Reflectometry at ILL

From 3D to 2D to 1D and back: Why we care…

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Bob Cubitt (cubitt@ill.fr)

P. Gutfreund, *et al.* Journal of Applied Crystallography **51**, 606-615 (2018).
T. Saerbeck, *et al.* Journal of Applied Crystallography **51**, 249-256 (2018).
R. Cubitt, *et al.* Journal of Applied Crystallography **48**, 2006-2011 (2015).
A. Vorobiev, *et al.* Neutron News **26**, 25-26 (2015)
A. Devishvili, *et al.* Review of Scientific Instruments **84**, 025112 (2013).
R. Campbell, *et al.* Eur. Phys. J. Plus **126**, 107 (2011).

The ILL Reflectometers:

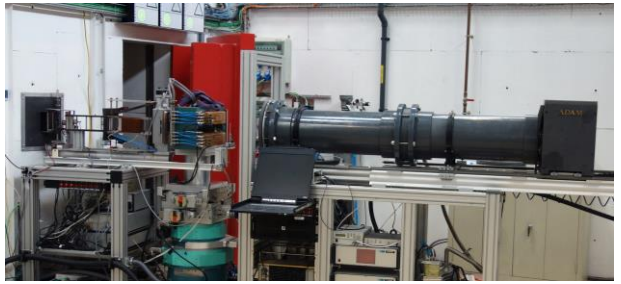
Figaro

- Time-of-flight
- Horizontal sample
- Reflection Up+Down
- Flexible resolution

D17

- Time-of-flight
- Monochromatic
- Vertical sample
- Polarized
- Flexible resolution

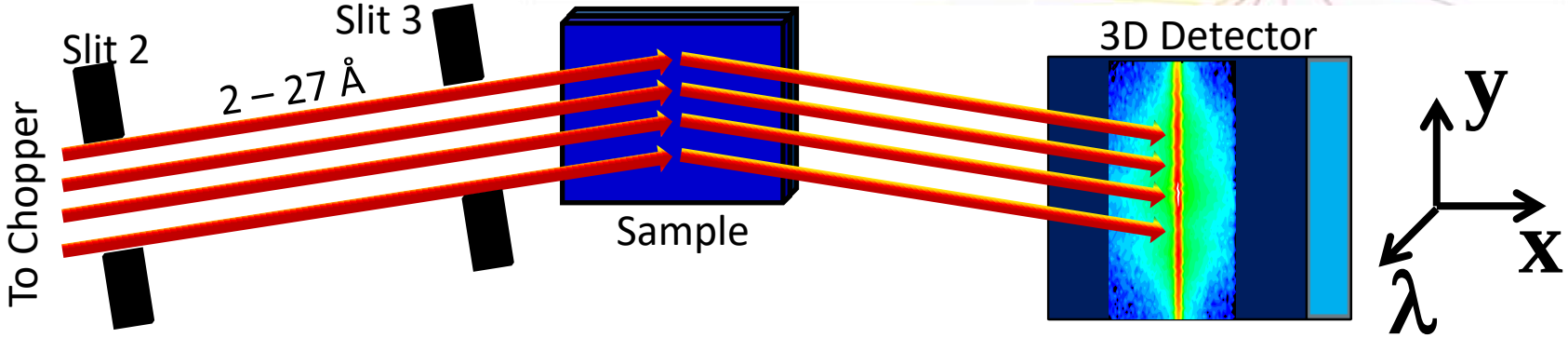
SuperAdam

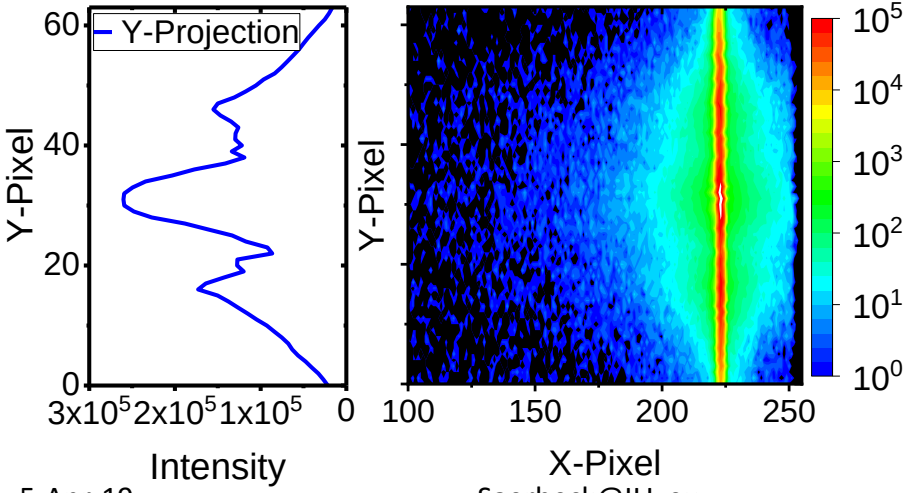


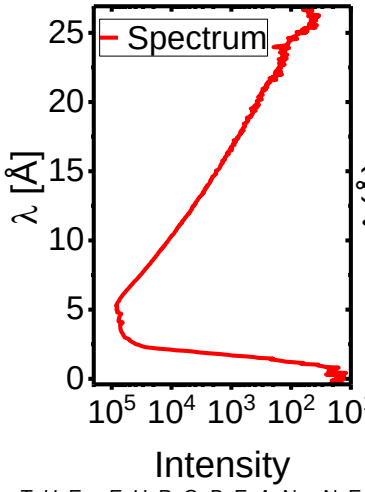
- Monochromatic
- Vertical sample
- Polarized
- High resolution or high intensity

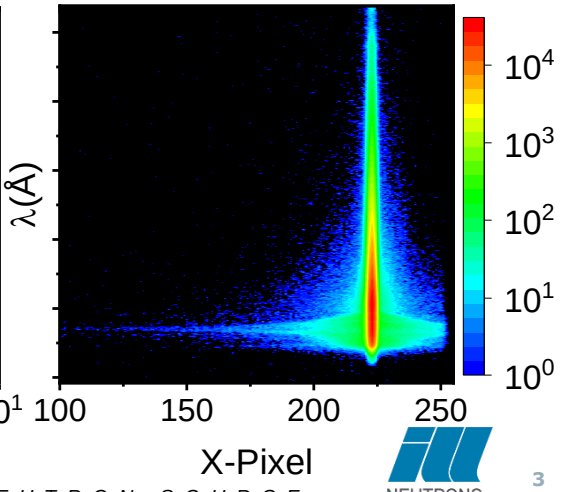
- Science: Soft matter, biology, biophysics/biochemistry, electrochemistry, materials science, magnetism, instrumentation

Time-of-Flight on 2D Detector

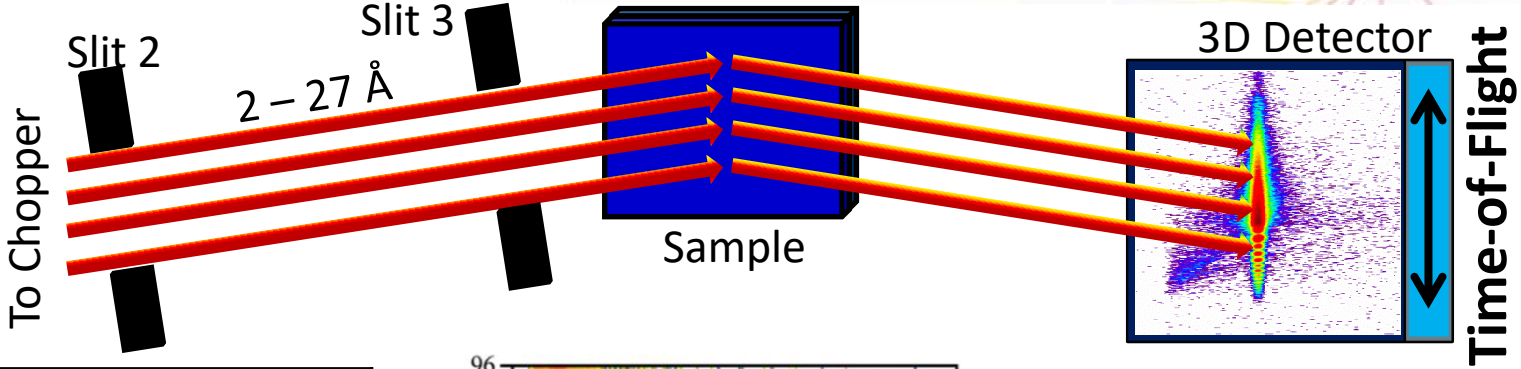


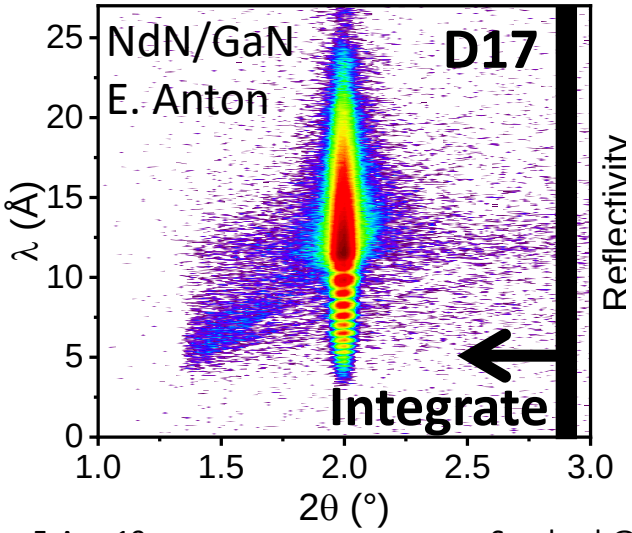


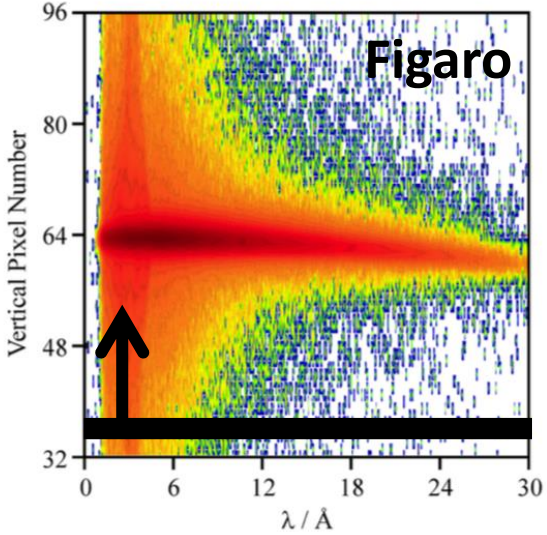


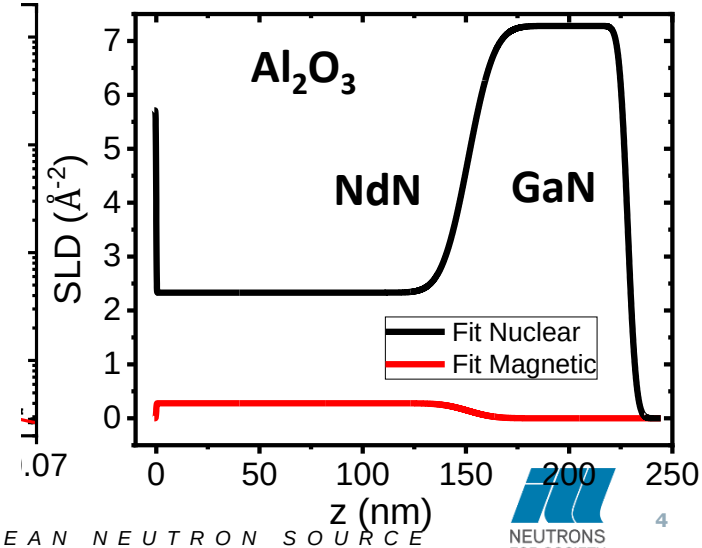


Time-of-Flight on 2D Detector



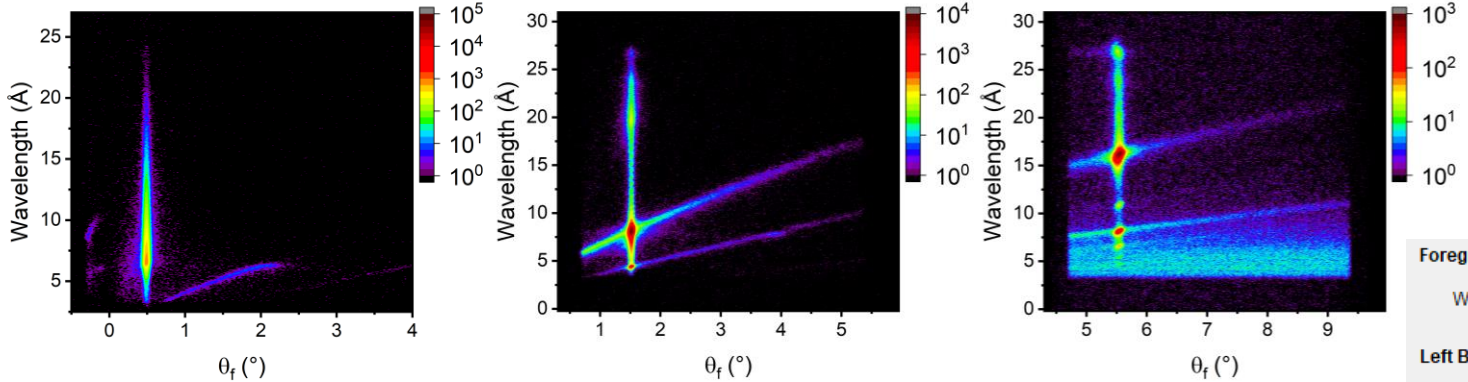


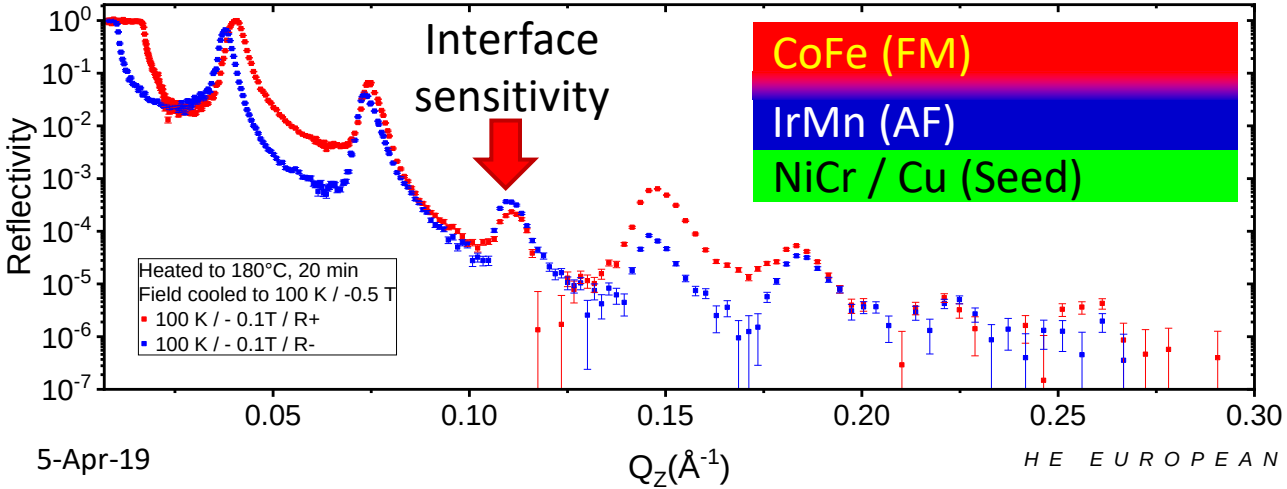




Challenge: Background Subtraction

Seed stabilized exchange bias multilayer (G.V. Fernandez, K. O'Grady; York)

