# Resolving the interfacial behaviour in complex magnetic nanostructures

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OSNS19

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## Alternatively…

God made solids, but surfaces were the work of the devil!

*Wolfgang Pauli 1900-1958*

en.wikipedia.org

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# Alternatively…

"There's Plenty of Room at the Bottom: An Invitation to Enter a New Field of Physics"

*RP Feynman 1918-1988*

en.wikipedia.org

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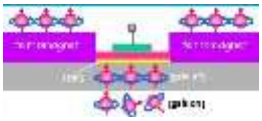
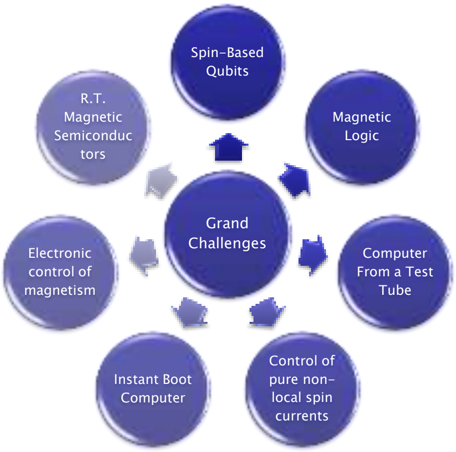
# Grand Challenges: Nanomagnetism





Spin-Based Qubits

Magnetic Logic

Computer From a Test Tube

Control of pure non-local spin currents

Instant Boot Computer

Electronic control of magnetism

R.T. Magnetic Semiconductors

Grand Challenges

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Adapted from S.D. Bader *Rev Mod Phys* **78** (2006)

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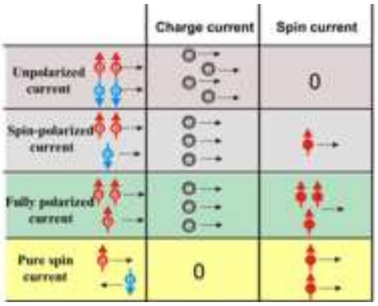
# (Super-)Spintronics

- Control and manipulation of the spin degree of freedom in the solid state environment

- Spin
  - **Transport**
  - Dynamics
  - Relaxation



Solid State Physics Lab., Kyushu Univ.

- Information storage
- Quantum Computing

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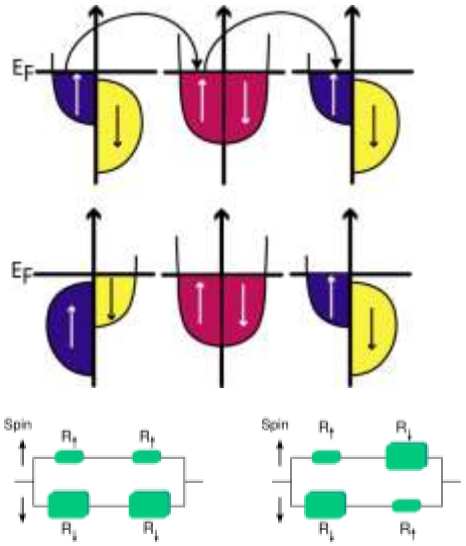
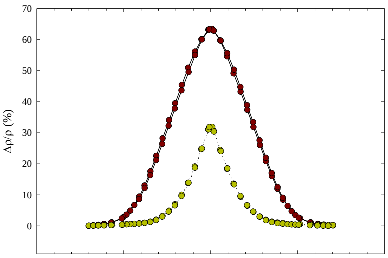
# Quantum Well State

- Band matching important *e.g.* Fe/Cr



Co/Cu GMR



THE NOBEL PRIZE

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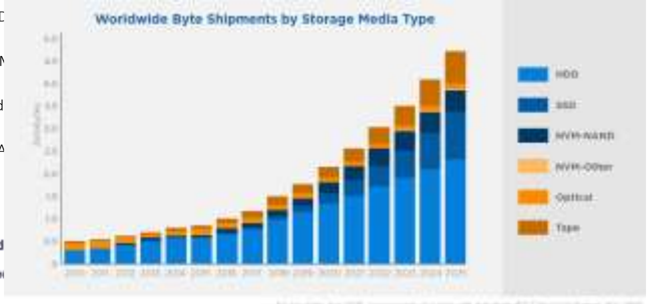
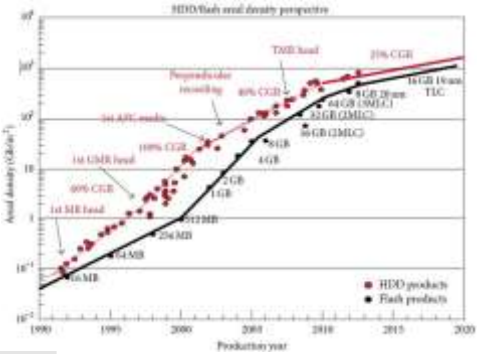
# A history of storage/spintronics

- 1956
  - RAMAC
- 1988
  - Discovery of GMR
- 1991
  - Spin Valve (IBM)
- 1994
  - MTJ
- 1997
  - SV in a HD
- 2004
  - Epitaxial M
- 2005
  - MTJ Read
- 2006
  - 4Mb MRA



Chappert, Fert, Nguyen van Dau Nat. Matl. **6**, 813 (2007)

Li Hei Thesis 2016 and refs therein



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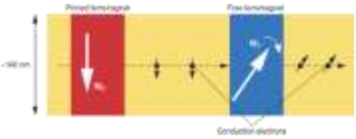
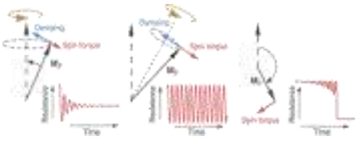
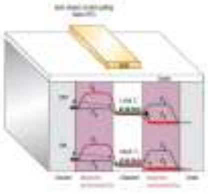
# The Importance of interfaces

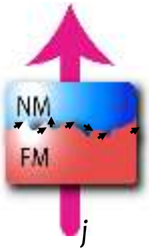


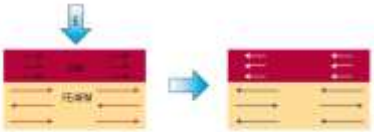


I. N. Krivorotov et al., Science **307**, 228 (2005).

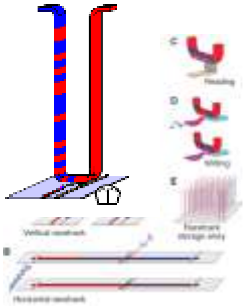


Awschalom and Flatté Nat. Phys. **3** 153 (2007)





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



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

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

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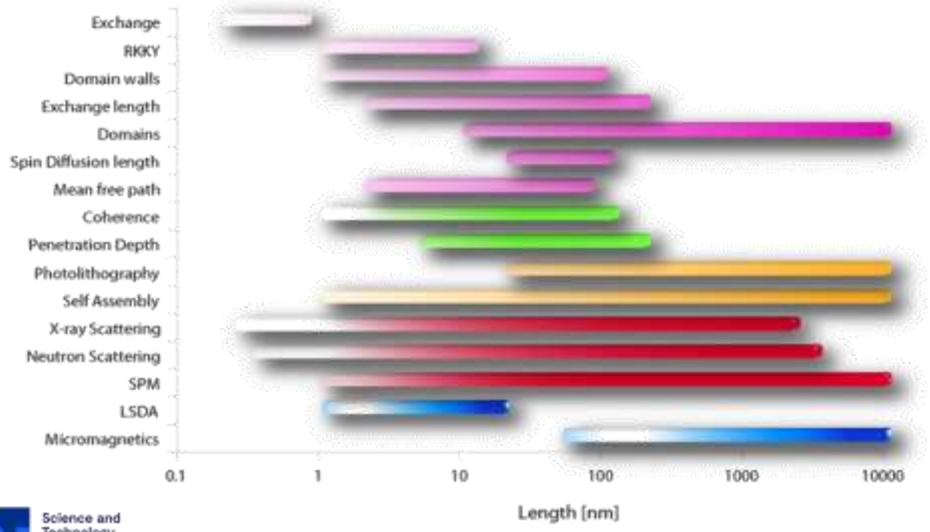
## Relevance of scattering techniques to interfacial effects



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Adapted from I.K. Schuller *et al.* J. Magn. Magn.Mater. 200 (1999) 571.

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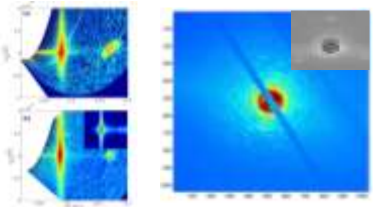
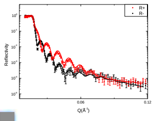
## Nanoscale Electronic Phenomena Research

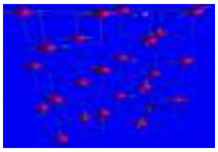
Domain Structures



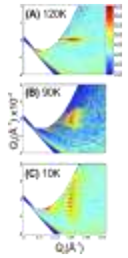
Heusler, DMS



Surface Magnetism

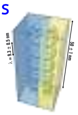


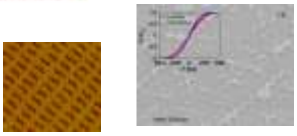
Exchange Bias
- Conventional EXB
- Synthetic EXB
- Frozen magnetism



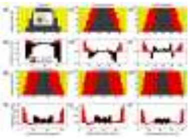
- To understand and control spin and interfacial phenomena
- Probe length scale (<1nm to >1000nm)
- Vector Magnetometry ~<0.1$\mu_B$ per f.u.

