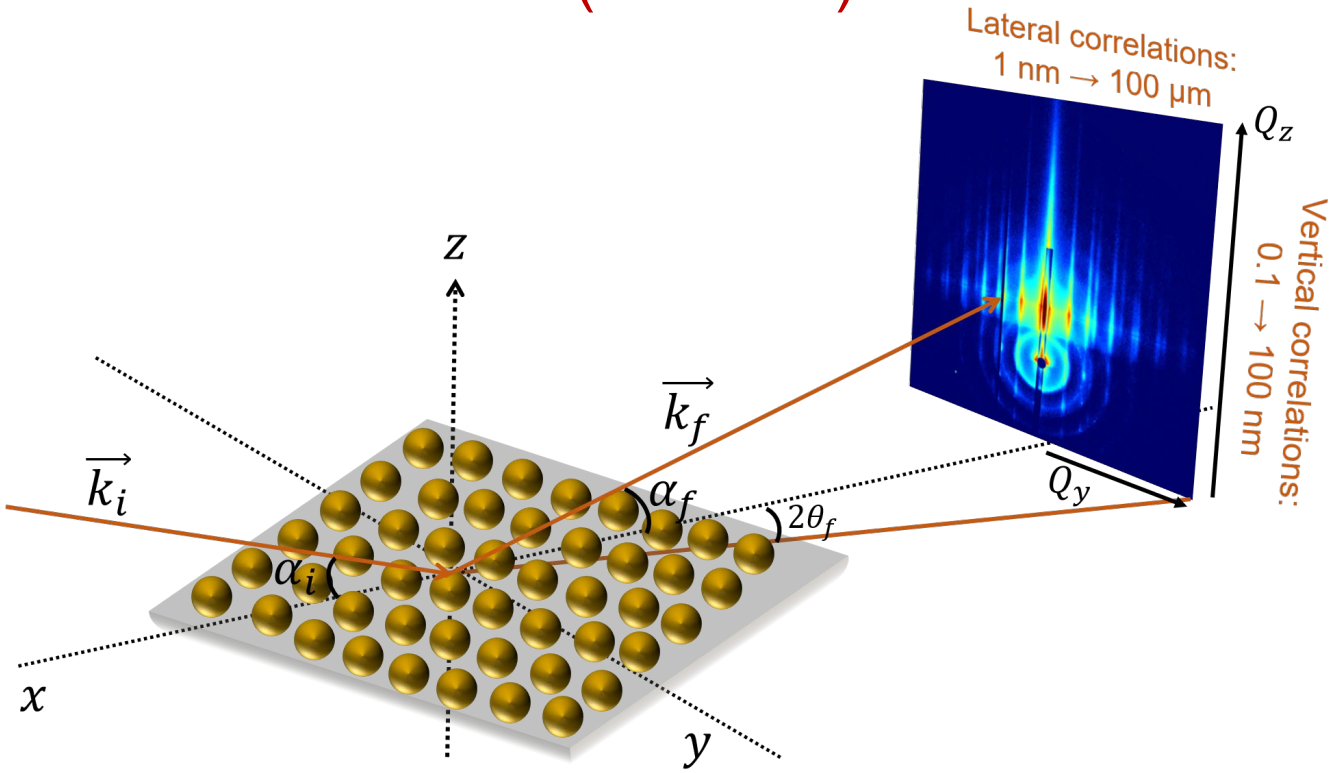
Experiments @ GALAXI

X-Ray Reflectometry (XRR)



- Specular reflectivity, $\alpha_f = \alpha_i$
- Laterally average density profile
- Out-of-plane structure
- Layer thickness / Interface roughness

Grazing Incidence Small Angle X-ray Scattering (GISAXS)



- $\alpha_f \neq \alpha_i$, $\vec{Q}$ has in-plane component
- In-plane structural correlation.
- Information about buried objects, object geometry, size distribution and spatial correlations

Sample environment

SAXS

Permanent Magnet

B1 = 0.78 T

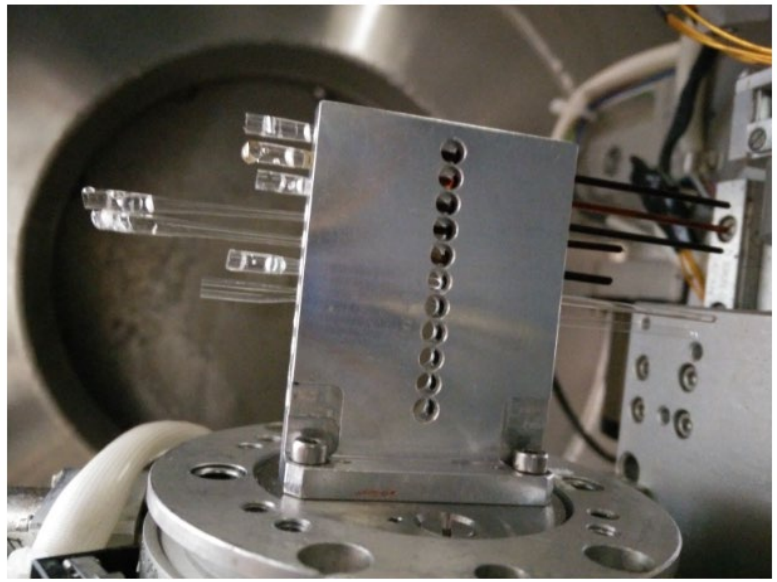
B2 = 0.9 T

Temperature

10°C – 70°C

Immobilized samples

Solution

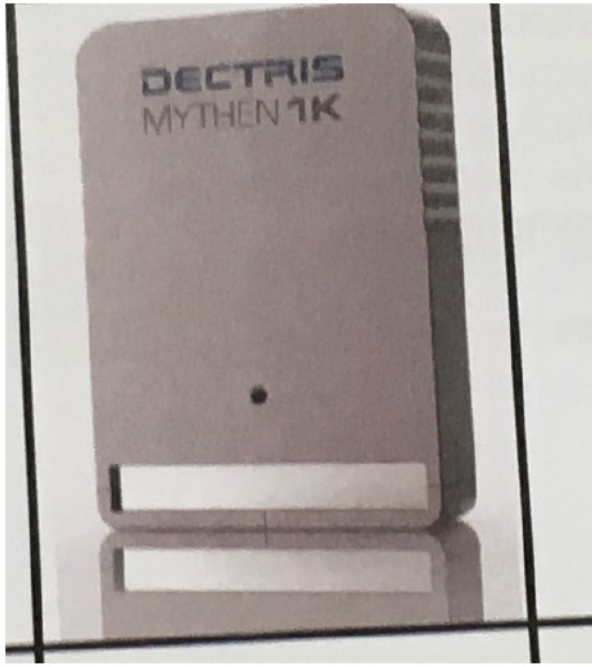


Quartz glass capillaries (Hilgenberg GmbH)

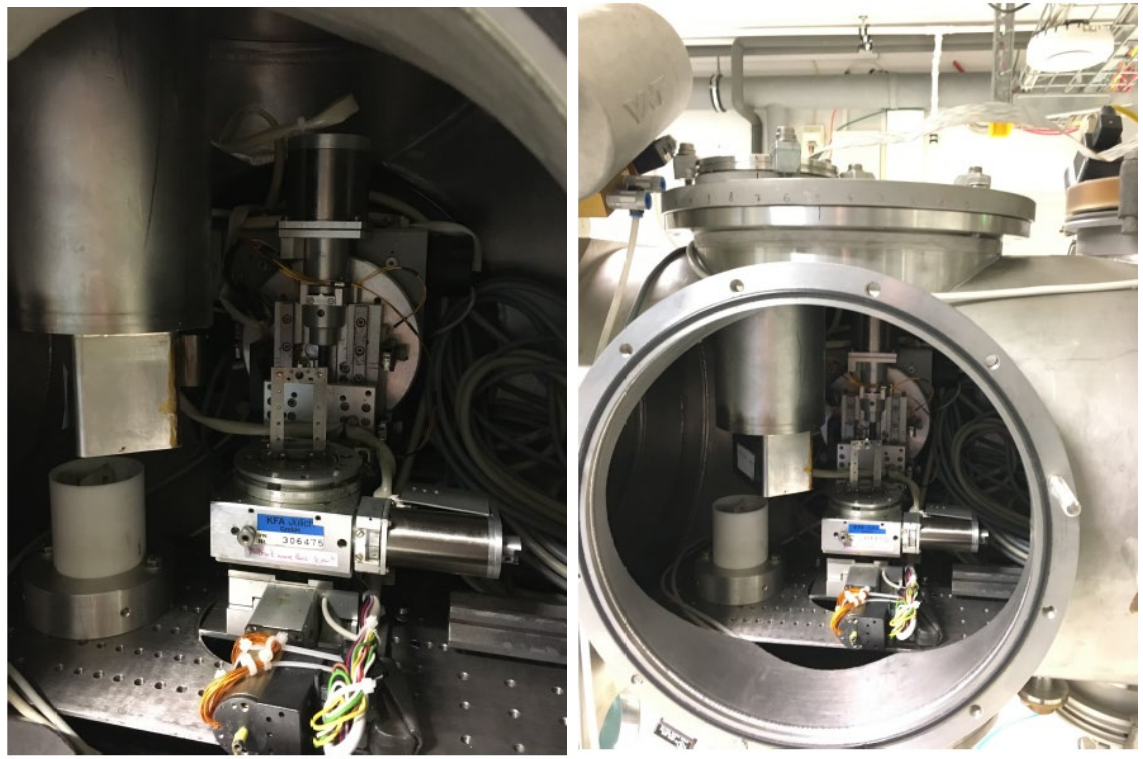
- Size and size distribution of NPs
- Self-assembly of the particles in dispersion at room temperature.
- Effect of size, concentration, and applied field in self-assembly of NPs

