



*The Abdus Salam
International Centre for Theoretical Physics*



1866-2

School on Pulsed Neutrons: Characterization of Materials

15 - 26 October 2007

Surfaces and Thin Films

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School on Pulsed Neutron Sources
Trieste - Italy, 15 - 26 October 2007



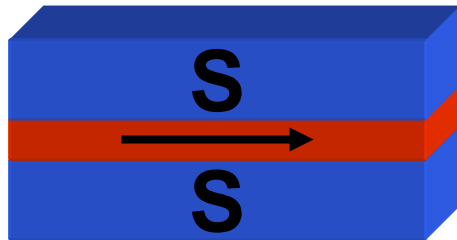
Surfaces, Interfaces and Thin Films

For fundamental properties in the area of
magneto- and spintronics

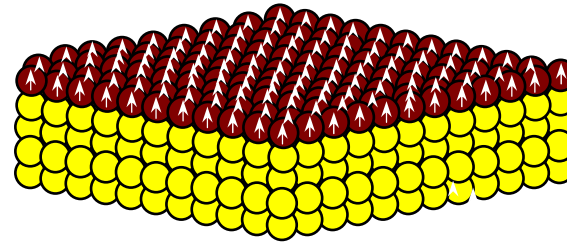
GMR heterostructures



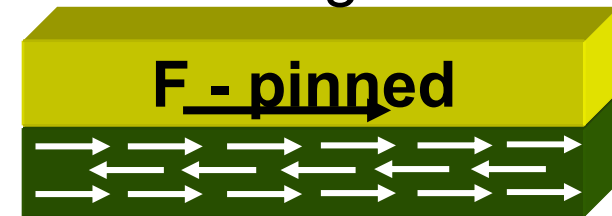
Proximity effects and tunneling



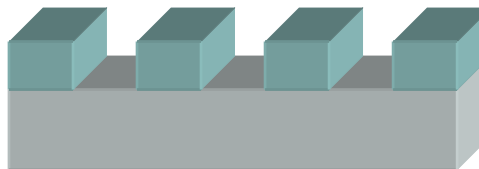
Magnetic films



Exchange bias



Lateral structures



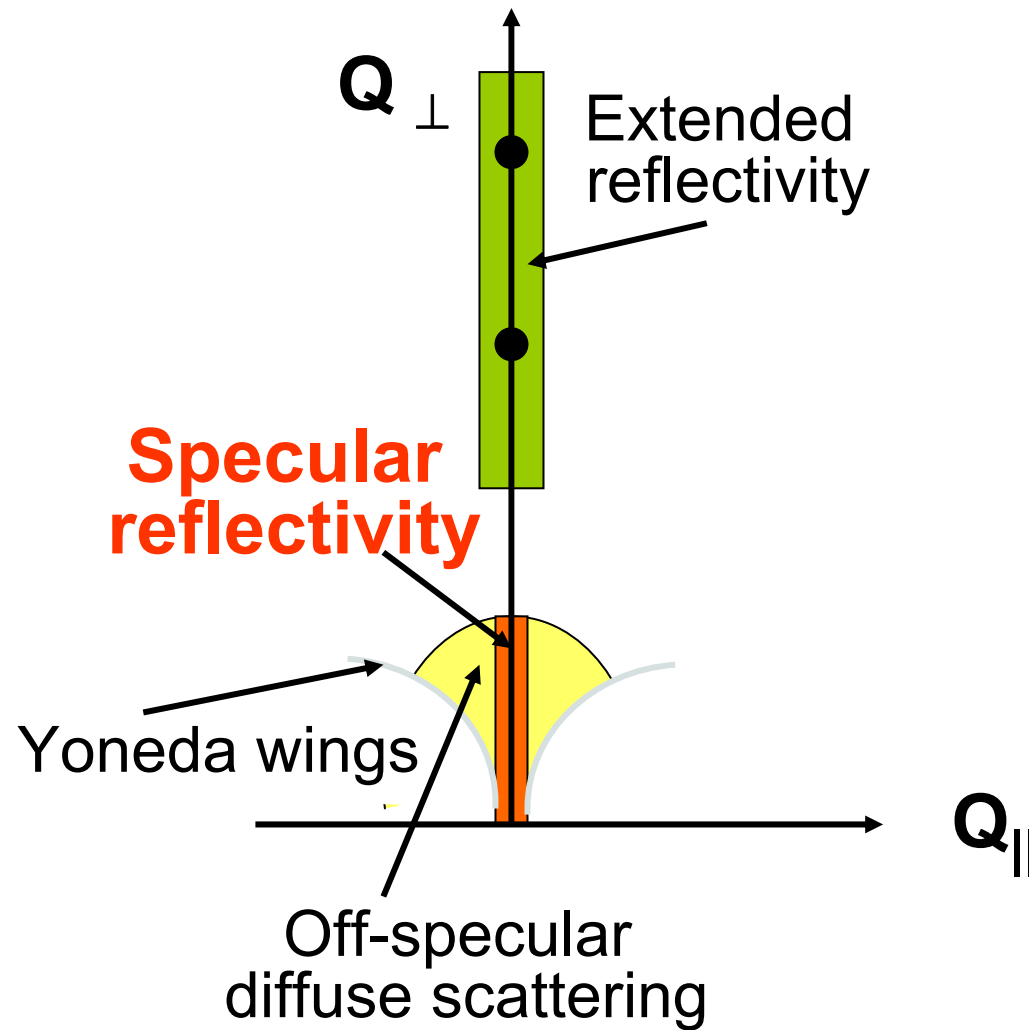
Content

- 1. X-ray reflectivity**
- 2. Neutron reflectivity**
- 3. Polarized neutron reflectivity (PNR)**
 - Methods
 - Instrumentation
 - Examples
- 4. Case study: do we still need neutrons?**

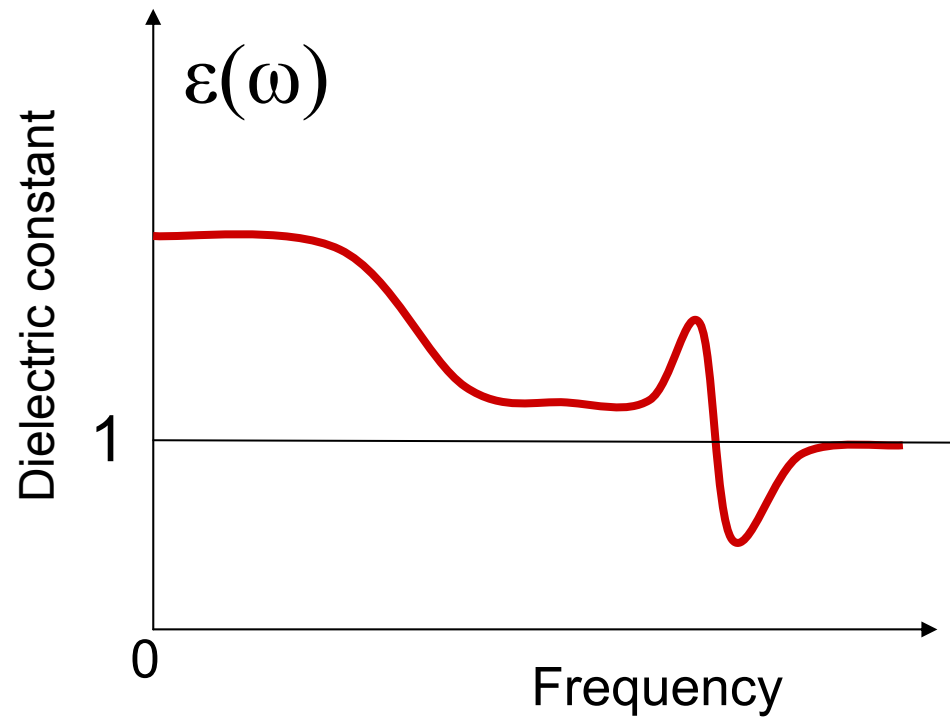


1. X-ray and neutron reflectivity

Specular reflectivity basics



Index of refraction for EM waves



$$\sqrt{\epsilon(\omega)} = n(\omega) = \text{refractive index} \Rightarrow \text{optics}$$



X-ray refractive index

Refractive Index:

$$n^2(\omega) = 1 + \frac{4\pi\rho_A e^2}{m_e} \sum_i \frac{f_i}{\omega_i^2 - \omega^2 + i\gamma}$$

For x-ray's, the refractive index is always smaller than 1:

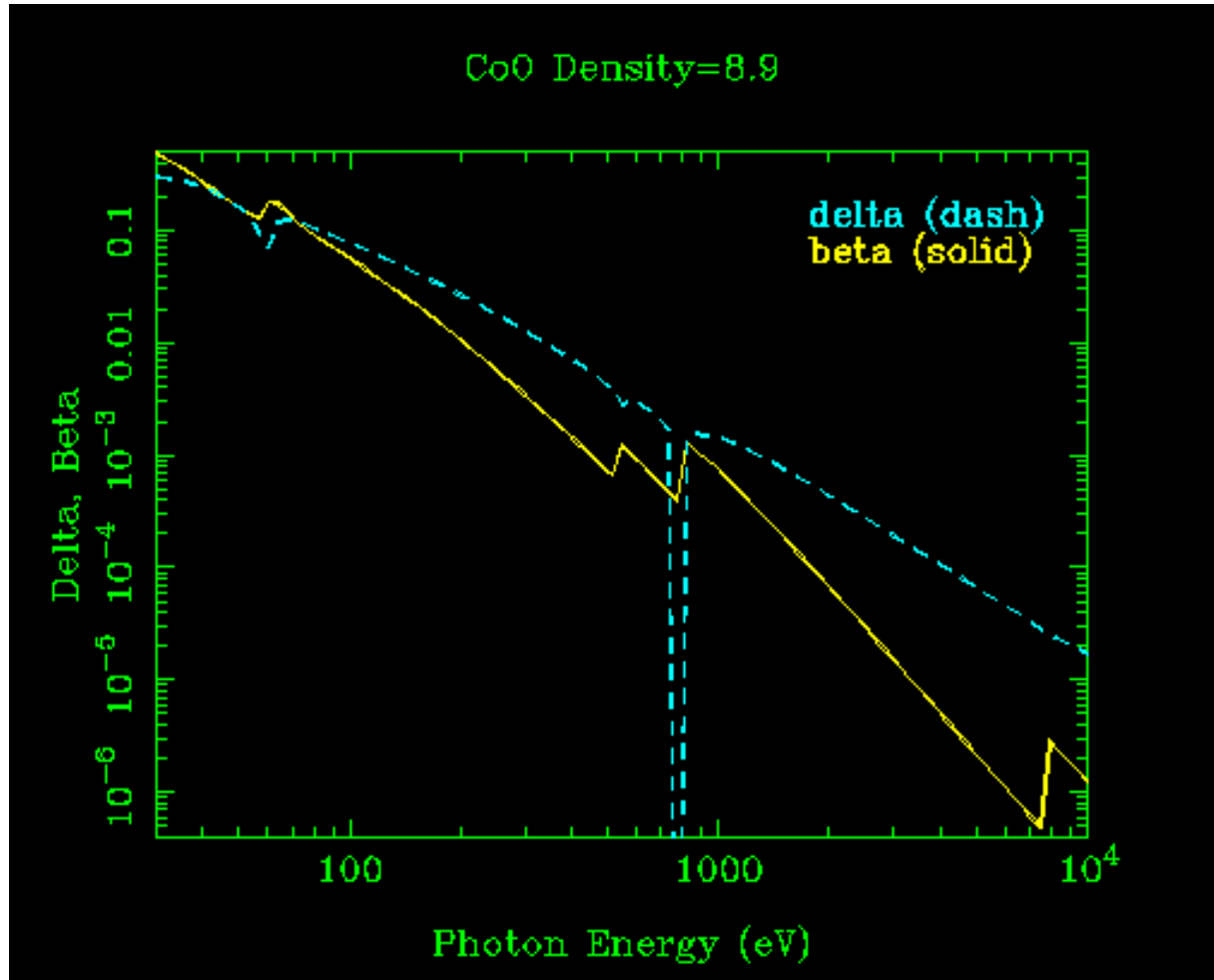
$$n(\lambda) = 1 - \frac{r_0}{2\pi} \rho_e \lambda^2$$
$$\approx 1 - 10^{-5} < 1$$

Adding dispersion and absorption correction:

$$n(Q_z) = 1 - \frac{2\pi r_0}{k_0^2} \rho_A [f(Q_z) + \Delta f] - \frac{i\mu}{2k_0}$$
$$= 1 \quad \quad \quad -\delta \quad \quad \quad -i\beta$$
$$\cong 1 \quad \quad \quad - \quad 10^{-5} \quad \quad \quad < \quad 1$$



Plot of δ and β for CoO



http://www-cxro.lbl.gov/optical_constants/getdb2.html 7

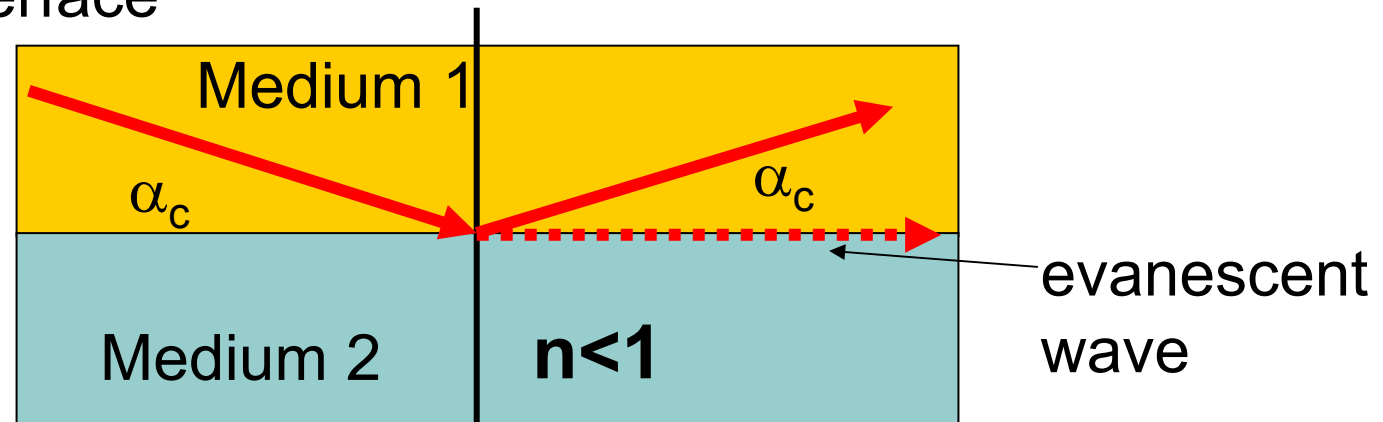


Critical angle for total reflection

Since $n < 1$, total reflection occurs at:

$$\frac{\cos \alpha_c}{\cos 0} = \cos \alpha_c = n$$

For all angles $\alpha < \alpha_c$ the wave can not penetrate into the medium, but at α_c there is an evanescent wave travelling along the interface



Critical Scattering Vector

Scattering vector is defined as:

$$Q = \frac{4\pi}{\lambda} \sin \alpha = 2k \sin \alpha$$

Accordingly the critical scattering vector is:

$$Q_c = \frac{4\pi}{\lambda} \sin \alpha_c = 2k \sqrt{1 - \cos^2 \alpha_c} = \sqrt{4k^2 (1 - n^2)}$$
$$\cong \sqrt{4k^2 2\delta} = \sqrt{16\pi r_0 \rho_e} \quad \boxed{Q_c \propto \sqrt{\rho_e}}$$

The critical scattering vector is no more a function of the wavelength. It is entirely determined by the property of the material and in particular by the electron density ρ_e .

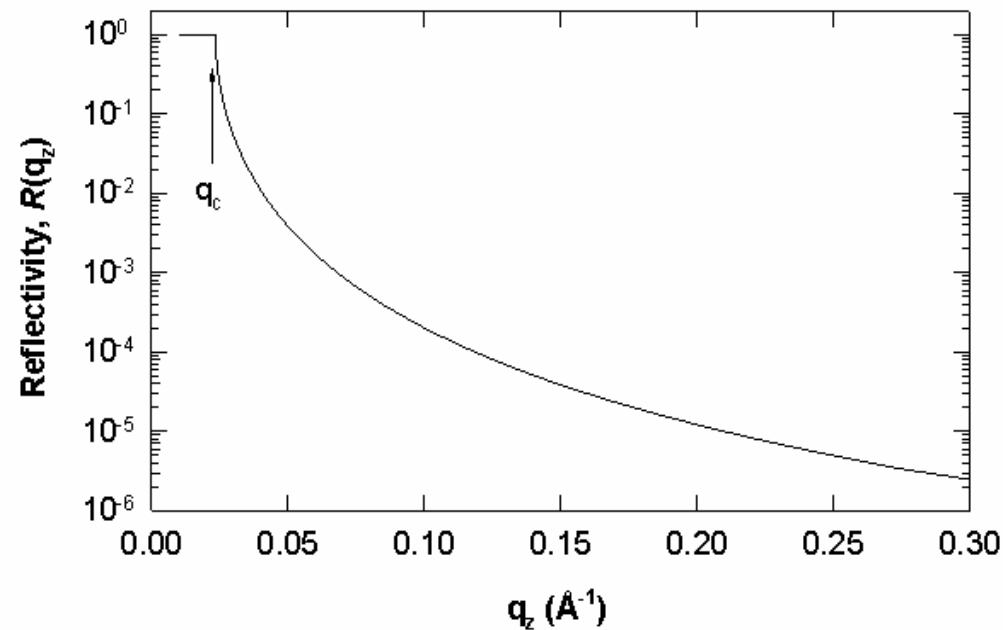
Fresnel reflectivity

For $Q_z < Q_c$: $R = 1$,

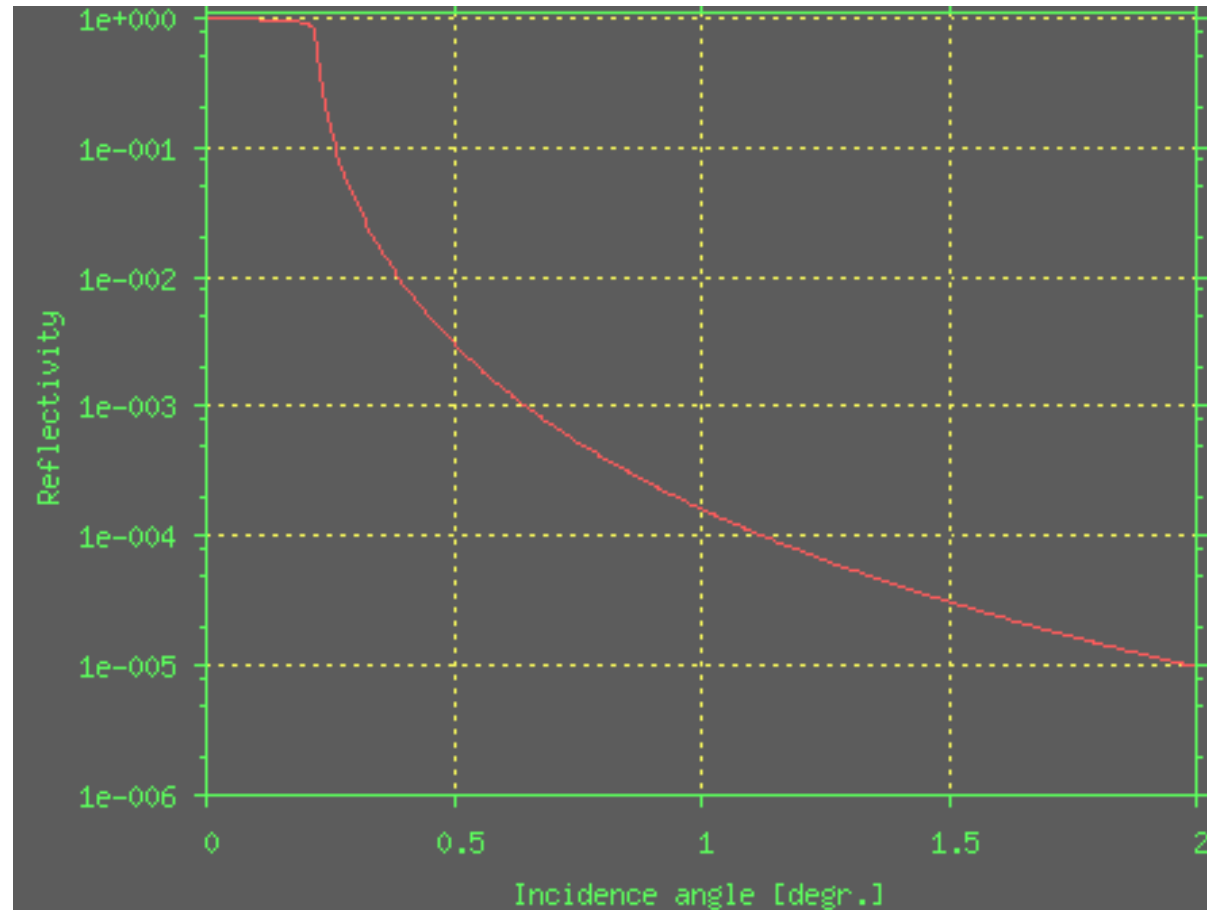
For $Q_z > Q_c$: $R = R_f$

The reflectivity drops with Q^4 , for scattering vectors $Q \gg Q_c$.

This applies for perfectly flat interfaces.



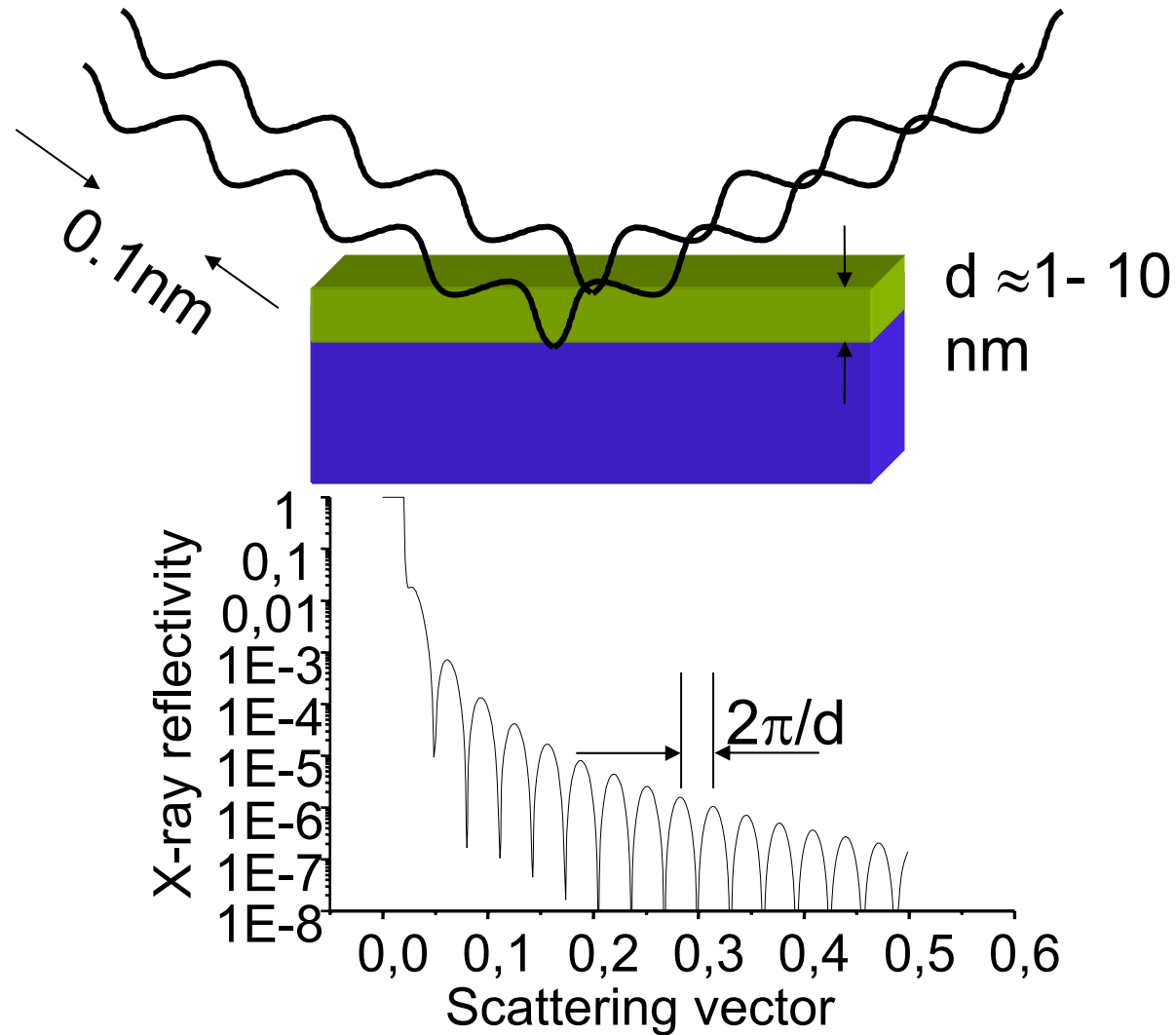
Fresnel reflectivity for Si



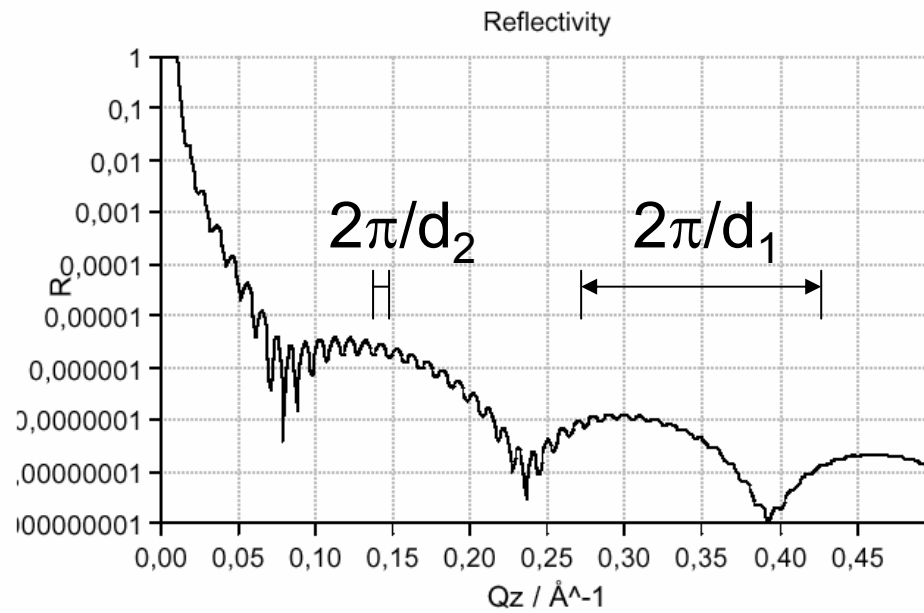
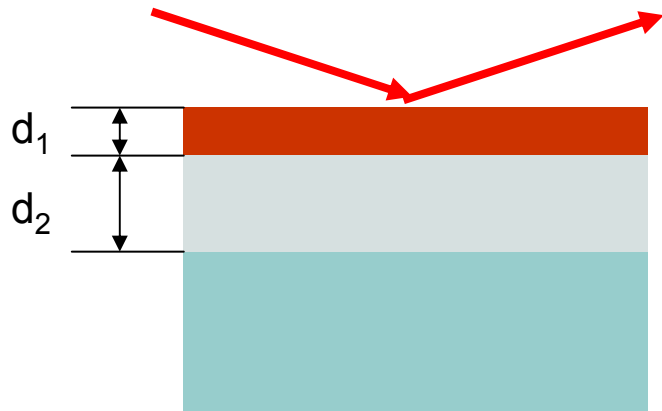
<http://sergey.gmca.aps.anl.gov/>