Helimagnetism-Skyrmions

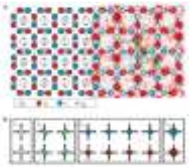




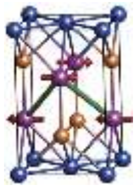
Interfacial Magnetism



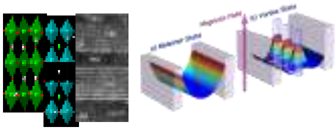
Frustration



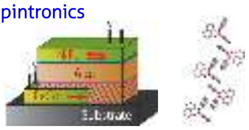
AF Semiconductors



Singlet-Triplet Superconductivity



Organic Spintronics



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# Motivation & Outline

- Motivation
  - *Unique* and *quantitative* description of complex electronic materials on the microscopic lengthscale
  - Relating the functional properties of materials to their atomic and nanoscale structure
- Introduction to Polarised Neutron Reflectivity (PNR)
- Ferrimagnetic insulators for spintronics/magnonics
  - *Understand interfacial behaviour in spin-current, magnonic systems*
  - *Understand some of the low-T anomalies in thin ferrimagnetic insulator films*
  - *Characterise the spin axis on FI/AFI systems*
- Chiral Magnetism
  - *Control of helical structures*
  - *Study of DMI in thin films*
- Magnetocaloric Material
- Towards Super-Spintronics
- Small Angle Scattering
- Summary & Conclusions

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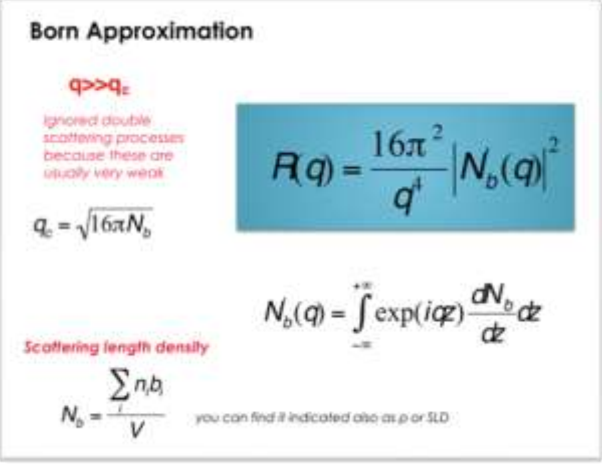
# Neutron reflectivity

- Recall Dr Fragneto's Talk Last Week



**Born Approximation**

$q>>q_c$

ignored double scattering processes because these are usually very weak

$$q_c = \sqrt{16\pi N_b}$$

$$R(q) = \frac{16\pi^2}{q^4}\left|N_b(q)\right|^2$$

$$N_b(q) = \int_{-\infty}^{+\infty} \exp(iqz)\frac{dN_b}{dz}dz$$

Scattering length density

$$N_b = \frac{\sum_i n_i b_i}{V}$$

you can find it indicated also as $\rho$ or SLD

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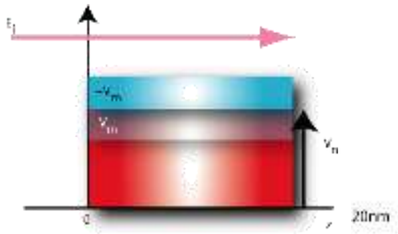
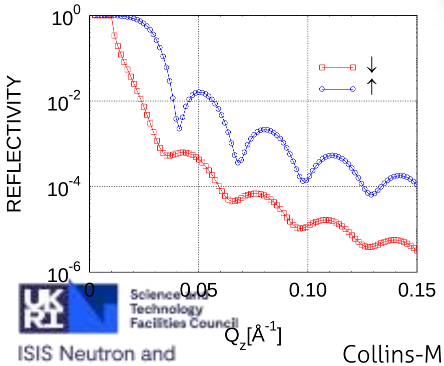
## PNR from a single layer

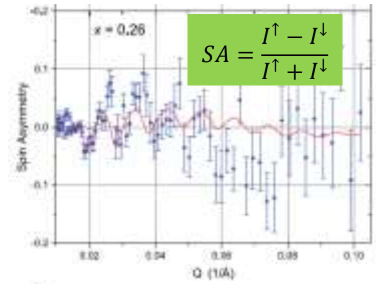


$$V = V_n + V_m$$

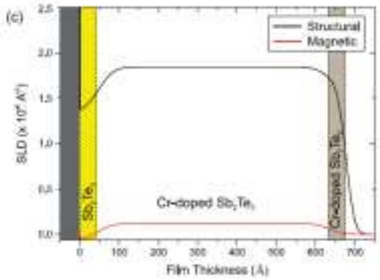
$$V = \frac{\hbar}{2\pi m} N(b \pm p)$$

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





$$SA = \frac{I^{\uparrow} - I^{\downarrow}}{I^{\uparrow} + I^{\downarrow}}$$



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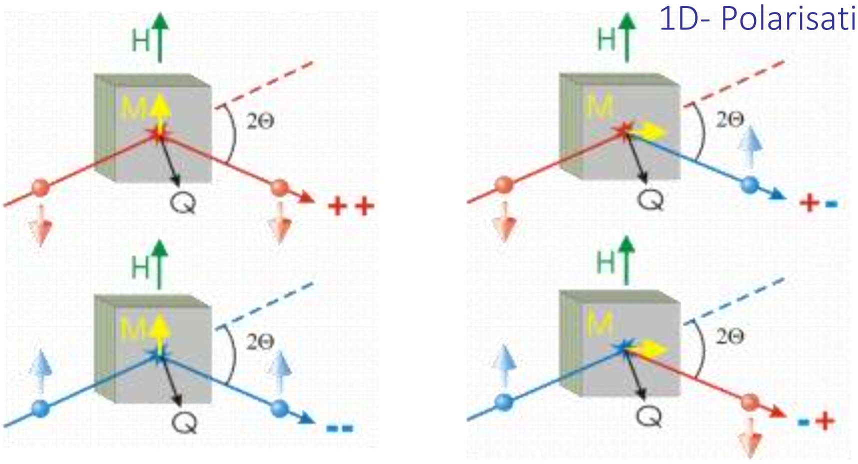
Collins-McIntyre, L. J. *et al.*
*Europhysics Lett.* **115,** 27006 (2016)

## 1D- Polarisation Analysis



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

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

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

## PolREF BEAMLINE

- TOF wavelength band 1Å – 16Å
- Vertical and horizontal geometry
- Non-polarised, polarised and polarisation analysis modes
- Sample point goniometer capable of moving 1000kg
- Experimental Setups:
  - GMW Magnet (± 1T).
  - Cryostat (2.5K -300K), (sub 1K fridge) ad hoc in-situ transport.
  - Vacuum furnace (300K – 800K).
  - PNR/PA Polarised modes.
  - Various soft matter setups
  - H loading

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## Generating ‘Pure’ Spin currents: Ferrimagnetic Insulators

- Understanding the interfacial behaviour

‘As spin pumping in heterostructures requires the transfer of spin information across an interface, the role of interface quality must be understood thoroughly, yet it remains unclear.’

Gray, M. T. *et al*. Phys. Rev. Appl. **9**, 064039 (2018).

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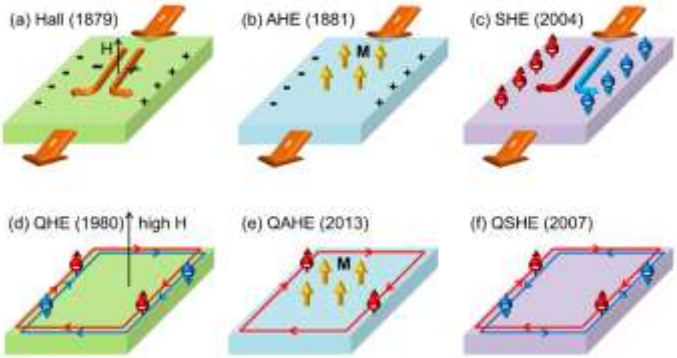
# Meet the Hall family…

‘On a new action of the Magnet on Electric Currents’
American Journal of Mathematics vol 2, 1879, p.287-292



- For a century only the two effects
- Extrinsic *vs.* intrinsic
  - SOC impurities
    - E.g. through spin injection
- Ideal for sensor technologies to low-power technologies
  - Spin transistors, spin logic, and spin quantum computing

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Chang, C.-Z. & Li, M. Quantum anomalous Hall effect in time-reversal-symmetry breaking topological insulators. J. Phys. Condens. Matter **28**, 123002 (2016).

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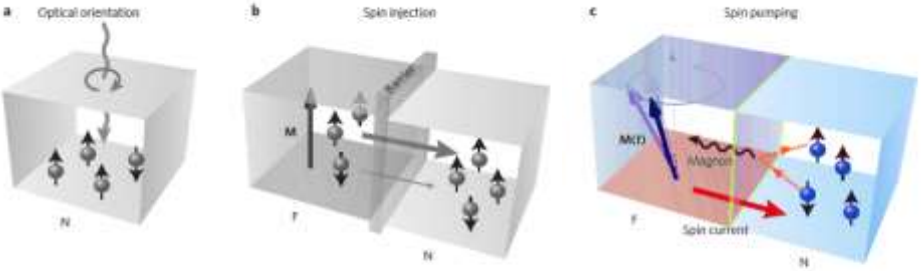
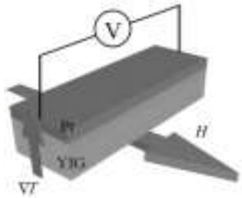
# Generate a spin imbalance

- Various mechanisms





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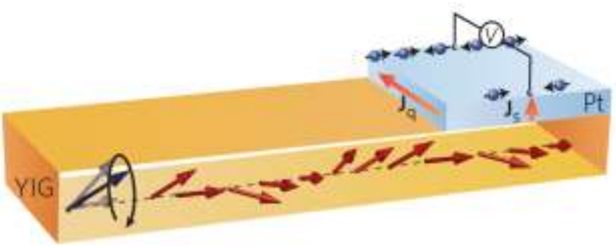
Qiu, Z., Hou, D., Uchida, K. & Saitoh, E. Influence of interface condition on spin-Seebeck effects. *J. Phys. D. Appl. Phys.* **48,** 164013 (2015).