WAXS

Sample environment & Setup



- 1D position-sensitive detector
  → Mythen 1k from Dectris
- Oriented Horizontally
- covers 41 degrees in 2Θ
- WAXS with applying voltage
- Thin film sample

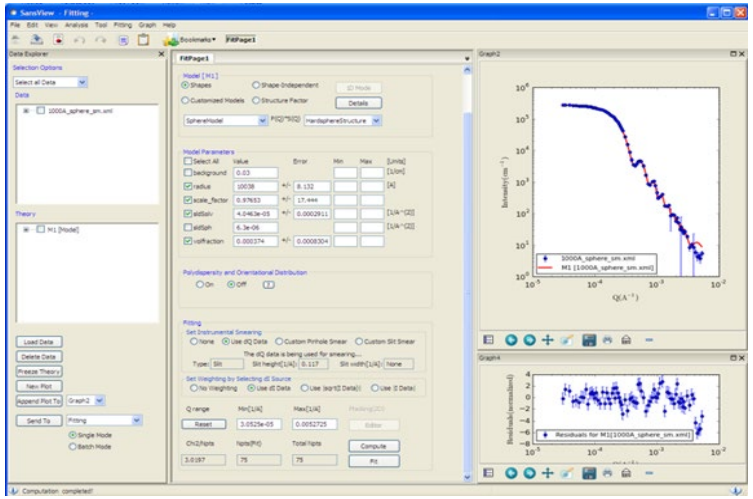


Sample-to-detector distance = 85 m

FUTURE → **GIWAXS measurement** → in-plane polycrystalline samples

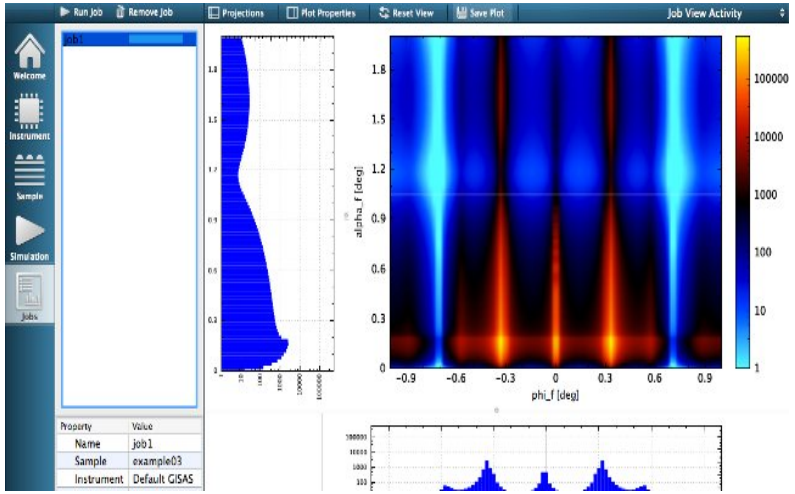
Modelling of SAXS, GISAXS and XRR data

SasView



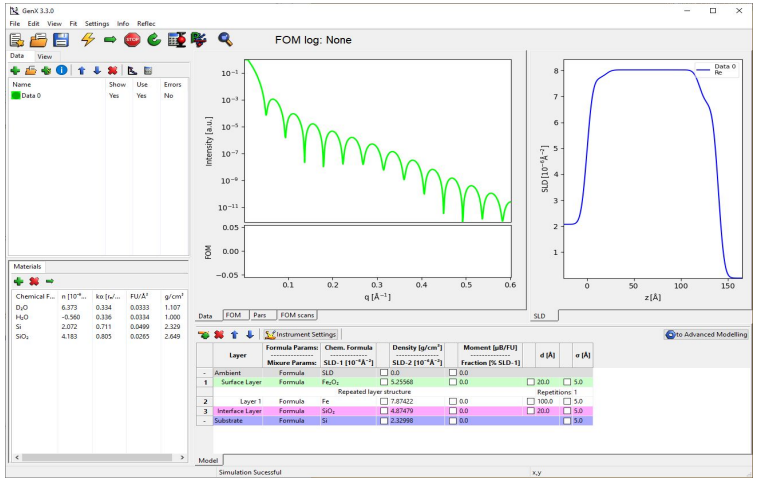
http://doi.org/10.5281/zenodo.3653469

BornAgain



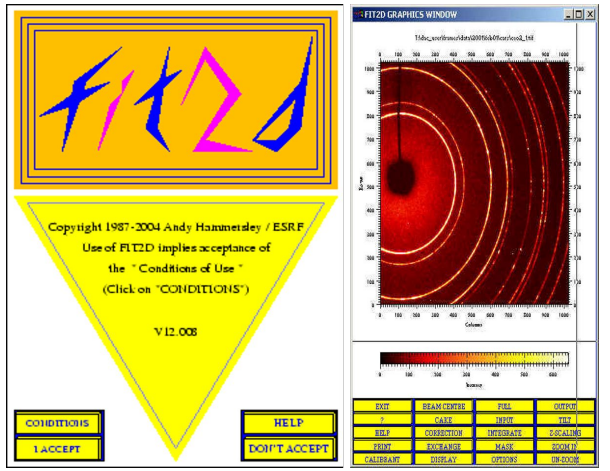
