High flux vertical sample reflectometer with full polarization analysis

IKON 5

25th and 26th of September 2013

Stefan Mattauch, Alexander Ioffe, Dieter Lott

Jülich Centre for Neutron Science

Science Case

JÜLICH FORSCHUNGSZENTRUM

Two workshops were held to discuss the science case for a (vertical sample) reflectometer at the ESS:

New Opportunities for Research on Hard and Soft Matter Nanostructures using Neutron reflectometry (Berlin April 17-18 2012)

Science case Workshop for the vertical sample reflectometer at the ESS (Garching 20th and 24th of June 2013)

Participants : more than 70 internationally well recognized scientists in the field of neutron reflectometry from universities and major neutron research centers all over the world

Science Case

Two workshops were held to discuss the science case for a (vertical sample) reflectometer at the ESS:

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Vertical sample reflectometer covers 70-75% of science case

Missing are e.g.: air / liquid-liquid interfaces, fast kinetics (<70ms = 1/14 Hz), etc.

Science Case

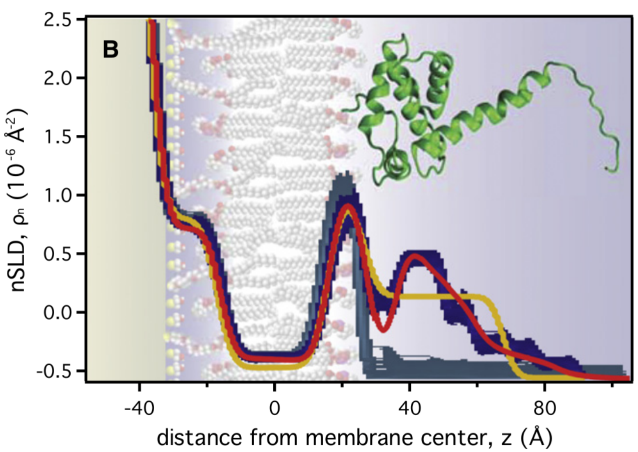
SOFT MATTER

- Hybrid materials (interface between two different classes of materials)
- Biomineralization and biocompatibility of materials (implants)
- Biology of membranes and membrane-associated proteins
- Engineered membrane-mimetic interfaces
- High throughput measurements and small samples
- Dynamic properties (time resolution)
- Performance under realistic conditions (in-vitro)
- Chemical reactions
- Processes in the drug or peptide delivery

HARD MATTER

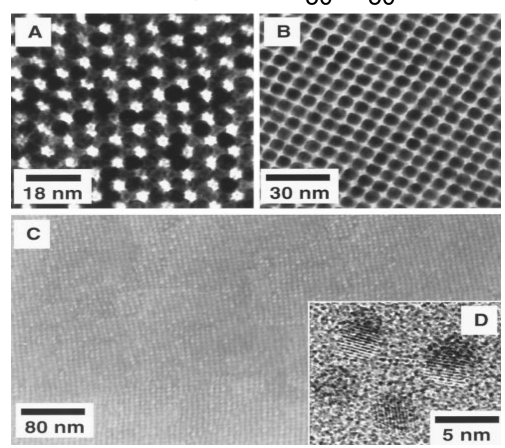
- Properties at interfaces between oxide materials
- Superconductivity between insulating materials
- Induced magnetism between non-magnetic layers
- Ferromagnetism between anti-ferromagnetic layers
- Understanding and controlling interfacial structures and interactions in the 1-10nm regime
- Interdiffusion mechanism at interfaces
- Ordered Lateral structures (magnetic and non-magnetic) on nm scale (GISANS) and µm scale (off-specular scattering)
- Small samples down to 1 x 1mm^2
- Dynamics and time resolved measurements

Hirsh Nanda et al., Biophysical Journal 99 (2010) 2516

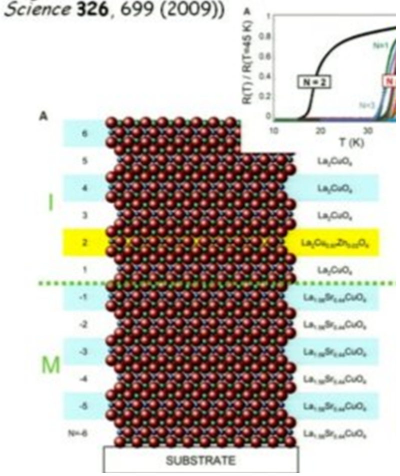


S. Sun et al., Science 287 (2000) 1989

3D assembly of $Fe_{50}Pt_{50}$ particles



Example: Interfacial superconductivity in La_2CuO_4 / $La_{1.56}Sr_{0.44}CuO_4$ (Logvenov, Gozar, and Bozovic, *Science* **326**, 699 (2009))



Science Case

SOFT MATTER | **HARD MATTER**

The most urgent common request from both communities:

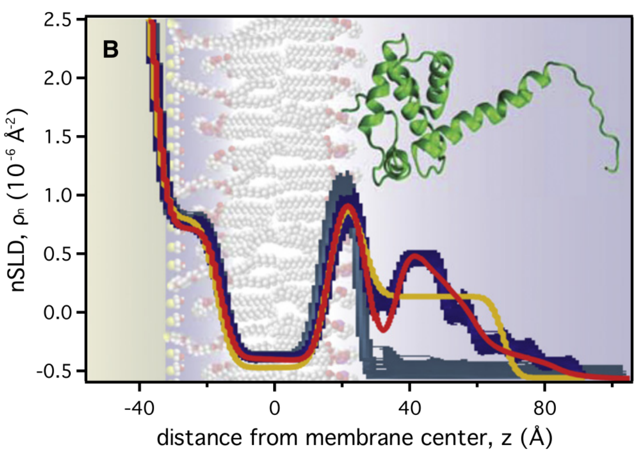
increase the **sensitivity to thin layers, interfacial regimes** in the **sub nm region**

high intensity (relaxed Q resolution)

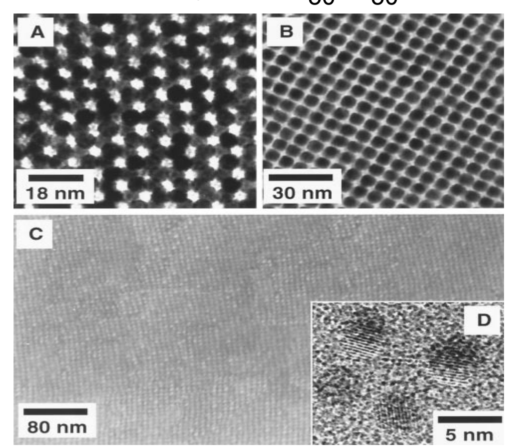
high dynamic Q range (low background)

implemented GISANS option (lateral structures in the nm range)

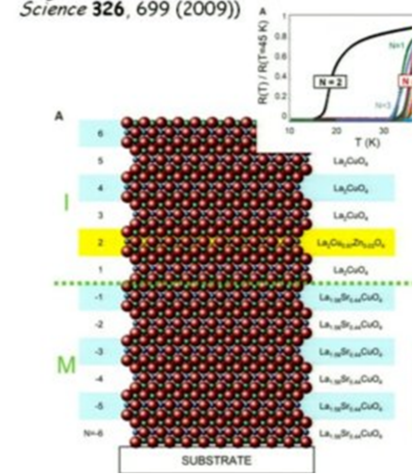
Hirsh Nanda et al., Biophysical Journal 99 (2010) 2516



S. Sun et al., Science 287 (2000) 198



(Logvenov, Gozar, and Bozovic, *Science* **326**, 699 (2009))



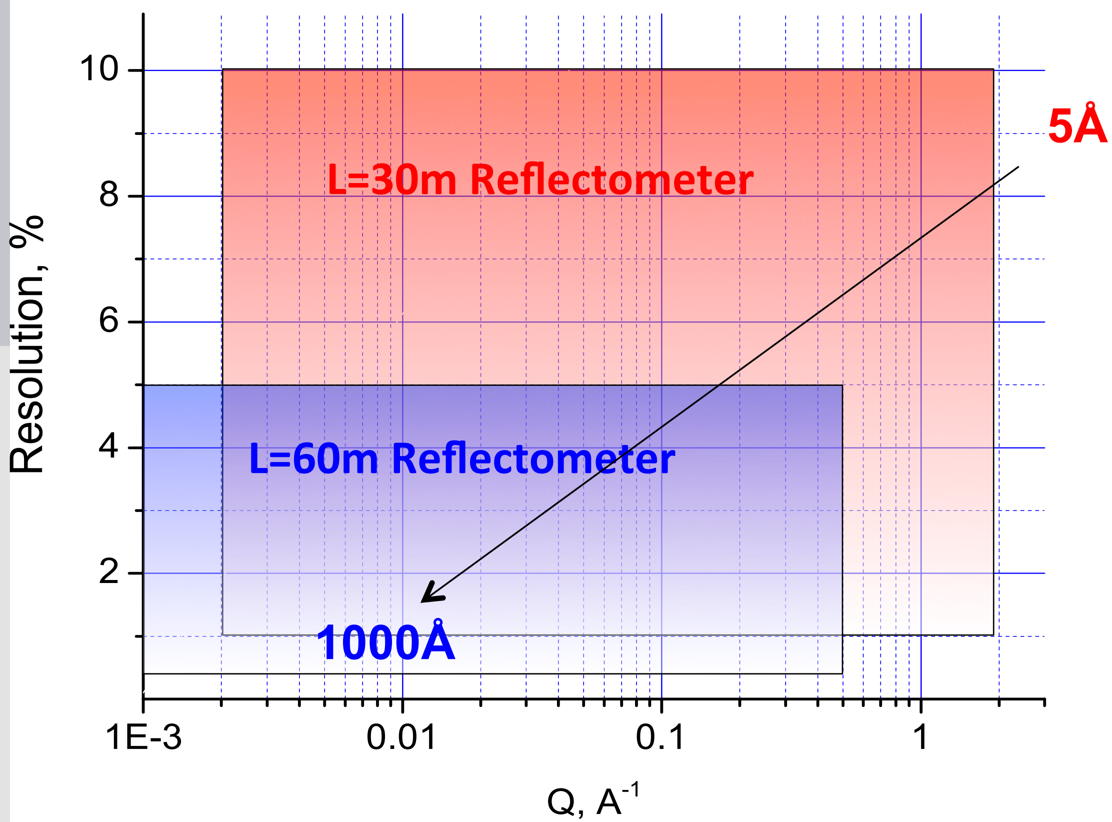
ESS reflectometers?

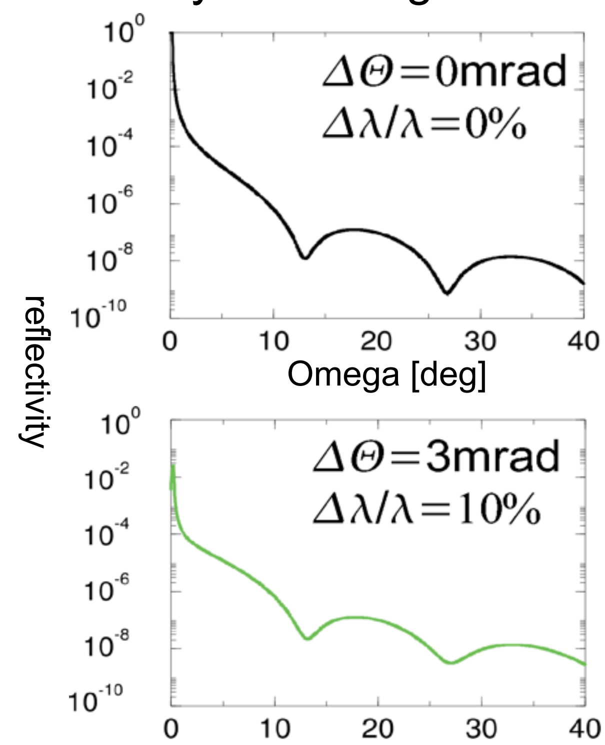
Physical limits are defined by the pulse width (***τ***) and the instrument length (***L***): their choice defines parameters of the instrument

Q-resolution: $\frac{\Delta Q}{Q} \propto \frac{\tau}{T} \propto \frac{\tau}{L}$

Wavelength band (Q-range for one setting): $\Delta\lambda \propto \frac{1}{L}$

The maximum use of neutrons from the source: ***τ = τ**$_{ESS}$*. Indeed, no instrument can provide maximum performance over the whole Q-range and for any resolution.

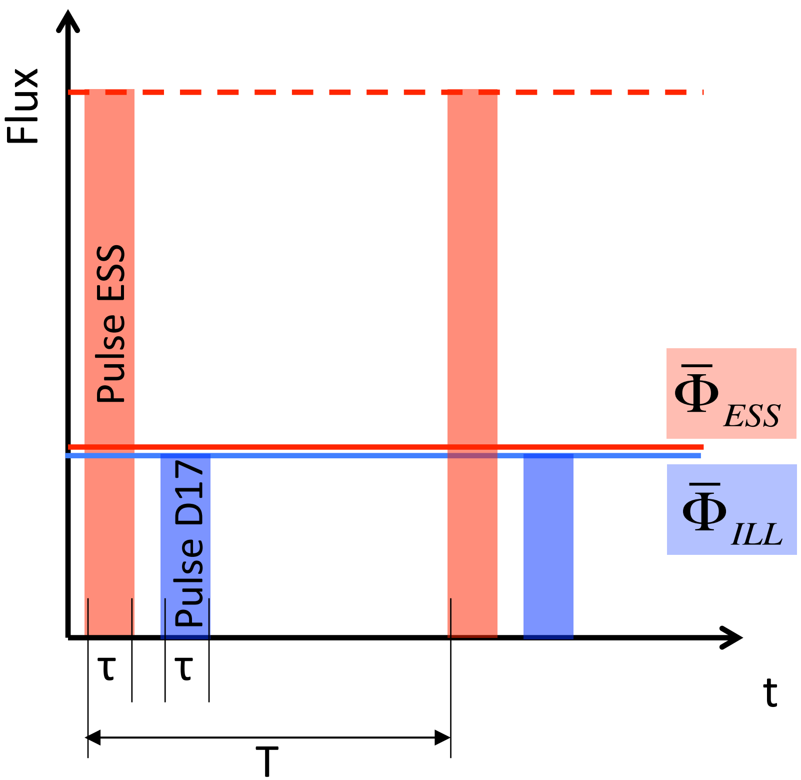




Back-of-envelope calculations

ESS reflectometer vs. D17 (TOF):

$$\bar{\Phi}_{ILL} \approx \bar{\Phi}_{ESS} \qquad \Phi_{D17} = \frac{\tau}{T}\Phi_{\text{ESS Refl}} \approx \frac{1}{25}\cdot\Phi_{\text{ESS Refl}}$$



	D17	ESS	ESS/D17
Pulse duration	7.5ms for 20% resolution	3ms for 10% resolution	Even at 10%
Repetition rate	16.6Hz	14Hz	even
Neutron Optics	Short (3m) parabolic guide	Long (26m) focusing elliptic guide	Should be a gain >2
Band	2.2-27 Å	2-10 Å	More than ½
Peak flux	1	25	25
Total gain			> 25

Effective gain is even more considering the sample sizes:
(1x1)cm^2 for the ESS vs. (1x5)cm^2 for D17

Ref.: R. Cubitt, G. Fragneto (2002); P. Gutfreund, private communication; D17 web page

Instrument design philosophy

Modes with best possible performance
!No compromise!

