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


@isisneutronmuon

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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!



## Specular Scattering

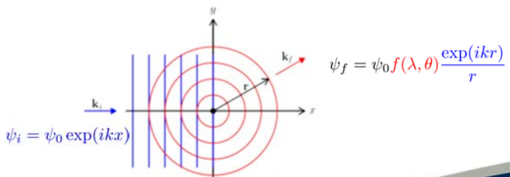
### Neutron and x-ray reflectivity

*How do we connect the scattering length profile with the reflectivity*



## Scattering from a single (fixed) atom

- atomic nuclei via the short-range (fm) strong force;
- unpaired orbital electrons via a magnetic dipole interaction

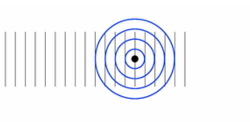





## Scattering length

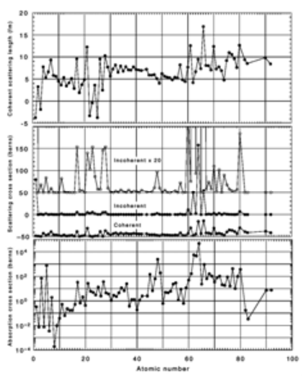
$$\sigma_{tot} = 4\pi b^2$$

Where *b* is the scattering length



- The sign of *b* is arbitrary
- A negative sign implies a change in the phase of the scattered wave
- *b* is sometimes complex and wavelength dependent due to resonant absorption
- *b* depends on the isotope
- *b* depends on the spin states of the neutron and nucleus






- Mass $1.67 \times 10^{-27}$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 |

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

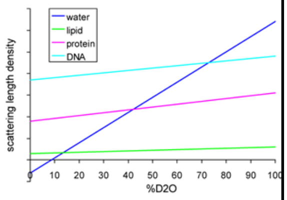
- Isotopic substitution for contrast
- Isotopic substitution to move peak positions in spectroscopy
- Incoherent scattering



# 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 |

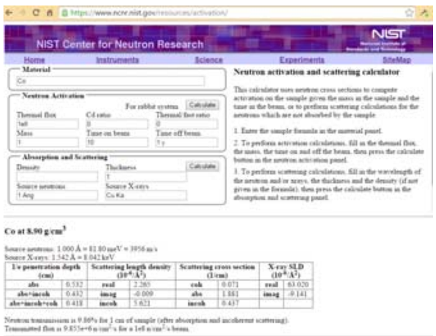



Intro to SANS SM King



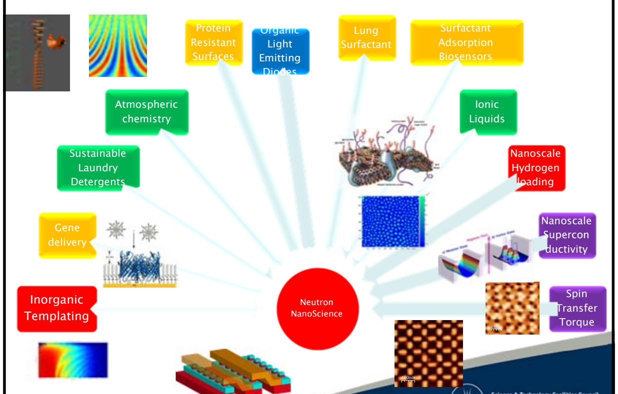
# SLD

http://www.ncnr.nist.gov/resources/sldcalc.html



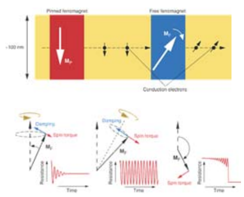



Fundamentally driven, *technologically relevant*

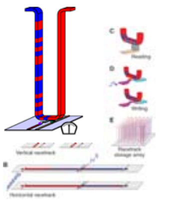





# The Importance of interfaces

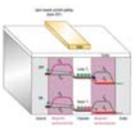


I. N. Krivorotov et al., Science **307**, 228 (2005).

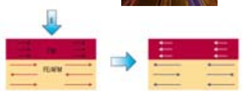



S.S.P.Parkin et al. Science **520** 5873 (2008)

Maccherozzi et al. Phys. Rev. Lett. **101**, 267201 (2008)



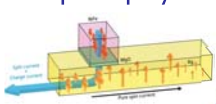
Awschalom and Flatté Nat. Phys. **3** 153 (2007)



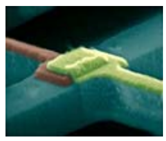
Ramesh and Spaldin Nat. Mat. **6**, 21 (2007)



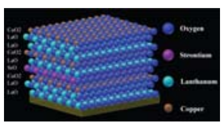
# Complex physics: advanced characterisation



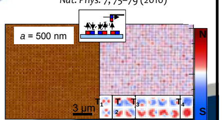
- x-section
- Polarisation
- Interfacial sensitivity

*Nat. Phys.* 7, 75–79 (2010)



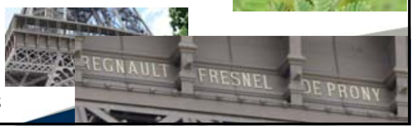

Nature Materials 11, 103–113 (2012)