Žutić, I. & Dery, H. Spintronics: Taming spin currents. *Nat. Mater.* **10,** 647 (2011)

# Inverse Spin Hall effect



- Exploit the SOC
  - Spin and orbital motion are coupled
- SOC limits spin diffusion length
  - Channel for spin relaxation
- SOC makes effective sink for the angular momentum
- Applying a field increases the number of magnons and the lattice acts as a source of spin current
- Use YIG as a source of spin current
- Pt as a spin converter

Žutić, I. & Dery, H. Spintronics: Taming spin currents. *Nat. Mater.* **10,** 647 (2011)

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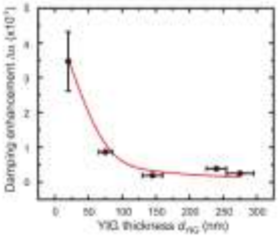
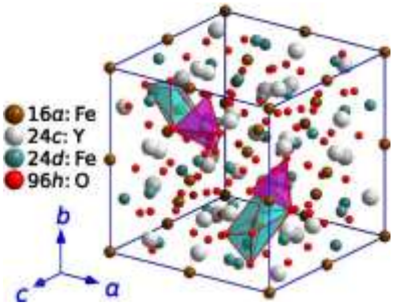
# YIG: $Y_3Fe_5O_{12}$

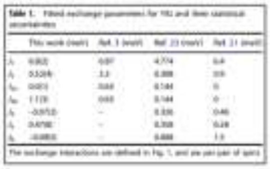
- YIG ferrimagnetic insulator
  - Ferrimagnetic insulator
    - Bandgap 2.58eV
    - $T_c$ = 560K
  - Transparent above 600nm
  - Low absorption in IR
  - Very low damping
  - Small linewidth for esr
  - Long spin wave decay length/ magnon damping
- Ideal for:
  - optical and magneto-optical applications,
  - microwave filters
  - Spin Seebeck/Peltier applications

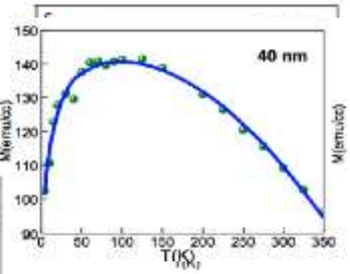


Jungfleisch, M. B. *et al.* Thickness and power dependence of the spin-pumping effect in YIG/Pt *Phys. Rev. B* **91,** 134407 (2015).



A. Kreisel, Goethe Univ.





- Princep, A. J. *et al.* The full magnon spectrum of yttrium iron garnet. *npj Quantum Mater.* **2,** 63 (2017).

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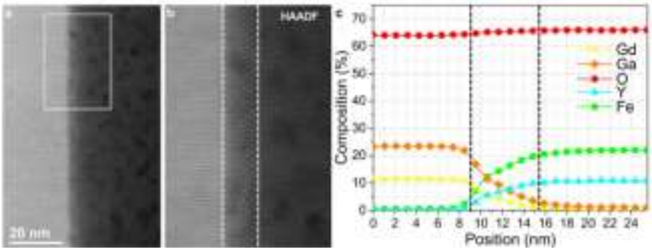
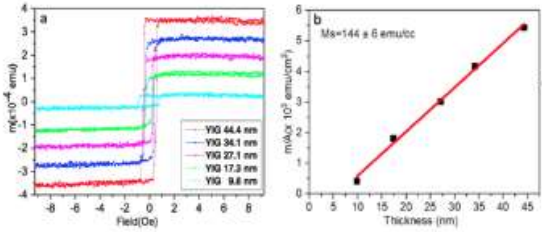
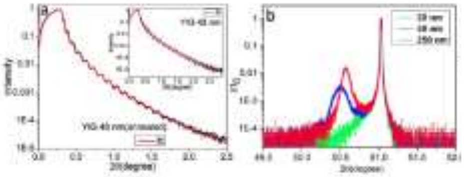
# Thin film YIG

- Numerous Growth techniques
- RF sputtered followed by anneal 850c for
- Grow on GGG ($Gd_3Ga_5O_{12}$)
- 0.06% lattice mismatch
- SuperSTEM
  - Abberation corrected
  - HAADF
  - 6nm diffusion
  - Diffusion co-efficient $1x10^{-17}cm^2s^{-1}$ (agrees with bulk)

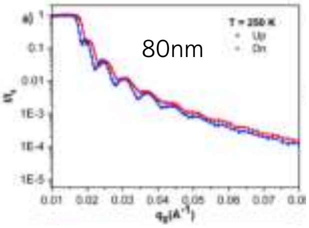






# PNR Data

- Analysed within a simple 2-layer model
  - 2 layer model
    - Pristine YIG
    - Gd doped YIG
    - $3.8\mu_B$/u.c.
  - Gd 6-7nm diffusion
  - Excellent agreement with x-ray and SuperSTEM
  - Gd absorption cross-section enhances sensitivity
  - Gd Iron Garnet: room temperature compensation



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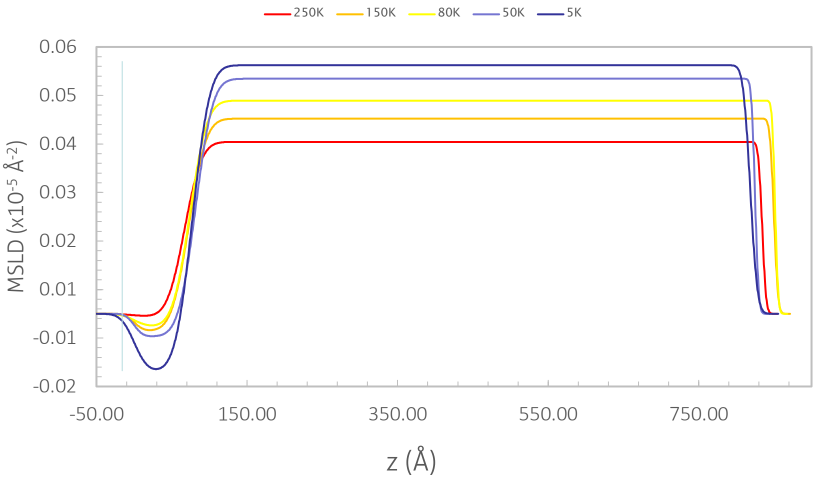
# Temperature Dependence

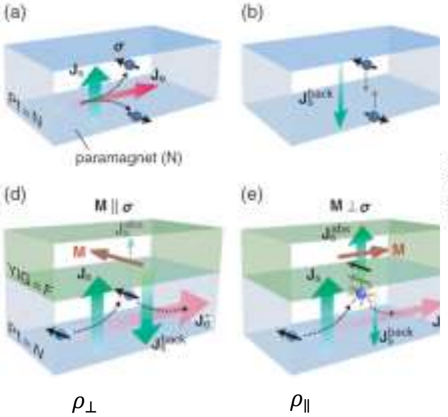
- Gd Iron Garnet: room temperature compensation
- M(T) YIG follows Bloch law
- Antiparallel moment develops at low temperature near to interface
- Gd substitutes on Y site
- Unusual magnetisation temperature dependence from Gd diffusion
- Effect on low-T FMR linewidth
- ISHE correlates with interface quality

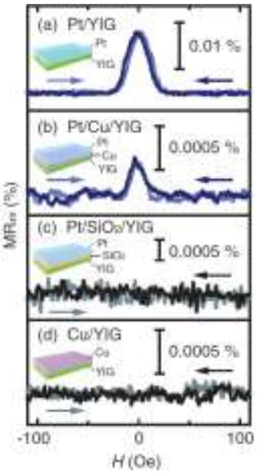
- Probably not the full story…



# Spin Hall Magnetoresistance (SMR)

- SHE
  - Conversion of electric current into a transverse spin current
  - Generates spin current and accumulation
- How can we control/observe this?
  - Ferr(i)omagnetic insulator
  - Interfacial spin mixing
  - Pt film resistance depends on the YIG orientation
- SMR
  - $\rho_{\parallel} > \rho_{\perp}$

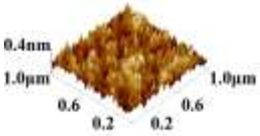


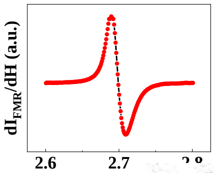


Nakayama, H. *et al.* Spin Hall Magnetoresistance Induced by a Nonequilibrium Proximity Effect. *Phys. Rev. Lett.* **110**, 1–5 (2013).