- **Un-/ Polarised specular reflectivity (thin interfaces areas)**
    - high neutron flux at sample with low background
    - high Q range high dynamical range (≥8 orders)

- **Un-/ Polarised off-specular scattering / GISANS mode (lateral structures)**
    - off-specular regime comes for free with above
    - for the instrument optimised GISANS mode

- **Smaller sample sizes**
    - from 10 mm x 10 mm down to 5mm x 5mm

Instrument design philosophy

Modes with good performance

(Add-ons with compromises)

- Highly focused beam down to 1 x 1 mm^2
- High wavelength resolution of 1% and 3%

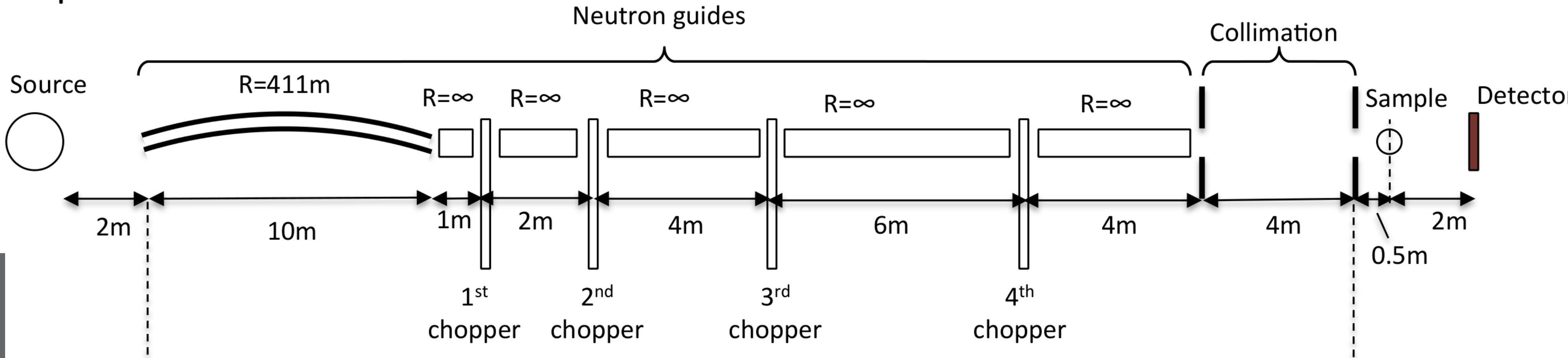
- High resolution GISANS

Layout of the instrument

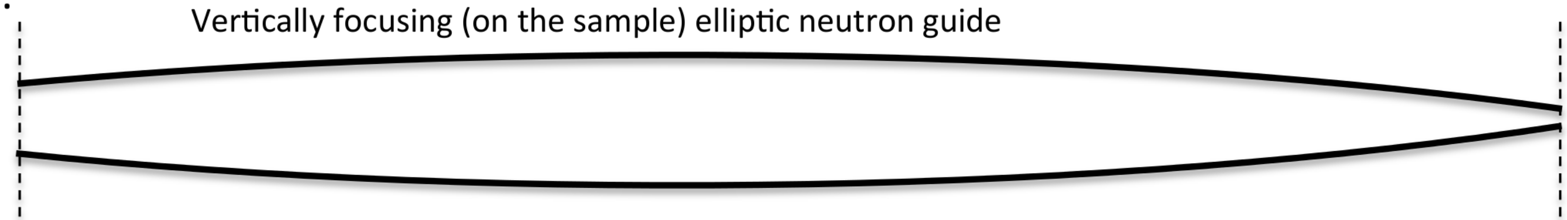
Wavelength band of 8Å
(out of the range of 2-20Å)

Chopper	1st	2nd	3rd	4th
Position	13m	15m	19m	25m
Radius	35cm	35cm	35cm	35cm
Frequency	14Hz	14Hz	14Hz	14Hz
Window	97°	118°	158°	217°
Type	Band	FO	FO	FO

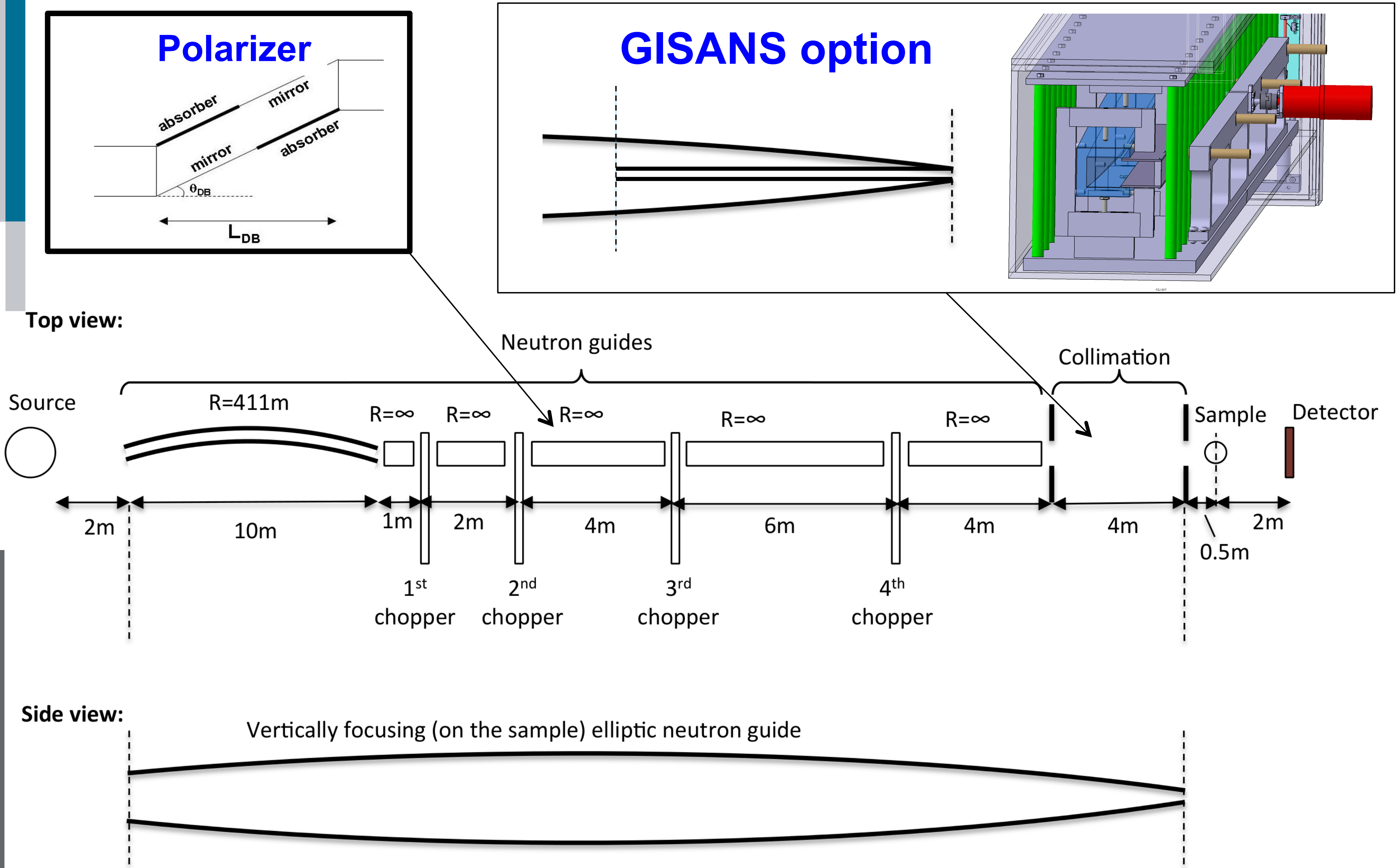
Top view:



Side view:



Layout of the instrument



Layout of the instrument

Three optimized setups for the detector

Off-specular and specular

- Large 2D-detector
- Spatial resolution <2x2mm^2

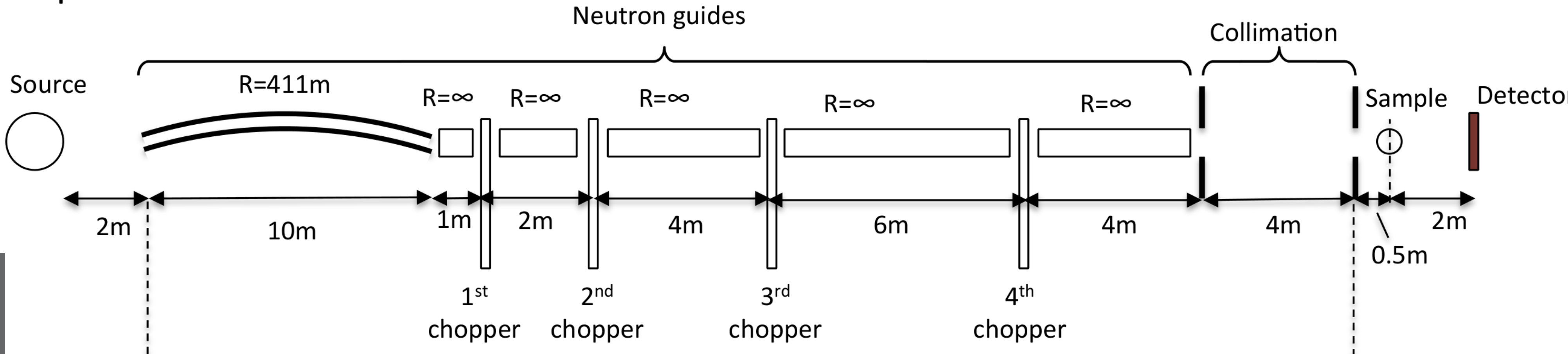
Pure specular

- Single detector in front of 2D-detector
- tight collimator

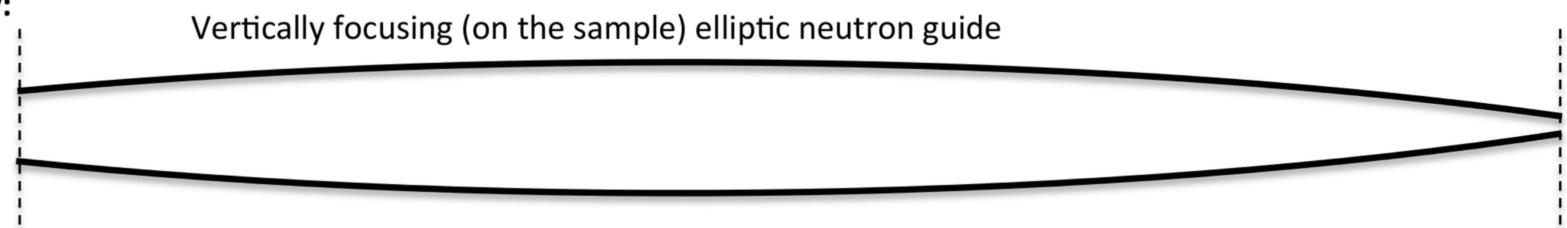
GISANS

- Variable distance 2-6m
- Tilted in height

Top view:

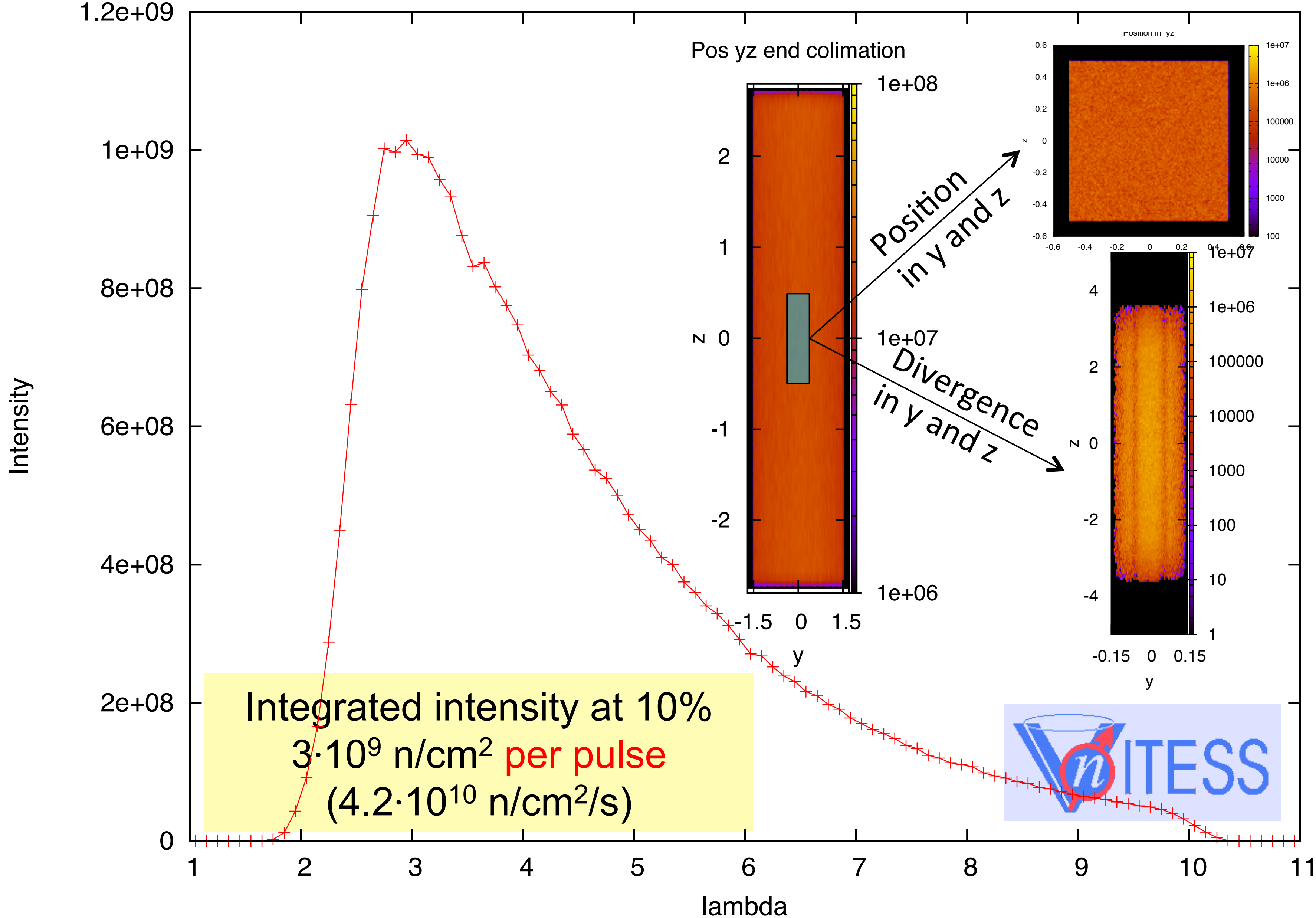


Side view:

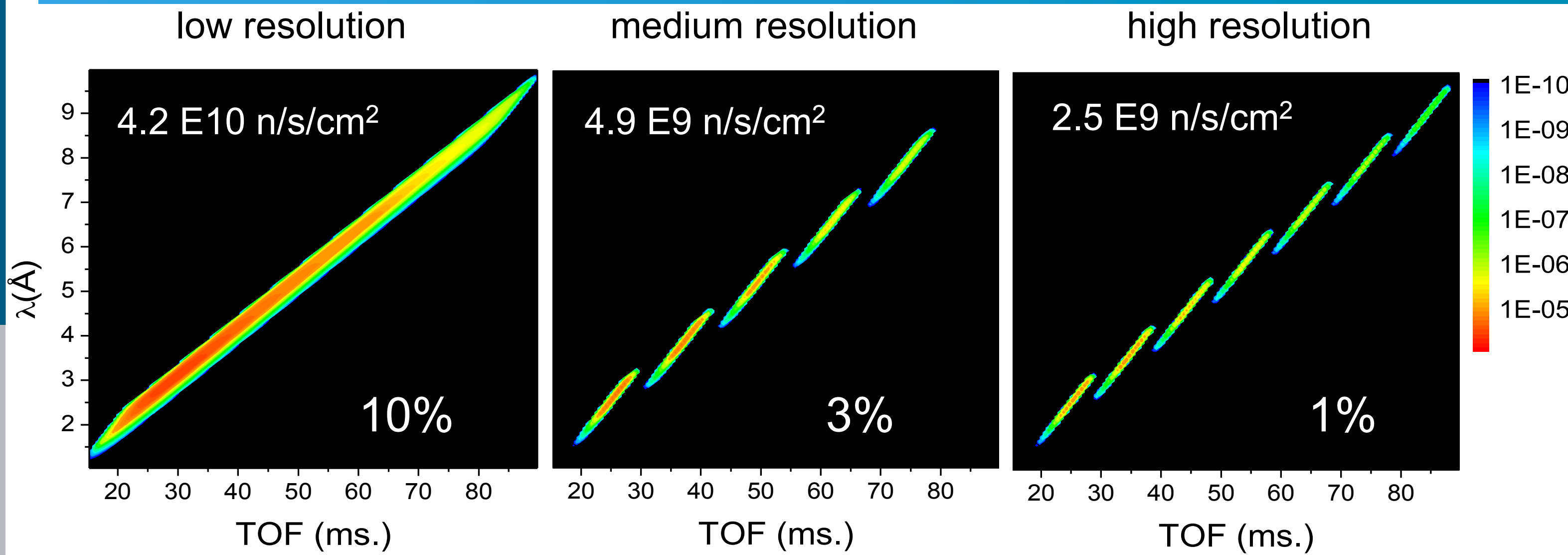


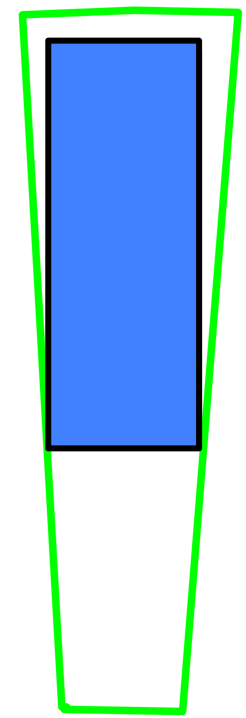
Instrument simulations

Flux at the sample position for 1cm^2 and 2 mrad (Gaussian equivalent FWHM) divergence



