Revealing the hidden microstructure of materials: *characterising surface and interfacial phenomena using reflectometry*

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Science & Technology Facilities Council
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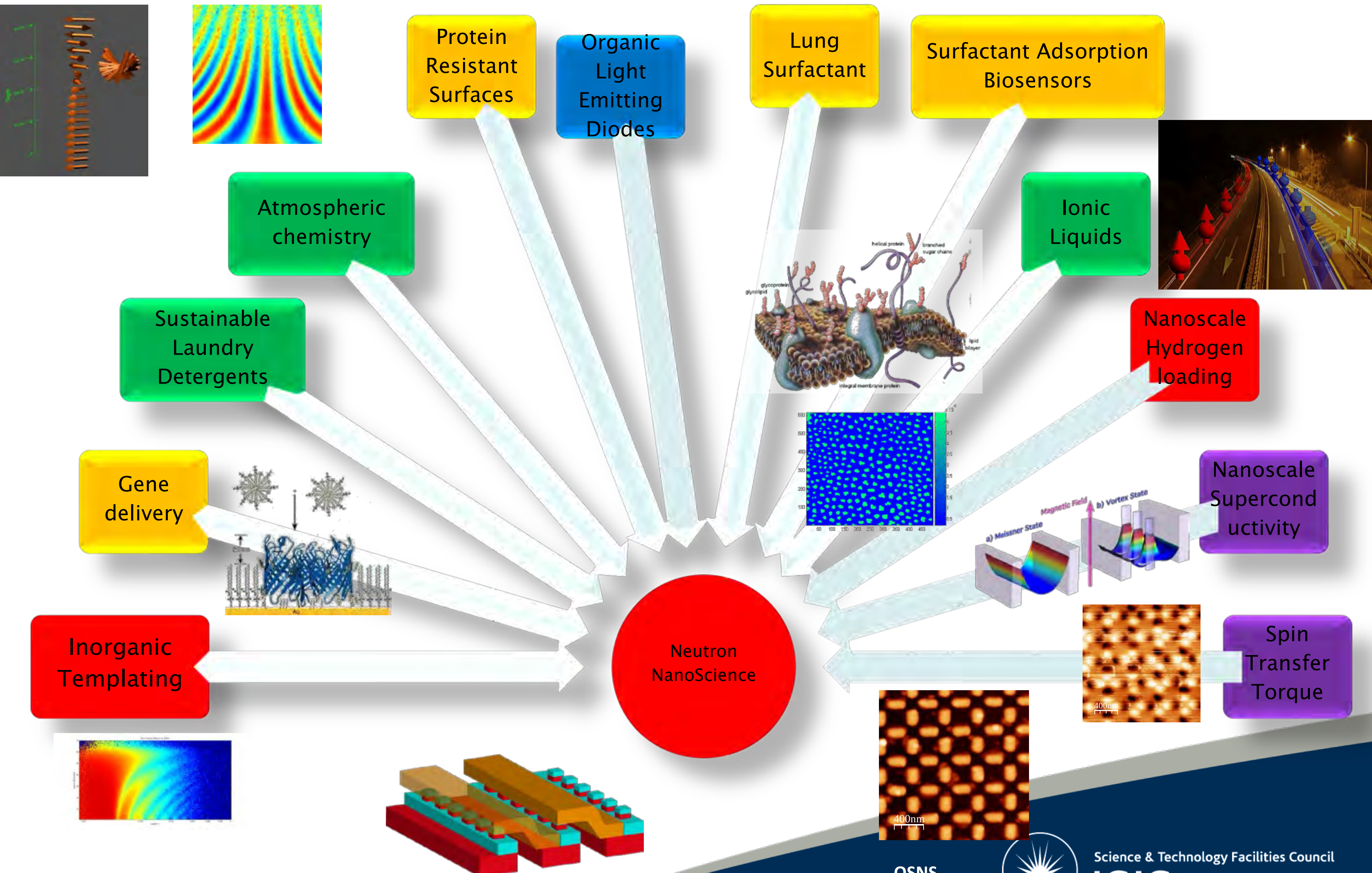
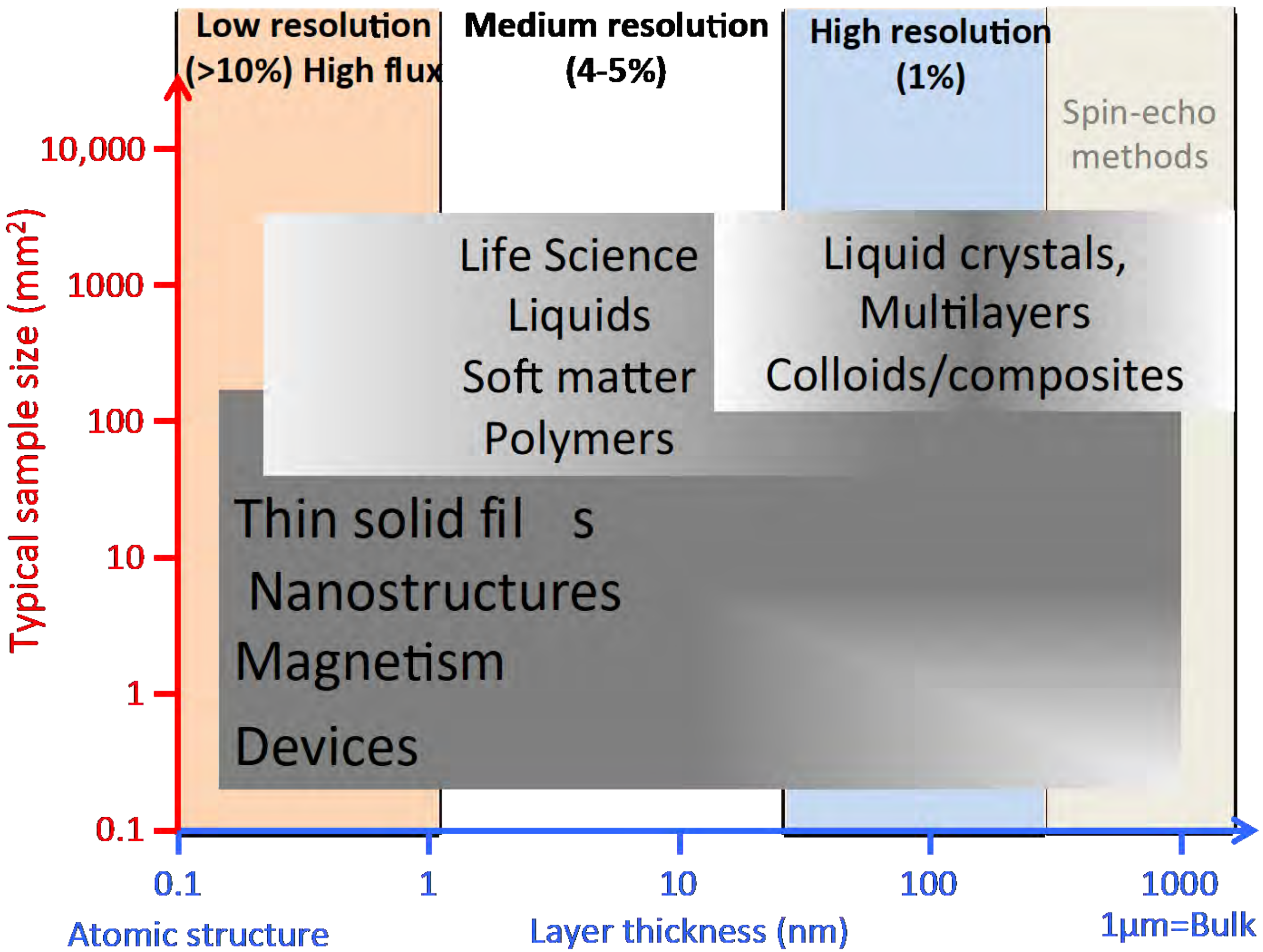
Outline

- Motivation
  - The importance of interfaces
- Reflectivity
  - Introduction to the basic Ideas
  - Information contained
    - Specular/Off-specular
  - Practical Considerations
- Examples
- Outlook
  - Bright!

Fundamentally driven, *technologically relevant*



Requirements



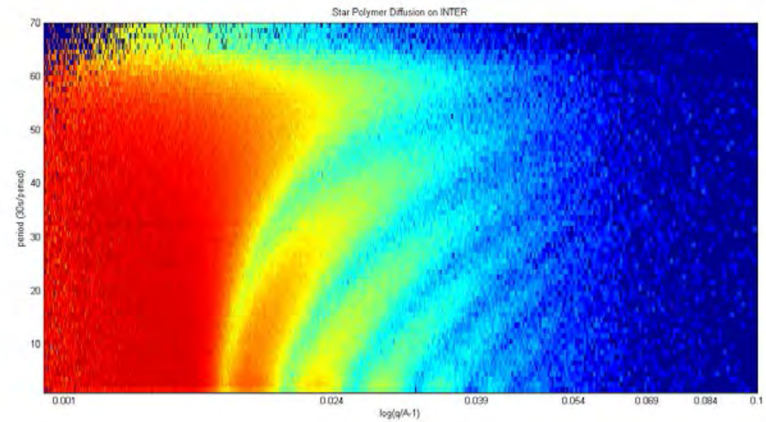
HW ESS (2015)

Scientific Drivers

- Weaker Effects
  - Magnetisation
    - 1nm (2u.c.) interface sensitivity
    - 2DEG, exchange bias, injected spin
    - 1emu cm^{-3}
  - Changes in scattering length density
  - Parametric Studies
- Smaller samples
  - Arrays
    - Spin injection
    - Spin-ice
  - Homogeneity
  - Compatible with TEM, x-ray, SQUID…
  - But some people have large domains

- Kinetics
  - new kinetics on sub-second timescale
- Excitations
  - Interfacial and surface magnon-phonons
- Complexity
- Black Box usage

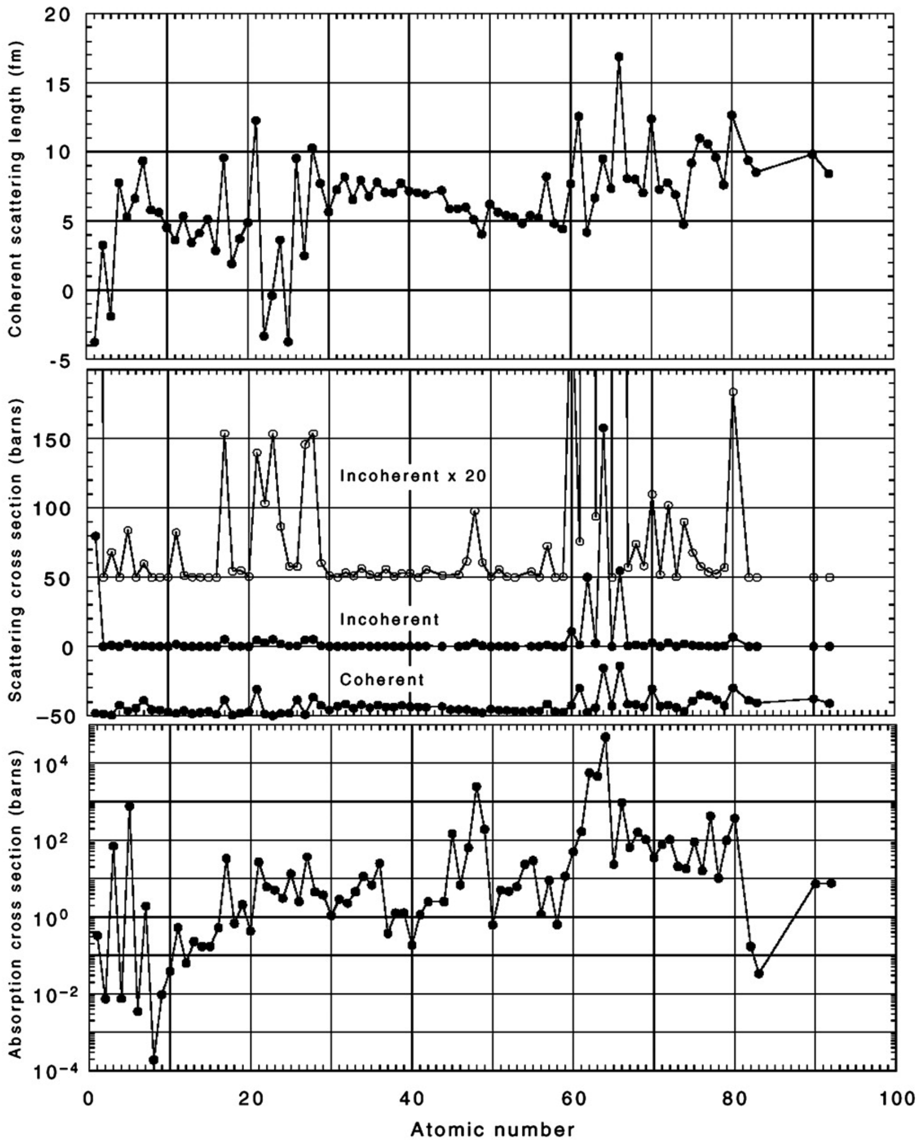
- Dilute systems
- Industrial relevance



Specular Scattering

Neutron and x-ray reflectivity

How do we connect the scattering length profile with the reflectivity



- Mass 1.67×10^{-27}kg 1.008665 atomic units
- Charge 0 $(1.5 \pm 2.2) \times 10^{-22}$proton charge
- Spin ½
- Magnetic moment $-1.913\mu_N$
- Electric dipole moment

$< 6 \times 10^{-25}$e-cm

$$\lambda = \frac{h}{mv}$$

$$E = \frac{h^2}{2m\lambda^2}$$

Isotope Dependence

Nickel Isotope	Scattering length *b (fm)*
^{58}Ni	15.0(5)
^{60}Ni	2.8(1)
^{61}Ni	7.60(6)
^{62}Ni	-8.7(2)
^{64}Ni	-0.38(7)

Hydrogen Isotope	Scattering length *b(fm)*
1H	-3.7409(11)
2D	6.674(6)
3T	4.792(27)
O	5.803

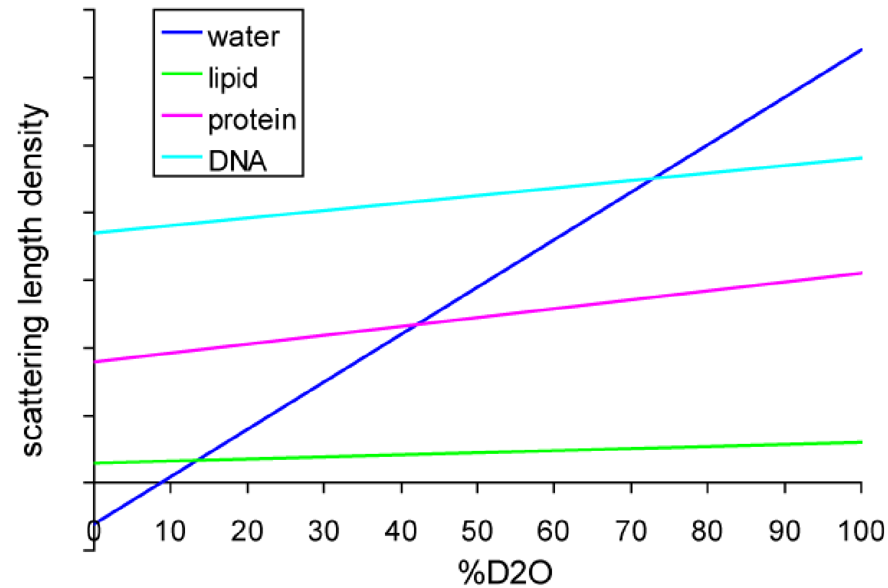
$$
\begin{aligned}
|11\rangle &= \uparrow\uparrow \\
|10\rangle &= (\uparrow\downarrow + \downarrow\uparrow)/\sqrt{2} \\
|1\ -1\rangle &= \downarrow\downarrow \\
|00\rangle &= (\uparrow\downarrow - \downarrow\uparrow)/\sqrt{2}
\end{aligned}
$$

- Isotopic substitution for contrast
- Isotopic substitution to move peak positions in spectroscopy
- Incoherent scattering

Scattering length density (SLD)

$$SLD = \sum_i b_i \frac{DN_a}{M_w}$$

Solvent	d (h form) (´ 10^{10} cm^{-2})	d (d form) (´ 10^{10} cm^{-2})	Polymer	d (h form) (´ 10^{10} cm^{-2})	d (d form) (´ 10^{10} cm^{-2})
Water	- 0.56	+ 6.38	PB	- 0.47	+ 6.82
Octane	- 0.53	+ 6.43	PE	- 0.33	+ 8.24
Cyclohexane	- 0.28	+ 6.70	PS	+ 1.42	+ 6.42
Toluene	+ 0.94	+ 5.66	PEO	+ 0.64	+ 6.46
Chloroform	+ 2.39	+ 3.16	PDMS	+ 0.06	+ 4.66
Carbon Tet.	+ 2.81		PMMA	+ 1.10	+ 7.22



SLD

https://www.ncnr.nist.gov/resources/activation/

https://www.ncnr.nist.gov/resources/activation/

NIST Center for Neutron Research

NIST National Institute of Standards and Technology

Home Instruments Science Experiments SiteMap

Material

Co

Neutron Activation

For rabbit system Calculate

Thermal flux	Cd ratio	Thermal/fast ratio
1e8	0	0
Mass	Time on beam	Time off beam
1	10	1 y

Absorption and Scattering

Calculate

Density	Thickness
	1
Source neutrons	Source X-rays
1 Ang	Cu Ka

Neutron activation and scattering calculator

This calculator uses neutron cross sections to compute activation on the sample given the mass in the sample and the time in the beam, or to preform scattering calculations for the neutrons which are not absorbed by the sample.

1. Enter the sample formula in the material panel.

2. To perform activation calculations, fill in the thermal flux, the mass, the time on and off the beam, then press the calculate button in the neutron activation panel.