J. Appl. Cryst. (2020). 53, 262–276

GenX



J. Appl. Cryst. (2022). 55, 1063-1071

Fit2d

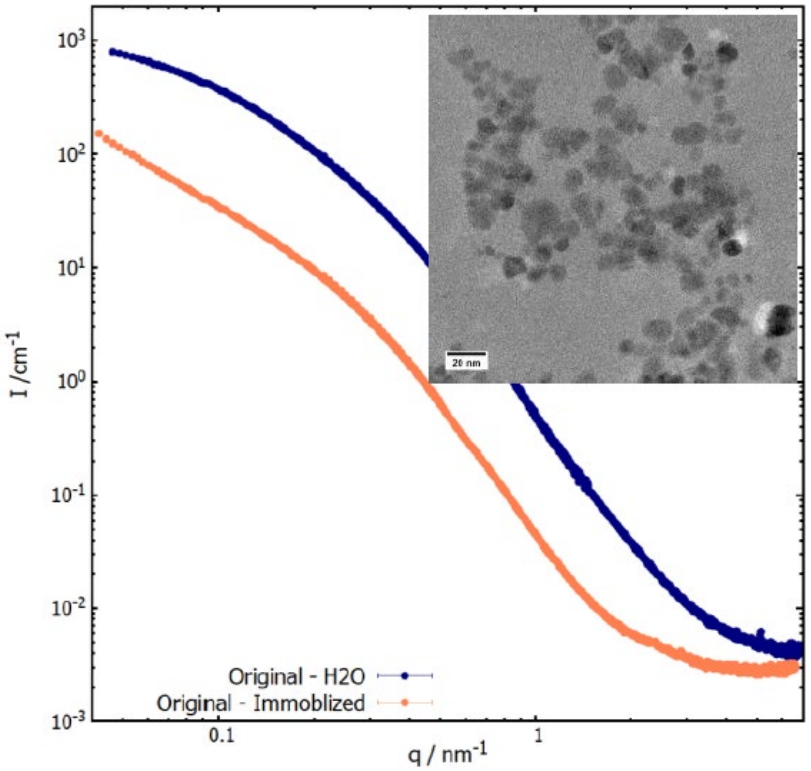


J. Appl. Cryst. (2016). 49, 646-652

Scientific results

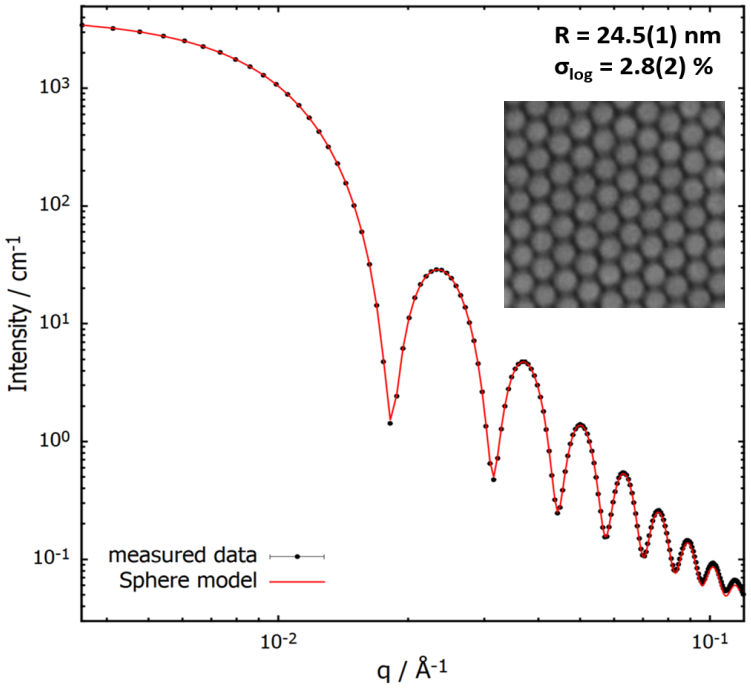
SAXS

- Statistically average information

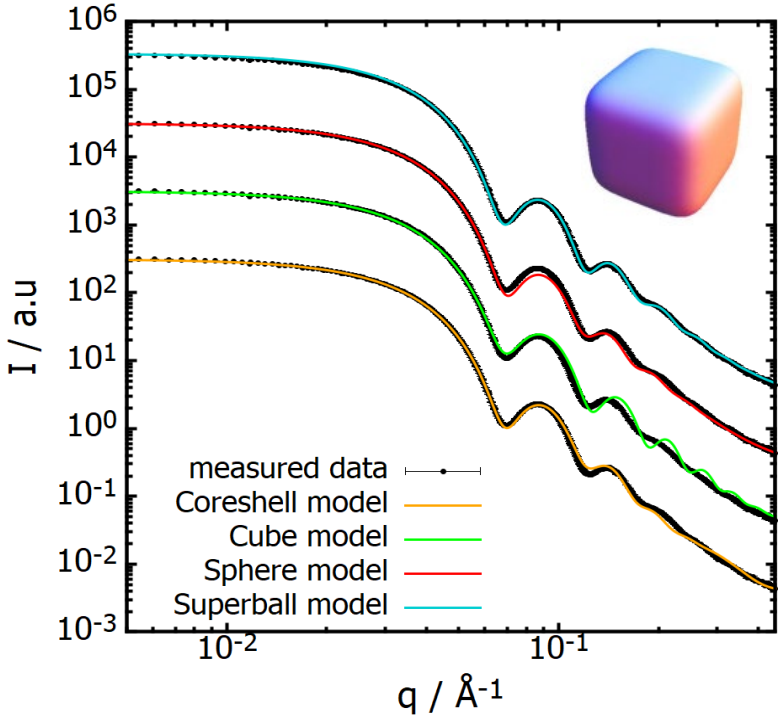


SPION^{Citrate} in water and immobilized in crosslinked polymer matrix

- Spherical nanoparticles
  - Silica NP
  - Grafted with Stearylalcohol
  - Dispersed in toluene



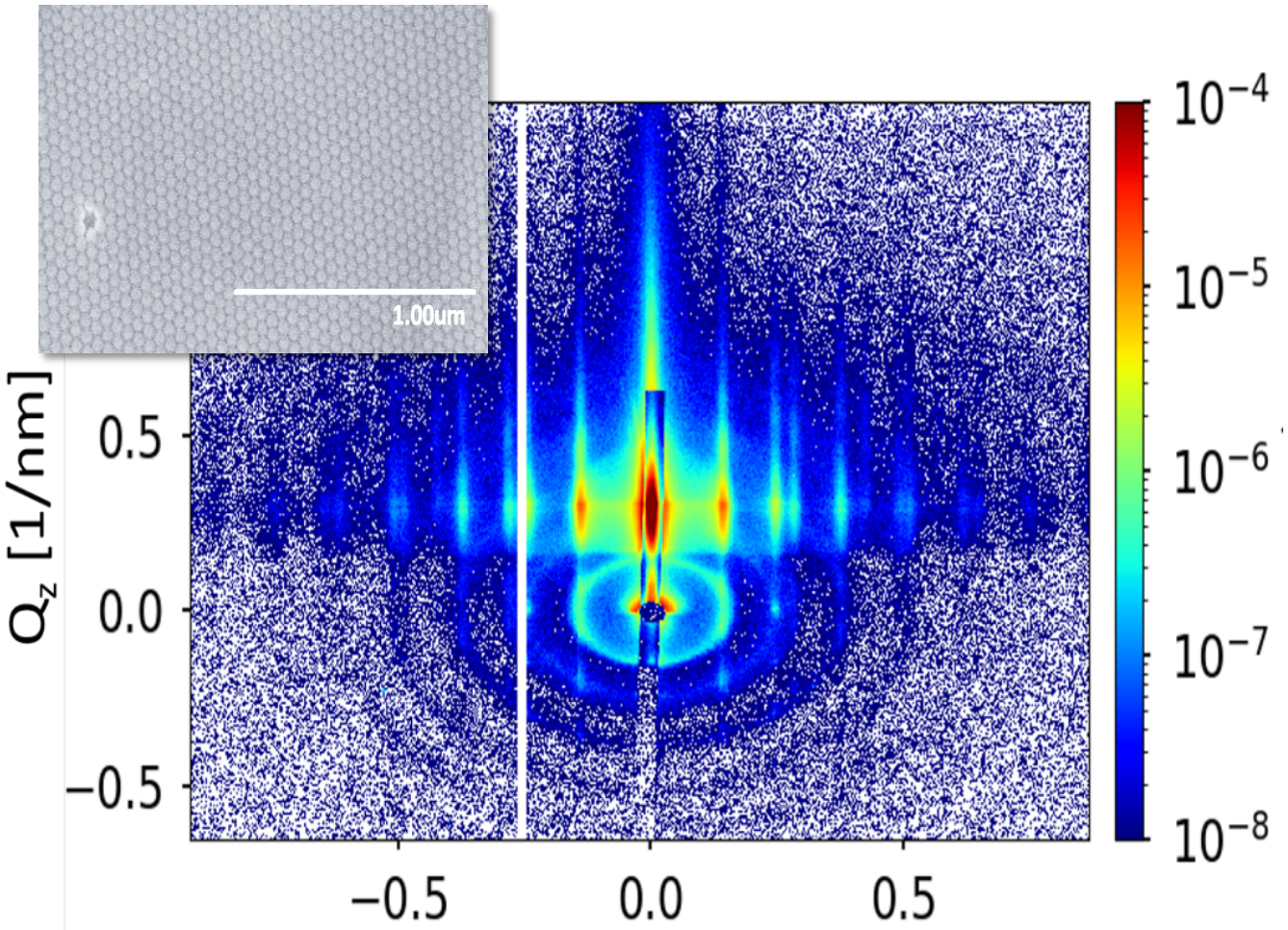
- Superball nanoparticles
  - Iron oxide (maghemite)

