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A Brief Introduction to Polarised Neutron Reflectivity

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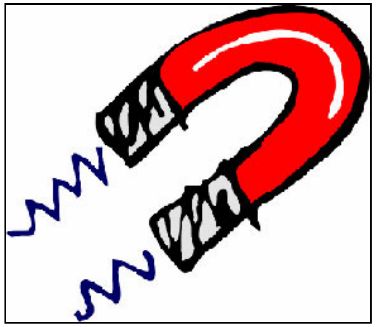

With almost constant reference to the published works of F. Ott, S. Sinha, R. Pynn, C. Majkrzak and D. Sivia

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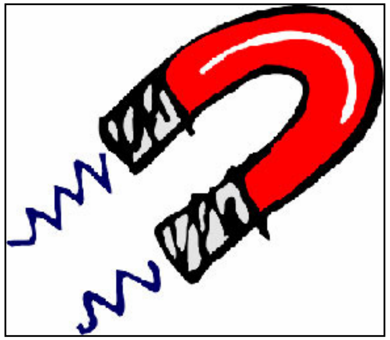
Polarised Neutron Reflectometry (PNR)

- NR measures the nuclear scattering length density profile.



- PNR measures the magnetic scattering lengthy density profile.

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Note - PNR is also called Half Polarised (half-pol)

What kind of scientific information can be obtained PNR:

- **Magnetometry (SQUID, VSM, MOKE):**
  - Measure the bulk magnetic response of a sample
  - No spatial information
- **Electrical Transport:**
  - Measures the conductive layers preferentially
  - Also no spatial information so very hard to interpret results beyond bulk response
- **Magnetic X-ray techniques (XMCD, XAS, XMLD, XRMS):**
  - Element specific, very high sensitivity.
  - **Results hard to interpret! (Spin-Photon interaction indirect).**
  - Light elements are difficult to measure.
  - Soft x-rays highly absorbing.
- **Microscopy (Electron, MOKE based, X-rays based):**
  - Surface sensitive only.
  - Sample environment limited.
- **Low Energy Muons:**
  - Poor depth sensitivity.
  - Difficulties large internal magnetic fields.
  - Fantastic sensitivity to very small magnetic moments.

- **PNR:**
  - Highly penetrating so can probe large volume. **Can probe buried interfaces.**
  - **Spin Neutron interaction direct. Results easy to interpret.**
  - **Strong magnetic scattering**
  - Contrast variation across the periodic table means light element easily measurable.
  - Isotope contrast variations available
  - Depth dependent magnetometer
  - Absolute moment
  - Vector magnetometer (in the plane of the sample)
  - Long coherence length
  - Low flux (relatively)
  - Needs big area samples

What kind of **PRACTICAL** scientific information can be obtained PNR:

Polarised Specular Reflectivity provides both the chemical/nuclear (isotopic) and the Magnetic scattering length density depth profile along the surface normal with a spatial resolution approaching half a nanometer (nm).

1. In NR you get three basic parameters assuming a box model is used to construct the SLD profile:

=> essentially **thickness, roughness, density (nSLD).**

2. In PNR these are joined by three more magnetic parameters:

=> essentially **magnetic thickness, magnetic roughness and magentic density (mSLD).**

a) Magnetic thickness: Magnetic dead layers, magnetic proximity effects, topological insulators.

b) Magnetic roughness: Canting, spirals, coupling, superconducting vortices.

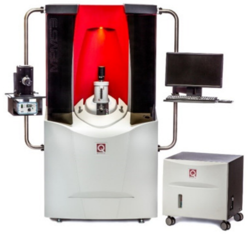
c) Magnetic Scattering Length Density: Total moment, interlayer coupling (RKKY AF coupled layers), inhomogeneities, magnetic transitions (AF/FM/P).

- Note: PNR is NOT sensitive to inter-atomic magnetic order like antiferromagnetism! You need diffraction for that.

Properties of Hysteresis loops (Terminology)

- Field (**H**) and Moment (**M**)

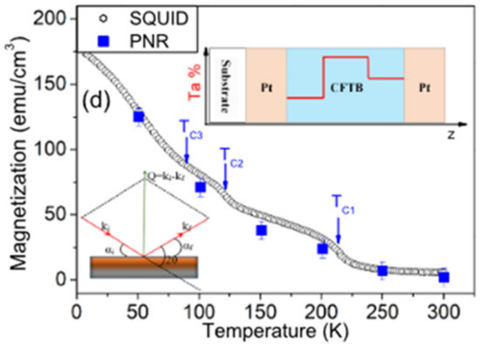
  SQUID, VSM (PNR/PA!)

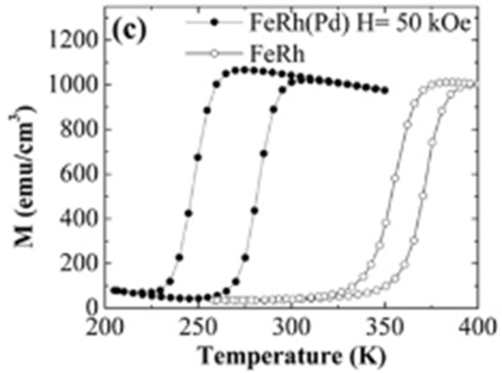


- Relative response to Field (**H**) only

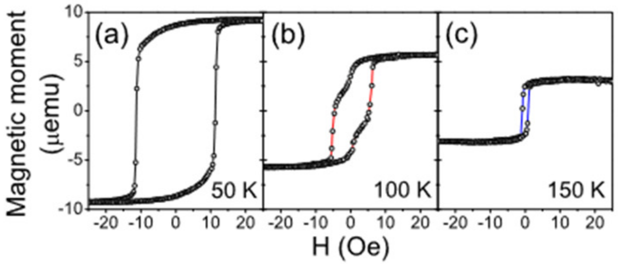
  MOKE, XMCD

- Magnetisation vs Temperature - (**M**vsT)





- Magnetisation vs Applied Field - (**M**vs**H**)



- The applied field and thermal histories are very important in magnetic materials.

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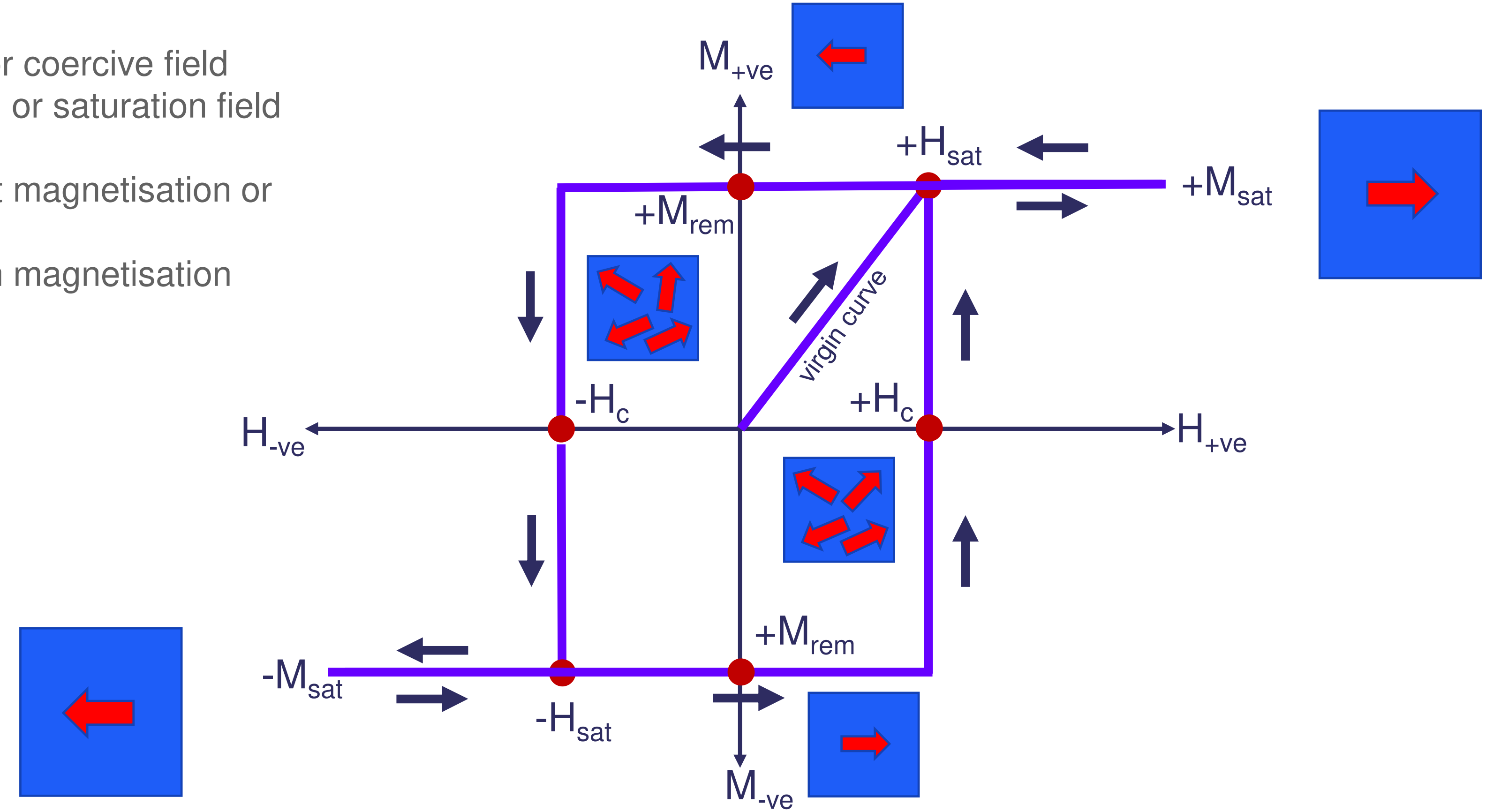
- C. J . Kinane *et al.* New Journal of Physics 16 (2014) 113073
- O Inyang *et al*, PRB 100, 174418 (2019)

Properties of Hysteresis loops (Terminology): Magnetic Hysteresis

H_c = Coercivity or coercive field
H_{Sat} = Saturation or saturation field

M_{Rem} = Remnant magnetisation or retentivity.
M_{sat} = Saturation magnetisation



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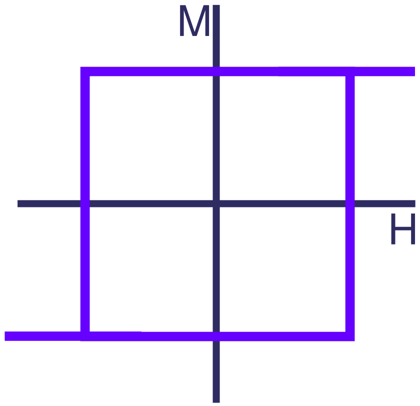
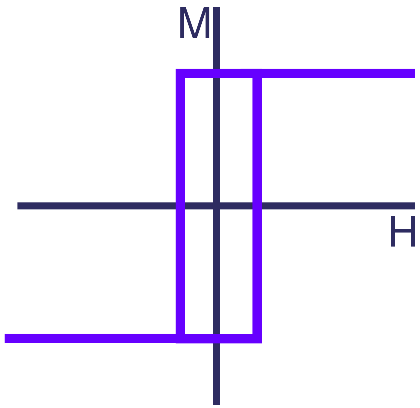
- The properties of a magnetic hysteresis loop are often thought of as scaler in nature (directionless) but in fact are vectorial. They apply to the direction the H field is applied in the sample.
- Hysteresis means the magnetic history of the sample is VERY important!

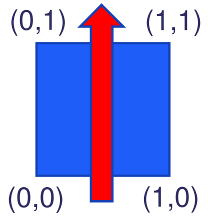
Properties of Hysteresis loops (Terminology):



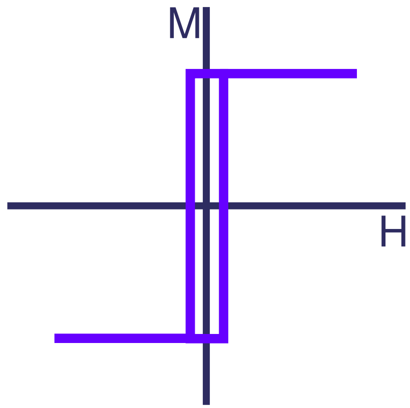
Hard Magnet



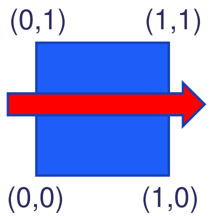
Soft Magnet



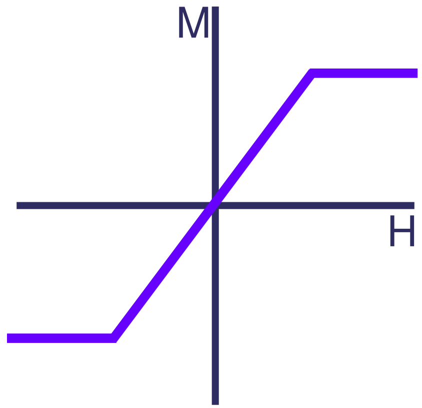
H field applied in the (0,1) direction



Easy Axis (EA)



H field applied in the (1,0) direction



Hard Axis (HA)

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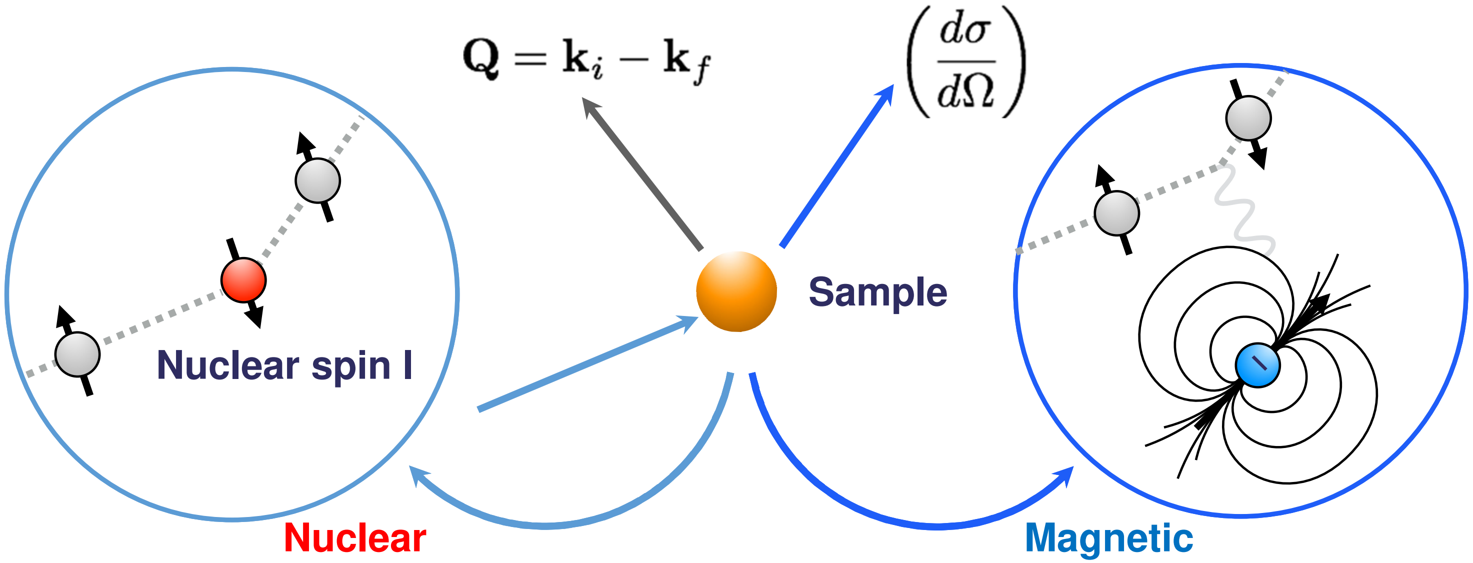
- Anisotropy has many forms – Magnetocrystalline, shape etc
- The direction that the H field is applied affects the values of H_c, H_{sat}, M_{rem}, M_{sat}
- Its very important to know the sample orientation vs the magnetic field!