3. To perform scattering calculations, fill in the wavelength of the neutron and/or xrays, the thickness and the density (if not given in the formula), then press the calculate button in the absorption and scattering panel.

Co at 8.90 g/cm^3

Source neutrons: 1.000 Å = 81.80 meV = 3956 m/s
Source X-rays: 1.542 Å = 8.042 keV

1/e penetration depth (cm)		Scattering length density (10^{-6}/Å^2)		Scattering cross section (1/cm)		X-ray SLD (10^{-6}/Å^2)	
abs	0.532	real	2.265	coh	0.071	real	63.020
abs+incoh	0.432	imag	-0.009	abs	1.881	imag	-9.141
abs+incoh+coh	0.418	incoh	5.621	incoh	0.437		

Neutron transmission is 9.86% for 1 cm of sample (after absorption and incoherent scattering).
Transmitted flux is 9.855e+6 n/cm^2/s for a 1e8 n/cm^2/s beam.

Interference effects

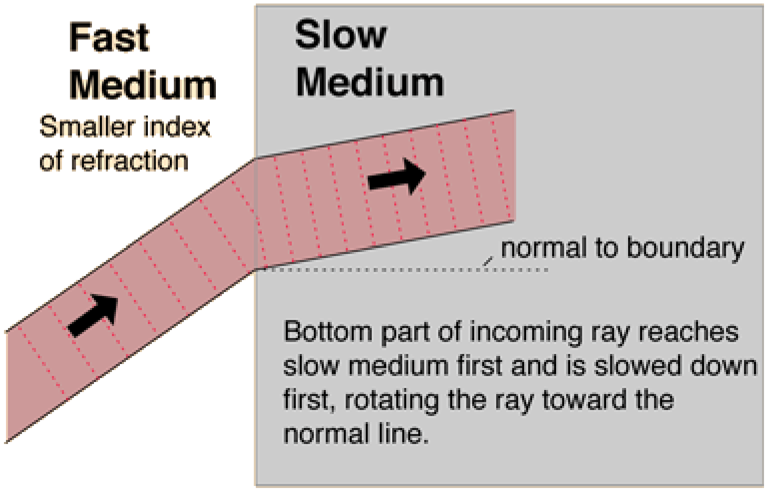
- Fresnel reflection 1815

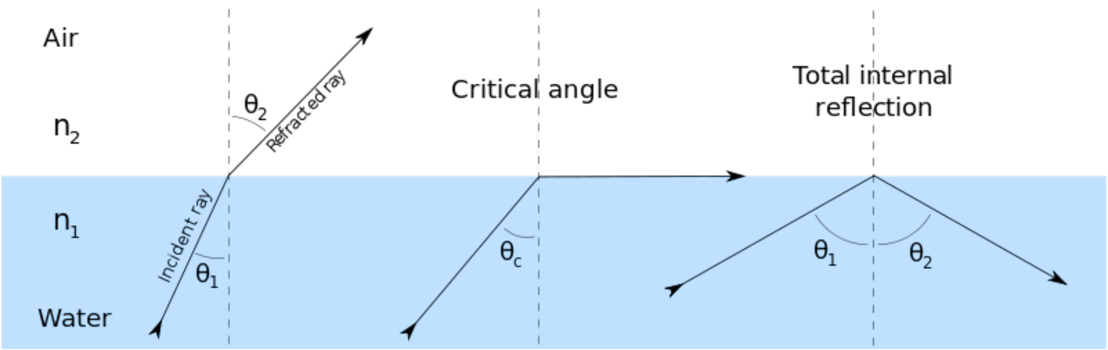
Refractive Index

$$n = \frac{c}{v}$$

- n varies with wavelength: dispersion

$$n_1 \sin\theta_1 = n_2 \sin\theta_2$$





http://hyperphysics.phy-astr.gsu.edu
en.wikipedia.org

$$n = 1 - \lambda^2 A - i\lambda B \qquad (1)$$

$$A = \frac{Nb}{2\pi} \qquad (2)$$

$$B = \frac{N(\sigma_a + \sigma_i)}{4\pi} \qquad (3)$$

$$n = 1 - \alpha - i\beta \qquad (1)$$

$$\alpha = \frac{N\lambda^2|r_e}{2\pi} \qquad (2)$$

$$\beta = \frac{\lambda\mu}{4\pi} \qquad (3)$$

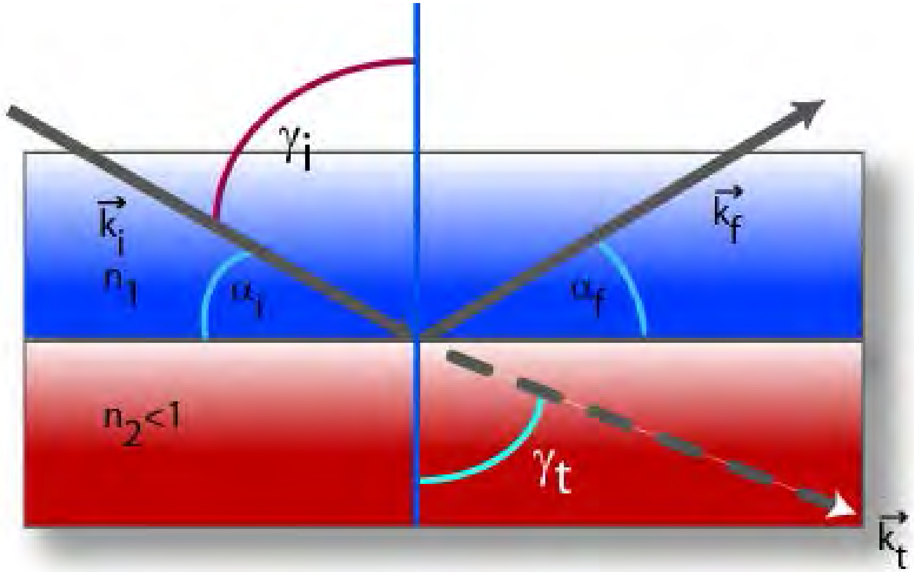
n <1 Total External reflection

Critical Reflection

- $$\frac{\cos \alpha_i}{\cos \alpha_f} = \frac{n_2}{n_1}$$
- At the critical angle
$$\frac{\cos \alpha_i}{\cos 0} = n$$

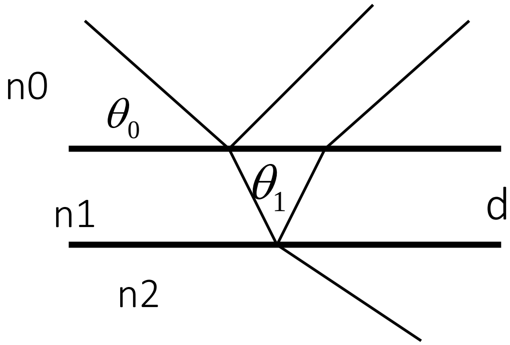
$$\begin{aligned}
Q_c &= \frac{4\pi}{\lambda} 2k \sin \alpha_c \\
&= 2k\sqrt{1 - \cos^2 \alpha_c} \\
&= \sqrt{4k^2\left(1 - n^2\right)} \\
&\cong \sqrt{4k^2 \cdot 2\delta} \\
&= \sqrt{16\pi N b}
\end{aligned}$$

- Q_c only depends on the material!



Material	θ_c / Å
Ni	0.1
Cu	0.083
Al	0.047
Si	0.047
D_2O	0.082

Fresnel's law for a surface and thin film



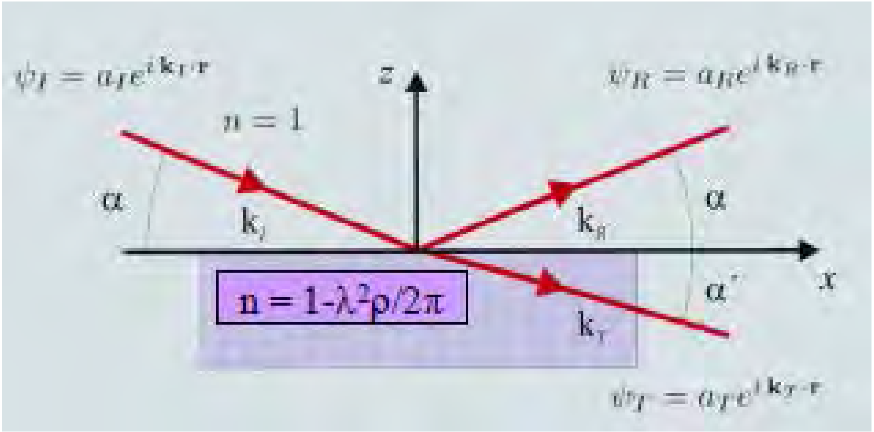
$$k_i = n_i \sin\theta$$

$$\beta_i = \frac{2\pi}{\lambda} n_i d_i \sin\theta_i$$

$$R(Q) = \left| \frac{r_{01} + r_{12}\exp(-2i\beta_i)}{1 + r_{01}r_{12}\exp(-2i\beta_i)} \right|^2$$

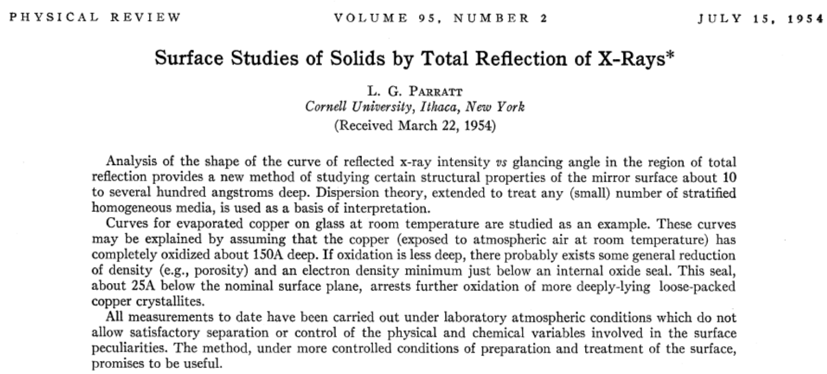
$$r_{ij} = \frac{a_r}{a_i} = \frac{k_i - k_j}{k_i + k_j}$$

$$t_{ij} = \frac{a_j}{a_i} = \frac{2k_i}{k_i + k_j}$$



http://www.che.udel.edu/cns/pdf/Reflectometry.pdf

Parratt Iteration



PHYSICAL REVIEW VOLUME 95, NUMBER 2 JULY 15, 1954

Surface Studies of Solids by Total Reflection of X-Rays*

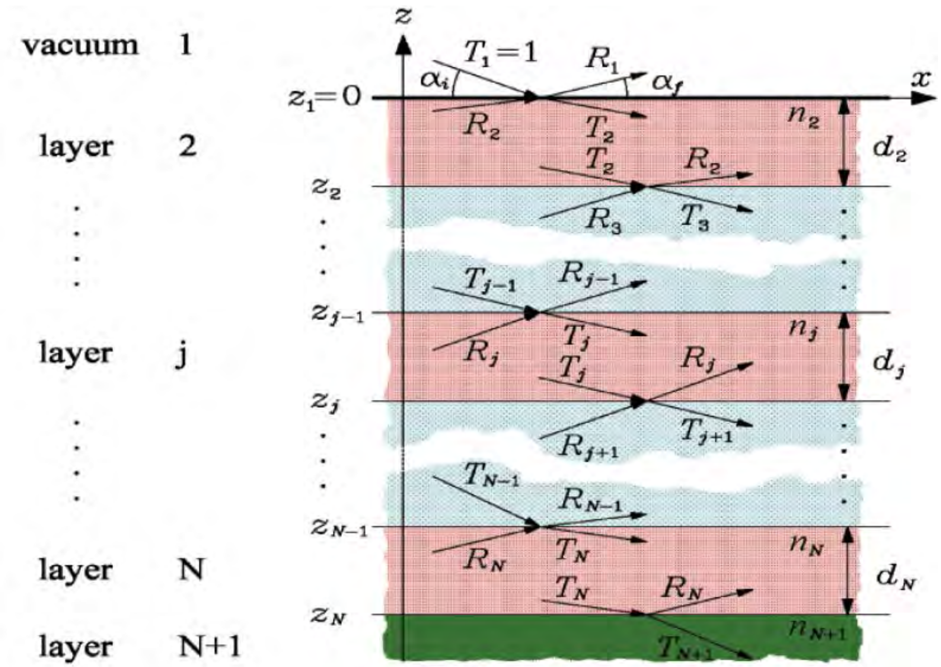
L. G. Parratt
Cornell University, Ithaca, New York

(Received March 22, 1954)

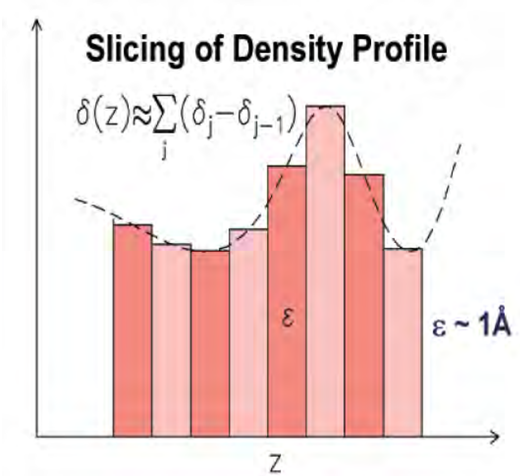
Analysis of the shape of the curve of reflected x-ray intensity *vs* glancing angle in the region of total reflection provides a new method of studying certain structural properties of the mirror surface about 10 to several hundred angstroms deep. Dispersion theory, extended to treat any (small) number of stratified homogeneous media, is used as a basis of interpretation.

Curves for evaporated copper on glass at room temperature are studied as an example. These curves may be explained by assuming that the copper (exposed to atmospheric air at room temperature) has completely oxidized about 150A deep. If oxidation is less deep, there probably exists some general reduction of density (e.g., porosity) and an electron density minimum just below an internal oxide seal. This seal, about 25A below the nominal surface plane, arrests further oxidation of more deeply-lying loose-packed copper crystallites.

All measurements to date have been carried out under laboratory atmospheric conditions which do not allow satisfactory separation or control of the physical and chemical variables involved in the surface peculiarities. The method, under more controlled conditions of preparation and treatment of the surface, promises to be useful.

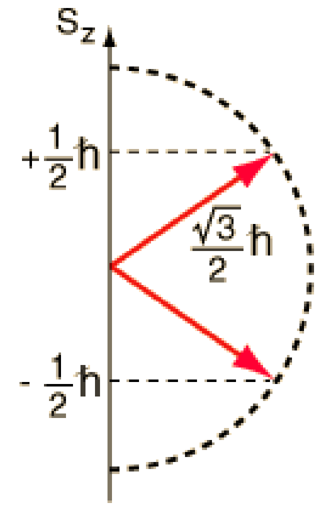


$$X_j = \frac{R_j}{T_j} = \exp(-2ik_{z,j}z_j)\frac{r_{j,j+1} + X_{j+1}\exp(2ik_{z,j+1}z_j)}{1 + r_{j,j+1}X_{j+1}\exp(2ik_{z,j+1}z_j)}$$

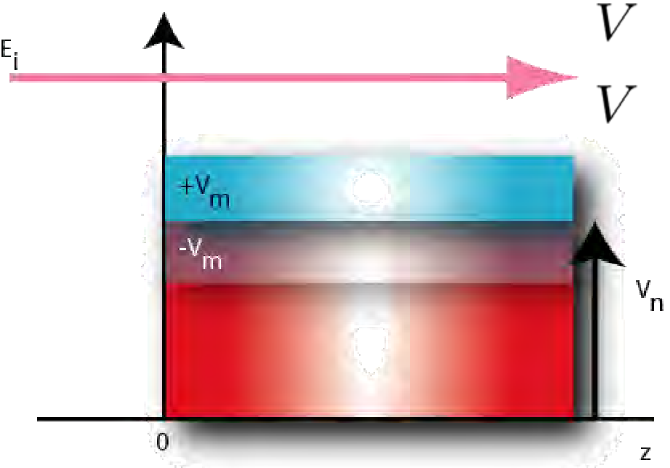


Can now simulate profile with a *"slice and dice"* approach

Using the neutron's spin



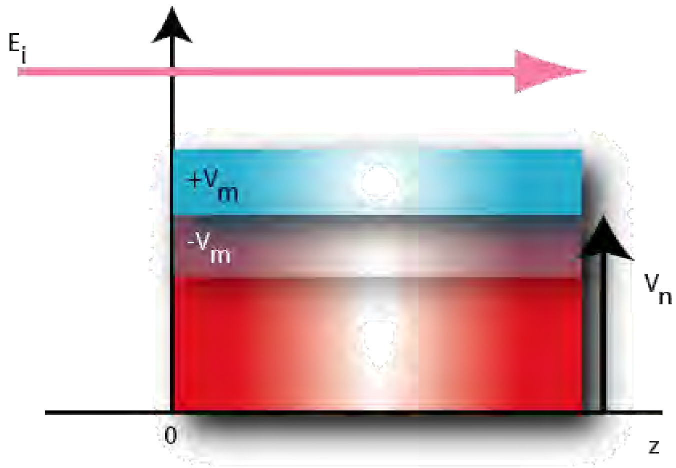
- The neutron is a spin ½ particle
- The neutron possesses an intrinsic magnetic moment: spin
- Caution…



Nuclear Magnetic

$$
\begin{aligned}
V &= V_n + V_m && (1)\\
V &= \frac{\hbar}{2\pi m}Nb \; -\mu\cdot\mathbf{B} && (2)\\
&= -4\pi\mu_n\cdot M && (3)\\
&= -\frac{2\hbar\pi^2}{m_n}p(z)\sin(\theta) && (4)\\
&= \frac{2\hbar\pi^2}{m_n}\frac{M_p}{M_s}N\frac{m_n\mu_n\mu_0}{2\pi\hbar^2}\mu_s && (5)
\end{aligned}
$$