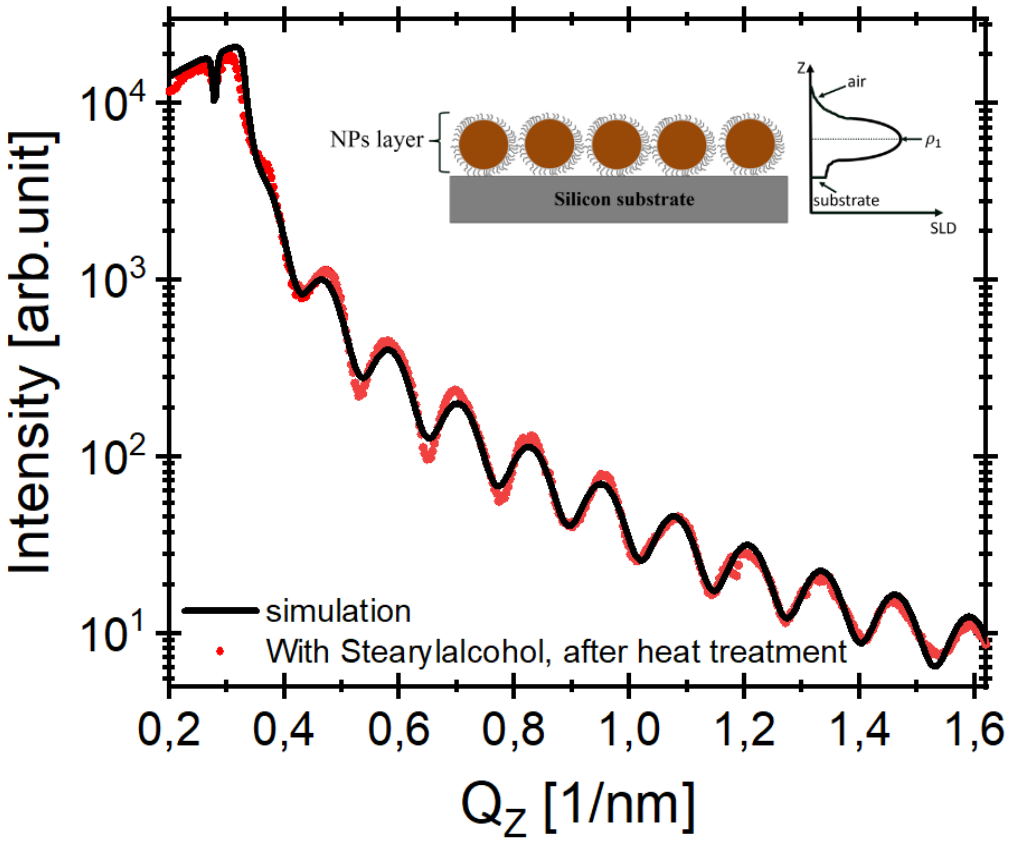


J. Phys. Chem. C 2021, 125, 42, 23356–23363

Reflectometry and grazing-incidence scattering

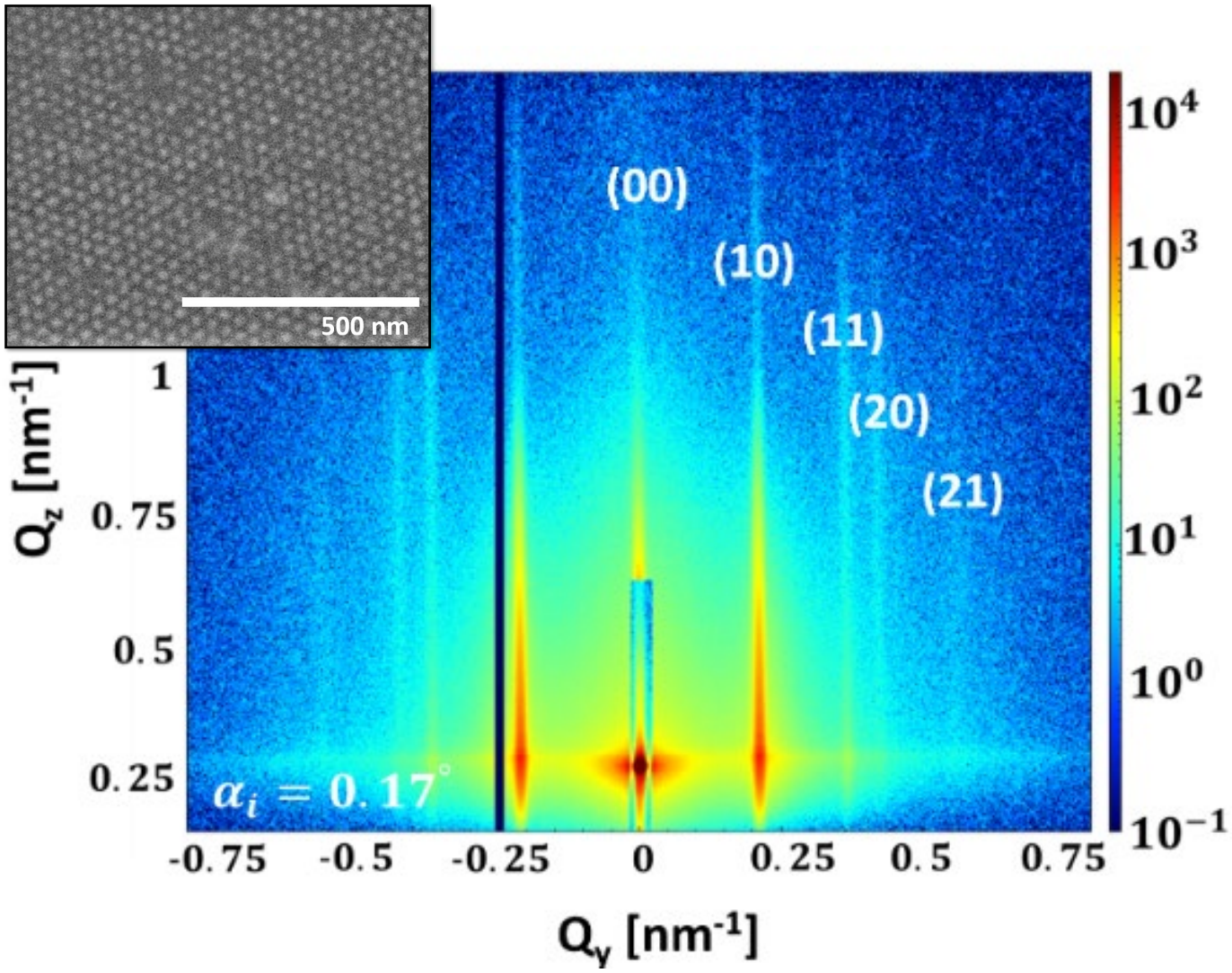
Self assembled monolayer of silica nanoparticles with improved order by drop casting

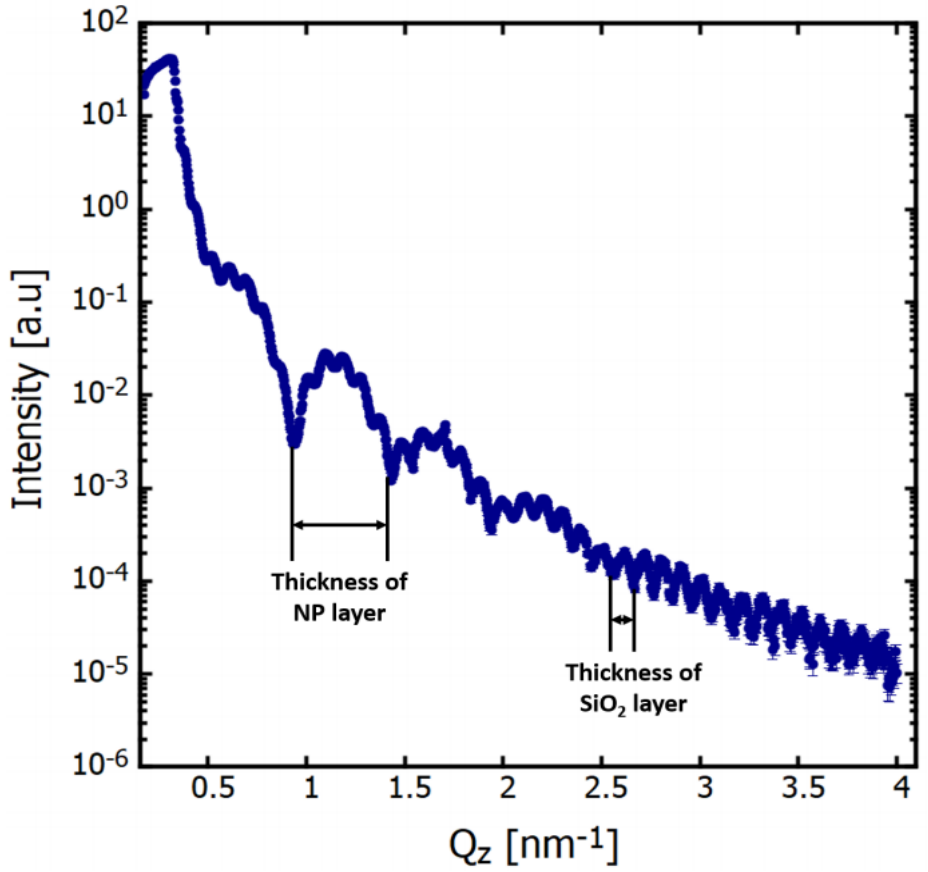


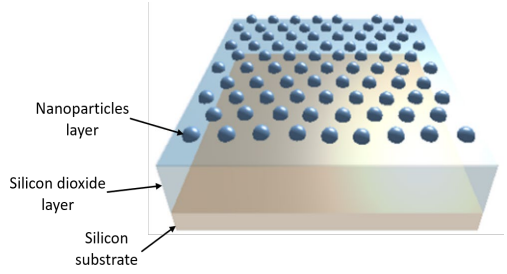


Qdemat, Asma et.al. (2020). "*Self assembled monolayer of silica nanoparticles with improved order by drop casting*". RSC Advances. 10. 18339-18347. 10.1039/D0RA00936A.