# Interference effects





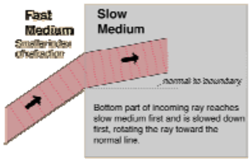

- Fresnel reflection 1815

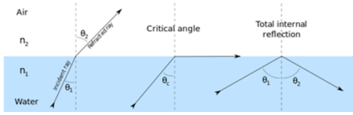
# Refractive Index

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

- n varies with wavelength: dispersion

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




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



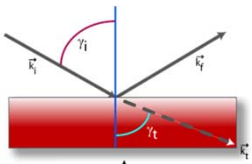
$$
\begin{aligned}
n &= 1 - \lambda^2 A - i\lambda B && (1)\\
A &= \frac{Nb}{2\pi} && (2)\\
B &= \frac{N(\sigma_a + \sigma_i)}{4\pi} && (3)
\end{aligned}
$$

$$
\begin{aligned}
n &= 1 - \alpha - i\beta && (1)\\
\alpha &= \frac{N\lambda^2 |r_e|}{2\pi} && (2)\\
\beta &= \frac{\lambda\mu}{4\pi} && (3)
\end{aligned}
$$

n <1 Total External reflection

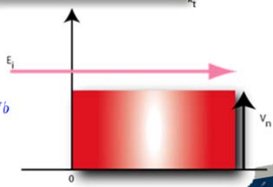
# Index of Refraction: Neutrons

$$
\begin{aligned}
n &= \frac{\sin\gamma_i}{\sin\gamma_t}\\
&= \frac{|\vec{k}_t|}{|\vec{k}_i|}
\end{aligned}
$$



$$
\begin{aligned}
n^2 &= \frac{|\vec{k}_t|^2}{|\vec{k}_i|^2}\\
&= \frac{E_t}{E_i}\\
&= \frac{E_i - V_n}{E_i}\\
&= 1 - \frac{4\pi}{k_i^2} Nb
\end{aligned}
$$

$V_n = \frac{2\pi\hbar^2}{m_n} Nb$

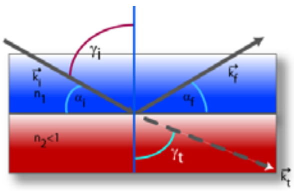




# Critical Reflection

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

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



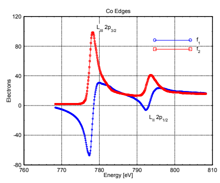
$$
\begin{aligned}
Q_c &= \frac{4\pi}{\lambda} 2k \sin\alpha_c\\
&= 2k\sqrt{1 - \cos^2\alpha_c}\\
&= \sqrt{4k^2\left(1 - n^2\right)}\\
&\cong \sqrt{4k^2 \cdot 2\delta}\\
&= \sqrt{16\pi Nb}
\end{aligned}
$$

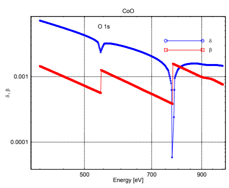
| Material | $\theta_c$ / Å |
|---|---|
| Ni | 0.1 |
| Cu | 0.083 |
| Al | 0.047 |
| Si | 0.047 |
| $D_2O$ | 0.082 |

- $Q_c$ only depends on the material!



# Index of Refraction: Photons

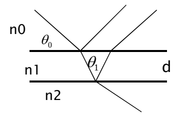



$$
\begin{aligned}
n &= 1 - \delta - i\beta\\
&= 1 - \frac{r_e}{2\pi}\lambda^2 \sum_i n_i f_i(0)\\
f(0) &= f_1 + i f_2\\
&= f_1 + i\frac{\sigma_a}{2 r_e \lambda}\\
&= Z^* + \frac{1}{\pi r_e h c}\int_0^\infty \frac{\epsilon^2 \sigma_a(\epsilon)}{E^2 - \epsilon^2}
\end{aligned}
$$

http://www-cxro.lbl.gov/optical_constants/

# Fresnel's law for a surface and thin film



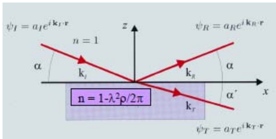

$k_i = n_i \sin\theta$

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

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

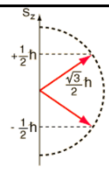
$r_{ij} = \frac{a_r}{a_i} = \frac{k_i - k_j}{k_i + k_j}$

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

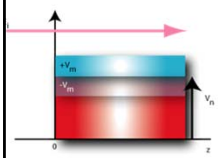



http://www.che.udel.edu/cns/pdf/Reflectometry.pdf

# Using the neutron's spin



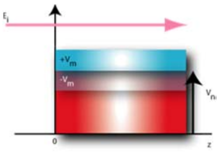

- The neutron is a spin ½ particle
- The neutron possesses an intrinsic magnetic moment: spin
- Caution…




$\vec{\mu_n} = \gamma\mu_N \vec{\sigma}$

$\sigma_x = \begin{pmatrix} 0 & 1 \\ 1 & 0 \end{pmatrix}, \sigma_y = \begin{pmatrix} 0 & -i \\ i & 0 \end{pmatrix}, \sigma_z = \begin{pmatrix} 1 & 0 \\ 0 & -1 \end{pmatrix}$

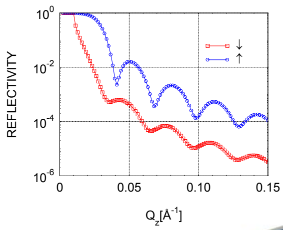
# PNR from a single layer




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

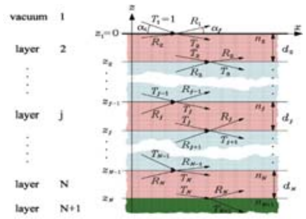
$$V = \frac{\hbar}{2\pi m} N(b \pm p) \quad (2)$$

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

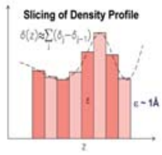




# Parratt Iteration

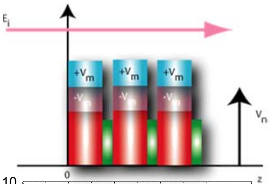



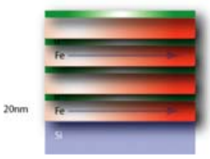

$X_j = \frac{R_j}{T_j} = \exp(-2ik_{z,j}z_j)\frac{r_{j,j+1} + X_{j+1}\exp(2ik_{z,j+1}z_j)}{1 + r_{j,j+1}X_{j+1}\exp(2ik_{z,j+1}z_j)}$