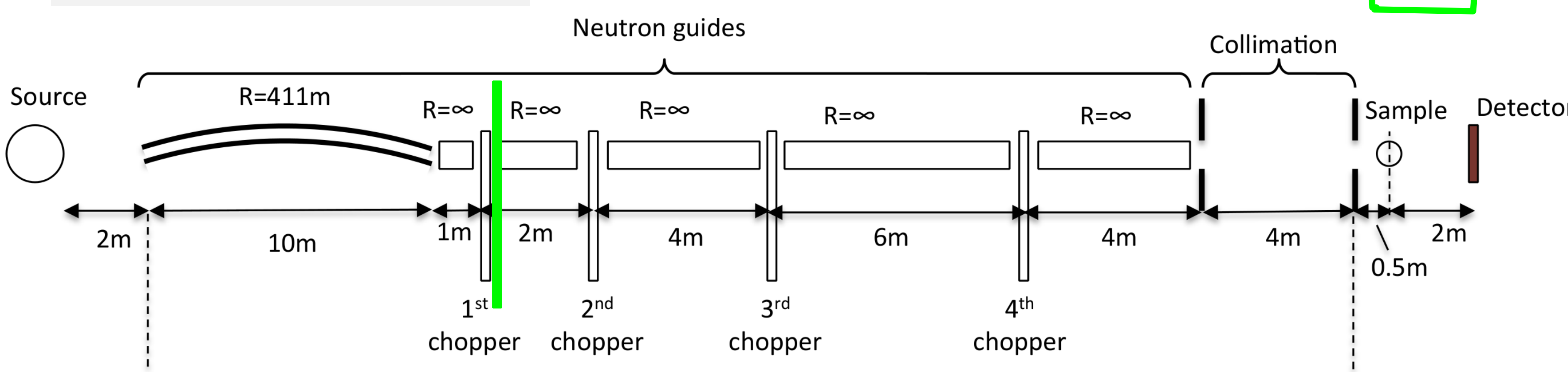
High resolution modes





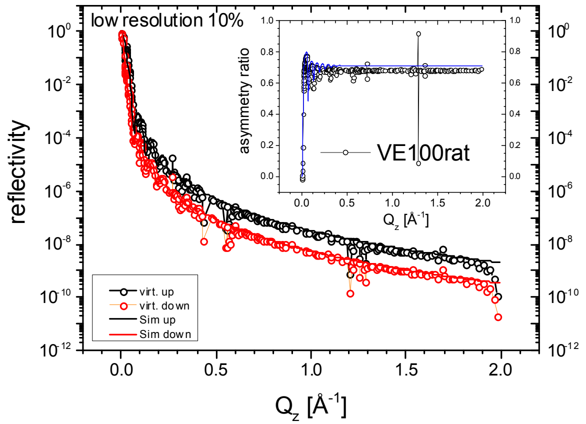
	$\Delta\lambda/\lambda$ at 25 ms. (%)	$\Delta\lambda/\lambda$ at 74 ms. (%)
Low-res	13.3	4.4
Mid-res	4.0	1.3
Hi-res	1.6	0.5

For high resolution the WFM-method is also applicable
M. Strobl et al., NIM A 705 (2013)

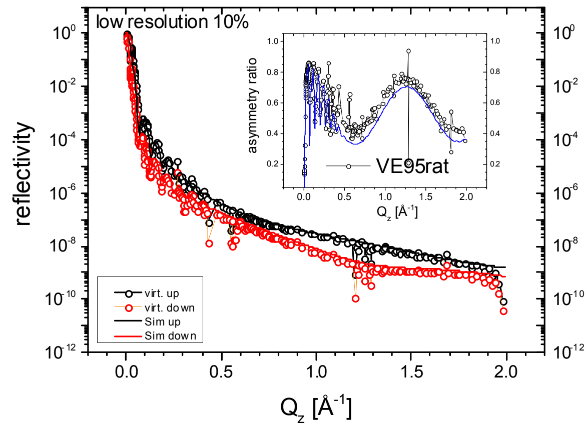


Virtual experiments

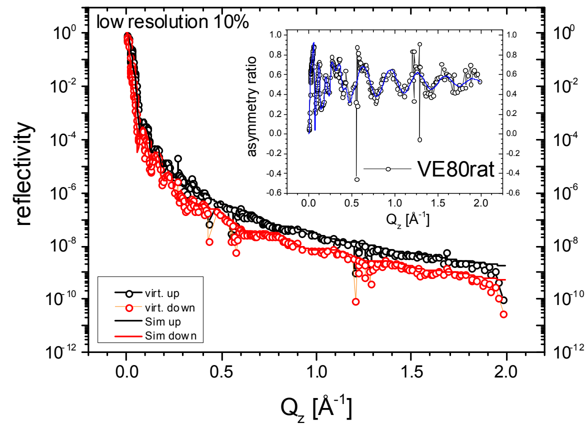
Simulations: Fe 100 on Si Substrate

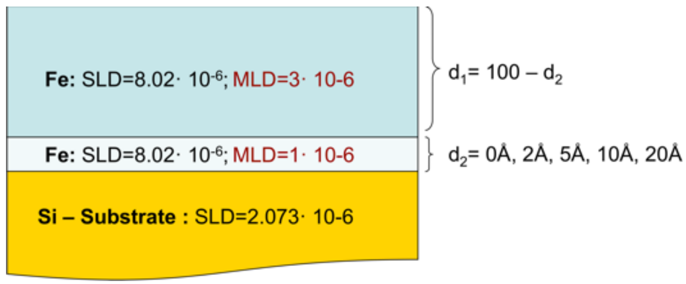


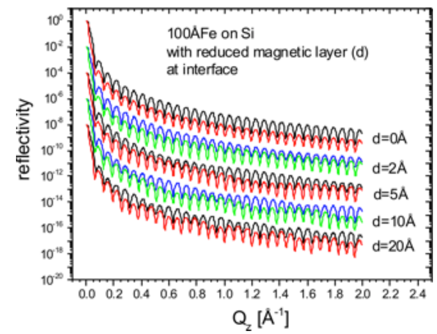
Simulations: Fe 95 - 5 on Si Substrate



Simulations: Fe 80 - 20 on Si Substrate







- perfect interfaces, no roughness
- 4 angular settings
- 2 polarization settings: ++ and –
- counting statistics > 50 counts/channel

=>measurement time: ~15 minutes

Conclusions

Low resolution reflectometer
optimized for thin interfaces and small scale lateral structures

Low risk
Based on tested principles / components

INTENSITY GAIN
25+
over today bests

About ¾ of science case will be served

Add on's
NOT COMPROMISING main modes

- High wavelength resolution 1% and 3%
- Small samples with 1 x 1mm^2

Non optimised BUT much better then presently available

Thanks to the contributers:

D. Rodrigues, Z. Medic, U. Rücker, Th. Brückel (FZ-Jüllich), J.-F. Moulin (HZG), S. Manoshin (JINR), F. Ott (LLB)

Strong commitment of the LLB for participation in the realisation of the proposal

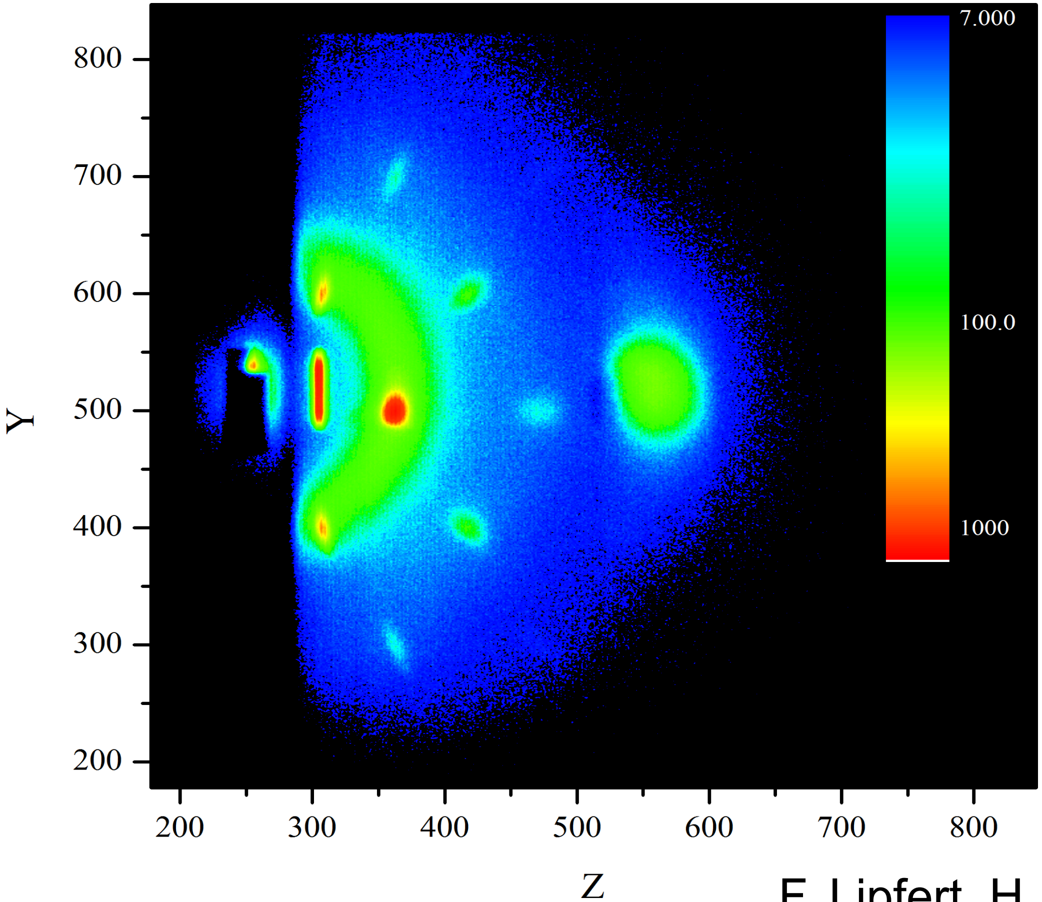
Thank you for your attention!

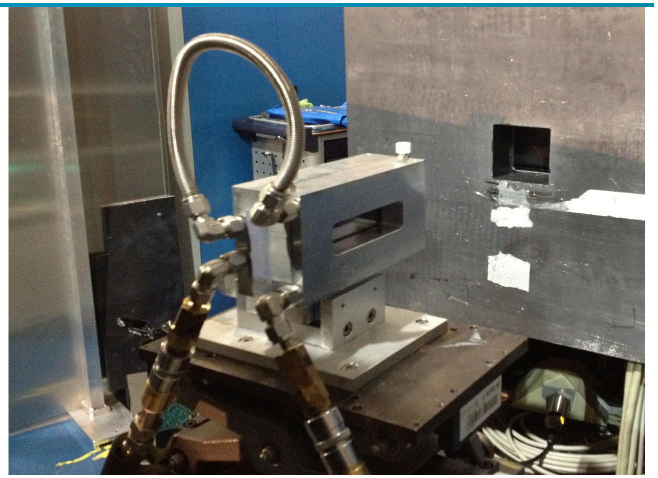
Key facts

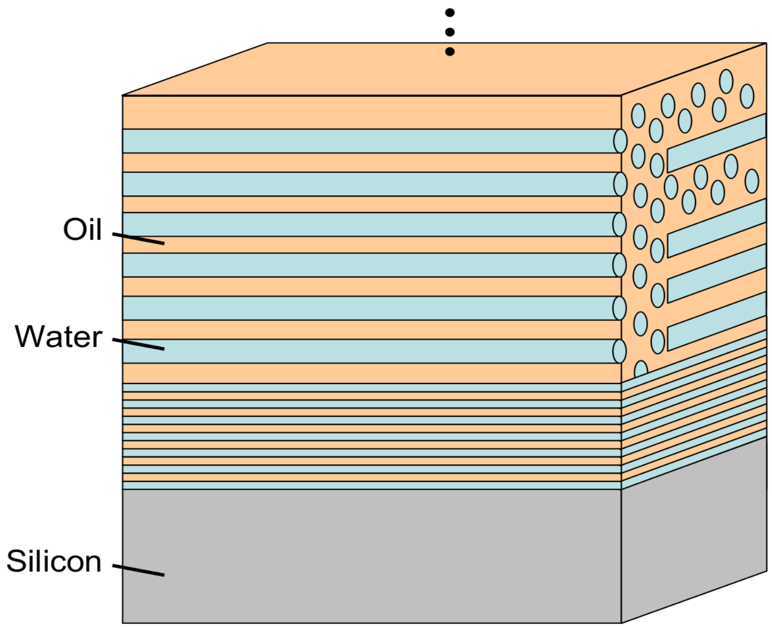
Wavelength band	8Å out of 2-20Å
Detector resolution	~2 x 2 mm^2
Sample detector dist.	2m
Max. detector angle	> 90°
Q_z-range	0.002 - 4.4 $Å^{-1}$
Q_x-range	6.10^{-5} – 0.01 $Å^{-1}$
α_i (incident angle)	-10° - + 90°
Polarisation	Double reflection polarizer
Polarisation analysis	^3He / Radial solid state polariser (depending on ESS policy)
Collimation	4000 mm long
Focusing	Vertically focusing elliptic guide
GISANS option	4000 mm long inside collimation
Q_y-range	0.002 - 0.8 $Å^{-1}$
Optimized for	Interfaces, thin layers and lateral structures

Soft matter GISANS at MARIA

Measurement of hexagonal phase ordering of a C10E4-based microemulsions near a hydrophilic, planar surface at high Q-values using GISANS