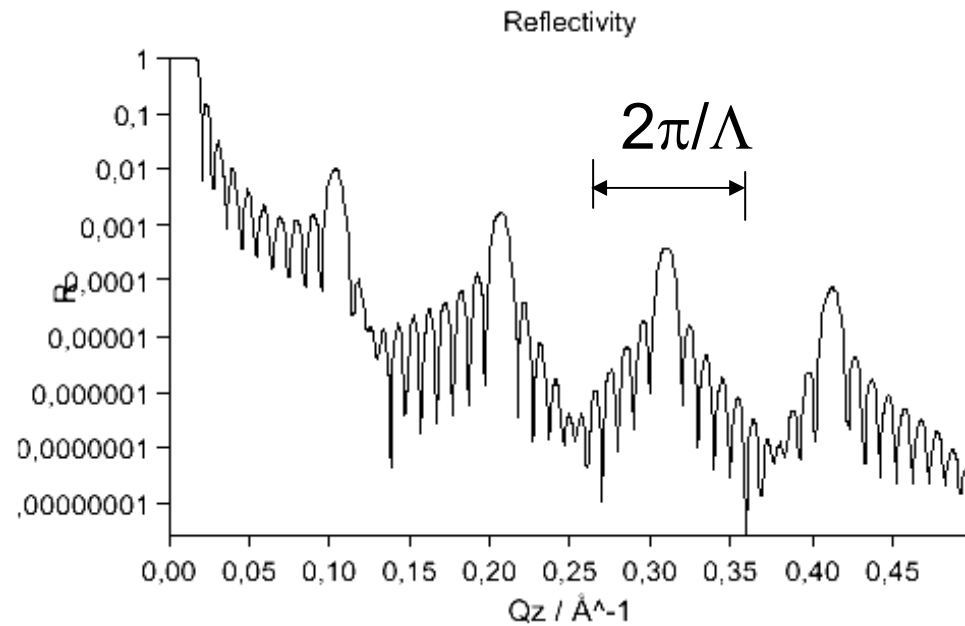
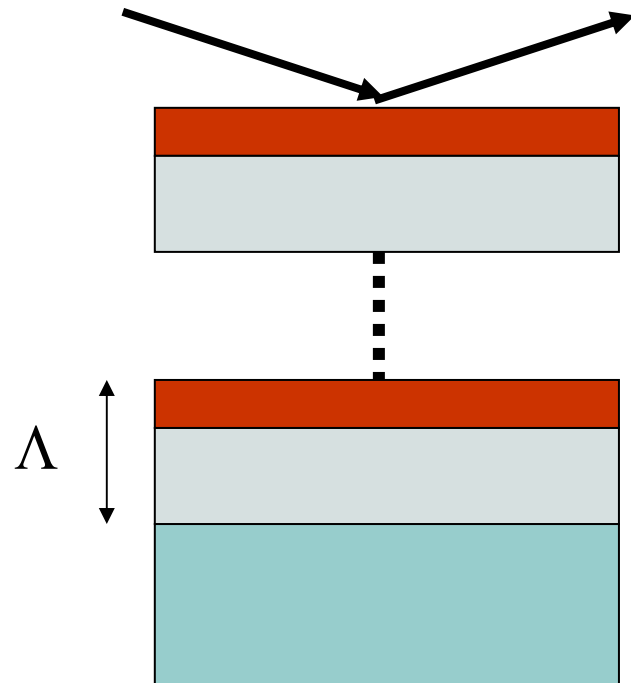
Reflectivity from a thin layer: Kiessig fringes



Reflectivity from a double layer

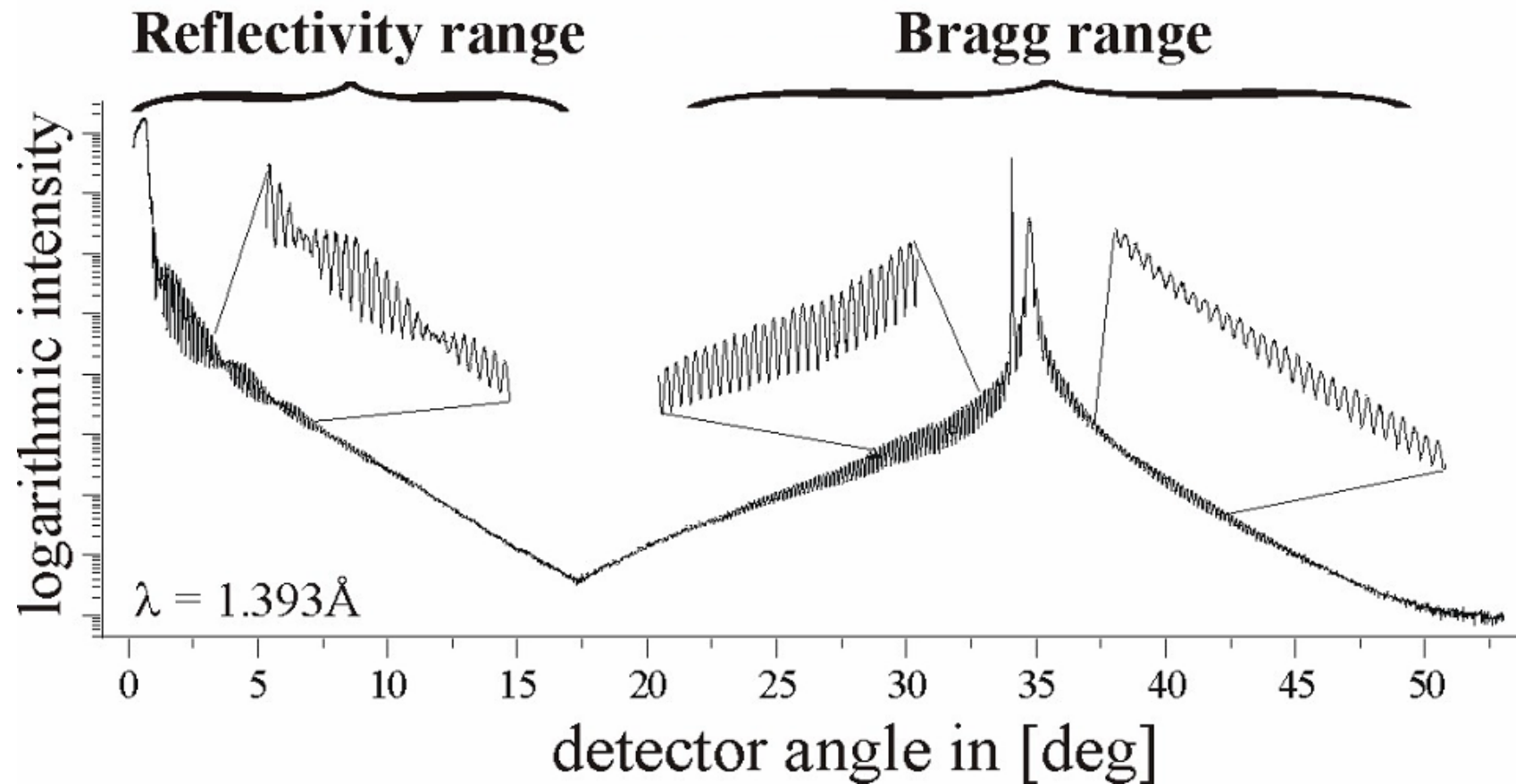


Reflectivity from a multilayer

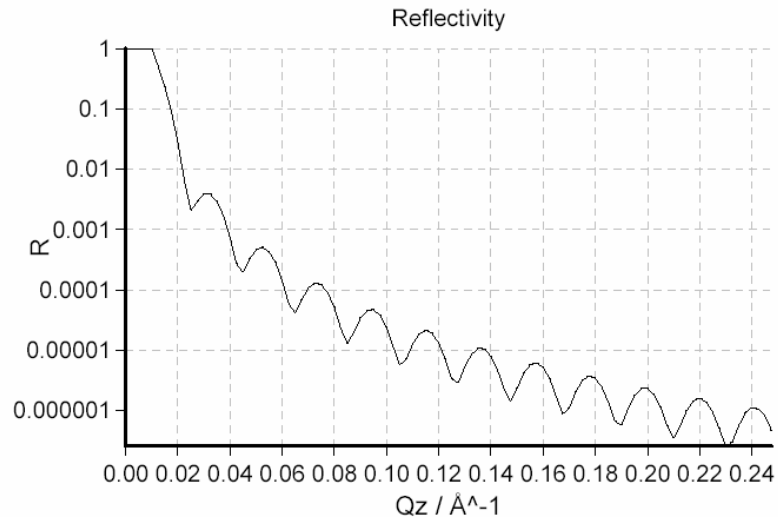


$$Q_l = \sqrt{Q_c^2 + l^2 \left(\frac{2\pi}{\Lambda} \right)^2}$$

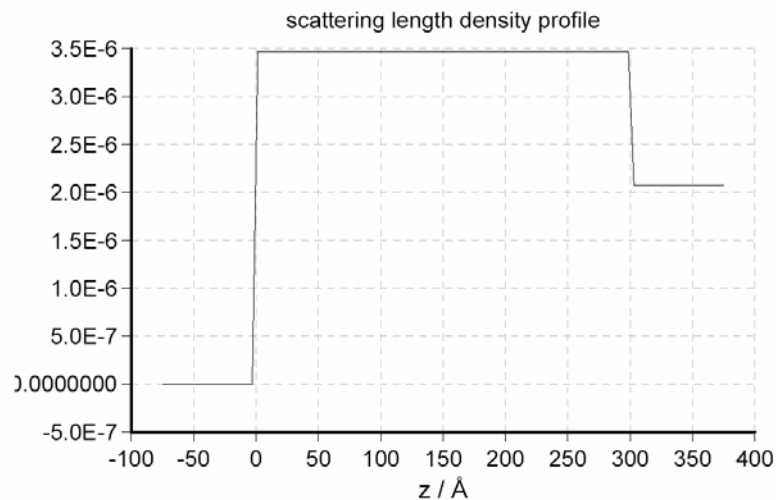
Reflectivity and Bragg range



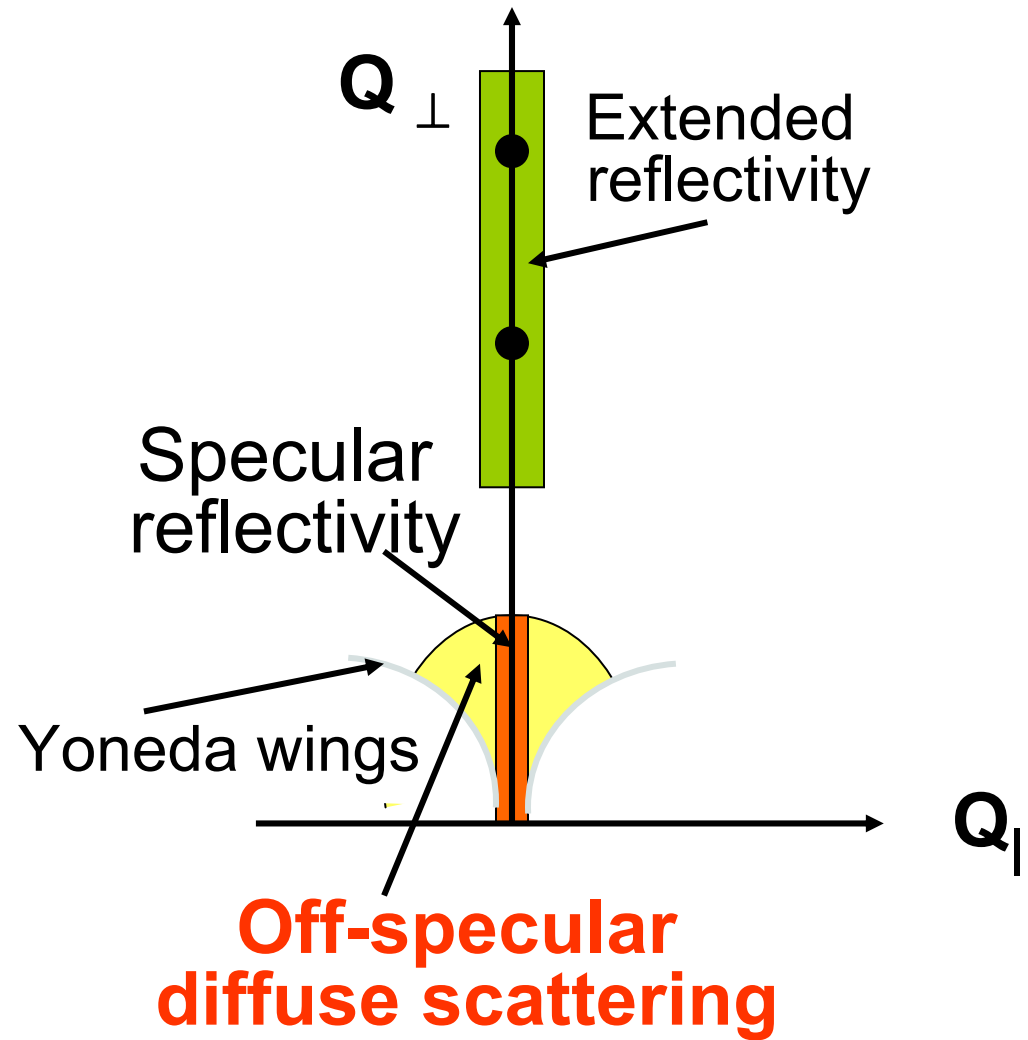
Electron density profile from reflectivity data



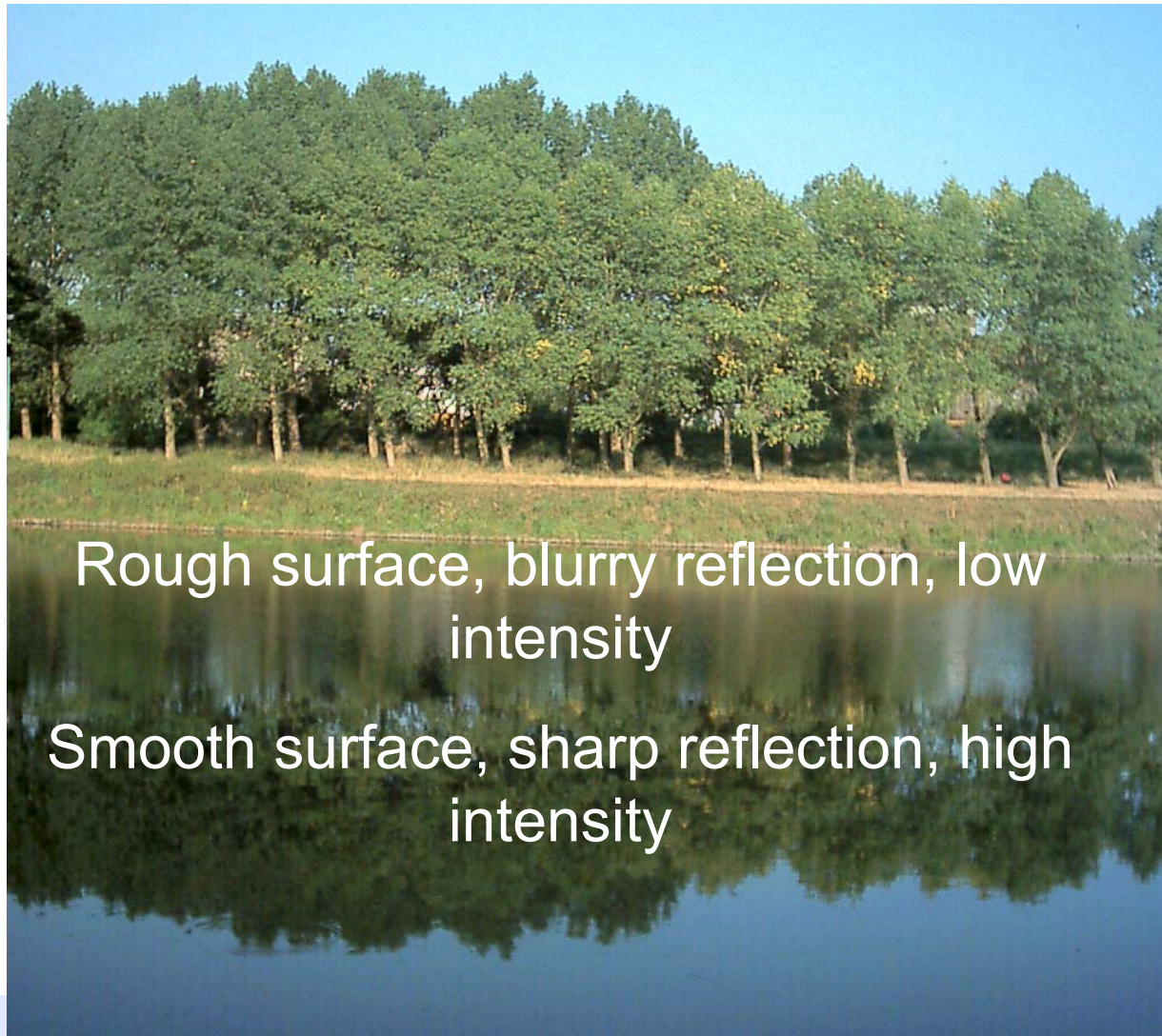
Back transformation from reflectivity data to electron densities and thickness profiles is the ultimate goal. However, the back transformation is not always uniquely possible.



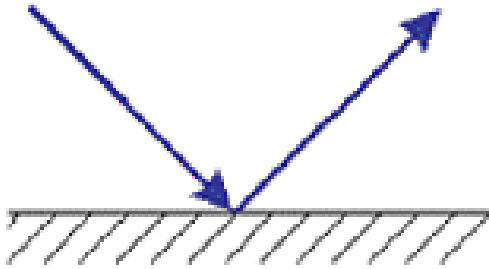
Reflectivity with surface roughness



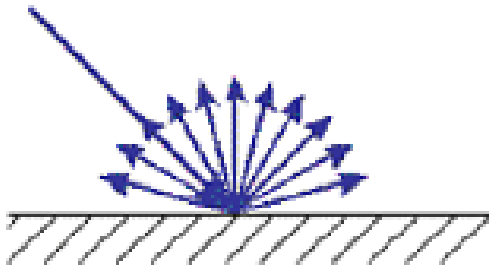
Smooth and rough surfaces



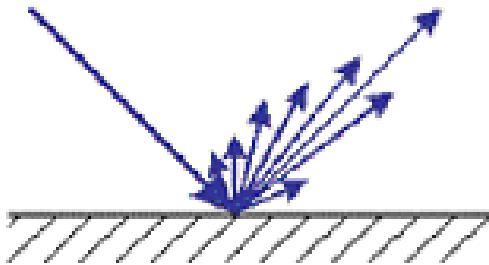
Off-specular scattering from rough interfaces



Perfectly specular surface,
100% reflection, mirror image



Perfectly rough surface,
100% diffuse scattering, projector wall



Partially reflecting and scattering from
rough surface

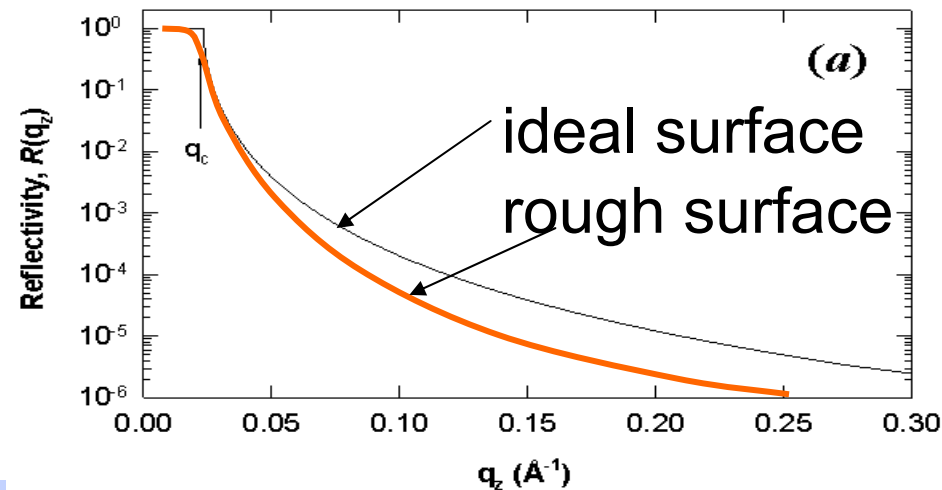
Reflectivity of rough surface

Master formular yields for a Gaussian roughness a damped Fresnel reflectivity:

$$R(Q_z) = R_F(Q_z) \exp(-Q_z^2 \sigma^2)$$

$R_F(Q_z)$ is the Fresnel reflectivity of the ideal surface.

Roughness adds a damping factor, similar to the Debye-Waller factor:



Diffuse Scattering

Scattering function in the Born approximation:

$$S(\vec{Q}) = \int \langle \rho(0) \rho(R) \rangle e^{i\vec{Q} \cdot \vec{R}} d^3 R$$

Pair correlation function:

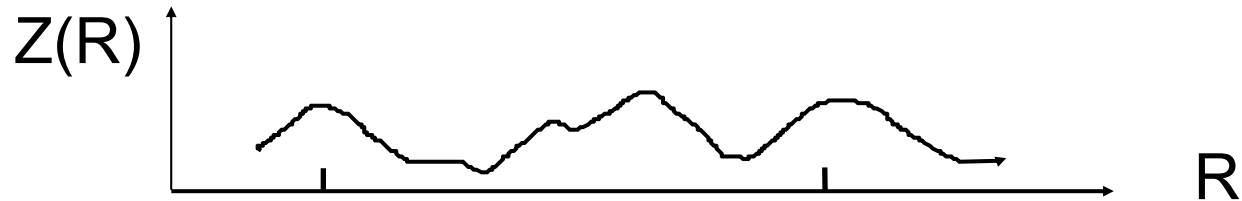
$$\begin{aligned} G(\vec{R}) &= \langle (\rho(0) - \langle \rho(0) \rangle) (\rho(\vec{R}) - \langle \rho(\vec{R}) \rangle) \rangle \\ &= \langle \rho(0) \rho(\vec{R}) \rangle - \langle \rho(0) \rangle \langle \rho(\vec{R}) \rangle \\ &= \langle \rho(0) \rho(\vec{R}) \rangle - \langle \rho(0) \rangle^2 \end{aligned}$$

Inserting:

$$\begin{aligned} S_{tot}(\vec{Q}) &= \underbrace{\langle \rho(0) \rangle^2 \int e^{i\vec{Q} \cdot \vec{R}} d^3 R}_{\text{Specular Reflection}} + \underbrace{\int C(\vec{R}) e^{i\vec{Q} \cdot \vec{R}} d^3 R}_{\text{Diffuse Scattering}} \\ &= S_{spec}(\vec{Q}) + S_{diff}(\vec{Q}) \end{aligned}$$



Height-height correlation function



Height-height correlation function for a single self-affine, fractal surface:

$$C(R) = \langle z(0)z(R) \rangle = \sigma^2 \exp[-(R / \xi)]^{2h}$$

σ = rms roughness

ξ = cut-off length:

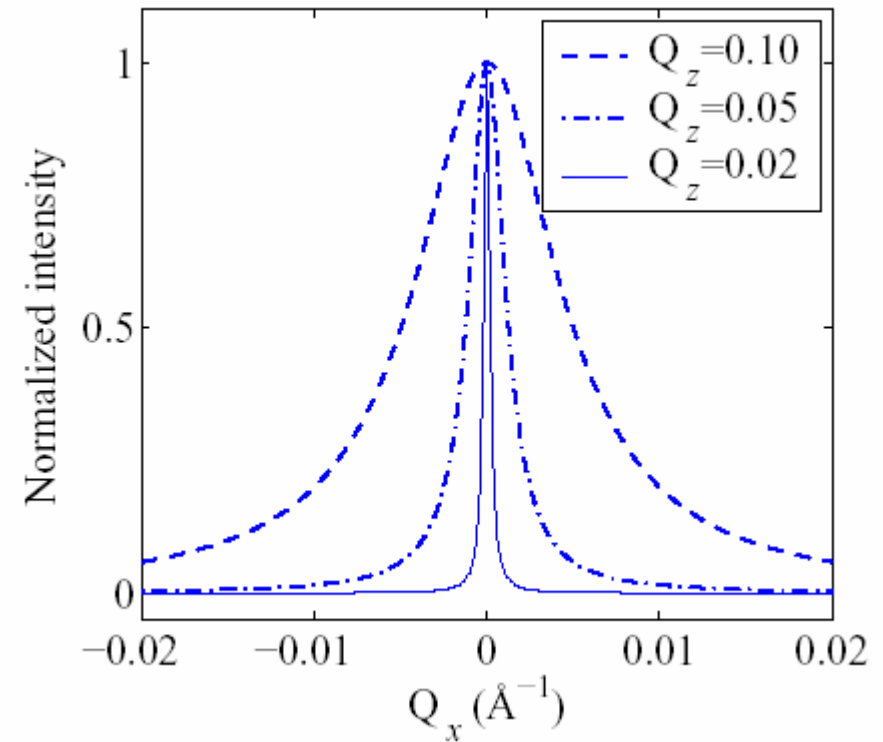
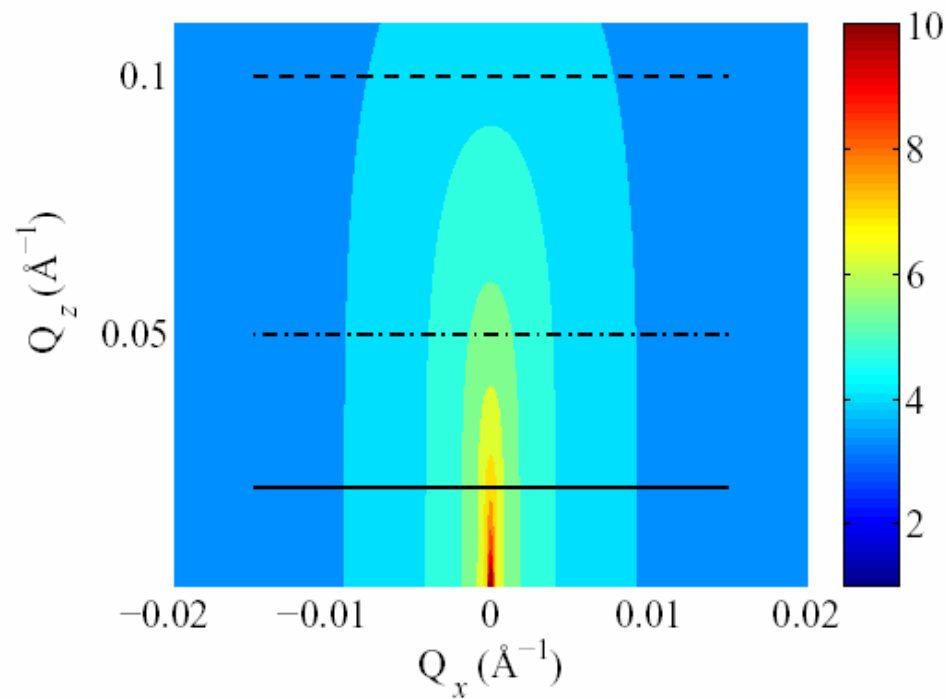
for $R > \xi$, interface appears smooth,

for $R < \xi$, interface appears rough, fractal behavior

$$S_{diff}(\vec{Q}) = \frac{\exp(-Q_z^2 \sigma^2)}{Q_z^2} \times \int [\exp(Q_z^2 C(R)) - 1] \exp(iQ_{\parallel} R) d^2 R$$

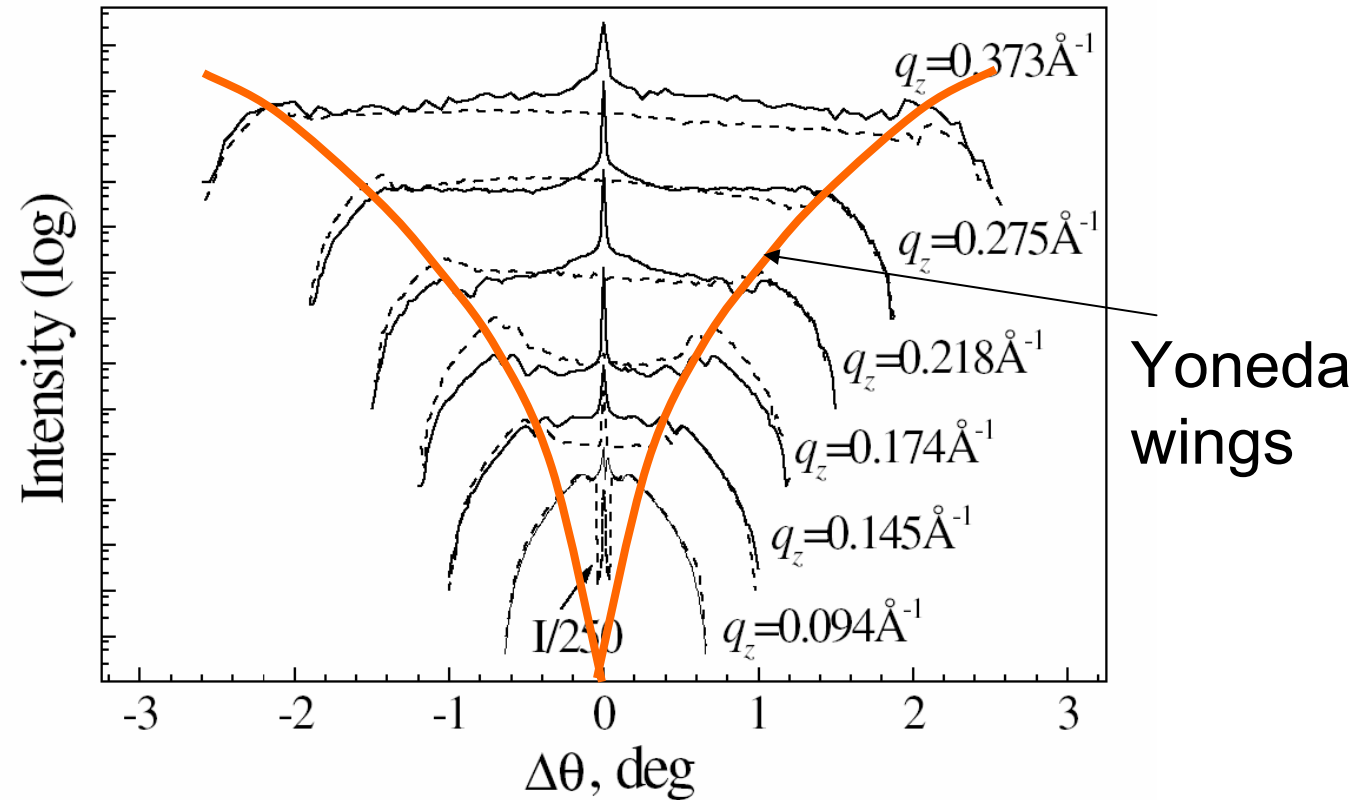


Specular and off-specular scattering



Transverse scans

Transverse scan from an FePt film on GaAs



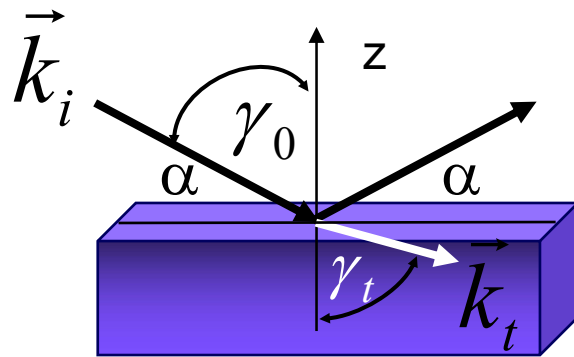
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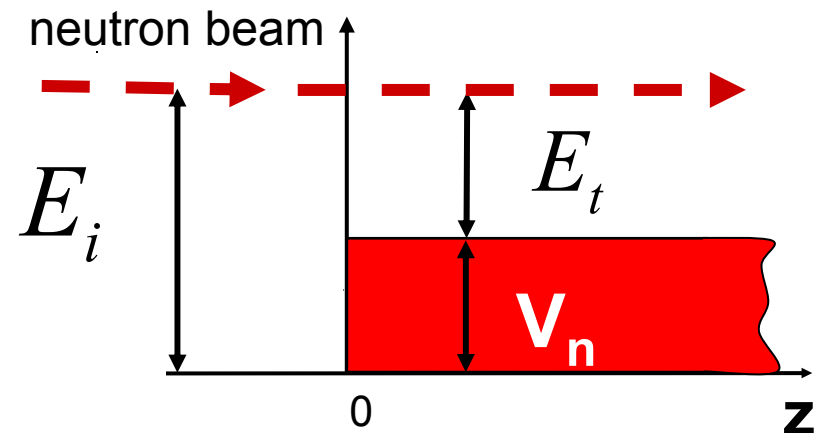
Refractive index for neutrons

Snell's law for specular reflection:



$$n = \frac{\sin \gamma_0}{\sin \gamma_t} = \frac{|\vec{k}_t|}{|\vec{k}_i|}$$

QM potential step for the z-component of the kinetic energy:



Nuclear potential:

$$V_n = \frac{2\pi\hbar^2}{m} N_A b_{coh}$$

Combining both

$$n^2 = \frac{\sin^2 \gamma_0}{\sin^2 \gamma_t} = \frac{|\vec{k}_t|^2}{|\vec{k}_i|^2} = \frac{E_t}{E_i} = \frac{E_i - V_n}{E_i} = 1 - \frac{4\pi}{k_i^2} N_A b_{coh}$$

N_A = nuclei number density

b_{coh} = coherent scattering length of nuclei A

Notice that $n \leq 1$, only for $b_{coh} \geq 0$

Total reflection only for $b_{coh} \geq 0$

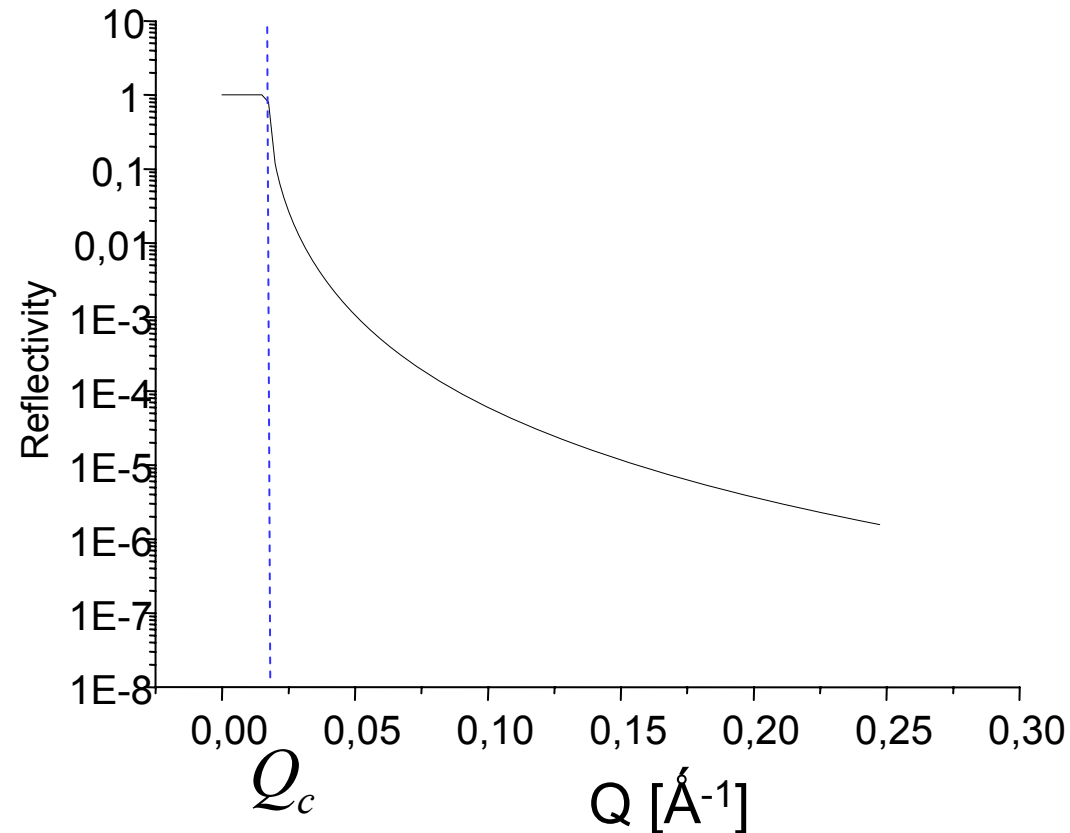
$$Q_c = \frac{4\pi}{\lambda} \sin \alpha_c = \sqrt{4k^2(1-n^2)} = \sqrt{16\pi N_A b_{coh}}$$

$$Q_c \propto \sqrt{N_A b_{coh}}$$



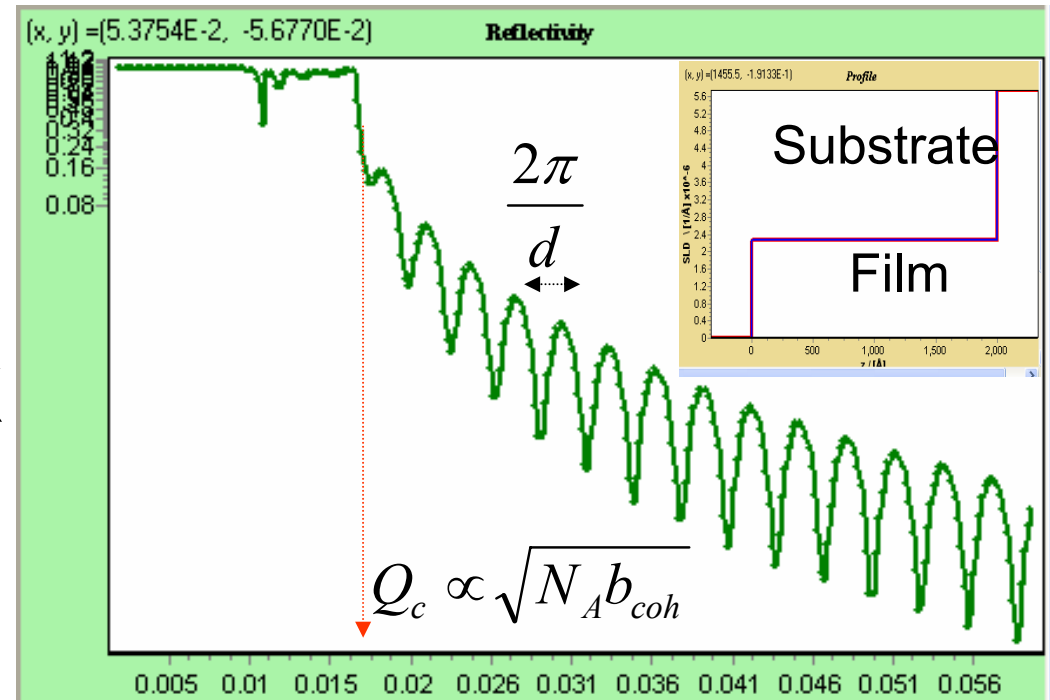
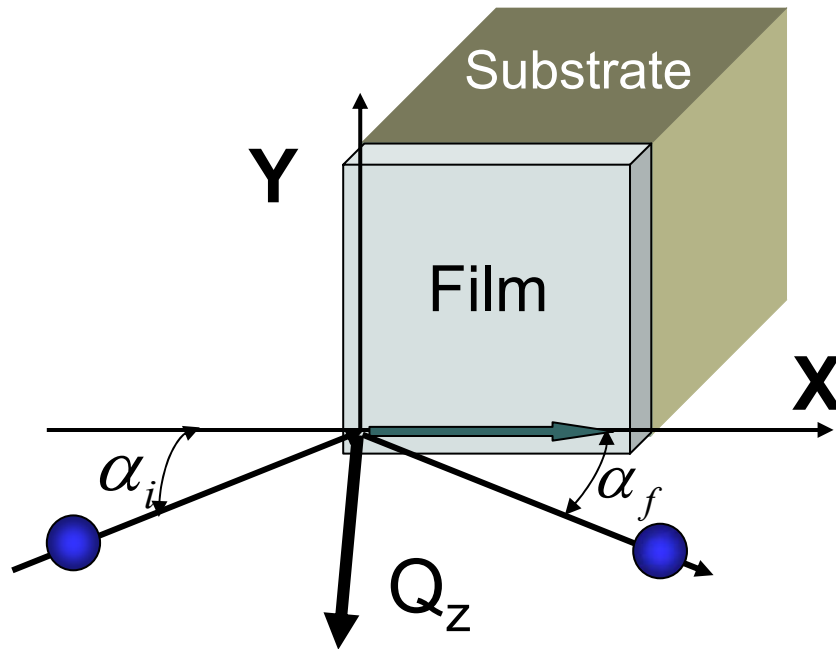
Example:

Neutron reflectivity from a non-magnetic, infinite thick and flat sample



For $Q_z < Q_c$: $R = 1$, only for $b > 0$, i.e. for coherent scattering length.

Neutron Reflectivity



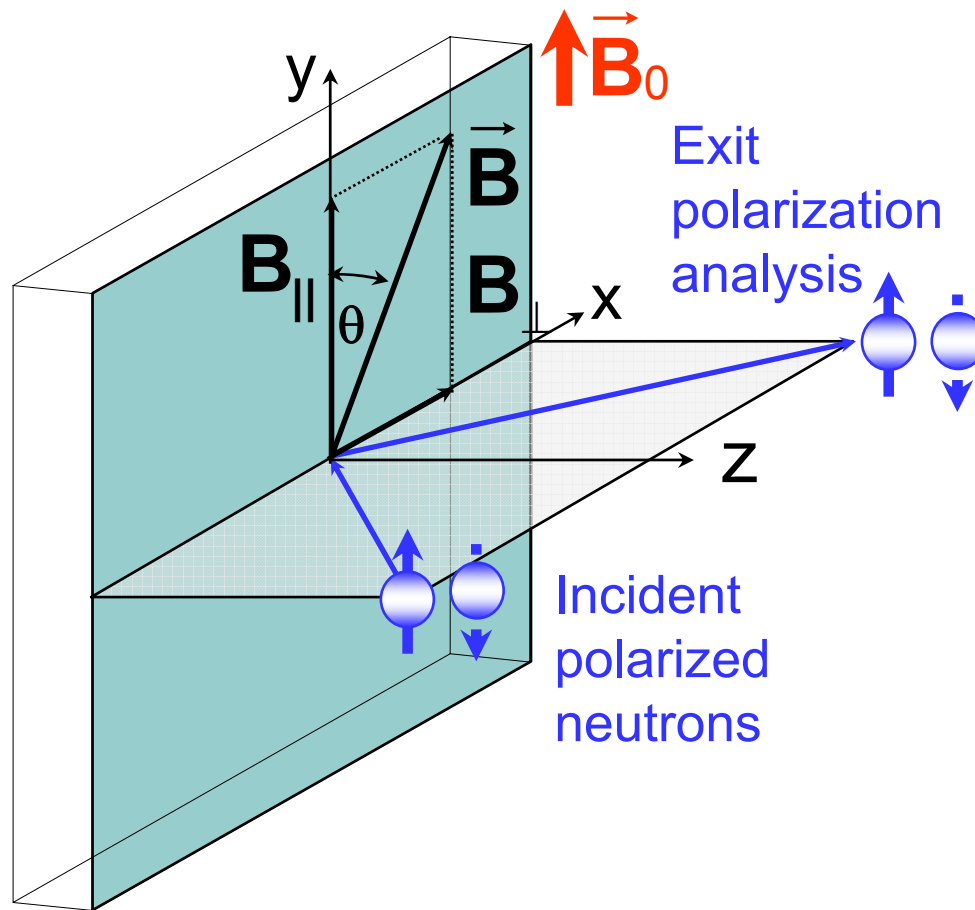
- ❖ Film thickness
- ❖ Interface roughness
- ❖ Density profiles

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Polarized Neutron Reflectivity: The four cross-sections



$$\vec{B} = (B_{\perp}, B_{\parallel}, 0)$$

Four cross sections:

$$R^{+,+} \quad R^{-,-} \quad R^{+,-} \quad R^{-,+}$$

$R^{\pm, \pm}$: Reflectivities

Quantum mechanical description of the scattering process

1D optical potential:

$$V = \underbrace{\frac{2\pi\hbar^2}{m} N_A b}_{\text{nuclear part}} - \underbrace{\vec{\mu} \cdot \vec{B}}_{\text{magnetic part}}$$

N_A : atomic number density

b : coherent neutron scattering length

$\vec{\mu}$: neutron magnetic momentum

$$\vec{\mu} = \gamma\mu_N\hat{\sigma}$$

μ_N : nuclear magneton

$\gamma = -1.913$: gyromagnetic ratio

$\hat{\sigma}$: Pauli spin operator

Schrödinger equation:

$$\left[-\frac{\hbar^2}{2m} \nabla^2 + V \right] \Psi(z) = E\Psi(z)$$

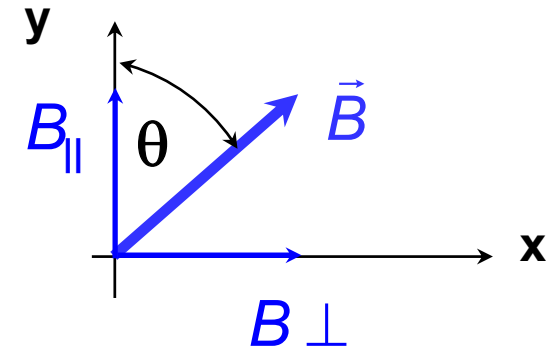
$$\sigma_{\perp} = \begin{pmatrix} 0 & 1 \\ 1 & 0 \end{pmatrix}; \quad \sigma_{\parallel} = \begin{pmatrix} 1 & 0 \\ 0 & -1 \end{pmatrix}; \quad \sigma_z = \begin{pmatrix} 0 & -i \\ i & 0 \end{pmatrix}$$

Polarized Reflectivity

- Non-Spin-Flip (NSF)

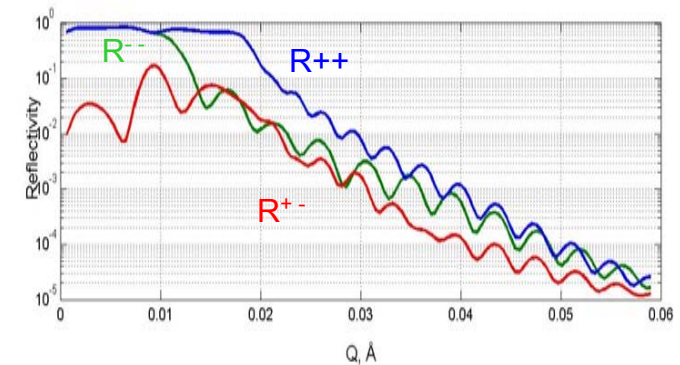
$$R^{\pm,\pm} = \underbrace{\frac{1}{2}(|r_+|^2 + |r_-|^2)}_{\text{nuclear}} \pm \underbrace{\frac{1}{2}(|r_+|^2 - |r_-|^2)}_{\text{magnetic}} \cos \theta$$

$$\longrightarrow R^{+,+} - R^{-,-} = (|r_+|^2 - |r_-|^2) \cos \theta \propto B_{\parallel}$$



- Spin-Flip (SF)

$$R^{\pm,\mp} = \frac{1}{4} |r_+ - r_-|^2 \sin^2 \theta \propto B_{\perp}^2$$



r_{\pm} : reflection amplitudes

Reflectivity and Asymmetry

$$R^+ = R^{++} + R^{+-}$$

$$R^- = R^{--} + R^{-+}$$

$$R^+ - R^- = R^{++} - R^{--}$$

$$R^+ + R^- = R^{++} + R^{--} + R^{+-} + R^{-+}$$

- Spin Asymmetry

$$SA = \frac{R^+ - R^-}{R^+ + R^-} = \frac{R^{++} - R^{--}}{R^{++} + R^{--} + R^{+-} + R^{-+}}$$



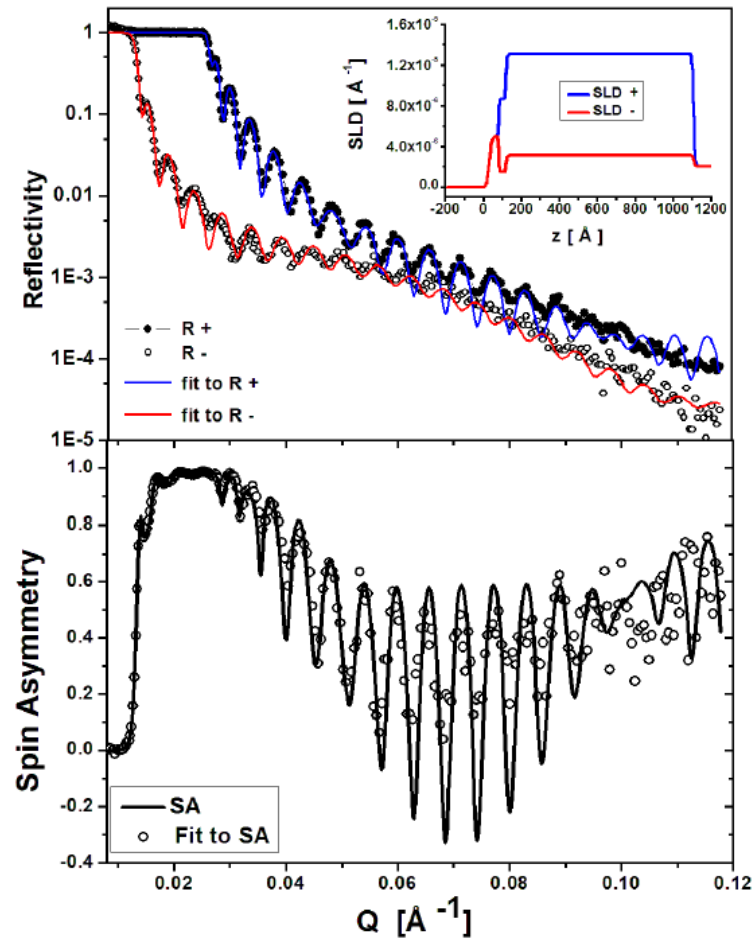
Reflectivity and Asymmetry of single Fe - layer

Reflectivities :