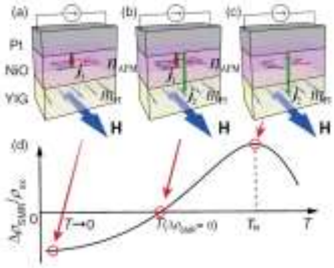
# GGG/YIG(15)/NiO(2)/Pt(4nm)

- PLD
- RT, 9.7798GHz
- Resonance field comparable to bulk
- M = 145emu/cm$^3$



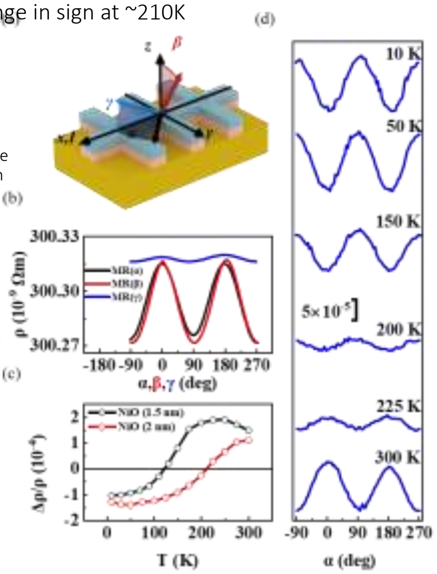


- Expect +ve SMR
  - Characteristic temperature (below T$_{Néel}$)
  - Changes sign at low temperature
    - Spin flip scattering→Inversion of Je$_{add}$ *PRL* **118**, 067202 (2017)
    - **Spin flop coupling** *PRL* **118**, 147202 (2017)
      - Spin axis of NiO ⊥M$_{YIG}$



- +ve SMR@RT
- Change in sign at ~210K

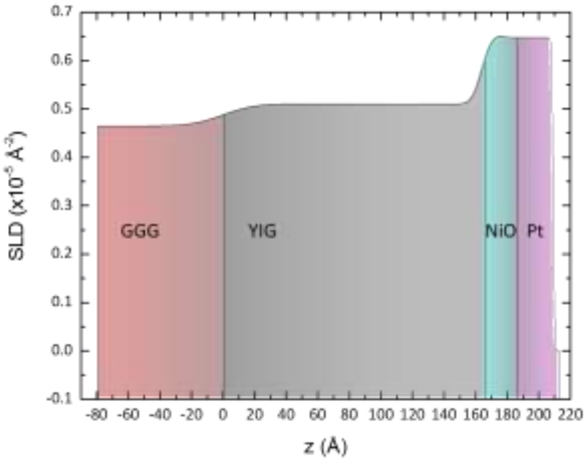
Hou, D. *et al.* Tunable Sign Change of Spin Hall Magnetoresistance in Pt/NiO/YIG Structures. *Phys. Rev. Lett.* **118**, 147202 (2017).



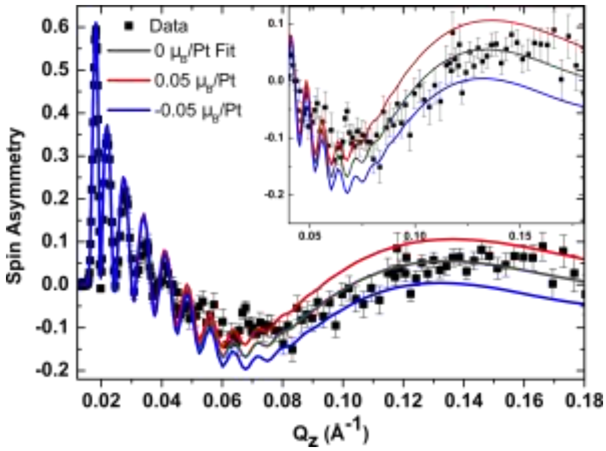
# Structural Profile

- Structural profile from PNR
- Pt (4 nm)/NiO (2 nm)/YIG/GGG
- Agreement with XRR & AFM

- Evidence for uncompensated moment?

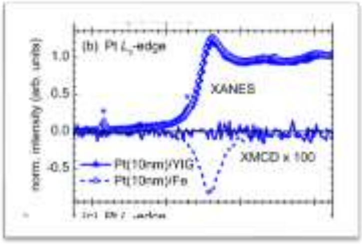


# Induced Pt moment (proximity/accumulation)



- Spin accumulation at the interfaces
  - Sensitivity to <0.05$\mu_B$/Pt
  - Less than 0.02$\mu_B$/Pt (within 1$\sigma$)
  - XMCD: 0.003$\mu_B$ averaged

S. Gebrägs *et al.* Appl. Phys. Lett. **101**, 262407 (2012);



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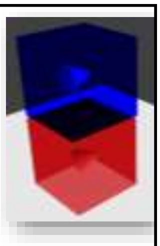
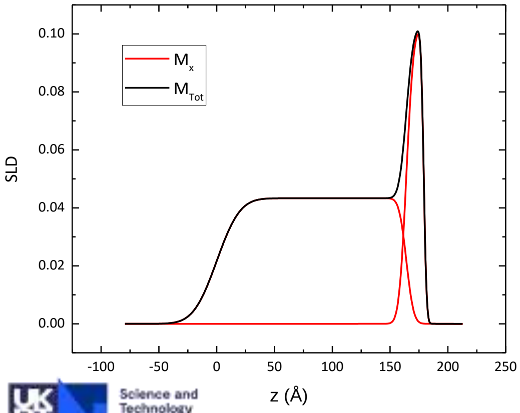
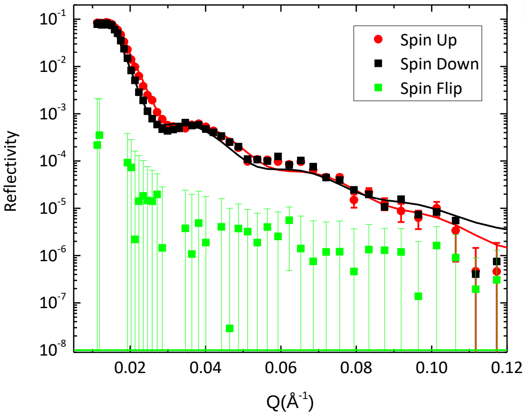
# Parallel Alignment



- Does not describe SF data





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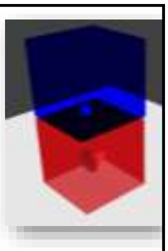
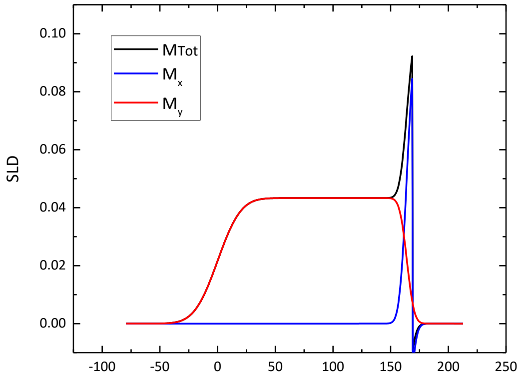
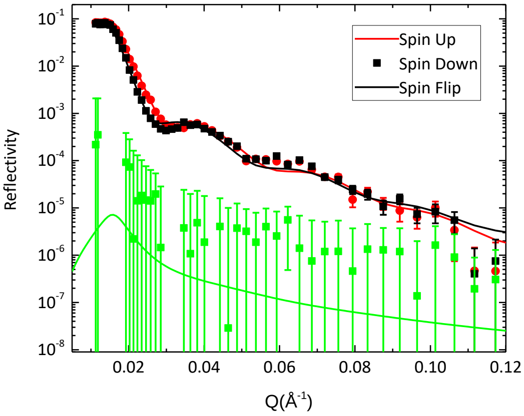
# Interfacial NiO layer



- Promising…





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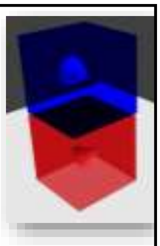
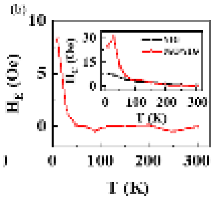
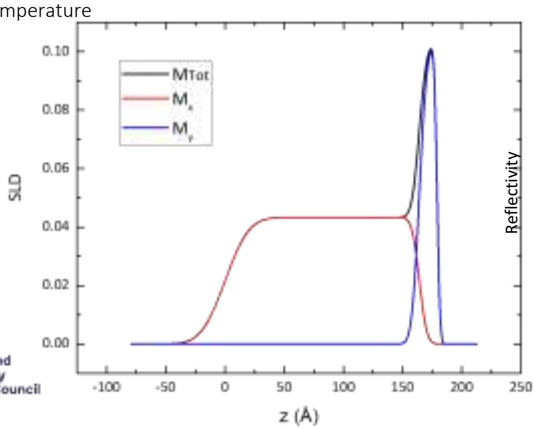
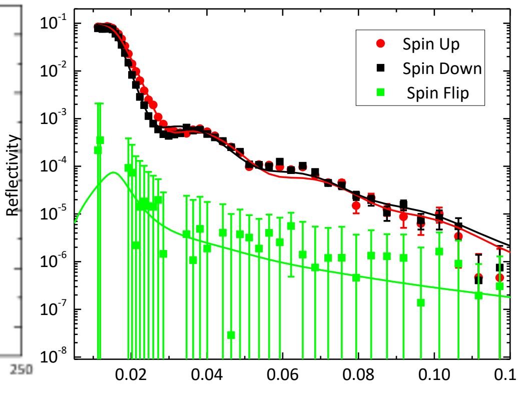


- Pt (4 nm)/NiO (2 nm)/YIG/GGG
- Significant Spin flip scattering
- NiO Quantization axis orthogonal to YIG
- Uncompensated moment in NiO
  - Domain state model
  - EB at low temperature







Nowak, U. et al. Phys. Rev. B **66**, 014430 (2002)

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# AFI Summary & Conclusions

- Unusual magnetisation temperature dependence arising from Gd diffusion
- Effect on low-T FMR linewidth
- Unambiguous evidence for orthogonal coupling of NiO to YIG: Spin-flop coupling responsible for negative SMR
  - Temperature independent
  - Exchange Bias

Mitra, A. *et al.* Interfacial Origin of the Magnetisation Suppression of Thin Film Yttrium Iron Garnet. *Sci. Rep.* **7**, 11774 (2017).