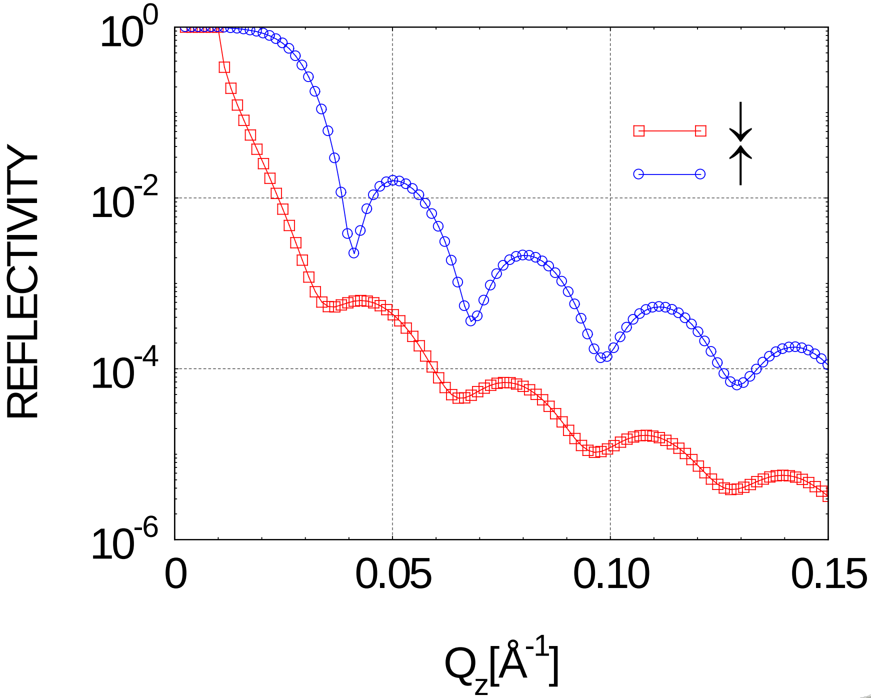
PNR from a single layer

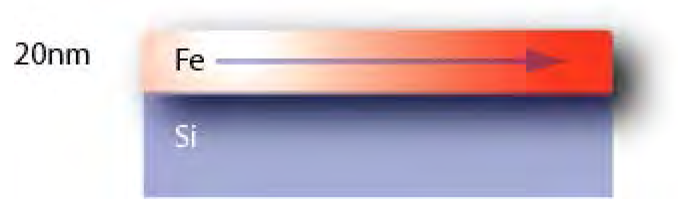


$$V = V_n + V_m \quad (1)$$

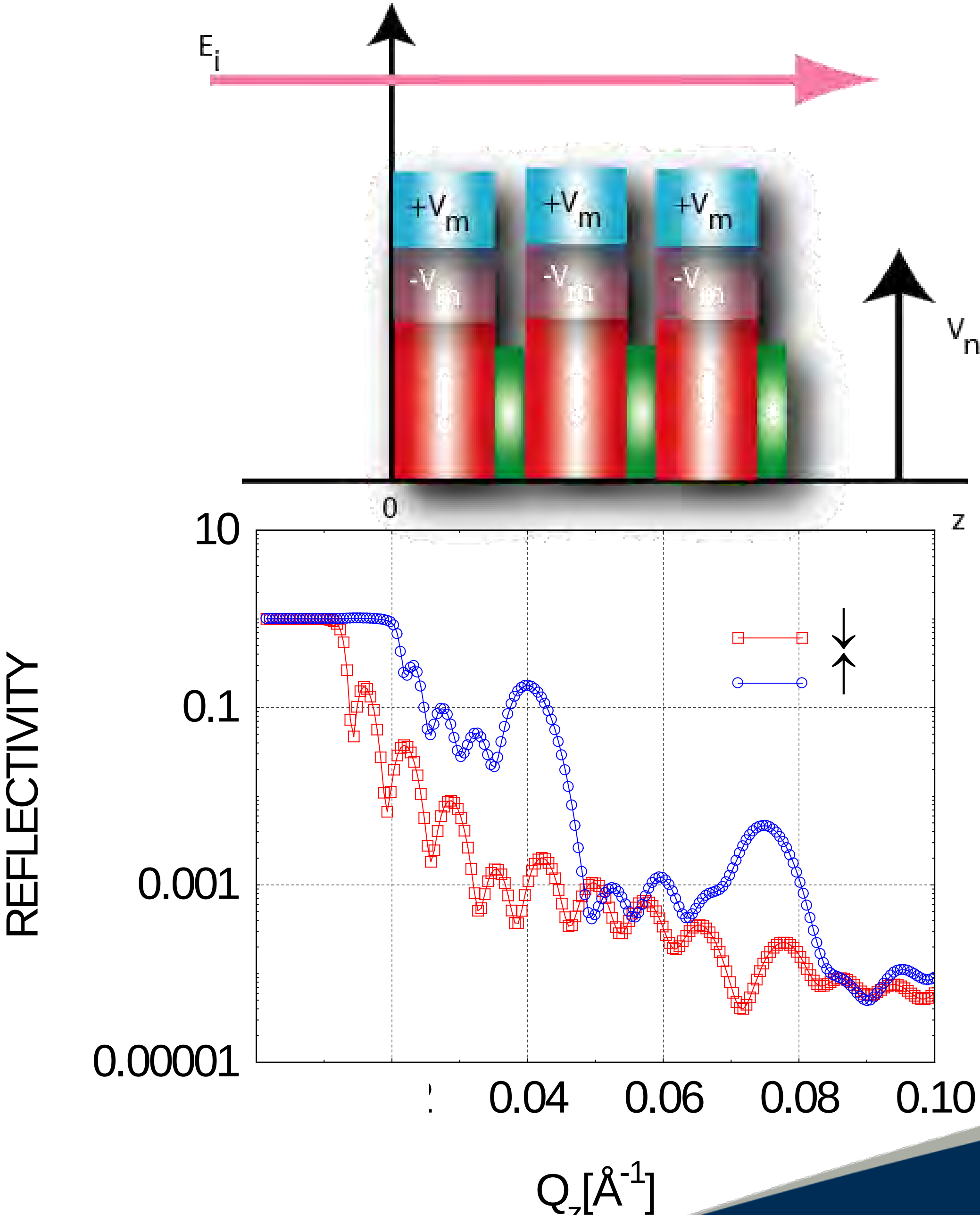
$$V = \frac{\hbar}{2\pi m} N(b \pm p) \quad (2)$$

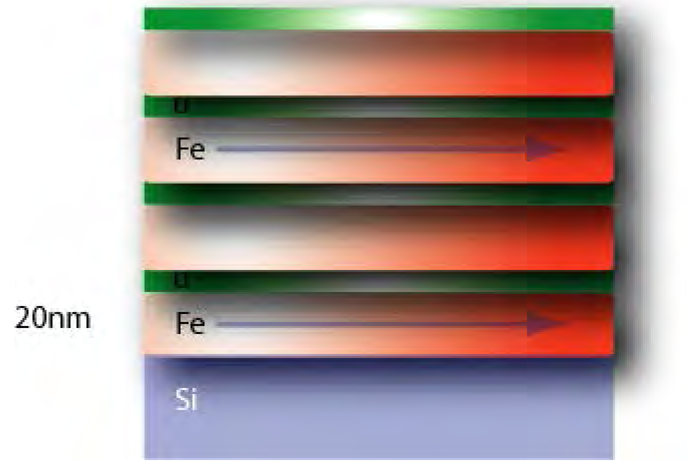
$$p = (2.695 \times 10^{-4} / \mu_B) \, |\vec{\mu_i}|$$





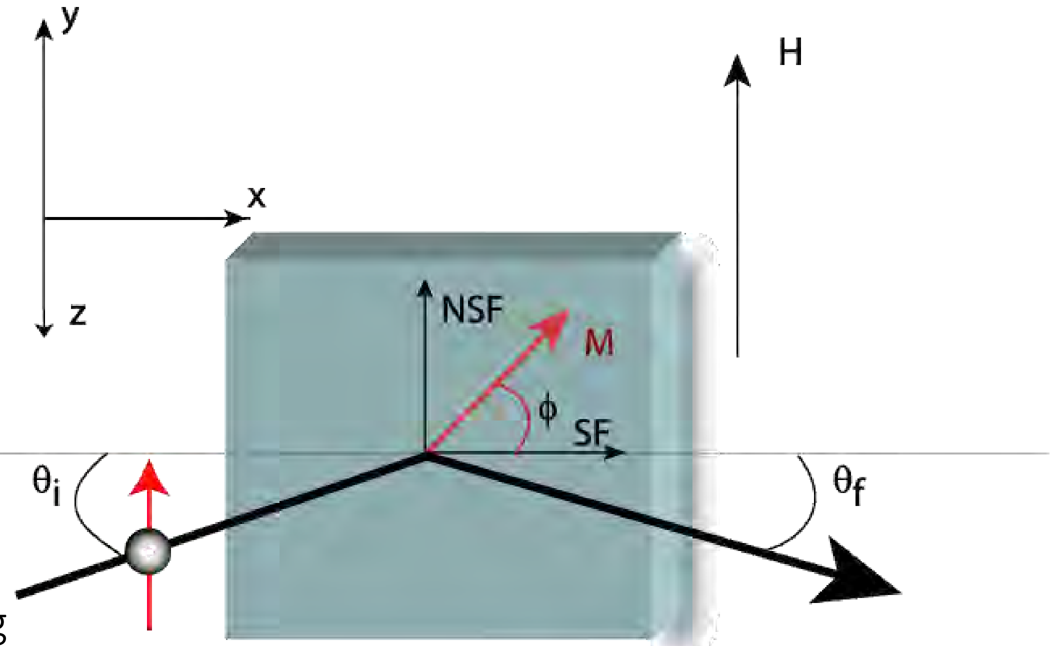
PNR from a multiple layers





Spin dependent cross-section

- In-plane orientation of magnetisation obtainable from 4 spin dependent cross-sections



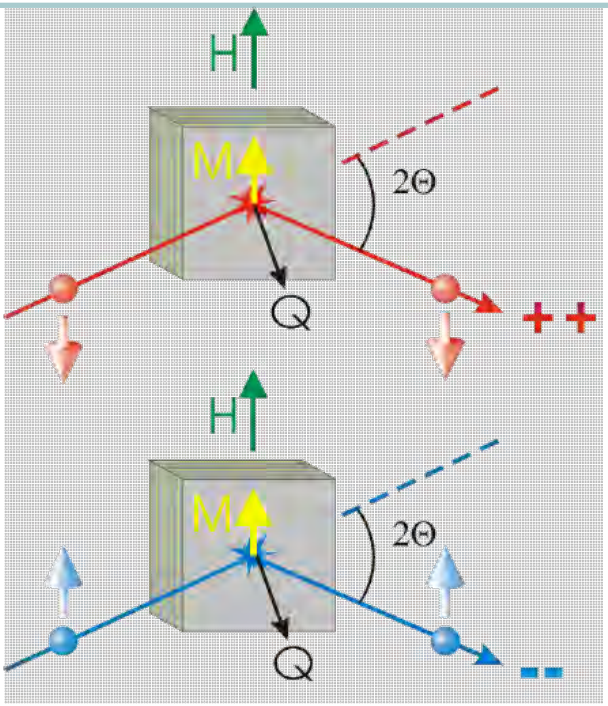
- Components of the magnetisation, m give rise to
- m || H: Non Spin Flip Scattering (NSF)
- m ⊥H: Spin Flip Scattering (SF)
- Dynamical analysis gives absolute depth dependence profile

$$b = b + p \sin \phi$$

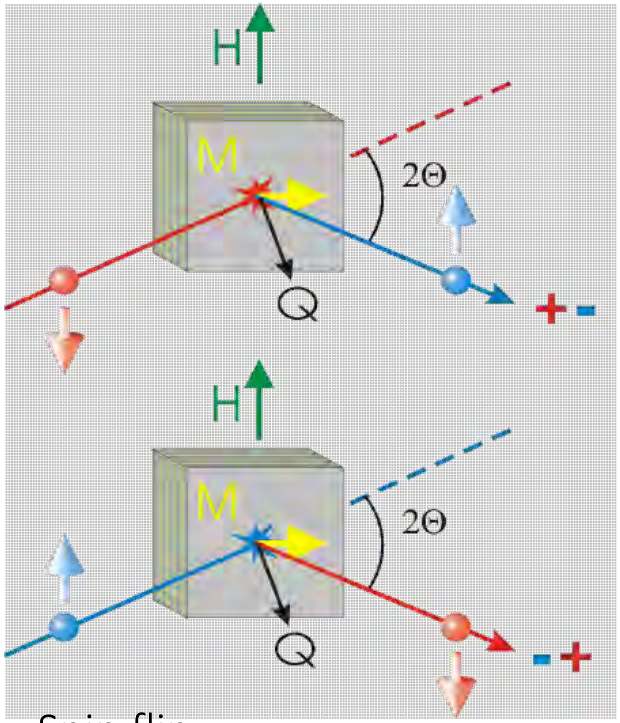
$$p_m \cos \phi = p_x$$

Polarisation

Polarised neutron reflection



Non spin flip
++ measures b + M_z
- - measures b - M_z



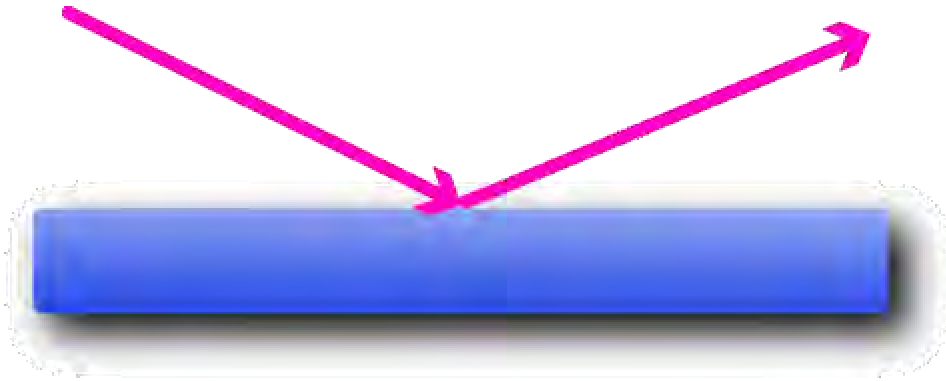
Spin flip
+ - measures M_x + iM_y
- + measures M_x - iM_y

By fitting all components the direction and strength of the magnetic moment can be measured as a function of depth

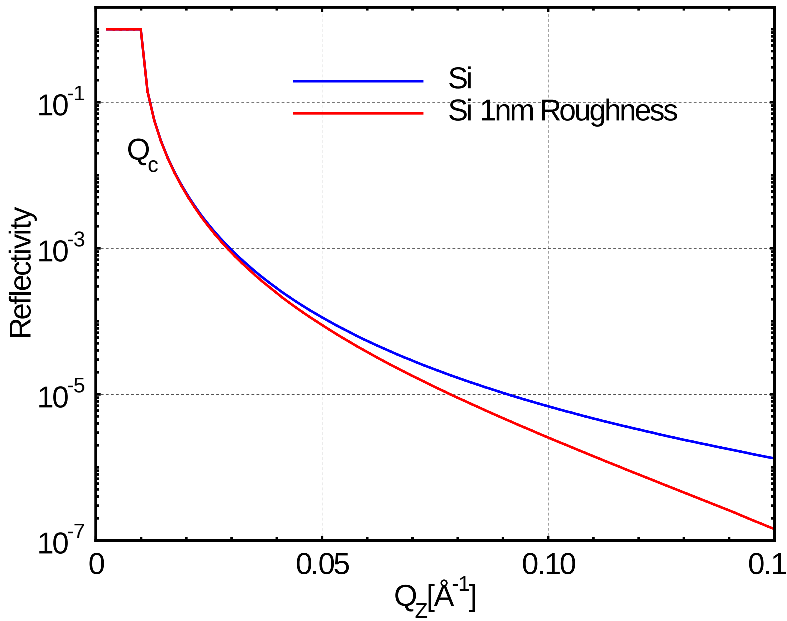
Roughness

Structural and magnetic interfacial phenomena

Structural Roughness



$$R(Q) = R_F \exp\left(-Q^2\sigma^2\right)$$



Simulation Packages

Simulation Packages: neutron

http://www.reflectometry.net/reflect.htm#Analysis

escriptions of Neutron Reflection Data Analysis

Software and Programs, Theory and Principles of Analysis, Data Formats

Software

The following links provide information about computer programs used for the analysis of neutron reflectivity data. In some case copies of the

- A catalogue of neutron reflectivity fitting programs written by Adrian Rennie that also use the Fitfun data fitting package developed at tl
  http://www.reflectometry.net/refprog.htm
- Neutron Reflection Analysis DRYDOC and LPROF by Adrian Rennie
  http://www.reflectometry.net/fitprogs/drydoc.htm
- Programs for X-ray reflectometry analysis by Adrian Rennie
  http://www.reflectometry.net/xrefprogs.htm
- AFIT written by Paul Thirtle (3.1) is available from Oxford.
  http://physchem.ox.ac.uk/~rkt/afit.html
- Neutron reflectometry programs available from the NIST Center for Neutron Research.
  https://www.ncnr.nist.gov/programs/reflect/data_reduction/software/
- Interactive, Web-Based Calculator of Neutron and X-ray Reflectivity, provided by Brian Maranville at NIST Center for Neutron Researc
  https://www.ncnr.nist.gov/instruments/magik/calculators/reflectivity-calculator.html.
  There is also an extended version for magnetic samples and polarised neutrons:
  https://www.ncnr.nist.gov/instruments/magik/calculators/magnetic-reflectivity-calculator.html.
- Calculate neutron reflectivity on the Web. Alan Munter has provided a WWW site at the NIST Center for Neutron Research.
  https://www.ncnr.nist.gov/resources/reflcalc.html
- Description of Norm Berk's Programs and Papers
  https://ncnr.nist.gov/staff/nfb/nxnsr.html
- Description of data reduction for CRISP at ISIS
  CRISP data analysis
- SURFace - A Package for Analysis of Reflectivity Data J. R. P. Webster and S. Langridge, RAL
  http://www.isis.stfc.ac.uk/instruments/surf/data-analysis/surf-data-analysis2565.html