Neutron-electron interaction:

The neutron has magnetic moment which can interact with sample moments:



Interaction is between dipolar fields of neutron and unpaired electron(s) → directional dependence.

Neutrons scatter magnetically for the B field of the magnetic material!

Strength of magnetic interaction similar to that of the neutron and nucleus (unlike X-rays).

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A neutron is a spin ½ particle with a magnetic dipole moment, effectively its a little bar magnet. (like a fridge magnet only smaller)

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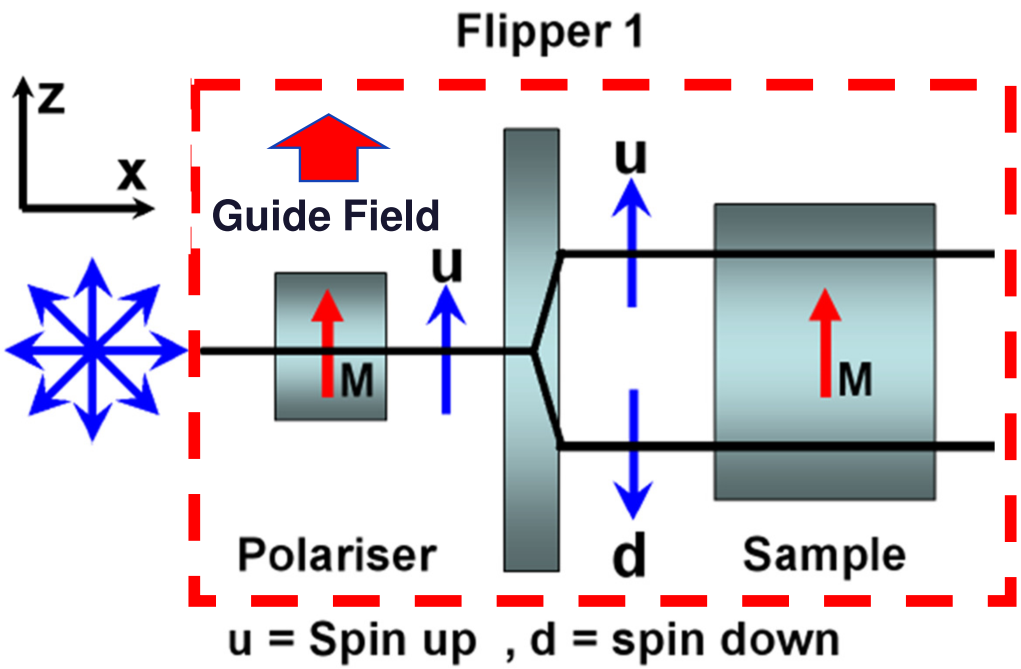
Thanks to G Nilsen for this slide

Polarised neutrons PNR or Half Polarised:

Unpolarised neutrons have no orientation



Now measure two reflectivity curves:

i) Spin up
ii) Spin down.

Experiments now takes 4 times as long to get similar statistics!

- There several ways of polarising and flipping neutrons, but that is beyond scope of this talk.
- It is the difference between the reflectivity curves of the two spin states that allow the magnetism to be probed.

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- W. Gavin Williams Polarized neutrons Oxford Science
- A.-J. Dianoux et al, Neutron Data Booklet (Institut Laue-Langevin, 2002), 1st ed.

PNR Terminology:

- In PNR we refer to measuring the spin states.
- These are referred to by a multitude of names but ultimately all the names are labels referring to the spin of the neutron being aligned “Parallel” or “Antiparallel” to the guide field. Labels include:
  - Parallel => Up, U, u, I^{+}, ↑, or just +
  - Antiparallel => Down, D, d, I^{-}, ↓, or just -
- It is important to note that in PNR the “Up” and “Down” states contain the Spin flip components as well. E.g. Up = UU + UD and Down = DD +DU but more on this later.
- Maintaining the guide field on a PNR beamline is VERY VERY important!

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Polarised Neutron Reflectivity (PNR):

- It is assumed that the polarisation vector and magnetisation are parallel (We set the beamline up so this is as true as possible)

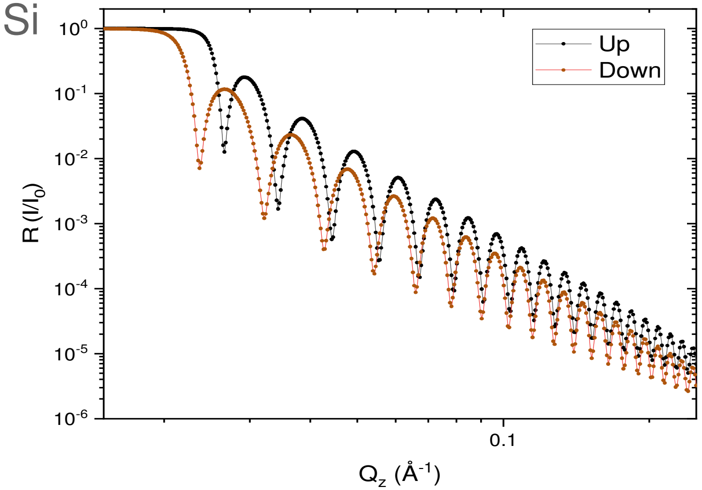
$$V = V_n \pm V_m \quad \text{where } V_n = \frac{2\pi\hbar^2}{m} Nb \text{ and } V_m = \frac{2\pi\hbar^2}{m} Np = \pm\mu_n B$$

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

For a single magnetic layer $$V = \frac{\hbar}{2\pi m} N(b_N \pm b_m)$$

- This essential means you get two reflectivity curves as the magnetic layer has two different values for its SLD depending it M is parallel or anti-parallel to the polarisation ***P*** direction established by the guide field.

- For example A 500 Å Ni layer on Si substrate



- Bland et al, Phys Rev B, 46, 3391 (**1992**)
- Zabel et al Physica B 276-278, 17 (**2000**)
- R. M. Moon et al Phys Rev, **1969**, 181, 920-931
- S. Blundell et al, JMMM, **1993**, 121, 185-188
- Bland et al, Phys Rev B, 46, 3391 (**1992**)
- G. L. Squires introduction to the Theory of Thermal neutron scattering
- Modern Techniques for Characterizing Magnetic Materials Edited by Yimei Zhu, Springer
- Neutron Scattering form Magnetic Materials, editor Tapan Chatterji, Elsevier

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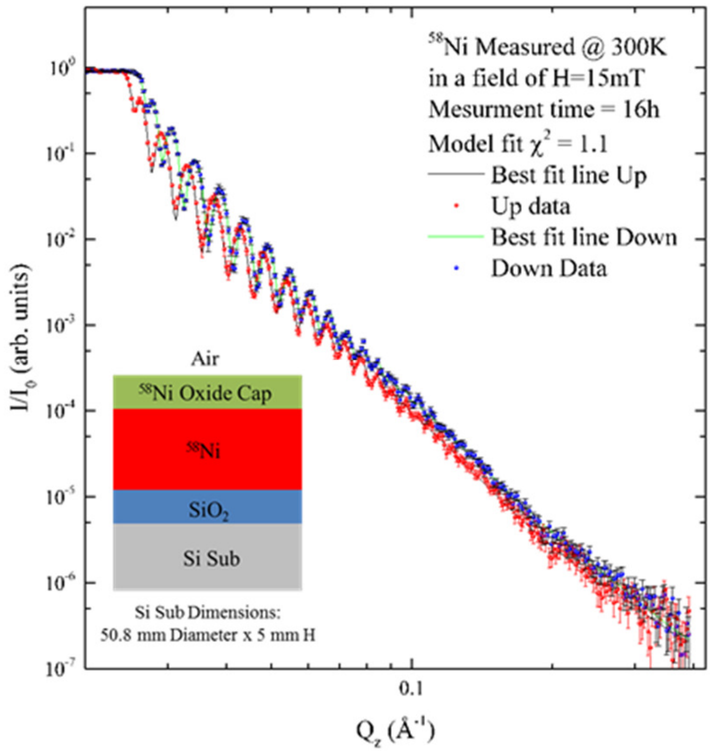
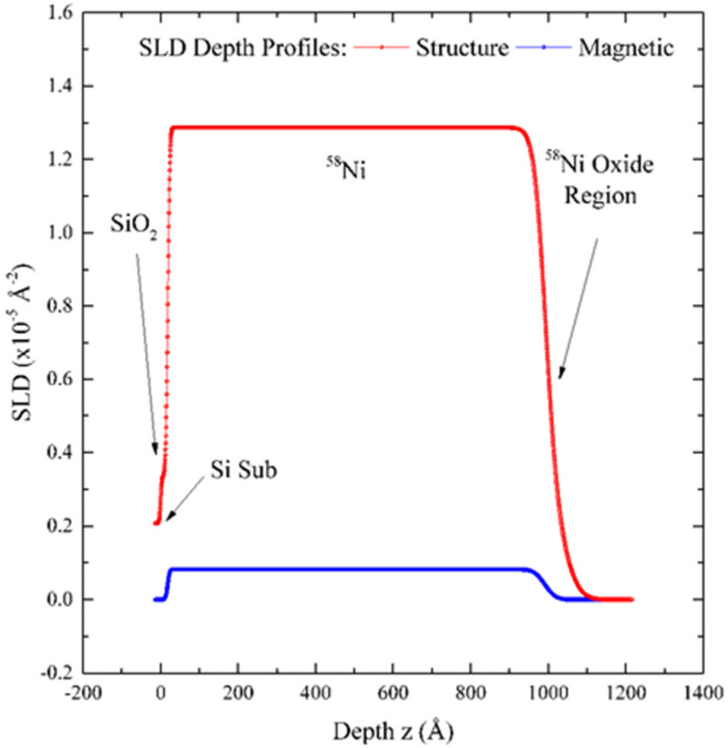
PNR: An example of PNR from a simple Ni film.





- PNR provides both the Nuclear (structural) and the magnetic SLD depth profile.
- Effectively functions as a depth dependent magnetometer
- But takes longer than NR by a factor 4 for similar statistics

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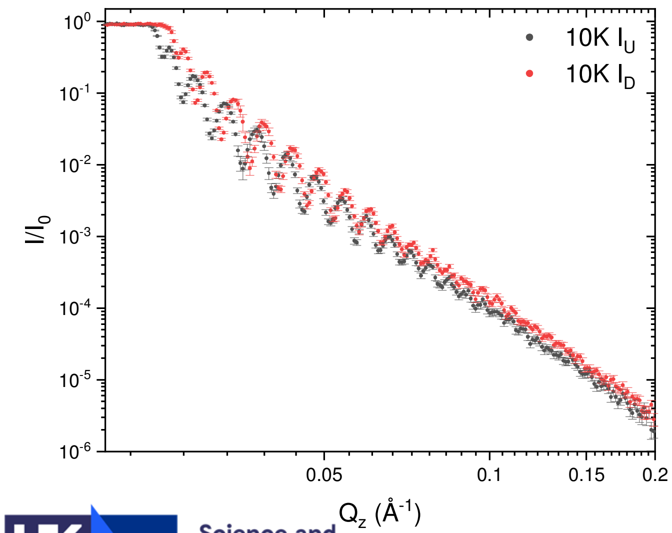
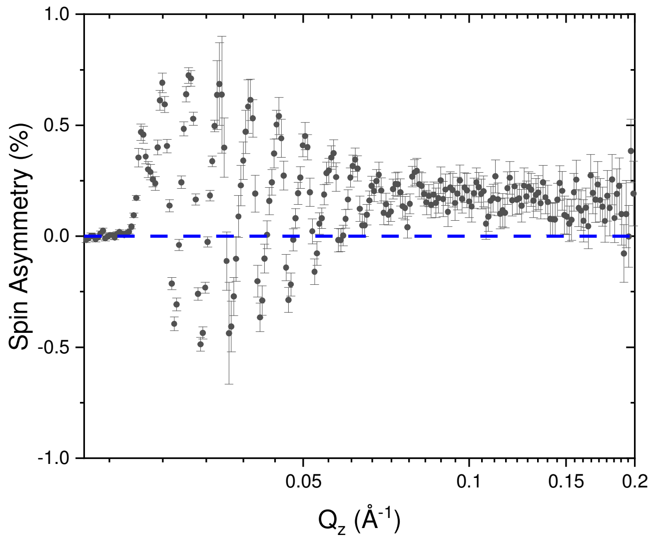
PNR: Spin Asymmetry