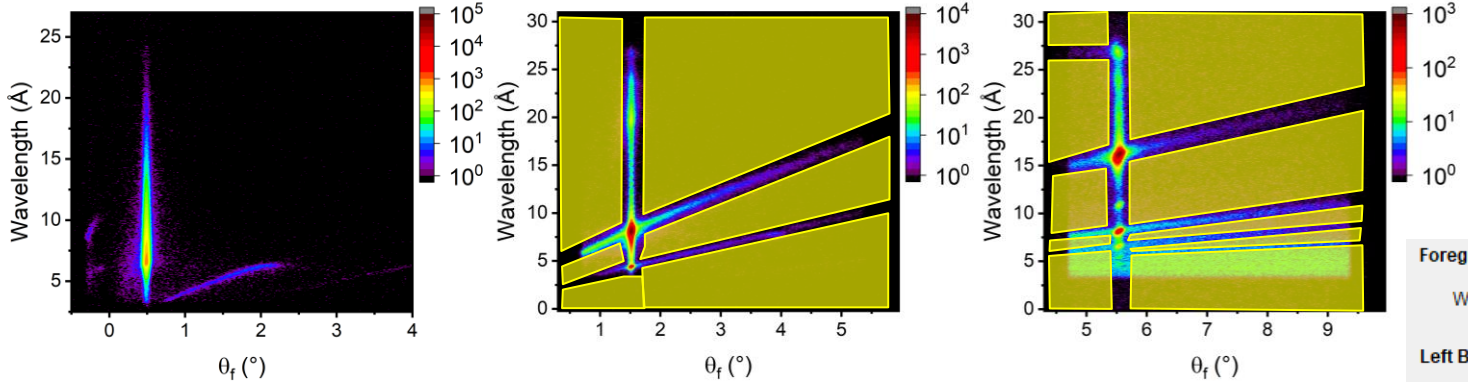


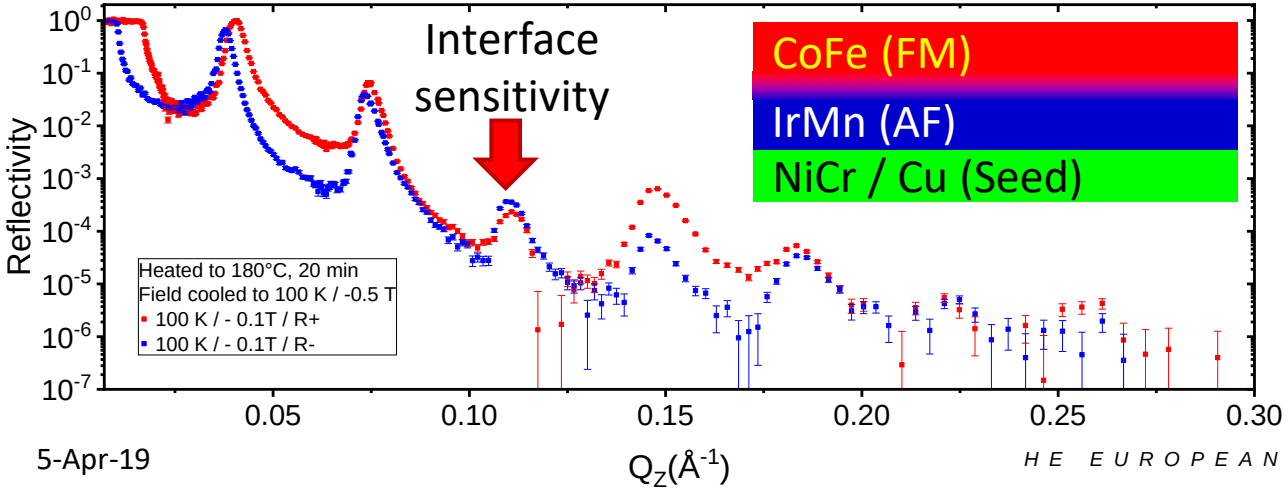


Foreground (pixel)
Width Angle 1 6,5
Left Background (pixel)
Width Angle 1 10
Offset Angle 1 100
Right Background (pixel)
Width Angle 1 0
Offset Angle 1 5
Lambda
Min Angle 1 4.00000
Max Angle 1 14.0000
Grouping Angle 1 0.500000

Challenge: Background Subtraction

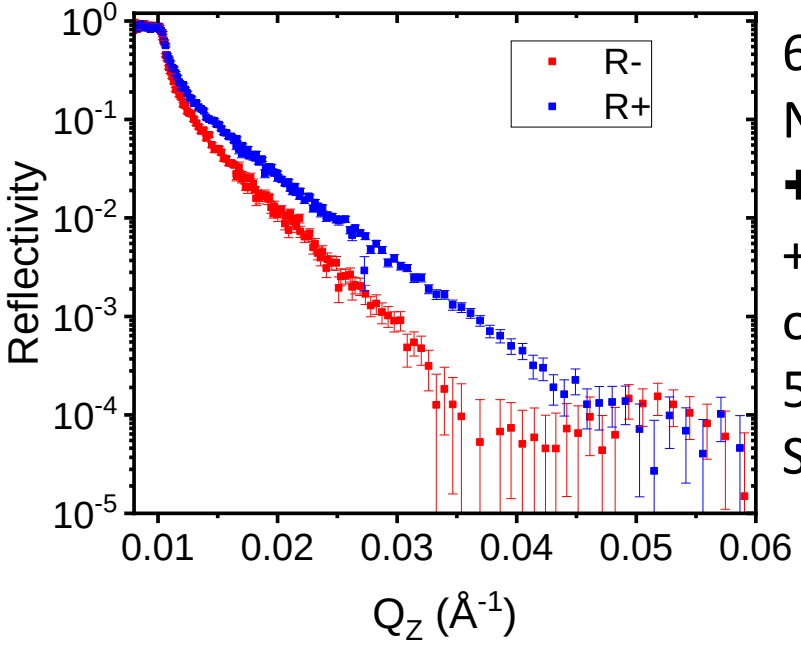
Seed stabilized exchange bias multilayer (G.V. Fernandez, K. O'Grady; York)





Foreground (pixel)
Width Angle 1 6,5
Left Background (pixel)
Width Angle 1 10
Offset Angle 1 100
Right Background (pixel)
Width Angle 1 0
Offset Angle 1 5
Lambda
Min Angle 1 4.00000
Max Angle 1 14.0000
Grouping Angle 1 0.500000

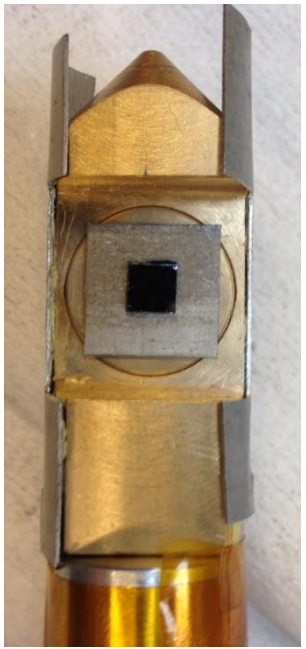
Challenge: Small samples/volume



6 nm Au
Nanoparticles
+ 1 nm Co
+ 2 nm CoO
on
5 mm × 5 mm
Substrate

Measure the Blue:

Co nanostructures
(K. Temst, M. Van Bael; Leuven)



7 T Vertical
2 – 340 K
50 mK

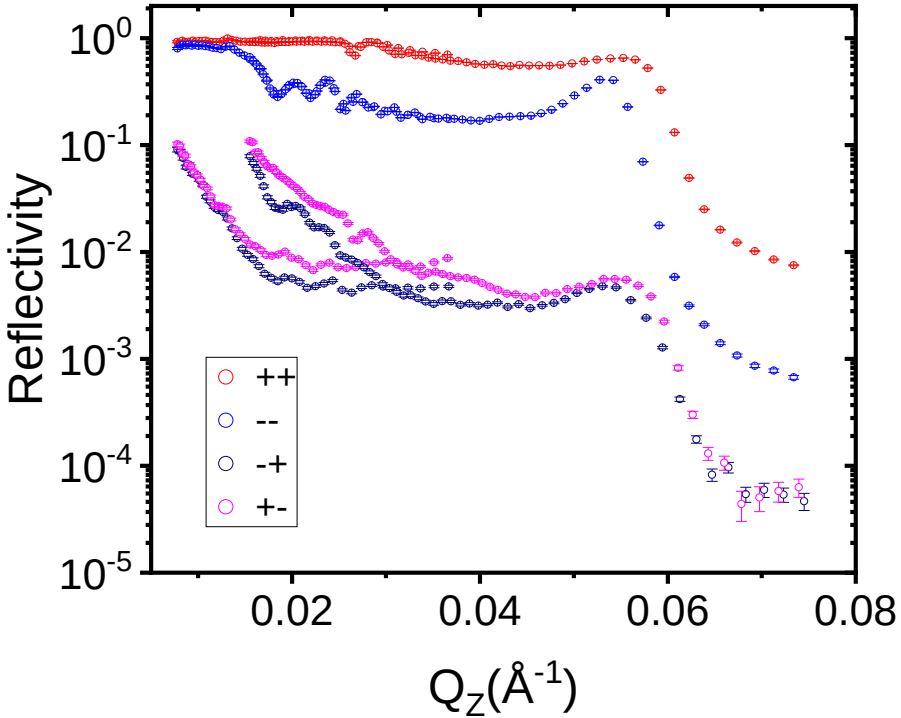
— 5 mm

J.K. Jochum *et al.*, JMMM, accepted (Mar 2019)