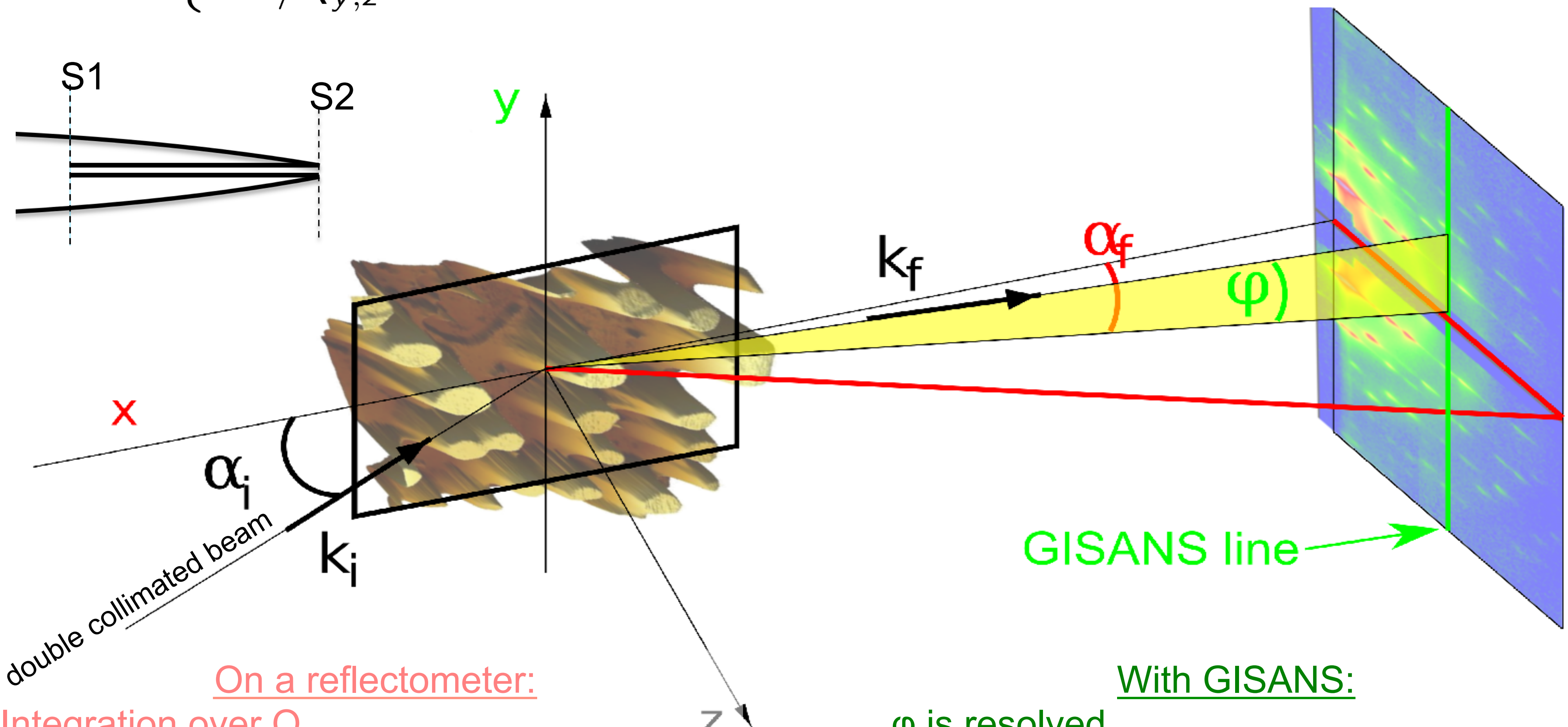




F. Lipfert, H. Frielinghaus, M. Kerscher

Geometry for GISANS

typically: $\begin{cases} 2\pi/Q_x & \simeq 1 - 10\mu m \\ 2\pi/Q_{y,z} & \simeq 1 - 100nm \end{cases}$

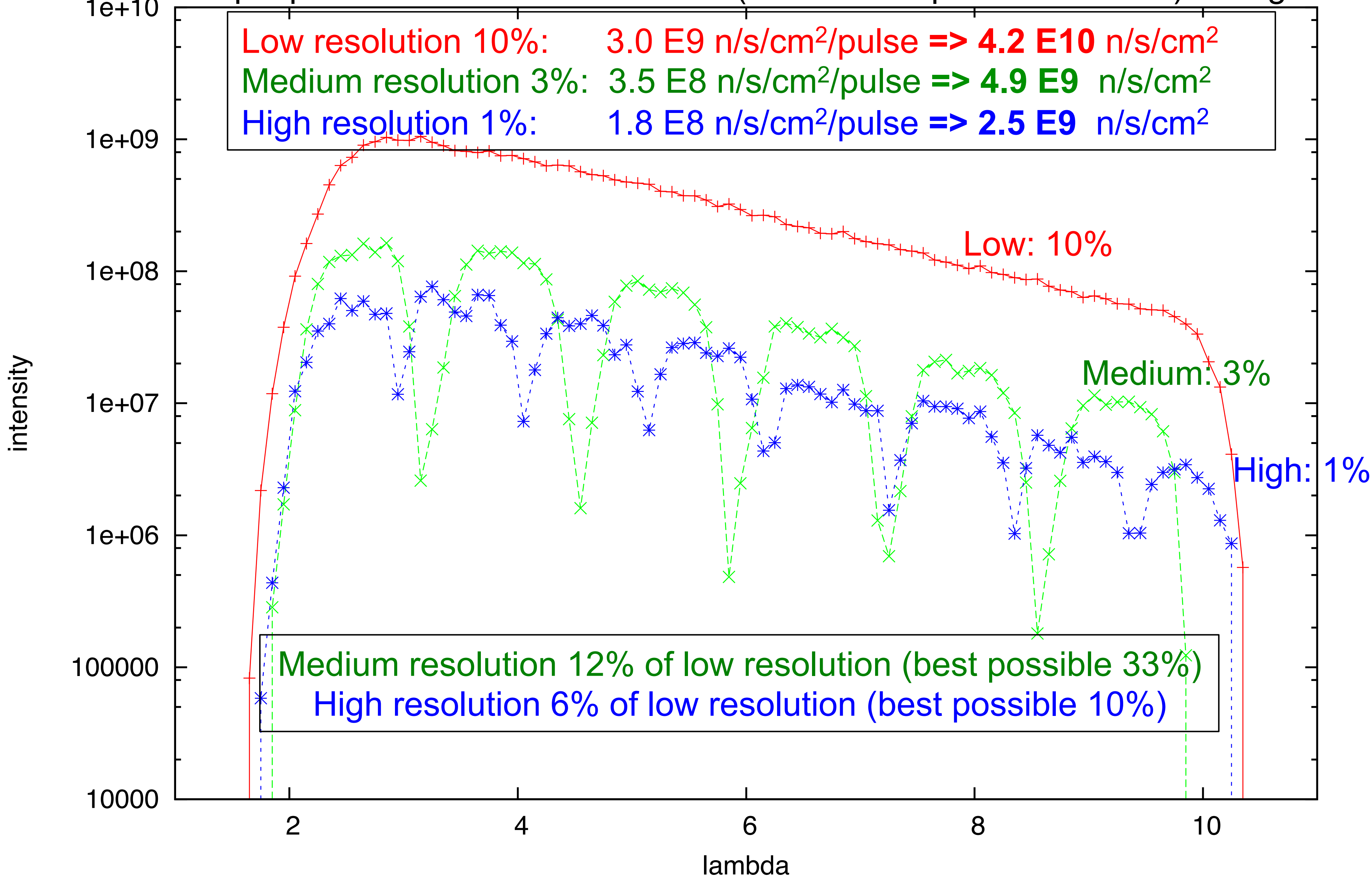


On a reflectometer:
Integration over Q_y
Lateral correlations are probed along Q_x
Layer structure along Q_z

With GISANS:
φ is resolved
Lateral correlations are probed along Q_x and Q_y !

High resolution modes

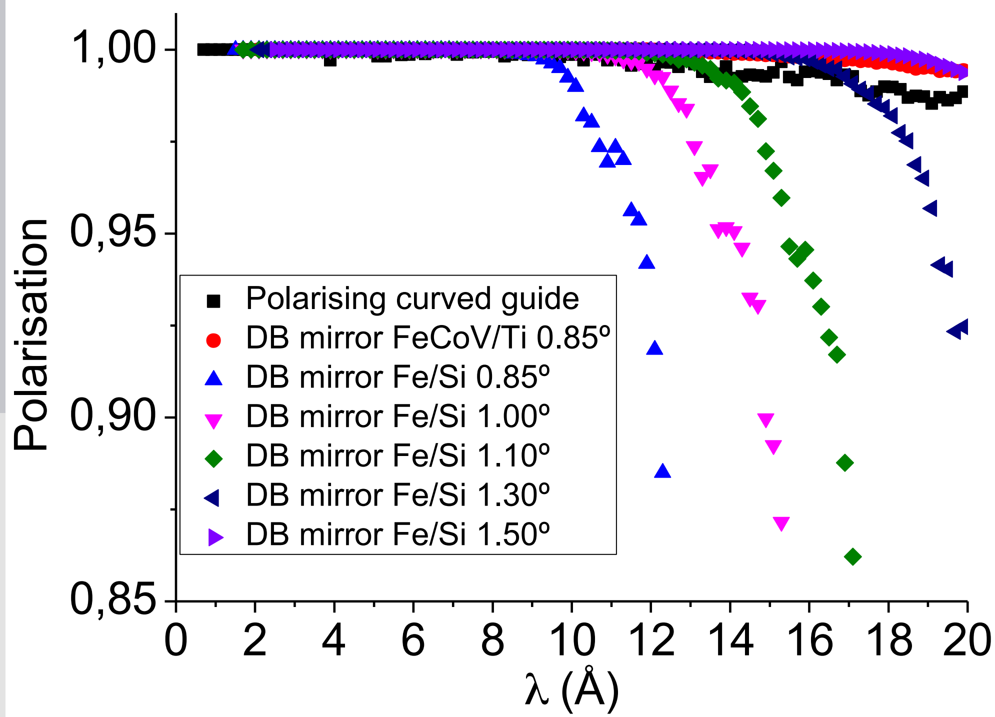
Flux at the sample position for 1cm² and 2 mrad (Gaussian equivalent FWHM) divergence

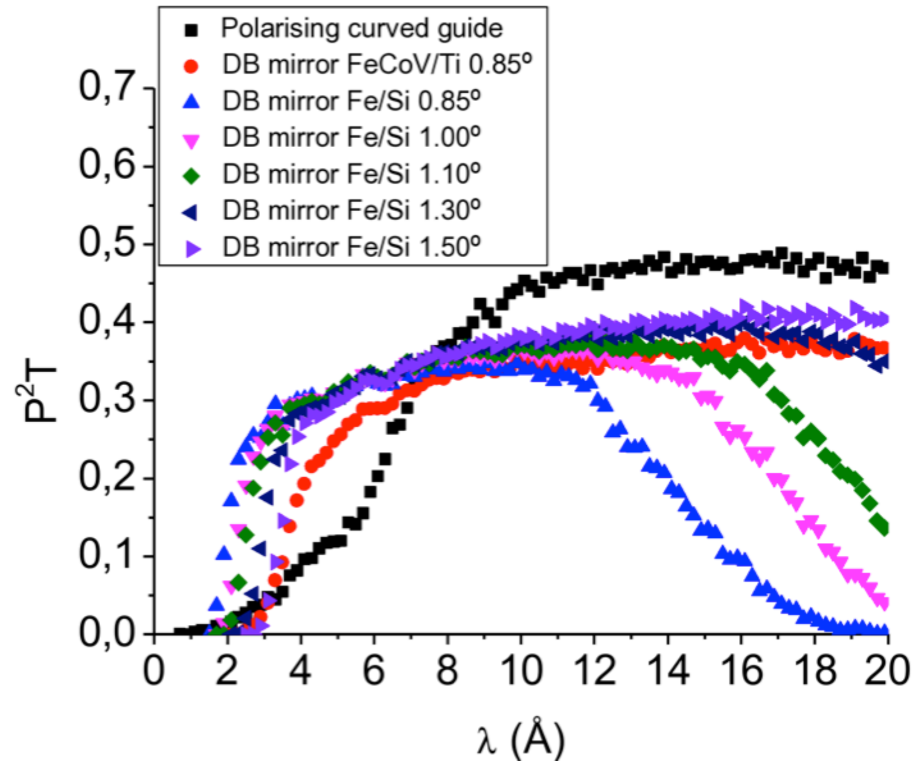


Polarising the beam

No change of geometry for unpolarized and polarised beam

curved guide
double bounce super mirror





Analysing the beam

For GISANS/SANS on large (500x500mm²) detectors

SEOP ^{3}He-analyzer (on beam pumping)

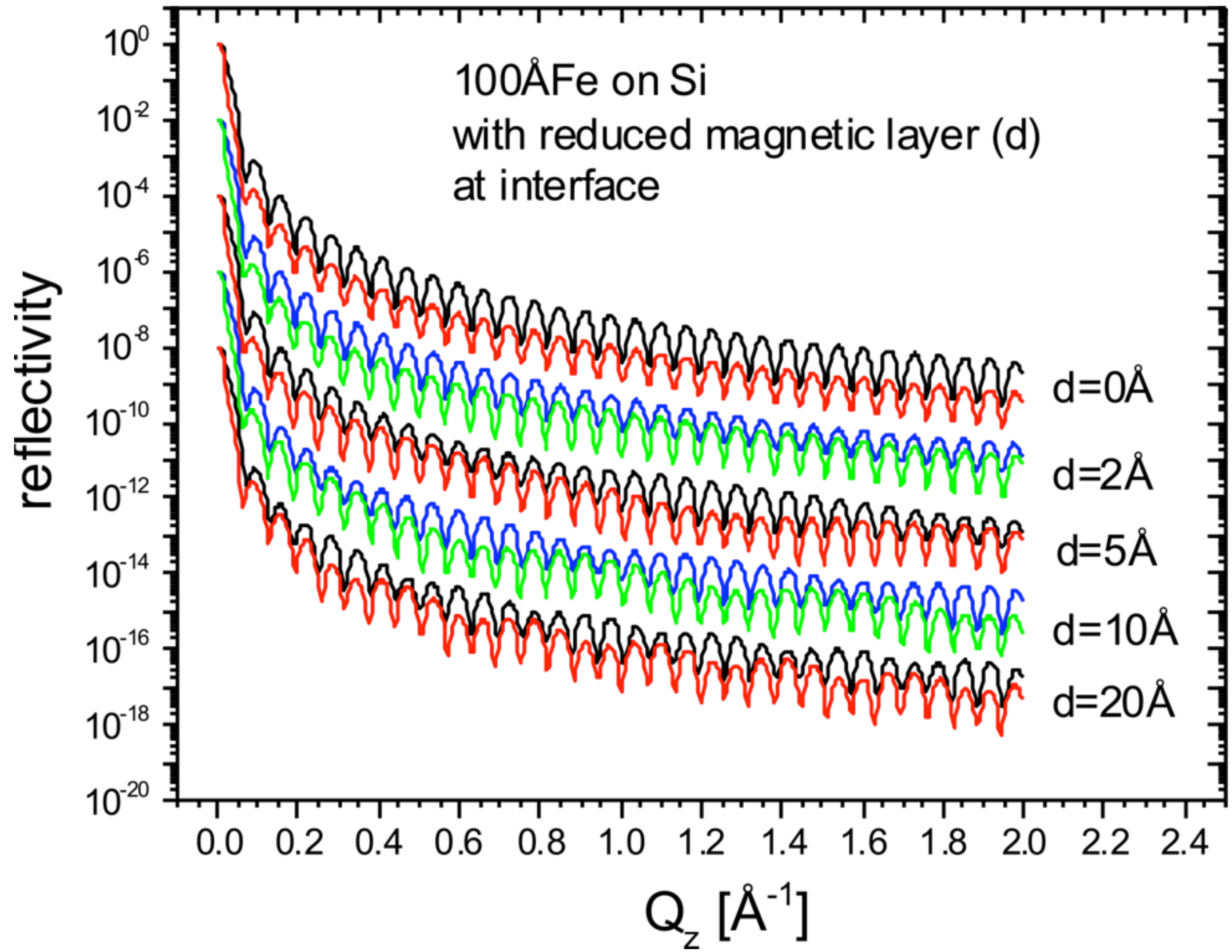
- (+) No loss of angular resolution in 2D
- (+) on beam pumping: No change of analyzing power over time! No corrections for data analysis needed!
- (+) Adjustable/tunable analyzing power
- (+) High efficency flipper inclusive (AFP)
- (-) Not as straight forward as SM (nowadays)

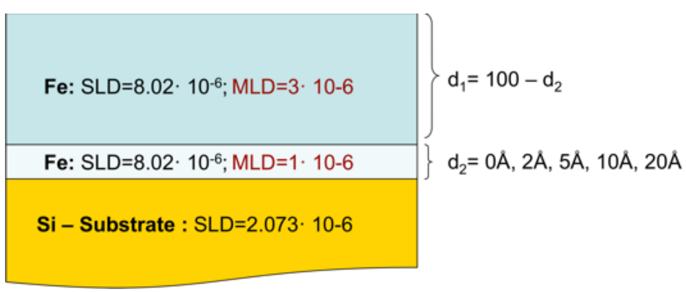
Polarizing SM

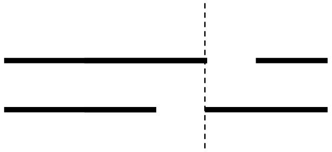
- (+) Mantainance free after setup !
- (-) Difficult to keep angular resolution in 2D (influencing the resolution)
- (-) Additional wide angle flipper (RF-Flipper) needed