- This magnetization-induced splitting between the Up-Up and Down-Down reflectivity's contains information regarding the magnetic depth profile of the thin film structure.
- Although not universally true, it is often the case that a larger splitting between the two cross sections indicates a larger magnetization of the film.
- Although we will always fit the reflectivity data itself, it is often helpful to plot the **spin asymmetry (SA)** of the reflectivity, defined as (U-D)/(U+D), to better emphasize the magnetic features in the scattering, as shown below:



$$Spin\ Asymmetry = \frac{U - D}{U + D}$$



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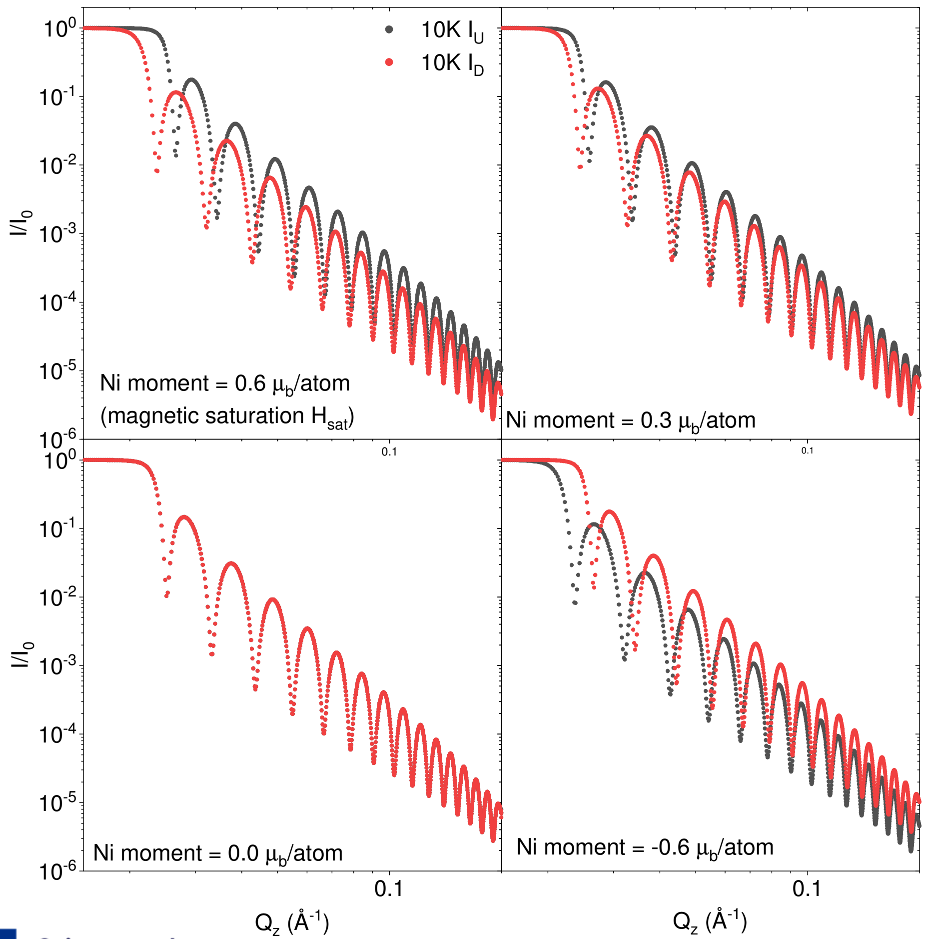
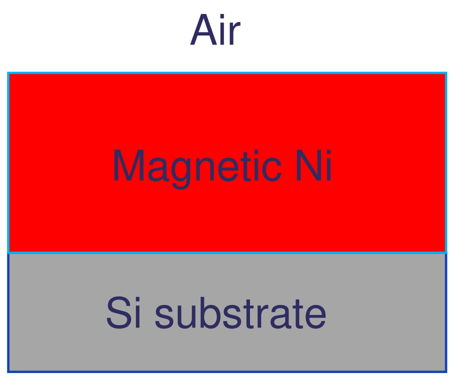
- This makes small magnetic changes a lot easier to see
- WARNING!!! Never fit the Spin Asymmetry exclusively on its own!!!!!

PNR Magnetic SLD: Magnetic saturation to Negative Magnetic Saturation





- The Ni layer is 500 Å thick with the Silicon substrate and top Ni interface roughness's set to 5Å rms (σ=5 Å).
- The edges of the magnetism are commensurate with the Ni nuclear structure, i.e. matching the thickness and roughness exactly.

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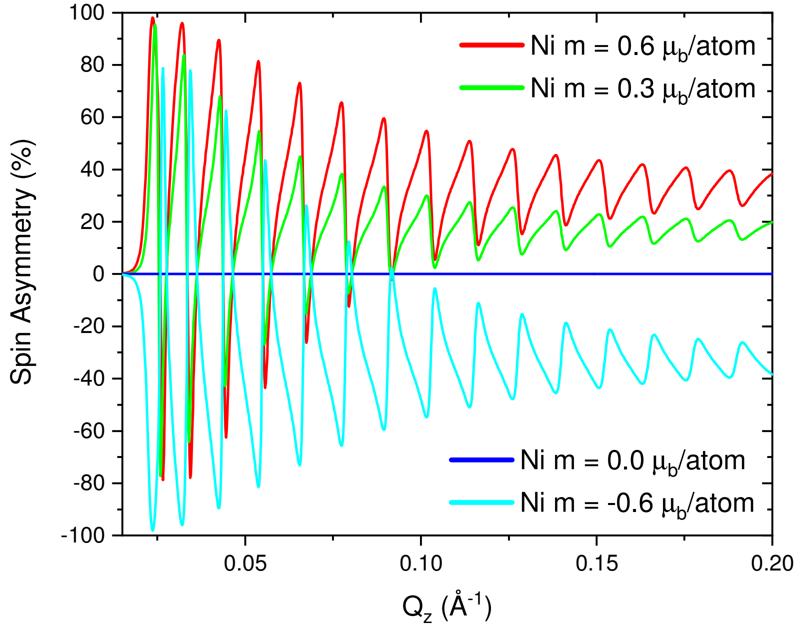
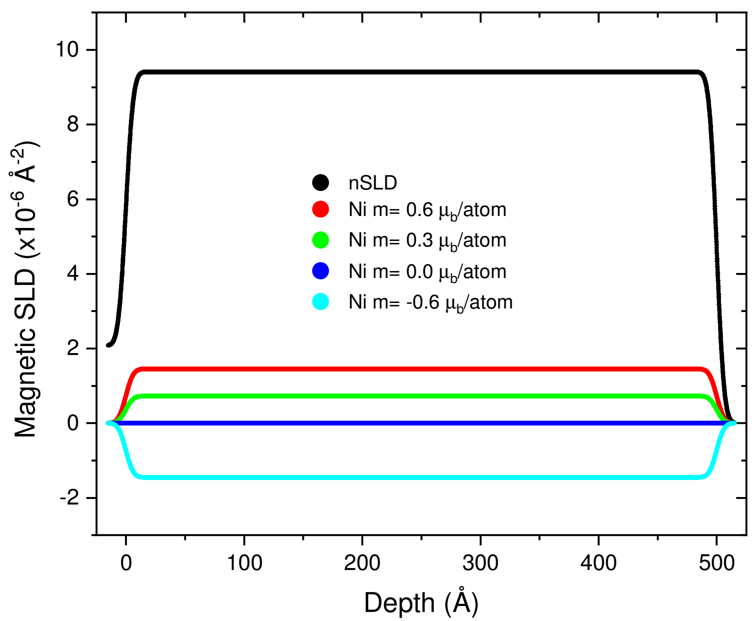
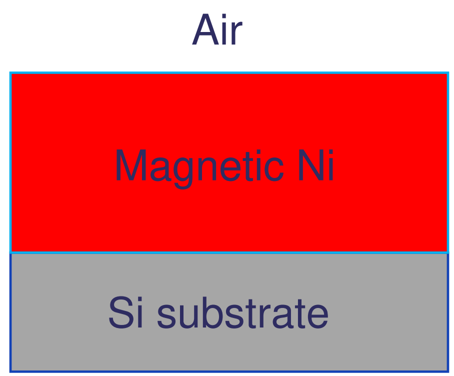
- Note the spin states have switched position for the negative magnetic moment -0.6μ_b/atom

PNR Magnetic SLD: Magnetic saturation to Negative Magnetic Saturation







- The Ni layer is 500 Å thick with the silicon and top interface roughness set to 5Å rms (σ=5 Å).
- It is much easier to see changes in the SA plot.
- The -0.6μ_b/atom SLD and SA are both flipped. This is the reverse saturated case the magnetisation of the sample is now antiparallel to the polarisation of the beamline.

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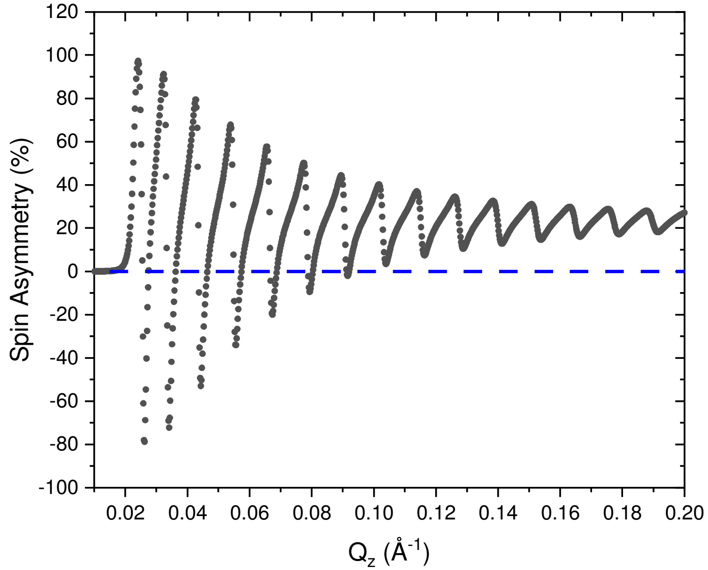
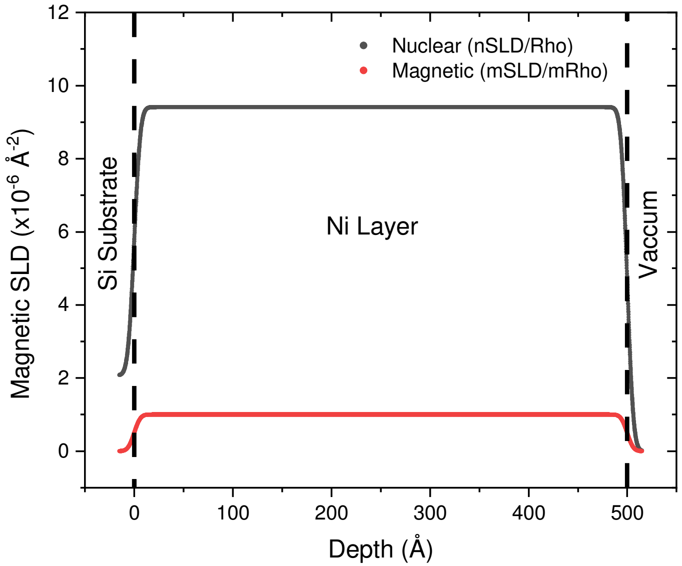
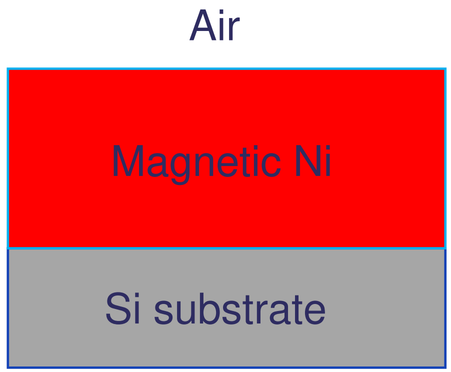
PNR :Magnetic Thickness: Commensurate Ni







- The Ni layer is 500 Å thick with the silicon and top interface roughness set to 5Å rms (σ=5 Å).
- The magnetism in the Ni is set to 0.6 μ_b/atom, the saturated case.
- The edges of the magnetism are commensurate with the Ni nuclear structure, matching the thickness.

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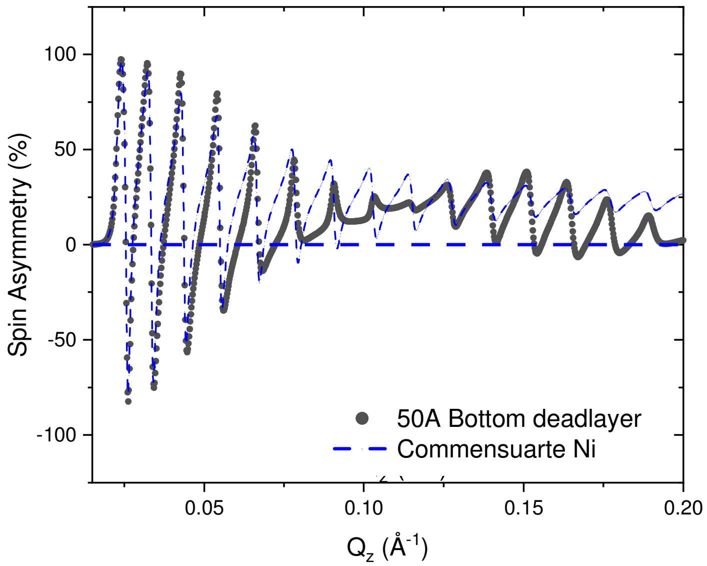
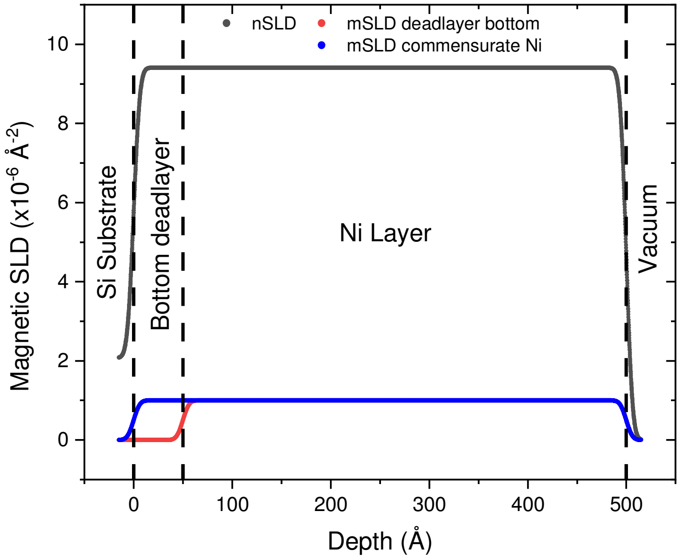
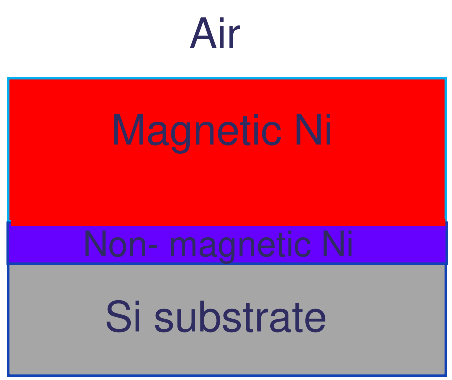
PNR Magnetic Thickness: Bottom magnetic dead-layer







- The Ni layer is 500 Å thick with the silicon and top interface roughness set to 5Å rms (σ=5 Å).
- 50 Å magnetic dead-layer at the bottom interface with the silicon substrate.
- The splitting in the PNR is modulated by a long range envelope function from the thinner dead-layer.