R^+ and R^-

Spin Asymmetry :

$$SA = \frac{R^+ - R^-}{R^+ + R^-}$$



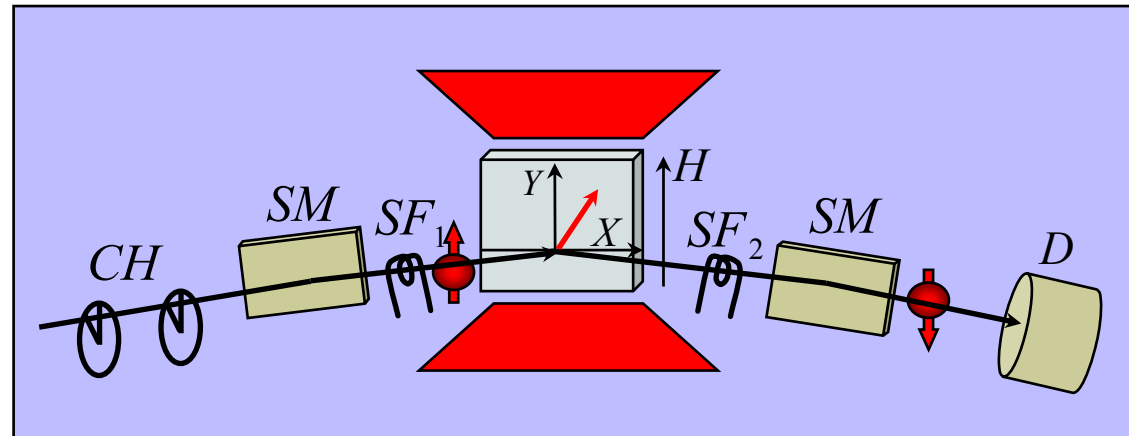
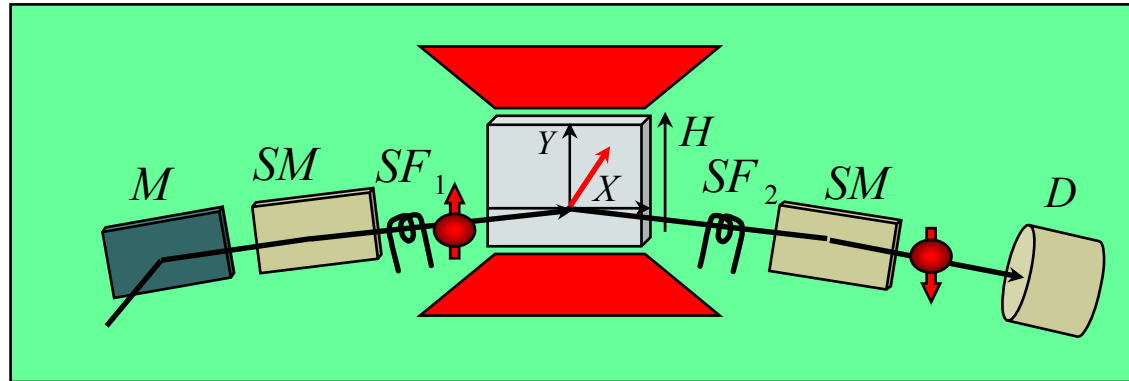
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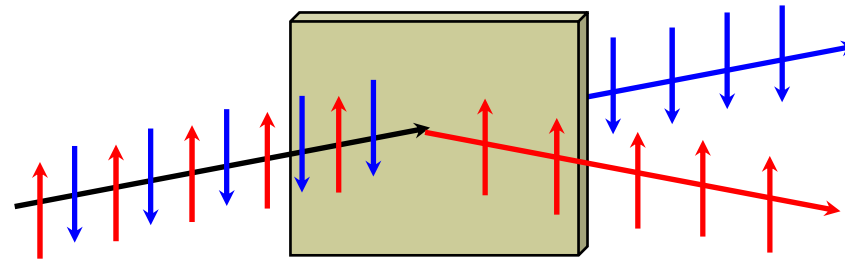
Instrumentation: two types

- Angle dispersive
- monochromatic beam
- scanning of θ
- Wavelength dispersive
- White beam
- TOF method,
- fixed θ

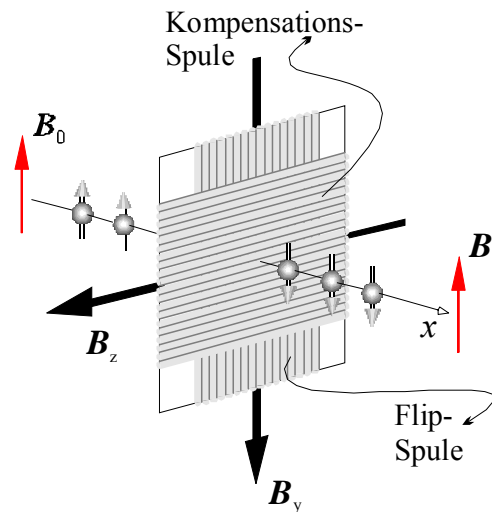


Polarizer, analyzer, spin flipper

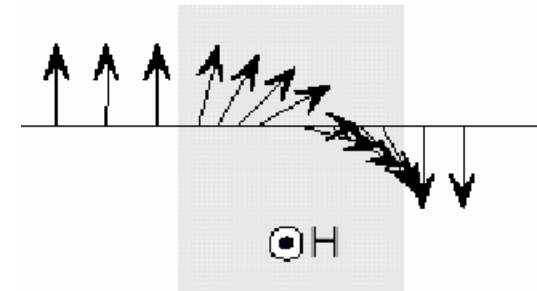
transmission
supermirror:



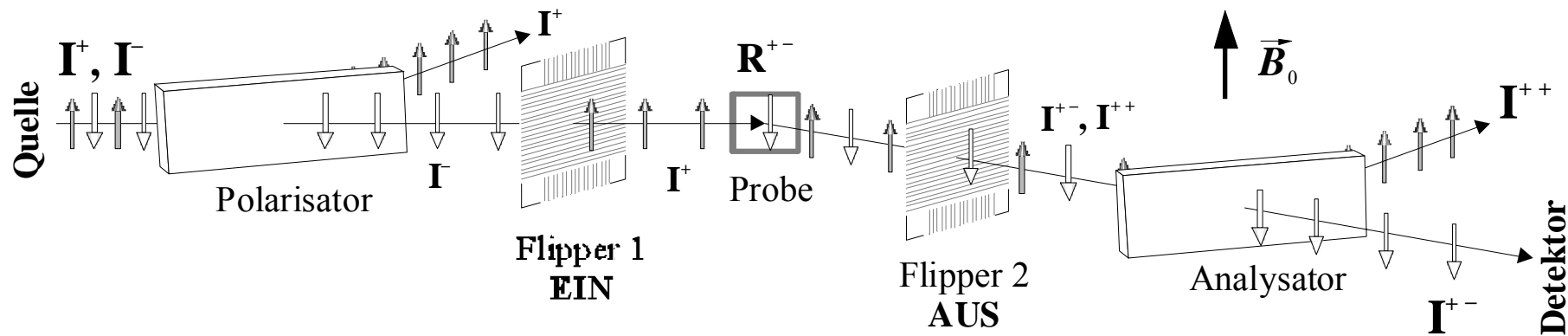
Mezei spin- π
neutron-flipper



Adiabatic spin rotation



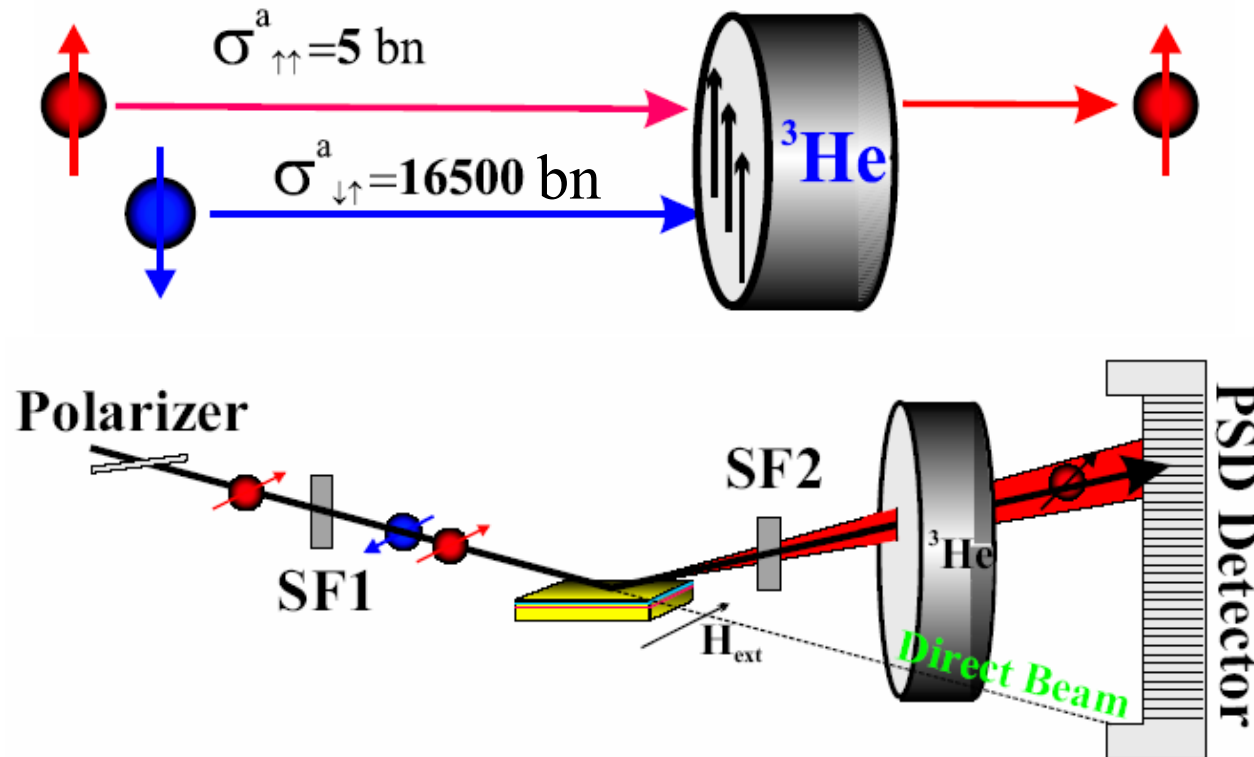
Schematics: neutron reflectometer with complete polarization analysis



This part is identical for angle dispersive and wavelength dispersive instruments

^3He Spin-Filter for the spin analysis of diffuse scattering

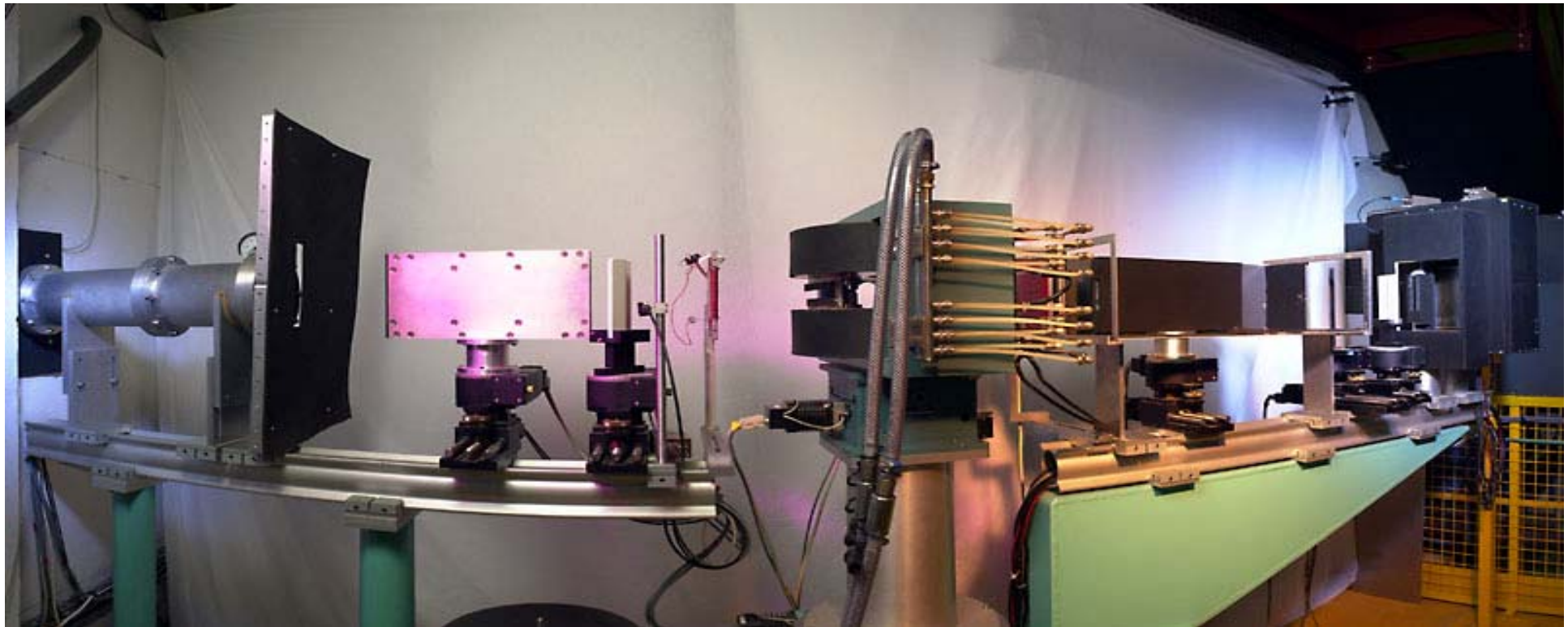
Ken Anderson, ILL



^3He spin-filter technique is very useful for the polarization analysis of off-specular scattering. Compared to solid-state analyzer, the spin-filter covers a wider angular range and is free of small angle scattering.

The ADAM Reflectometer at the ILL

Polarizer-
SM Front
flipper Sample in
magnet Back
flipper Analyzer-
SM Detector

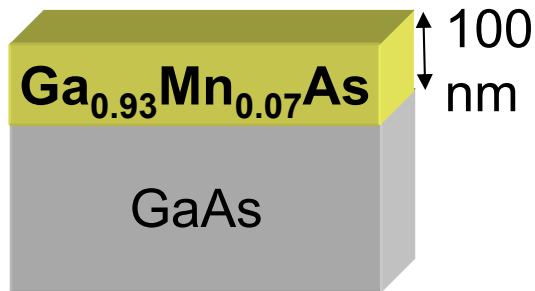


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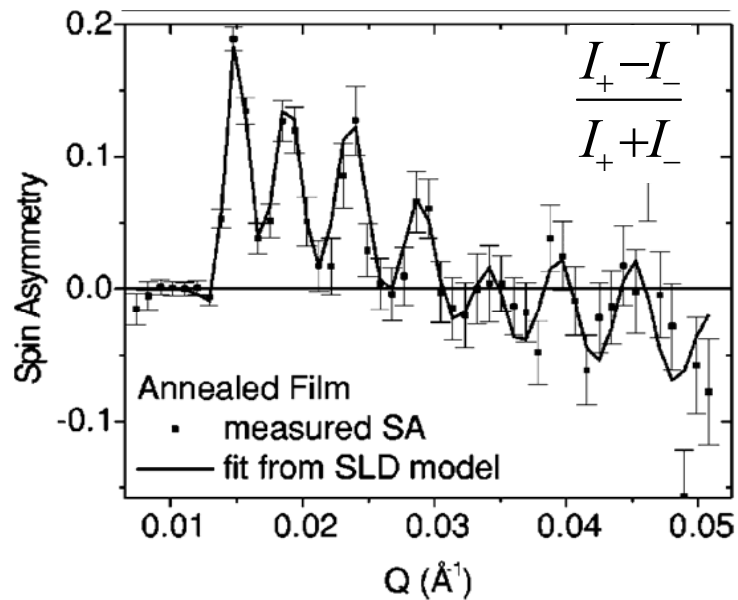
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 - ▶ Specular and off-specular scattering
 - ▶ Lateral magnetic nanostructures
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Nuclear and magnetic density profile in (GaMn)As

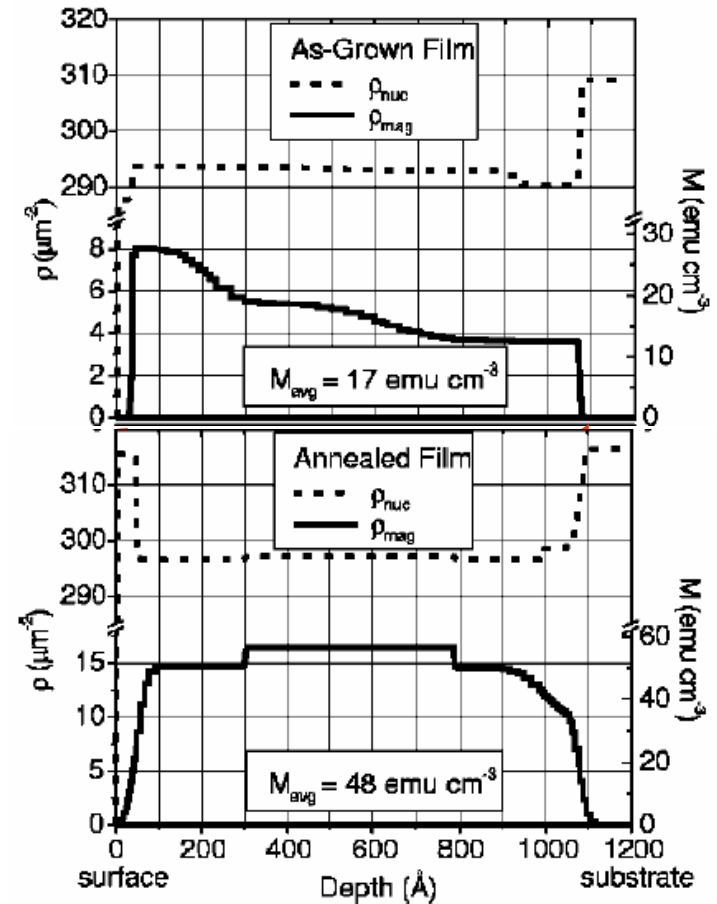


Spin asymmetry

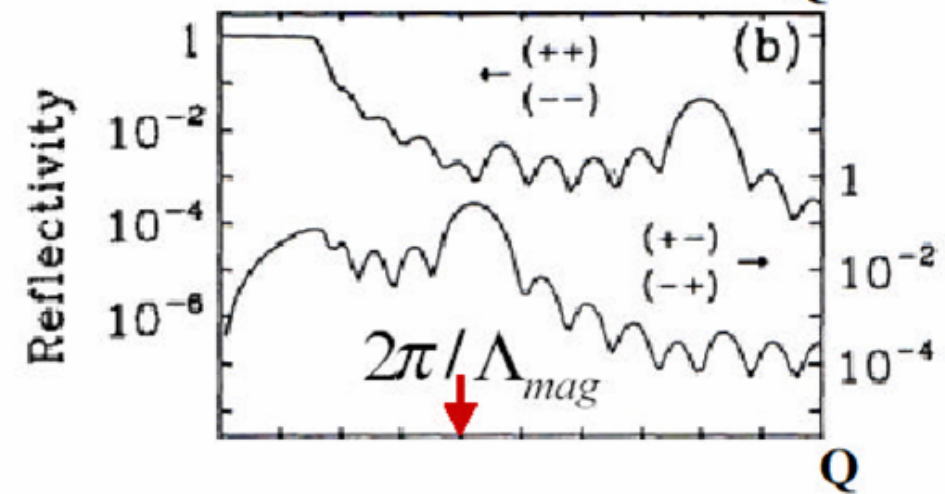
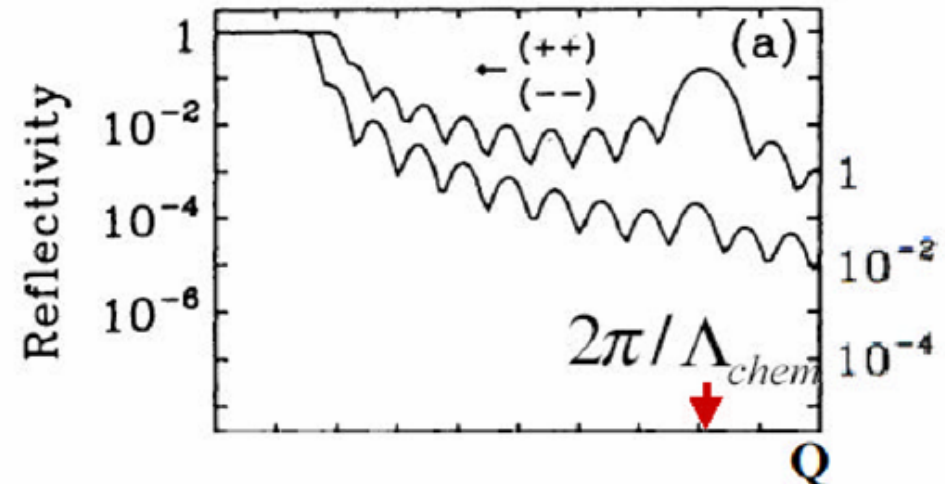
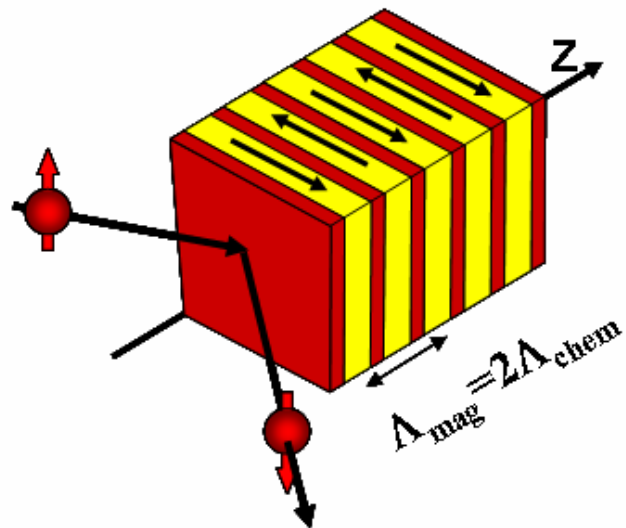
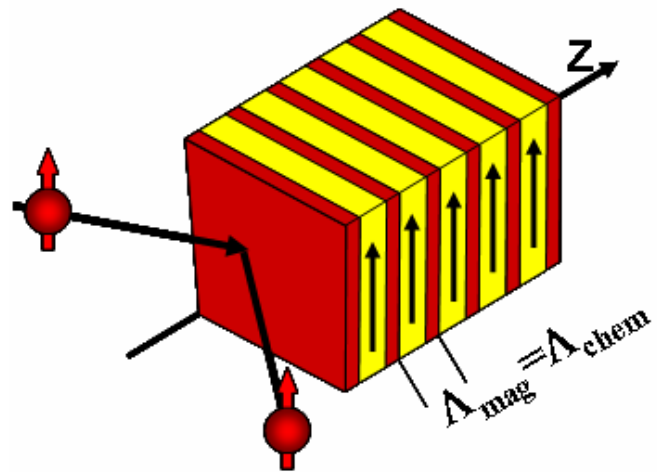


before annealing

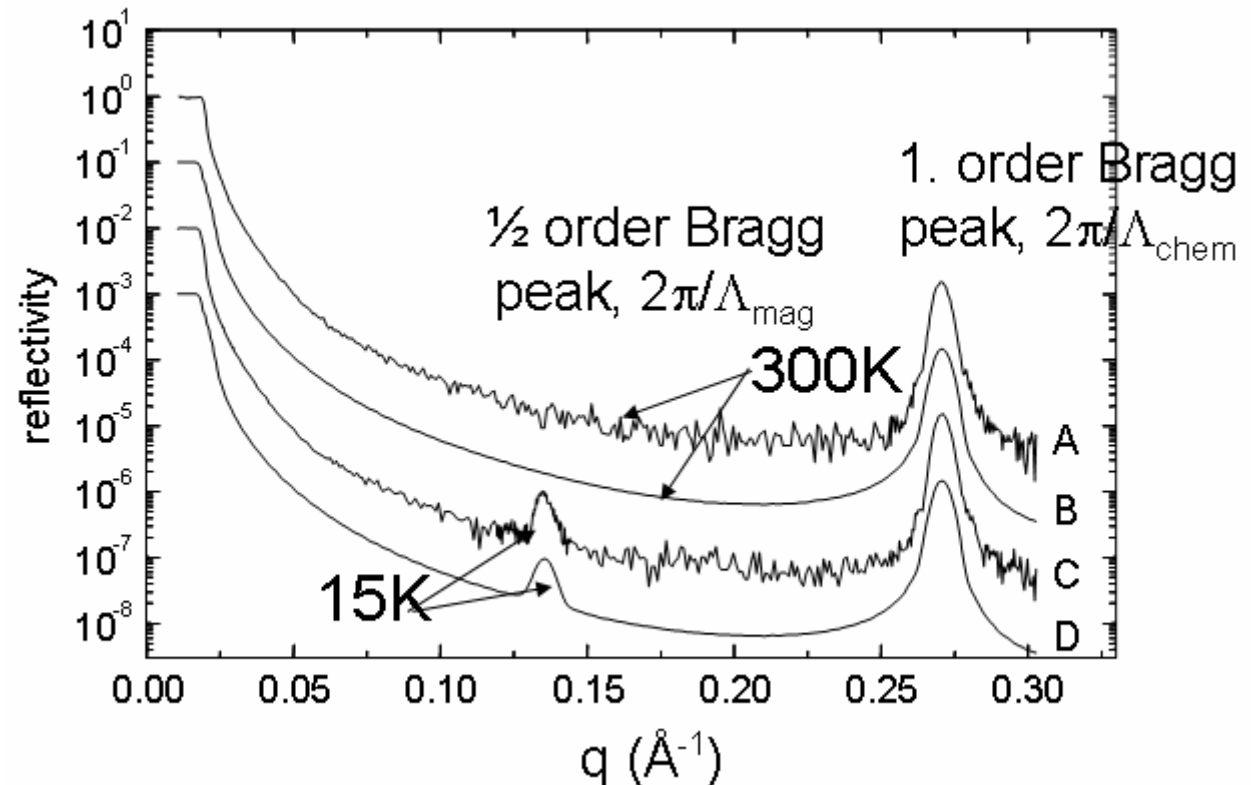
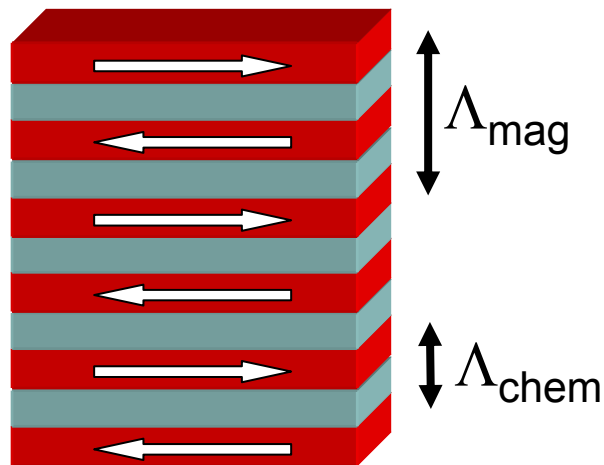
after annealing at 280°C for 1 h



PNR from model-spinstructures in magnetic multilayers



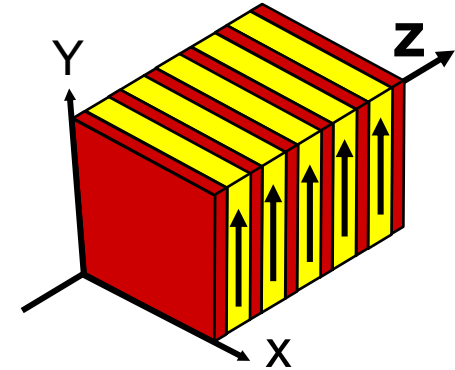
AF case: NR of Fe(2ML)/V(13ML) above and below $T_N = 100K$