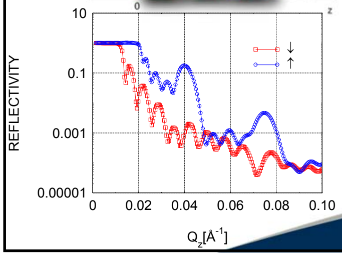



Can now simulate profile with a "*slice and dice*" approach

# PNR from a multiple layers

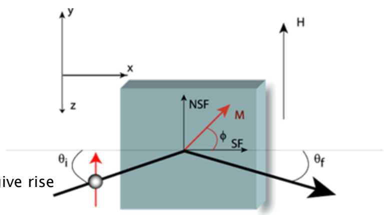



# Spin dependent cross-section

- 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

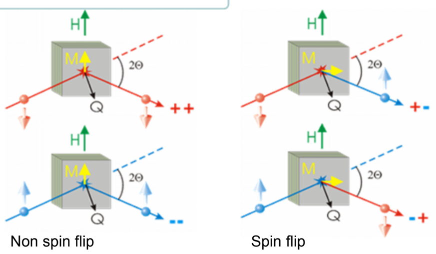
$b = b + p\sin\phi$

$p_m \cos\phi = p_x$

$\left[\frac{-\hbar^2}{2m_n}\nabla^2 + V(r)\right]\Psi^{\uparrow,\downarrow} = E\Psi^{\uparrow,\downarrow}$

## Polarisation

Polarised neutron reflection



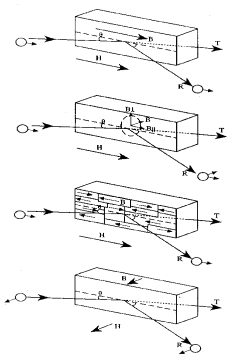

Non spin flip
++ measures b + $M_z$
- - measures b - $M_z$

Spin flip
+ - measures $M_x$ + i$M_y$
- + measures $M_x$ - i$M_y$

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



## Effects of magnetisation on neutron reflectivity



(a) Spin dependent reflectivity, potential is Zeeman split

(b) Rotation of the neutron polarisation. Resonance or spin flip channel

(c) Average of the 2 spin states

$$\nabla \cdot \vec{B} = 0$$

(d) Pure nuclear, the component of B normal to the surface is continuous across the surface

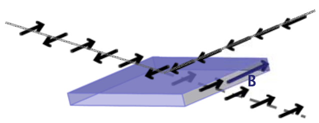
G.P. Felcher, Physica B, **192**, (1993), 137



## Polarisation

Polarising supermirrors

Magnetic materials have a spin dependent term in their refractive index




An ~60%/40% Fe/Co mirror works well at saturation



## Caveat to Classical Description

PHYSICAL REVIEW B 71, 214423 (2005)

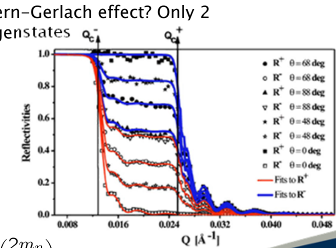
**Quantum states of neutrons in magnetic thin films**

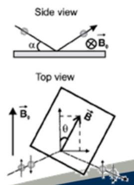
F. Radu,[1,*] V. Leiner,[2] M. Wolff,[1,3] V. K. Ignatovich,[4] and H. Zabel[1]
[1]Department of Physics, Ruhr-University Bochum, D-44780 Bochum, Germany
[2]Institut für Werkstoffforschung WFN, GKSS Forschungszentrum GmbH, 21502 Geesthacht, Germany
[3]Institut Laue-Langevin, F-38042 Grenoble Cedex 9, France
[4]Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, 141980, Dubna Moscow Region, Russia
(Received 12 April 2005; published 27 June 2005)

- CD predicts a continous variation of critical edge

$$\frac{4\pi \sin(\alpha_c^{\pm})}{\lambda} = Q_c^{\pm} = \sqrt{\frac{2m_n}{\hbar^2}\left(V_n \pm |\mu|\,|B_s|\cos(\theta)\right)}$$

- Stern-Gerlach effect? Only 2 eigenstates




$$k_{\pm}^2 = \left(\frac{2m_n}{\hbar^2}\right)(V_n \pm |\mu|\,|B|)$$

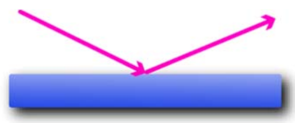


## Roughness

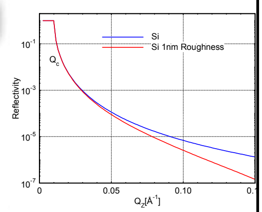
Structural and magnetic interfacial phenomena



## Structural Roughness



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



PHYSICAL REVIEW B VOLUME 38, NUMBER 4 1 AUGUST 1988

**X-ray and neutron scattering from rough surfaces**

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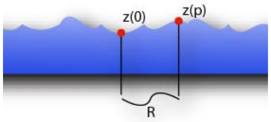
H. B. Stanley†
*University of Maryland, College Park, Maryland 20742*
(Received 30 November 1987)

$$C(R) = \left\langle \left[ z(x,y) - z(x',y') \right]^2 \right\rangle = \sigma^2 \exp(-r/\xi)^{2h}$$




- σ= roughness
- ξ= cut-off length:
  - for R > ξ, interface appears smooth,
  - for R < ξ, interface appears rough, fractal behaviour
- h=3-D Hurst parameter for jaggedness(0<h<1)
  - smooth: D=2, h=1
  - very rough D=3, h=0



## Simulation Packages



## Simulation Packages: neutron

http://neutronreflectivity.neutron-eu.net/main/SimulationPrograms



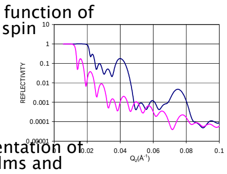
## Simulation Packages: photon

- X-ray
  - http://sergey.gmca.aps.anl.gov/
  - ESRF
  - RefTool



## Spin polarised Neutron Reflectivity

- Measure the reflected neutrons as a function of their perpendicular momentum and spin eigenstate
- $k^{\pm} = \sqrt{k - 4\pi N(b \pm cB)}$

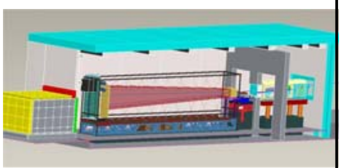



- Can observe the magnitude and orientation of atomic magnetic moments in thin films and multilayer media.
- Probe length scale (<1nm to >1000nm): covers many aspects of thin film structure and magnetism
- Complementary to:
  - VSM/SQUID
  - MOKE average magnetisation over the sample thickness
  - SEMPA, Lorentz surface domain magnetisation
  - XMCD/XRMS-element specific
- PNR gives the microscopic *in-plane vector* magnetisation *depth* profile.