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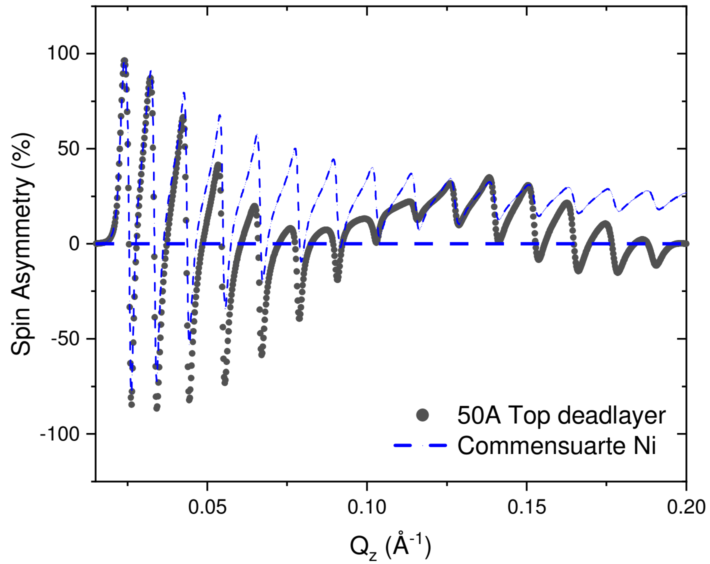
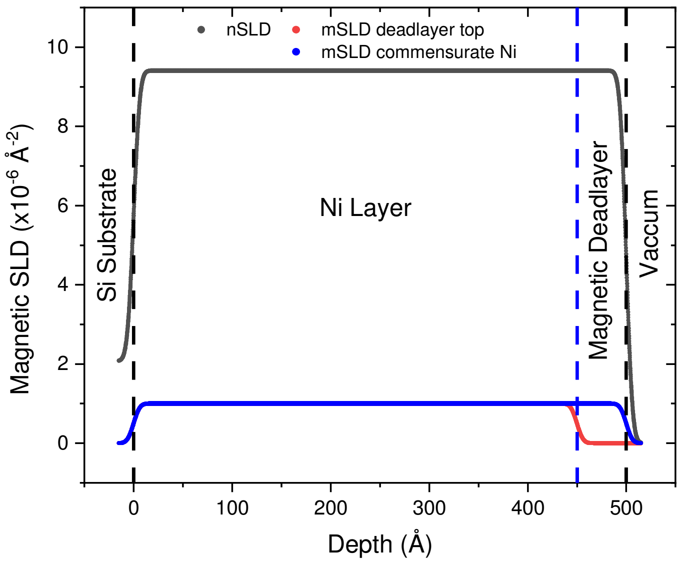
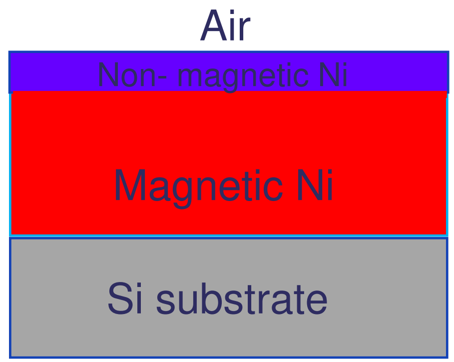
PNR Magnetic Thickness: Top magnetic dead-layer







- The Ni layer is 500 Å thick with the silicon and top interface roughness set to 5Å rms (σ=5 Å).
- 50 Å magnetic dead-layer at the top interface with the air.
- Easy to see using the SA that the envelope function shifts its modulation.

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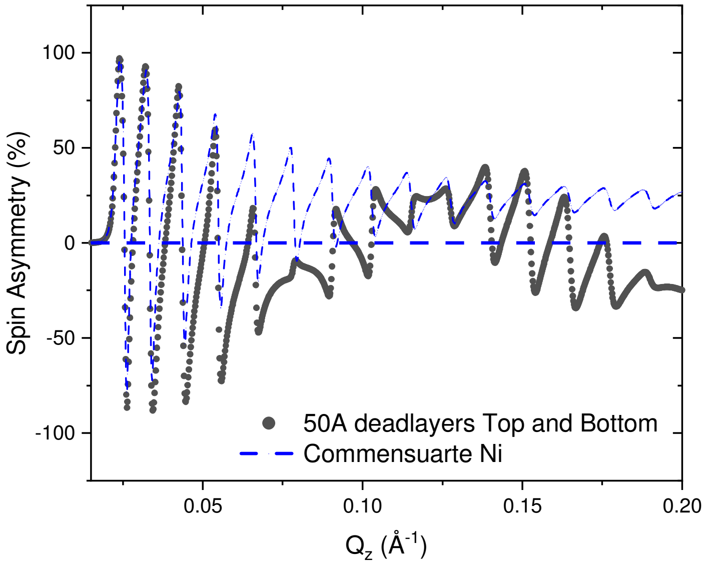
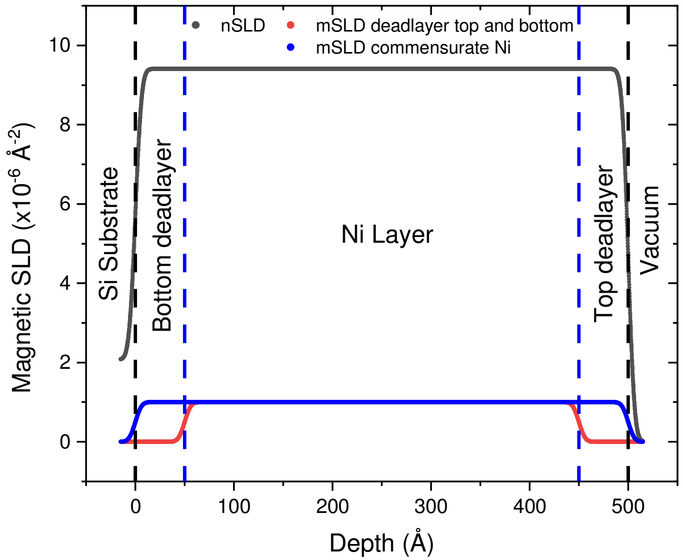
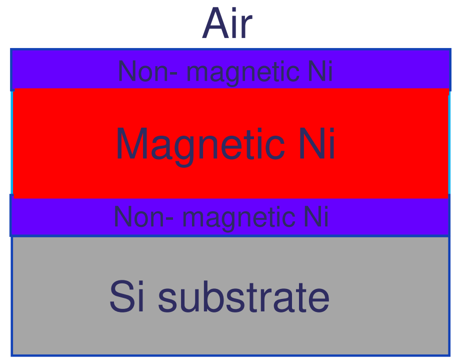
PNR Magnetic Thickness: Top and bottom dead-layers







- The Ni layer is 500 Å thick with Silicon and top interface roughness of 5Å (σ=5 Å).
- 50 Å magnetic dead-layer at the top and bottom interfaces with the silicon substrate and air respectively.
- Easy to see using the SA that two envelope functions combine to change the modulation. Effectively now have three magnetic layers living in the same nuclear layer.
- NOTE: Dead-layers can be inverted, where the bulk becomes non magnetic and the interfaces remain magnetic.

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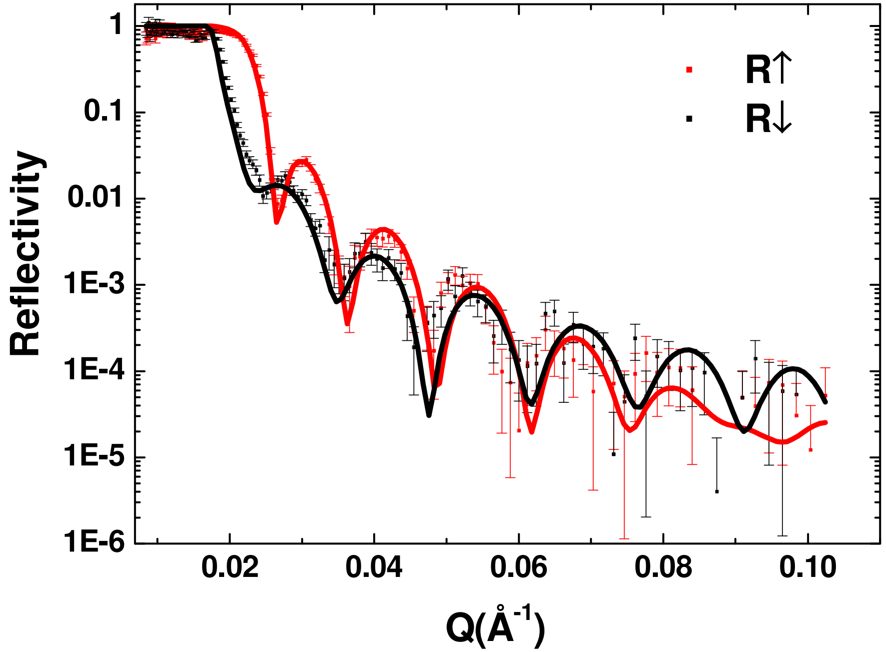
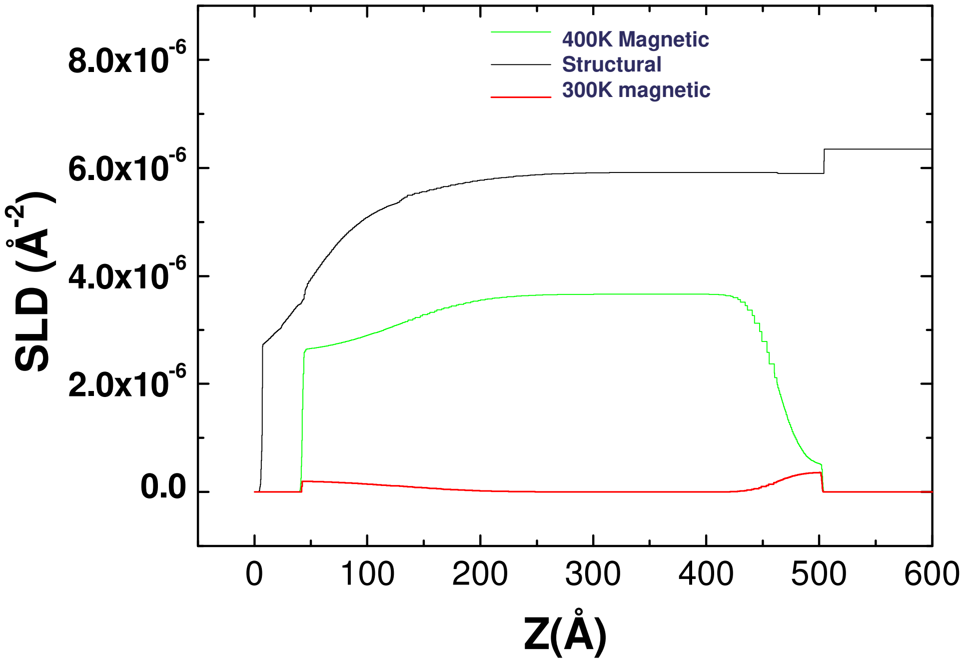
PNR example: MgO(sub)/FeRh(50nm)/MgO(5nm)





- FeRh has a AF/FM/P set of transitions with temperature. The T_c for the FM is 400K (100C).
- Magnetic SLD depth profile, shows how influence of substrate and surface effect magnetism in AF phase. FM persists at in the interface regions to low temperatures.

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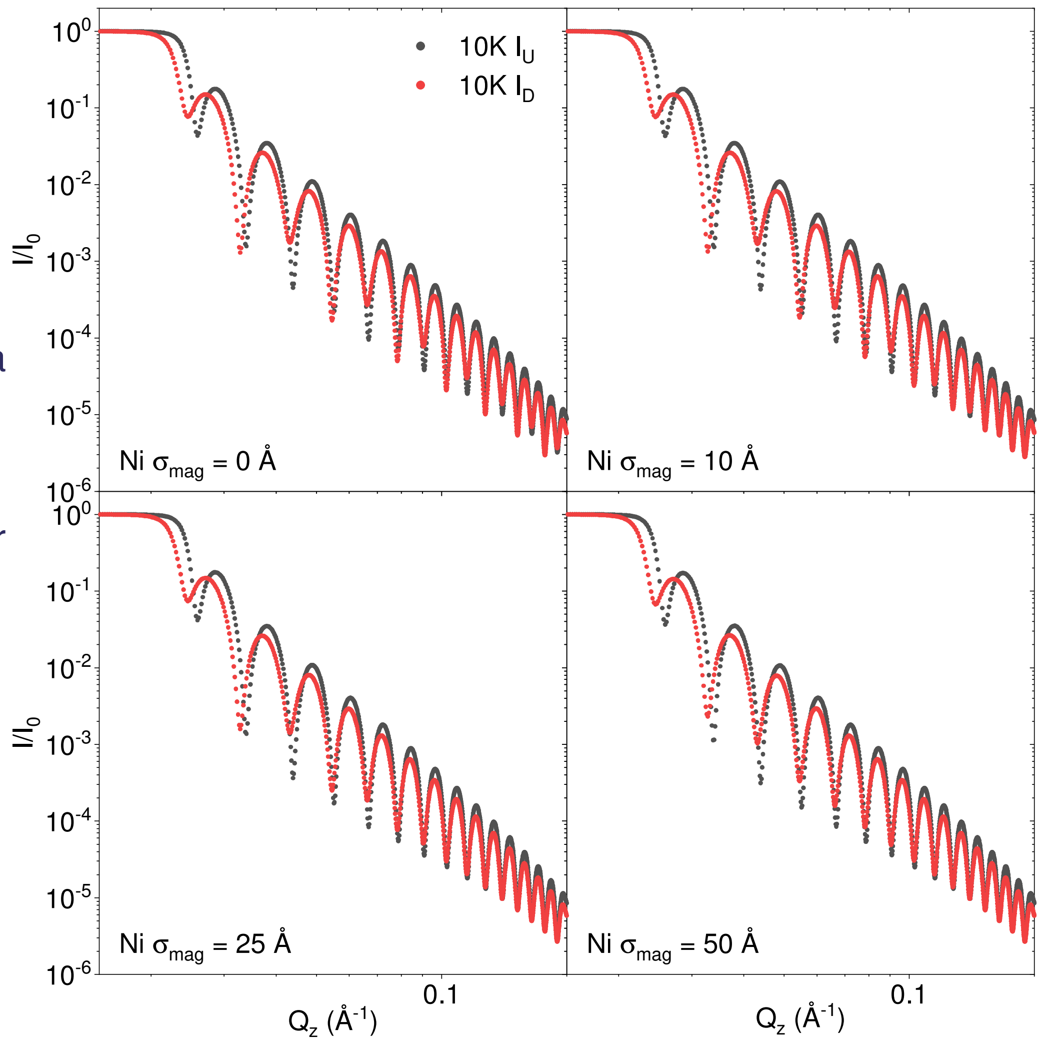
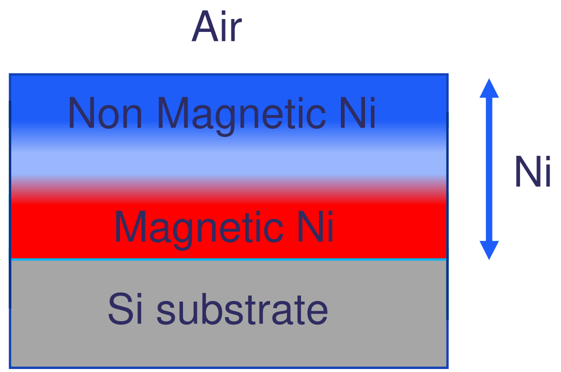
R. Fan *et. al*. Phys. Rev. B 82, 184418 (2010)

PNR Magnetic roughness:

- Magnetic roughness treated the same as structural roughness. Applied to the mSLD only.
- Modelled as a gaussian interface.
- Not the only functional form for a magnetic interface.
- Effects are much more subtle in the reflectivity curves and harder to spot.