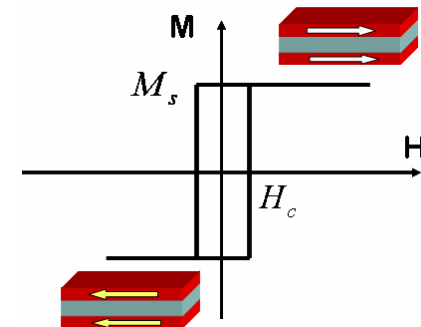
Antiferromagnetic coupling below the Néel temperature
Intensity = M^2

Three steps to FM sample analysis

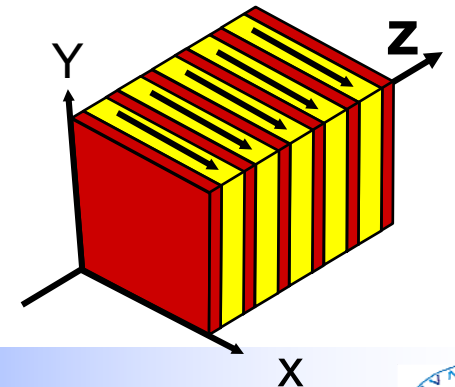
1. Align sample with easy axis parallel to Y-axis



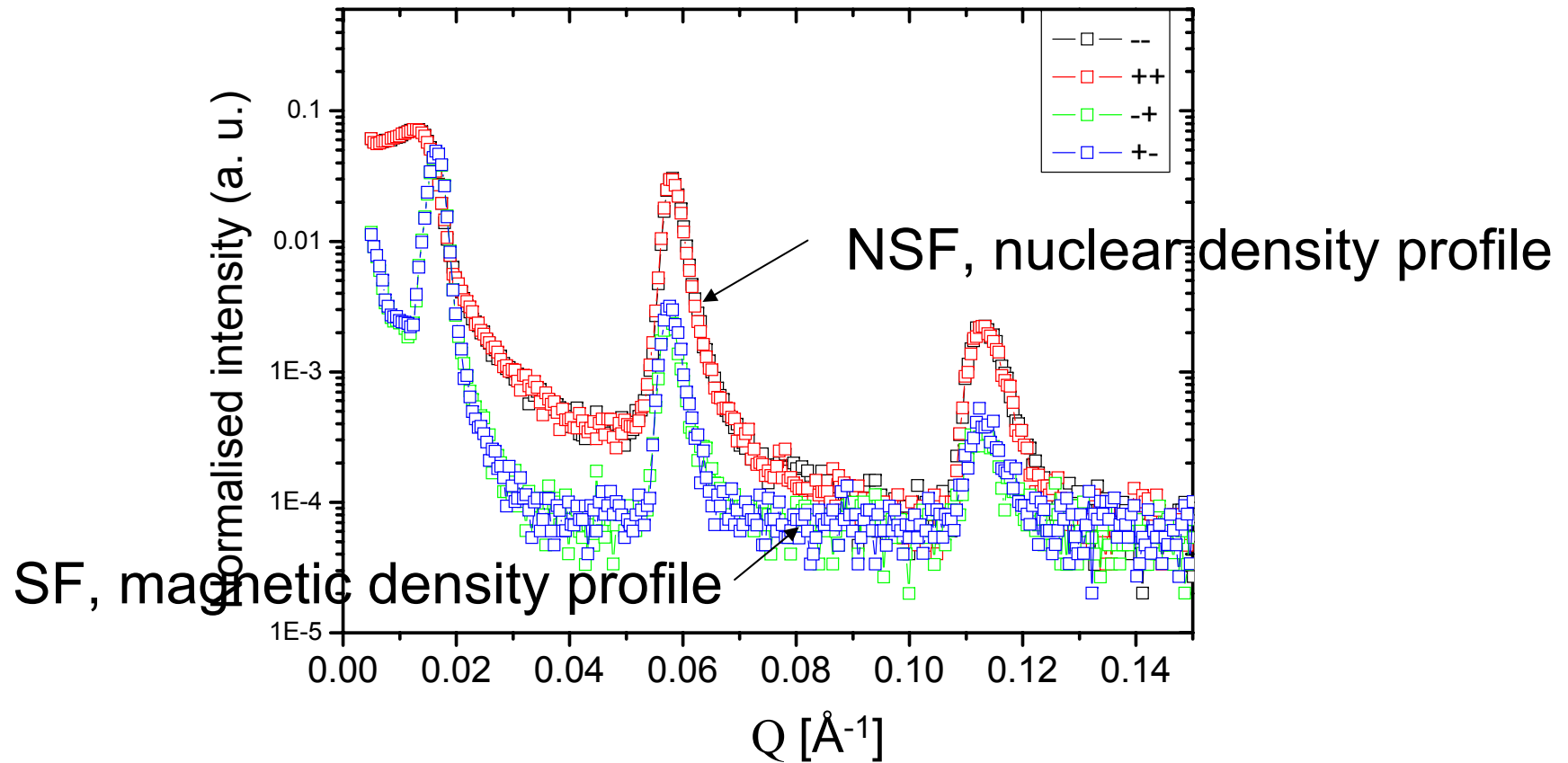
2. Go to saturation and then back to remanence



3. Rotate sample in remanence by 90° . Now the NSF reflectivity is only nuclear, and the SF reflectivity is only magnetic.



PNR in remanence



Nuclear and magnetic density profiles have the same periodicity but different shape.

M. Vadala, K. Zhernenkov, M. Wolff, H. Zabel, August 2006, ADAM reflectometer

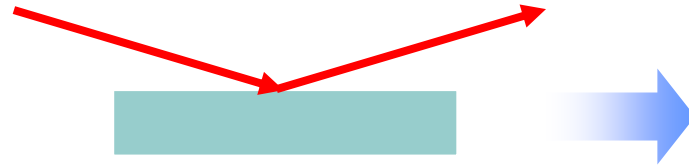
Content

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 - ▶ **Specular and off-specular scattering**
 - ▶ Lateral magnetic nanostructures
4. Case study: do we still need neutrons?

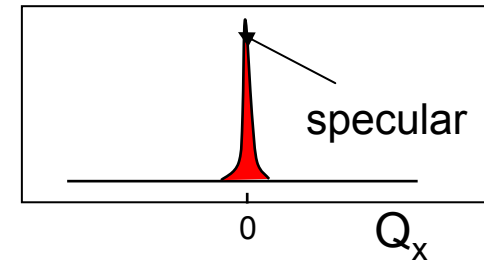


Transverse reflectivity

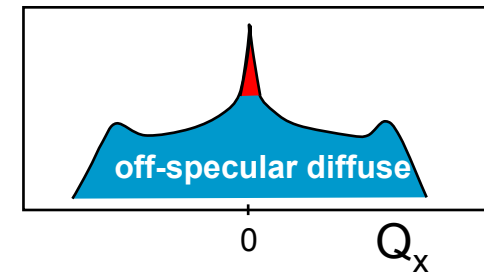
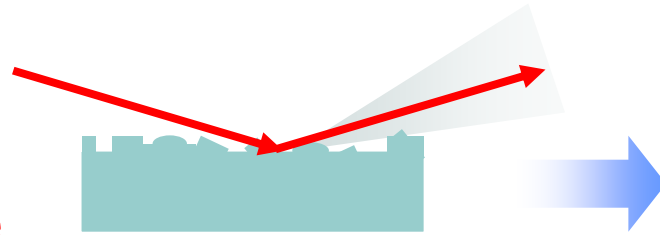
**Flat film:
Specular**



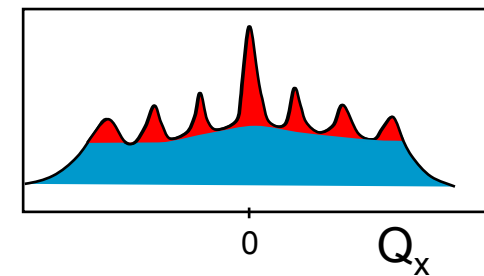
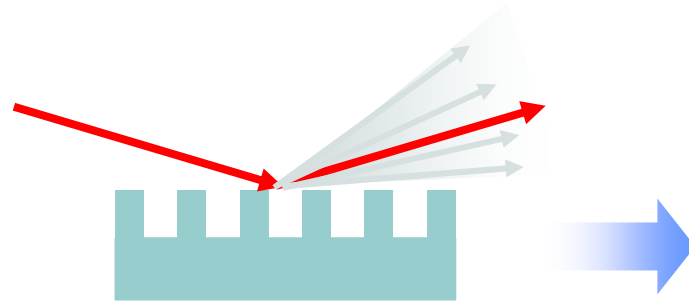
Transverse scan



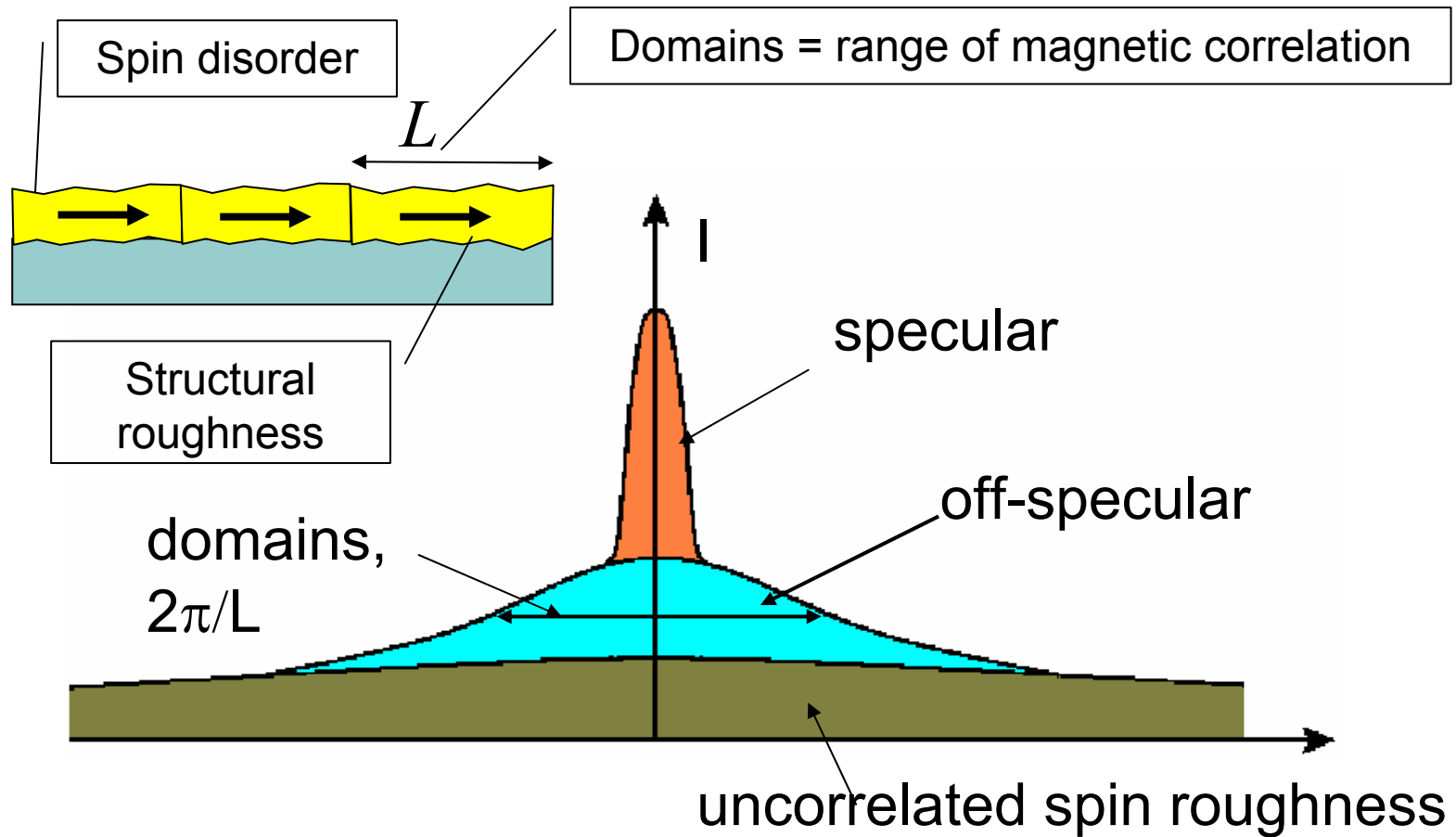
**Rough surface:
Specular + Diffuse**



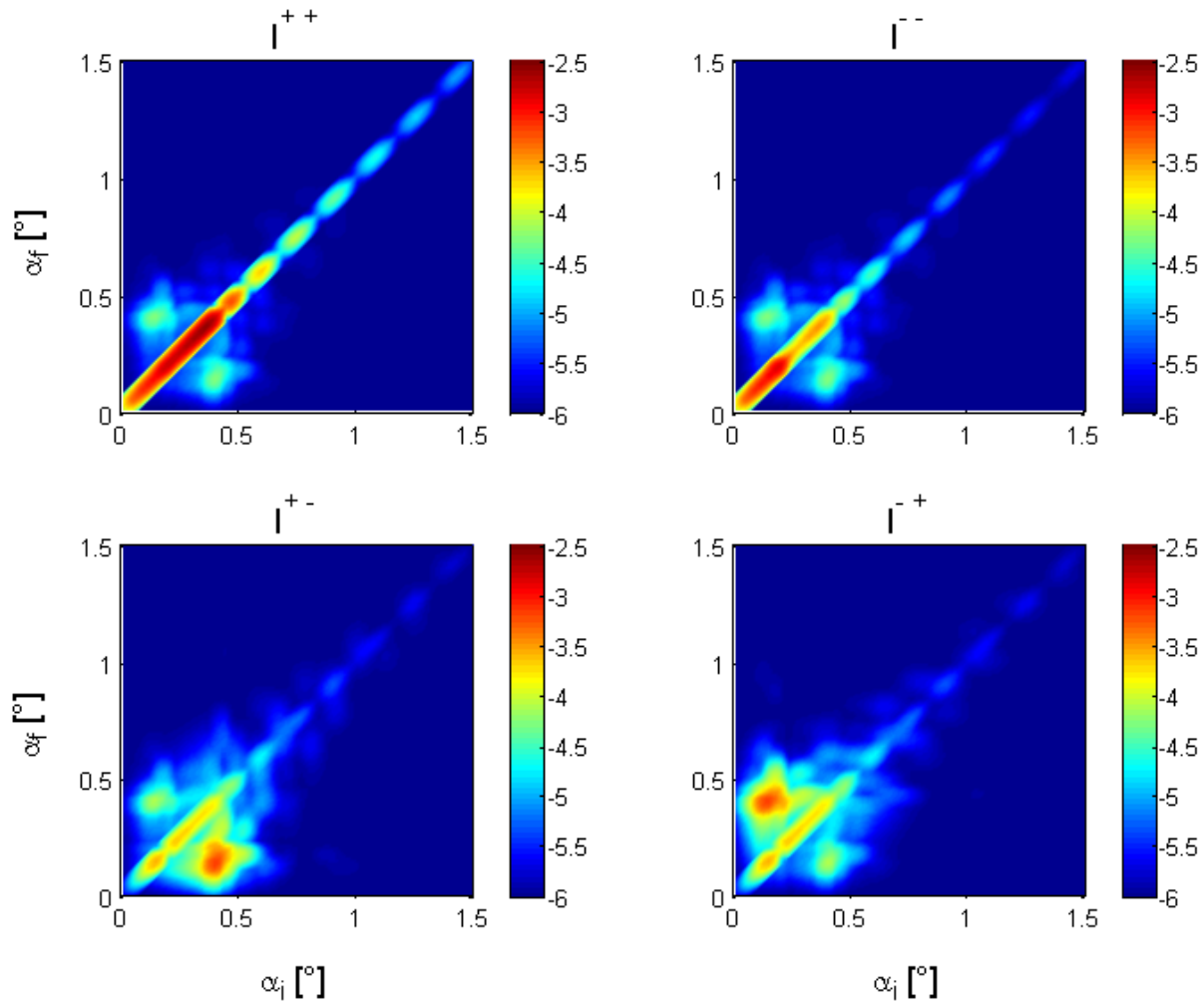
**Lateral periodic
pattern:
Diffuse + Bragg**



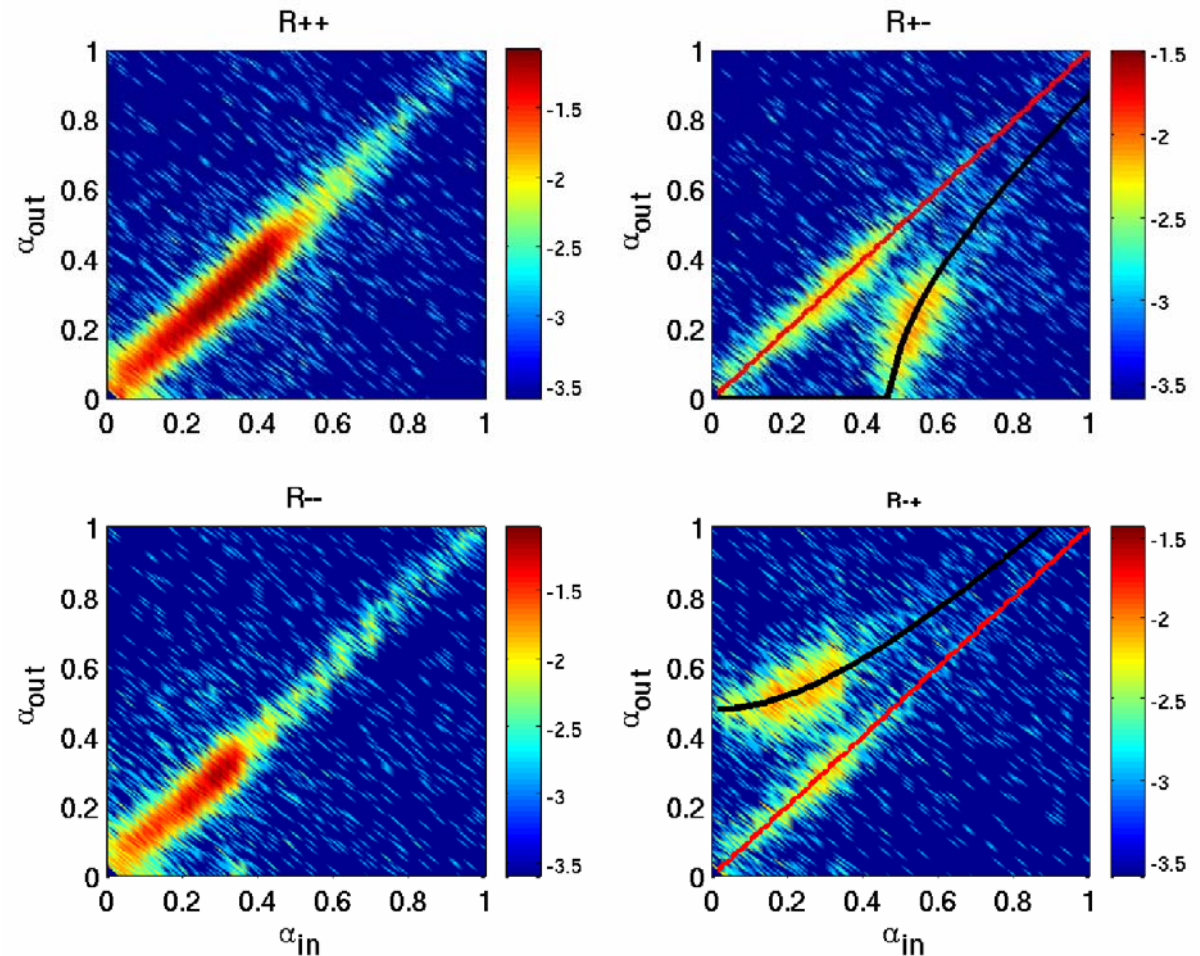
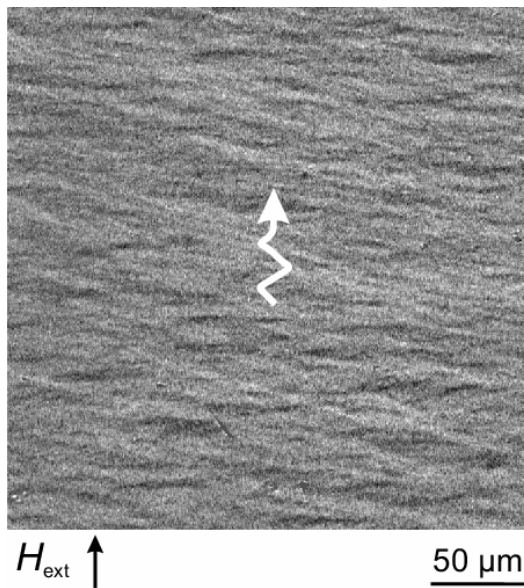
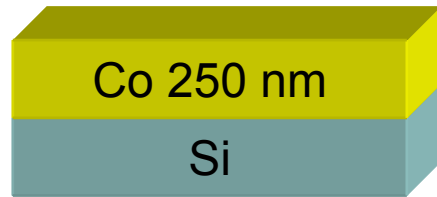
Types of magnetic roughness



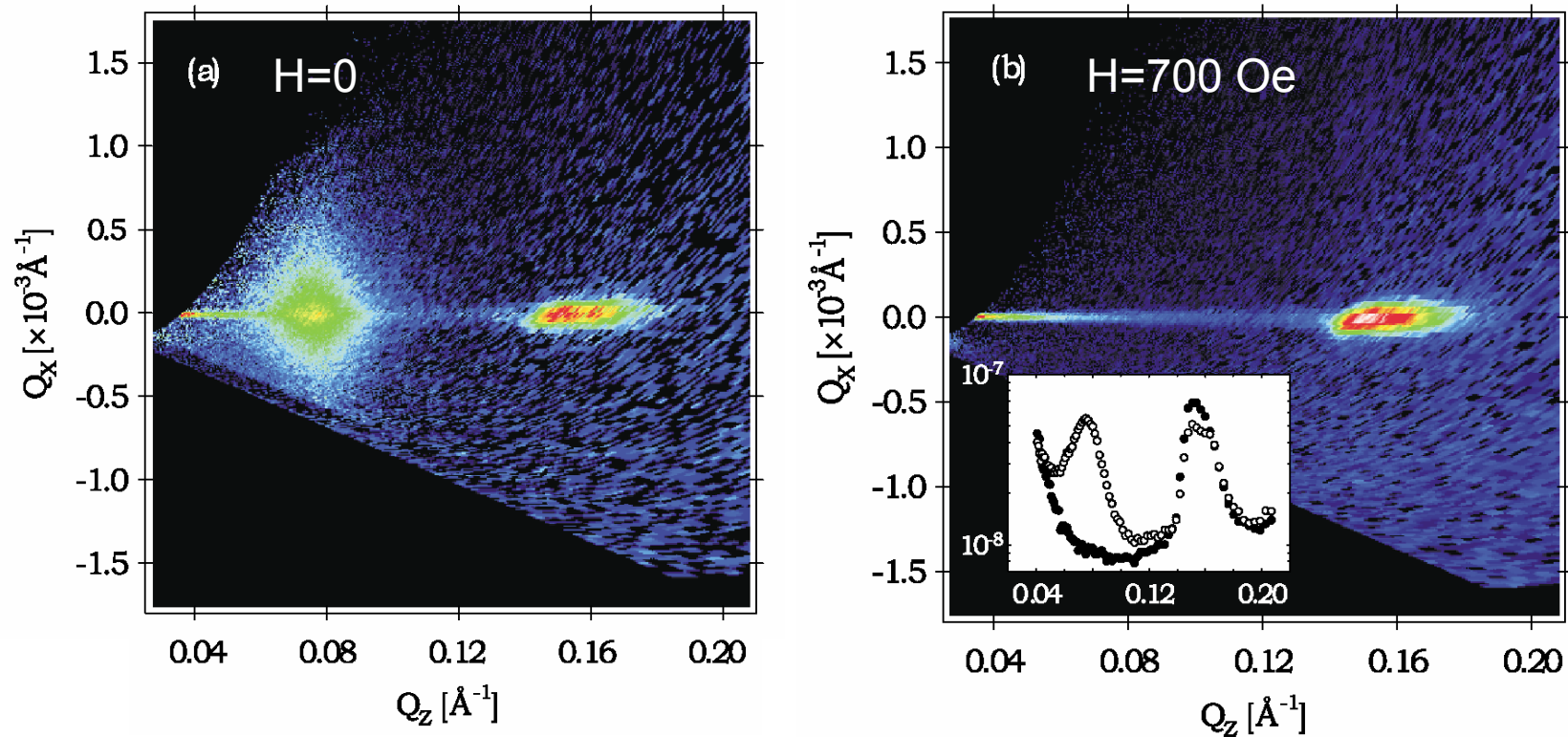
Domain size: $1 \mu\text{m}$ ($\ell \ll L_D$)



Single ferromagnetic film in the domain state



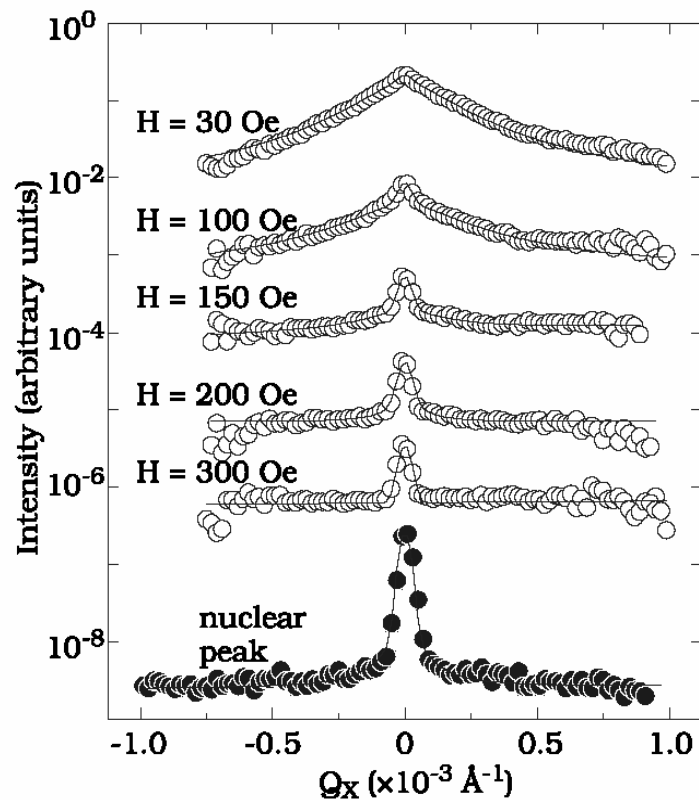
Magnetic roughness in Co/Cu superlattices