Large wavelength bands are not easy to analyse

Virtual simulations

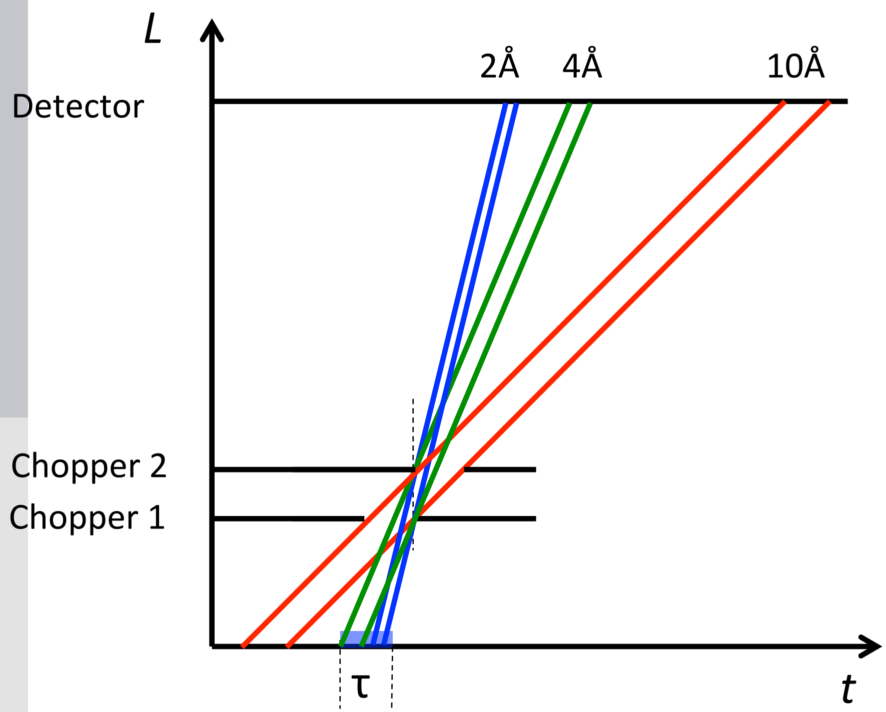


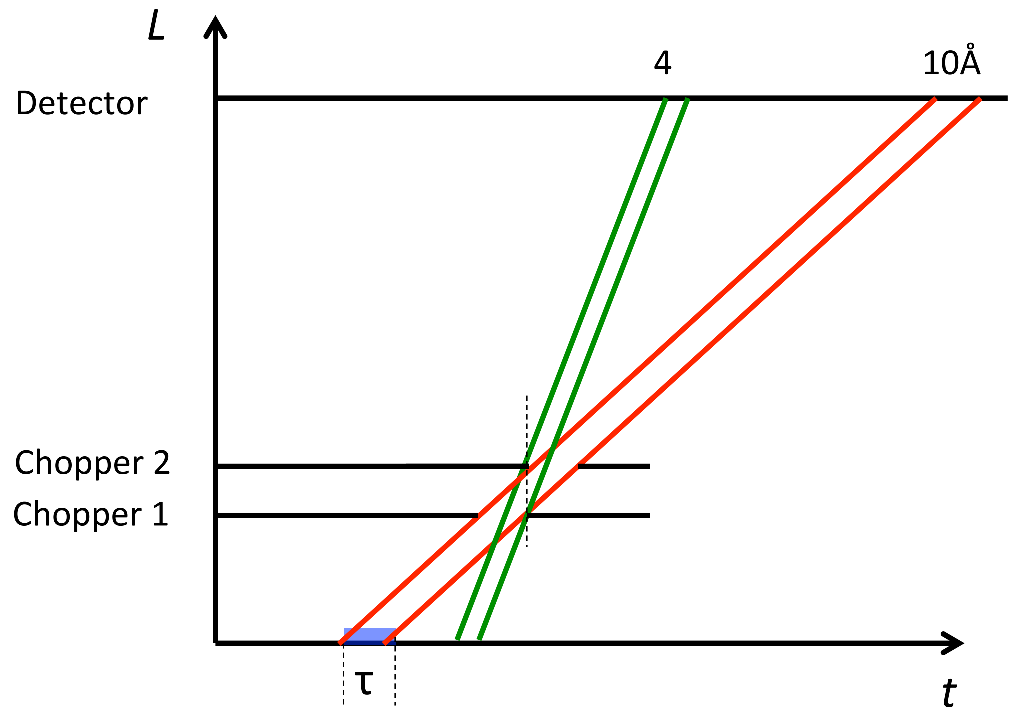




optically blind double chopper (A. van Well, 1992) ⇨ $\Delta\lambda/\lambda$=const

<u>Pulsed Source:</u> the wavelength band that can be covered by the optically blind double chopper is limited by the pulse length.

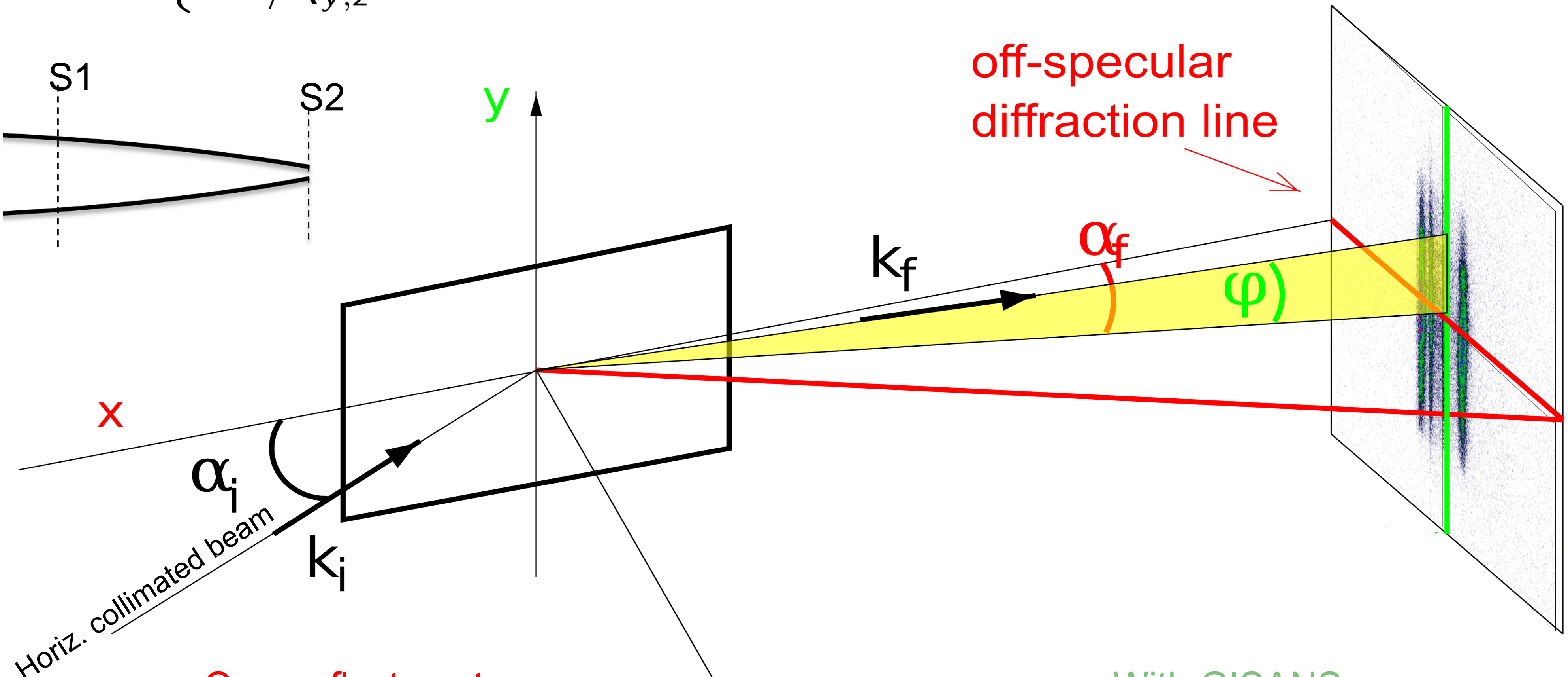




Unfortunately, this technique is not suitable for the 8Å wavelength band

Geometry for reflectometry

typically:
$$\begin{cases} 2\pi/Q_x & \simeq 1-10\mu m \\ 2\pi/Q_{y,z} & \simeq 1-100nm \end{cases}$$



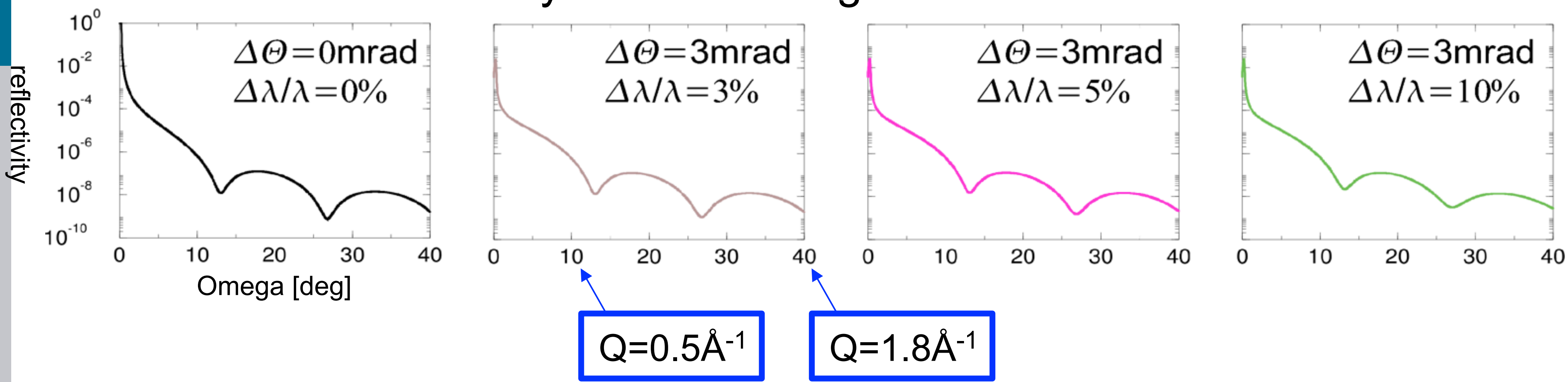
On a reflectometer:
Integration over Q_y
Lateral correlations are probed along Q_x
Layer structure along Q_z

With GISANS:
φ is resolved
Lateral correlations are probed along Q_x and Q_y !

Wavelength resolution

Specular reflectivity for Spin-up neutrons

10Å Fe-layer on a 10Å Ag substrate for 4.5Å



Dynamic range of 8 orders => $\Delta\lambda/\lambda$ has to be relaxed!

For characterization of thin interfaces
$Q=0.5 Å^{-1}$
is NOT enough!

Softmatter

Physica B 406 (2011) 2448–2452

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Polarized ^{3}He neutron spin filter program at the JC

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Keywords:
Polarized ^{3}He
Neutron spin filter
Spin-exchange optical pumping
Polarized neutrons

ABSTRACT

Polarized neutron instruments will occupy abou
instrument park. A successful polarized ^{3}He prog
have been focusing the developments on spin-exc
situ. Where possible, in situ polarization using
performance of the instruments. Further this allo
^{3}He to be developed optimized for each instrumen
for the advantages of in situ polarization; descri
tests of its performance; describe testing of pola
biological samples, and our plans for an in situ p

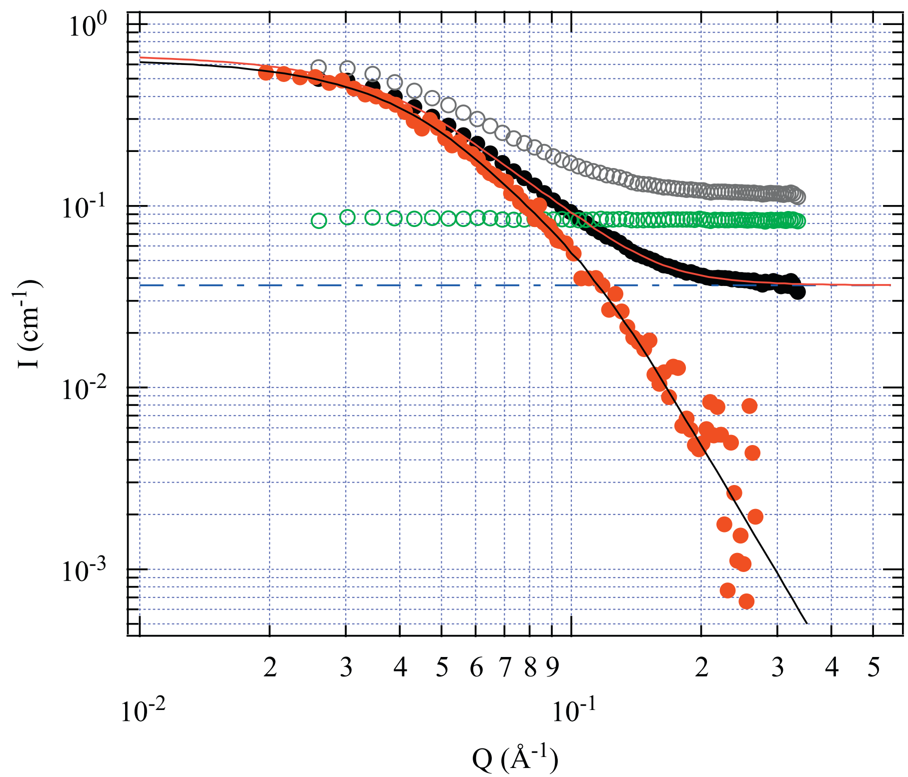
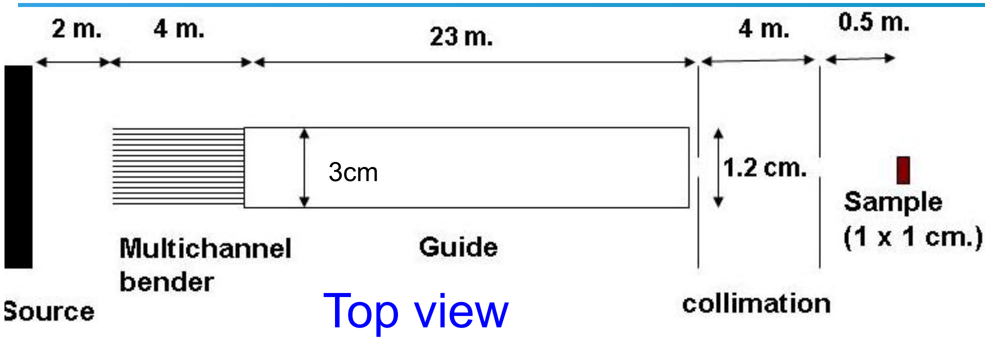
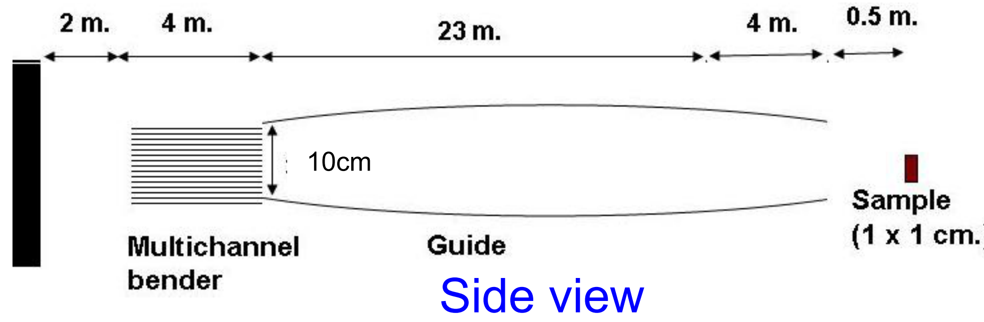