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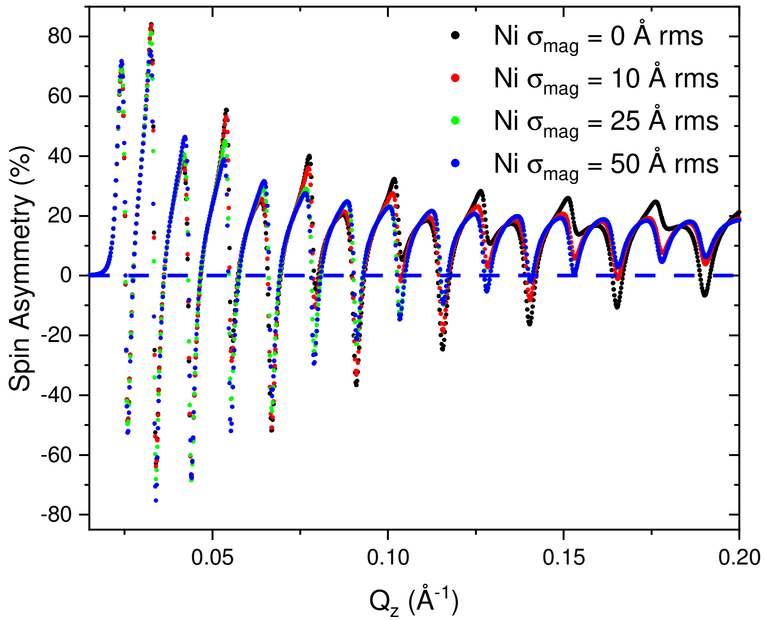
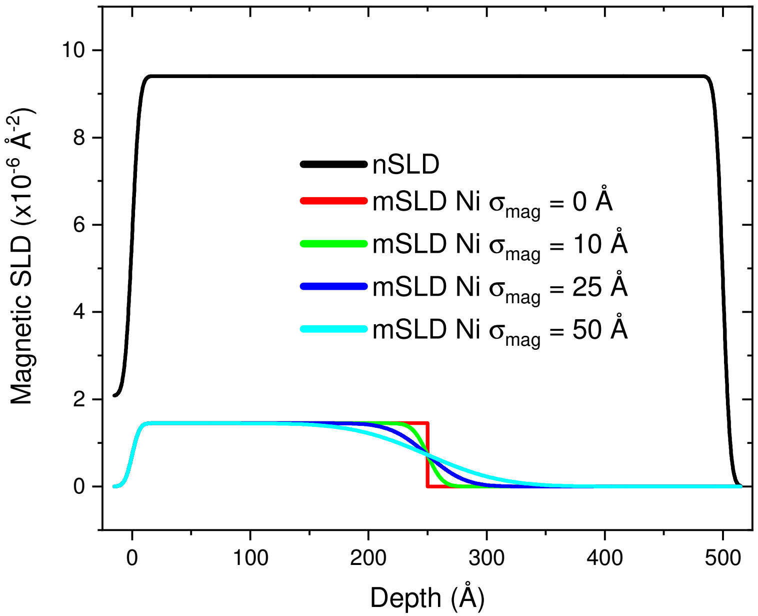
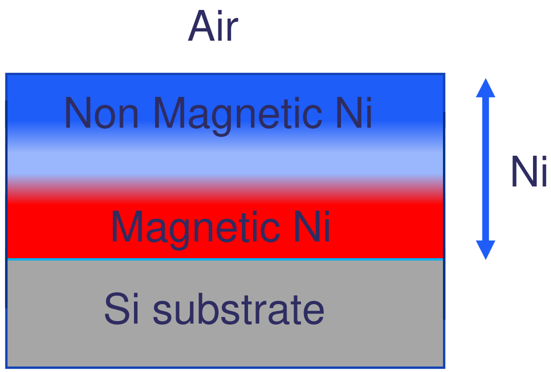
PNR Magnetic roughness:







- The Ni layer is 500 Å thick with the silicon and top interface roughness set to 5Å rms (σ=5 Å).
- 250 Å magnetic dead-layer starting in the middle of the layer which is magnetically roughened via a Gaussian profile.
- Effects are more subtle but easily seen at High Q in the SA

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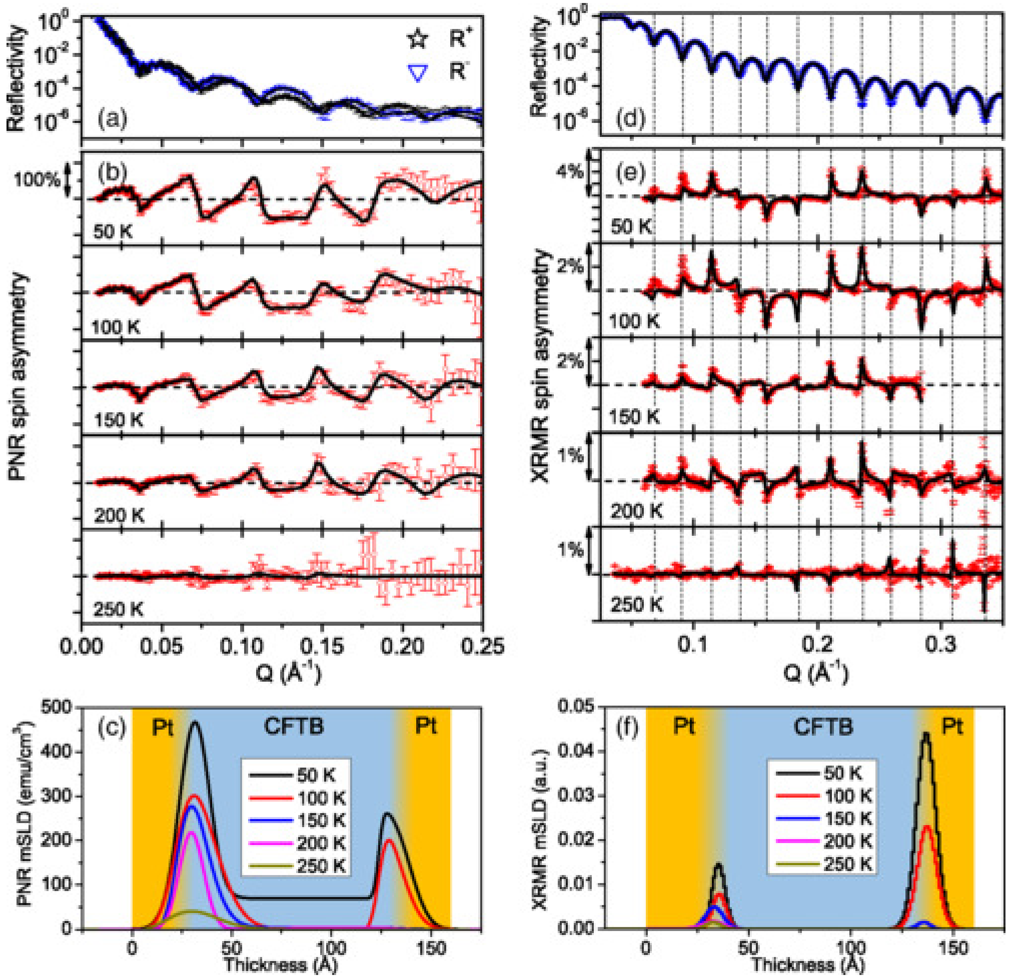
PNR examples CFTB/Pt proximity effects

- CoFeTaB – amorphous FM, low magnetisation and tuneable T_C with tantalum concentration
- Polarised neutron reflectivity (PNR) using Polref instrument @ ISIS, data fitted using GenX
- Scattering sensitive to structure and magnetism
- Magnetic signal here only from CoFeTaB layer: moment on Pt is below sensitivity limit.
- Authors use XRMS scattering to probe the Pt. This is a good example of the complementary use of different scattering techniques.
- The effects are a combination of density changes and magnetic roughness.



O. Inyang *et. al.* Phys. Rev. B 100, 174418

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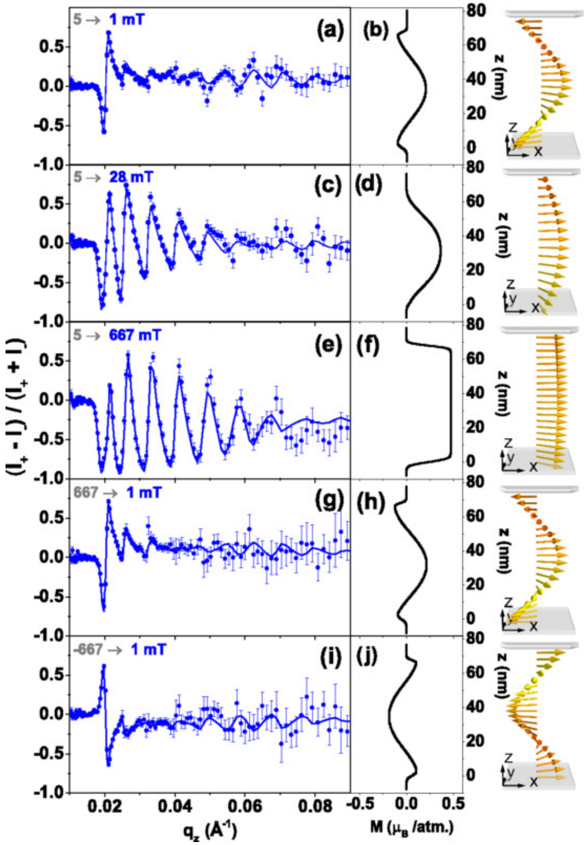
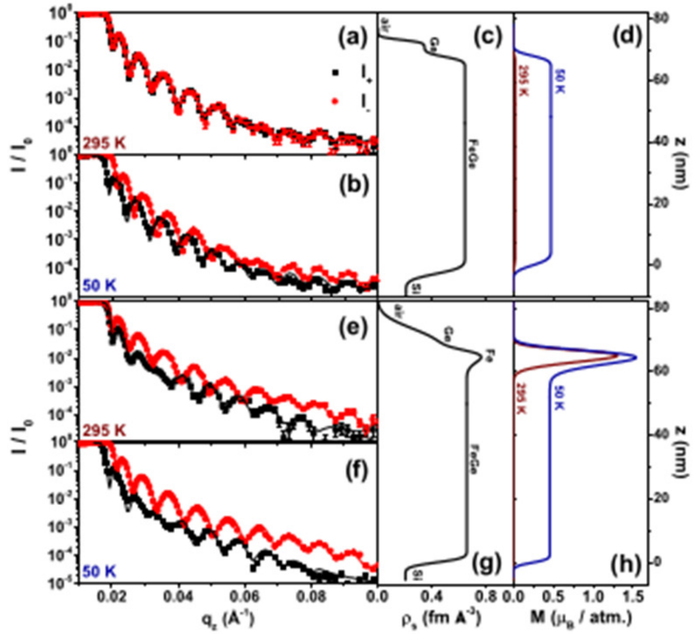
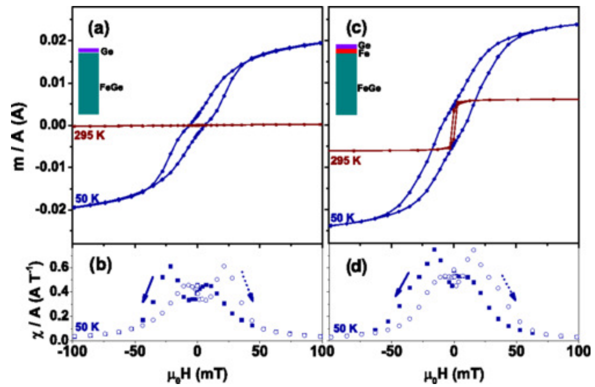
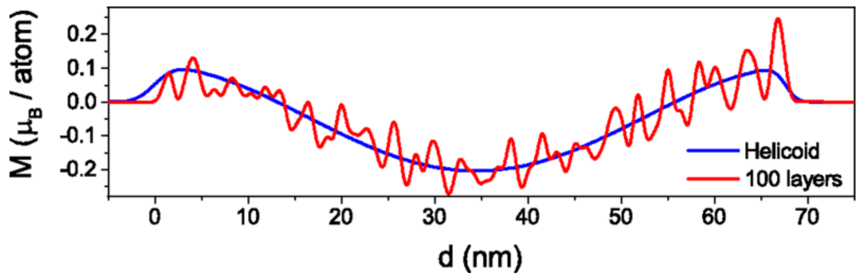
PNR Examples: Spin Helix in FeGe thin films and FeGe/Fe multilayers (DMI)