T. Zhu et al. *Interfacial coupling and negative spin Hall magnetoresistance in Pt/NiO/YIG. Appl. Phys. Lett.* ***113****, 072406 (2018).*

- What is happening in the thinnest YIG films
- Proximity to TI
- Alternatives to YIG

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# Chiral Magnetism

- Porter, N. A. et al. Manipulation of the spin helix in FeGe thin films and FeGe/Fe multilayers. Phys. Rev. B **92**, 144402 (2015)
- Spencer, C. S. et al. Helical magnetic structure and the anomalous and topological Hall effects in epitaxial B20 $Fe_{1-y}Co_yGe$ films. Phys. Rev. B **97**, 214406 (2018).

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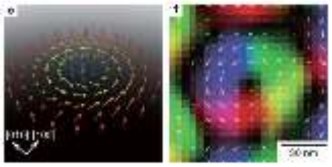
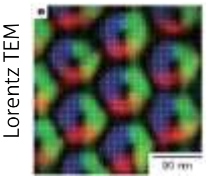
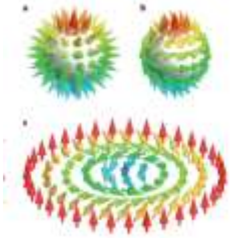
# Spin Textures



simulation Lorentz TEM

Skyrmion crystal



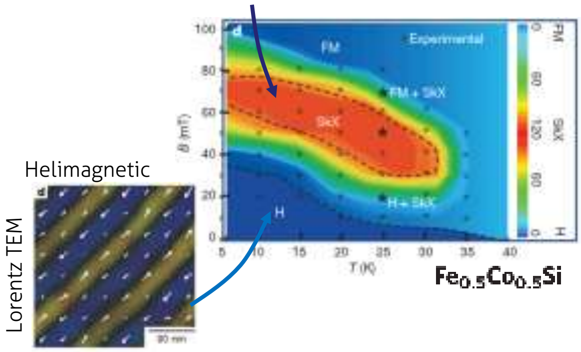


For bulk expect a helical magnetic ground state

In out of plane magnetic field obtain hexagonal skyrmion crystal lattice

Skyrmion is topologically stable soliton, here (x = 0.5) ~90 nm in diameter.

Helimagnetic



**Fe$_{0.5}$Co$_{0.5}$Si**

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X. Z. Yu et al., Nature **465**, 901 (2010).
C. Pfleiderer, Nature Physics **7**, 673 (2011).

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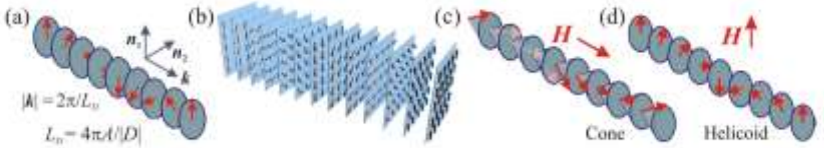
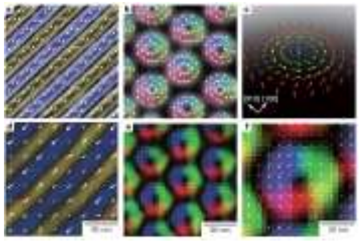
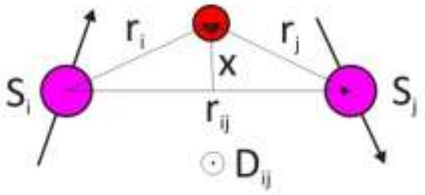
- Can be created by
  - Competition of anisotropies
  - Relativistic DM Interaction (DMI)
  - Frustration



Ler, U. K. R., Leonov, A. a. & Bogdanov, A. N. Chiral Skyrmionic matter in non-centrosymmetric magnets. J. Phys. Conf. Ser. **303**, 012105 (2011)



Yu, X. Z. et al. Real-space observation of a two-dimensional skyrmion Nature **465**, 901–904 (2010)



- $H = \boldsymbol{D}_{ij} \cdot (\boldsymbol{S}_i \times \boldsymbol{S}_j)$

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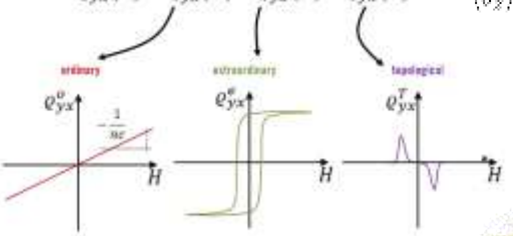
Fert, A., Cros, V. & Sampaio, J.. Nat. Nanotechnol. **8**, 152–156 (2013).

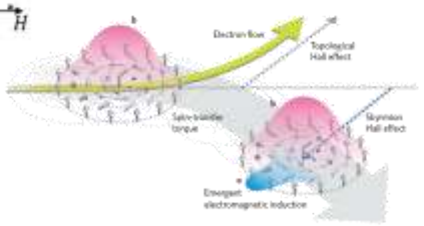
# Topological Hall Effect

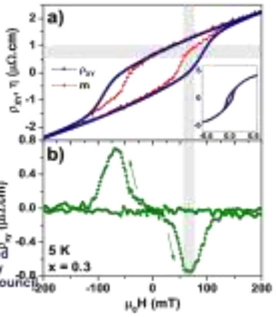
$$\varrho_{yx}(H) = \varrho^{o}_{yx}(H) + \varrho^{e}_{yx}(H) + \varrho^{T}_{yx}(H)$$



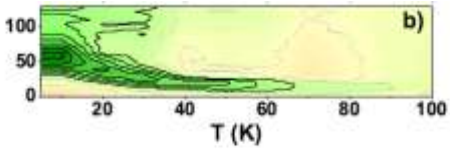
$$\langle b_z \rangle = \frac{\sqrt{3}\phi_0}{2\lambda^2}$$

$$n = \frac{1}{4\pi}\hat{M}\cdot\frac{\partial\hat{M}}{\partial x}\times\frac{\partial\hat{M}}{\partial y}$$





ρ$^{T}$ = 820nΩcm



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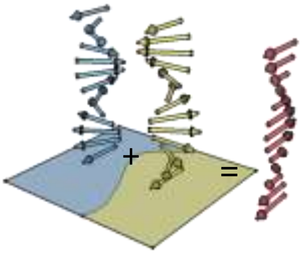
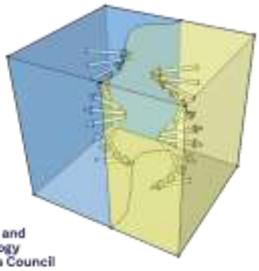
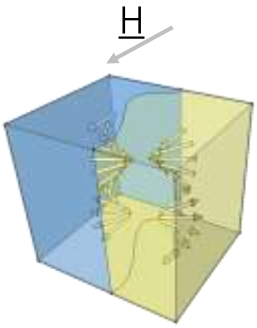
# Neutron scattering - field cooling

If we zero field cool our system the domains will point in any direction. The net moment is zero so expect no magnetic scattering.

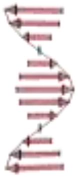
If we field cool our system helices are aligned. Opposite chiral helices will be aligned.







Net sum of the helices of opposite chirality is spin density wave



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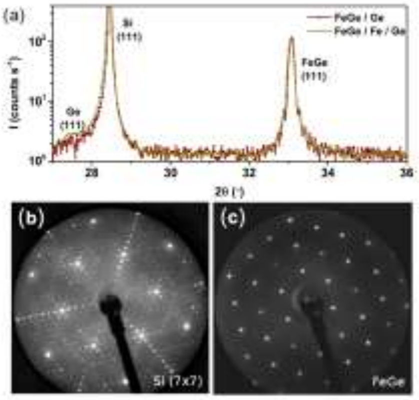
Wilson, M. N. *et al.* *Phys. Rev. B* **88**, 214420 (2013).

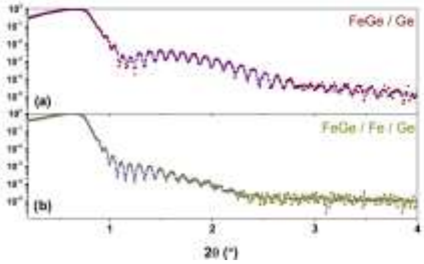
## Exchange coupling to a Ferromagnet

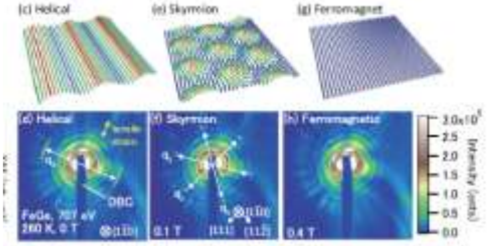
Controlling the helix

## Epilayer Characterisation

- $T_C$ ~
- 70n



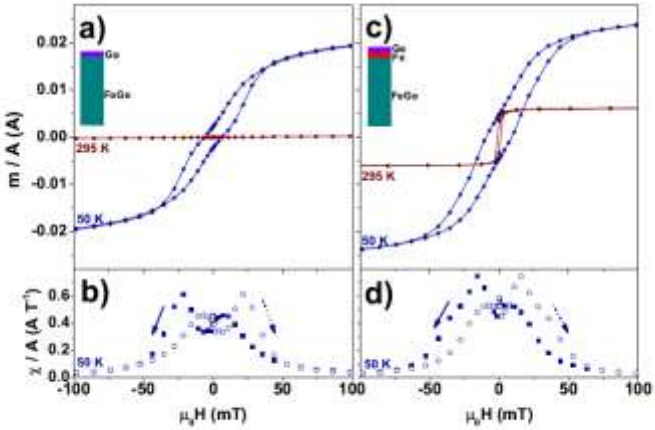




Yamasaki Y et al. (2015) Phys. Rev. B **92** 220421

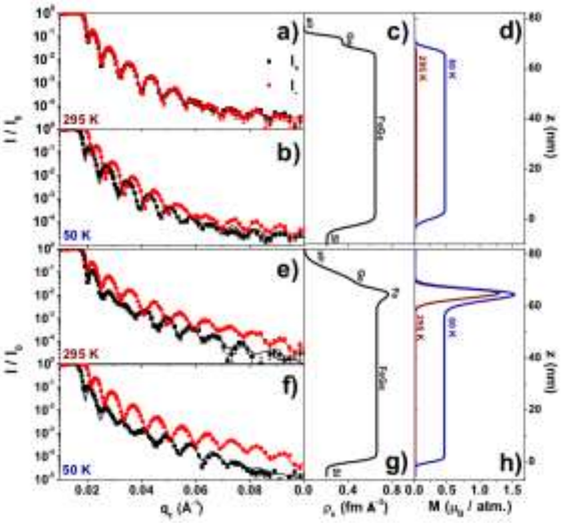
## Manipulation of the spin helix: FeGe/Fe