Fig. 4. Sample of the data obtained. The grey/green markers are the data obtained from standard measurements for the sample+solvent and a separate measurement of the solvent, respectively. After data treatment one obtains the black/red markers which are the standard SANS signal, and the signal with PA, respectively. The red/black lines are fits to the data, where for the PA data the background parameter is held to 0. The blue dotted line is the presumed background level from the fit of the standard data. The data shown is from a sample prepared by C. Sill (FZ-Jülich) who assisted the measurements. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

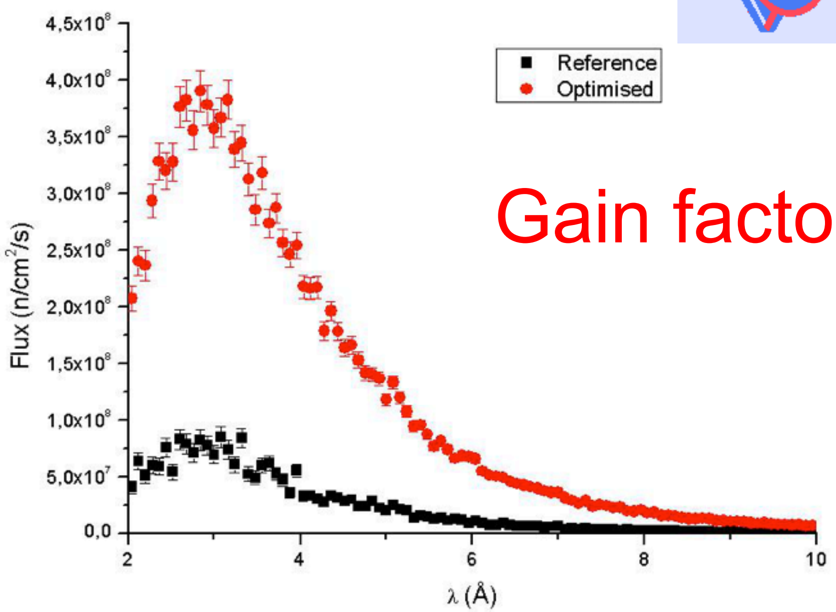
Focusing the beam



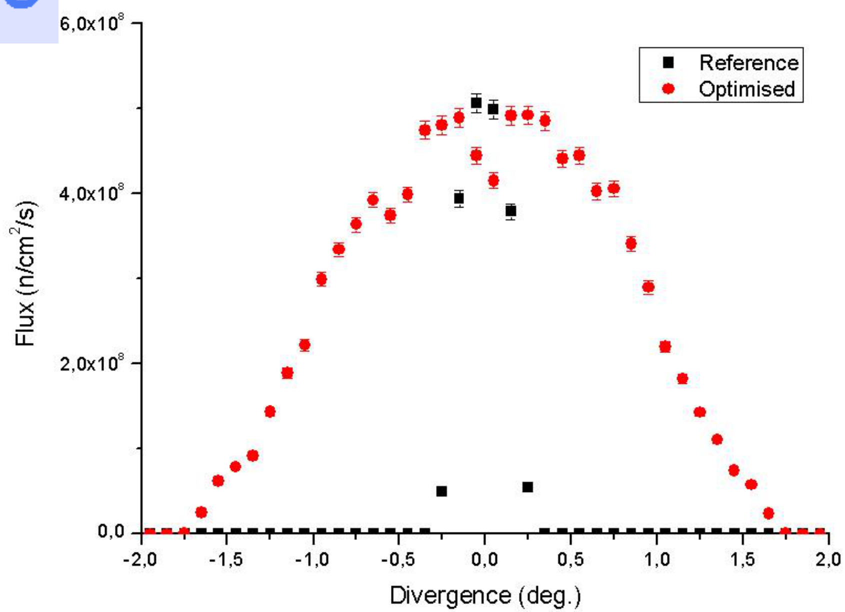


Reference: straight guide

Optimised: elliptic focusing guide

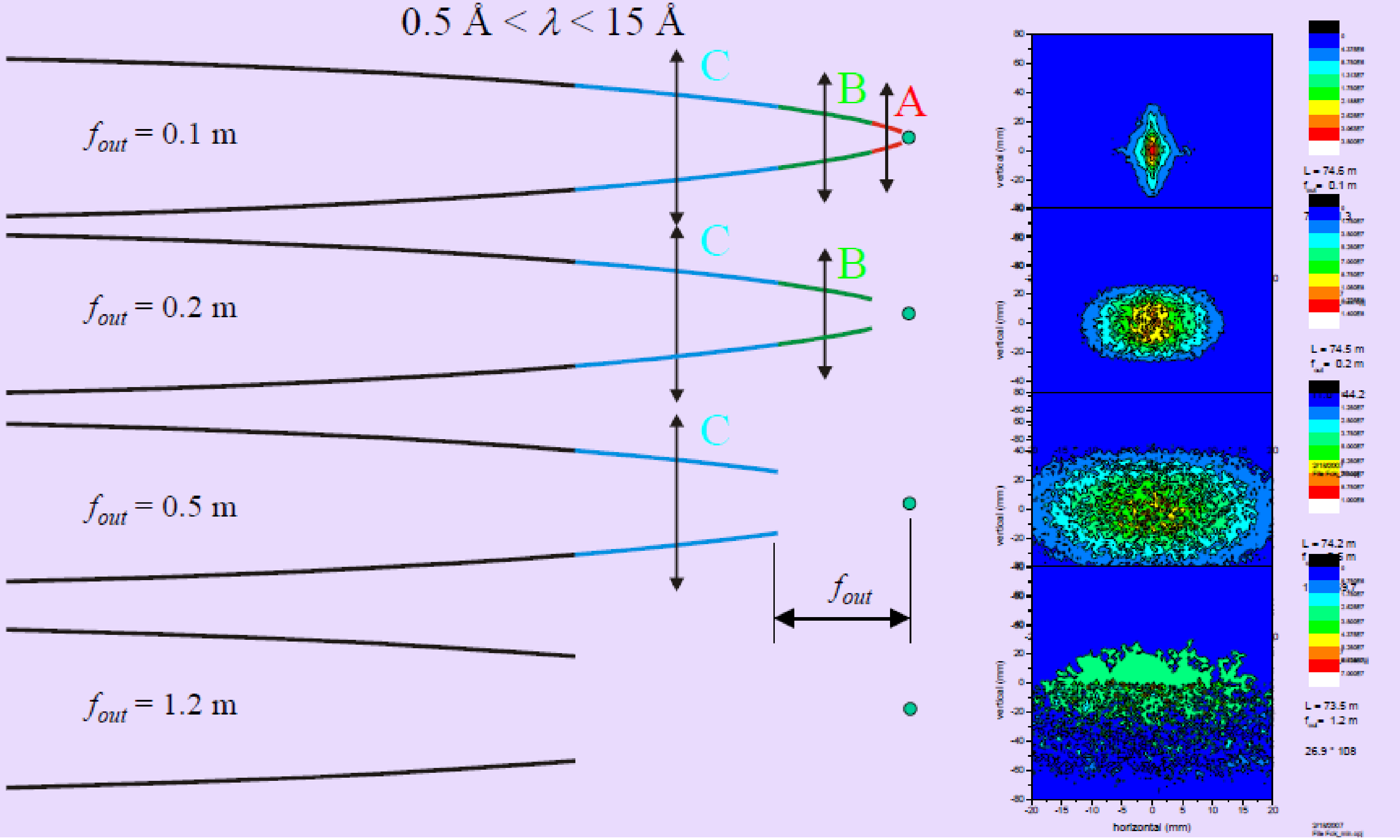


Gain factor ~4

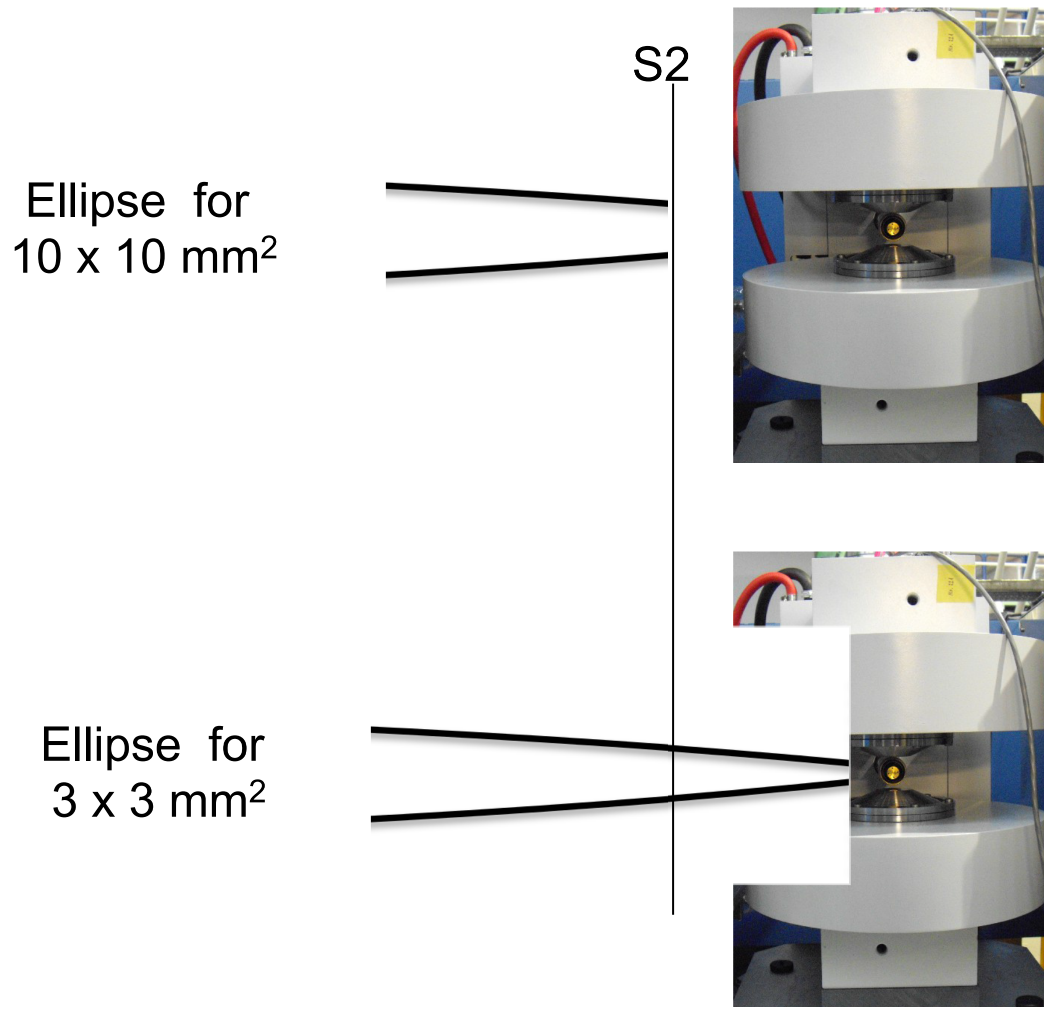


Focusing the beam

Liouville: strong focusing – large divergence of the beam: G ≈ D^2:
→ may be a problem for high resolution experiments!



Higher focus = less place



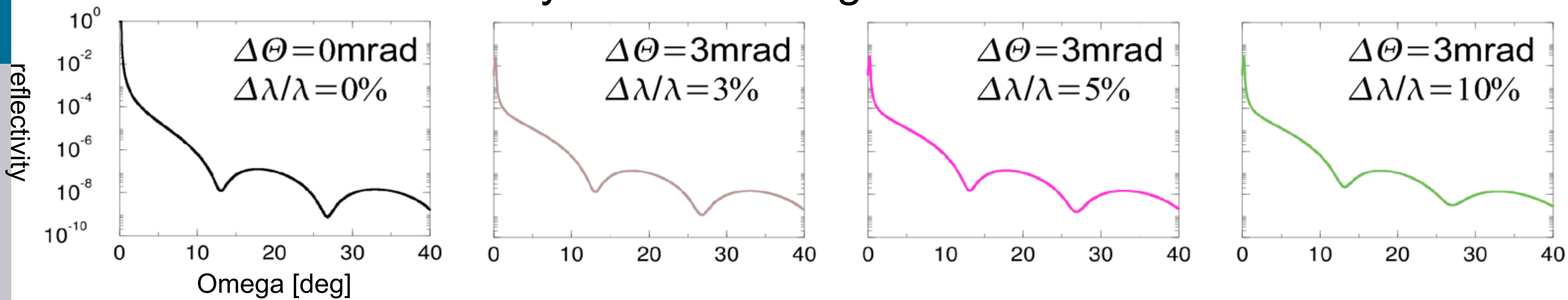
One has to find a compromise

Can't come close to the sample (SE)
=>no micro focusing

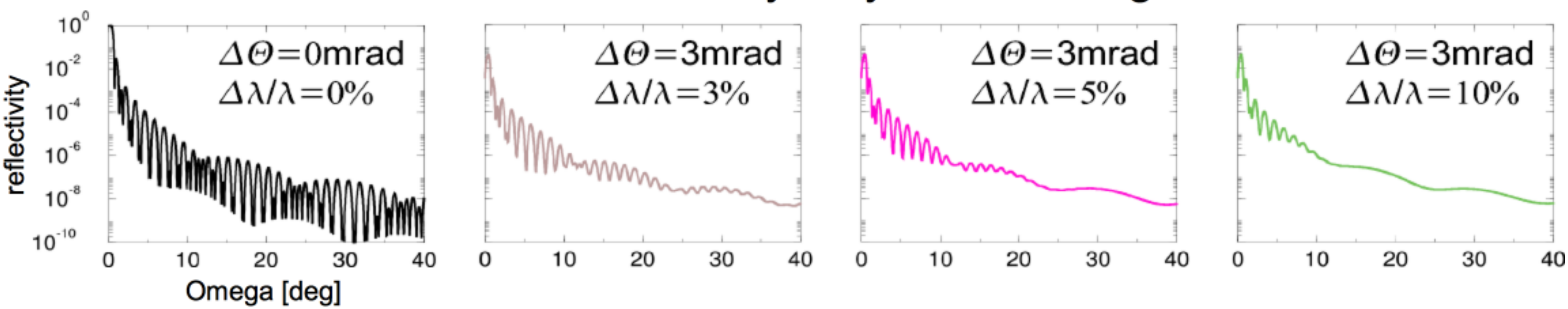
Wavelength resolution II

Specular reflectivity for Spin-up neutrons

10Å Fe-layer on a 10Å Ag substrate for 4.5Å

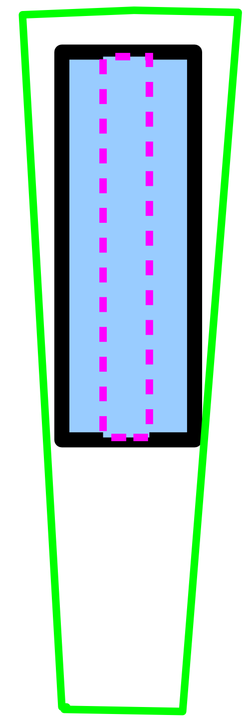


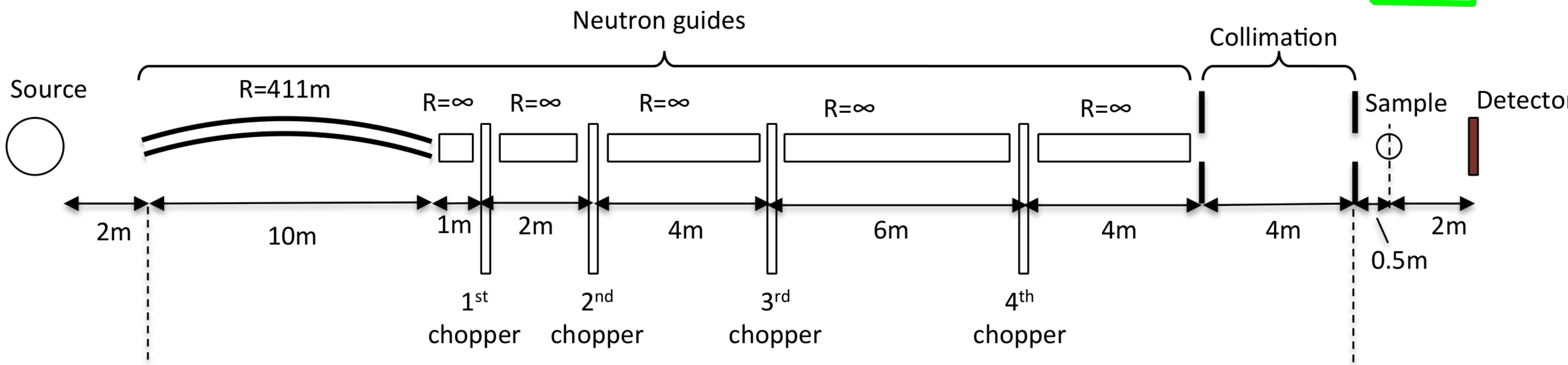
100Å Fe – 11Å Cr – 100Å Fe layer system on a Ag substrate