## Inter

- Designed for the study of chemical interfaces, with a particular emphasis on the air-water interface
- >10 times the flux of SURF
- Much wider dynamic range
- Tuneable resolution

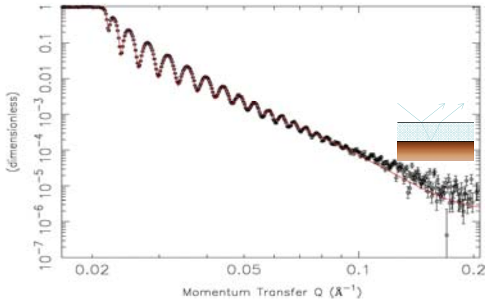


**Scientific Opportunities**

- Biology

| | |
|---|---|
| **D-spacing range** | 1 – 16 (22) Å |
| **Moderator** | Coupled s-$CH_4$ grooved – 26K |
| **Primary flight path** | 17m (m=3 supermirror guides) |
| **Secondary flight path** | 3-7 m |
| **Beam size** | 60(h) x 30(v) mm |
| **Flux at sample** | ~$10^7$ n/s/cm² |

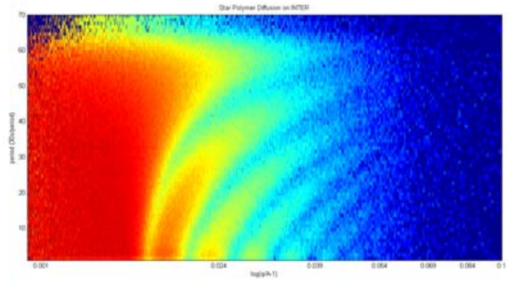
## First Results 29 sep 08




First reflectivity measured on INTER. Nickel/Carbon film (1216 Å) on glass



## Kinetics



- Star Polymer
- D.G. Bucknall *(2010)*



## PolRef

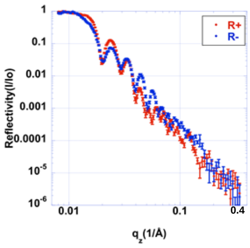
- Wavele
- Unpola
- Polariz
- Source
- Beam i 2.3°
- Well shielded Helium tube
- 640 channel linear gas detector with 0.5mm pixel

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



## Reflectometry Village

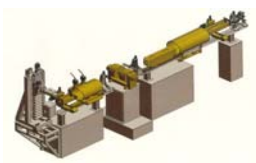





ASF08 T=10K, 3 angles (θ=0.4, 1.6, 3.2)

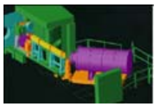


## Reflectometers



- Continuous or tof mode of operation
- 5Å Monochromator (6Å Polarised)
- White beam flux $9.6x10^9$ n/s/cm²

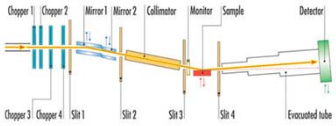
- The CRISP reflectometer at ISIS is a white beam time of flight (tof) polarised neutron reflectometer viewing a 20K hydrogen moderator.



http://www.ill.fr/YellowBook/D17/

http://www.ill.fr/YellowBook/ADAM/

## 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/



## Polref schematic

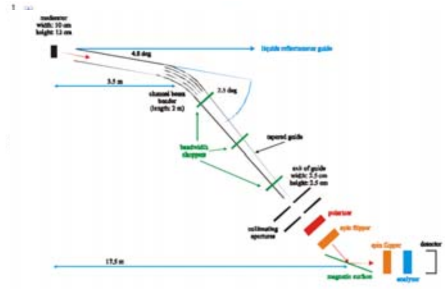


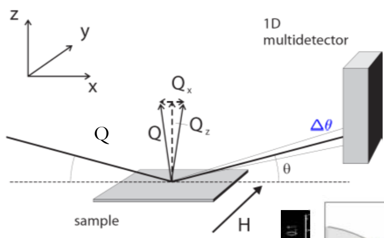
Figure 1 An outline side schematic of polREF.



## Off-specular scattering

Probing in-plane lengthscales

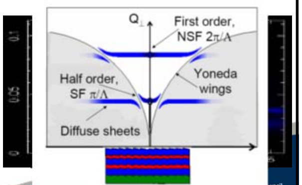
## Experimental Geometry




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

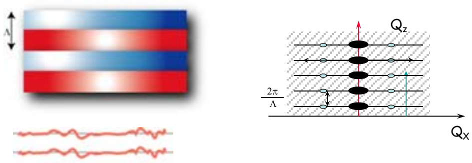
- λ range 0.5–6.5Å
- 1-d Detector
- Typical acquisition~2hours/field
- Measured coherence length ~30μm

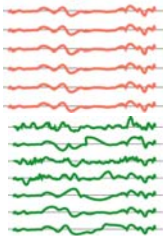
$\theta$



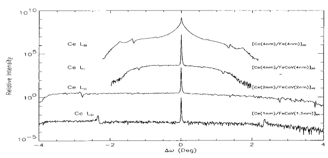



## Diffuse scattering



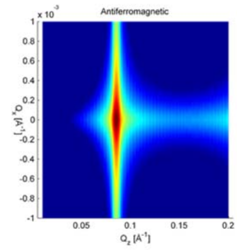
Holý and Baumbach, prb, 49, 10668, (1994)



(Ce/Fe) Tixier, Mannix et al.