PolRef

- Wavelen
- Unpolari
- Polarizec

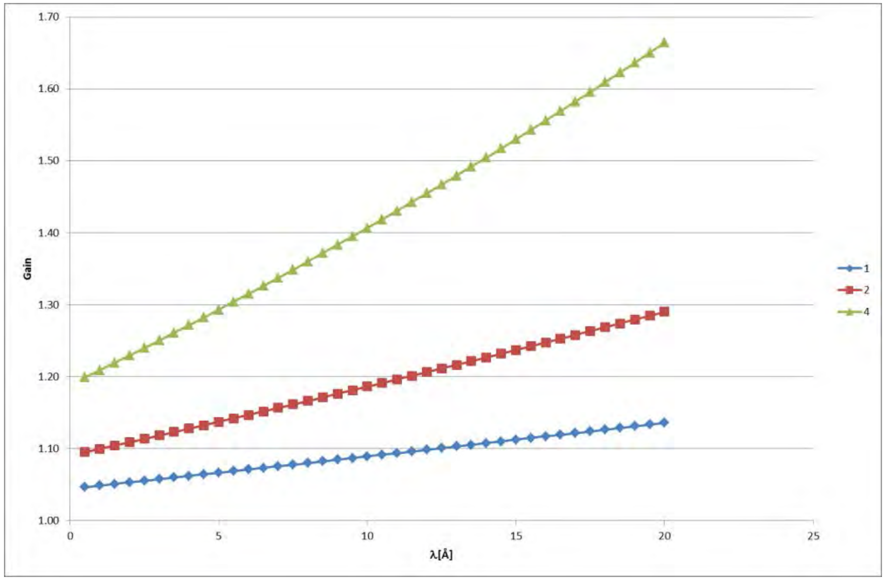
- Source t
- Beam in

- Well shie
- 640 channel linear gas detector with 0.5mm pixel

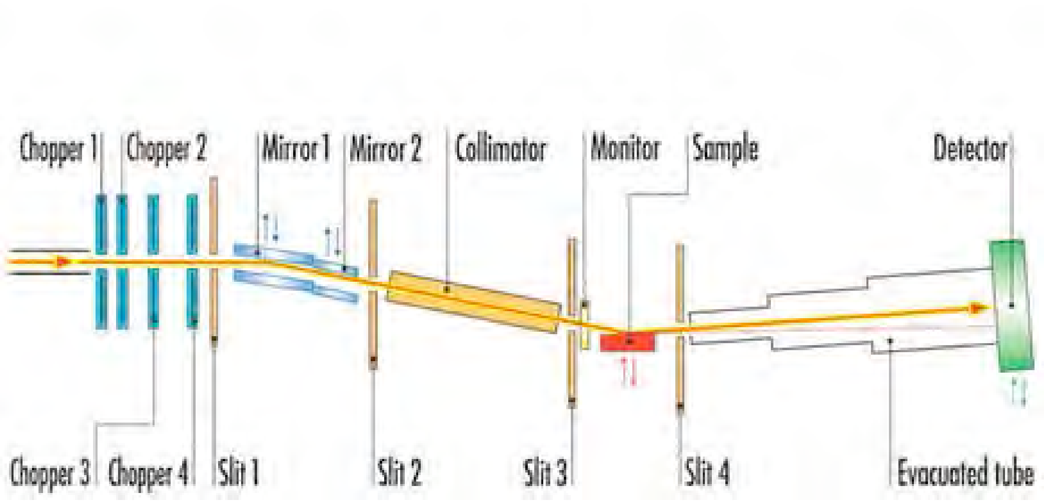
- Vertical 2θ 7.5°
- Horizontal 2θ 22°

- Moderator
  - Cryogenic moderator (relaxed time resolution)
- Guide System
  - Couple efficiently to the moderator
  - Efficiently transport neutrons to the sample/detector
  - Bender
    - Move from line of sight
    - Multi-channel (~6)
    - Vertical and horizontal guide coating
    - Alignment

- Vacuum
- Chopper system
  - T0
    - Only necessary for line of sight
    - Bandwidth chopper (control $\Delta\lambda$)
- Data Acquisition
- Polarisation
  - High degree of polarisation required
- Detector
  - High count rate 10^6 per pixel
  - Low gamma sensitivity
  - Magnetically insensitive
  - High efficiency
  - 2D spatial resolution <1mm
  - Large dynamic range



FIGARO – horizontal sample reflectometer at ILL



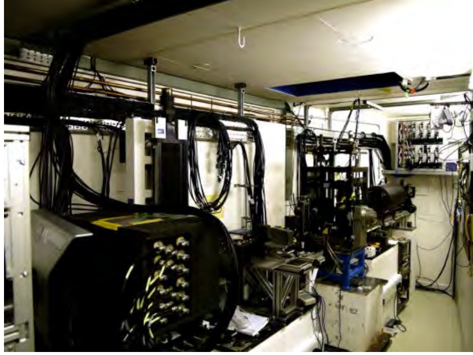
Q range=0.005 $Å^{-1}$ < Q < 0.4 $Å^{-1}$
Horizontal samples
Free liquids
Liquid/liquid interfaces
TOF

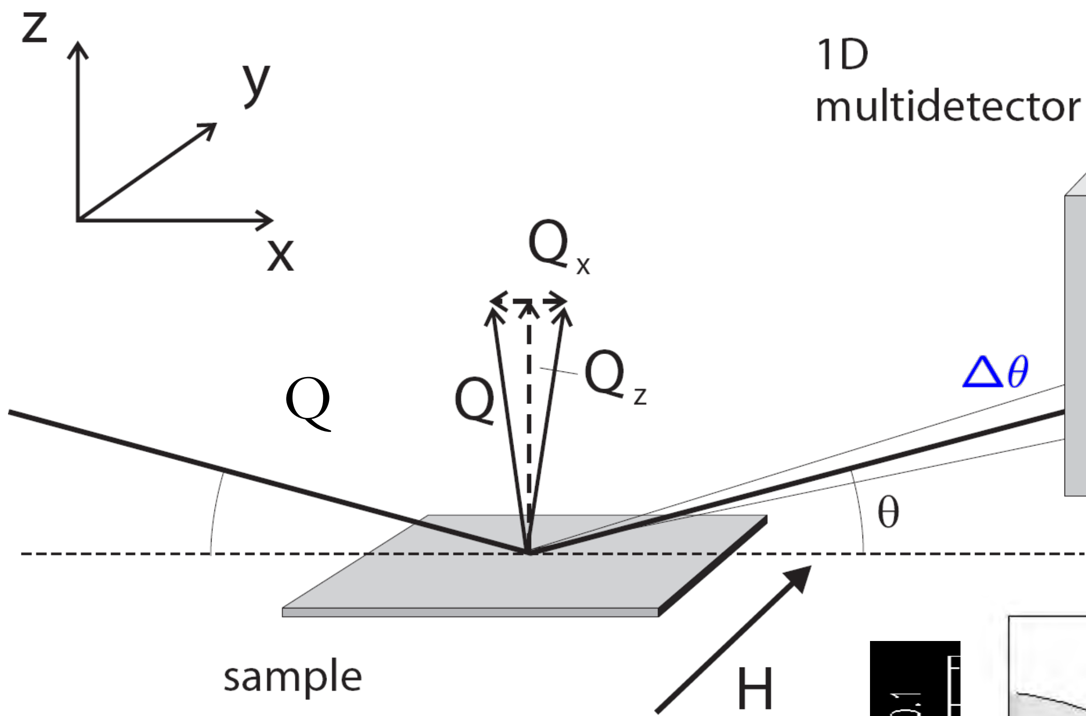
http://www.ill.eu/instruments-support/instruments-groups/instruments/figaro/

Off-specular scattering

Probing in-plane lengthscales

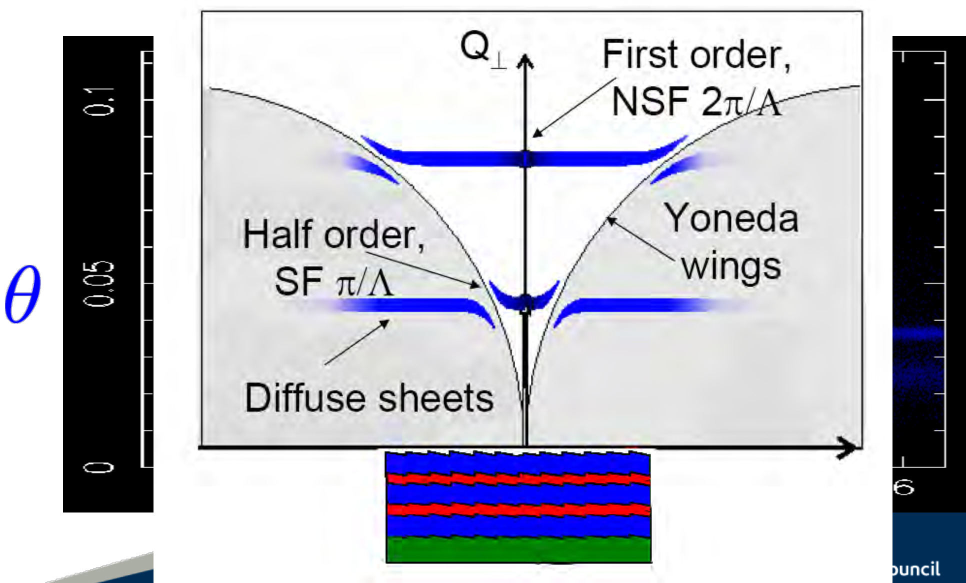
Experimental Geometry





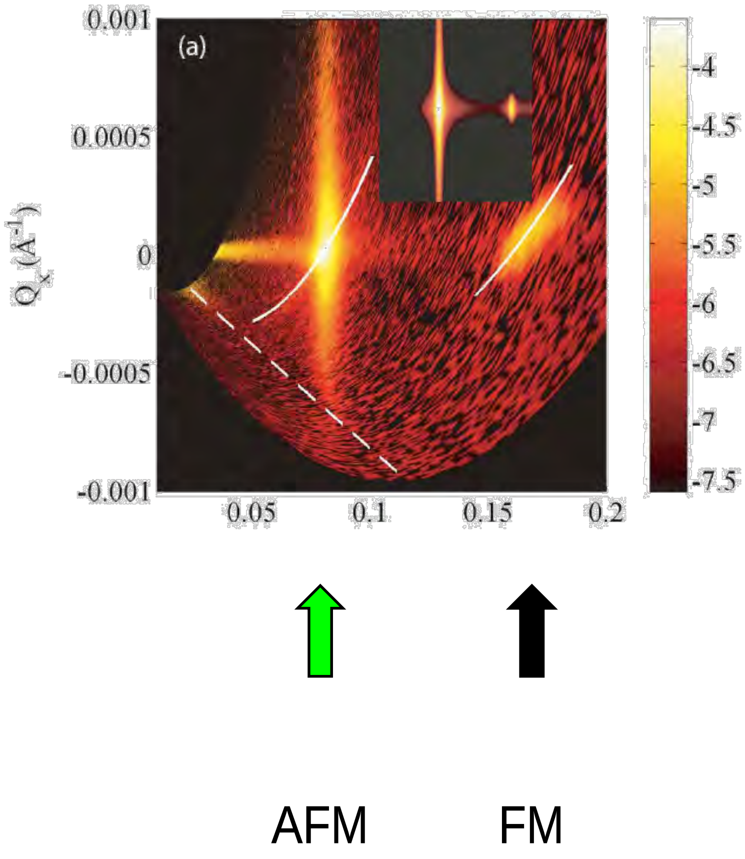
$$Q_x = |Q| \sin\theta \times \Delta\theta$$
$$Q_y = |Q| \times \Delta\phi$$

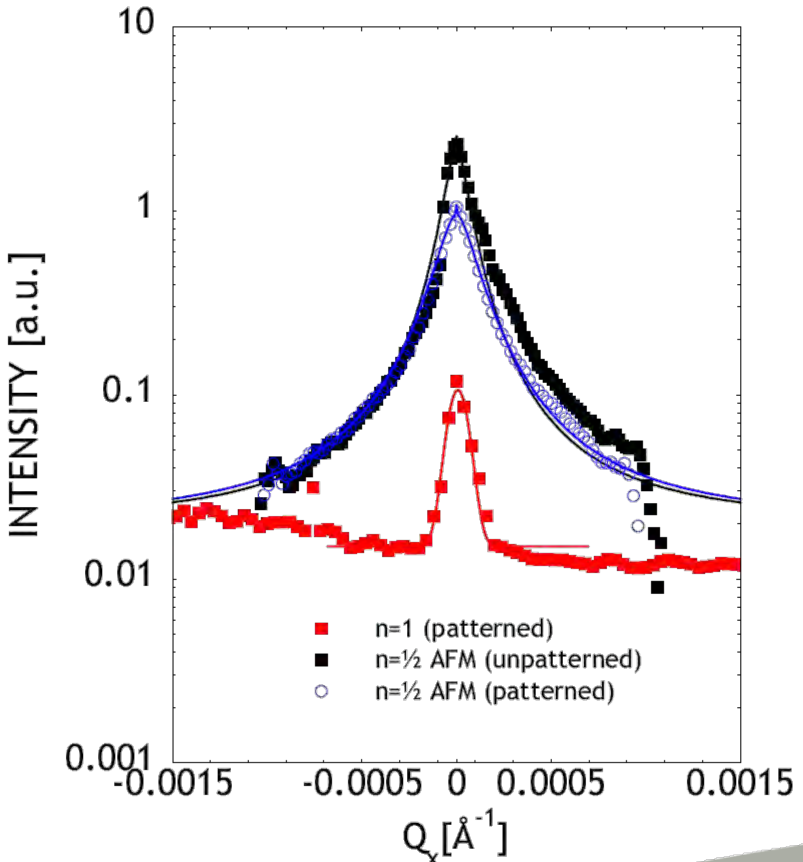
- λ range 0.5-6.5Å
- 1-d Detector
- Typical acquisition~2hours/field
- Measured coherence length ~30μm



- Coercive Field

$$\theta = \frac{Q_z}{2Q_i} - \frac{Q_x}{2Q_z}$$





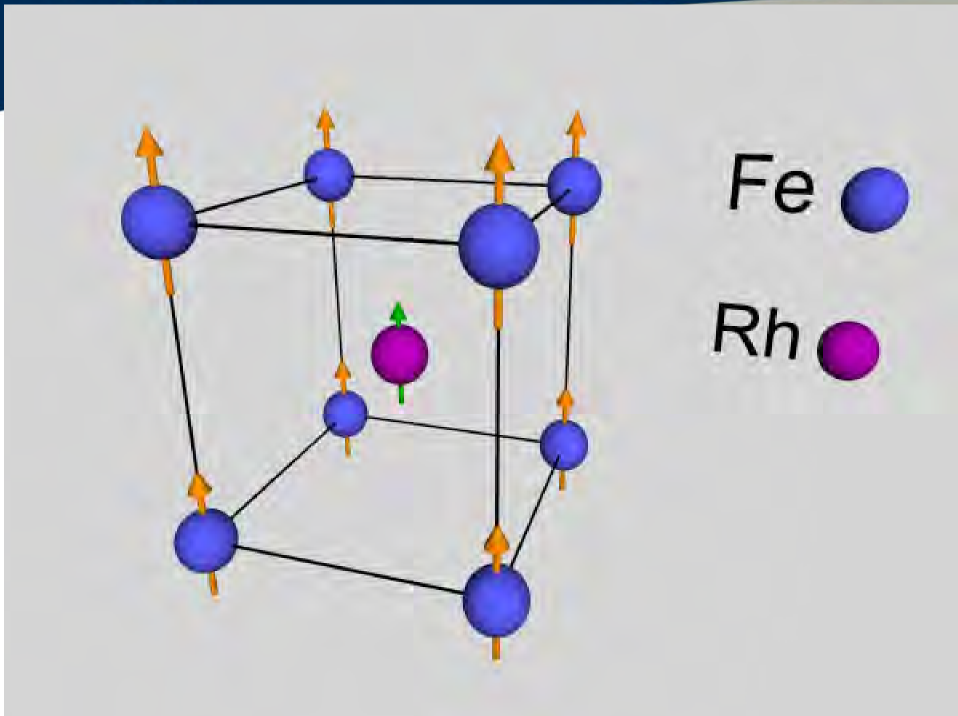
Examples of reflectivity

Science & Technology Facilities Council
ISIS

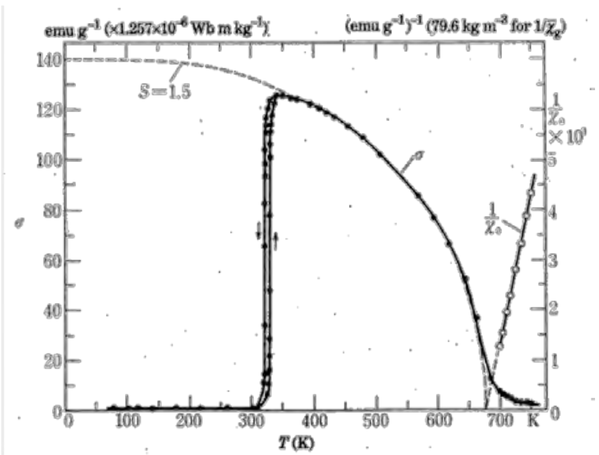
A Magnetic controllable interface

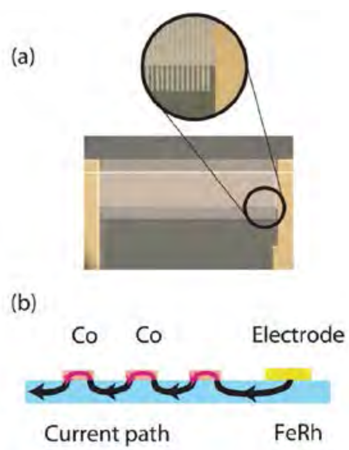
doped-FeRh

FeRh: Bulk Properties



- CsCl structure
  - a~2.99Å
  - α’ phase
- 300K: Type G AF
- Fe: ~3.3 μ_B
- Rh: no moment
- FM alignment within <111> planes
- AF alignment between <111> planes
- 350 K: AF → FM
- Fe: ~3.1 μ_B
- Rh: ~1 μ_B





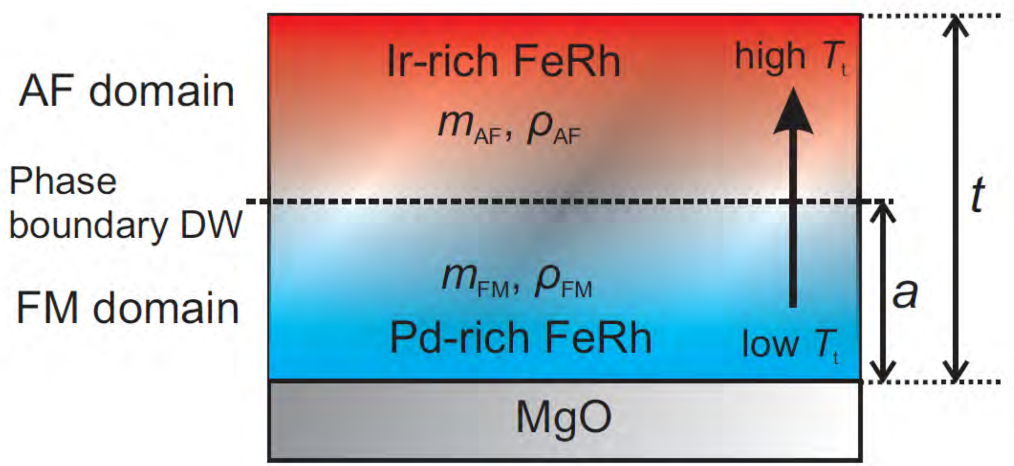
- J. S. Kouvel and C. C. Hartelius, JAP, **33**, 1343 (1962).
- G. Shirane *et al*. Phys. Rev. **134**, A1547 (1964)
- T. Naito et al., J Appl Phys **109**, 07C911 (2011)
- R. Fan, SL et al. Phys Rev B **82**, 184418 (2010).