High resolution modes

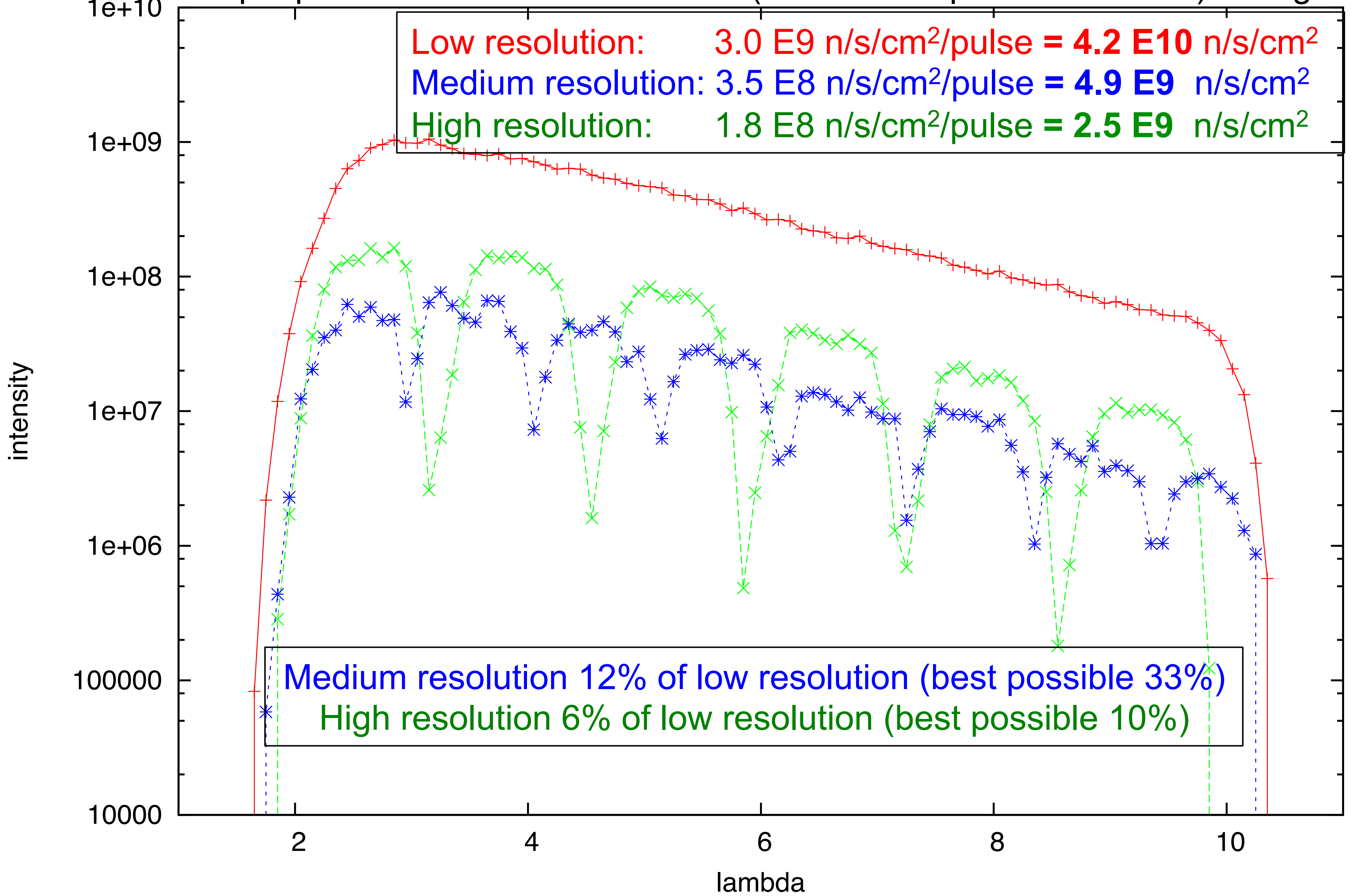
- For the beam we use (3 x 10 cm^2) the minimum angular aperture of the chopper has to be 10º
- In order to obtain the desired pulse length we need to rotate the chopper *n* times the source frequency and cover as much as possible of the wavelength band
- For a medium resolution mode ($\Delta\lambda/\lambda$ = 3%) the best option is a chopper with four symmetrically placed 10° apertures rotating 4 times faster than the source frequency
- For a high resolution mode ($\Delta\lambda/\lambda$= 1%) the best option is a chopper with two symmetrically placed 10° apertures rotating 10 times faster than the source frequency
- Intensity compared with the reference instrument



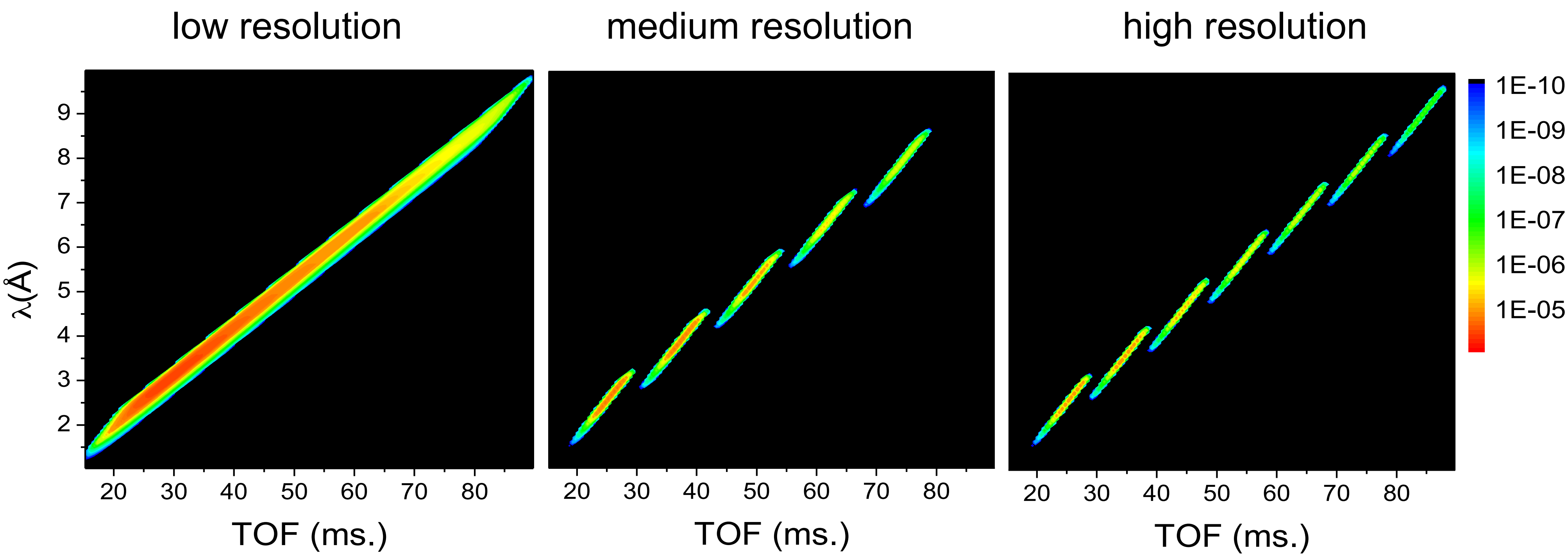


High resolution modes

Flux at the sample position for 1cm² and 2 mrad (Gaussian equivalent FWHM) divergence



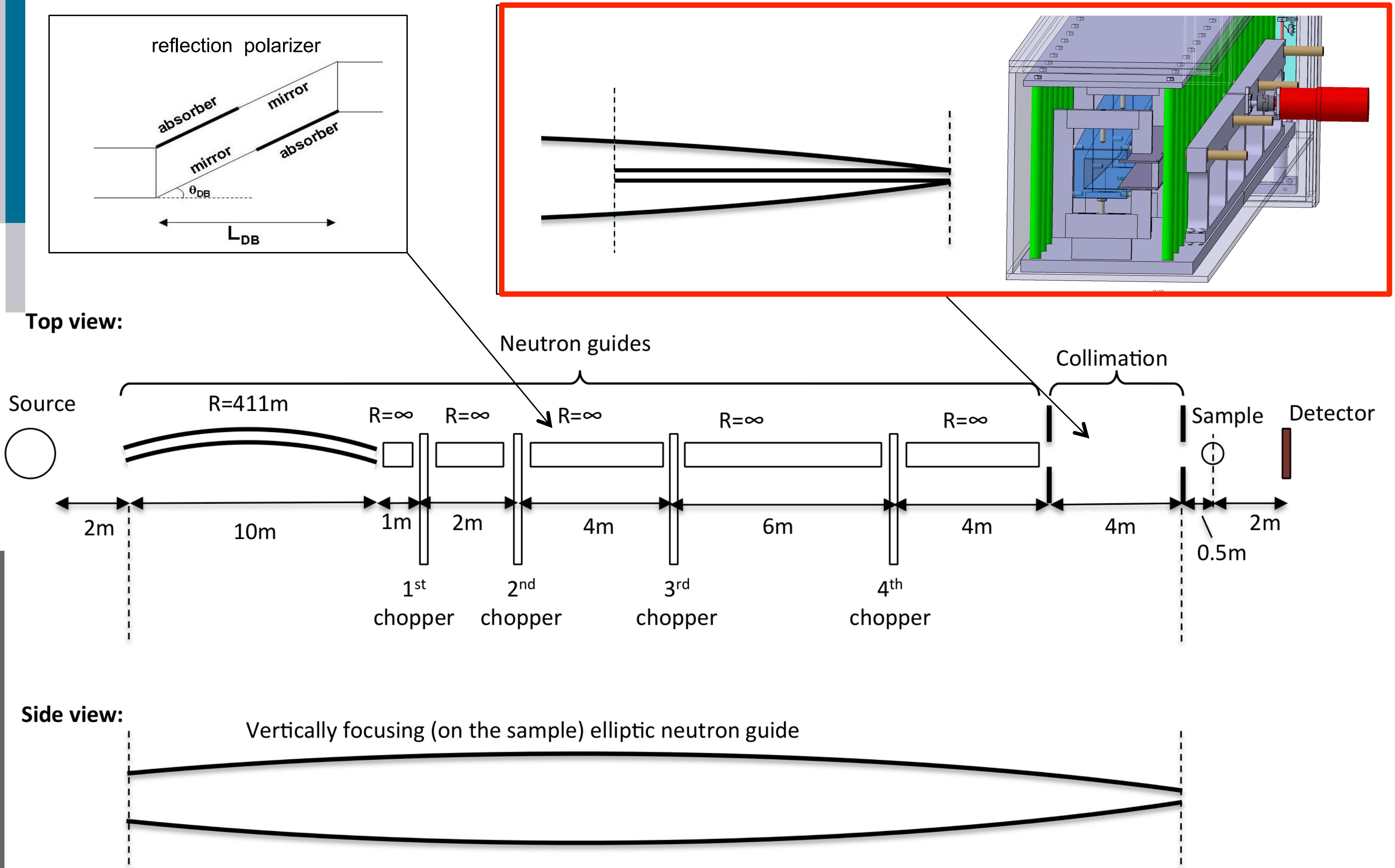
High resolution modes



- There is no time-of-flight overlap

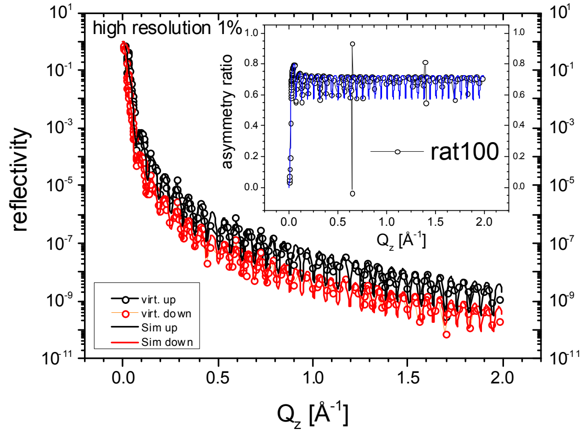
	Δλ/λ at 25 ms. (%)	**Δλ/λ at 74 ms. (%)**
Low-res	13.3	4.4
Mid-res	4.0	1.3
Hi-res	1.6	0.5