- $\frac{1}{2}$ order AF peak, only diffuse peak \Rightarrow small domain size
- weak out-of-plane correlation, gradient in periodicity



Transverse scans across half-order AF peak



$$S_{Diff}(Q) = DW \int d^2\vec{r} e^{i\vec{Q}_{\parallel} \cdot \vec{r}} [s + m + sm]$$

s = structural roughness

m = domain distribution roughness

sm = cross term contains magnetic roughness

Diffuse scattering due to:

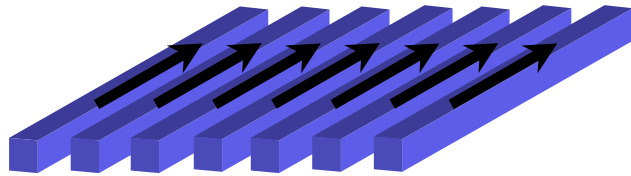
- domain size distribution
- orientational domain distribution
- Lorentzian profile changes into Gaussian profile with increasing domain size
- Diffuse scattering diminishes in high fields

Content

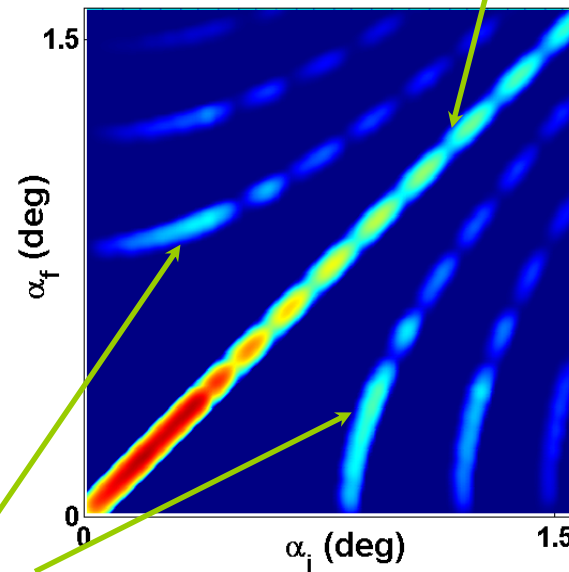
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PNR from patterned films



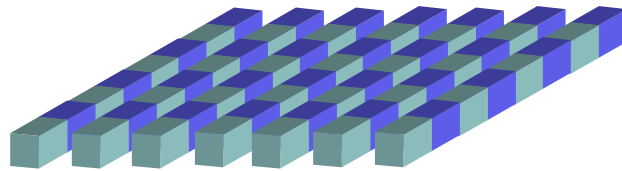
Simulation:



Specular reflectivity

Bragg reflections

PNR from patterned films: domains

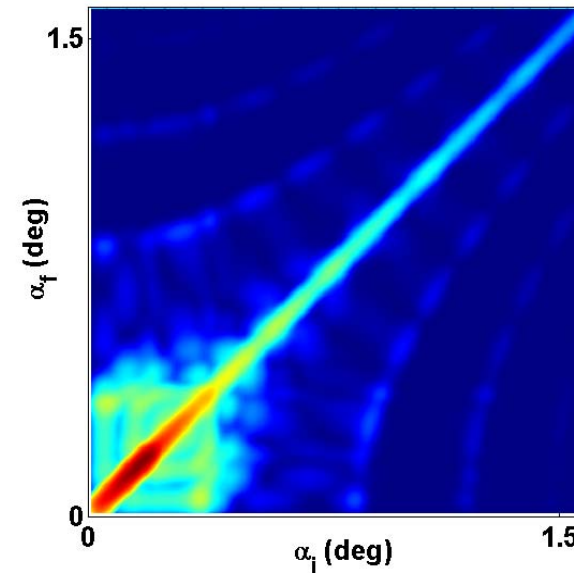
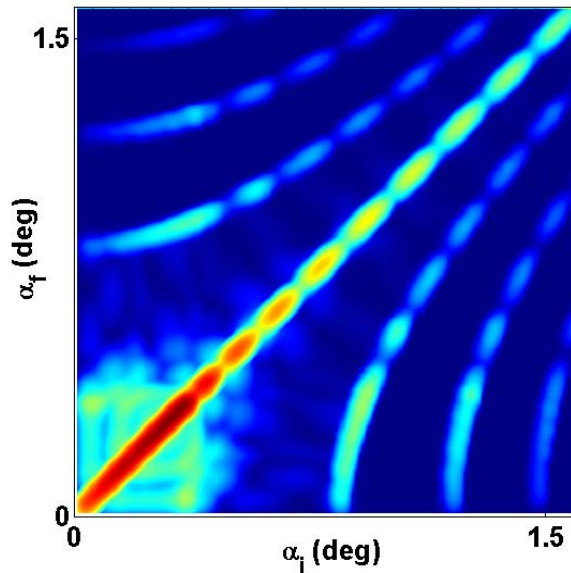


Non-spin-flip channels

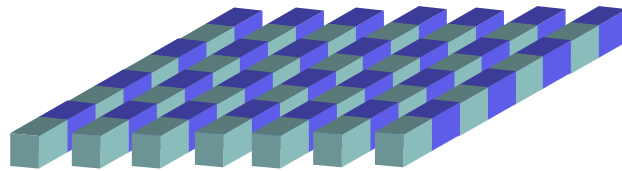
R⁺⁺

Simulation:

R⁻⁻



PNR from patterned films: domains

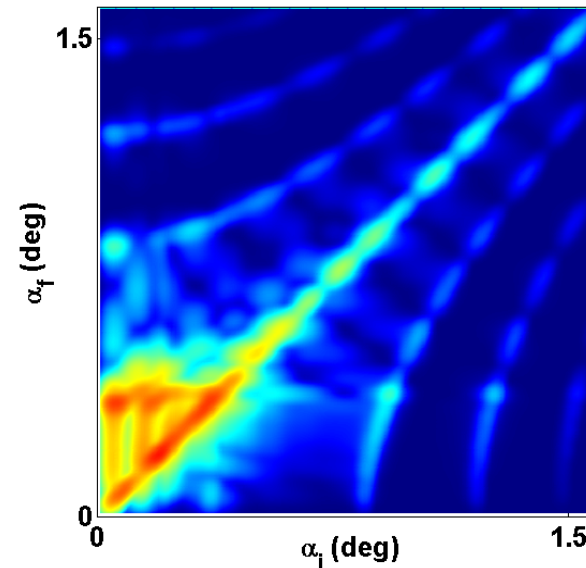
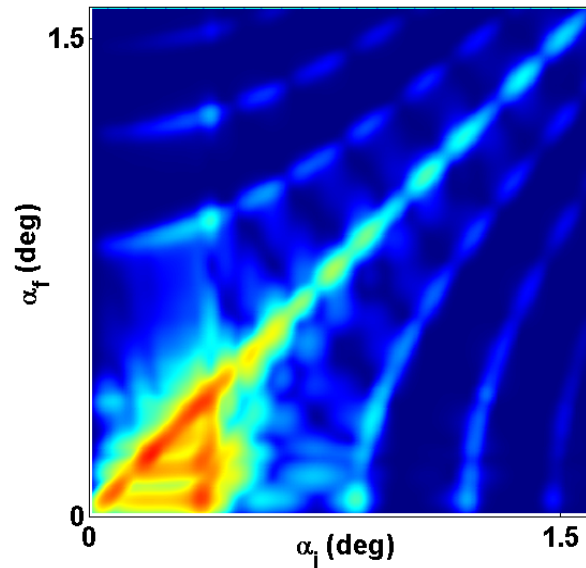


Spin-flip channels

R^{+-}

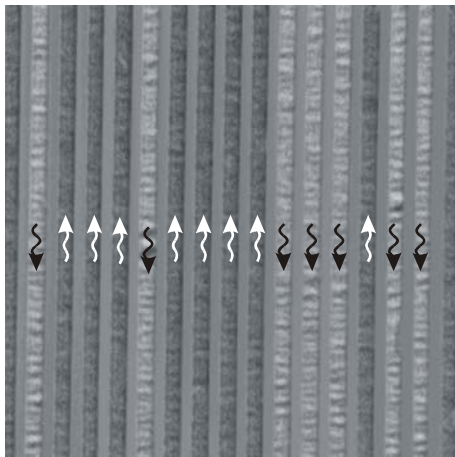
Simulation:

R^{-+}

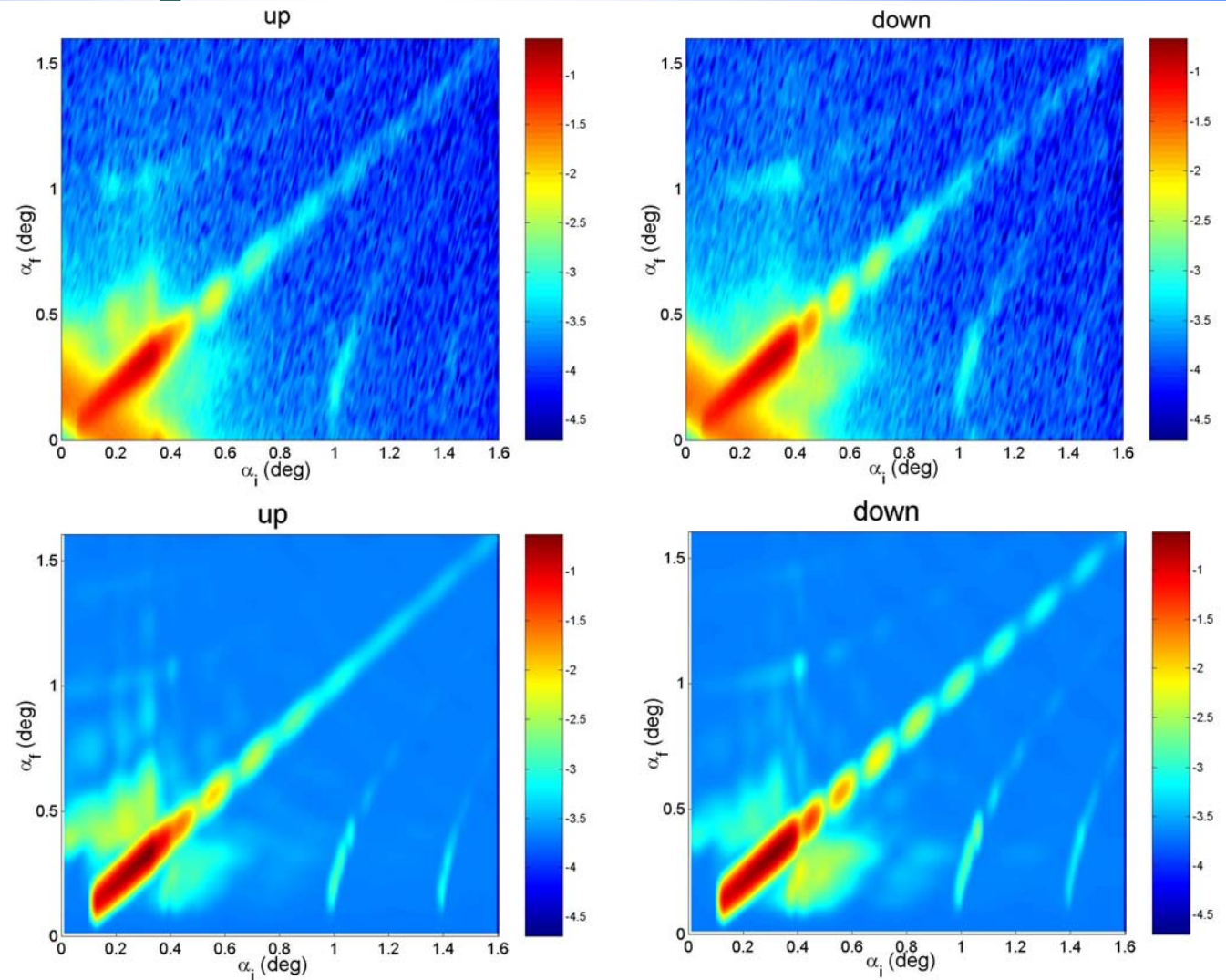


Reciprocal space map from a stripe array

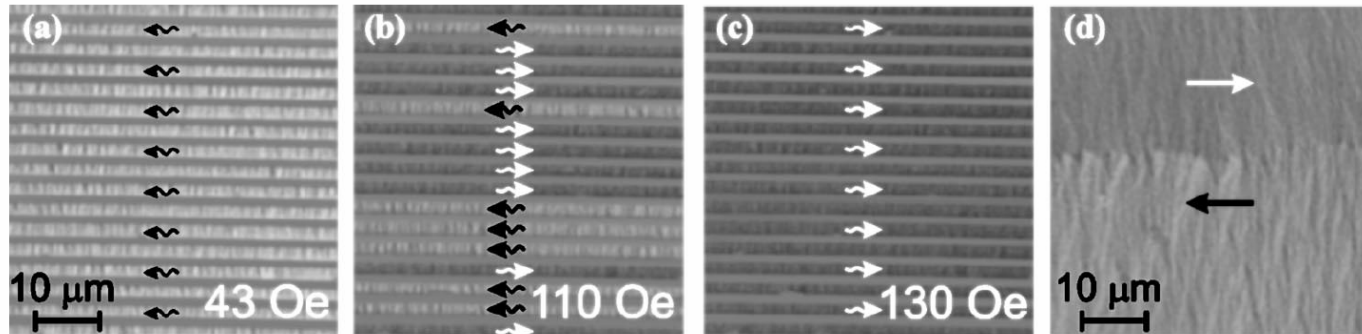
Experiment:



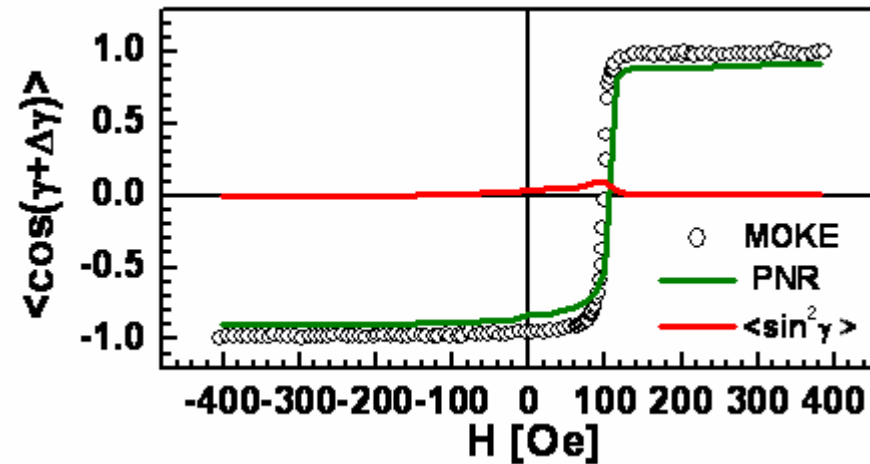
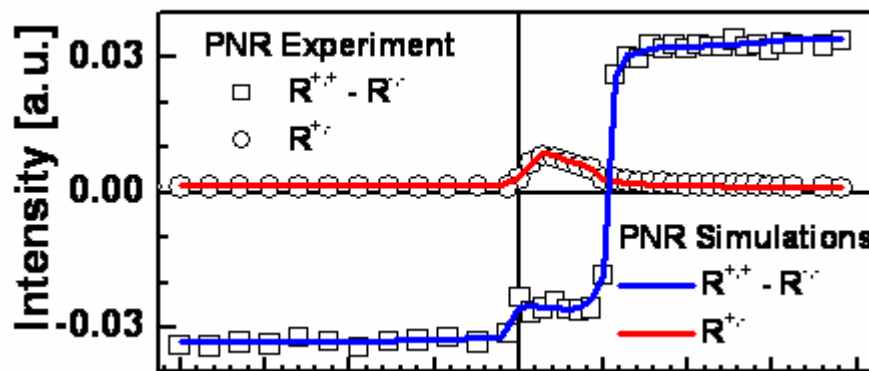
Simulation:



Magnetization reversal parallel to stripes

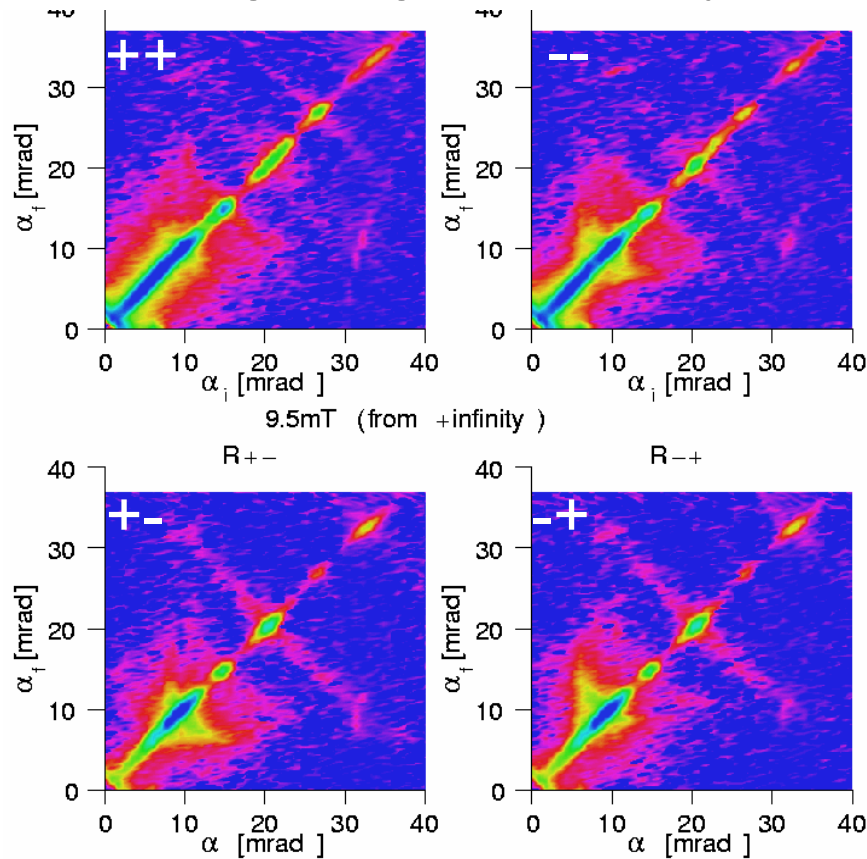
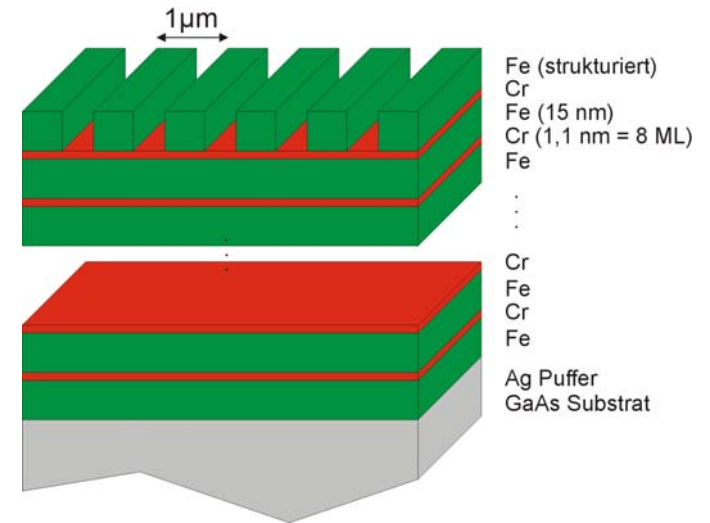


Average hysteresis and quantitative determination of fluctuations



Laterally structured Fe/Cr multilayer

- **Fe stripes on AF-Fe/Cr ML** $\Lambda = 1 \mu\text{m}$
Ziegenhagen et al., Physica B **335** (2003) 50



Maps:

- specular PNR (half- and full-order peaks)
- off-specular diffuse scattering
- Bragg sheets
- first-order lateral Bragg diffraction

Measurements @ FRJ2, HADAS

Content

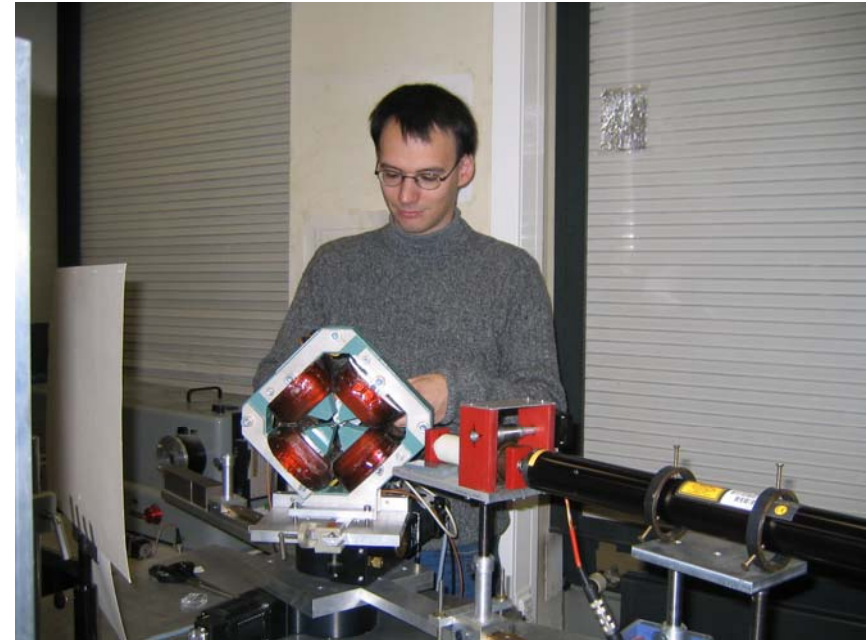
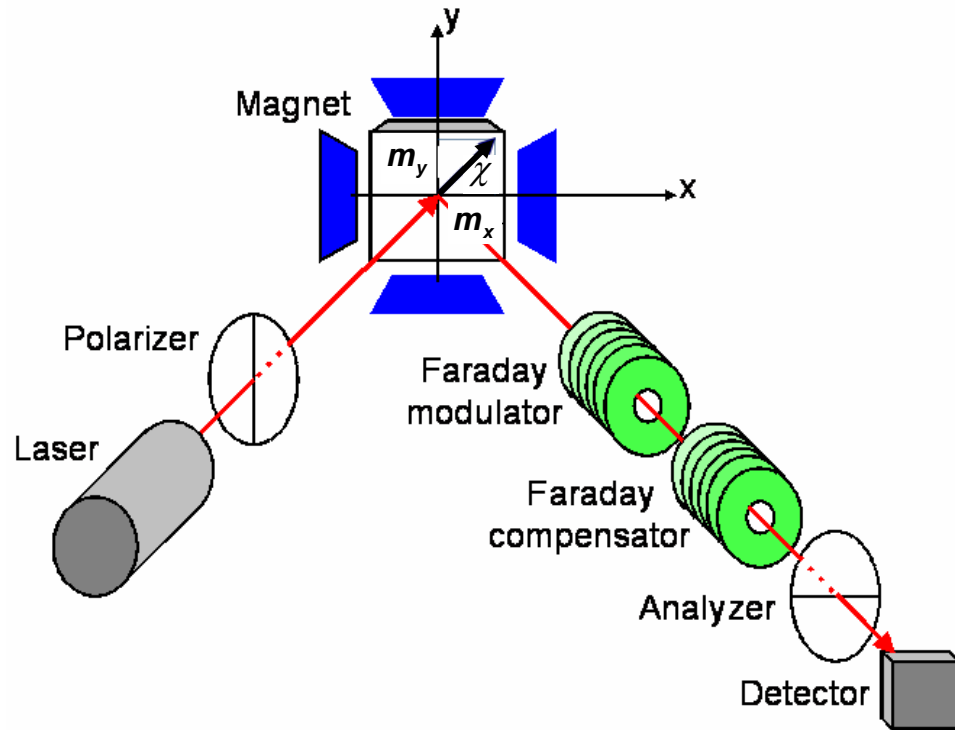
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Analytical tools

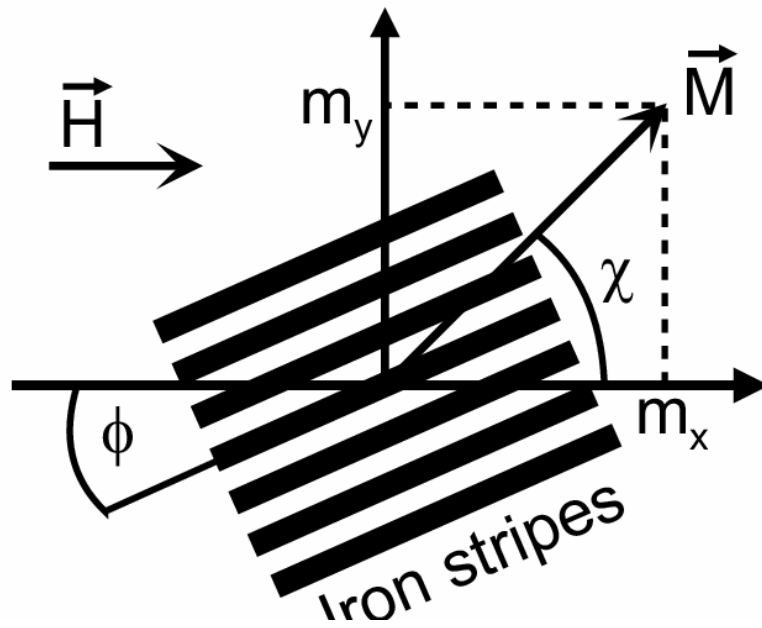
- Magneto-optic Kerr effect (MOKE):
Vector – magnetometry
- Polarized neutron reflectivity (PNR):
Vector – magnetometry
+ depth resolution
- X-ray resonant magnetic scattering (XRMS):
Vector – magnetometry
+ depth resolution
+ element selectivity
+ time resolution

Longitudinal vector - MOKE



Kerr angle $\theta_K^x \sim m_x = |\vec{m}| \cos \chi$
 $\theta_K^y \sim m_y = |\vec{m}| \sin \chi$