- Example of the combination of PNR with Transport/Magentometry and XRD/XRR





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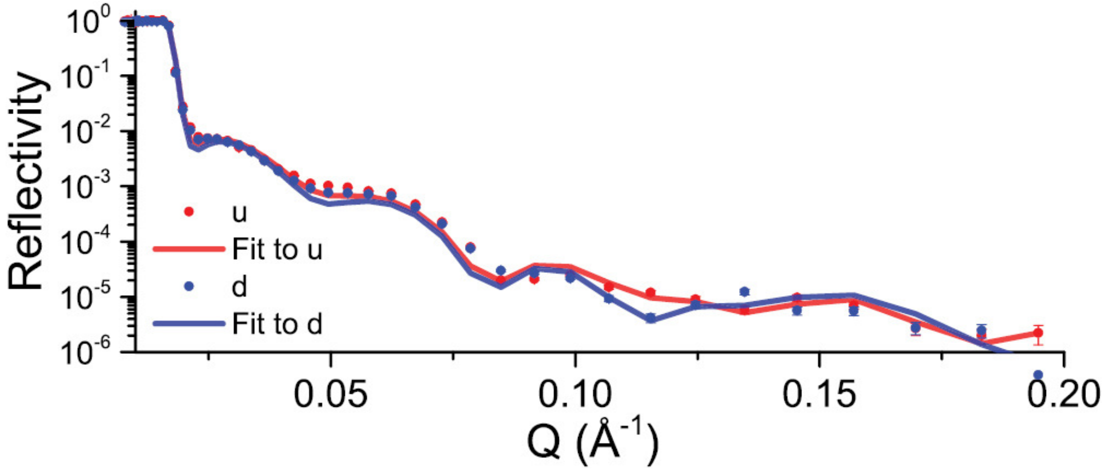
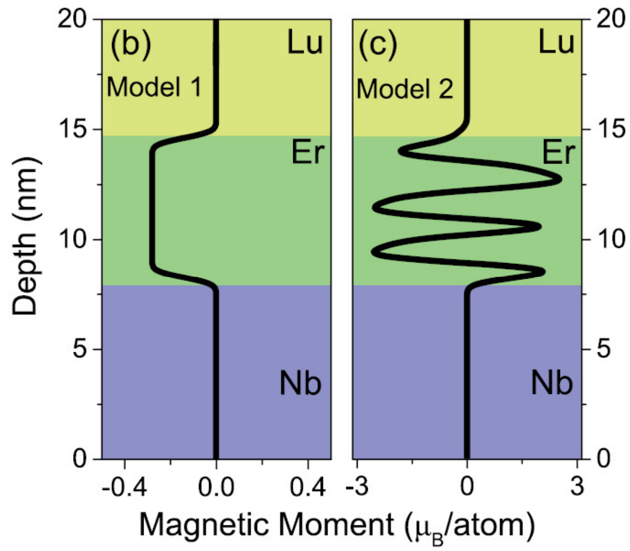
- N.A. Porter et al PRB **92**, 144402 (2015)
- C. S. Spenser *et al PRB 97, 214406, (2018)*

PNR example: Er Spin Spiral





- Er based Spin spiral in a think magnetic layer.
- Good example of the use of a Null hypothesis to indicate the presence of a magnetic structure.

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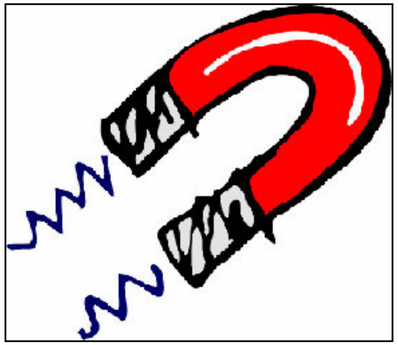
N Satchell *et. al.* Journal of Physics: Condensed Matter, Volume 29, Number 5 2016

Polarisation analysis (PA)

- NR measures the Nuclear scattering length density profile.
- PNR measures the Magnetic scattering length density profile.



- PA measures the Magnetic scattering length density profile and Magnetic vector in the xy plane of the sample as a function of depth!

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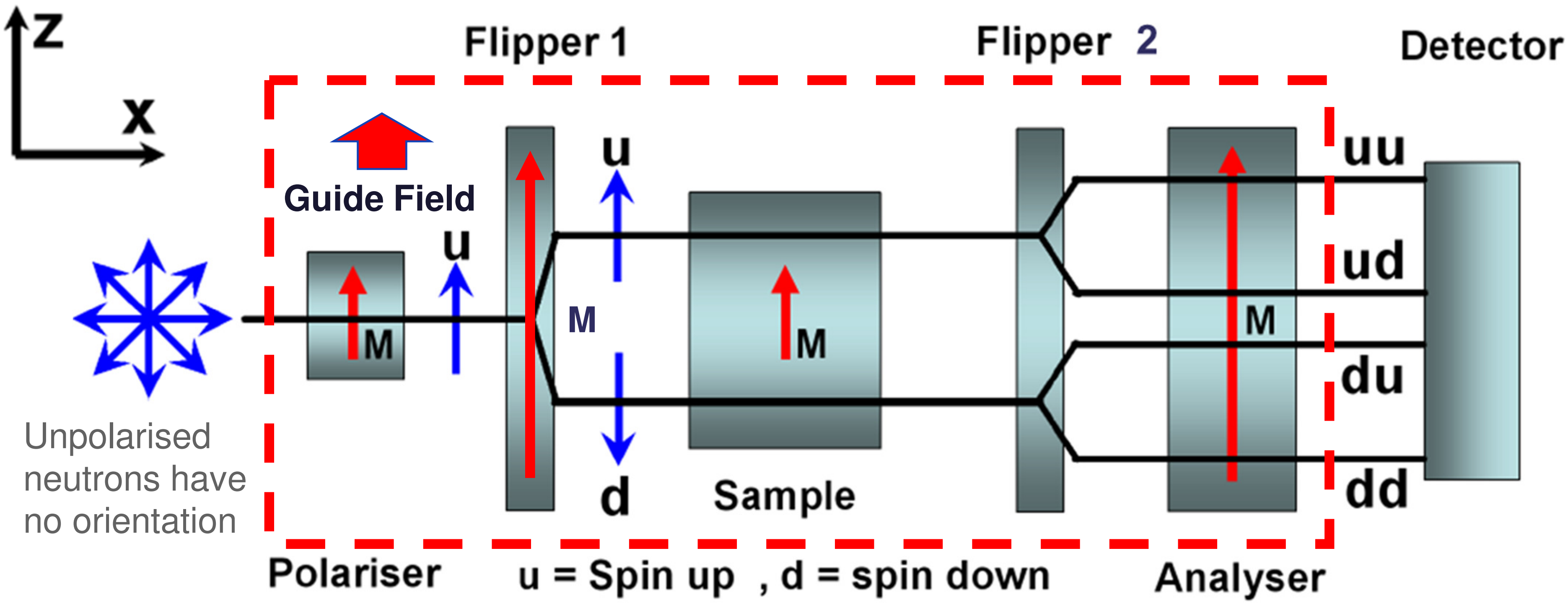
Polarisation analysis (PA):



- Second flipper and Analyser added to the system. **Experiments now takes a factor 8 to 16 times longer!**
- Now measure 4 spin states with 50% of the initial flux! PA experiments very tough!
  - UU and DD – Non Spin flip channels
  - UD and DU – Spin flip channels

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W. Gavin Williams Polarized neutrons Oxford Science
A.-J. Dianoux et al, Neutron Data Booklet (Institut Laue-Langevin, 2002), 1st ed.

Polarisation analysis (PA):

- Only magnetic scattering can cause spin flip scattering.

$$\mathbf{U}^{\mathrm{uu}} = N + M_{\perp,z},$$

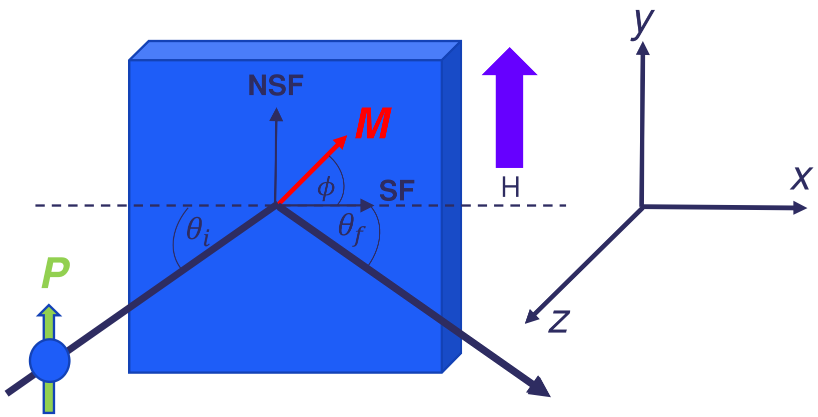
$$\mathbf{U}^{\mathrm{dd}} = N - M_{\perp,z},$$

Non Spin Flip scattering (NSF)

$$\mathbf{U}^{\mathrm{ud}} = M_{\perp,x} + iM_{\perp,y},$$

$$\mathbf{U}^{\mathrm{du}} = M_{\perp,x} - iM_{\perp,y},$$

Spin Flip scattering (NSF)



- By measuring the 4 spins states uu, dd, ud and du, the in-plane orientation of the magnetism ***M*** is obtainable.
- The ratio of Non Spin flip to Spin flip scattering scales with the angle of ***M*** in the plane of the sample.

$$b = b + p \sin \phi \qquad p_m \cos \phi = p_x$$

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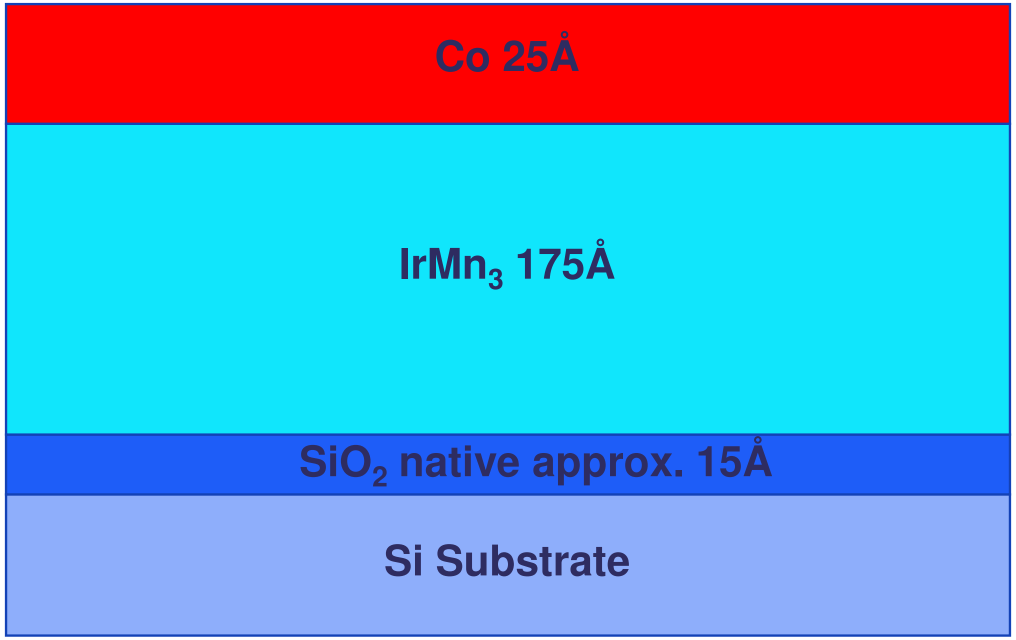
- Bland et al, Phys Rev B, 46, 3391 (**1992**)
- Zabel et al Physica B 276-278, 17 (**2000**)
- R. M. Moon et al Phys Rev, **1969**, 181, 920-931
- S. Blundell et al, JMMM, **1993**, 121, 185-188
- Bland et al, Phys Rev B, 46, 3391 (**1992**)
- G. L. Squires introduction to the Theory of Thermal neutron scattering
- Modern Techniques for Characterizing Magnetic Materials Edited by Yimei Zhu, Springer
- Neutron Scattering form Magnetic Materials, editor Tapan Chatterji, Elsevier

Polarisation Analysis (PA): Test system



- Test Case: proto exchange bias (EB) sample
- EB happens when and antiferromagnet (AFM) is put next to a FM.
- The effect is to pin the FM so it is always pointing in one direction. This is set by a growth field or a thermal anneal above T_c and cool down in a guide field.
- If a small guide field approx. 10 Oe (0.001T) is used at the sample point then the sample can be rotated without ***M*** rotating to follow the guide field.
- Normally sputtered Co is very soft and amorphous/polycrystalline so has very low anisotropy, so it has no preferred direction (easy axis) and will rotated to follow any applied magnetic field.