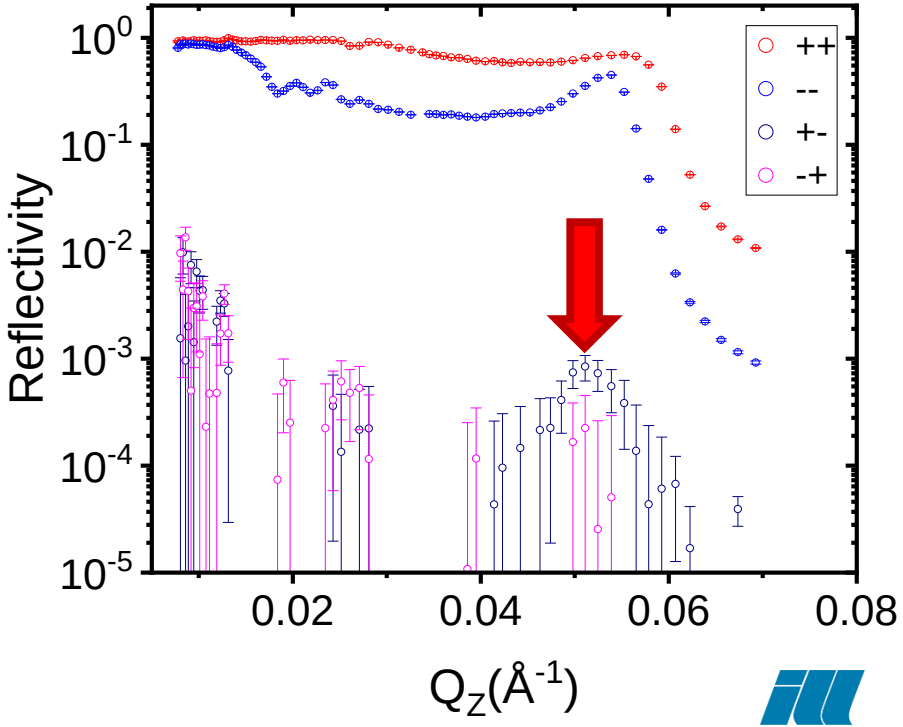
Challenge: Polarization efficiency

Badly compensated FeTi supermirror

Semi raw data



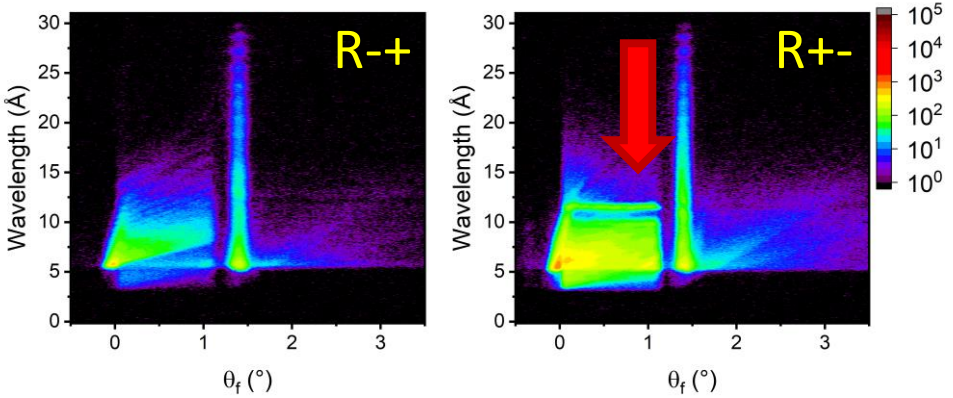
Corrected data

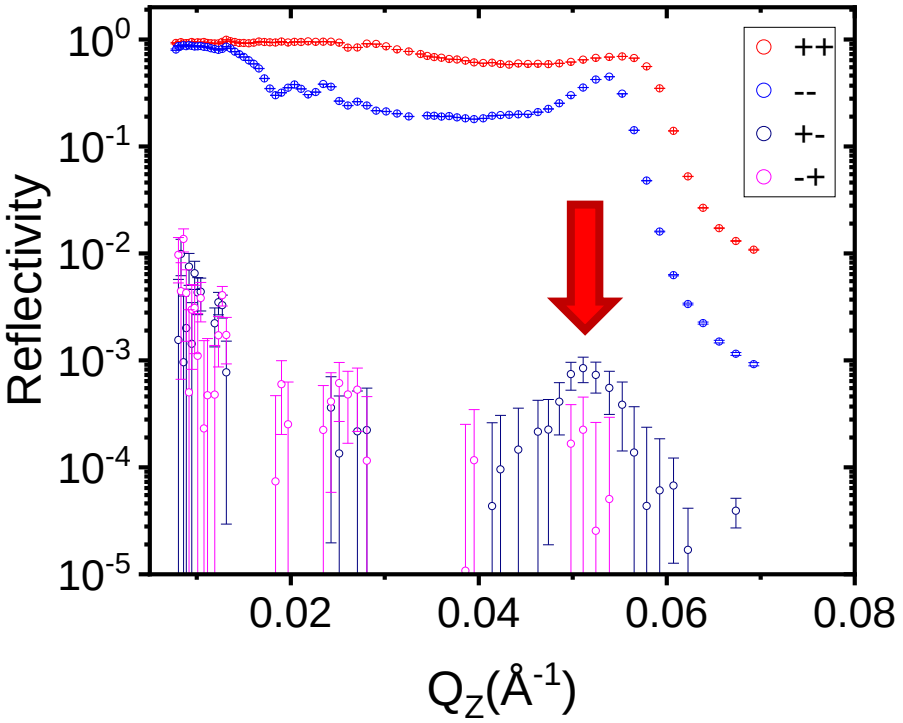


Challenge: Polarization efficiency

Badly compensated FeTi multilayer

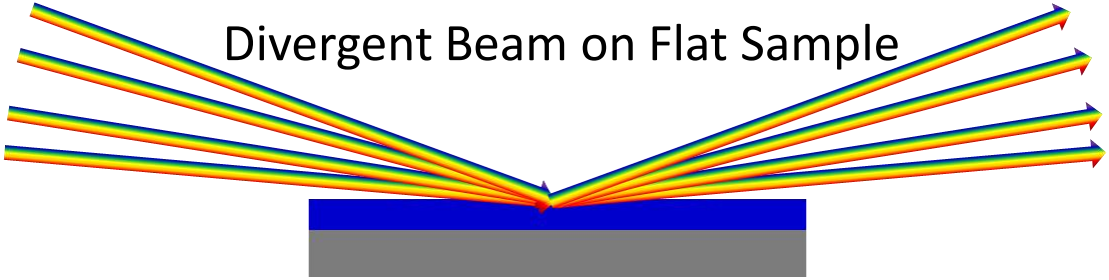
Background from scattering



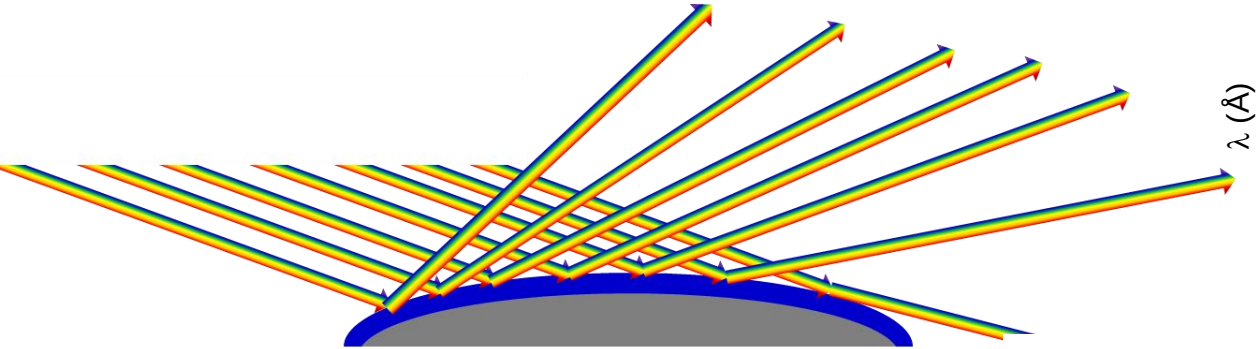


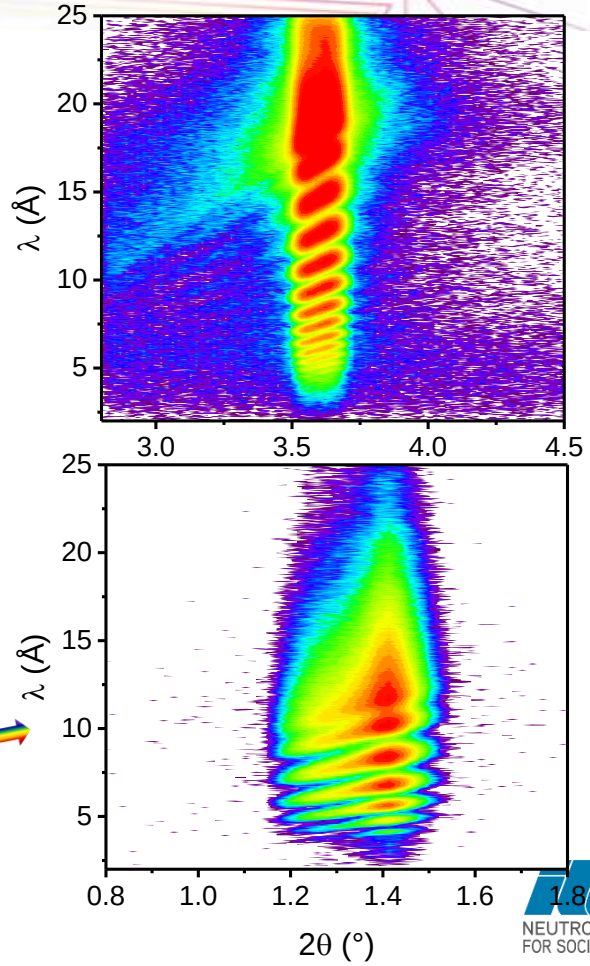
Challenge: Diffuse Reflections

Mirror Reflection of
Divergent Beam on Flat Sample



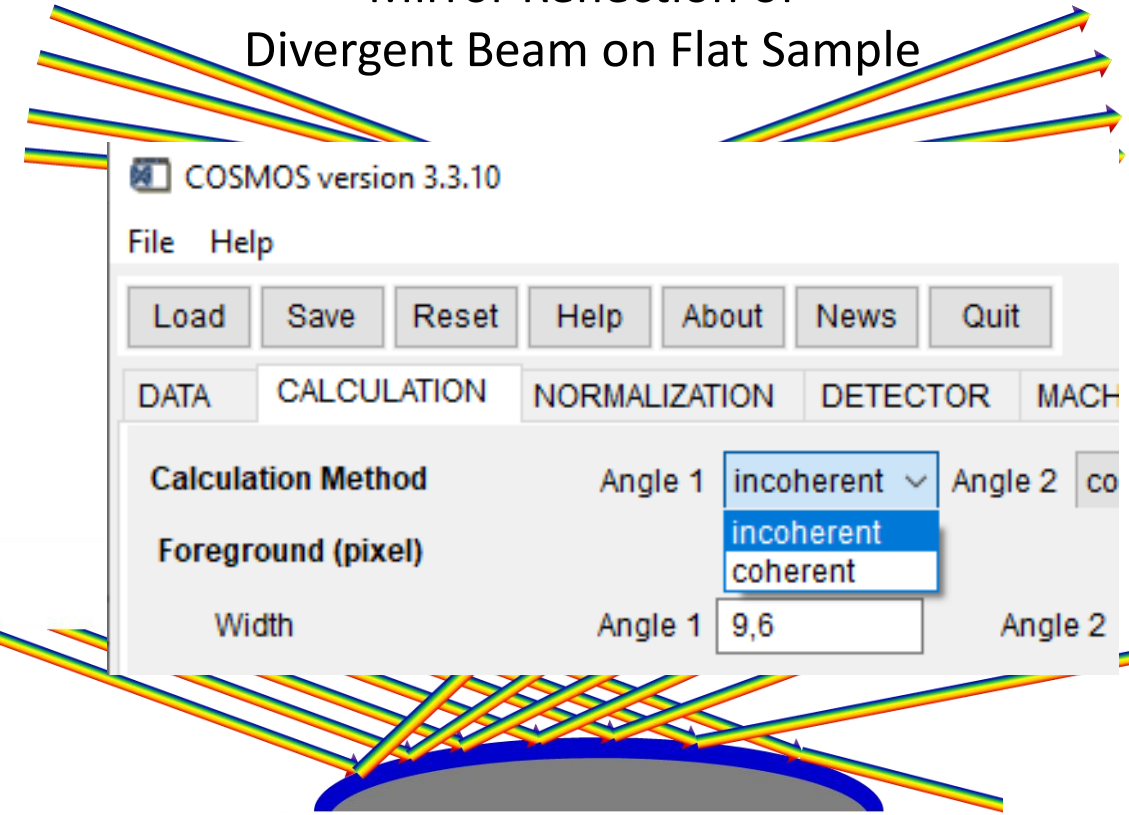
Reflection of
Collimated Beam on Curved Sample

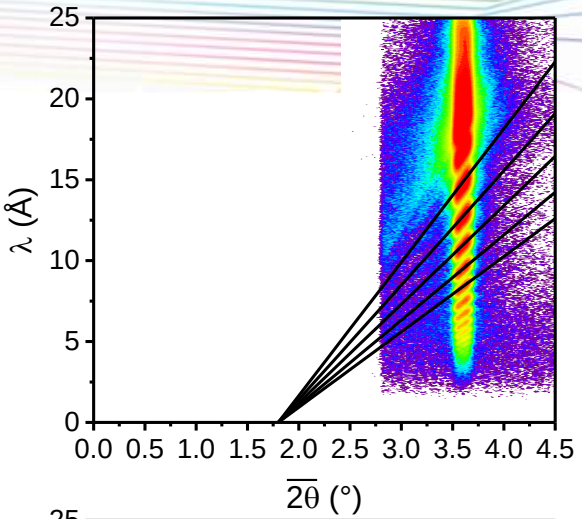


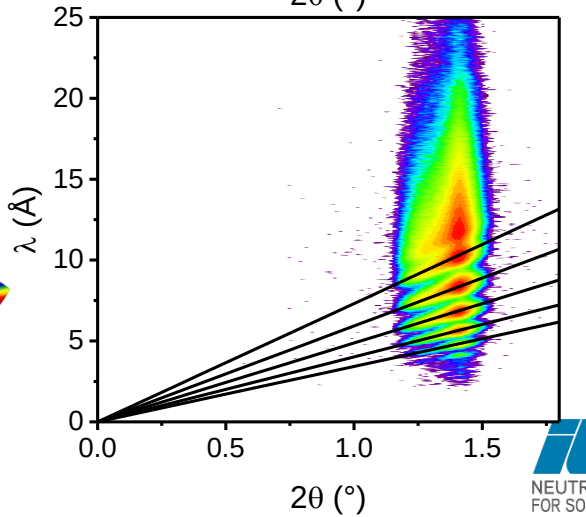


Diffuse Reflections

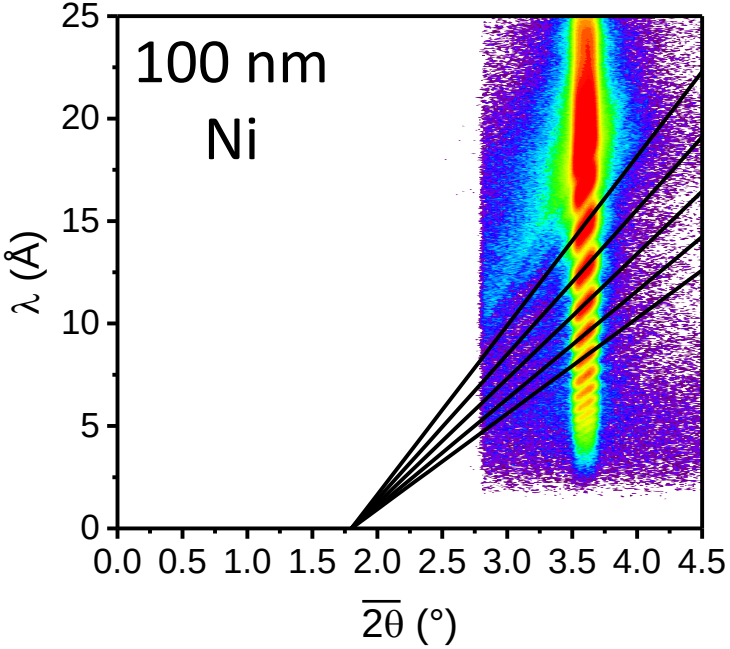
Mirror Reflection of
Divergent Beam on Flat Sample