- Two step helix distortion
- Strong interfacial coupling for Fe/FeGe
- $\chi = \mu\, dm/dH$
- Differing switching mechanism
- Wilson, M. N. *et al.* Discrete helicoidal states in chiral magnetic thin films. *Phys. Rev. B* **88,** 214420 (2013)



| Sample | [illegible] | $t_{FeGe}$ (nm) | $t_{Fe}$ (nm) | $t_{Ge}$ (nm) |
|---|---|---|---|---|
| FeGe/Ge | 469.38 ± 0.03 | 67.8 ± 0.1 | - | 4.77 ± 0.07 |
| FeGe/Fe/Ge | 467.58 ± 0.03 | 64.2 ± 0.4 | 5.4 ± 0.2 | 2.1 ± 0.1 |

- FeGe behaves similarly with temperature
- Can ‘switch off’ FeGe with temperature
- 0.025$\mu_B$/atom effective paramagnetic moment!



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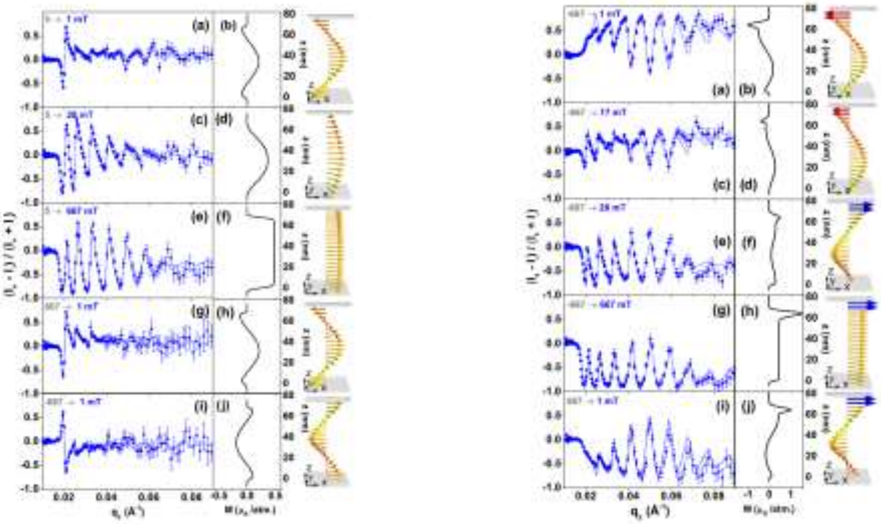

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$$M = M_0 + M_1 \sin\left(\frac{2\pi z}{\Lambda} + \phi_0\right) + M_2 \cos^2\left(\frac{2\pi z}{\Lambda} + \phi_0\right) \quad (1)$$

# Summary

- A controllable system
  - Doping
  - Planar
  - Grown on Si
- Demonstrated the behaviour of the helix under different boundary conditions
- $t < \Lambda$ Antinode in the centre of the layer
- Field pushes the nodes out to the interfaces and gives a peak in $\chi$
- Weaker peak are the nodes reentering the film
- Fe layer breaks the symmetry of the helicoidal state
- Prevents a node exiting via the top interface
- SANS investigation desirable

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# doped-FeRh

A Magnetic controllable interface

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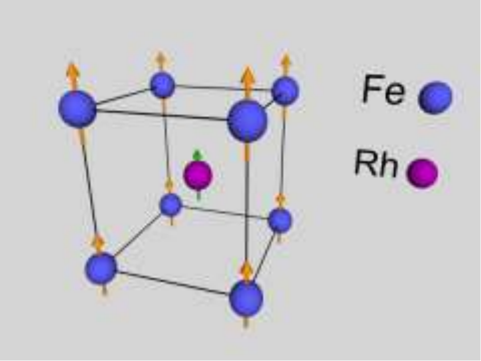

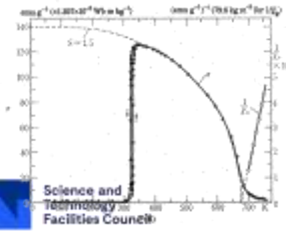

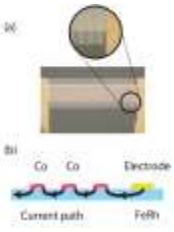

D. Arena, BNL

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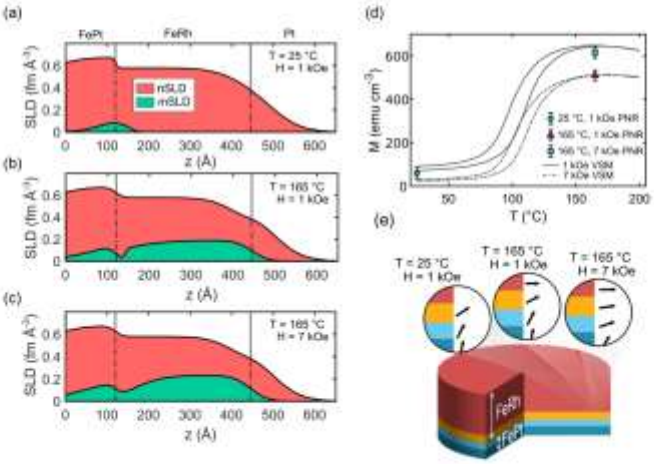
- CsCl structure
  - a~2.99Å
  - α' phase
- 300K: Type G AF
- Fe: ~3.3 $\mu_B$
- Rh: no moment
- FM alignment within <111> planes
- AF alignment between <111> planes
- 350 K: AF → FM
- Fe: ~3.1 $\mu_B$
- Rh: ~1 $\mu_B$

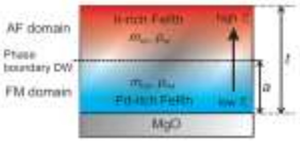
- J. S. Kouvel and C. C. Hartelius, JAP, **33**, 1343 (1962).
- G. Shirane *et al.* Phys. Rev. **134**, A1547 (1964)
- T. Naito et al., J Appl Phys **109**, 07C911 (2011)
- R. Fan, SL et al. Phys Rev B **82**, 184418 (2010).
- Lewis, L. H., Marrows, C. H. & SL. Coupled magnetic, structural, and electronic phase transitions in FeRh. *J. Phys. D. Appl. Phys.* **49**, 323002 (2016).

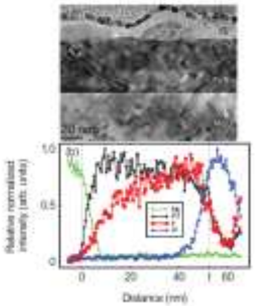
# **Motivation:** Tuneable magnetostructural phase transition

- Transition cont
  - Temperature
  - Field (H)
  - *Doping – Pd, I*
  - Strain - R. Fan
  - Pressure – S. Y
- Possible to crea

Basis for a field/ter







10.1103/PhysRevMaterials.6.024403

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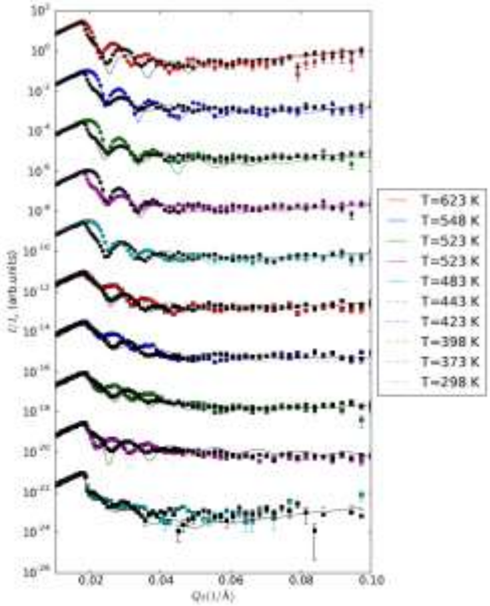
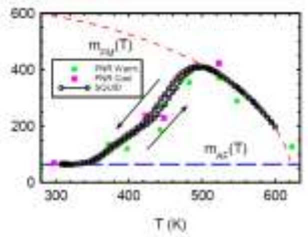
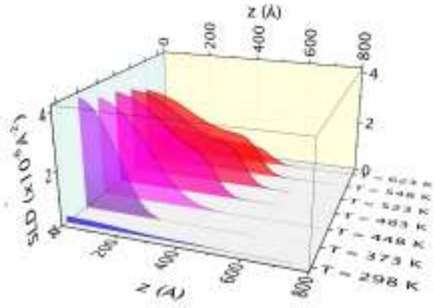
# Neutron Measurements