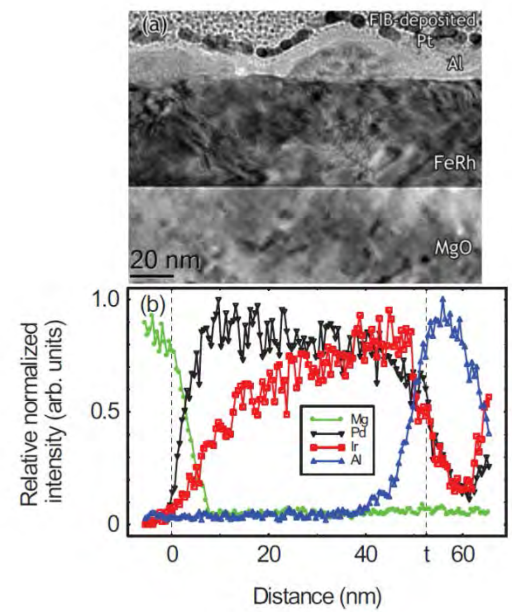
D. Arena, BNL

Motivation: Tuneable magnetostructural phase transition

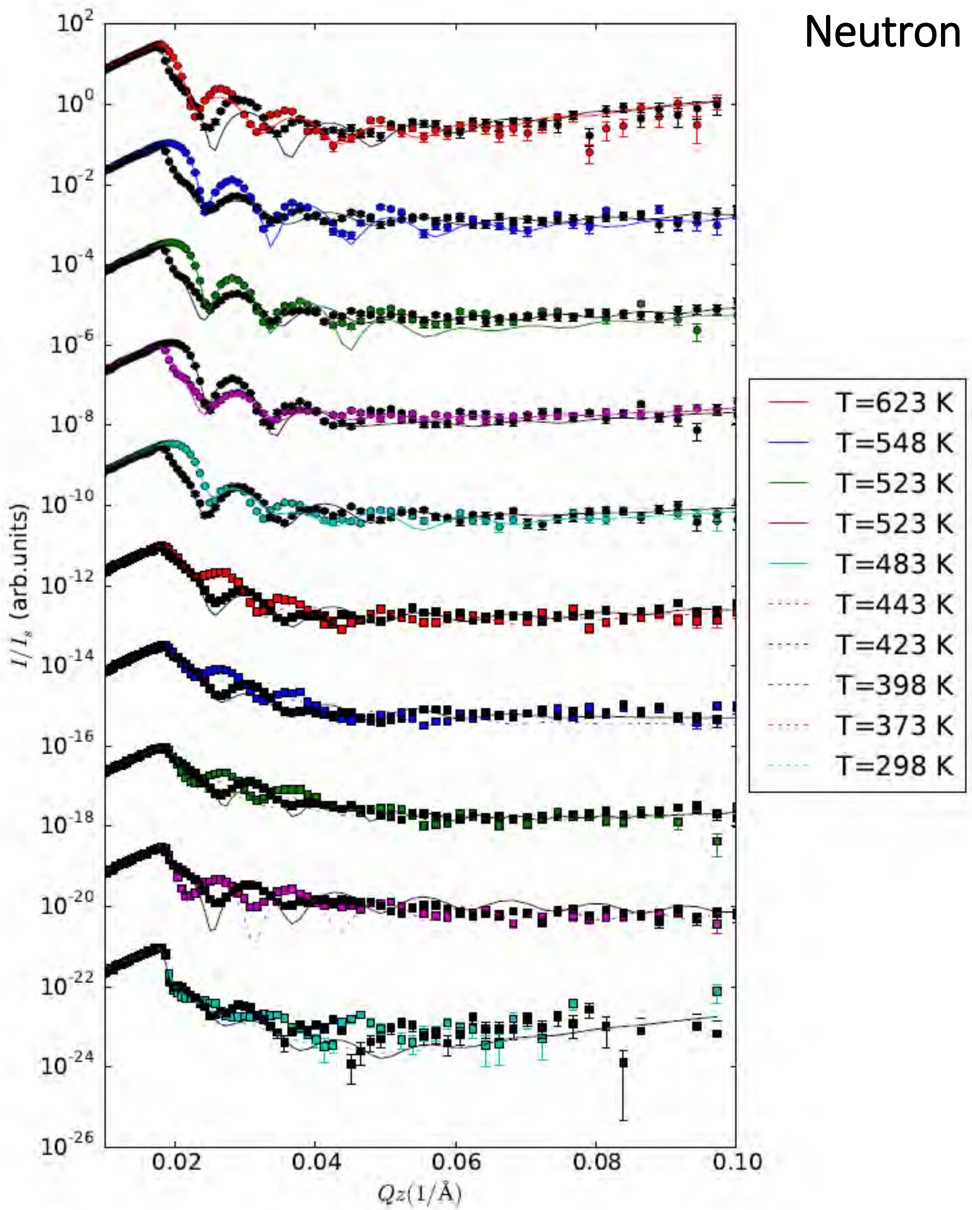
- Transition controllable by:
  - Temperature
  - Field (H)
  - *Doping – Pd, Pt (decrease T_c) or Ir, Ni (increase T_c)*
  - Strain - R. Fan, *et. al.* Phys Rev B 82, 184418 (2010)
  - Pressure – S. Yuasa *et. al.* Phys. Soc. Jap. 63, 3, 855-858(1994).

- Possible to create a controllable magnetic interface via a doping gradient.



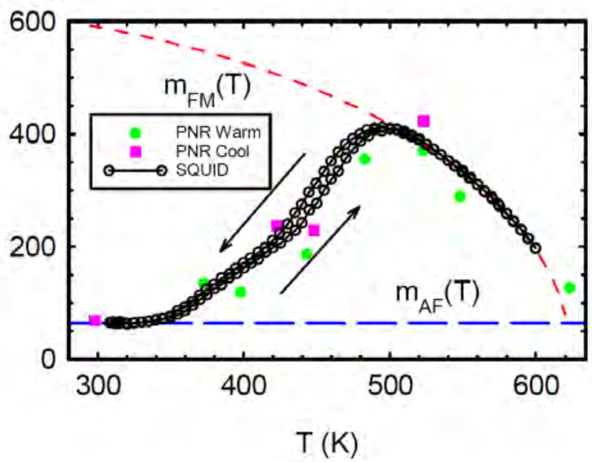


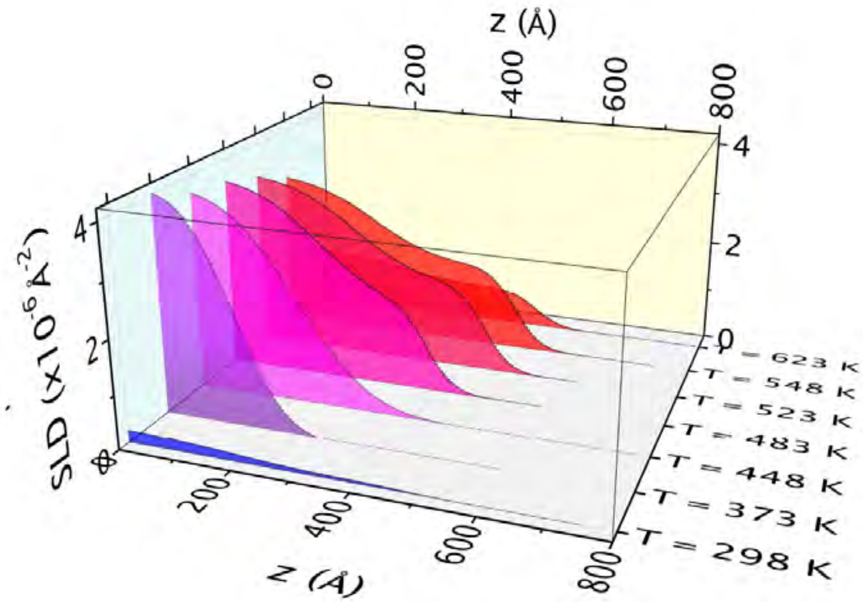
- Basis for a field or temperature sensor.

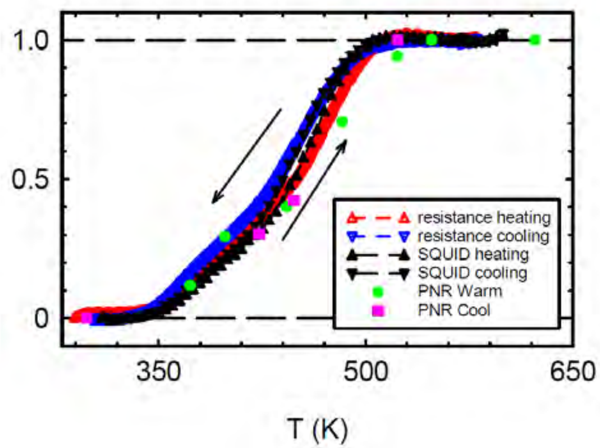


Neutron Measurements

- Saturating Field
- Constrained two layer model
  - The minimum to describe the data





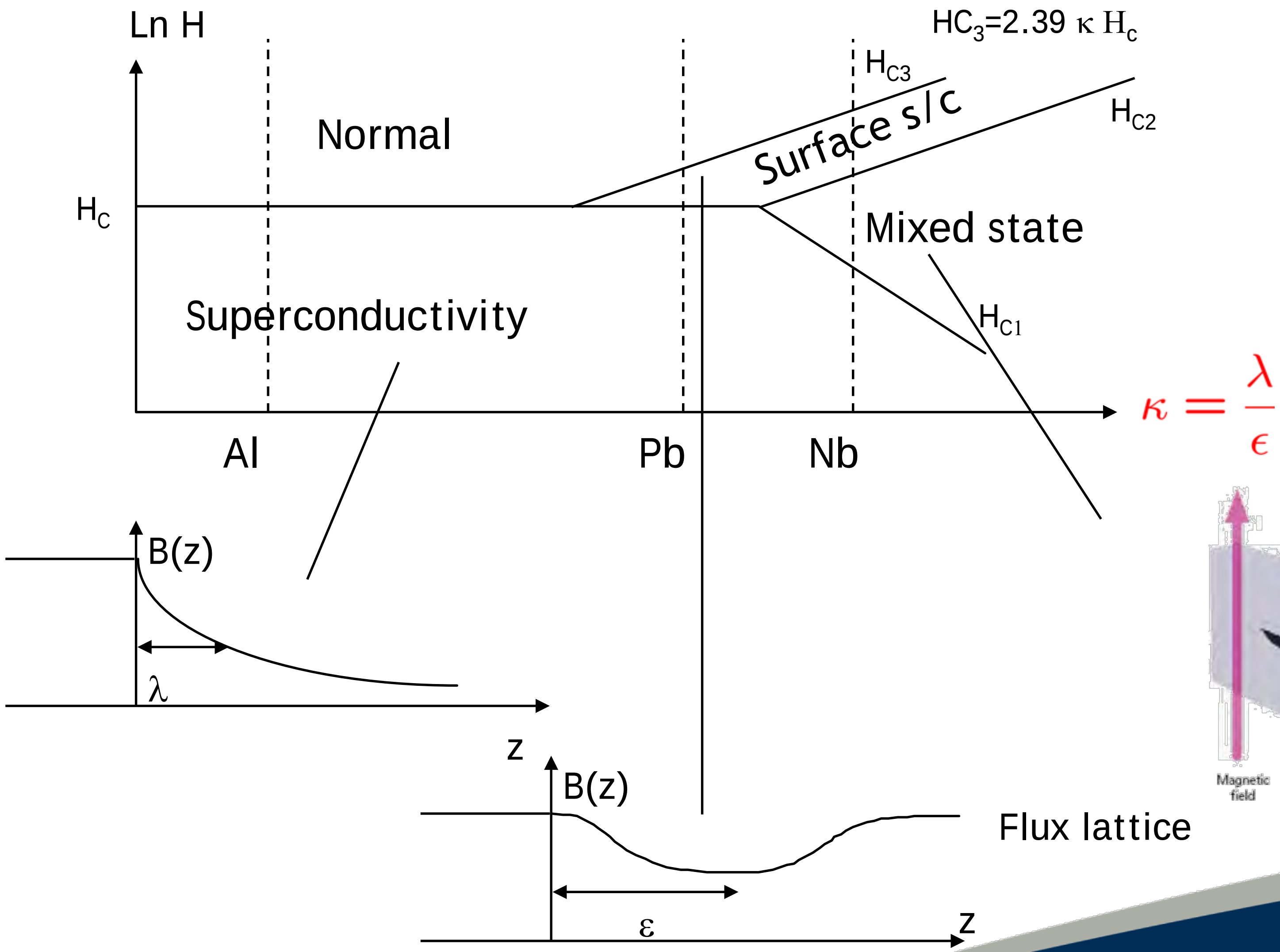


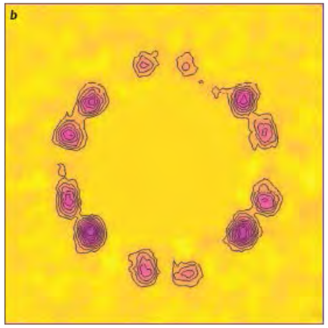
- Evidence of a controllable, mobile magnetic (electronic) domain wall
- Complex structure
- Neutrons essential to quantitatively resolving the complex magnetic structure

Superconductivity and reflectivity

Towards superconducting spintronics

Flux penetration in superconductors





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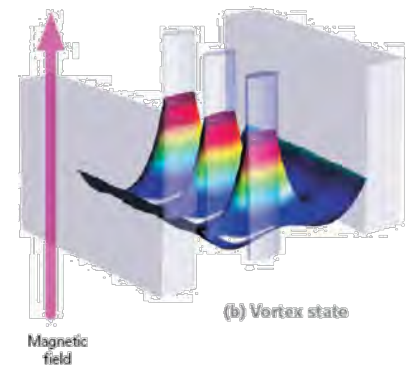


Flux penetration in superconductors

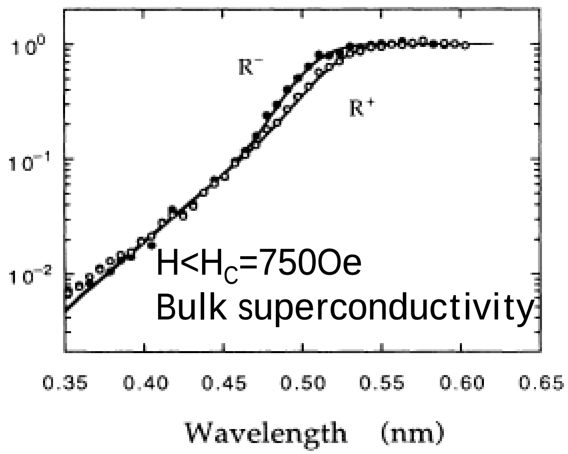
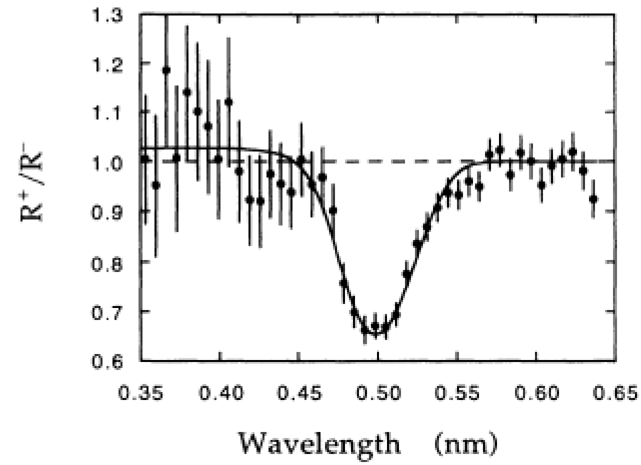


FIG. 2. The spin-dependent reflectivities R^+ and R^- measured in an applied magnetic field of 6.0×10^4 A/m (750 Oe). The continuous lines are the reflectivities calculated for the two polarization states, with the same instrumental and surface parameters as in Fig. 1, and an exponential decay of magnetic induction with a penetration depth of 39 nm.