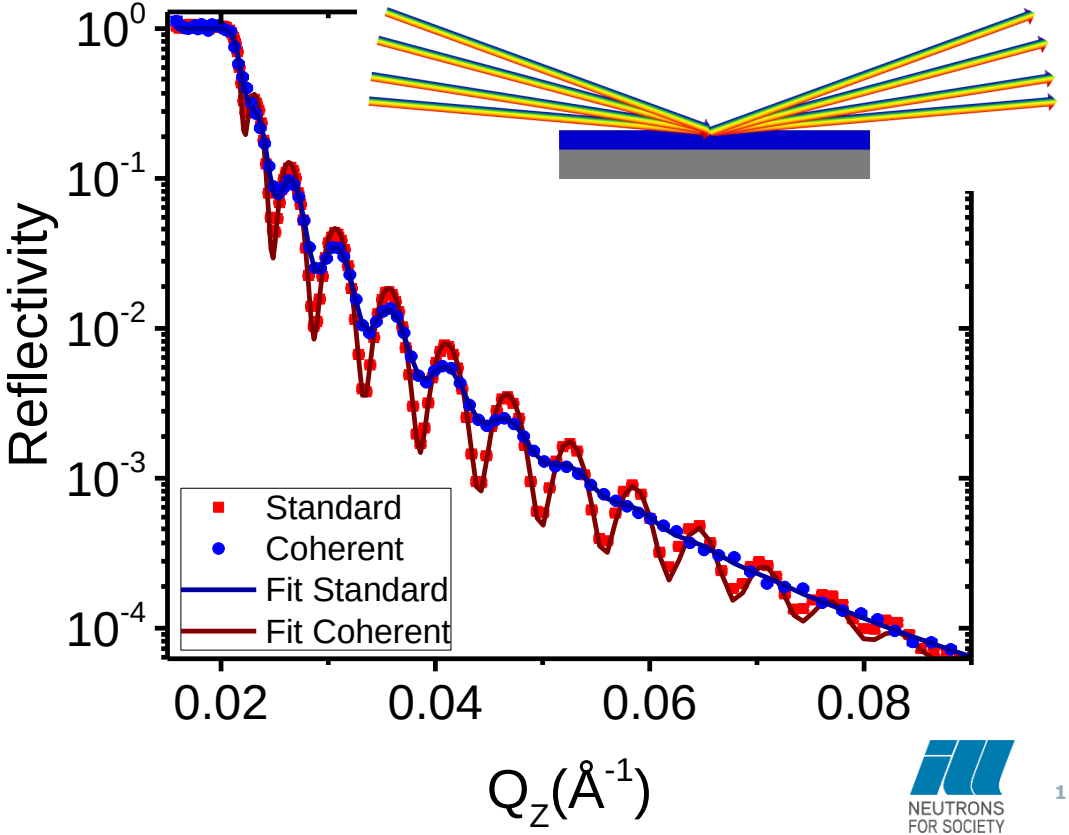


Divergent Beams: Fast single shots

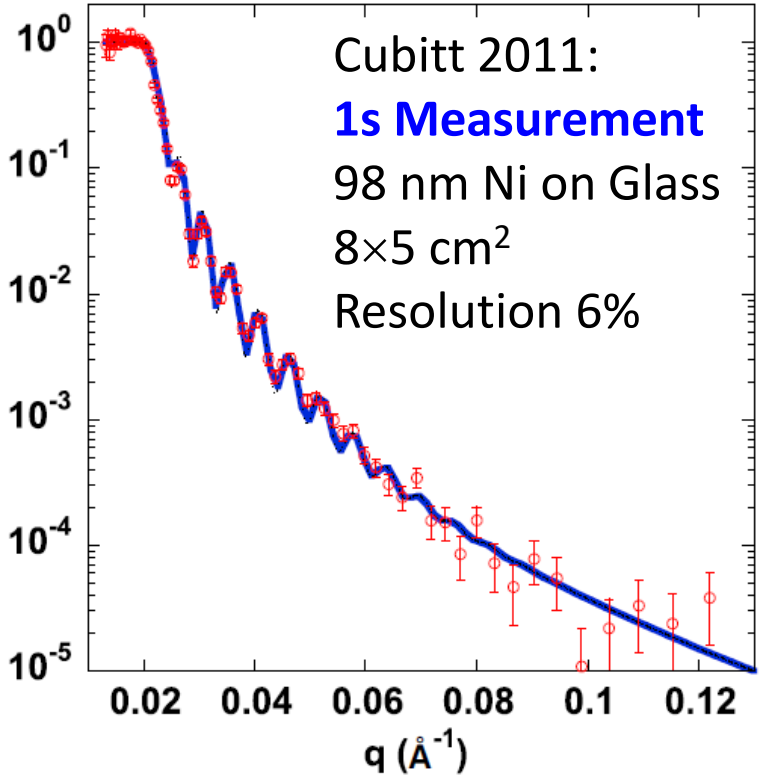


Recovery of **resolution** and
flux increase
with **coherent method**

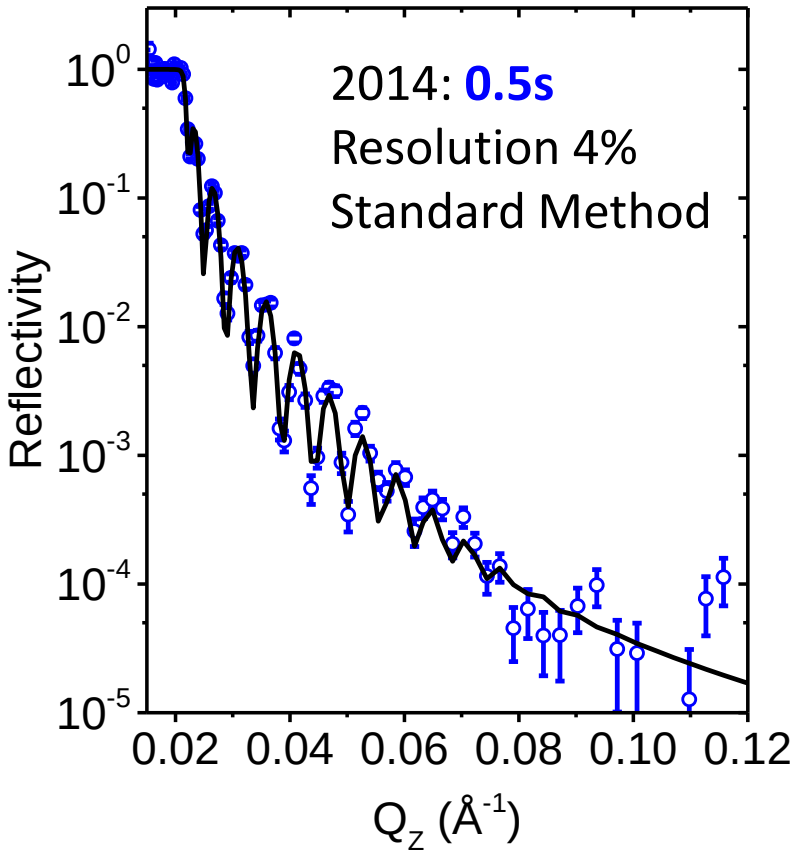
Requirement: Flat sample, no off-specular scatter



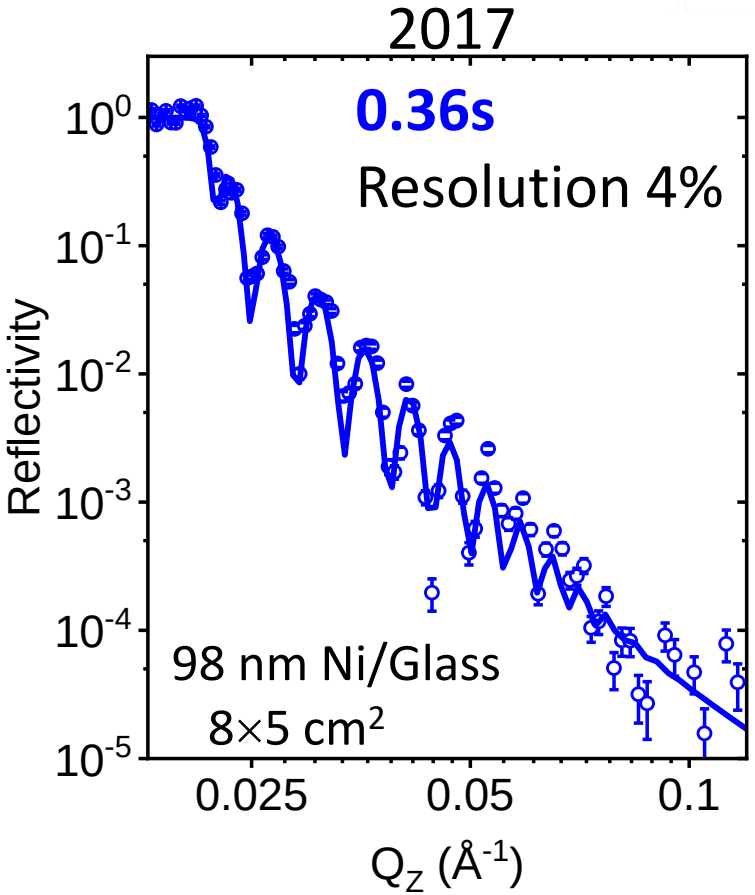
Single Shot Kinetics:

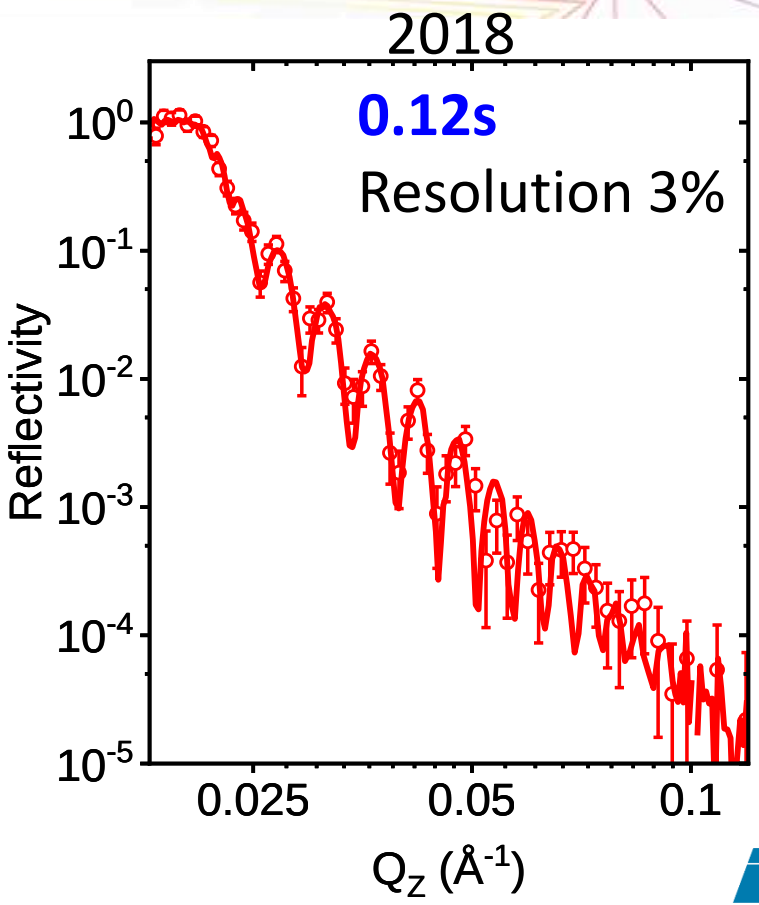


Cubitt & Stahn Eur. Phys. J. Plus (2011)