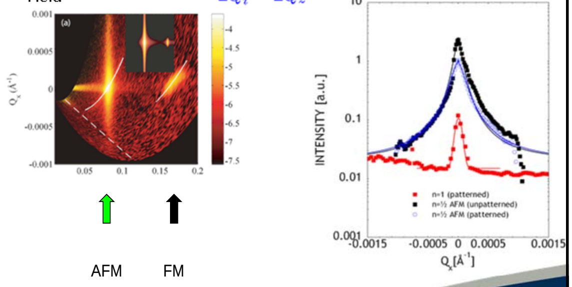
## Kinematic calculation




- Langridge *et al. Phys. Rev. Lett.* (2000) **85**, 4964

Coercive Field

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



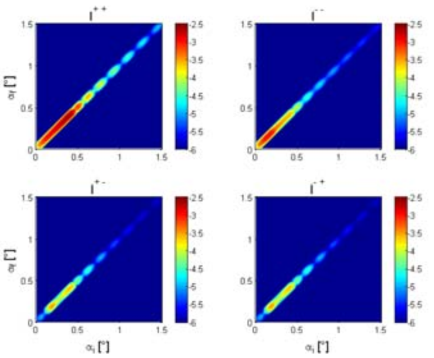



## Domains and the coherence volume

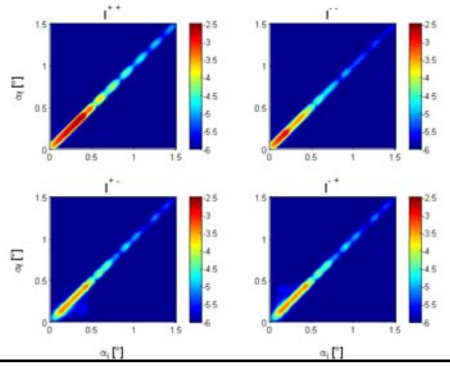
Many thanks to H. Zabel for slides



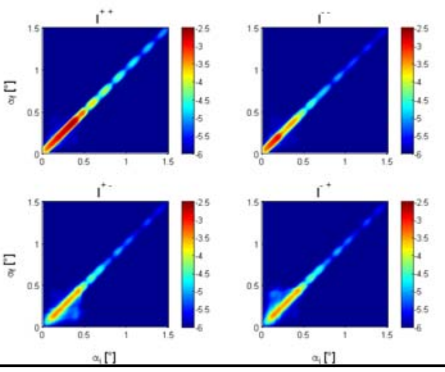
## *Domain size: 100 μm ( > $L_c$)*



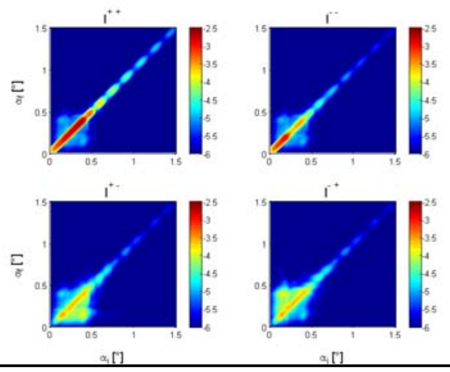
## *Domain size: 100 μm*



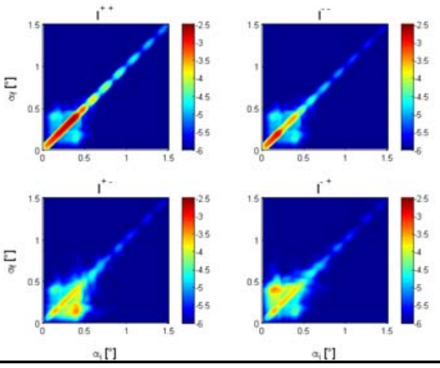
## *Domain size: 50 μm*



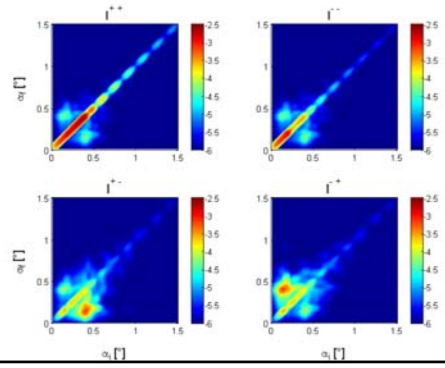
## *Domain size: 10 μm*



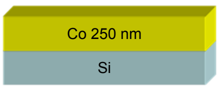
## *Domain size: 5 µm*

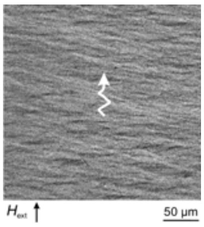



## *Domain size: 1 µm*




## Single ferromagnetic film in the domain state

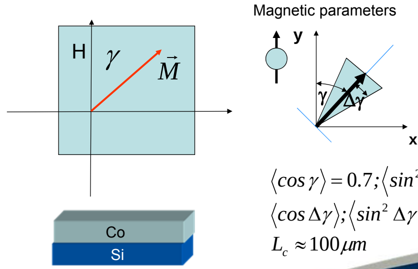



F. Radu *et al*, J. Phys.: Condens. Matter **17** (2005) 1711-1718



## Simulation of a Co film on a substrate



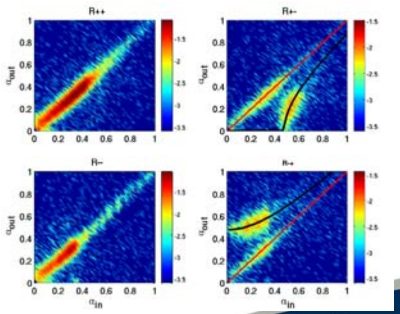

$\langle \cos\gamma \rangle = 0.7; \langle \sin^2\gamma \rangle = 0.25;$

$\langle \cos\Delta\gamma \rangle; \langle \sin^2\Delta\gamma \rangle$

$L_c \approx 100\mu m$

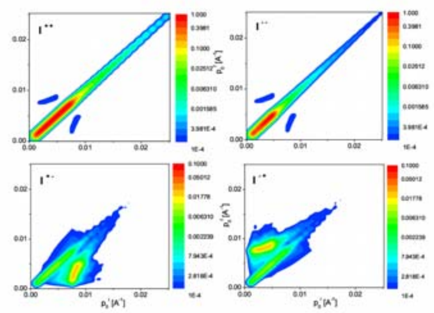


## Banana shape off-specular scattering from domain state






## Simulation of domain state



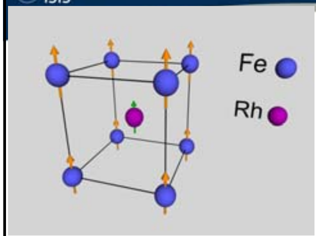
B.P. Toperverg Physica B **297** (2001) 160-168