2D Ordered Arrays of Ferrimagnetic Cobalt Ferrite nanodots

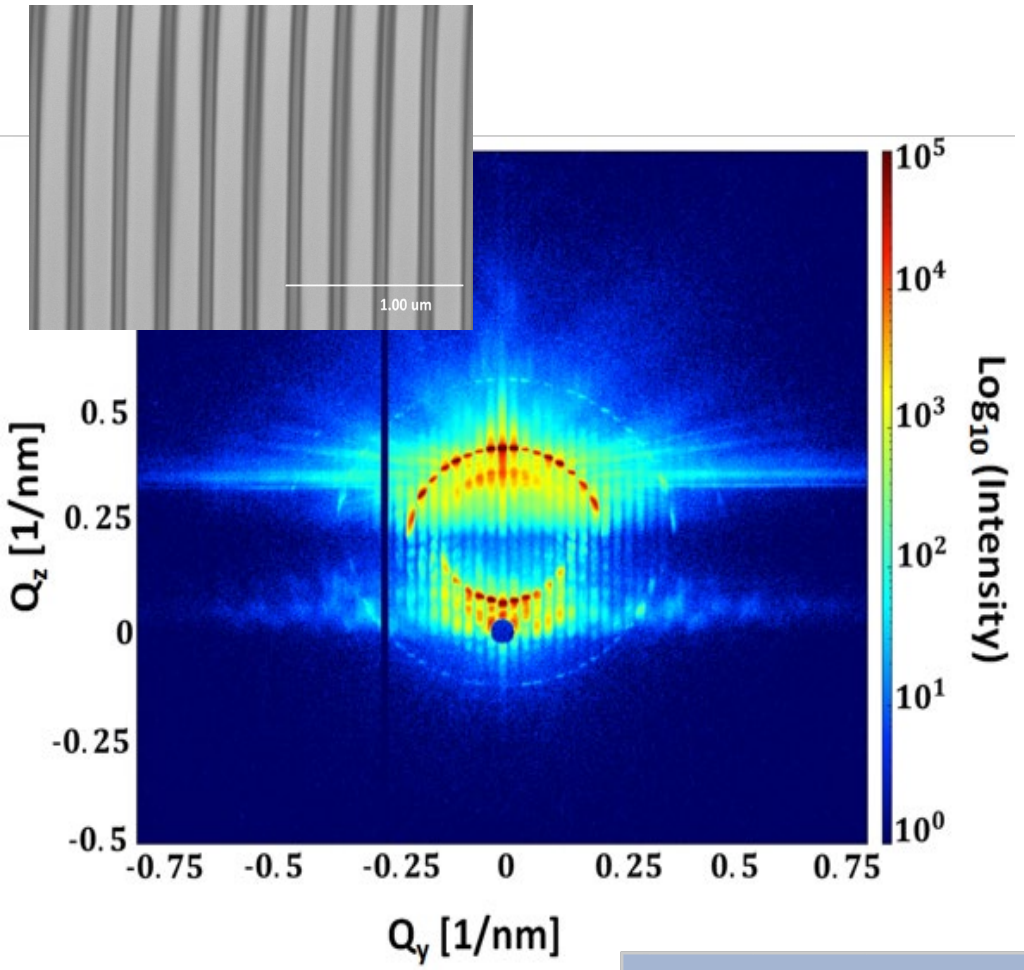


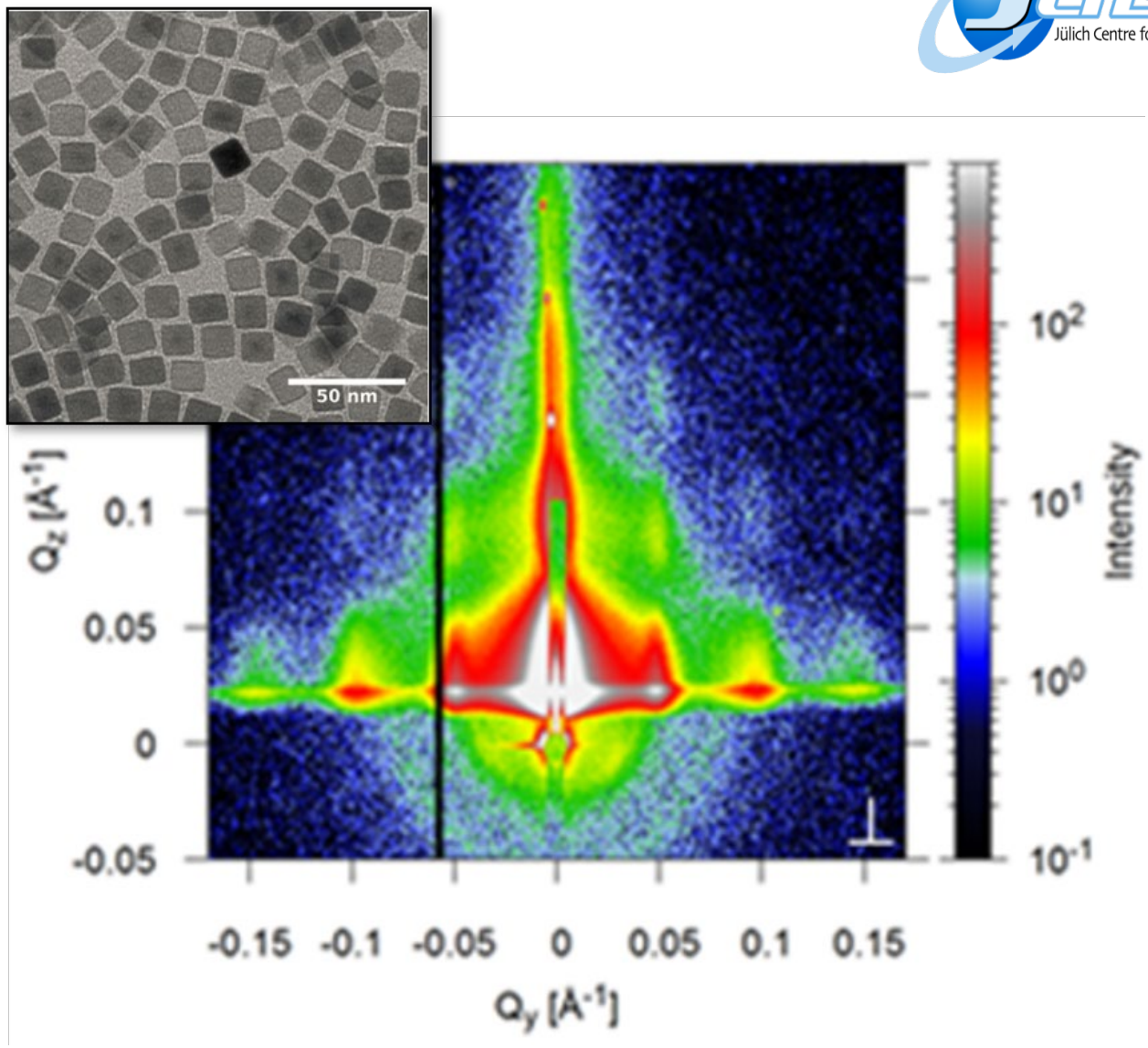




Directed self-assembly of magnetic nanoparticles on patterned substrates

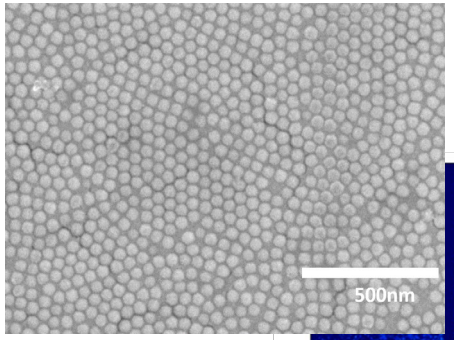
patterned surfaces

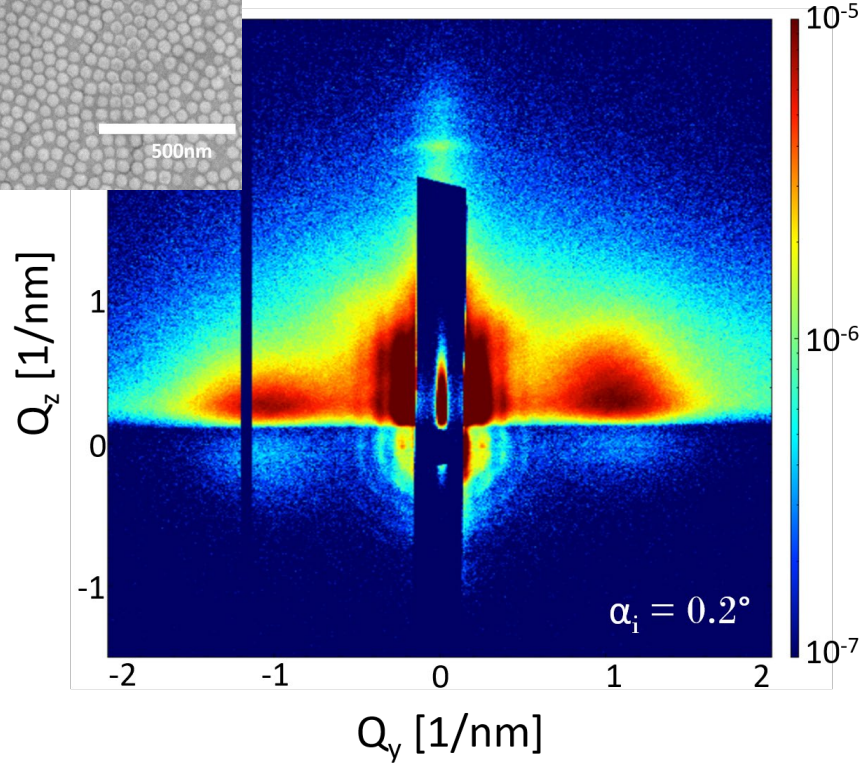


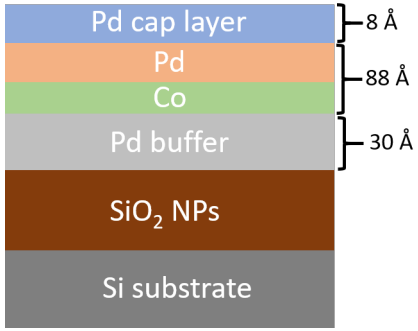


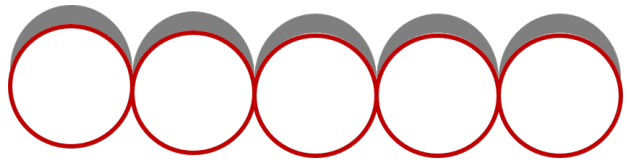
No correlation between the structural arrangement of nanoparticles and the geometry of the trench-patterned substrates