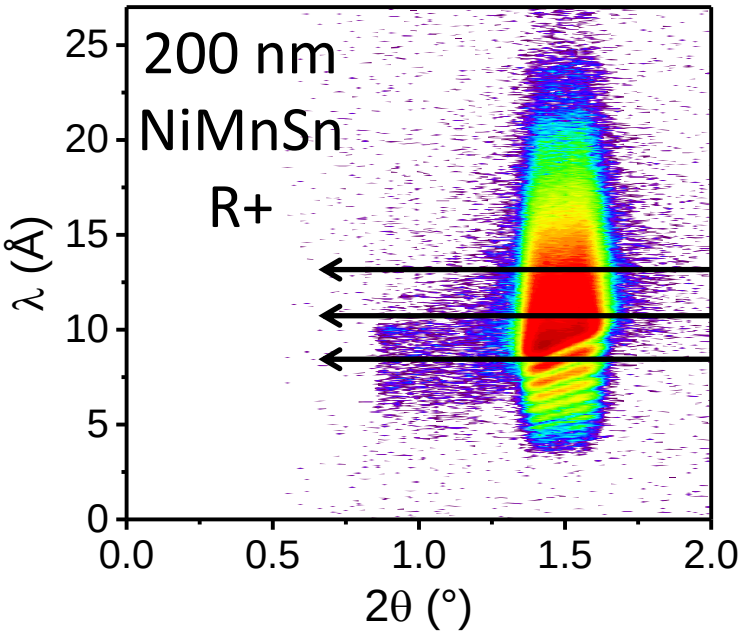


Single Shot Kinetics

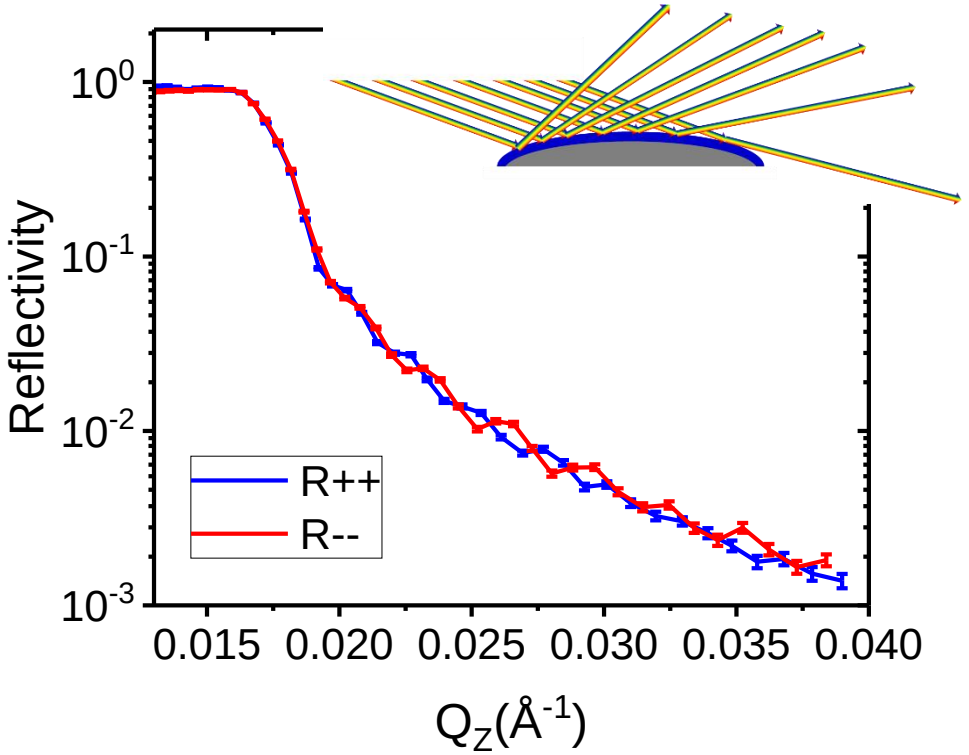




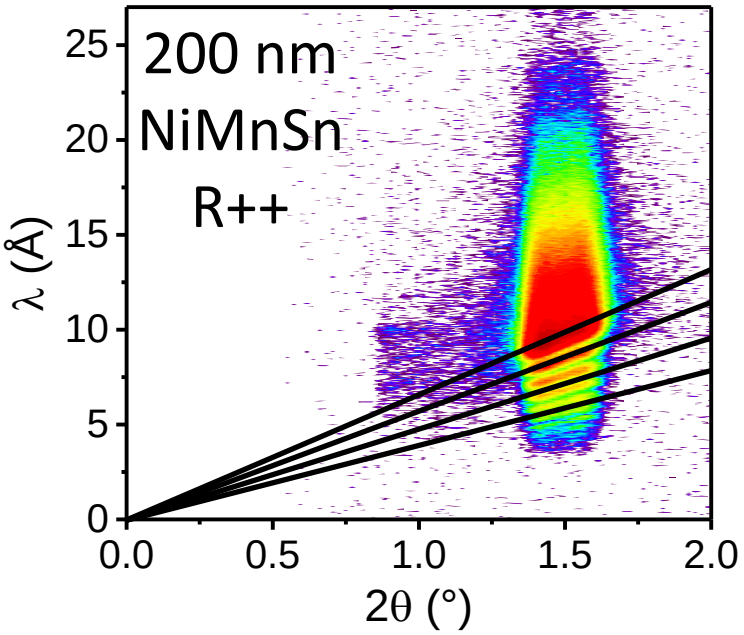
Diffuse Reflections: Bent Samples



Full **loss** of **resolution** (fringes) and **magnetic contrast**

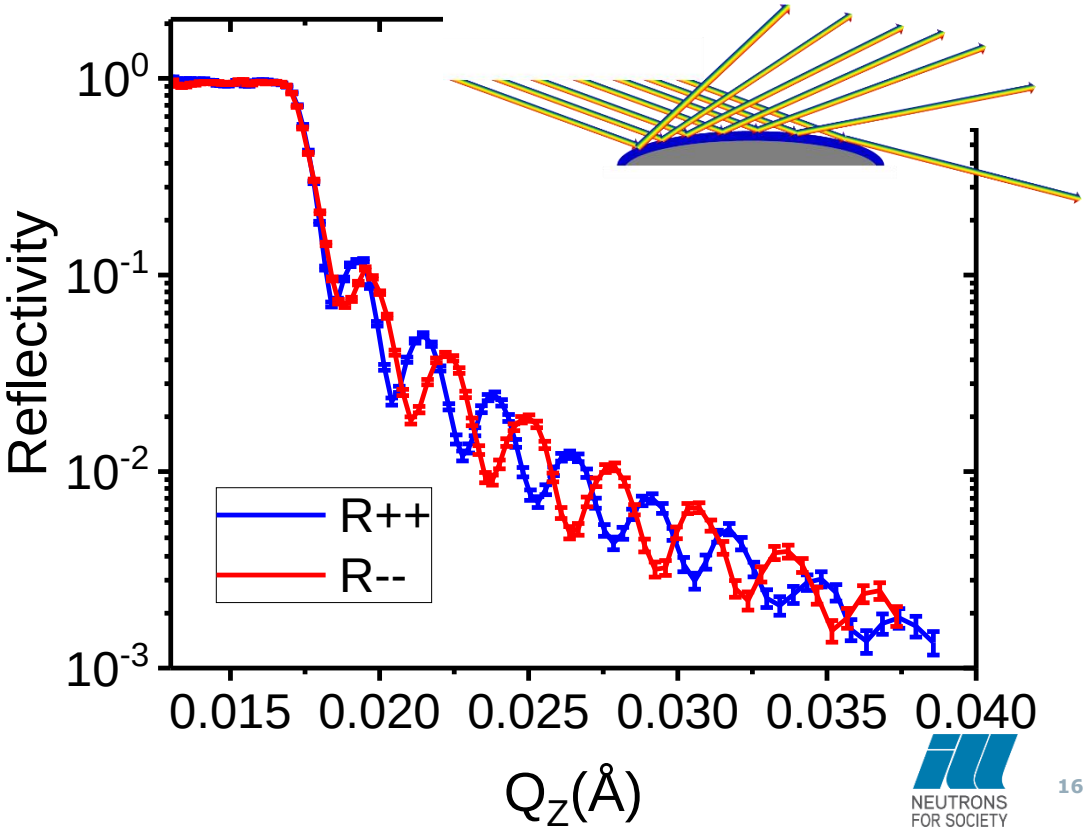


Diffuse Reflections: Bent Samples



Full **recovery** of **resolution** and **contrast** with **coherent method**

Requirement: Collimated beam, no off-specular



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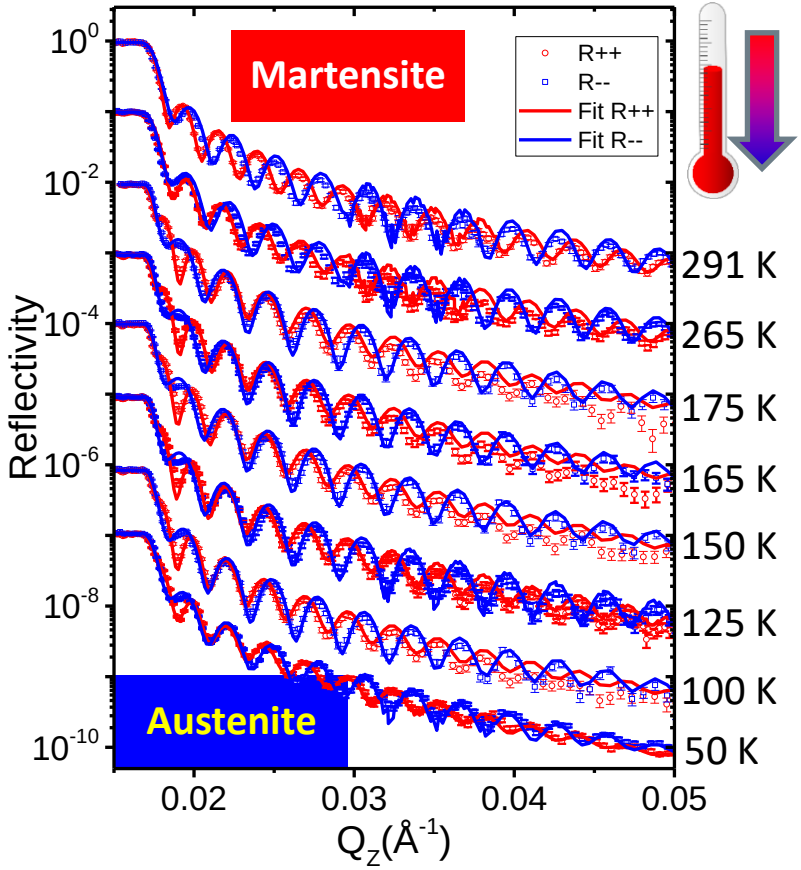
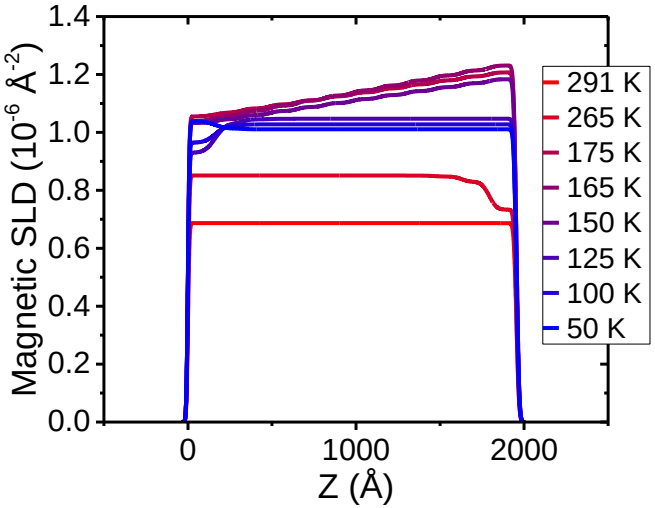
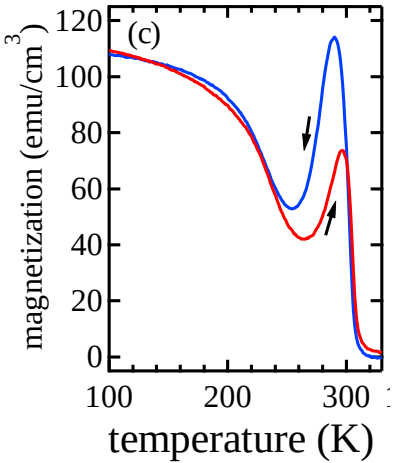
Magnetic Gradient in NiMnSn: PNR



Gradient transition Heusler alloy
(A. Hütten; Bielefeld)

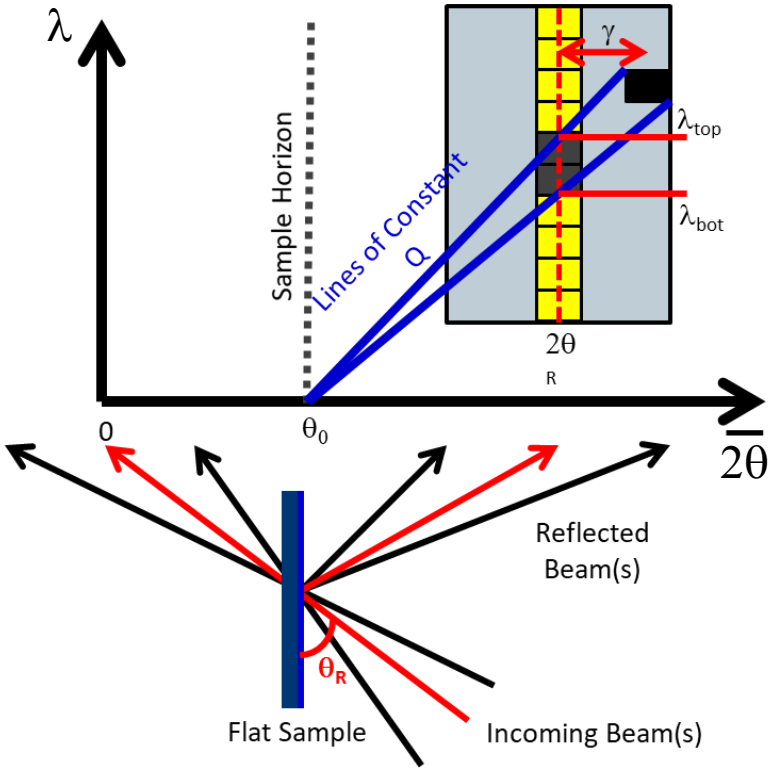
$Ni_{52}Mn_{36}Sn_{12}$ ▶ ▶ ▶ $Ni_{50}Mn_{34}Sn_{16}$



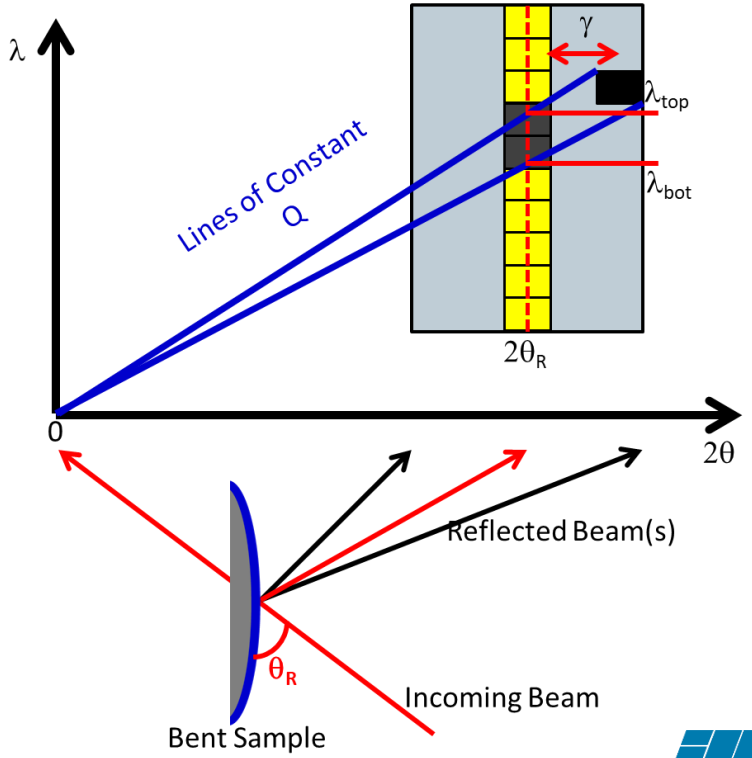


Diffuse Reflections:

Divergent beam, flat sample



Collimated beam, bent sample



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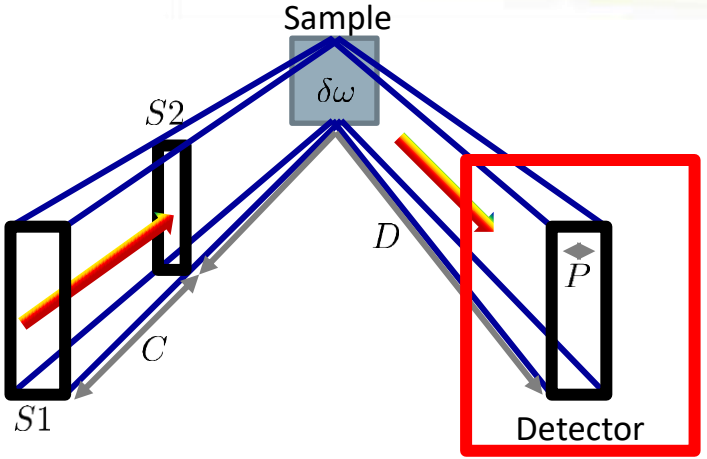
Full Resolution Treatment

R. Cubitt, T. Saerbeck, R. Campbell,
R. Barker, P. Gutfreund,
J. Appl. Cryst. **48**, 2006 (2015)



Divergent Beam on Flat Sample:

$$(\delta\theta)^2 = \begin{cases} (S1/C)^2 + (S2/C)^2 & \text{if } (S1/C)^2 + (S2/C)^2 < (S2/D)^2 + (P/D)^2 \\ (S2/D)^2 + (P/D)^2 & \text{if } (S1/C)^2 + (S2/C)^2 > (S2/D)^2 + (P/D)^2 \end{cases}$$

Collimated Beam on Bent/Non-Flat Sample:

$$(\delta\theta)^2 = \begin{cases} (S2/D)^2 + (P/2D)^2 + (S1/C)^2 & \text{if } S1/C < 2\delta\omega \\ (S2/D)^2 + (P/D)^2 + (\delta\omega)^2 & \text{if } S1/C > 2\delta\omega \\ (S1/C)^2 + (S2/C)^2 + (P/2D)^2 + (\delta\omega)^2 & \text{if incoherent} \end{cases}$$

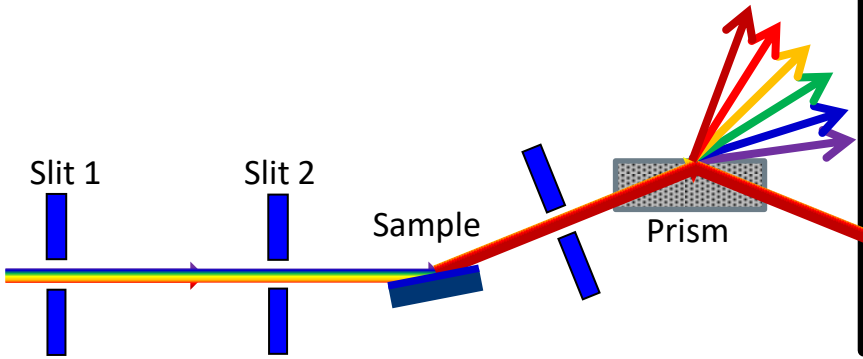
RAINBOWS: D50, Figaro, D17

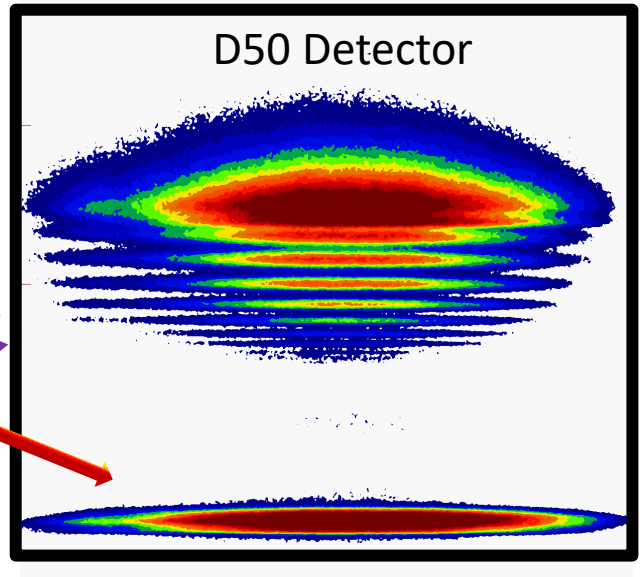
Based on **Refractive Encoding** of Neutron **Wavelength**

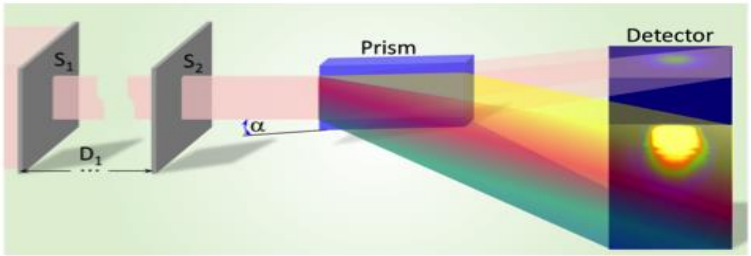
- By Refraction of a Prism Instead of Using a Chopper