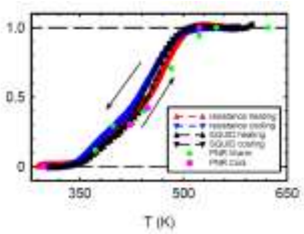
- Saturating Field
- Constrained two layer model
  - The minimum to describe the data



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







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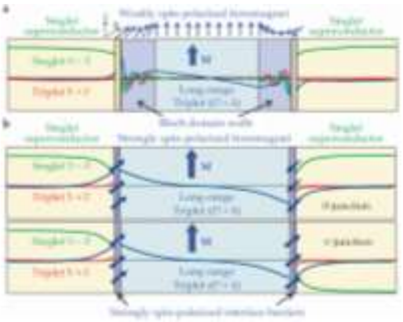
# Towards Superconducting Spintronics

- *Controllable Generation of spin-triplets*



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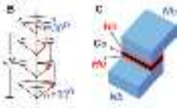
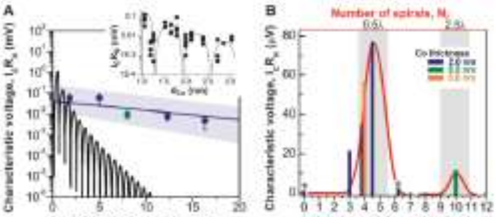
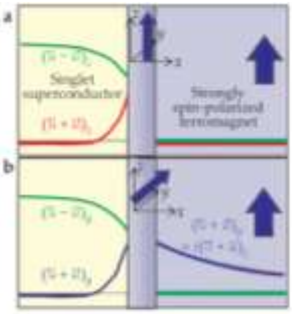
M. Eschrig Physics Today (2011)

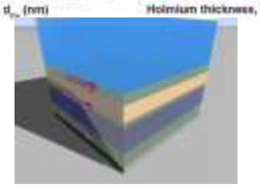
# Generating a triplet state

- 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









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ISIS Neutron and Muon Source

- Robinson *et al. Science* **329**, 59 (2010)
- Banerjee *et al.* Nat. Commun. **5**, 1 (2014).
- M. Eschrig Physics Today (2011)

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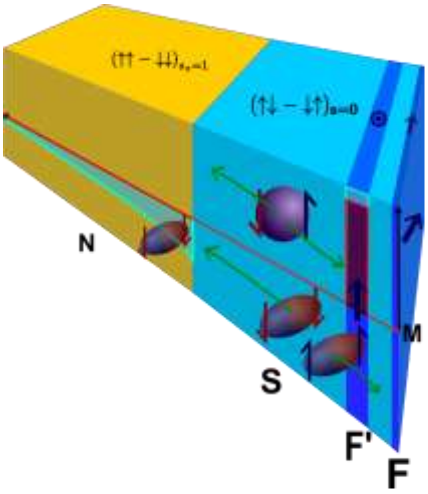
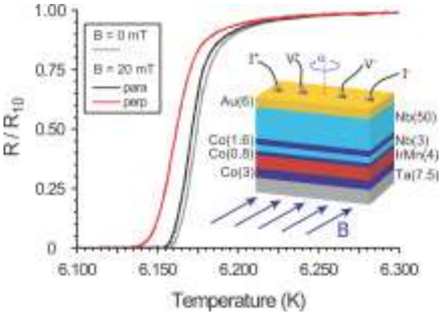
# Generating a triplet state





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- F. S. Bergeret, A. F. Volkov, and K. B. Efetov, Rev. Mod. Phys. **77**, 1321 (2005)
- M. G. Flokstra, **CJK, JFKC, SL**, *et al.* Phys. Rev. B **060501(R)**, (2015)

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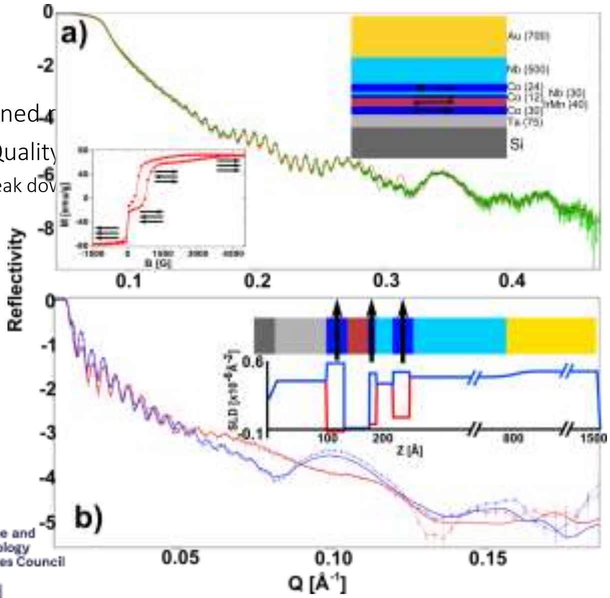
- Combined r
- High Quality
  - Break dov



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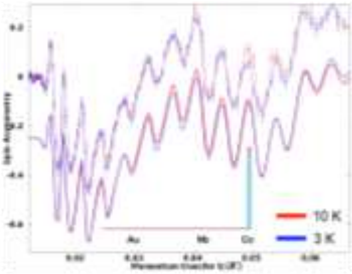
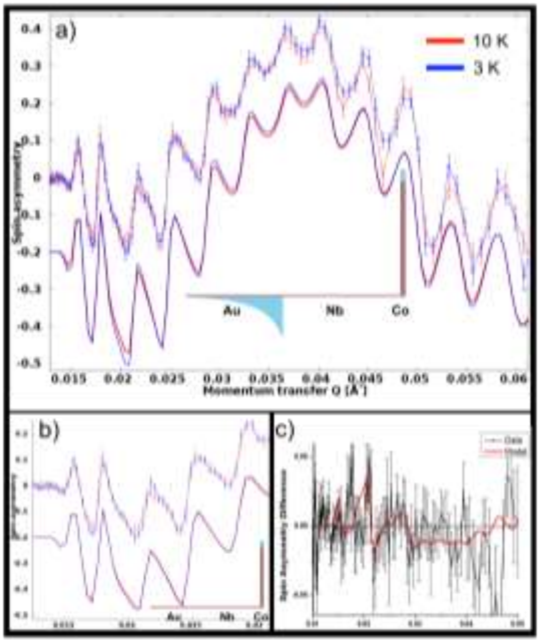
- Negative moment in the Au layer
- ~0.1$\mu_b$ change in Co

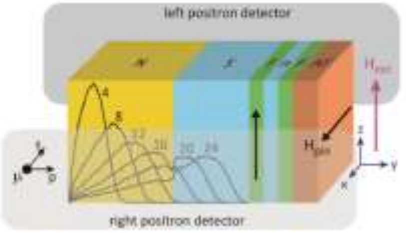


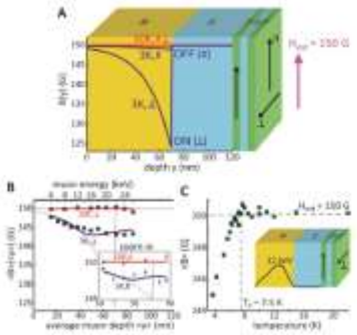


M. G. Flokstra, **CJK, JFKC, SL**, *et al.* Phys. Rev. B **060501(R)**, (2015)

## Remotely Induced Magnetism from a superconducting spin valve

- Singh, A., Voltan, S., Lahabi, K. & Aarts, J. Colossal Proximity Effect in a Superconducting Triplet Spin Valve Based on the Half-Metallic Ferromagnet $CrO_2$. *Phys. Rev. X* **5,** 021019 (2015).
- Leksin, P. V *et al.* Superconducting spin valve effect and triplet superconductivity in $CoO_x$/Fe1/Cu/Fe2/Cu/Pb multilayer. *arXiv* 1–14 (2015).





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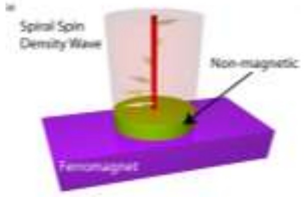
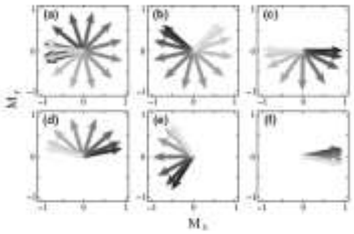
- M. G. Flokstra, **CJK, JFKC, SL**, *et al.* 10.1038/nphys3486 (2016)

## 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)

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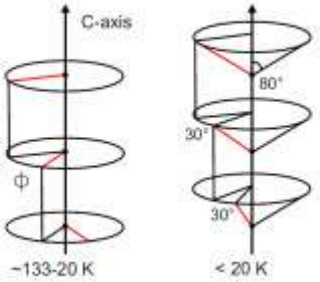
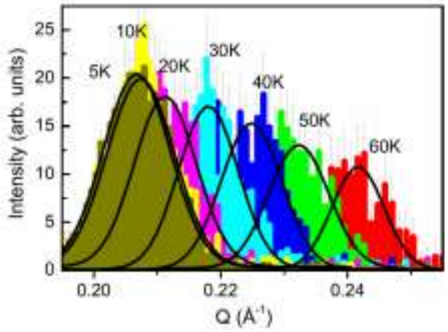
# Magnetic Spirals in Holmium thin films





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.