Magnetic multilayers on Silica nanospheres

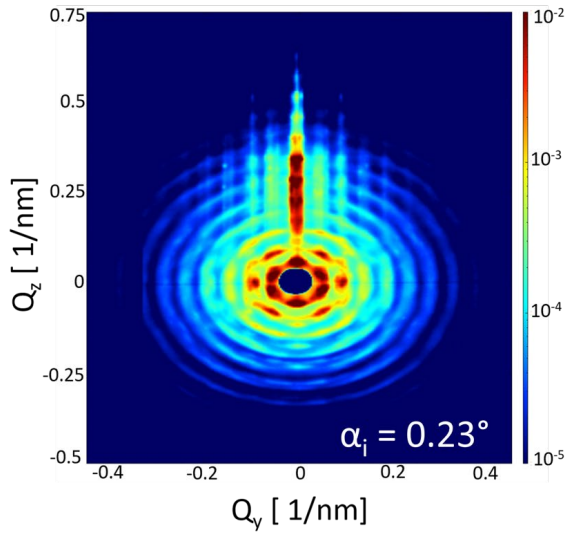


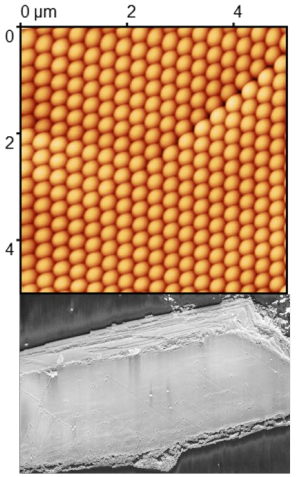


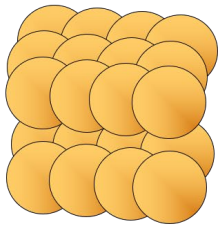


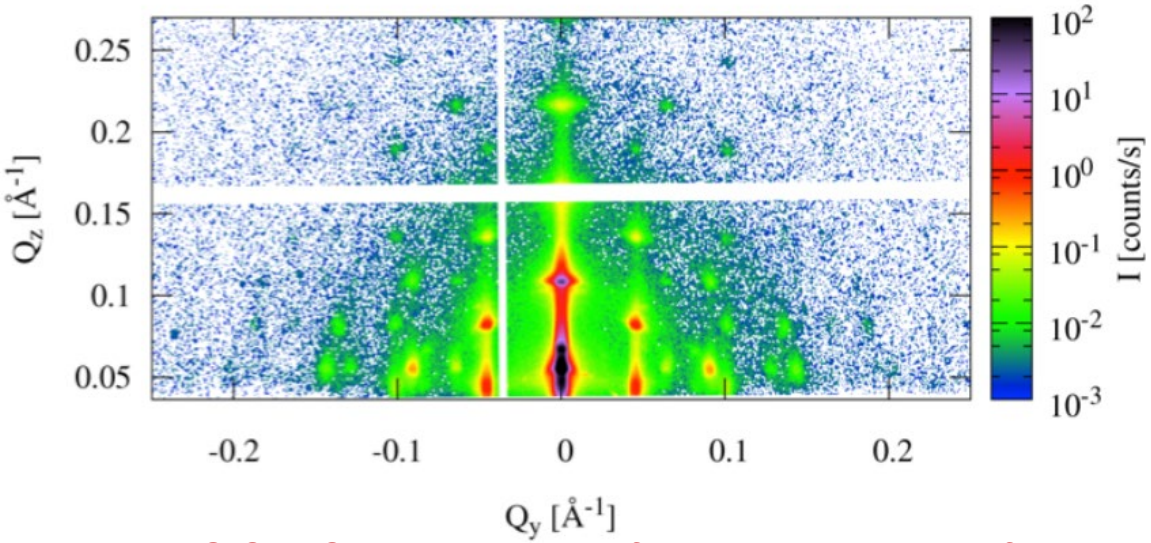


Self-Assembled Mesocrystals of silica Nanospheres







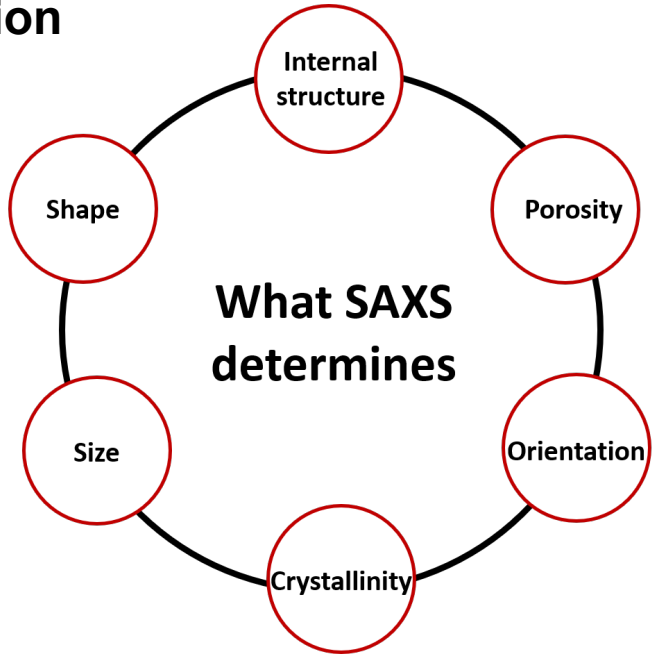


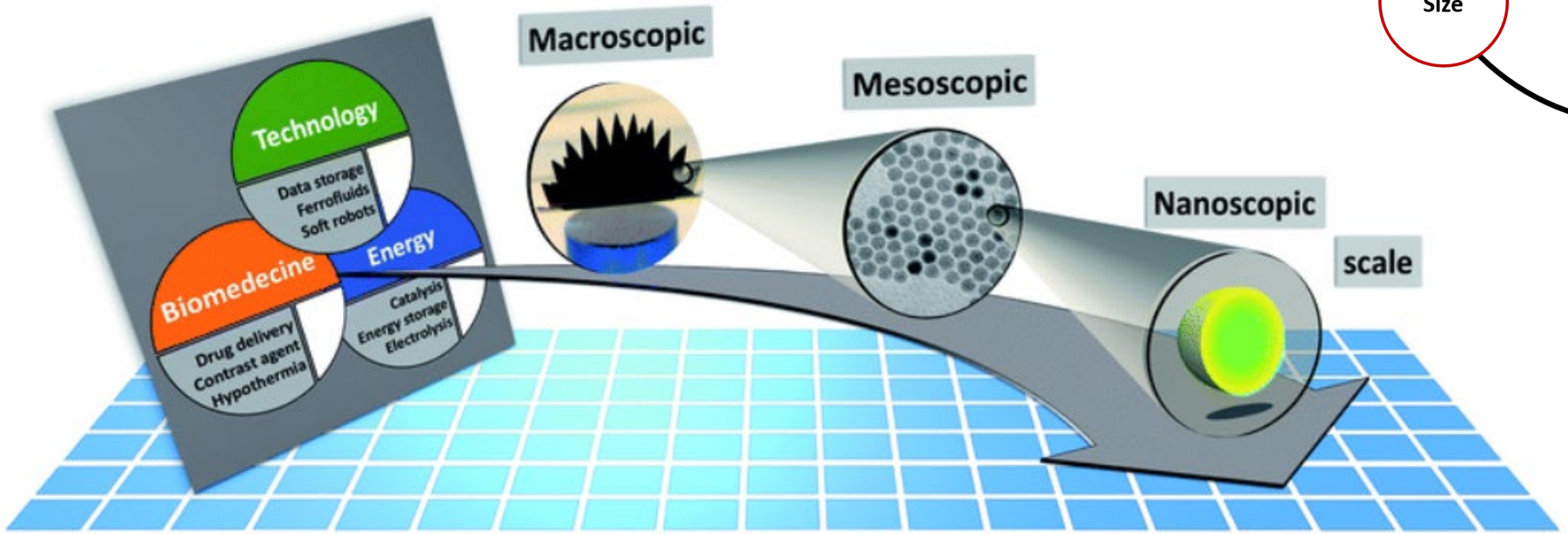
GISAXS measurement from an assembly of magnetic nanoparticles of cubic shape deposited on a substrate

(E. Josten et al. Nanoscale Horizons 5, 1065 (2020))

Summary

- **SAXS / GISAXS: open new possibilities of advanced sample characterization**
- **SAXS / GISAXS : reciprocal space analysis technique**
  - non-destructive structural probe
  - Low sample preparation efforts
  - Yields excellent sampling statistics
    (averages over macroscopic regions to provide information on nanometer scale)



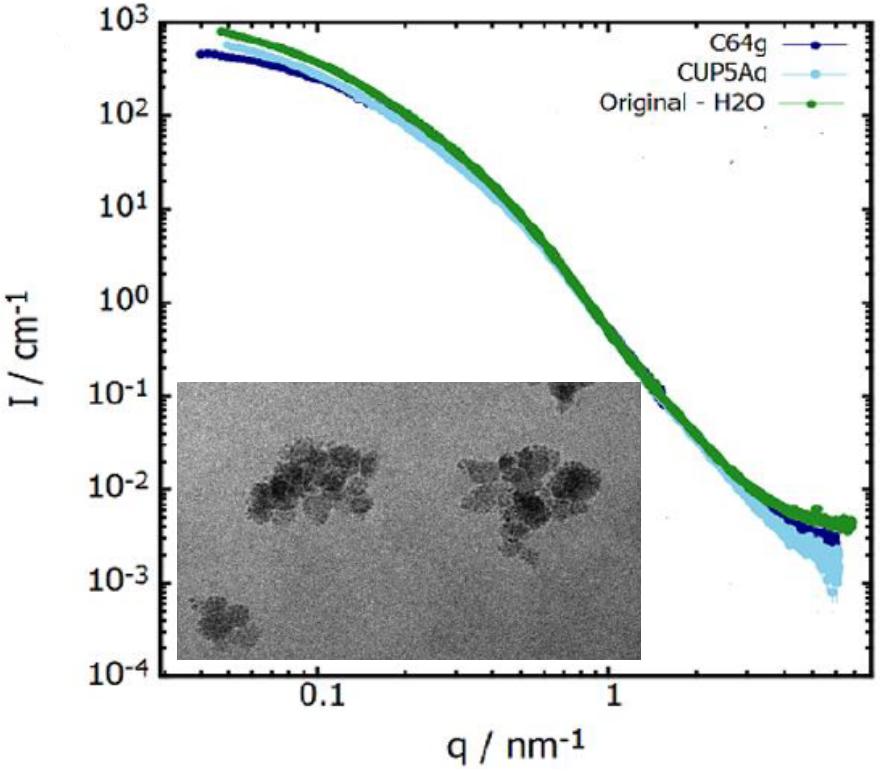


Nanoscale Adv., 2022,4, 1026-1059

PDF @ ID09-MS - SESAME

Amal Atari

structural characterization of citrate coated superparamagnetic iron oxide nanoparticles for magnetically controlled immune therapy