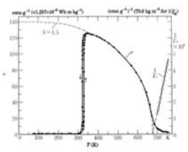
## Examples of reflectivity

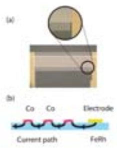
A Magnetic controllable interface

## doped-FeRh

### FeRh: Bulk Properties





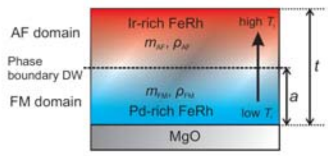
- CsCl structure
  - a~2.99Å
  - α' phase
- 300K: Type G AF
- Fe: ~3.3 $\mu_B$
- Rh: no moment
- FM alignment within <111> planes
- AF alignment between <111> planes
- 350 K: AF → FM
- Fe: ~3.1 $\mu_B$
- Rh: ~1 $\mu_B$

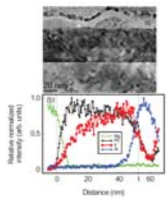

- J. S. Kouvel and C. C. Hartelius, JAP, **33**, 1343 (1962).
- G. Shirane *et al*. Phys. Rev. **134**, A1547 (1964)
- T. Naito et al., J Appl Phys **109**, 07C911 (2011)
- R. Fan, SL et al. Phys Rev B **82**, 184418 (2010).


D. Arena, BNL

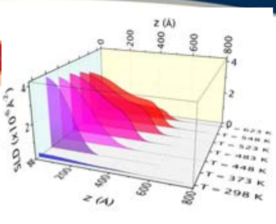
### Motivation: Tuneable magnetostructural phase transition

- 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.

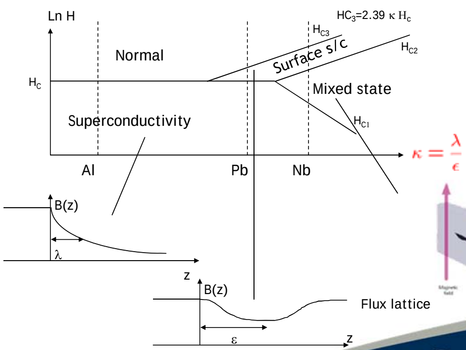


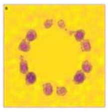

- Evidence of a controllable, mobile magnetic (electronic) domain wall
- Complex structure
- Neutrons essential to quantitatively resolving the complex magnetic structure

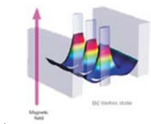
## Superconductivity and reflectivity

*Towards superconducting spintronics*

## Flux penetration in superconductors





R.J. Cubitt *et al* Phys. Rev. Lett. **91** 047002 (2003)



## Flux penetration in superconductors

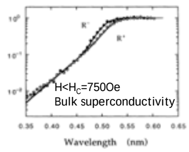



FIG. 2. The spin-dependent reflectivities $R^+$ and $R^-$ measured in an applied magnetic field of $6.0\times10^4$ A/m (750 Oe). The continuous lines are the reflectivities calculated for the two polarization states, with the same instrumental and surface parameters as in Fig. 1, and an exponential decay of magnetic induction with a penetration depth of 39 nm.

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

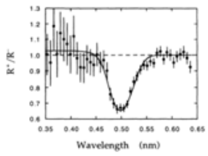
**Magnetic-induction profile in a type-I superconductor by polarized-neutron reflectometry**

M. P. Nutley*
*Institut Laue-Langevin, 156X, 38042 Grenoble CEDEX, France*

A. T. Boothroyd and C. R. Staddon
*Department of Physics, Clarendon Laboratory, University of Oxford, Parks Road, Oxford, OX1 3PU, United Kingdom*

D. McK. Paul
*Department of Physics, University of Warwick, Coventry, CV4 7AL, United Kingdom*

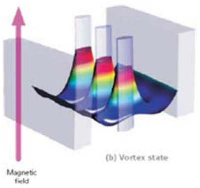
J. Penfold
*ISIS Science Division, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon., OX11 0QX, United Kingdom*
(Received 17 February 1994)

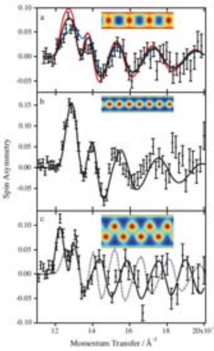



- 1μm Pb film
- PbO surface layer
- $B(z)=\mu_0H\exp(-z/\lambda)$
- $\lambda$=39±1nm
- No field dependence



## Visualising the Flux distribution

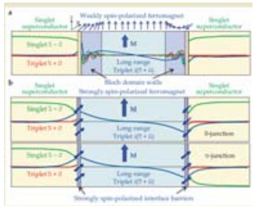


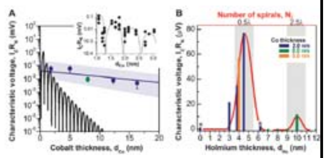



A Drew *et al* Phys. Rev. B. **80** 134510 (2009)