Vector-MOKE



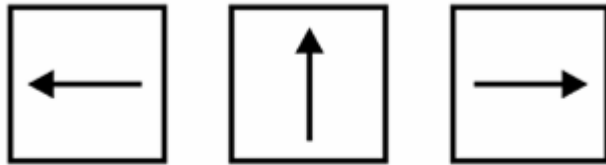
$$\frac{m_x}{m_y} = \frac{\cos \chi}{\sin \chi} = \frac{\theta_K^x}{\theta_K^y}$$

$$\chi = \text{arc cot} \left(\frac{\theta_K^x}{\theta_K^y} \right)$$

$$\frac{|\vec{M}|}{|\vec{m}|_{x,sat}} = \frac{\theta_K^x}{\theta_K^{x,sat}} \frac{1}{\cos \chi}$$

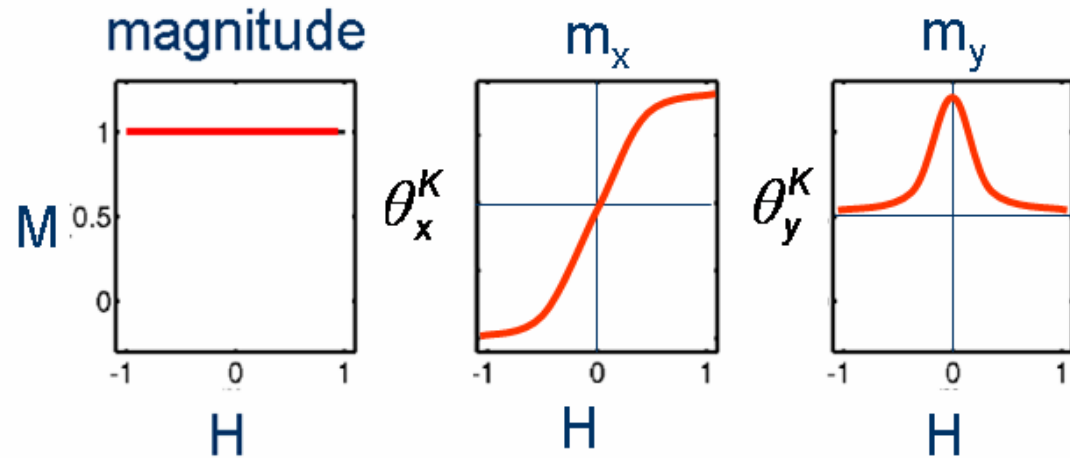
Two limiting cases

Coherent rotation:



$$m_x = -1 \quad = 0 \quad = +1$$

$$m_y = 0 \quad = +1 \quad = 0$$

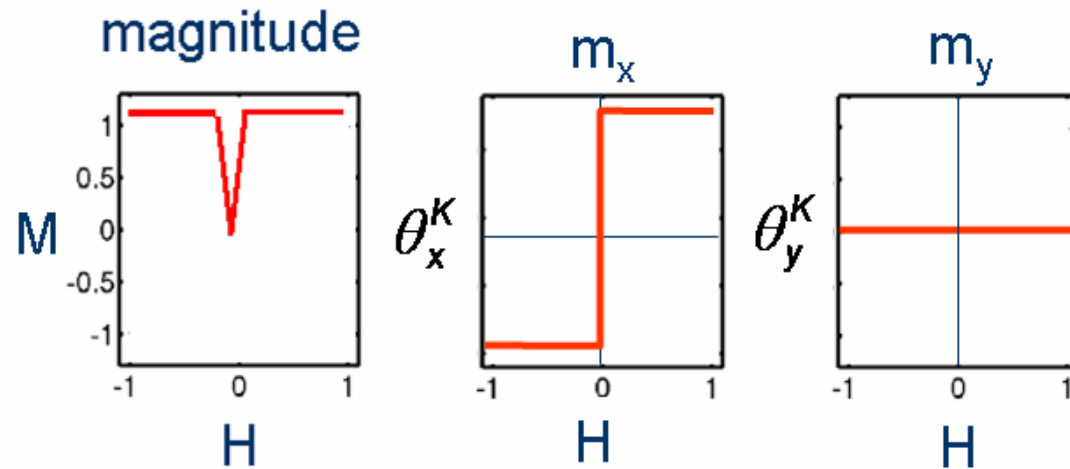


Domain formation:

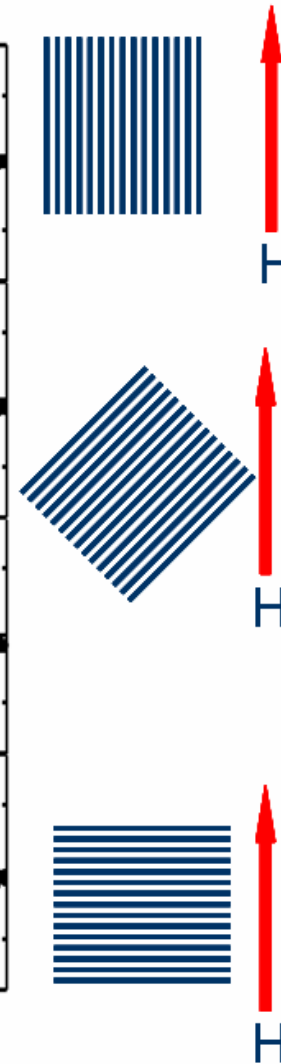
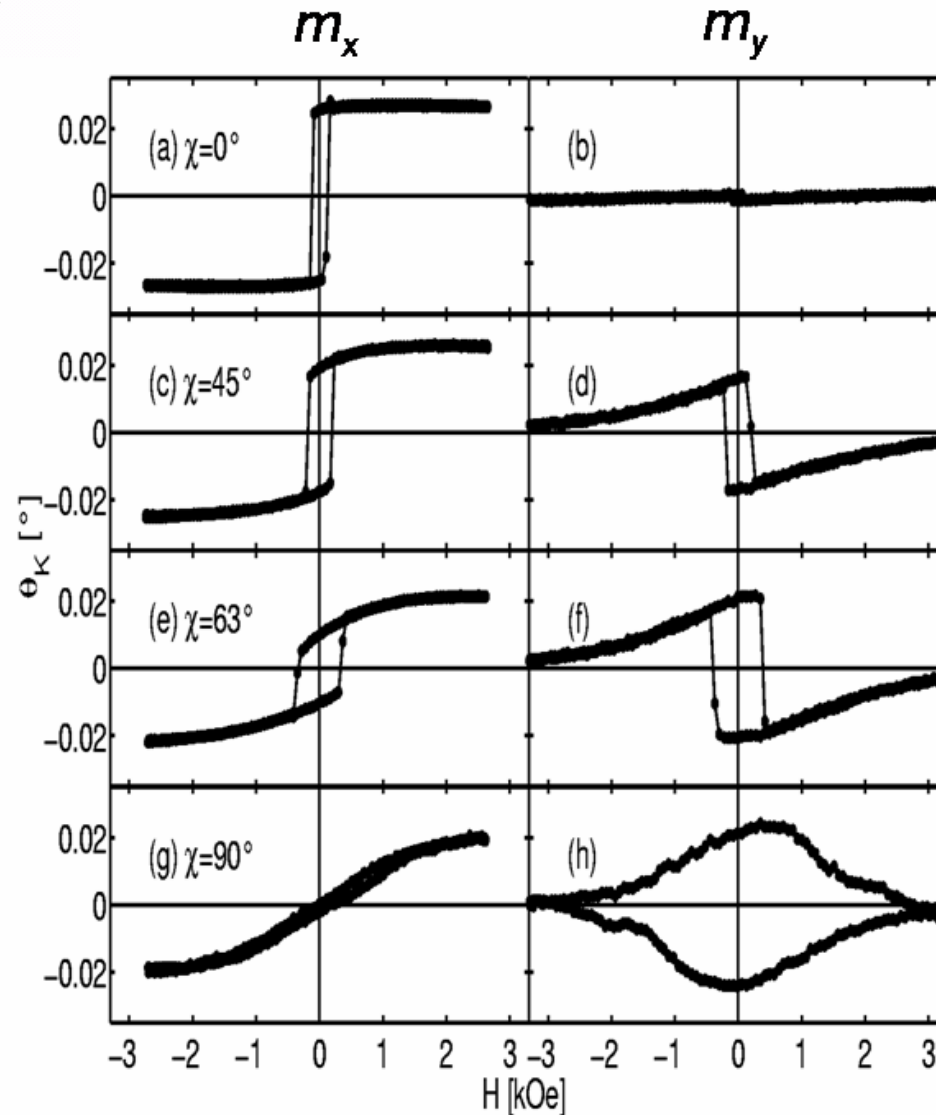
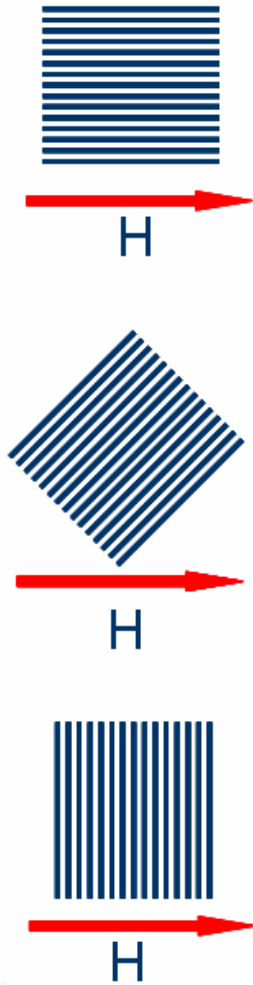
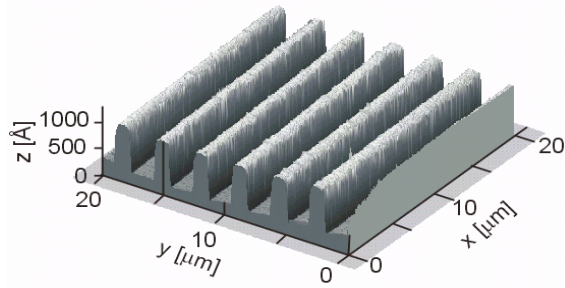


$$m_x = -1 \quad = 0 \quad = +1$$

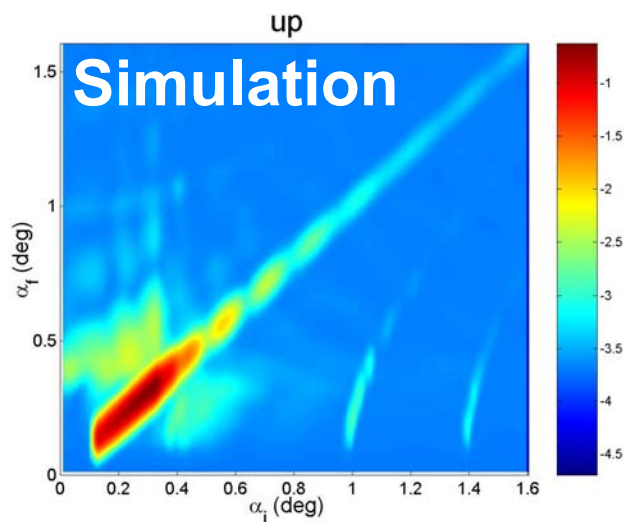
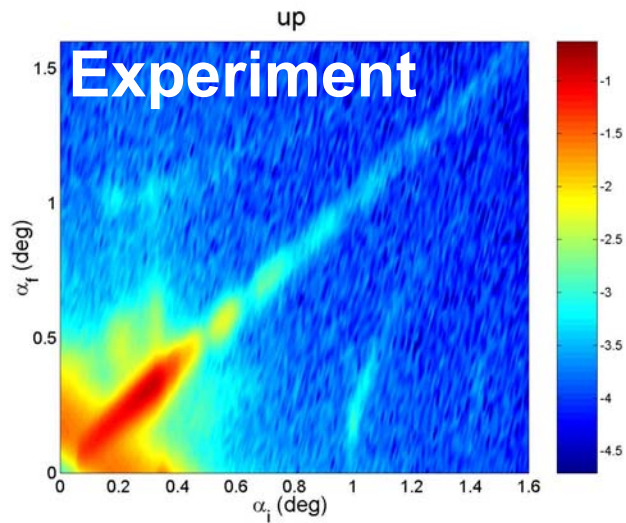
$$m_y = 0 \quad = 0 \quad = 0$$



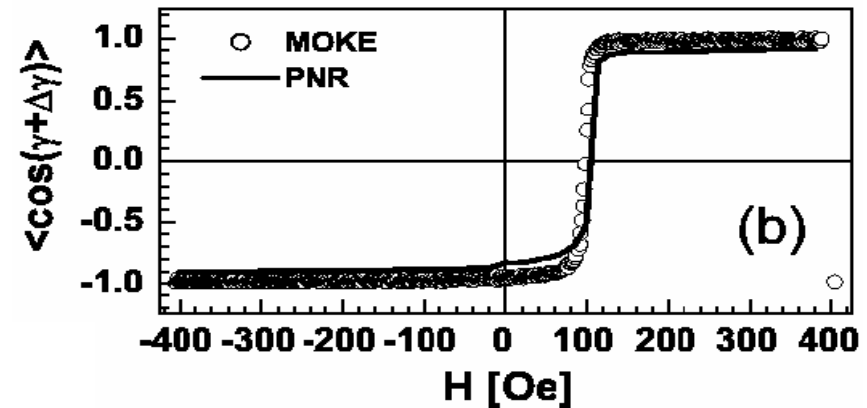
Magnetization reversal in a stripe array



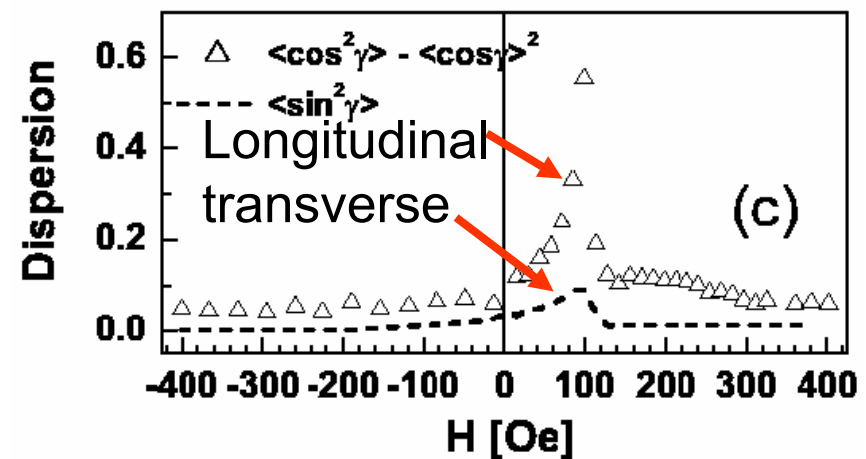
Magnetization reversal of a stripe array



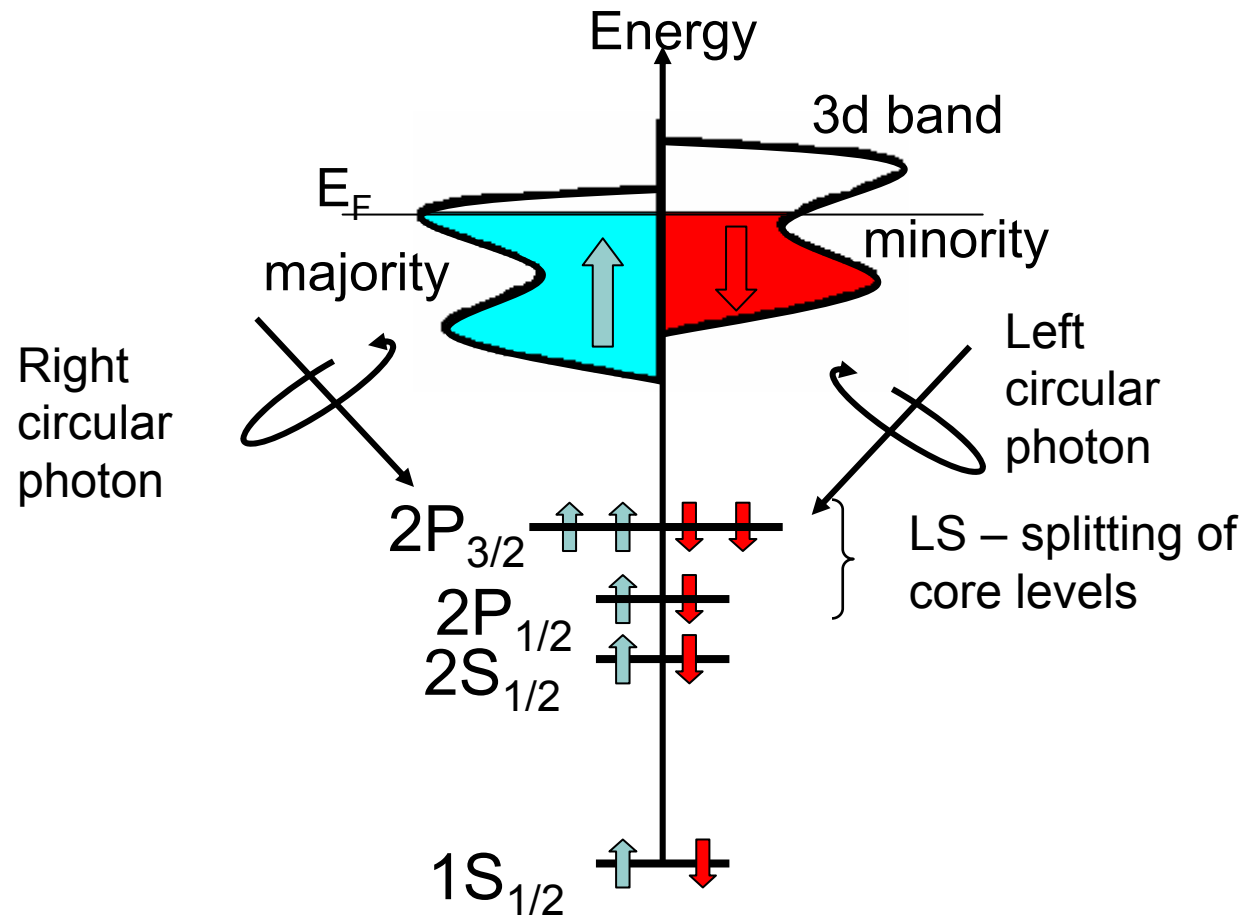
Normal component \parallel H:



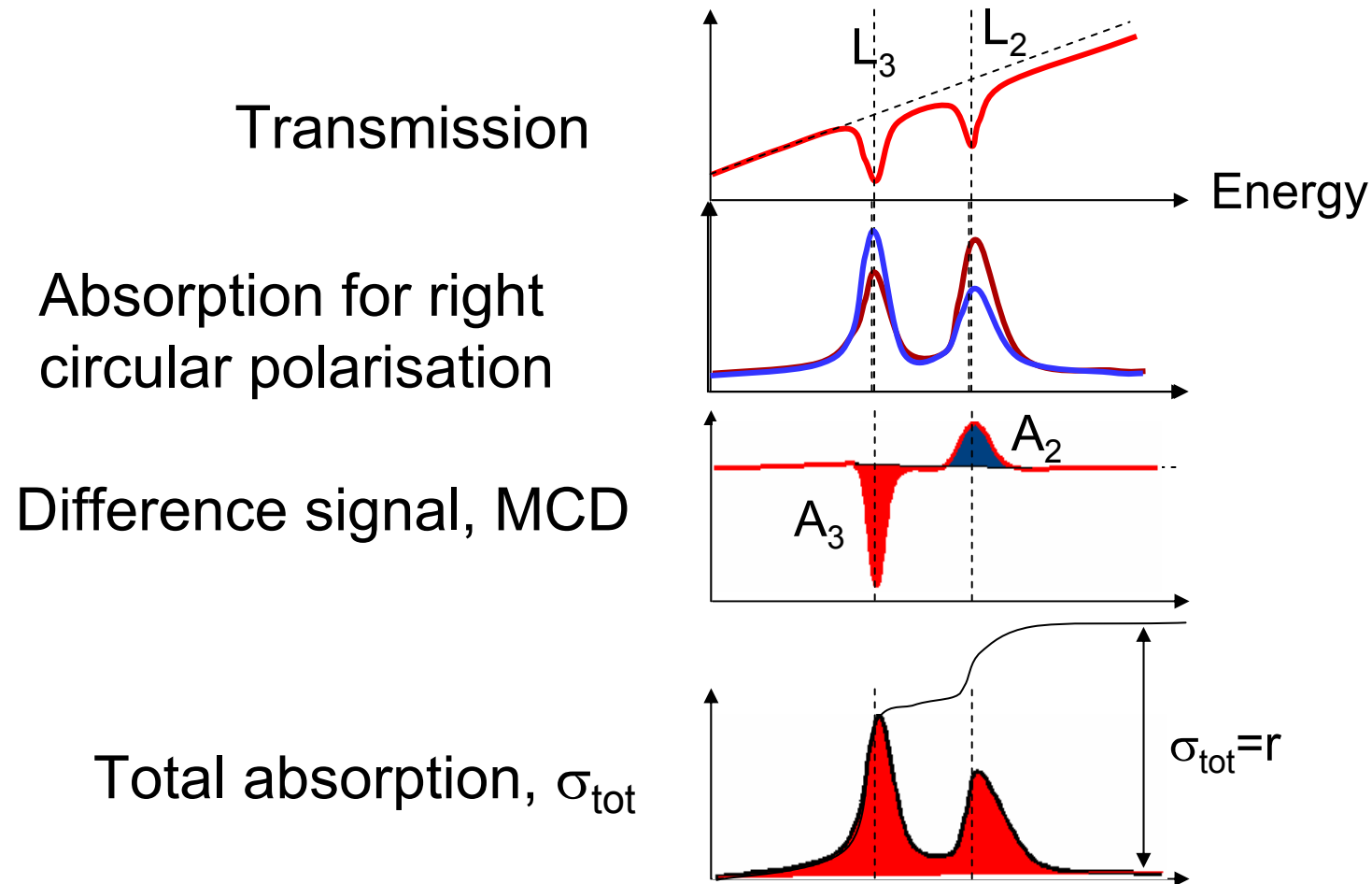
Fluctuations:



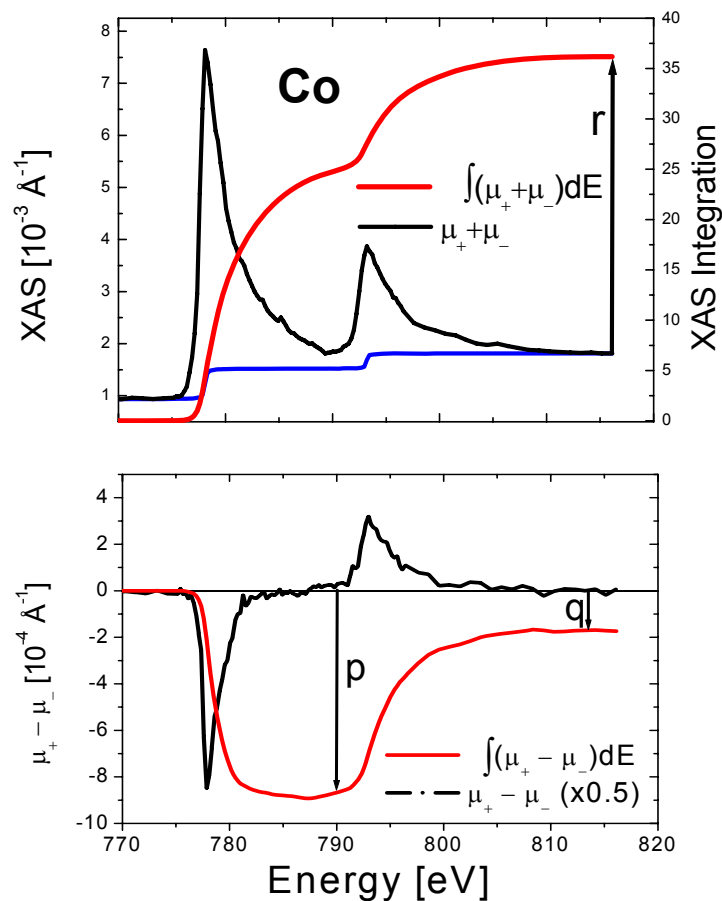
MCD for ferromagnetic metals



MCD for ferromagnetic metals



Spin and orbital moments from sum rules

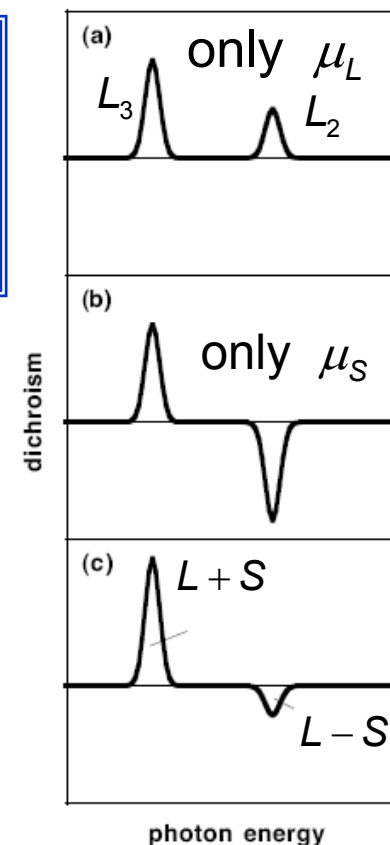


$$m_{\text{orb}}[\mu_B/\text{atom}] = -\frac{4qh}{3r}$$

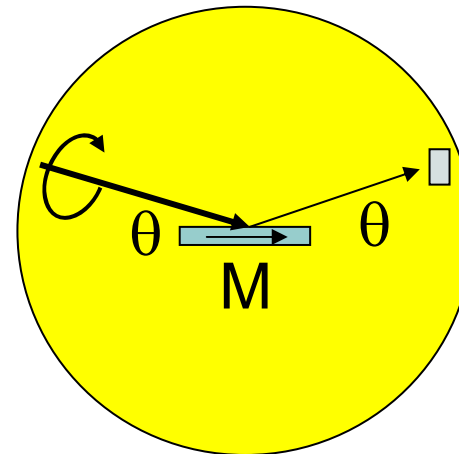
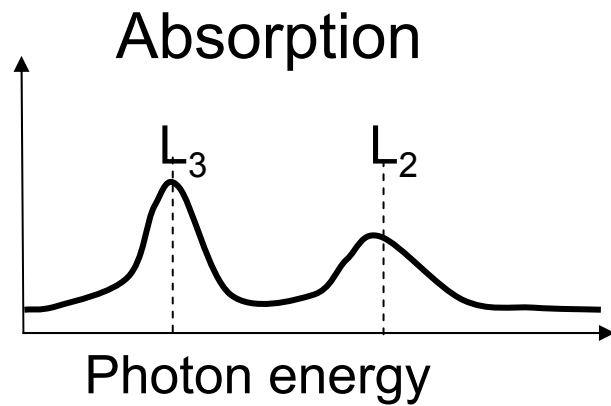
$$m_{\text{spin}}[\mu_B/\text{atom}] = -\frac{(6p-4q)h}{r}$$

$$\Rightarrow \frac{m_{\text{orb}}}{m_{\text{spin}}} = \frac{2q}{9p-6q}$$

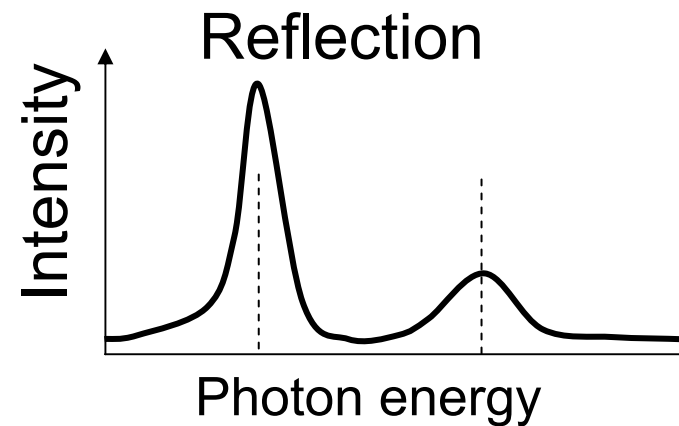
$$h = (10 - 3d)$$



From absorption to reflection



Kramers-Kronig
Transformation



X-ray magnetic scattering (XRMS)

$$f = (\mathbf{e}_f^* \cdot \mathbf{e}_i) \left\{ -r_e Z + \frac{3}{8\pi} \lambda [F_1^1 + F_{-1}^1] \right\}$$