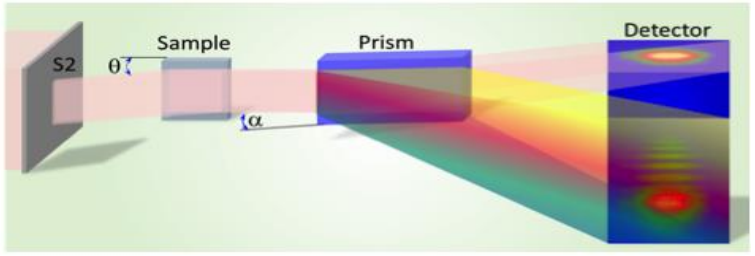
Bob Cubitt

Chopper is open for
≈ 8 min / 24 h

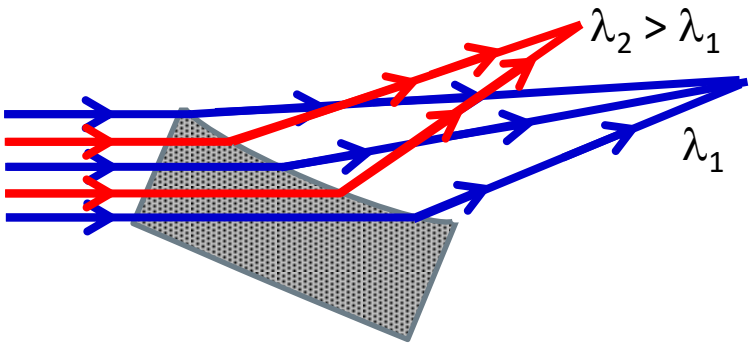


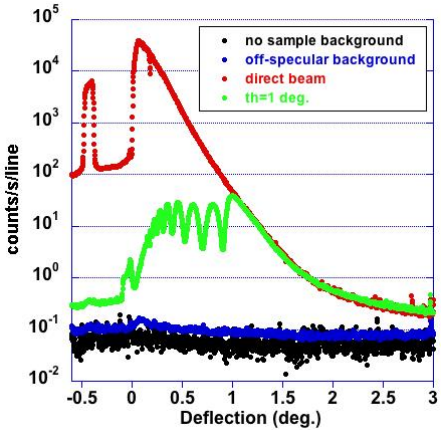


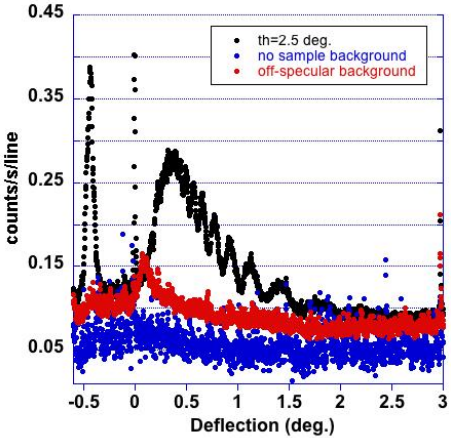


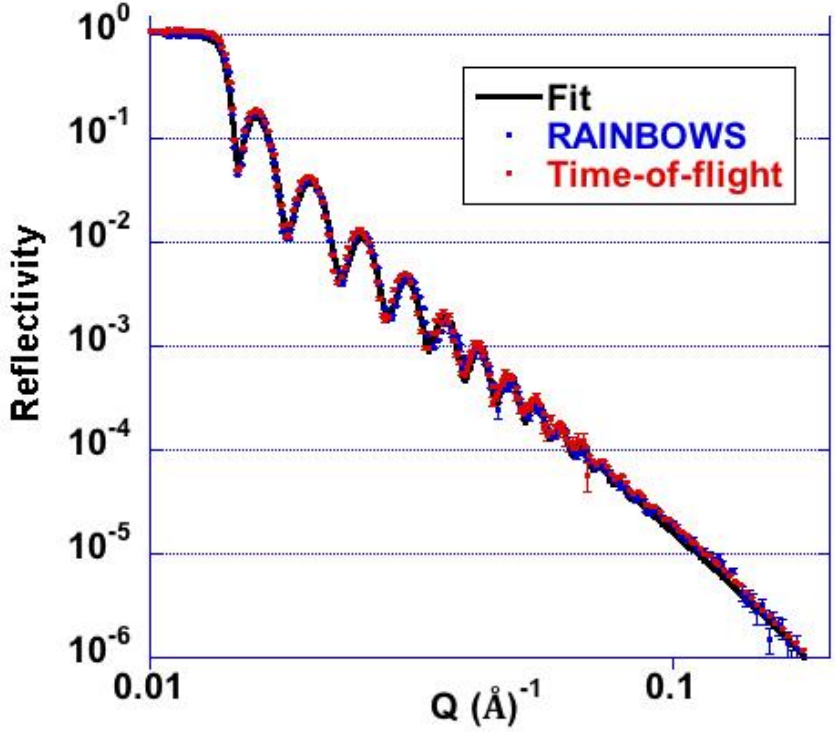


RAINBOWS: D50 Results







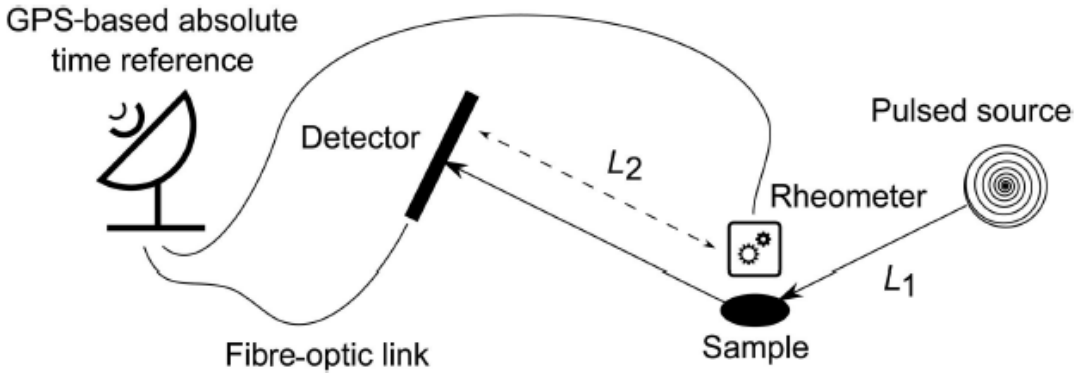


For Faster Kinetics: Event Mode

Towards neutron scattering experiments with sub-millisecond time resolution

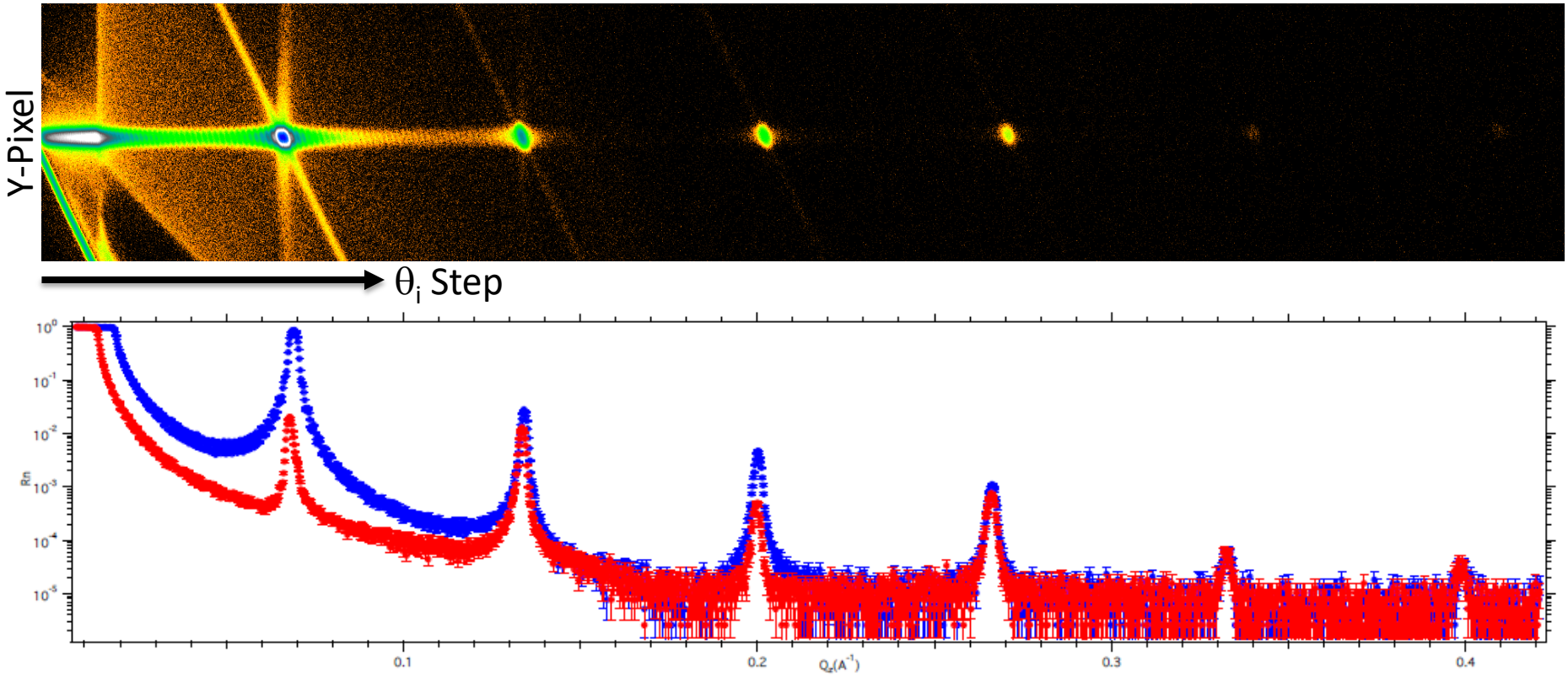
F.A. Adlmann *et al.*, J. Appl. Cryst. **48**, 220 (2015).

Stroboscopic Excitation and Acquisition

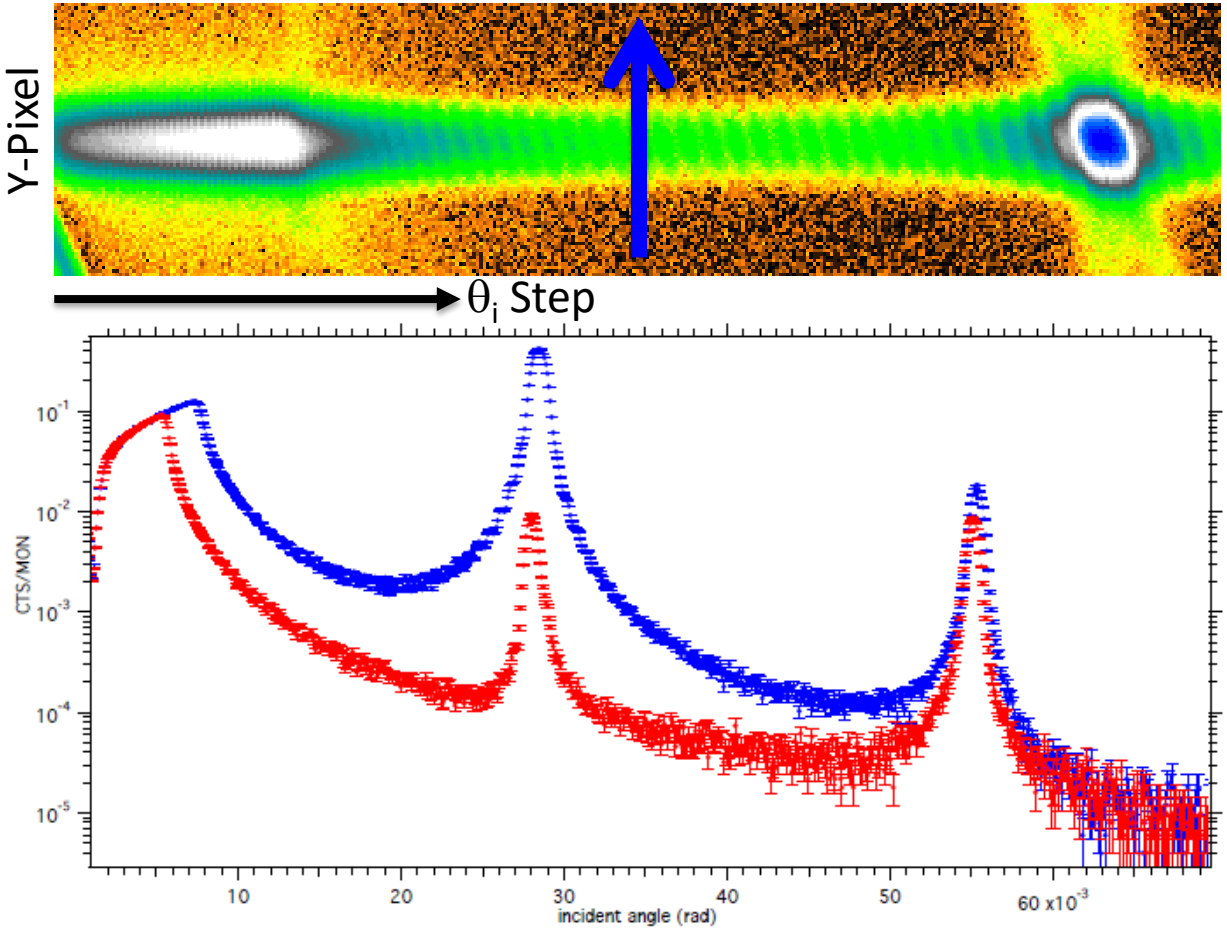


- Detection of events on absolute time scale
- Synchronized with oscillatory excitation
- NOT in-phase with Chopper => No biasing

Monochromatic (Super-Adam)



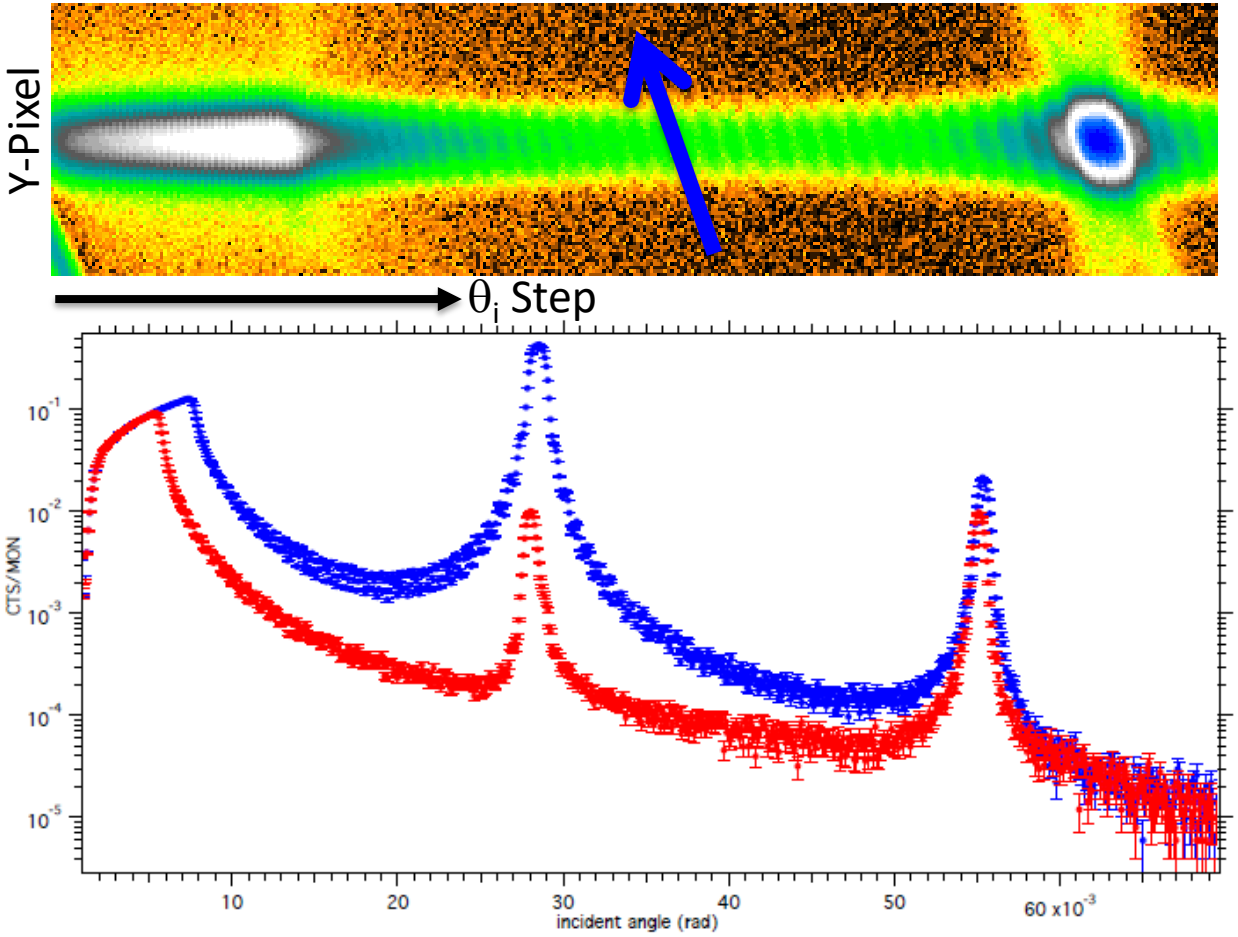
Monochromatic (Super-Adam)



Constant k_i integration

Loss of resolution

Monochromatic (Super-Adam)



"Constant Q_Z" integration

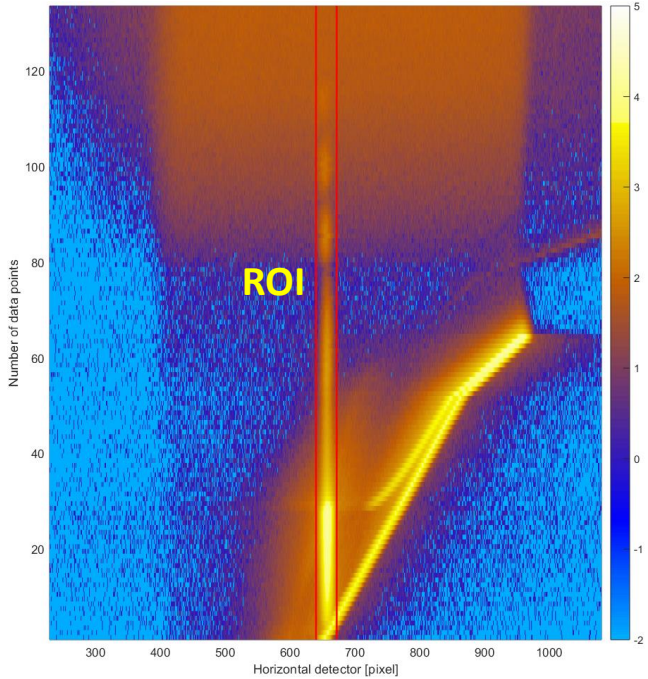
Recovery of resolution

Challenge: Solid-liquid cell over-illumination

Raw 2D data

x = horizontal detector pixel

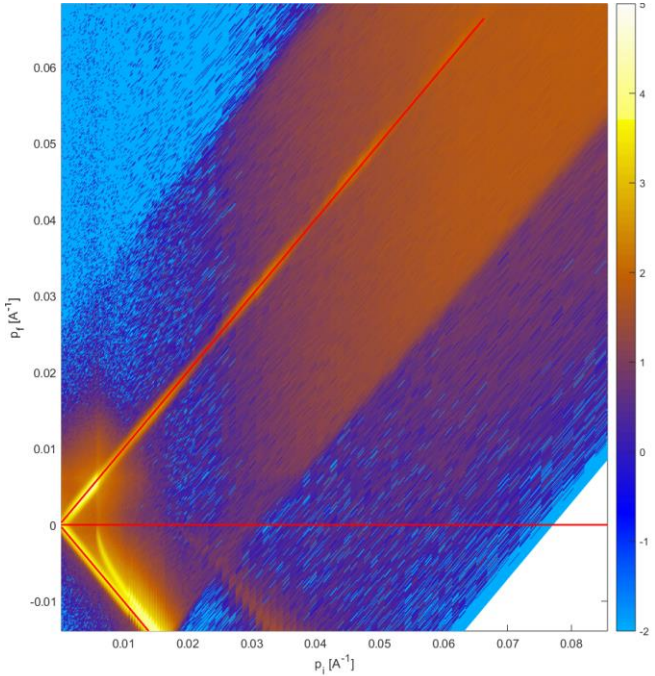
y = data point number



Transformed 2D data

x = pi

y = pf



Each scan varies

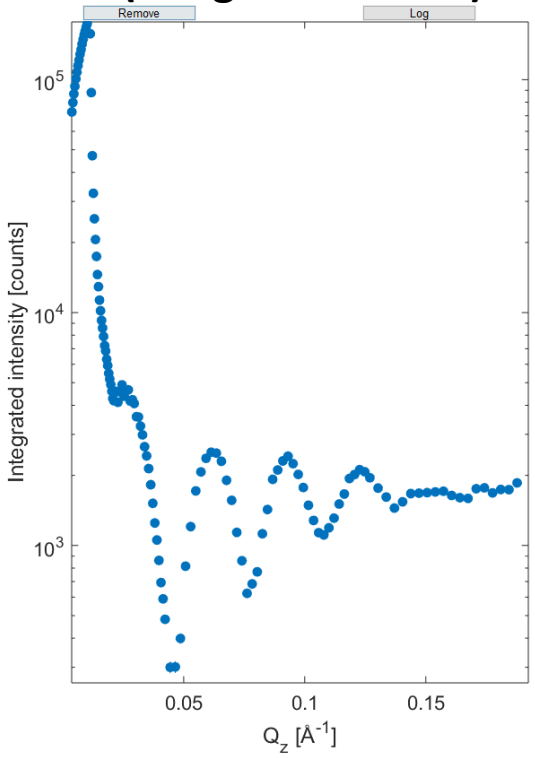
- **ω and 2θ**
- **Counting time**
- **Slit opening (footprint)**
- **Q_z stepsize**