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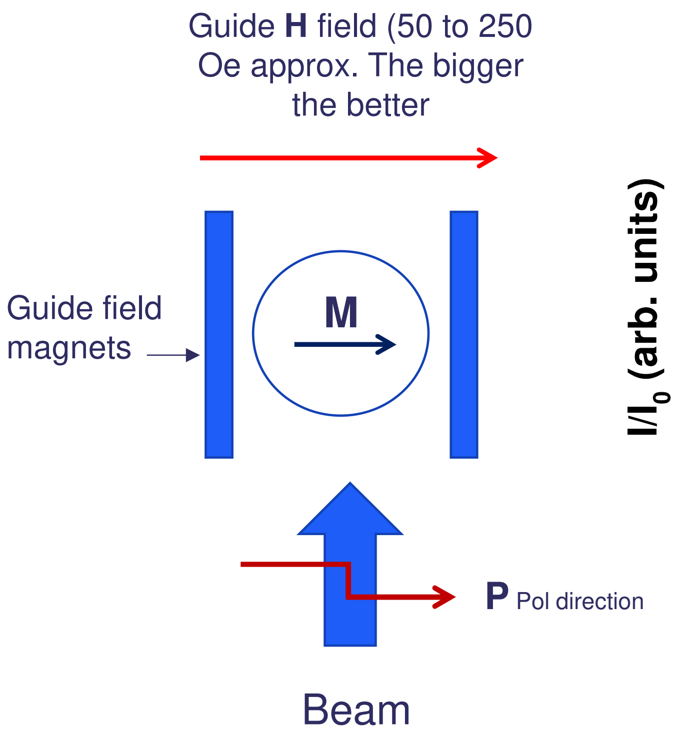
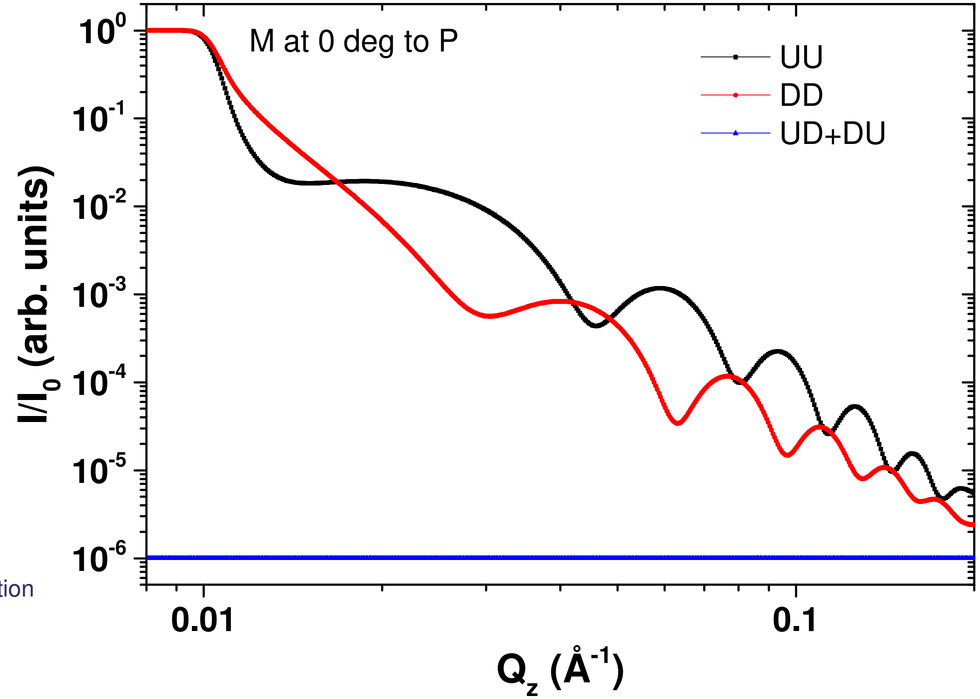
Polarisation Analysis Example 1: 0 Deg rotated M





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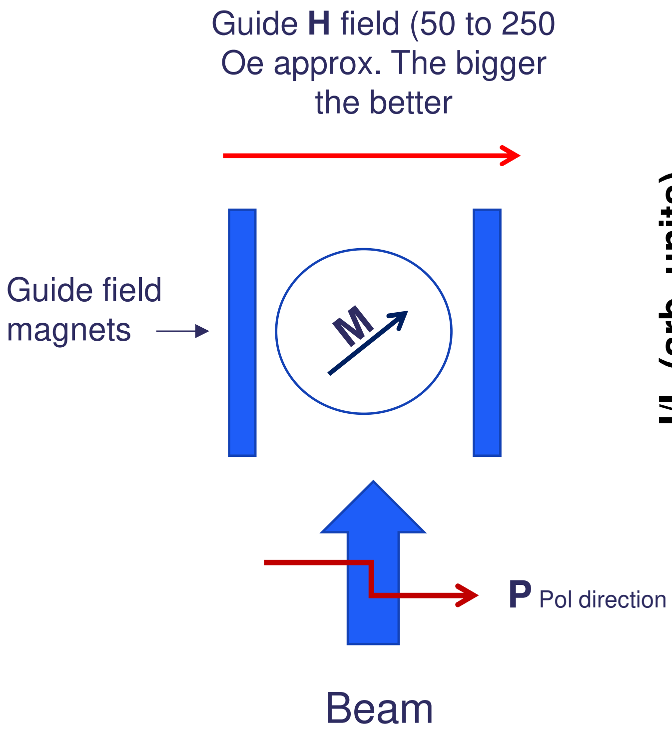
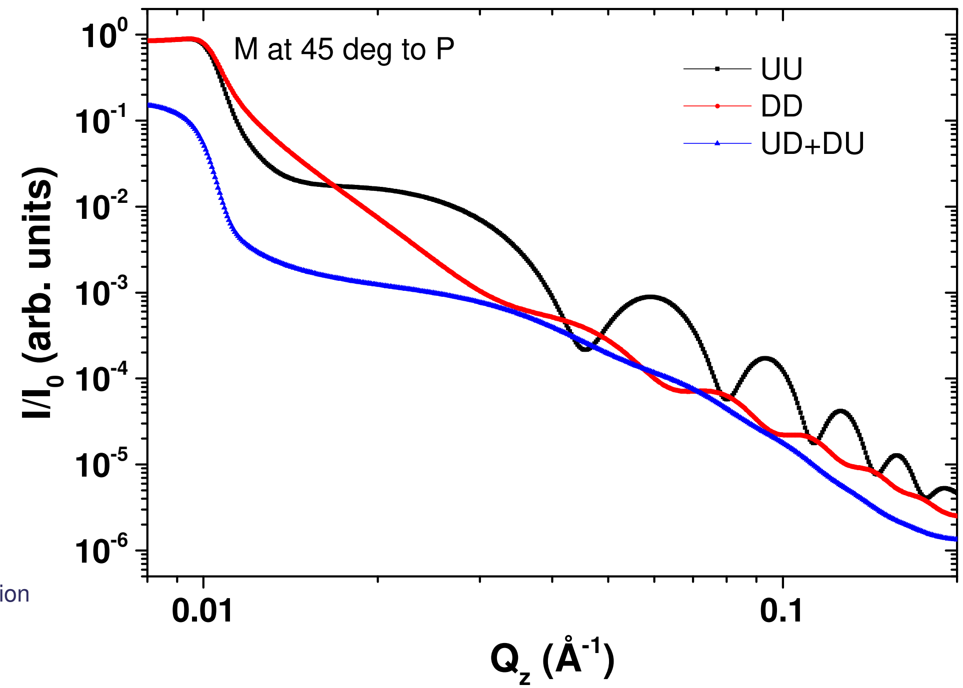
- Non spin flip case, only UU, DD states will can scatter.
- Zero spin flip
- This is similar to what is going on in PNR only any spin flip scattering would be filtered out by the analyser.

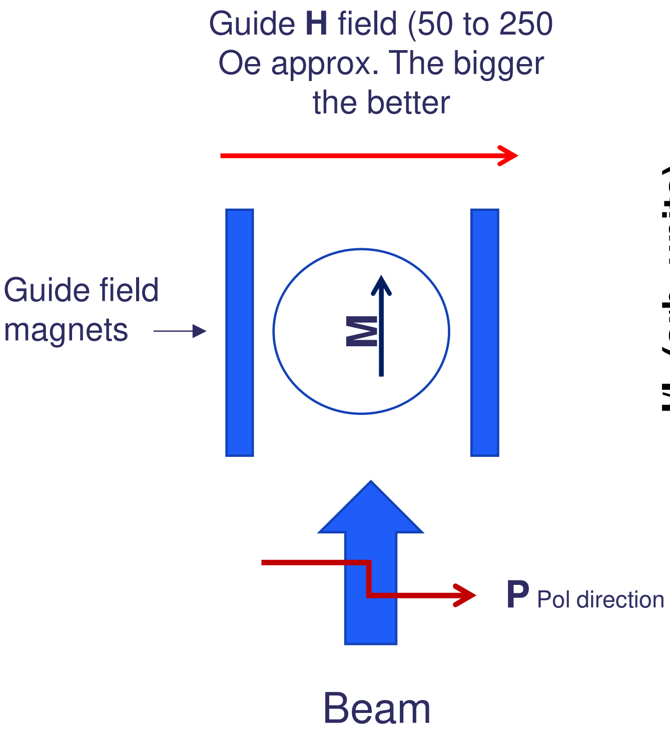
Polarisation Analysis Example 2: 45 Deg rotated M

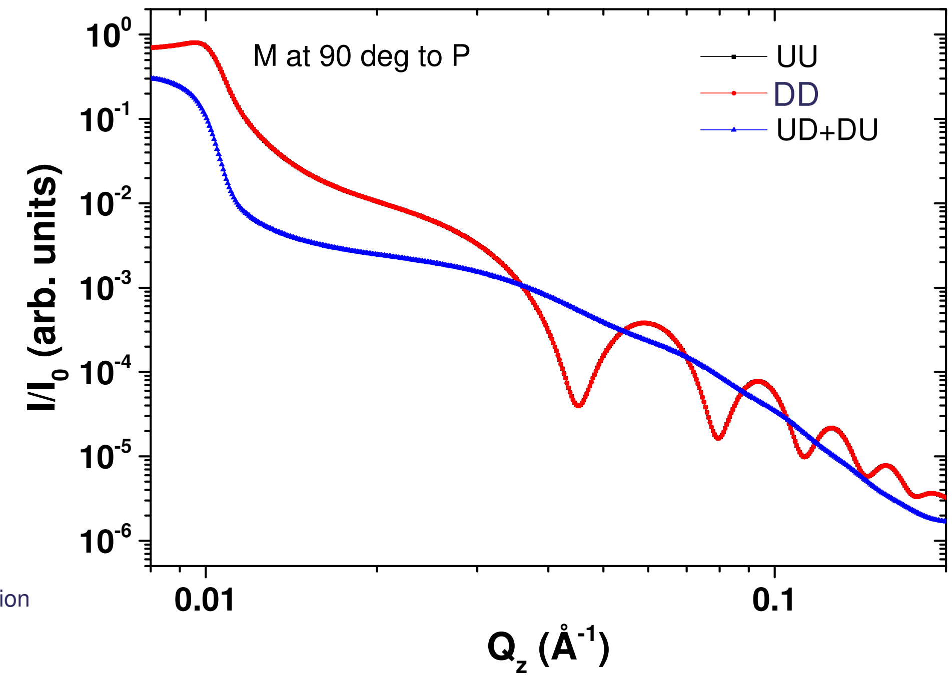




- Non spin flip UU, DD splitting reduced.
- Spin flip UD, DU is increased. In the specular case they should be the same.

Polarisation Analysis Example 3: 90 Deg rotated M





- Non spin flip scattering now purely non magnetic and UU and DD show no spin splitting nuclear scattering only.
- Spin flip UD, DU scatter is maximised

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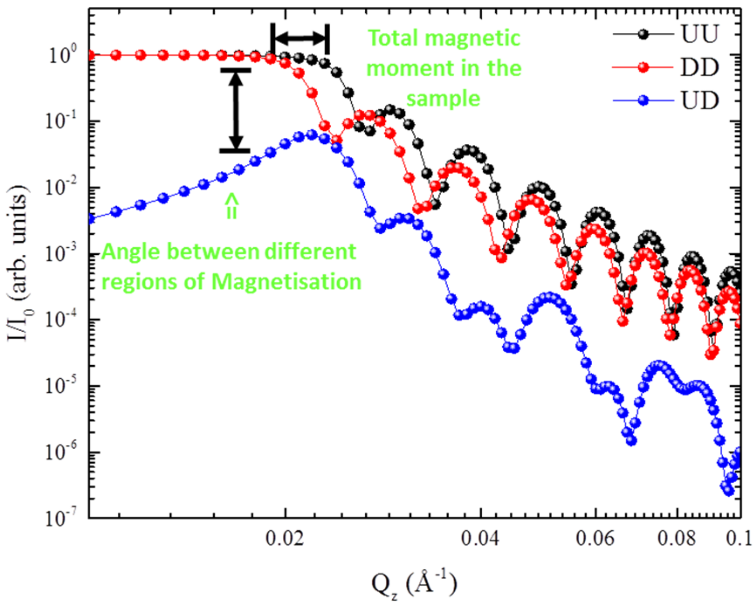
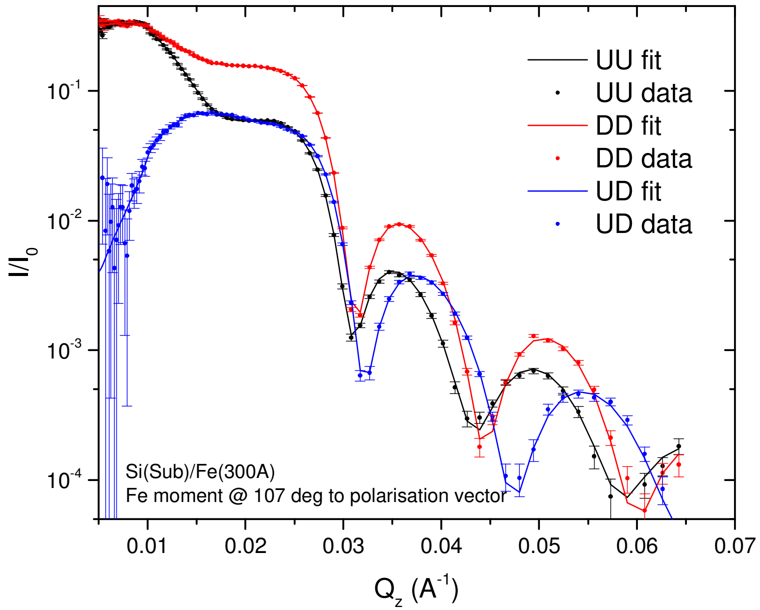
Polarisation Analysis (PA): Summary





- Polarisation analysis tells you the angle of the Magnetisation in the plane of your sample as a function of depth.
- It also provides the nuclear (structural) and magnetic density depth profile.
- But it takes longer than PNR taking a factor 8-16 longer than NR for equivalent statistics.
- Warning PA experiments are tough going! You really **need** to have to do the measurement and really need to know at what fields and temperatures to measure at!

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Magnetic Scattering Rule:

The non-spin-flip scattering is sensitive only to those components of the magnetisation parallel to the neutron spin

The spin-flip scattering is sensitive only to those components of the magnetisation perpendicular to the neutron spin

The ratio of non spin-flip to spin-flip scattering give the angle in the plane of the sample.