Charge scattering
(non-resonant and
resonant)

$$+ i(\mathbf{e}_f^* \times \mathbf{e}_i) \cdot \hat{\mathbf{m}} \frac{3}{8\pi} \lambda [F_1^1 - F_{-1}^1]$$

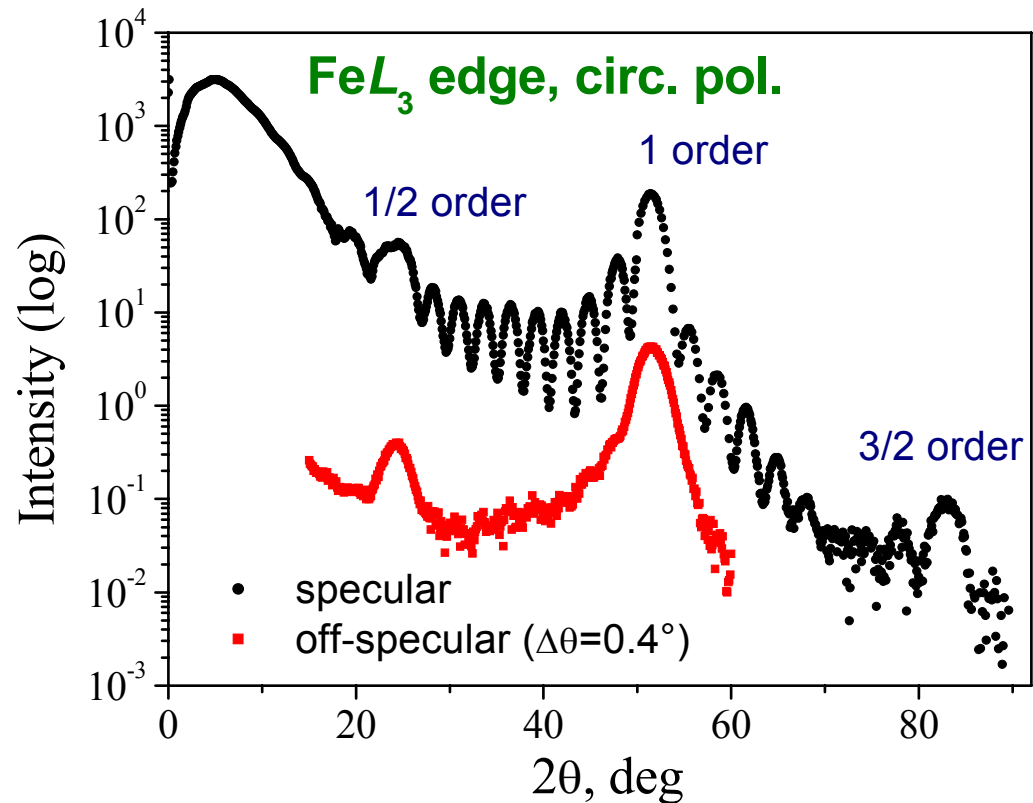
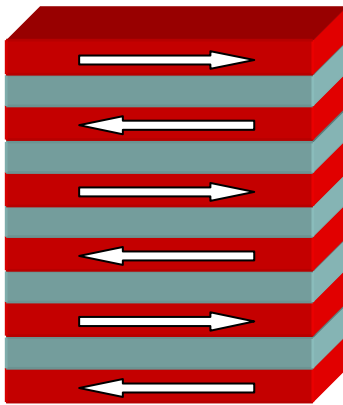
1st order in \mathbf{M} → Circular Dichroism

$$+ (\mathbf{e}_f^* \cdot \hat{\mathbf{m}})(\mathbf{e}_i^* \cdot \hat{\mathbf{m}}) \frac{3}{8\pi} \lambda [2F_0^1 - F_1^1 - F_{-1}^1]$$

2nd order in \mathbf{M} → Linear Dichroism

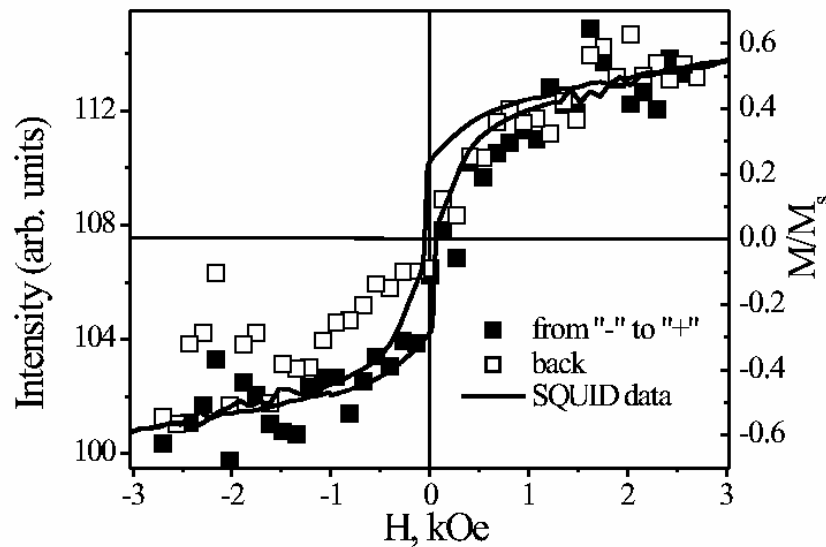
Resonant
magnetic
scattering

Fe/Cr superlattices: resonant magnetic scattering

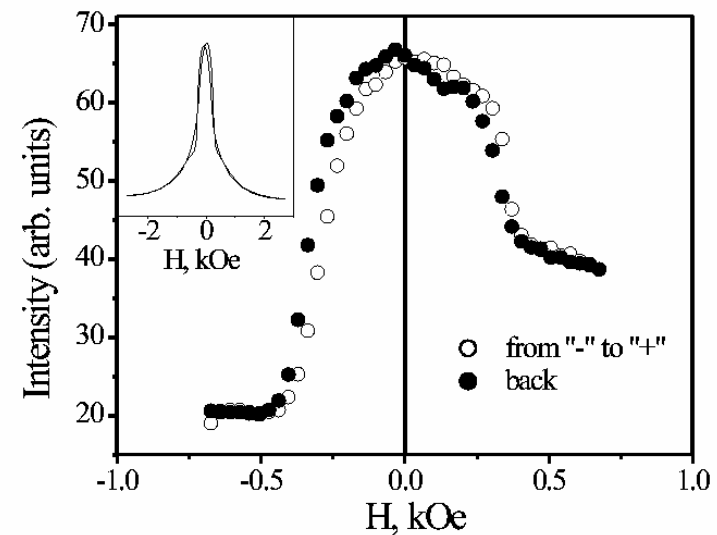


Fe/Cr superlattices: hysteresis

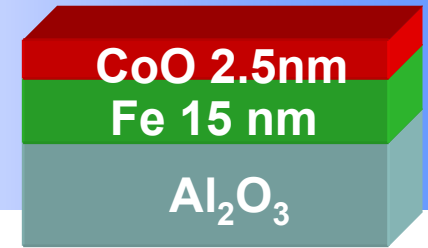
FM-hysteresis loop at
1. order peak, circular pol



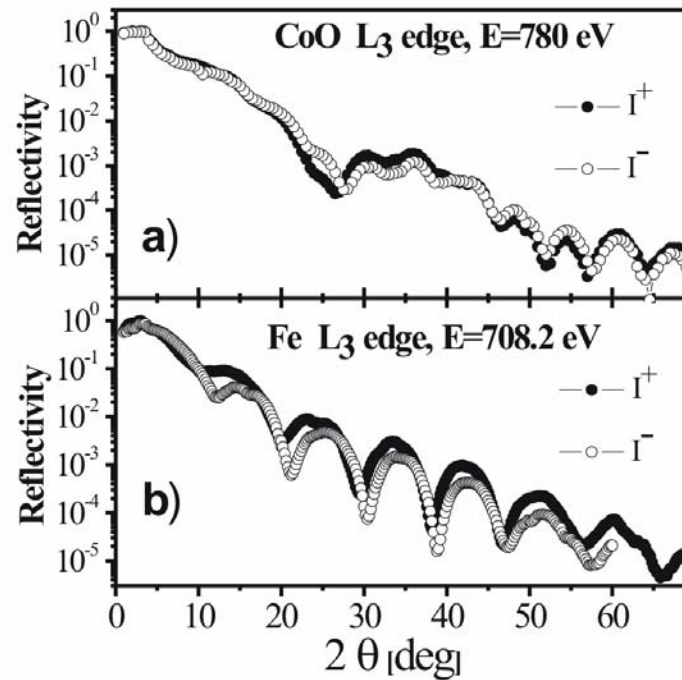
AFM-hysteresis loop at
1/2 order peak, linear pol



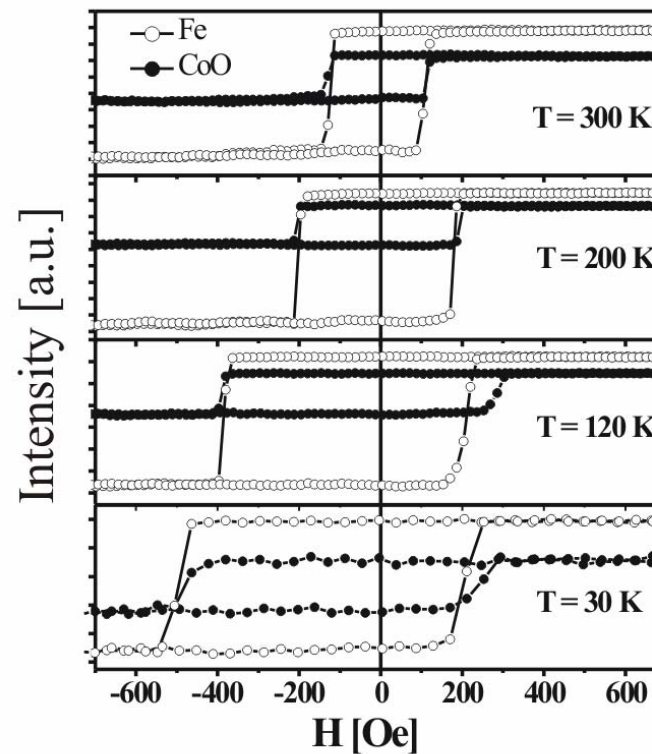
Element specific reflectivities and hysteresis of EB system



XRMS after field cooling
in $H_{FC} = + 2$ kOe at 30 K



Hystereses



$$T > T_N$$

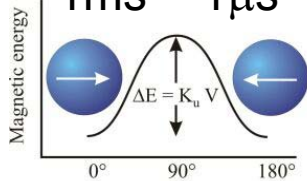
$$T < T_N < T_B$$

$$T_N > T > T_B$$

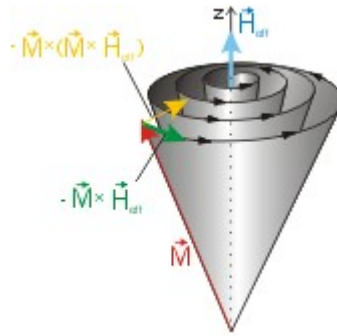
$$T < T_B$$

Time scales in magnetic materials

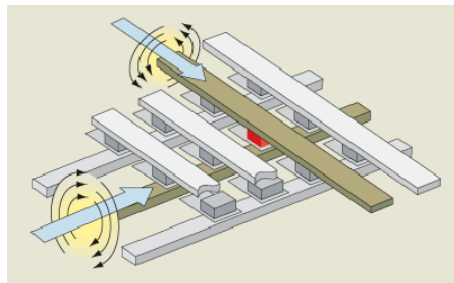
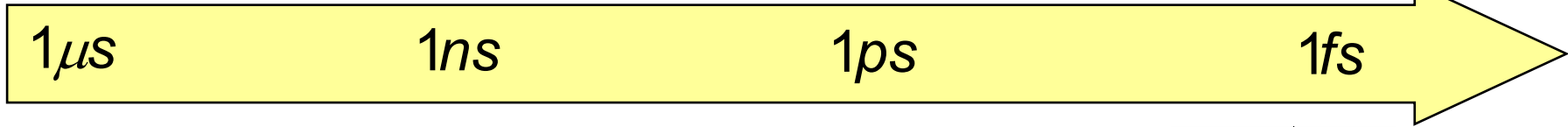
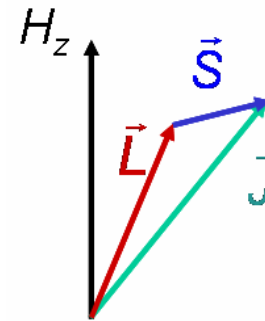
Thermal activations
1ms – 1μs



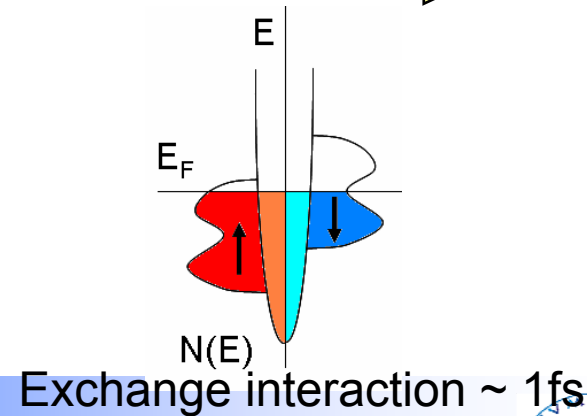
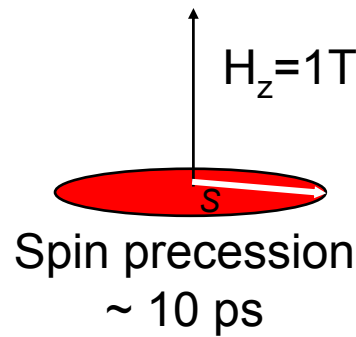
Spin-lattice relaxation and damping ~ 100ps



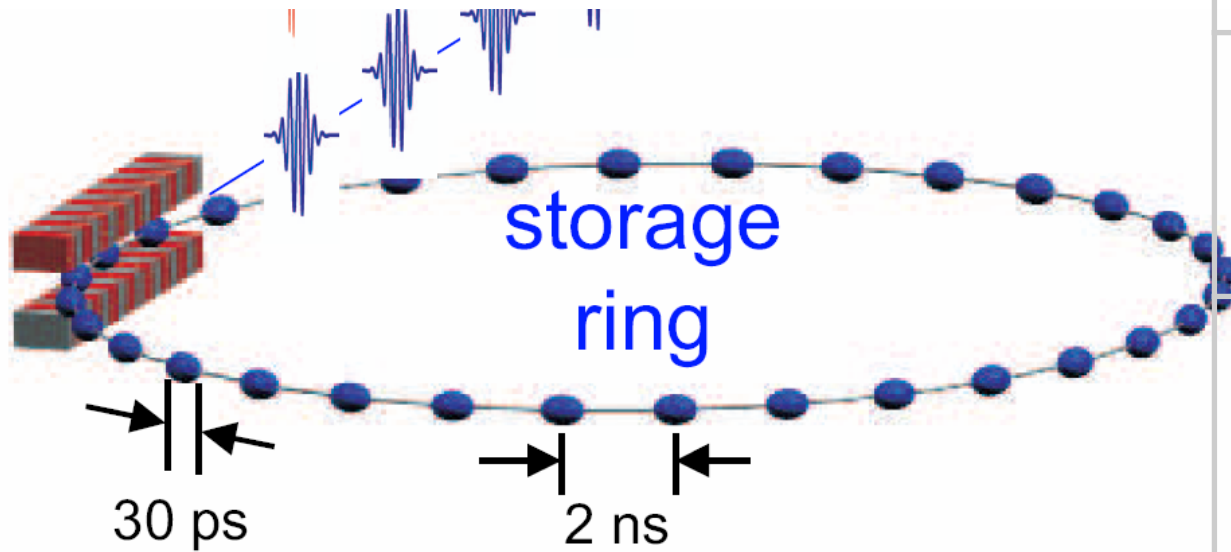
SL-coupling ~ 100fs



Domain nucleation + propagation ~ 1 ns

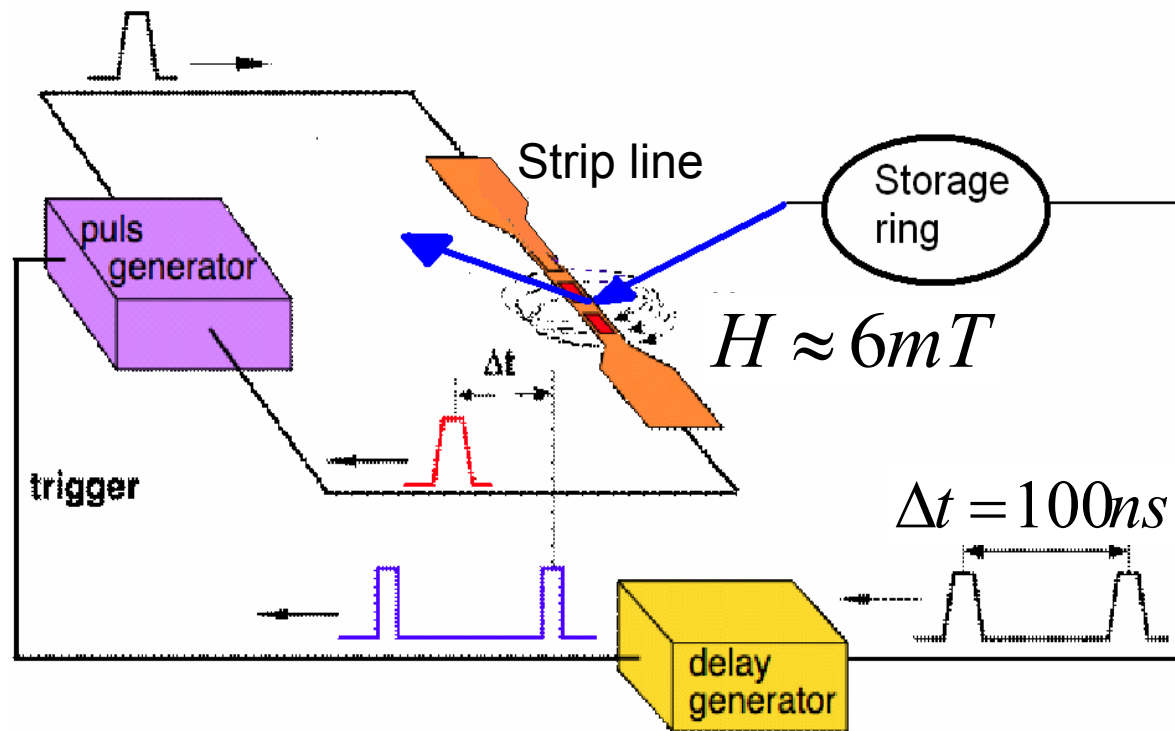


Time resolved reversal XRMS

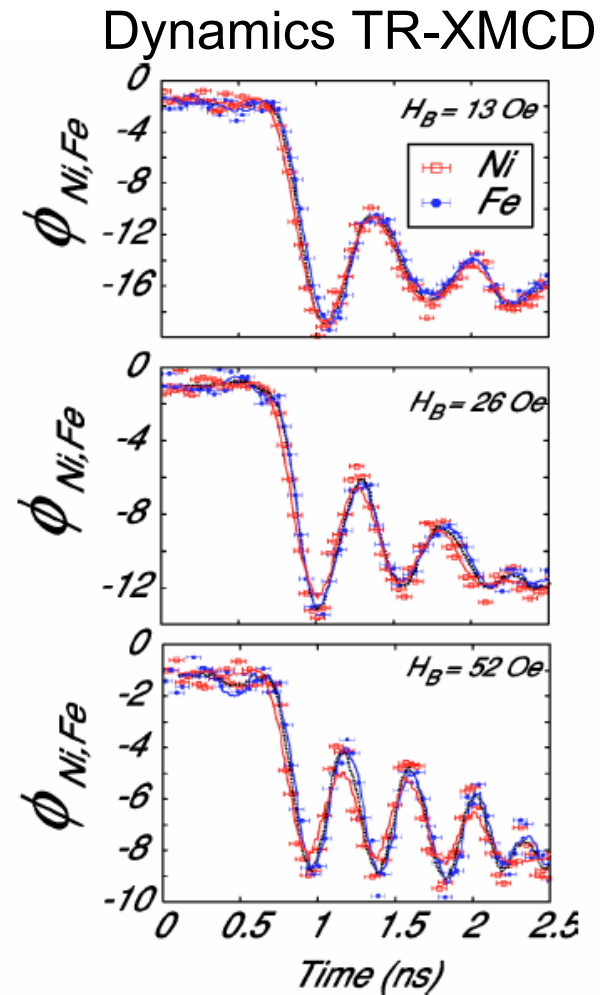
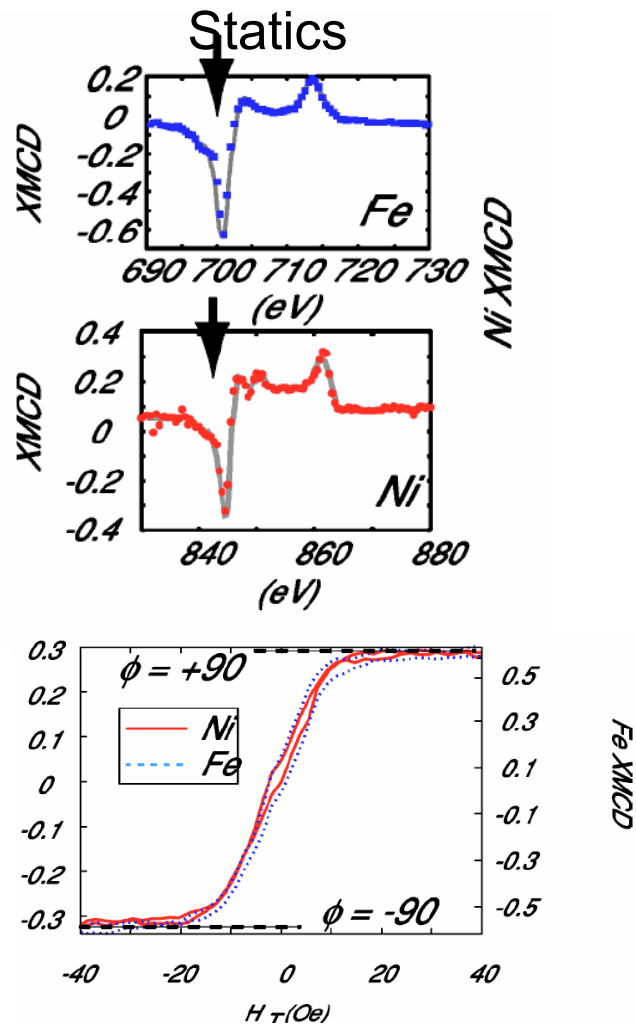


	BESSY low α
pulse width	30 ps
repeat time	2 ns
n_{bunch} current	400 0.5 μA

Time domain: pump probe technique



Precessional dynamics of elemental moments in a ferromagnetic alloy

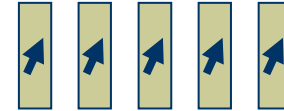


Summary: uses of PNR

- Layer resolved magnetization vector
- Magnetization profile independent of structural/chemical profile
- Absolute moment evaluation not obscured by substrate effects
- Distinction between different types of magnetization reversal
- High field measurements
- Domain distribution, domain sizes, domain walls
- Ferro- and antiferromagnetic structures
- Non-collinear magnetism
- Magnetic correlation lengths

Topics for PNR studies

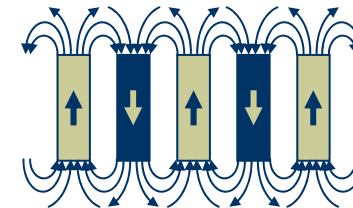
- **Quantitative vector magnetometry**



- **Domain configurations**

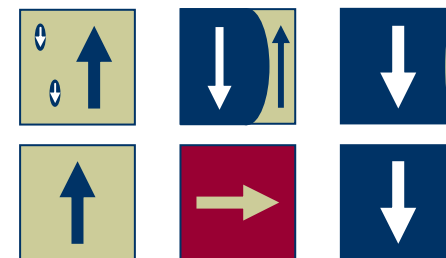


- **Interactions between elements**



- **Details of remagnetization**

- domain nucleation/
wall movement
- vs. rotation



Literature

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- *J.F. Ankner and G.P. Felcher*, J. Magn. Magn. Mater. **200**, 751 (1999)
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Advantages and disadvantages: XRMS

- 😊 High flux and large q
- 😊 Element specific
- 😊 Distinguish L and S
- 😞 Cross sections not known
- 😞 Indirect probe of the magnetisation
- 😞 Measure interference term between charge and magnetism
- 😊 Time resolved experiments
- 😞 Penetration depth

Thank you for your attention