- 1μm Pb film
- PbO surface layer
- $B(z)=\mu_0 H \exp(-z/\lambda)$
- λ=39±1nm
- No field dependence

PHYSICAL REVIEW B VOLUME 49, NUMBER 22 1 JUNE 1994-II

Magnetic-induction profile in a type-I superconductor by polarized-neutron reflectometry

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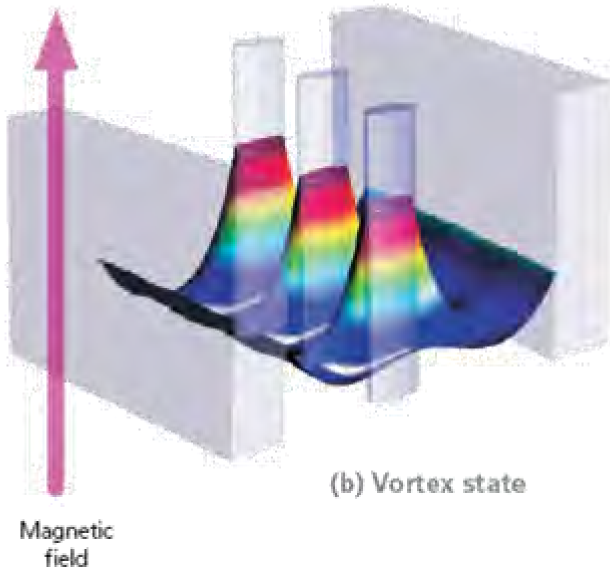
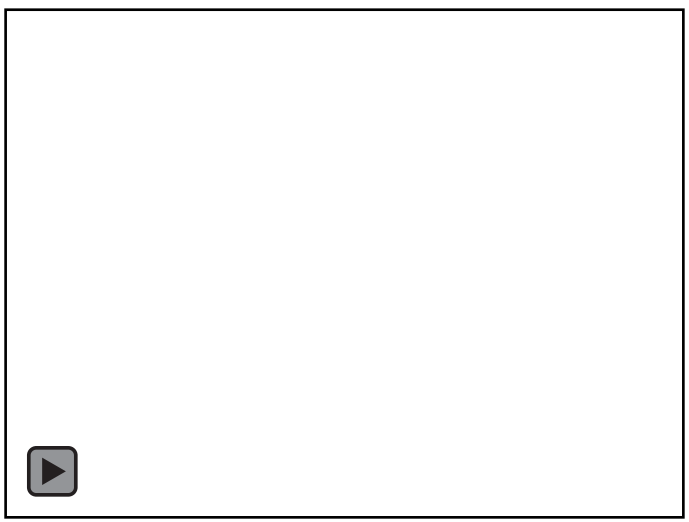
(Received 17 February 1994)

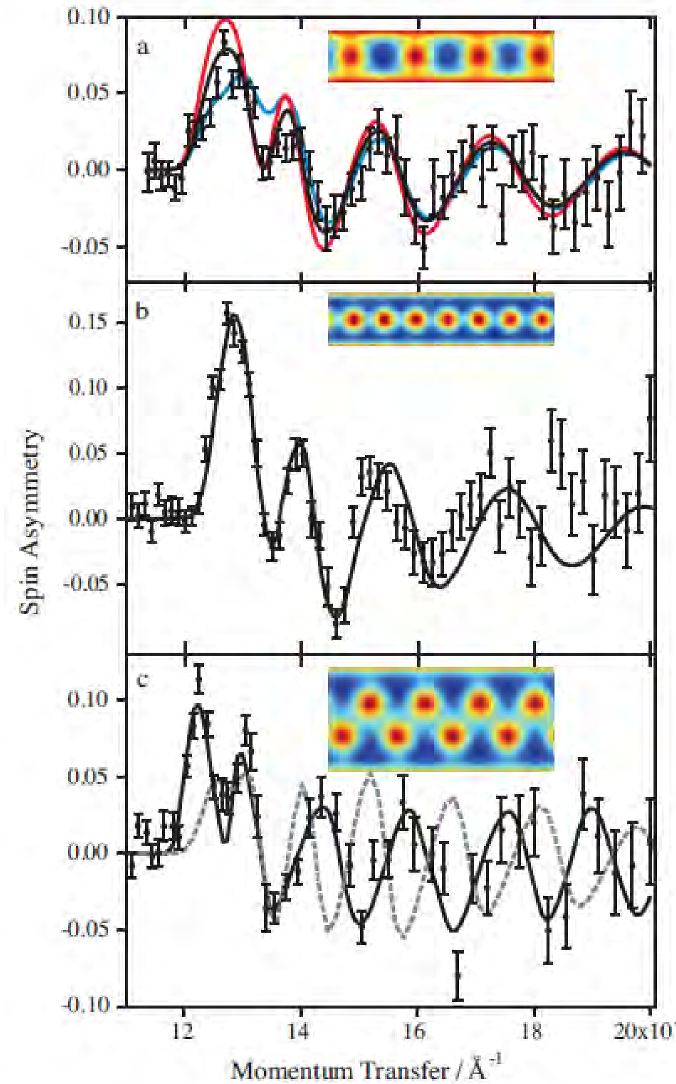
Visualising the Flux distribution





$$d = 195nm$$

$$d = 250nm$$



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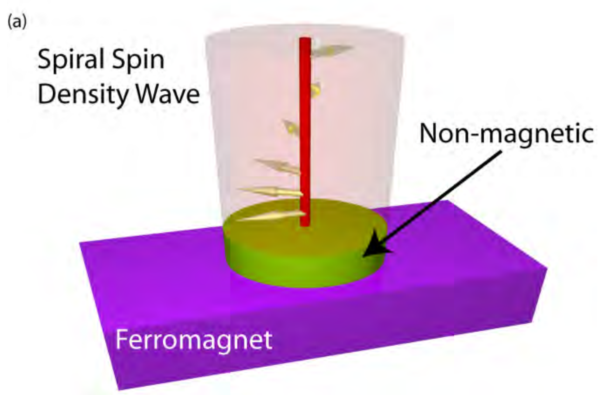
Motivation

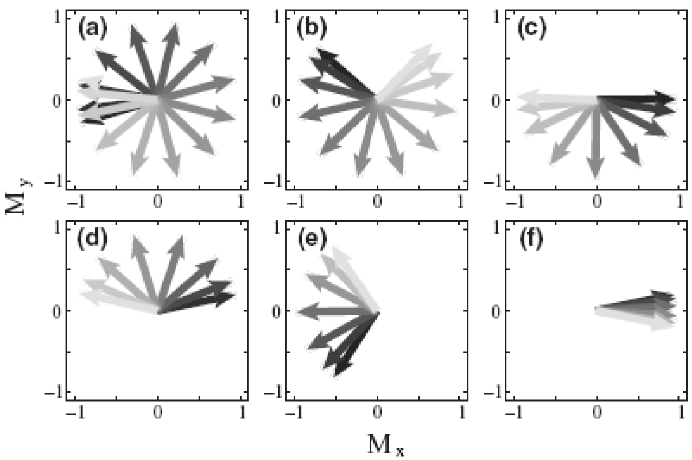
Bulk Spin Transfer Torque

- PRL **96**, 256601 (2006)
- PRB **79**, 104433 (2009)



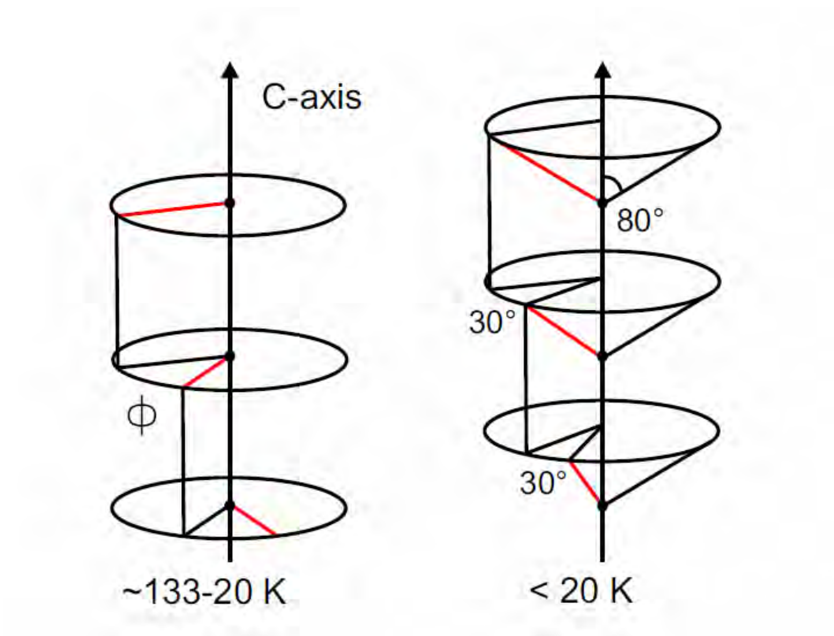
Exotic Phase

- PRB **78**, 020402(R) (2008)
- PRB **79**, 134420 (2009)

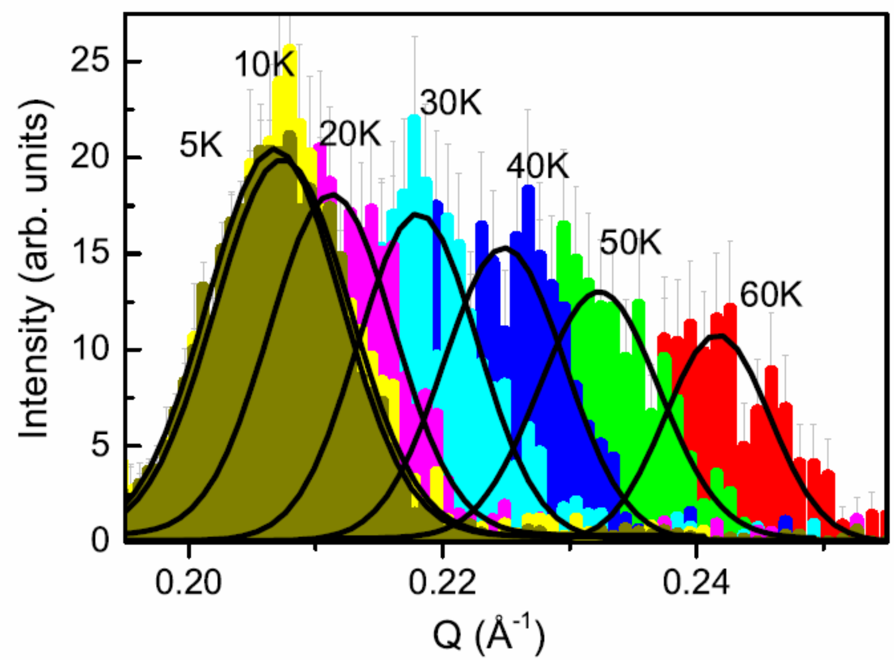


- Spin-triplet SC Science, **329**, 59 (2010)
- RMP **77** 1321 (2005)

Magnetic Spirals in Holmium thin films

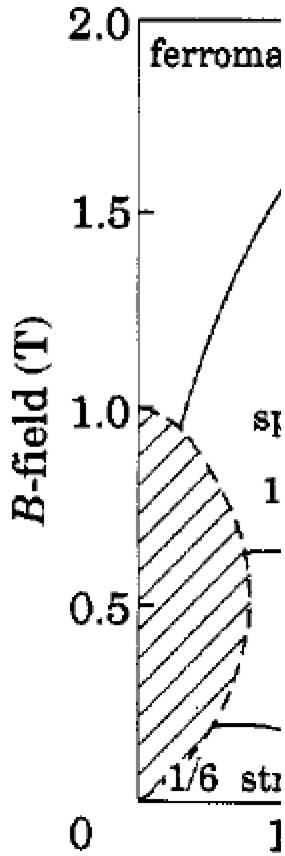


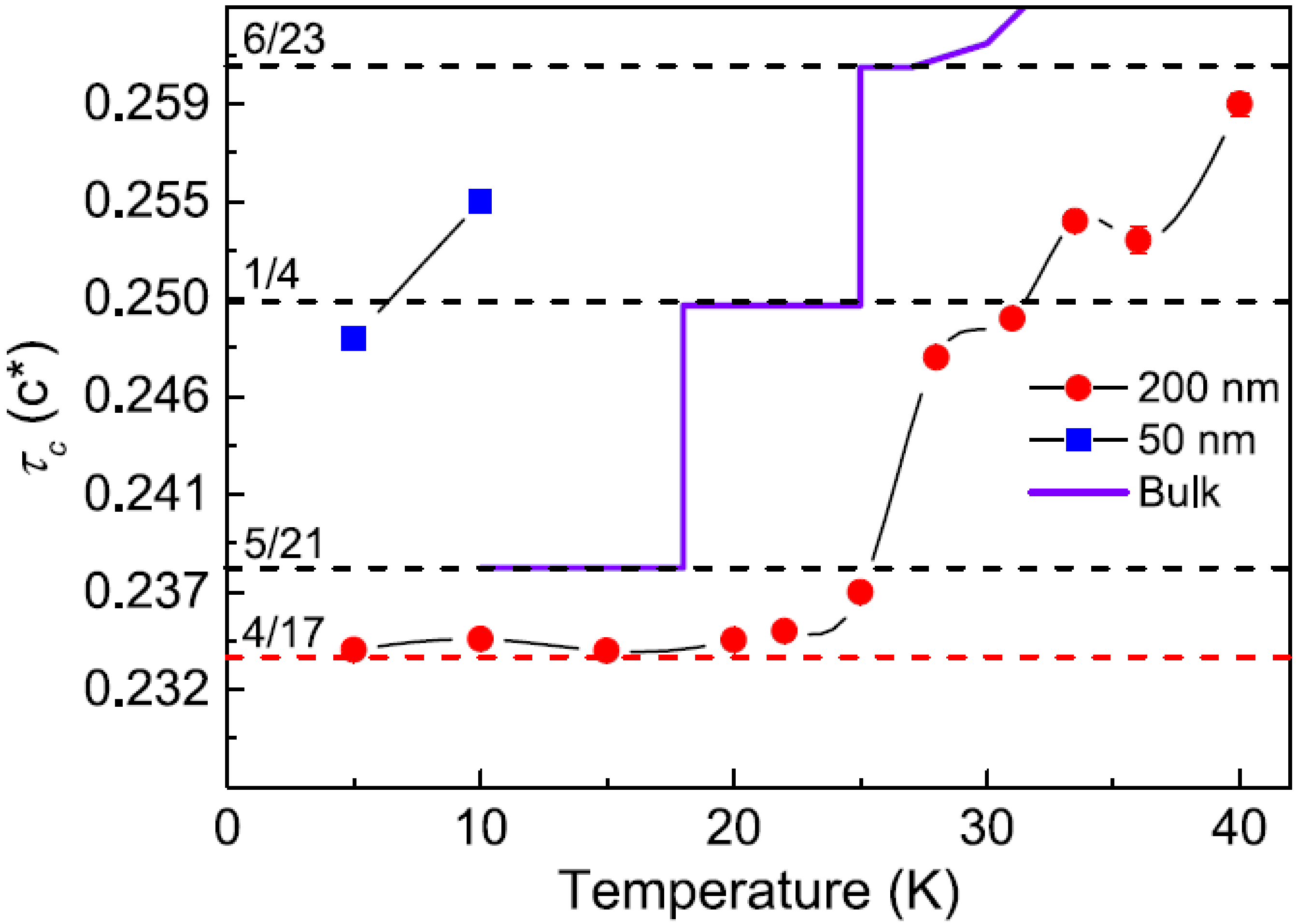
The representative magnetic structure of bulk Ho below its Néel temperature (left) and below its Curie temperature (right).



The momentum transfer versus the intensity of the magnetic diffraction peak for the 50 nm thick Ho sample as a function of temperature.