Layout of the intrsument

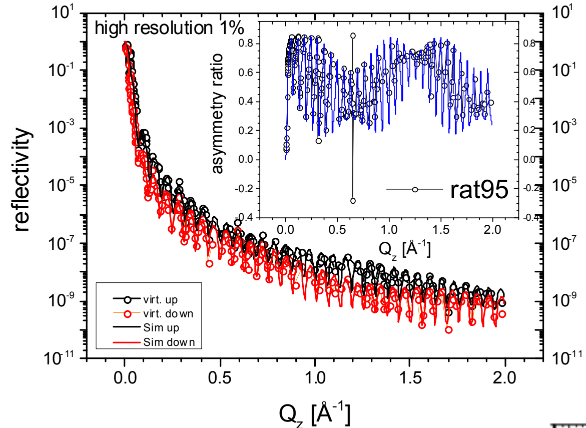


Virtual Simulations

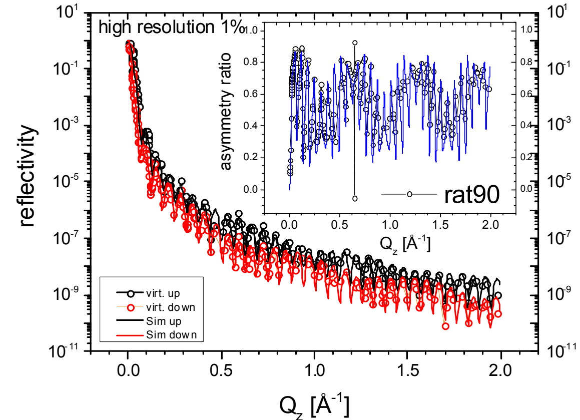
Simulations: Fe 100 on Si Substrate

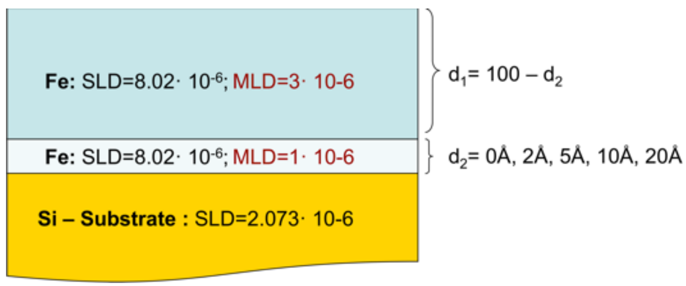


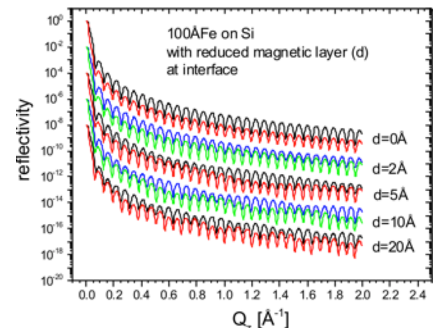
Simulations: Fe 95 - 5 on Si Substrate



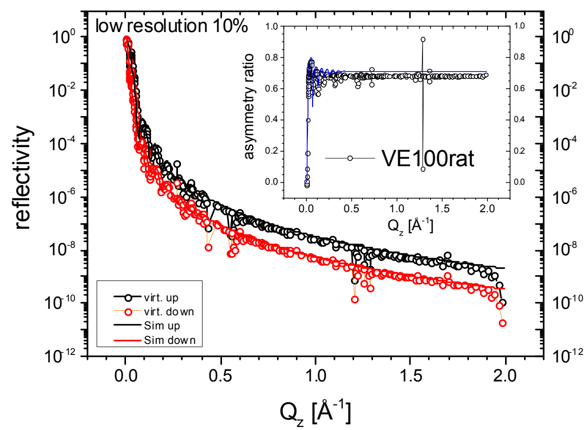
Simulations: Fe 90 - 10 on Si Substrate



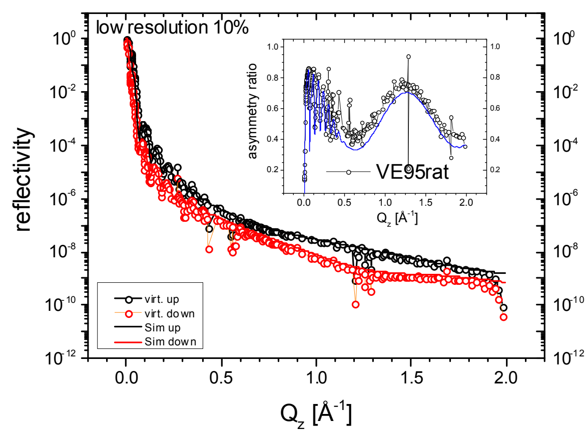




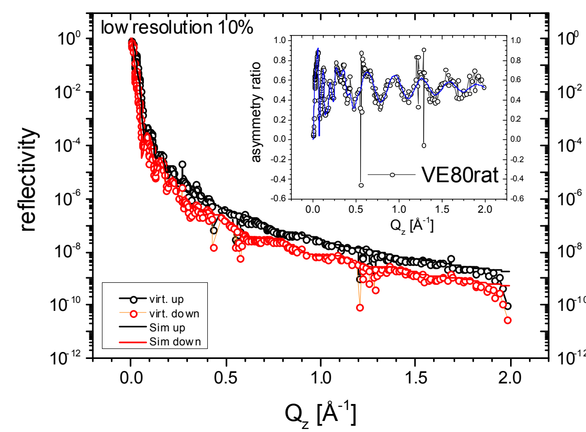
Simulations: Fe 100 on Si Substrate



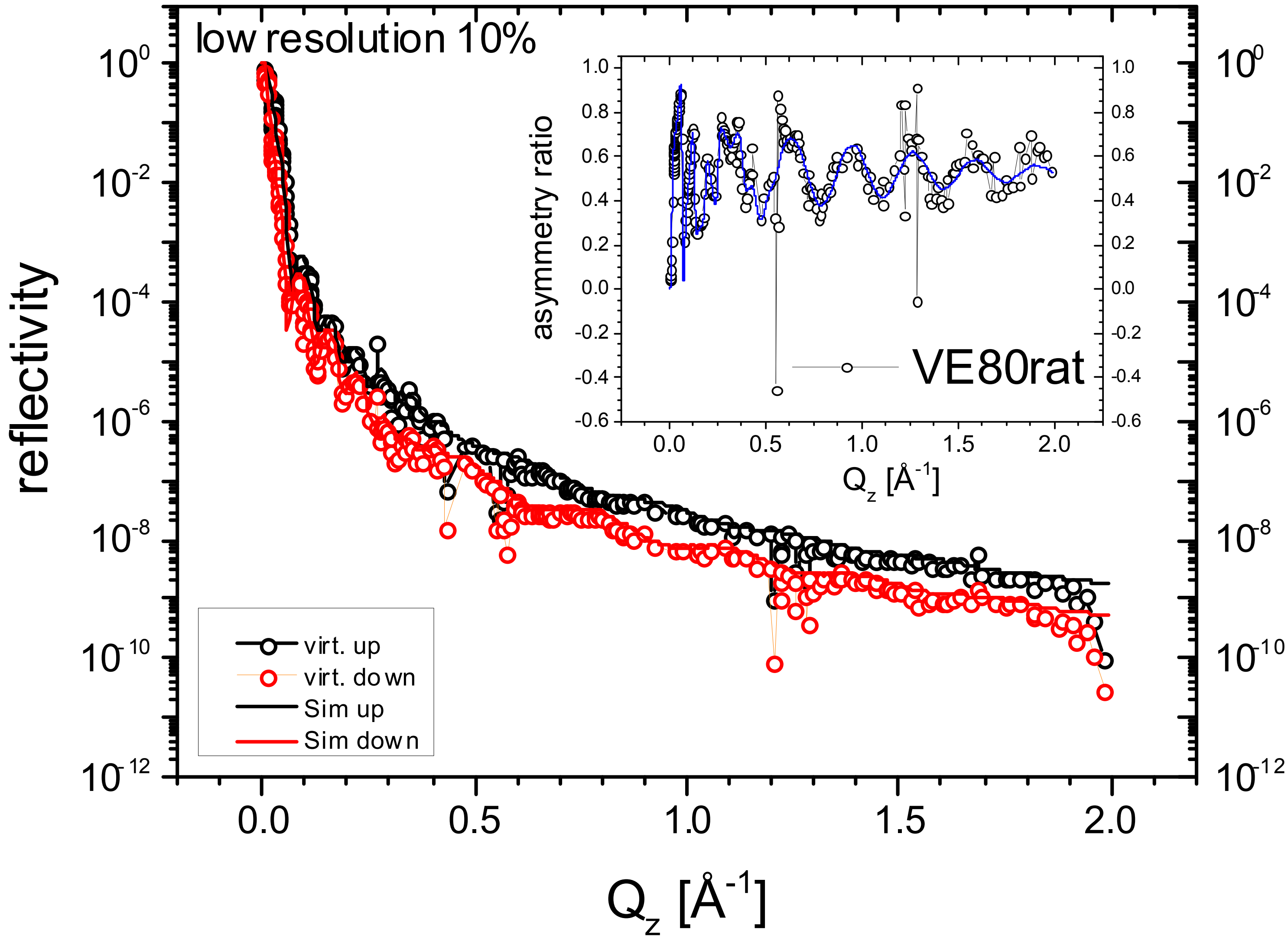
Simulations: Fe 95 - 5 on Si Substrate



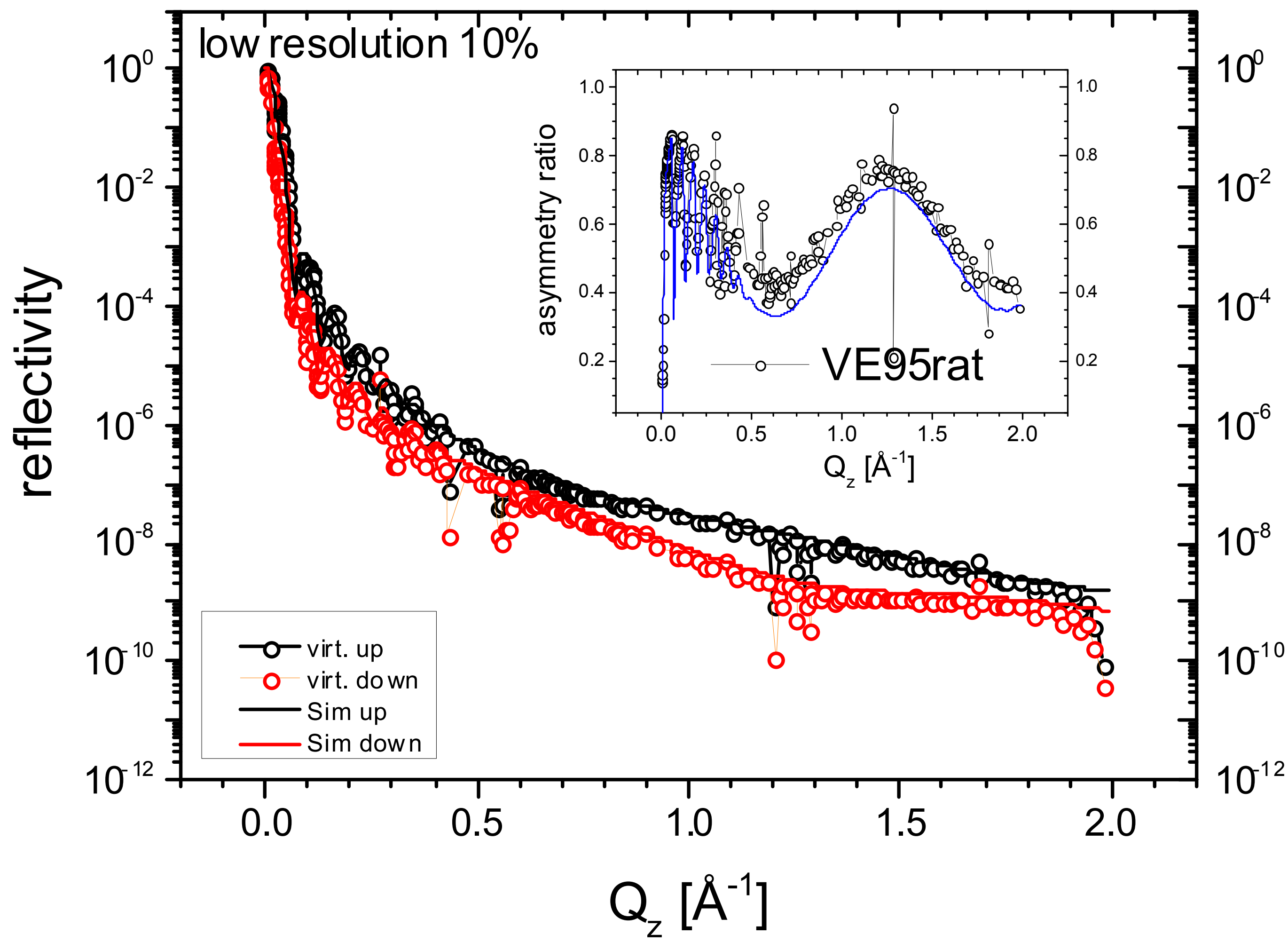
Simulations: Fe 80 - 20 on Si Substrate



Simulations: Fe 80 - 20 on Si Substrate



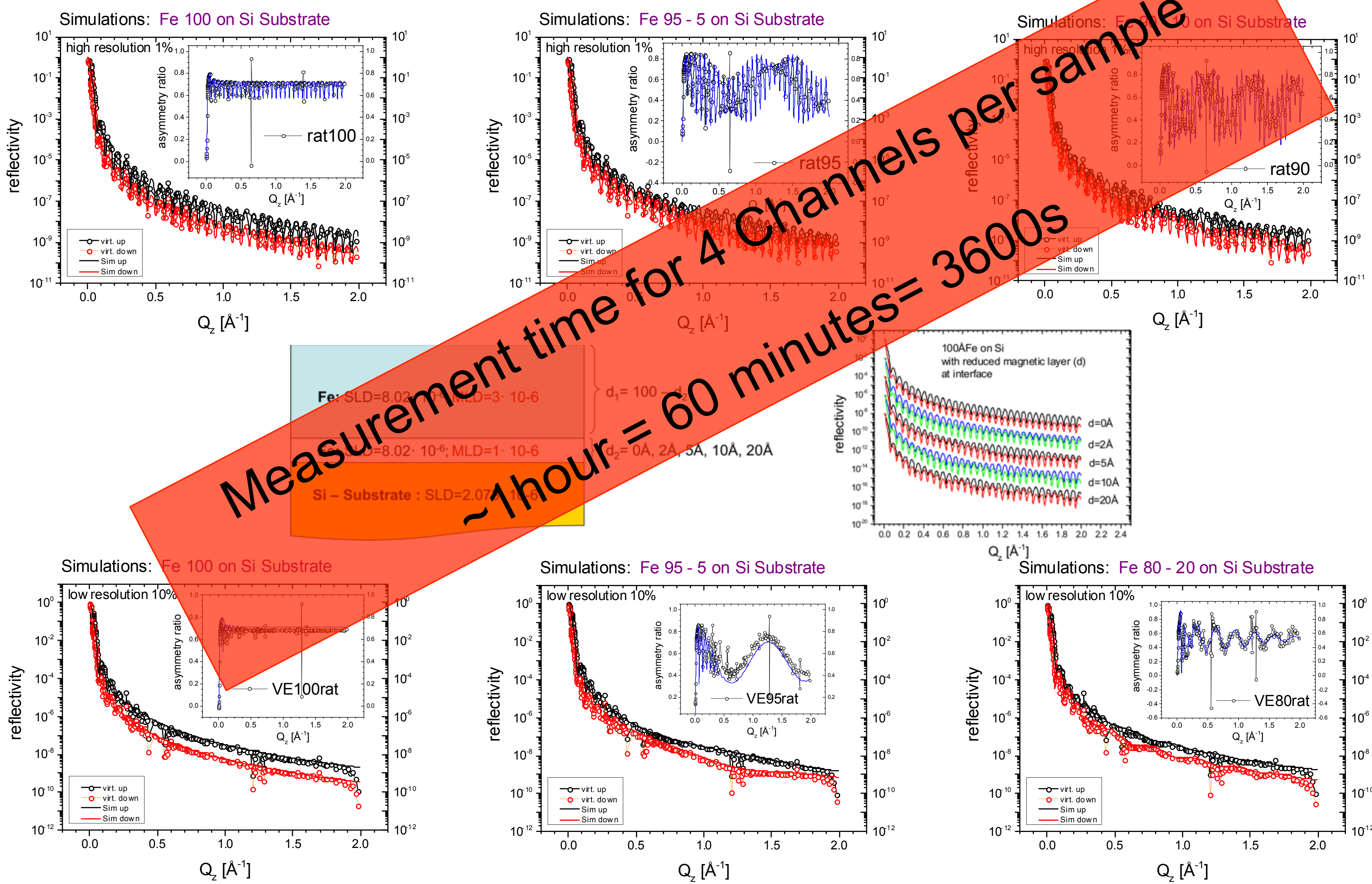
Simulations: Fe 95 - 5 on Si Substrate



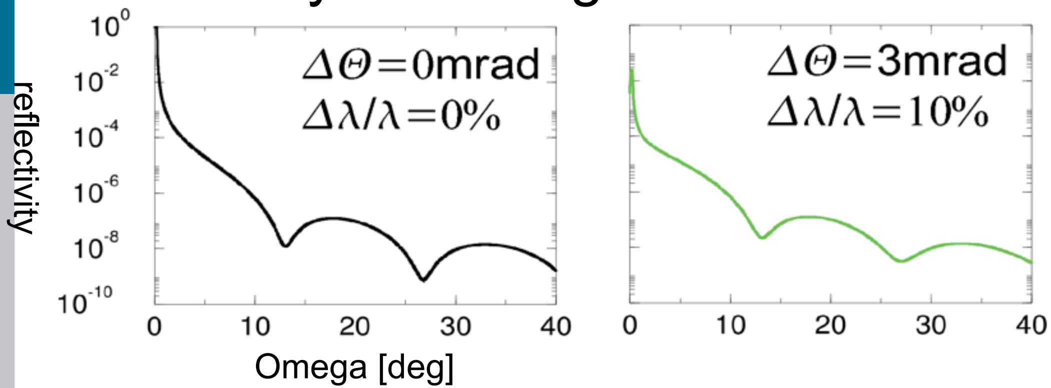
Virtual Simulations

Measurement time for 4 Channels per sample

~1hour = 60 minutes= 3600s



10Å Fe-layer on a Ag substrate for 4.5Å



10^{0}

10^{-2}

10^{-4}

10^{-6}

10^{-8}

10^{-10}