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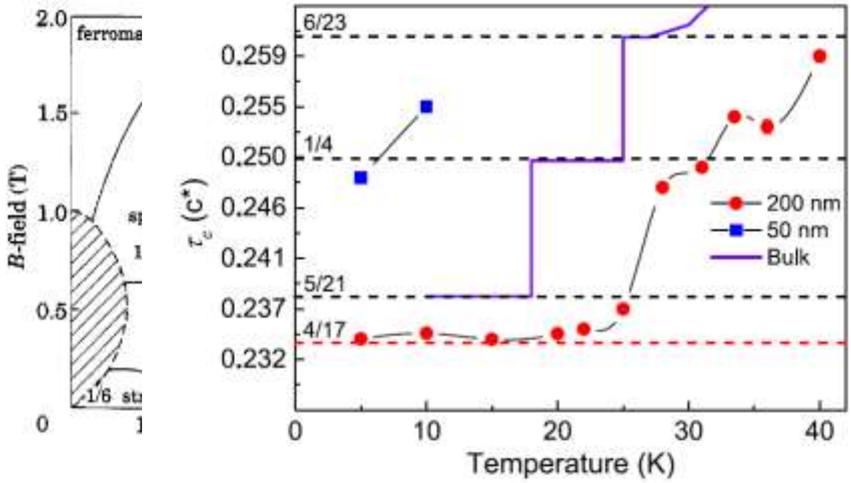
# Bulk Ho Field Phase diagram



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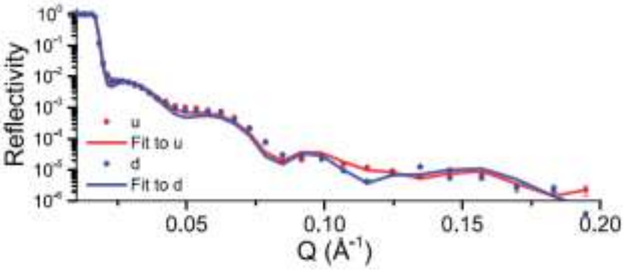
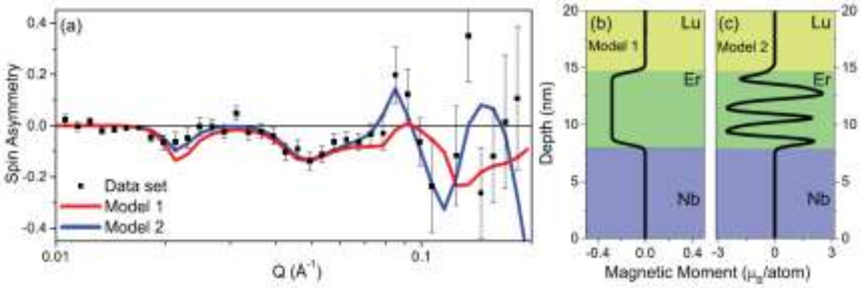
D. A Jehan *et al.*, Europhys. Letts. 17 553 (1992)

# Thin film Er

- Witt, J. D. S. *et al.* Magnetic Phases of Sputter Deposited Thin-Film Erbium. *Sci. Rep.* **6,** 39021 (2016).
- Satchell, N. *et al.* Probing the spiral magnetic phase in 6 nm textured erbium using polarised neutron reflectometry. *J. Phys. Condens. Matter* **29,** 55801 (2017).





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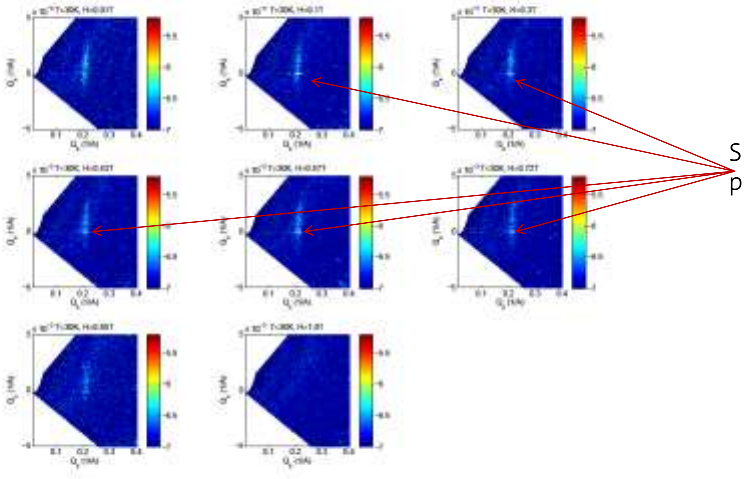
# Polref: Diffuse scattering from a holmium spin spiral: 7.5T



Spin Spiral peak

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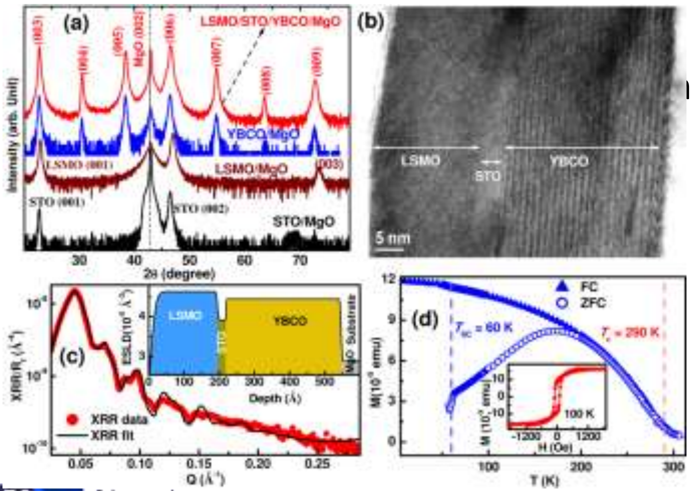
J. D. S. Witt et al. J Phys: Condens Matter **23**, 416006 (2011).

# Half metal-Insulator-d-wave superconductor



ıge of systems (tunnelling

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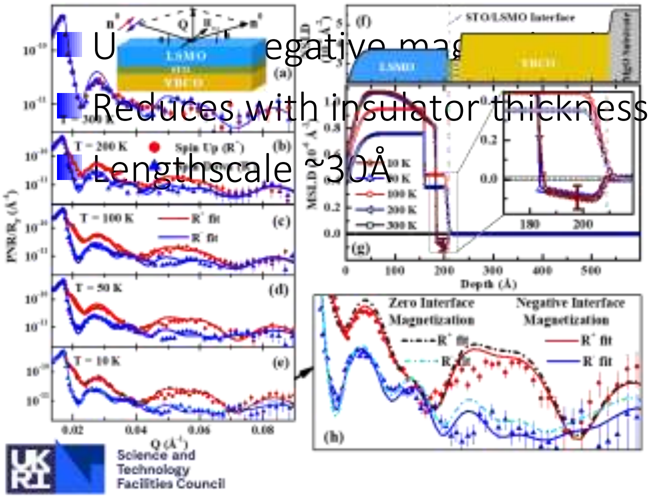

- U egative mag observed
- Reduces with insulator thickness
- Lengthscale ~30Å

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S. Singh *et al.* Under Review

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# PNR Summary

- Spin Valve
  - Length scale of induced magnetisation too long for inverse proximity.
    - Flokstra PRB 89 054510
  - Profile is not the parabola shape expected for Meissner screening.
    - Khaydukov arXiv:1405.0242
  - Induced moment in the Au layer has to be a result of triplet formation.
  - We therefore conclude that as with the muon result this induced moment is as a result of the triplet superconducting state
- Half-metal/d-wave
  - Large length-scale effect: possible triplet
  - Unusual tunneling driven proximity effect

# Small Angle Scattering

- Grazing Incidence to give depth selectivity
- Difference gives the interference term

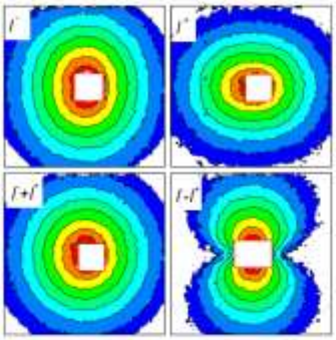

**Figure 1**
SANSPOL patterns in $Fe_3O_4$ for neutron spins antiparallel ($I^-$) and parallel ($I^+$) to the horizontal field. The arithmetic mean $[(I^-) + (I^+)]/2$ corresponds to the 2D pattern of non-polarised neutrons. The difference $(I^-)-(I^+)$ yields the interference term [equation (1c)].

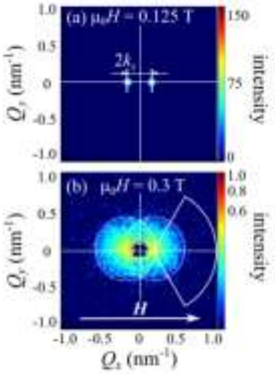

FIG. 1. Maps of the small-angle neutron scattering at $T = 15$ K for $Fe_{0.7}Co_{0.3}Si$: (a) in the conical phase at magnetic field $\mu_0 H = 0.125$ T, (b) in the induced ferromagnetic phase at magnetic field $\mu_0 H = 0.3$ T.

- A. Wiedenmann J. Appl. Cryst. (2000). 33, 428-432
- Grigoriev et al. Phys Rev B **100**, 094409 (2019)
- Mühlbauer, S. *et al.* Magnetic small-angle neutron scattering. Rev. Mod. Phys. **91**, 015004 (2019).

# Summary & Outlook

- The neutron possess a range of characteristics well matched to both ***curiosity driven*** and ***technologically*** relevant nanoscale research
  - Absolute, quantitative results provide a robust test of theory
  - From atomic and nano through to micro-macroscopic lengthscales
- Examples drawn from:
  - Ferromagnetic Insulators
  - Chiral Magnetism
  - Magnetocaloric
  - *Biomedical applications, Clean Energy*
- Frontier Research
  - Thin film quantum matter
  - Novel sample environment
  - Smaller Samples
  - Increasing connection with calculation/theory

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