Bulk Ho Field Phase diagram





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Thin film Er

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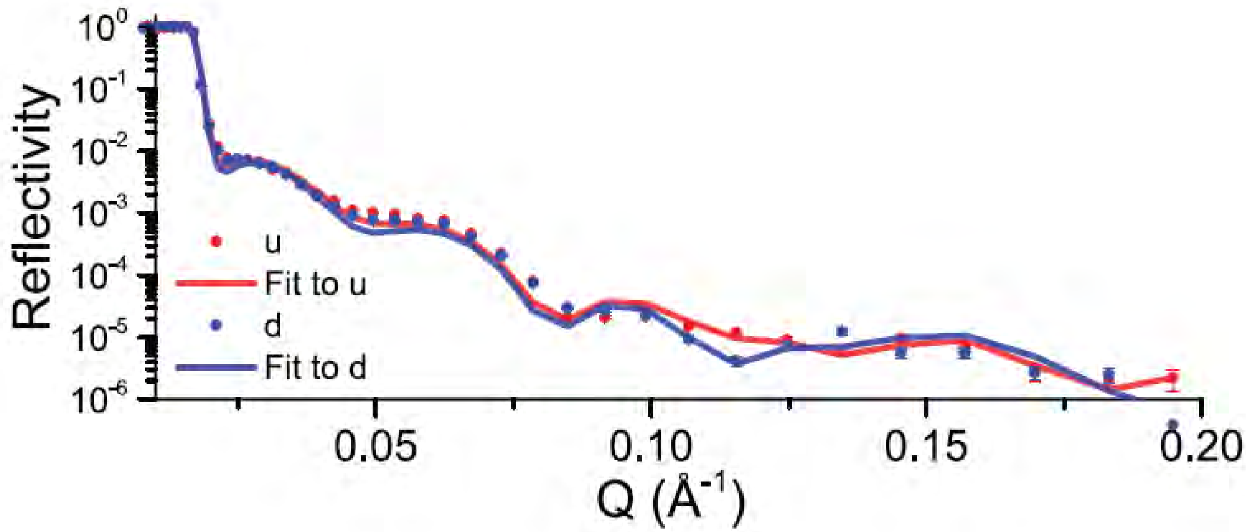
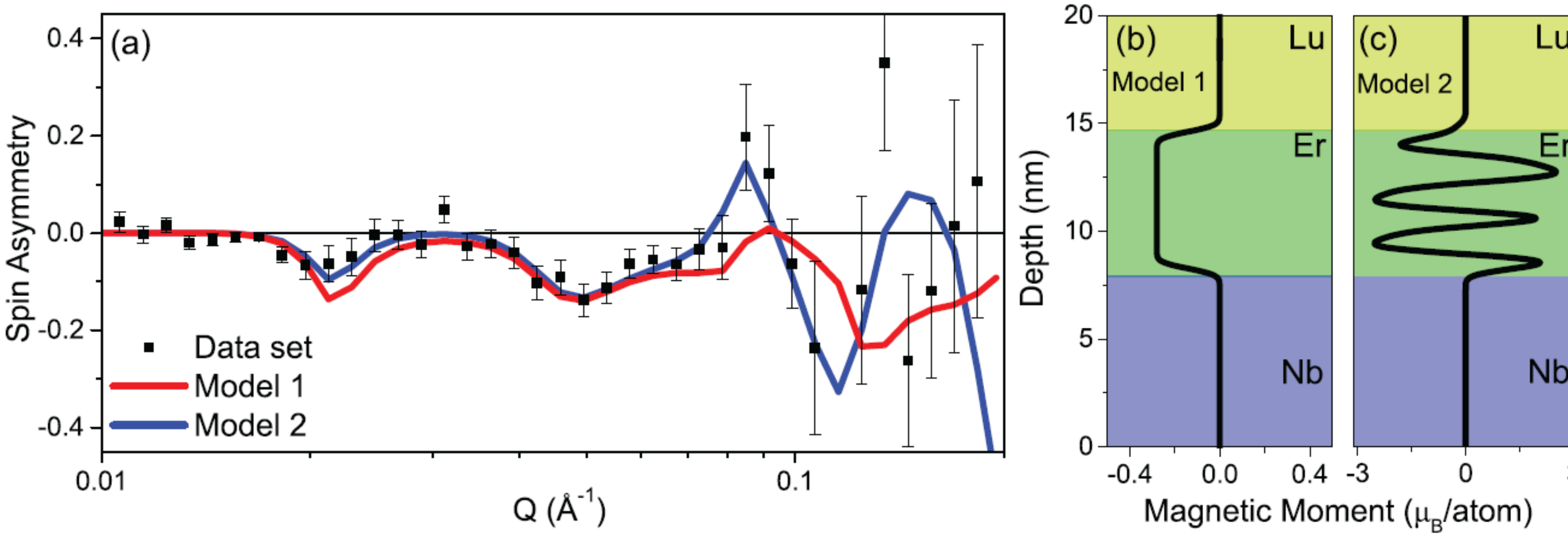
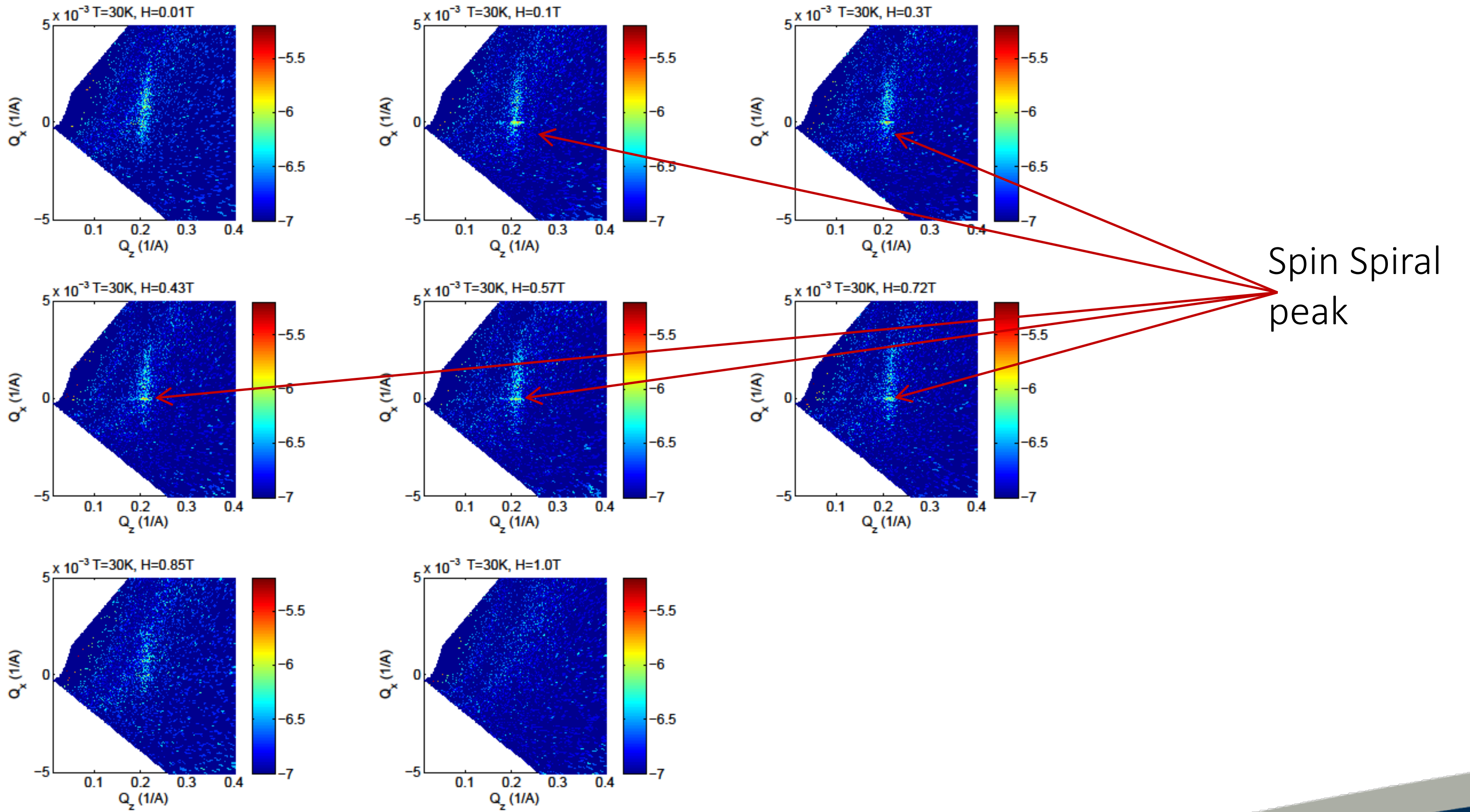


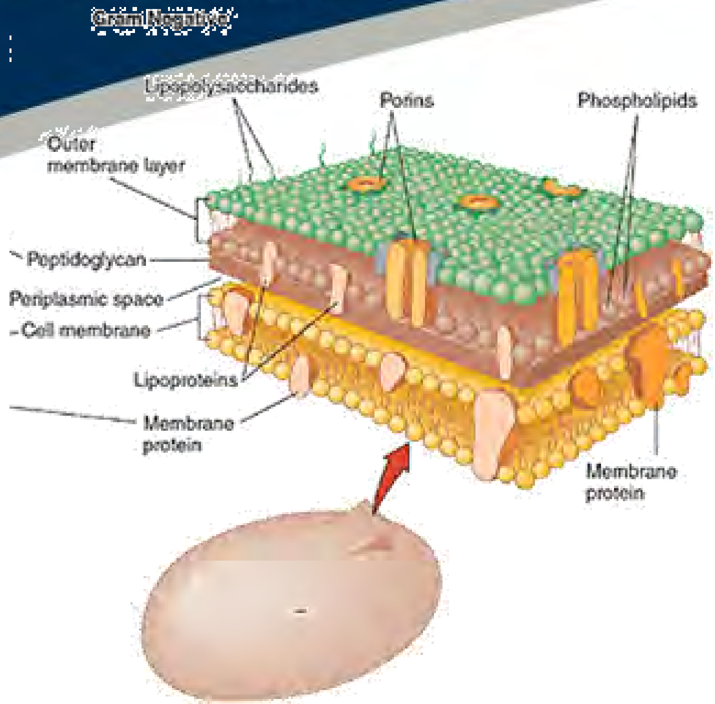


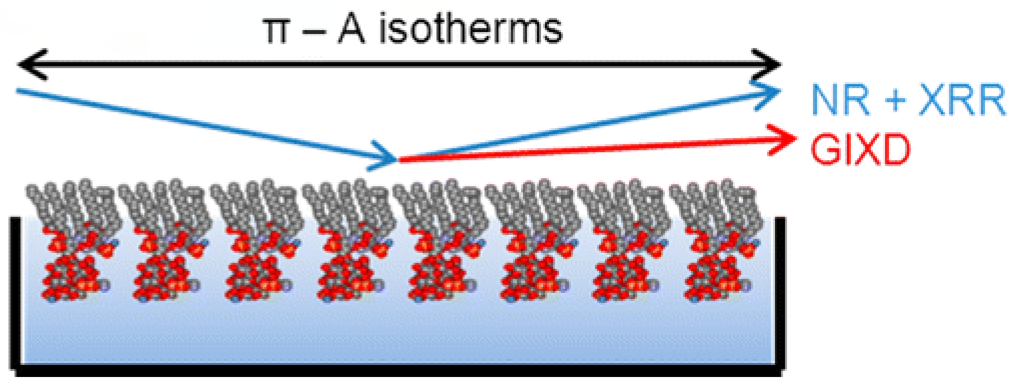
Polref: Diffuse scattering from a holmium spin spiral: 7.5T

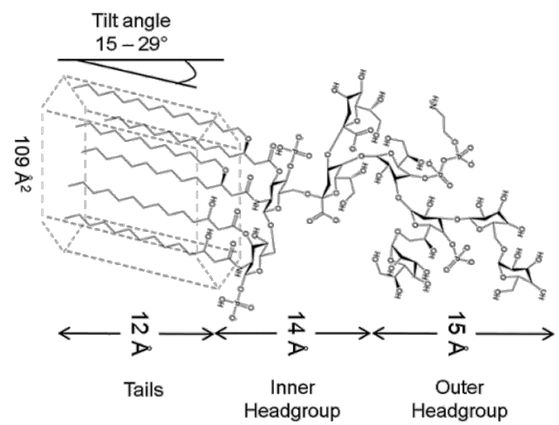


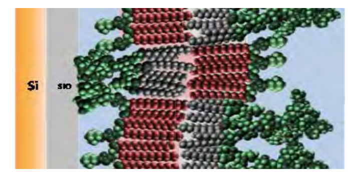
J. D. S. Witt et al. J Phys: Condens Matter **23**, 416006 (2011).

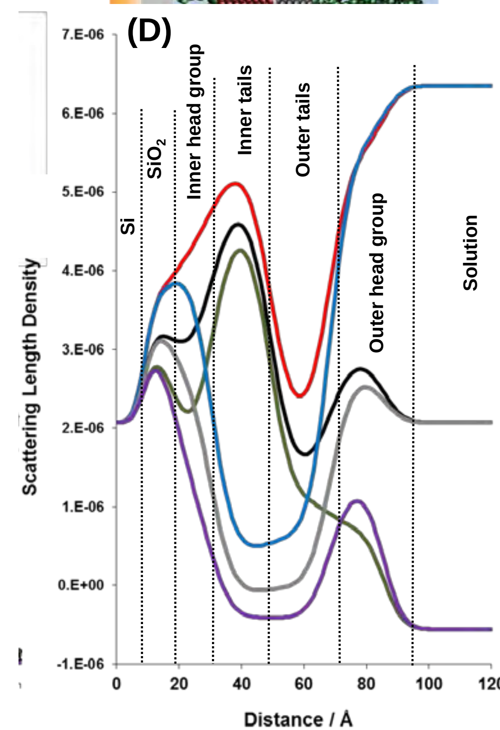
Building model gram negative bacterial membranes









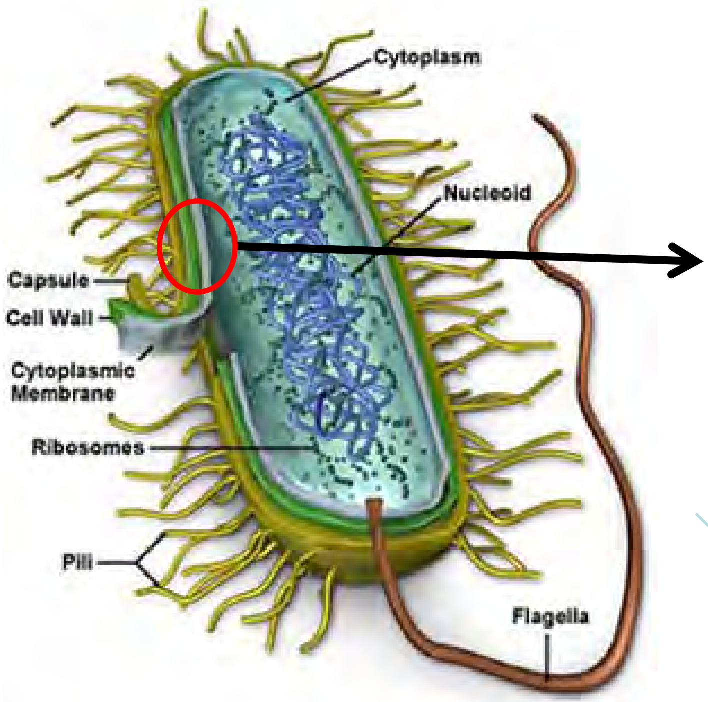


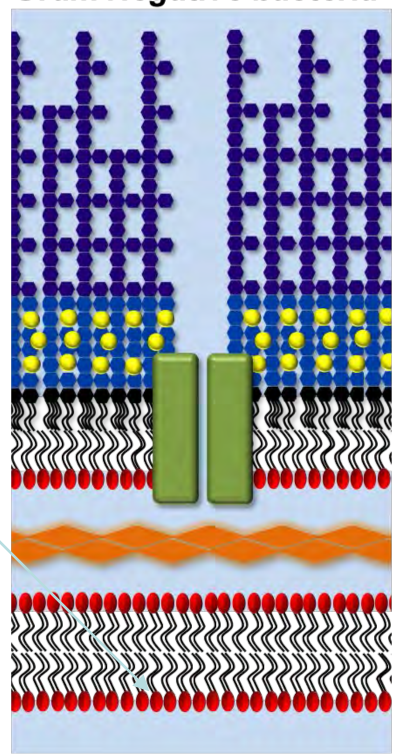
- Gram negative bacteria due to their increasing antibiotic resistance and their role in biotechnological processes.

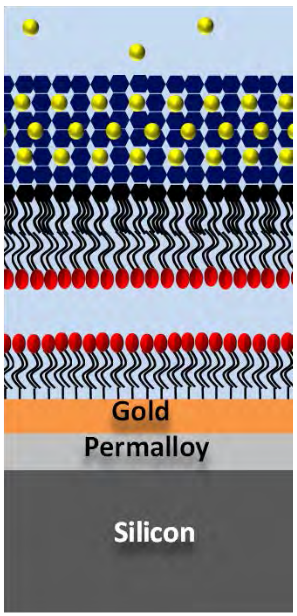
- The asymmetric membrane is the outer surface.

- Work has been on going to produce accurate models of the gram negative bacterial membrane using extracted bacterial lipopolysaccharides for future use in biophysical studies on membrane behaviour and interactions.