$$p_i = \frac{2\pi}{\lambda} \sin\theta_i$$

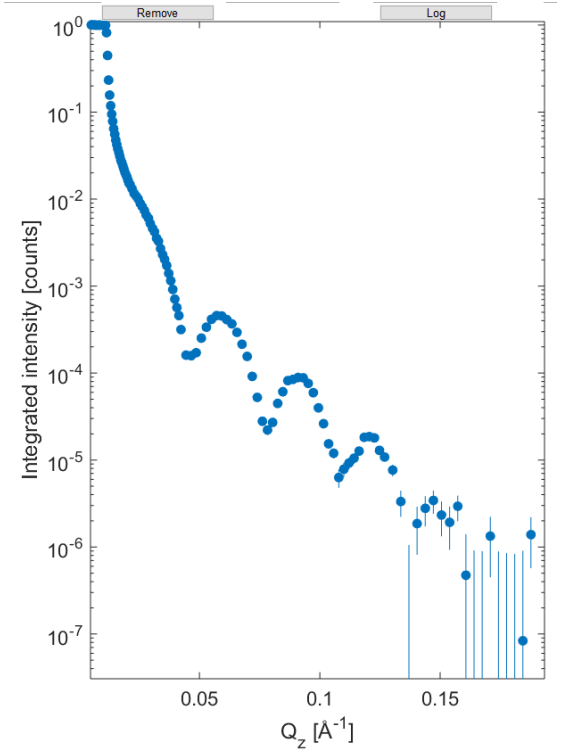
$$p_f = \frac{2\pi}{\lambda} \sin\theta_f$$

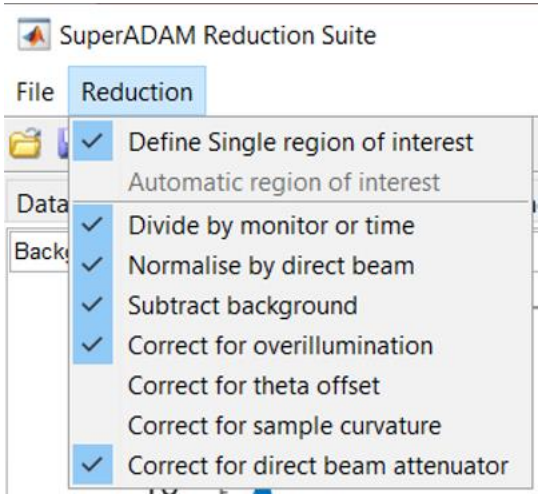
Monochromatic (Super-Adam)

raw reflectivity curve (integral over ROI)



reduced reflectivity curve

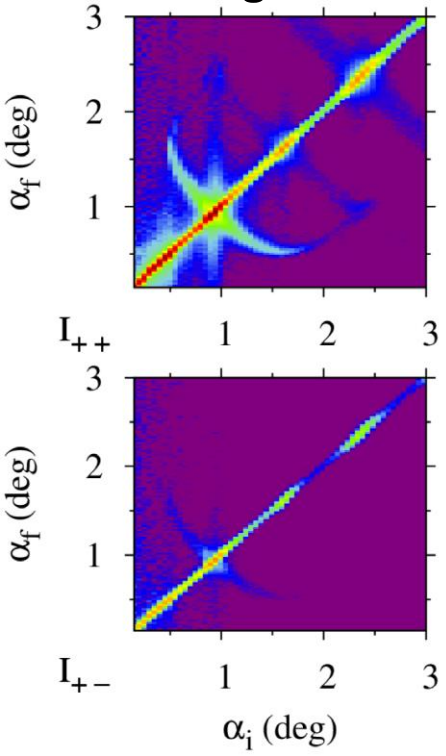




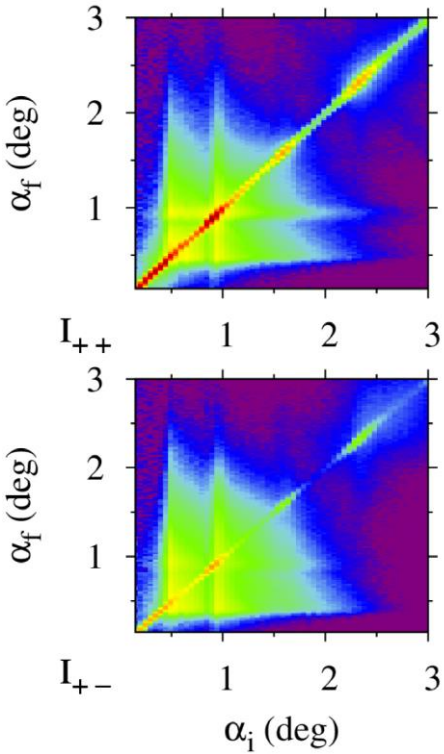
Off-specular Scattering

Monochromatic

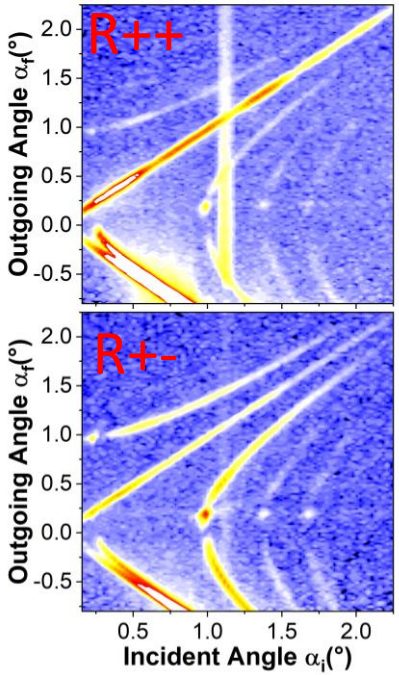
Roughness

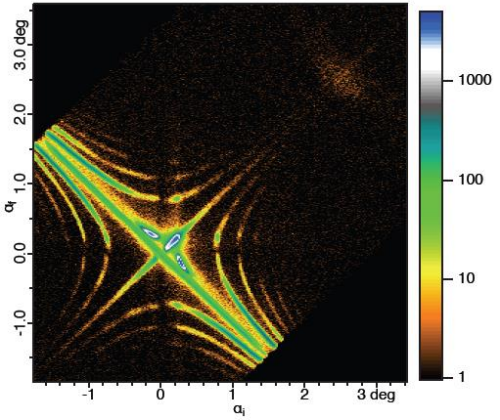


Domains



Patterns

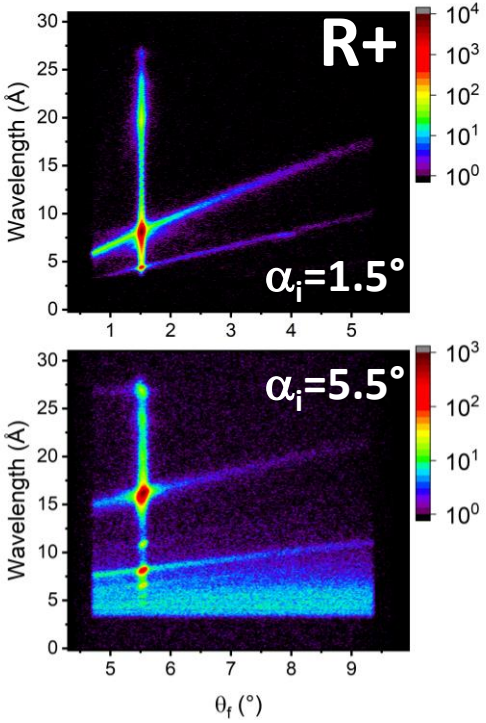




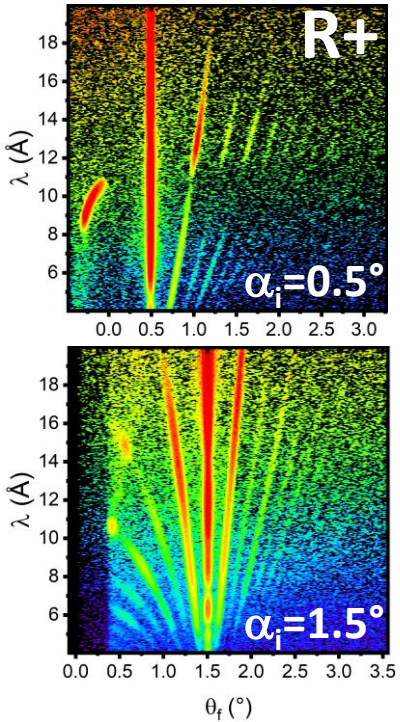
Off-specular Scattering

Time-of-flight

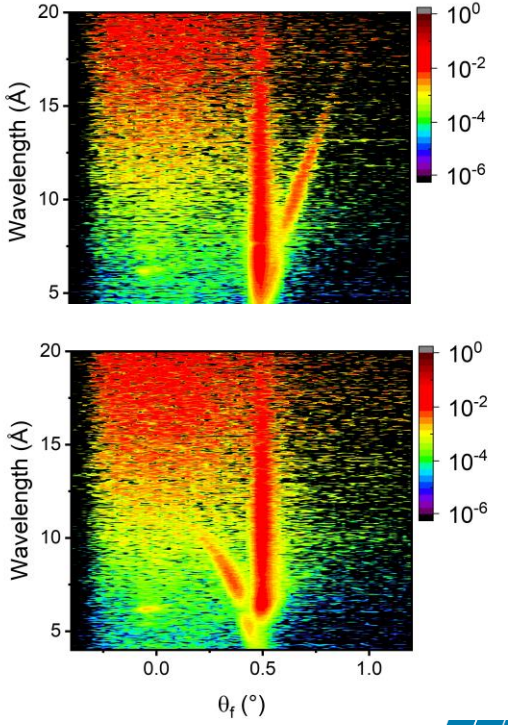
Roughness



Patterns



Zeeman / High Field



OSS Contour Plots

Instrumental

$$[\theta_f, \lambda]$$

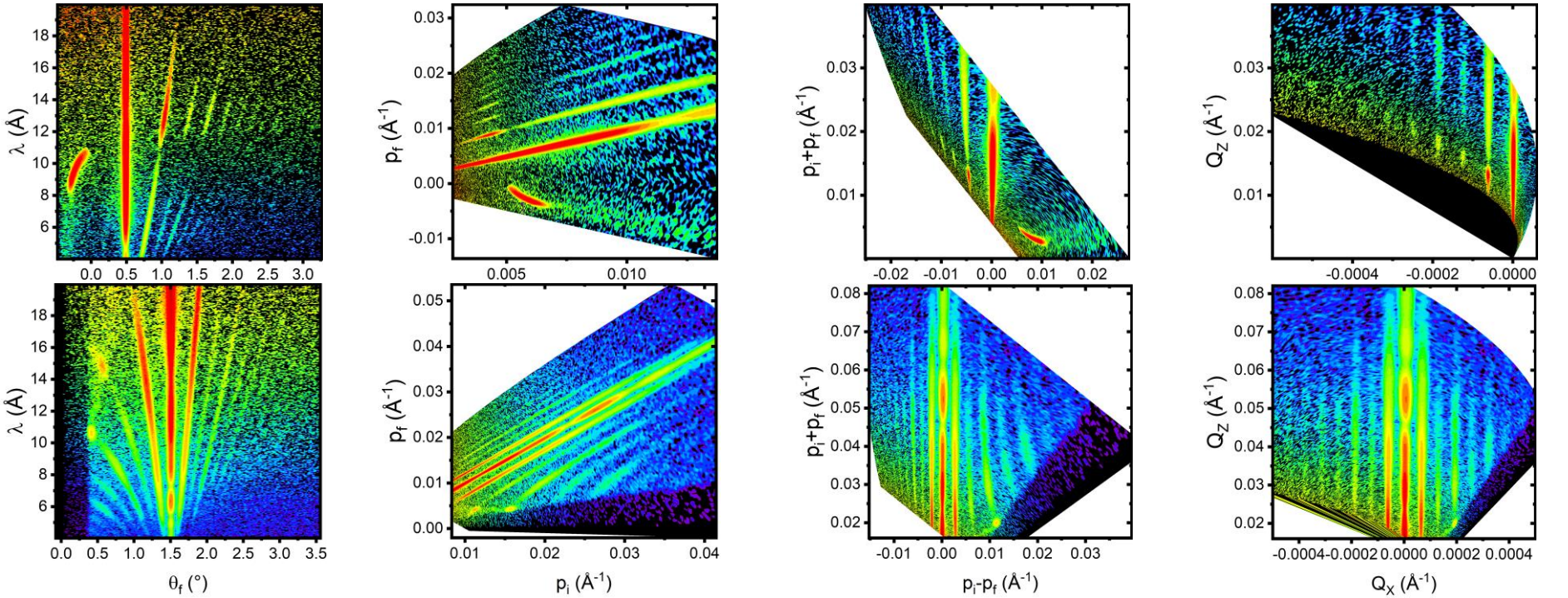
Intermediate

$$p_i = \frac{2\pi}{\lambda}\sin\theta_i$$
$$p_f = \frac{2\pi}{\lambda}\sin\theta_f$$

$$[p_i - p_f, p_i + p_f]$$

Reciprocal

$$Q_Z = \frac{2\pi}{\lambda}(\sin(\alpha_i) + \sin(\alpha_f))$$
$$Q_X = \frac{2\pi}{\lambda}(\cos(\alpha_f) - \cos(\alpha_i))$$

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