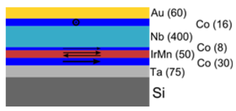


## Generating a triplet state






- first found in heavy fermion materials such as $UGe_2$ or URhGe
- *orbital* and *spin* components of the wavefunction are both *even* with respect to electron exchange, thus they must be an *odd-frequency*, odd with respect to time reversal
- non-collinear magnetism within a distance of the coherence length required



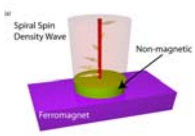

- Robinson et al *Science* **329**, 59 (2010)
- M. Eschrig Physics Today (2011)



## Motivation

### Bulk Spin Transfer Torque

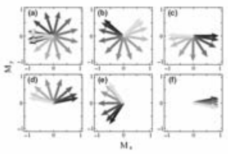
- PRL **96**, 256601 (2006)
- PRB **79**, 104433 (2009)




$$|f| = (1/2\pi J\hbar)\sqrt{(C_{23}j_3)^2 - (6K_6^6)^2}$$

### Exotic Phase

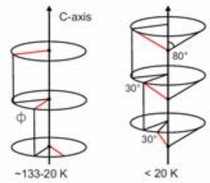
- PRB **78**, 020402(R) (2008)
- PRB **79**, 134420 (2009)



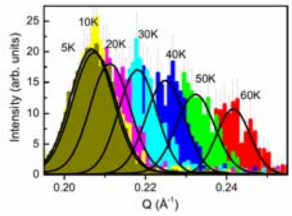
- Spin-triplet SC Science, **329**, 59 (2010)
- RMP **77** 1321 (2005)



## Magnetic Spirals in Holmium thin films

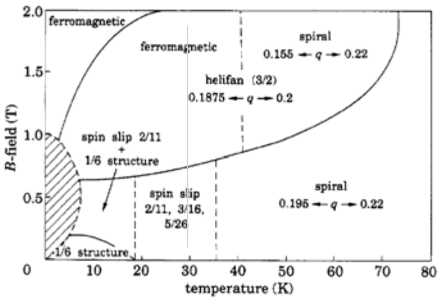



The representative magnetic structure of bulk Ho below its Néel temperature (left) and below its Curie temperature (right).




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

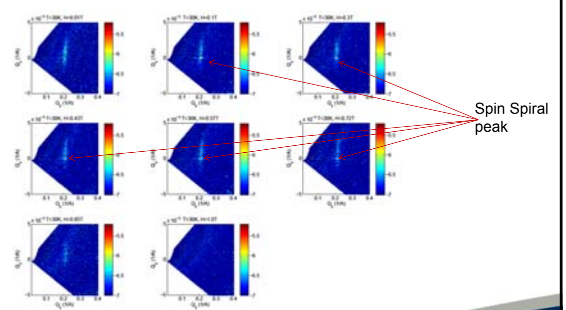
# Bulk Ho Field Phase diagram




D. A Jehan *et al.*, Europhys. Letts. 17 553 (1992)

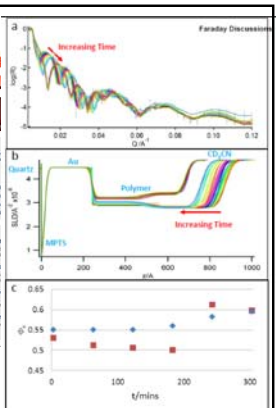


# Polref: Diffuse scattering from a holmium spin spiral: 7.5T




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




- Combining Figaro and INTER

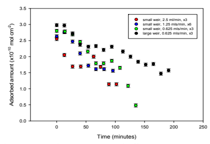
A. R. Hillman et al. Faraday Discussions (2013).



# Kinetics of surface desorption

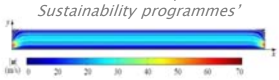
Kinetics of anionic surfactant desorption followed by neutron reflectivity on INTER

Small trough, d-LAS4/nrw

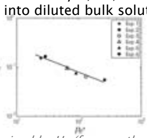



C. Morgan, C. Breward, I. Griffiths, P. Howell, J. Penfold, R. Thomas, I. Tucker, J. T. Petkov, and J. R. P. Webster, Langmuir **28**, 17339 (2012).

*'Understanding and optimising surfactant rinse mechanisms important for Sustainability programmes'*



Diffusion from a near-surface stagnant layer, Hc,
into diluted bulk solution

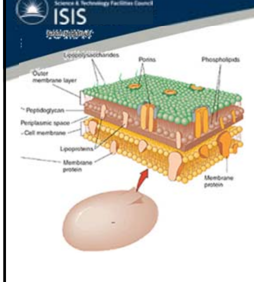


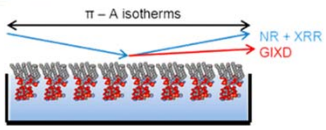
*Kinetics determined by Hc (from mathematical model) and scales with Peclet number (flow conditions)*

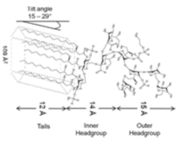


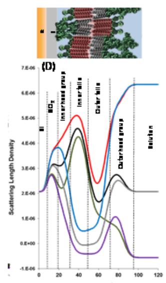


Building model gram negative bacterial membranes



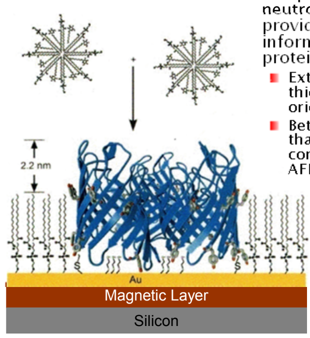



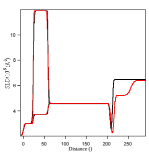



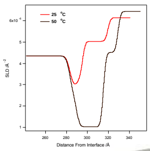

- **Gram negative bacteria due to their increasing antibiotic resistance and their role in biotechnological processes.**
- **The asymmetric membrane is the outer surface.**
- **Work has been on going to produce accurate models of the gram negative bacterial membrane using extracted bacterial lipopolysaccharides for future use in biophysical studies on membrane behaviour and interactions.**