Realistic Models of the Gram Negative bacterial Outer Membrane: a test bed for new antibacterials

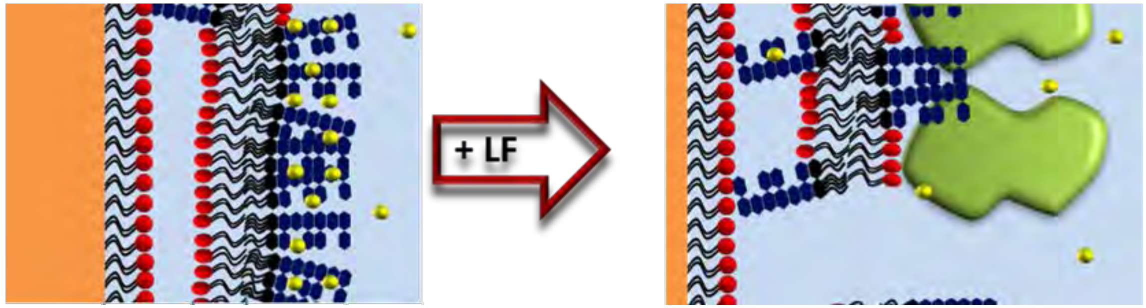






The detailed molecular structure of this dynamic complex layer can only be resolved by neutron reflectometry

Antibacterial protein disruption of the membrane



Angew. Chem. Int. Ed. DOI: 10.1002/anie.201504287

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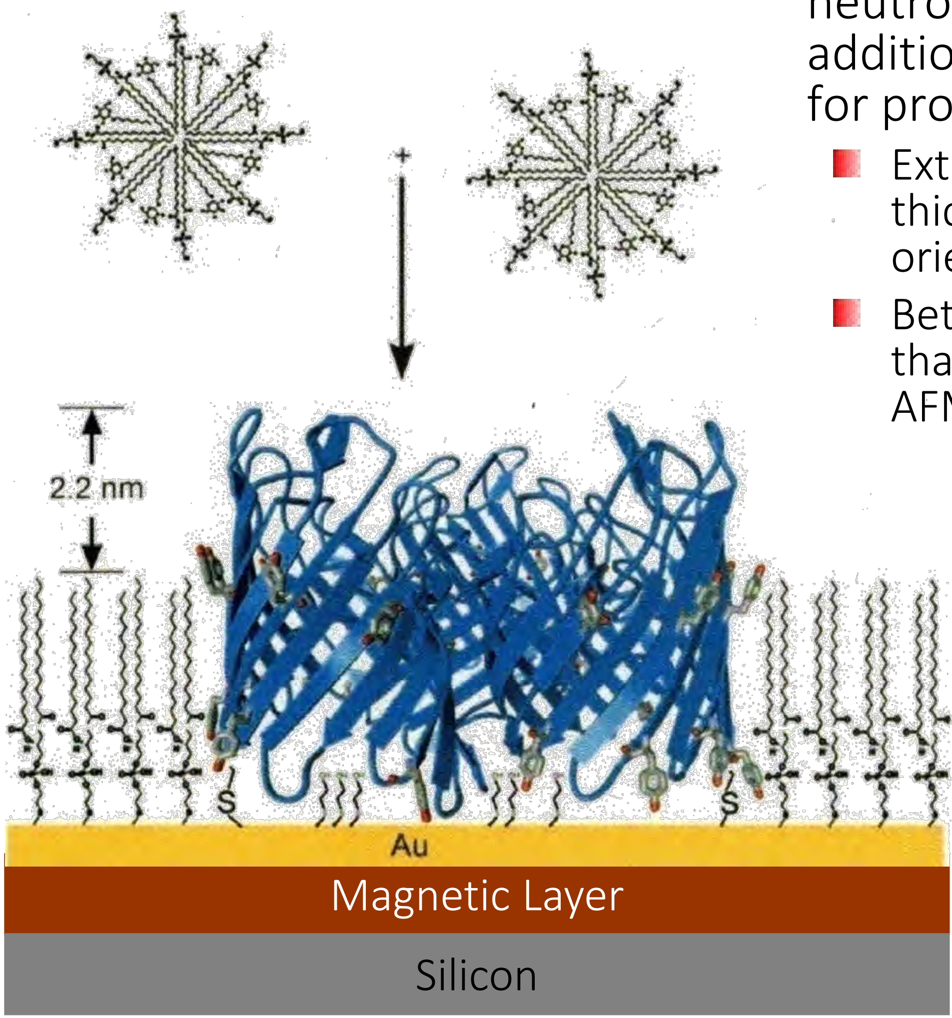
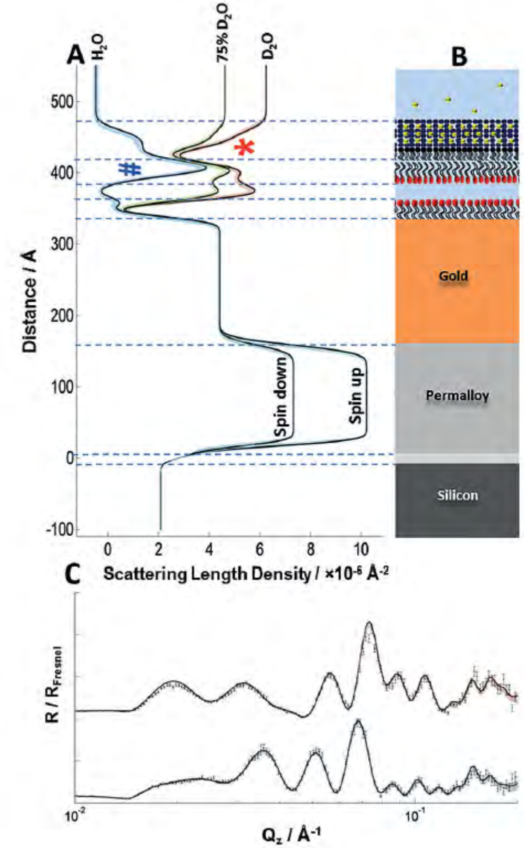
Polarised Neutrons for Biology



- Use polarised neutrons to provide additional informatio for protein absorptio
  - Extract protein thickness and orientation
  - Better resolution than conventional AFM studies



Seeing hydrogen

Nanoscale Storage

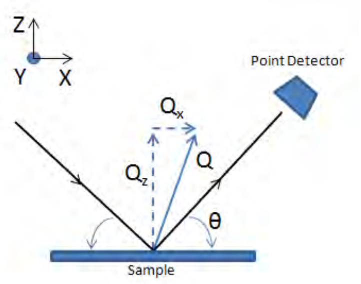
Mg/Ti thin film Hydrogen loading

- These films have gravimetric hydrogen storage capacities up to 6.5 wt% and fast and reversible kinetics of hydrogen absorption and desorption.

- Multilayer thin films of Mg and Ti offer a geometrically well-defined system for the study of the hydrogen absorption properties of these metals.

- Neutron reflectometry (NR) is an ideally suited method for tracking composition changes in thin film samples as well as the changes in the thin film dimensions. Owing to the large negative coherent neutron scattering length of hydrogen ($b_H = -3.74$ fm, compare to $b_{Mg} = 5.38$ fm and $b_{Ti} = -3.44$ fm).

- With this in mind a low pressure hydrogen cell was developed for the CRISP reflectometer.



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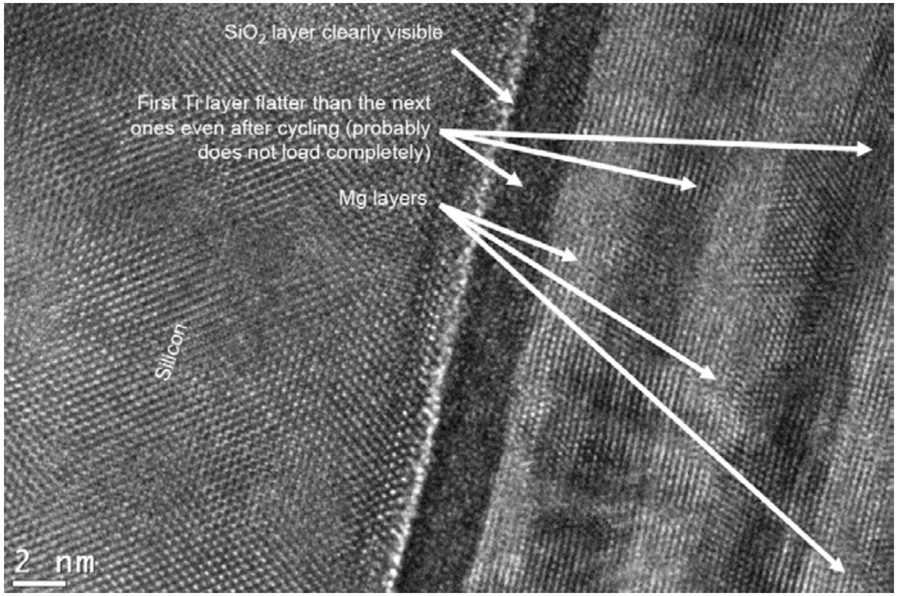
- Christian Kinane, Maximilian Skoda,Raymond Fan, Sean Langridge and William I. F. David

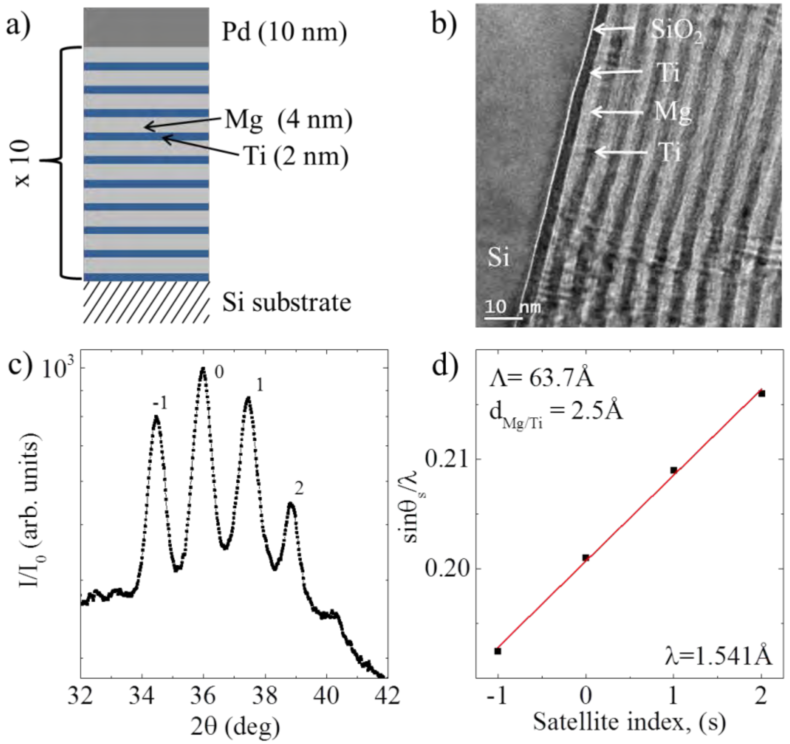
- ISIS Facility, Rutherford Appleton Laboratory, Chilton, Oxfordshire, OX11 0QX, U.K.

- Philippe C. Aeberhard

- Department of Chemistry, ICL, University of Oxford, South Parks Road, Oxford OX1 3QR, U.K.

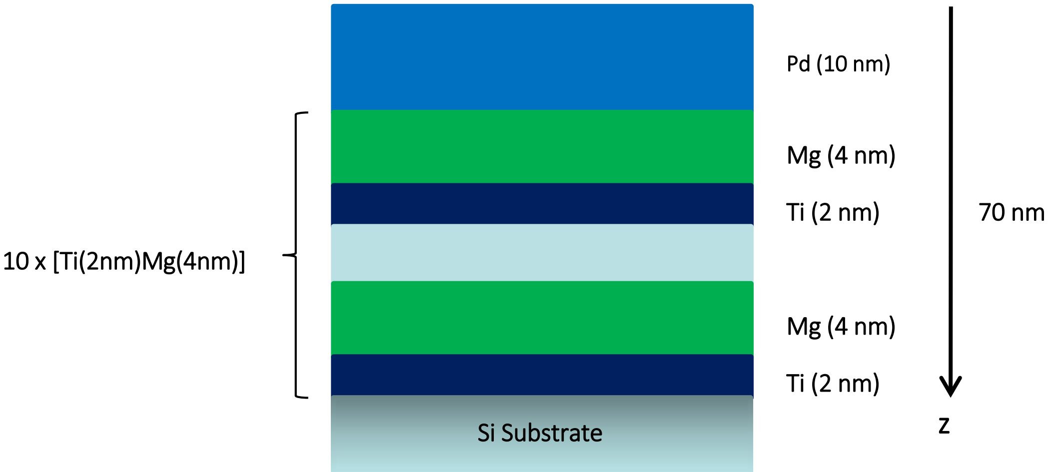
Science & Technology Facilities Council ISIS

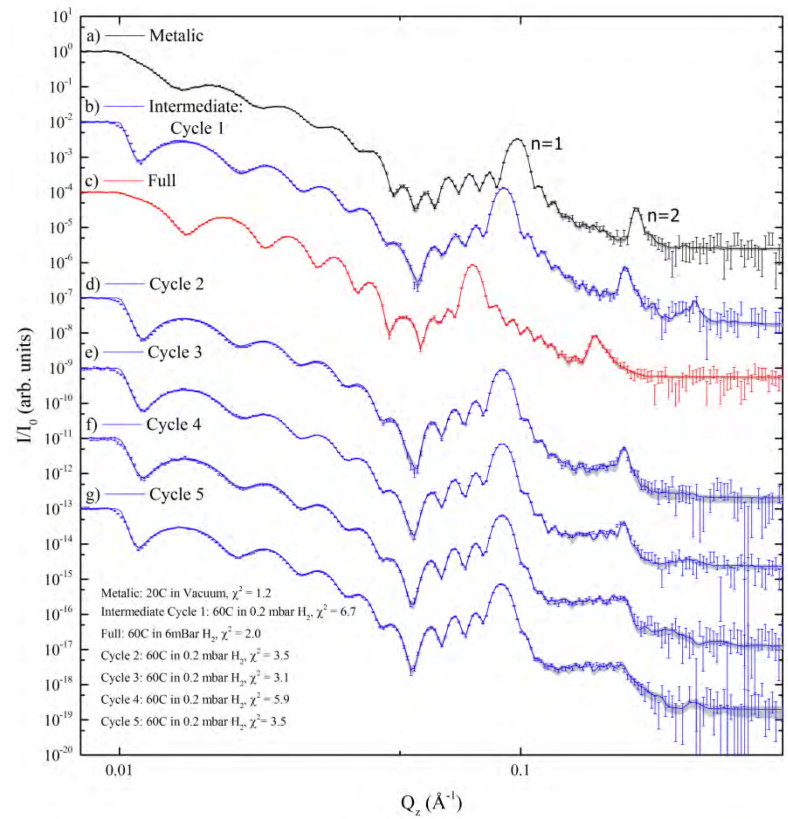




- Multilayer thin films of Mg and Ti offer a geometrically well-defined system for the study of the hydrogen absorption properties
- Neutron reflectometry (NR) is an ideally suited method for tracking composition changes in thin film samples as well as the changes in the thin film dimensions. Owing to the large negative coherent neutron scattering length of hydrogen (b_H = −3.74 fm, compare to b_{Mg} = 5.38 fm and b_{Ti} = −3.44 fm).

Sample Structure

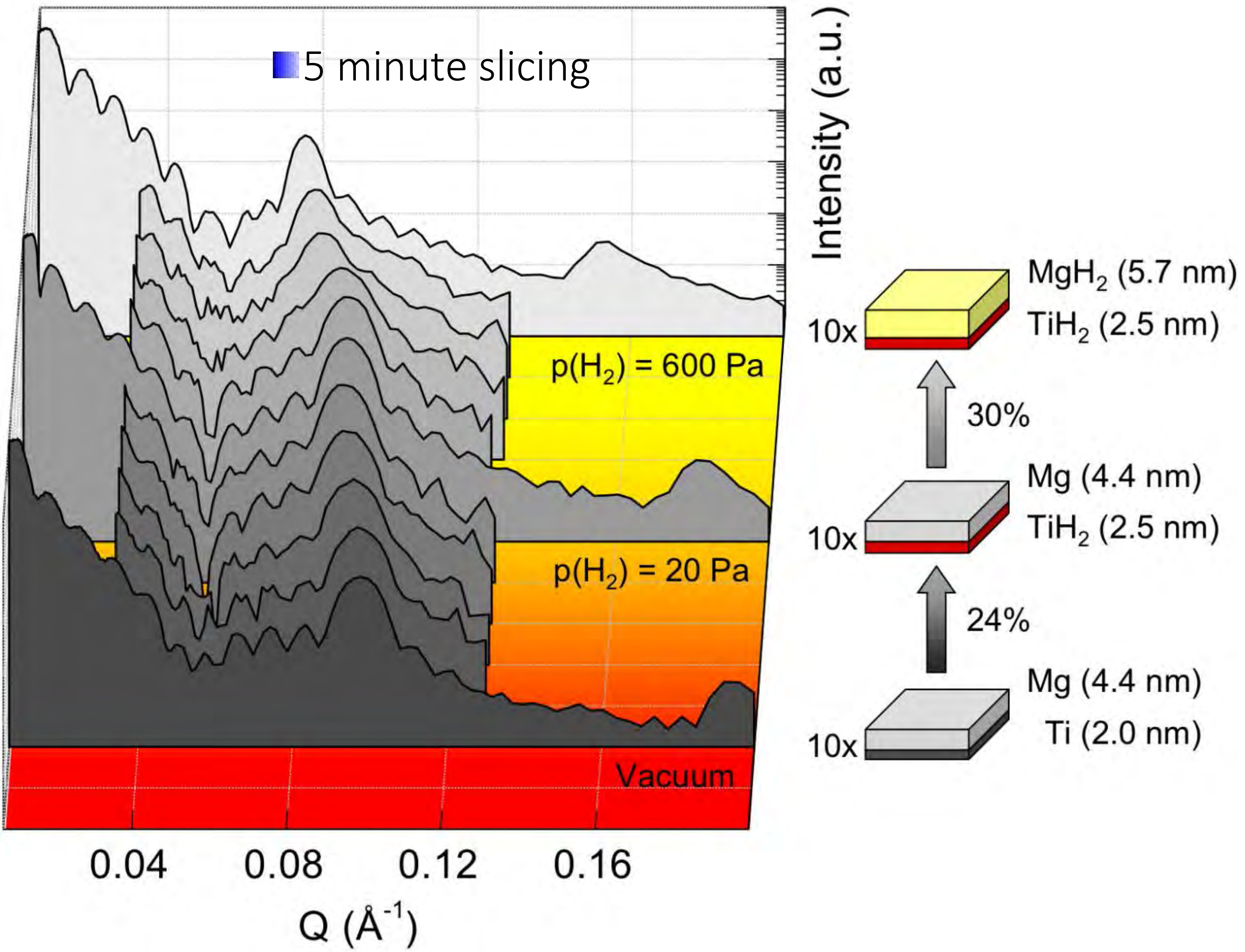




- Mg/Ti Multilayer Grown on is substrate by DC Magnetron sputtering.

- Clear shifting due to 30% lattice expansion between the Hydrogen loaded and unloaded states.

Hydrogen loading in Mg/Ti



Neutron reflectometry patterns measured during hydrogenation of the 10x{Ti/Mg} multilayer at 333K, in vacuum and after exposure to 20 and 600 Pa of H_2.

Summary

- Can take advantage of (*i.e.* control) the refractive index (polarised neutrons, deuteration, isotopic substitution)
- Can extract magnetic structures
- Realistic sample environments
  - Time resolution
- Sub nm resolution for structural systems
- Lengthscales (out of plane) monolayer to ~100nm
- New opportunities in pinhole and refractive optics

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