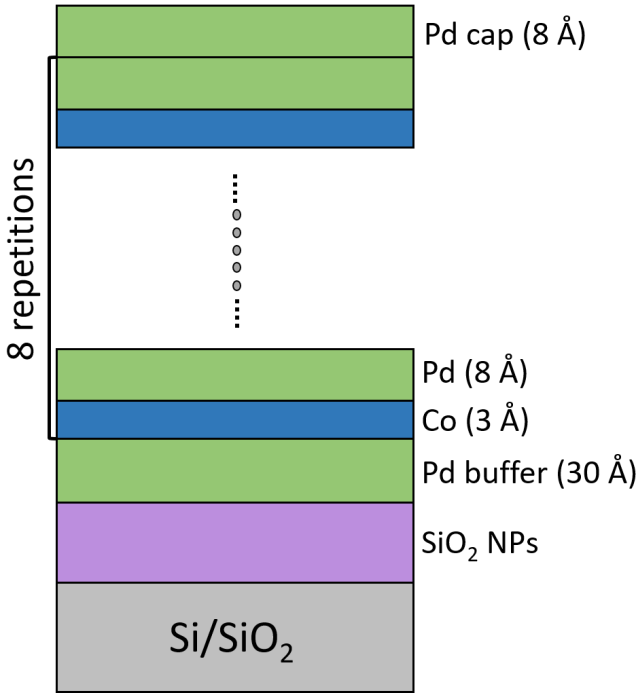
Time-resolved in situ PDF experiments during annealing

Track changes in the crystalline structure of the iron oxide core and determine its composition

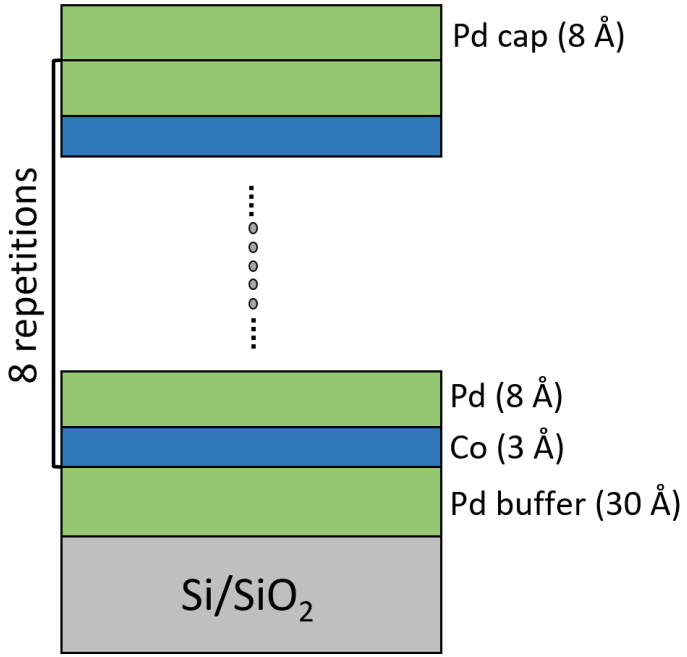
Future plans at SESAME

Tuning shape-imposed anisotropy via magnetic multilayers on self-organized nanospheres

Aim: investigate the influence of using curved surfaces as a substrate on the deposited magnetic thin film properties



Magnetic multilayers on nanospheres



Magnetic multilayers on Si substrate

XMCD @ ID11- HESEB

Element-specific orbital magnetic moments and their anisotropies **using** Co L-edge and Pd M-edge angle-dependent XMCD

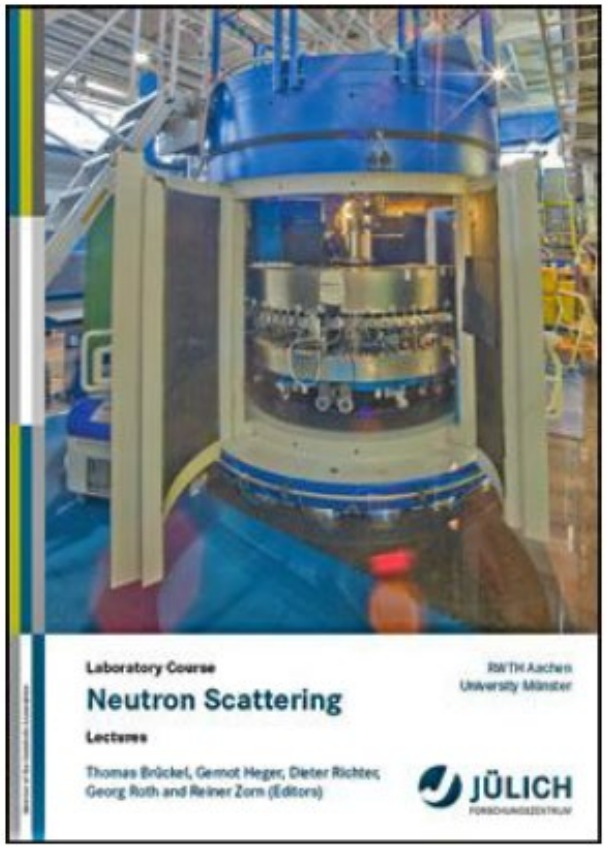
Okabayashi, J.,et al. Sci Rep 8, 8303 (2018). https://doi.org/10.1038/s41598-018-26195-w

Future vision

SAXS / WAXS / GISAXS beamline @ SESAME

25th JCNS Laboratory Course - Neutron Scattering 2023

- 04 - 15 September 2023 Jülich / Garching – Germany



Contact: neutronlab@fz-juelich.de

https://www.fzjuelich.de/jcns/EN/Expertise/ConferencesAndWorkshops/LabCourse/_node.html

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Helmholtz Nanoelectronic Facility, HNF

Electronic Materials (PGI-7)

Institute for Biological Information Processes (IBI-4)

university of groningen

RWTH AACHEN UNIVERSITY

Mitglied der Helmholtz-Gemeinschaft

JÜLICH Forschungszentrum

Thank you for your attention!

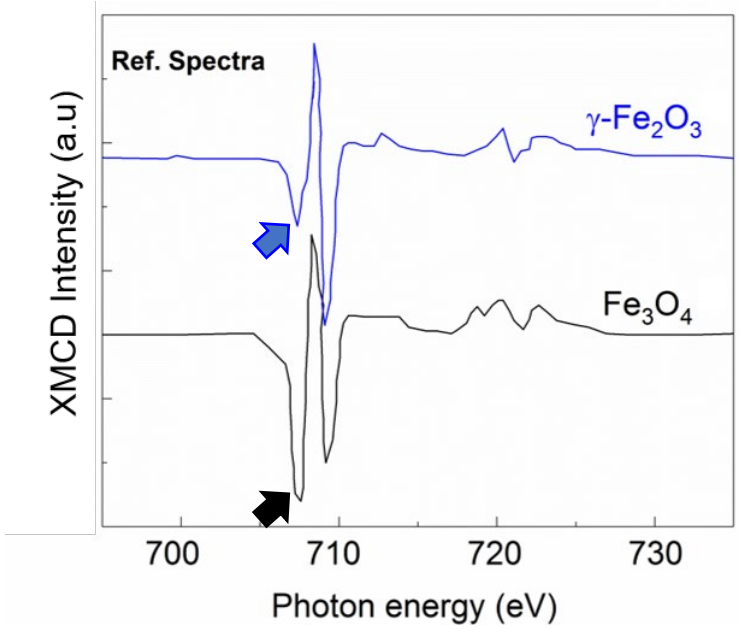
If you want to perform your own SAXS
or GISAXS or WAXS experiment with GALAXI:

Info: https://www.fz-juelich.de/en/jcns/jcns-2/expertise/in-house-x-ray/galaxi

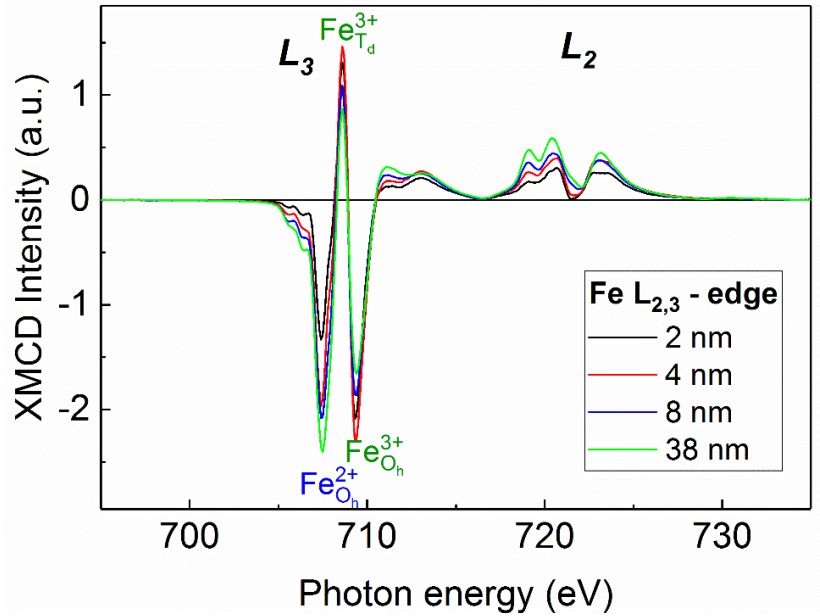
you are also welcome to e-mail me at

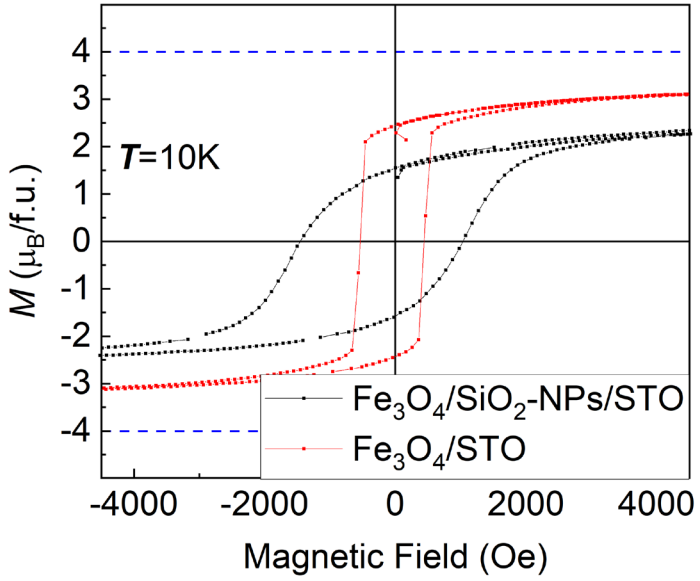
a.qdemat@fz-juelich.de

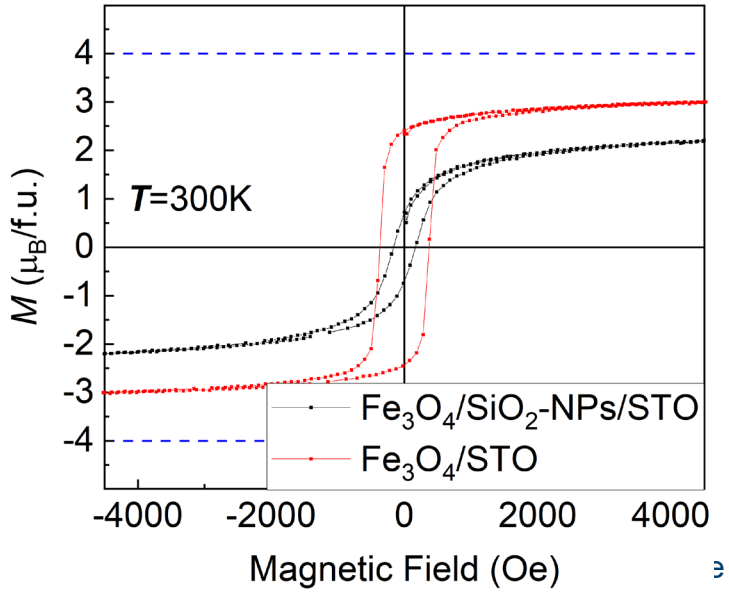
Interface Magnetic Properties (XMCD)

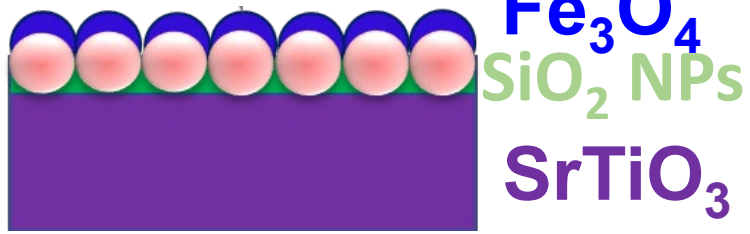


Physica status solidi (b) 215, (1999)797



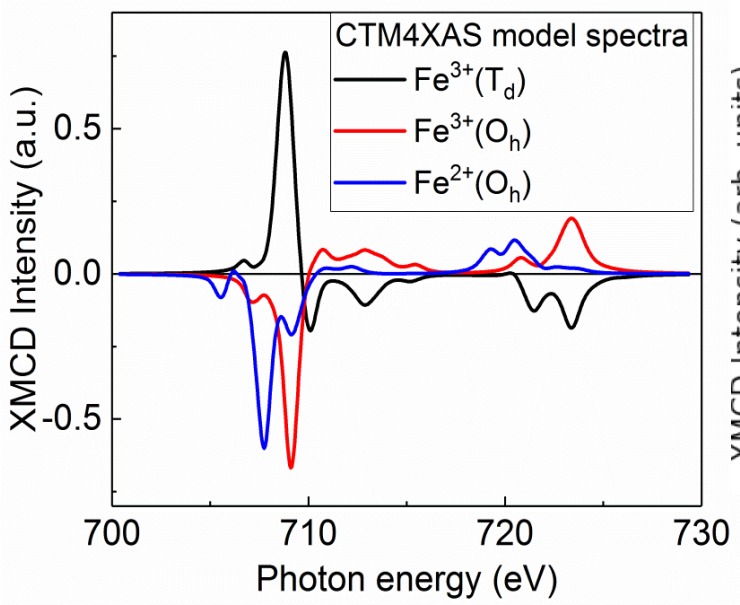


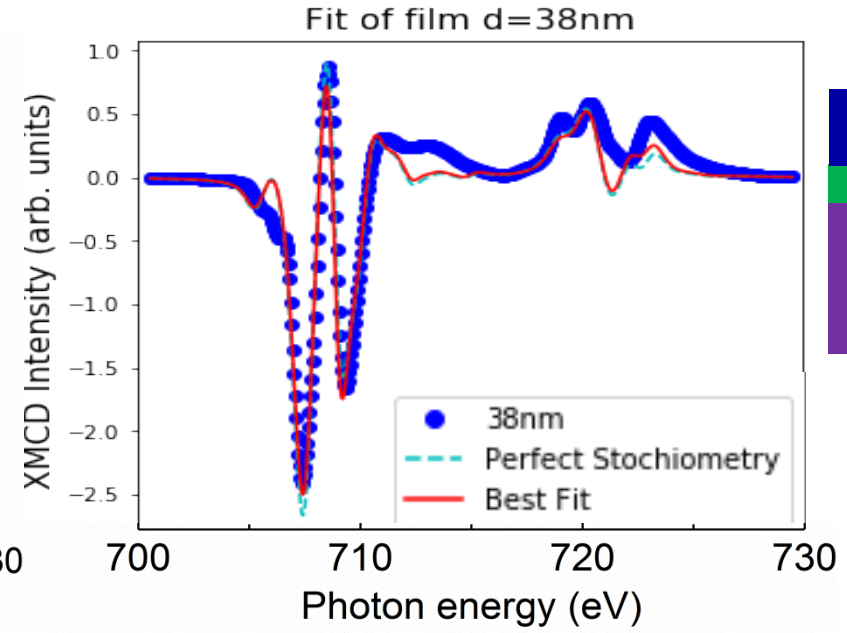




M.H. Hamed et al. ACS Applied Materials & Interfaces, 11(7), 7576, 2019.

Intralayer Thickness (XMCD)

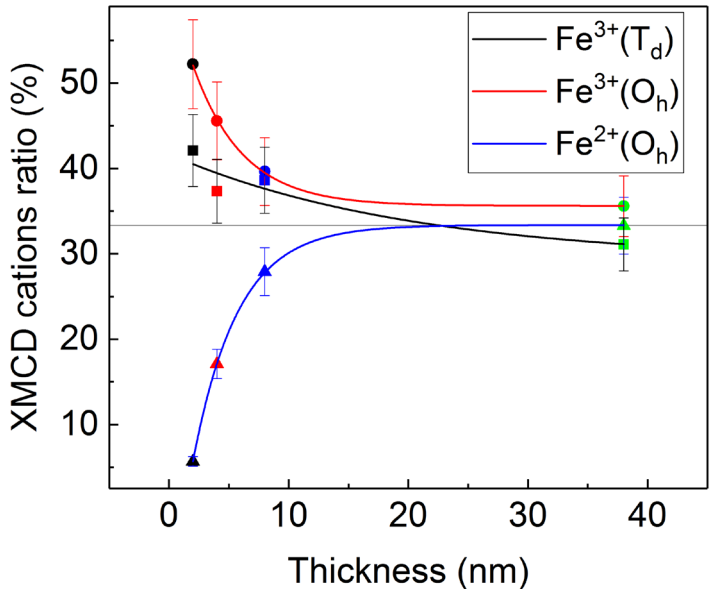


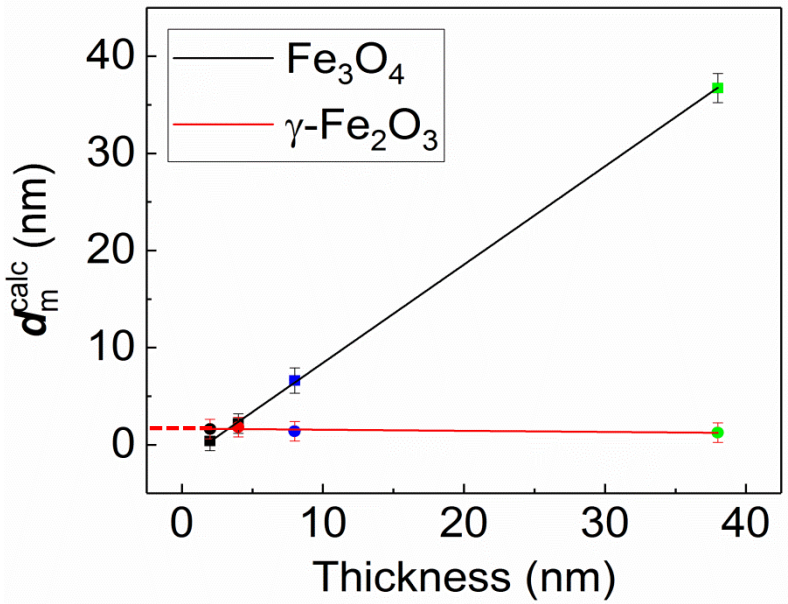


Fe_3O_4

γ-Fe_2O_3

$SrTiO_3$





γ-Fe_2O_3 intralayer
1.3±0.3nm (~ 2u.c.)