# Polarised Neutrons for Biology




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



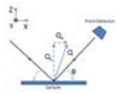


Seeing hydrogen

# 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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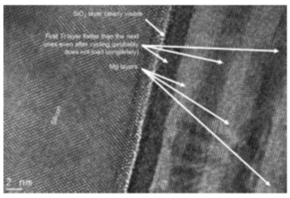
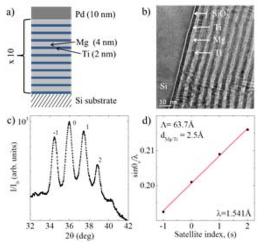
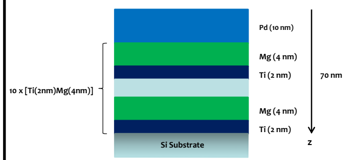
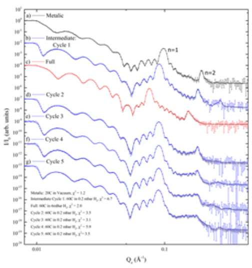


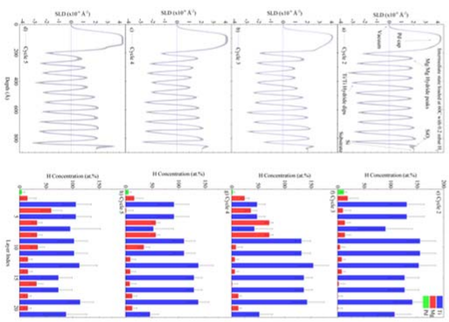




## Sample Structure and Initial Measurement



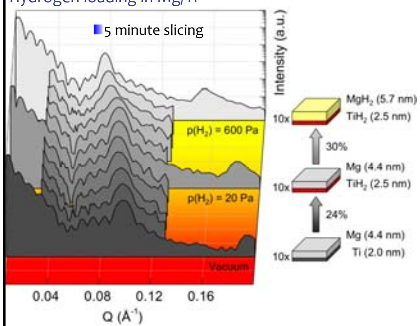



- Mg/Ti Multilayer Grown on is substrate by DC Magnetron sputtering.
- Clear shifting due to 30% lattice expansion between the Hydrogen loaded and unloaded states.



## Hydrogen loading in Mg/Ti

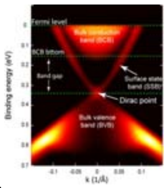
- 5 minute slicing



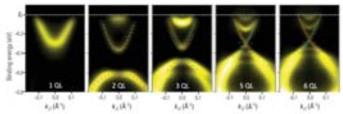

Neutron reflectometry patterns measured during hydrogenation of the 10x{Ti/Mg} multilayer at 333K, in vacuum and after exposure to 20 and 600 Pa of $H_2$.

## QUANTUM MATTER

## QAHE & Topological Insulators (TIs)



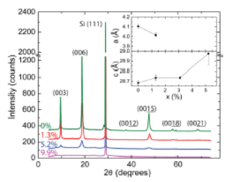

- Quantum Hall effect observed in 2DEG
  - $\sigma = v \frac{e^2}{h}$
- Anomalous Quantum Hall effect (QAH)
  - Band inversion
  - Ferromagnetic insulator breaking TRS
- TIs possess insulating bulk gap and gapless edge states
  - Magnetic dopants

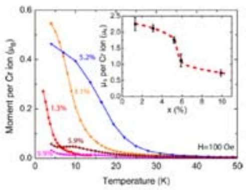



Yu et al. Science **329** (2010) 61
Chang et al. Science **3340** (2013) 167
He, K., Wang et al. *Natl. Sci. Rev.* **1**, 38–48 (2013)

## Cr doped $Bi_2Se_3$

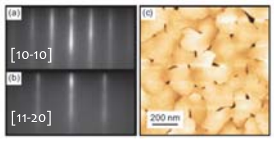
- Tc~20K for 5%
- Good crystalline quality ~<5%

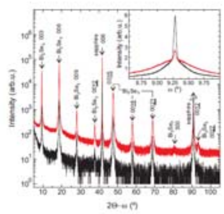


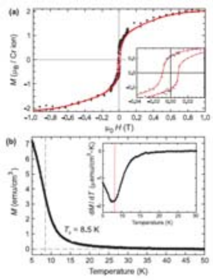




Haazen et al. APL **100** (2012) 082404

## $(Cr_{0.12}Bi_{0.88})_2Se_3$



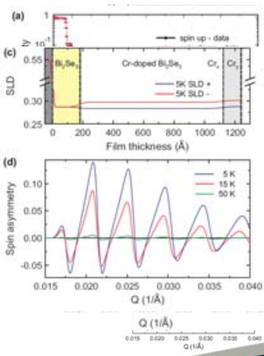




x = 0.12

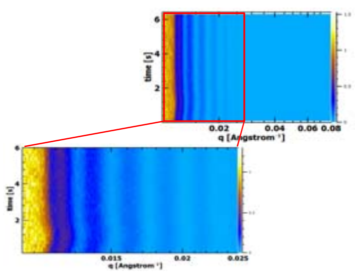
- Cr concentration fixed by RBS
- $1.5\mu_B$/Cr @0.7T
- No enhancement observed in the near surface region
- Moment less than $3.78\mu_B$ of $Cr^{3+}$ substituted on Bi site

- **Summary**
- Cr doping up to 12% without a loss of crystallinity (substitutional doping)
- Moment lower (2.1) than expected for $Cr^{3+}$
- Homogeneous ferromagnetism
- Precursor to observing the QAHE (lower temperatures)




Collins-McIntyre, L. J. *et al.* Magnetic ordering in Cr-doped $Bi_2Se_3$ thin films. *Europhys. Lett.* **107,** 57009 (2014).

## New capability, new science




- Visible change in $Q_c$ and shift in Kiessig fringes

- Stroboscopic measurement on INTER
- Courtesy of A. Glidle *et al.*
- Time slices are 200 milliseconds
- Polymer redox system

OSNS
Sept 2015

## 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



## References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