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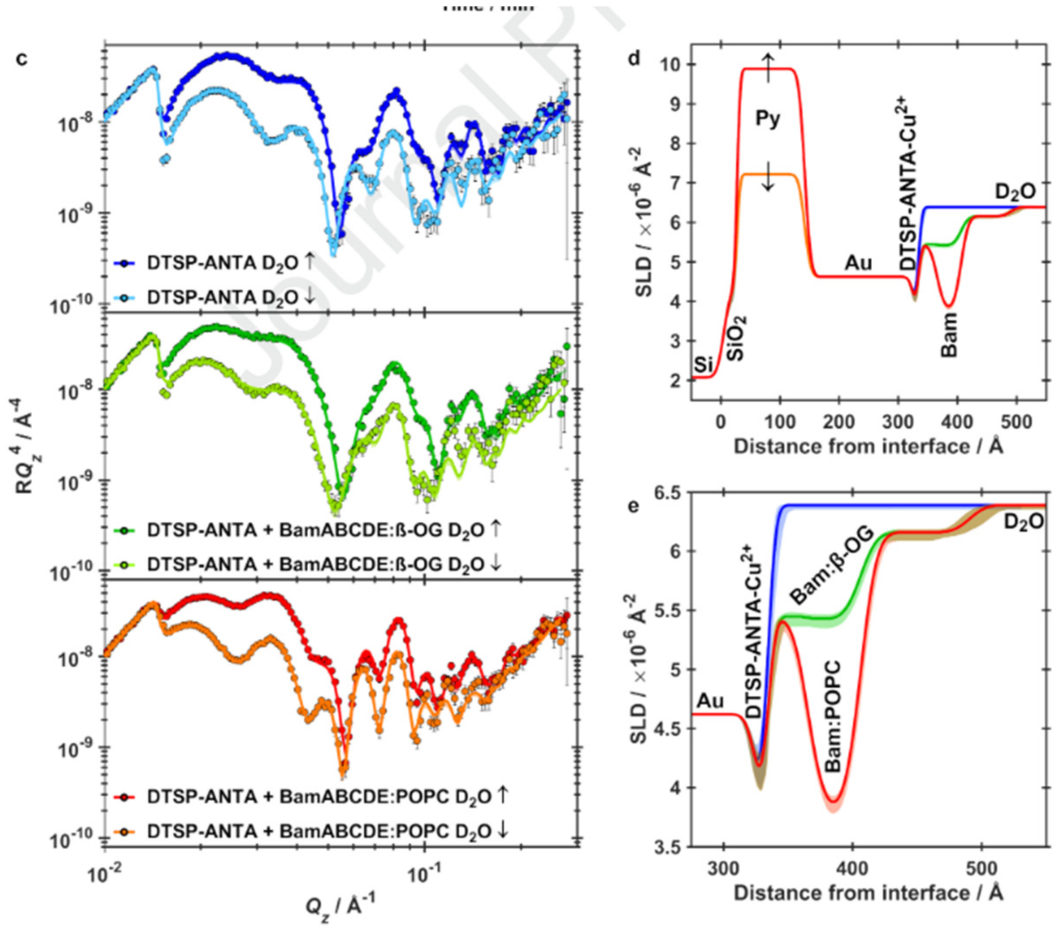
NB This is one of those points that I would like you to take away with you. It is the basis of many magnetic polarization analysis techniques.

Courtesy of Ross Stewart, ISIS

PNR/PA Practical points for experiments: Soft matter Magnetic contrast.

- **Reflectivity is basically a battle for contrast!**
- In NR for Chemistry and Biology isotopic contrast is a real boon. Can mix H_2O and D_2O to contrast to match out layers and beat the inverse phase problem. Makes fitting a lot easier!
- Deuteration not possible for some systems. To expensive or too difficult.
- Magnetic contrast can then be used. Magnetic reference layer is used like Py or Fe. This gives two contrast via that magnetic layer.
- The technique can be used with isotopic contrast as well to reduce the ambiguity of analysis further.
- Warning PNR takes longer to measure (x4) it might be worth measuring more isotopic contrasts rather than one magnetic contrast.



Why reference layers and Isotopic/magnetic contrast variation works:

- A. Koutsioubas, J. Appl. Cryst. (2019). 52, 538–547
- *C.F. Majkrzak et. al., Langmuir* **2003,** *19,* 7796-7810

S.C. Hall *et. al.*,Surface-Tethered Planar Membranes Containing the β-Barrel Assembly Machinery: a platform for Investigating Bacterial Outer Membrane Protein Folding
Biophysical Journal, **2021**

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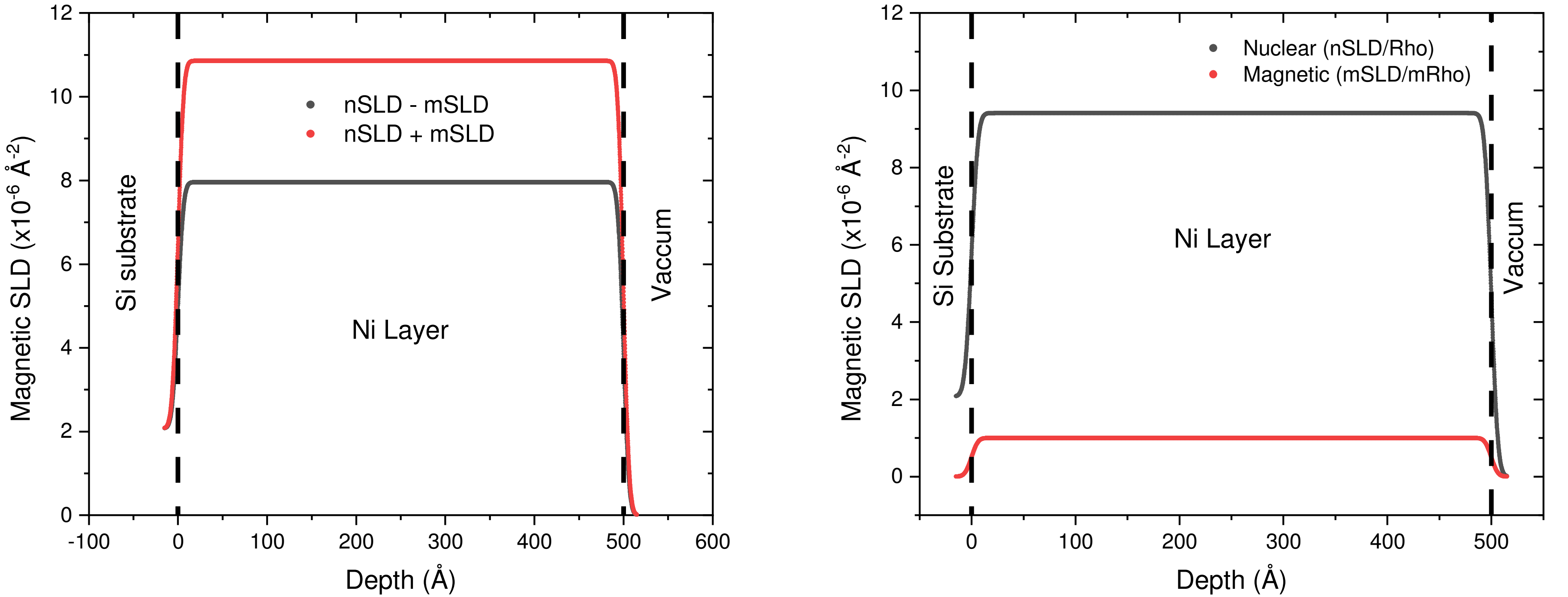
PNR/PA Practical points for experiments:

- Plotting PNR SLD profiles for Hard Condensed Matter (HCM) and Soft Matter (SM) experiments is different as they have different aims.



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- In SM the mSLD is added to the nSLD as the magnetic profile is effectively irrelevant its just there to add another contrast. This is what the spin up and down neutrons actually interact with in reality.
- In HCM the magnetic mSLD profile is the thing that is actually wanted so it is best to deconvolve the two into separate mSLD and nSLD curves.

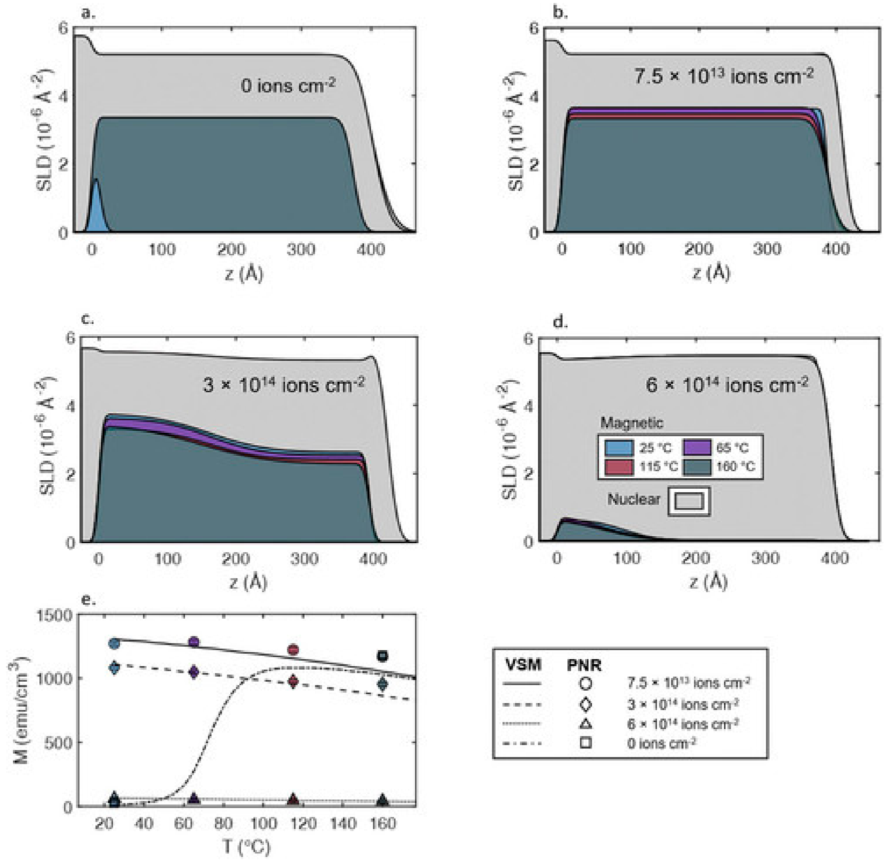
PNR/PA Practical points for experiments:

Experimental planning: Count times and picking measurements!

- **Technique:**
  - NR is quick
  - PNR is x4 time slower than NR
  - PA is x8 -x16 times slower than NR (is it really needed? Pick your measurements very carefully, beamtime is limited and precious, ***its your Thesis!***
- **Sample area: Count time scales linearly with area**
  - SM samples sizes tend to by 80mm x 50 mm in area or bigger.
  - PNR/PA samples tend to be 20 x 20 mm 10 x 10 mm or even 5 x 5 mm in area!
  - A 20 x 20 mm sample will require a **factor 10** in count time to get the same level of statistics as an 80mm x 50mm SM Si block!
  - **Point to take home for PNR/PA the larger the surface area the better!**
- **Sample area: Other effects of small surface areas on data quality**
  - Smaller Q range – its not possible to count out to high Q usually within the time allowed for an experiment.
  - **Small sample often replace slit 2 as the final resolution optic! Think carefully about what resolution you need! This can have dramatic effects on resolution and count times!**
  - Can you redesign your sample, not affecting the physics/chemistry/biology, to push the measurable effects to lower Q. Tricks include:
    - Resonance effects.
    - Bilayer/multilayering of the system to boost the signal into a Bragg peak.
    - Thick buffer/capping layers to shift the signal to lower Q in a radio carrier wave analogy.
    - Thickness variation of the various layers to make sure features of interest are not in intensity minima.
  - **When designing the experiment simulating the sample and what you what to see is KEY to a successful experiment!**

PNR/PA Practical points for experiments:

- **Contrast is key for HCM**
  - Like in SM, HCM experiments need to think about contrasts.
  - Trying to fit one PNR data set in isolation is very difficult as can get multiple solutions.
  - If possible measure at H_{sat} and H_{rem} or at any other magnetic of interest and simultaneously fit the data.
  - This also works if there is a variation of M in the sample with temperature.
  - Measuring multiple fields or temperatures and fitting simultaneously is analogous to solving a simultaneous equations. This can vastly reduce ambiguity! (more in the lectures to come on this)



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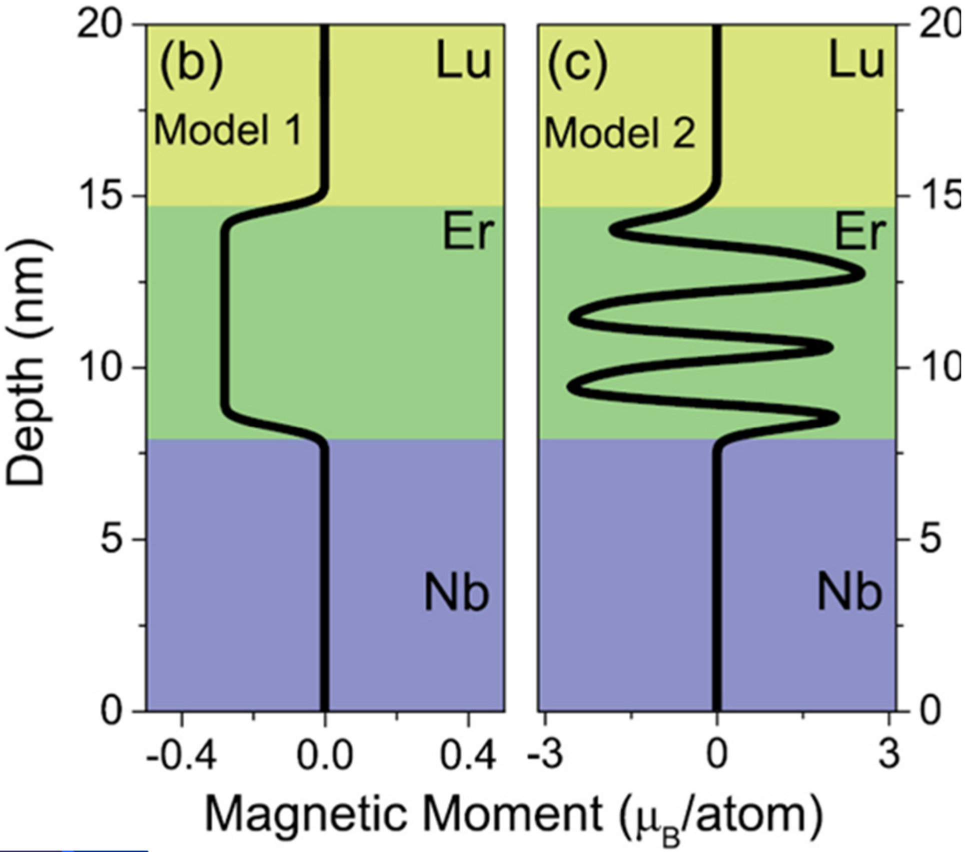
W. Griggs, *et. al.*, Depth selective magnetic phase coexistence in FeRh thin films, *APL Materials*, **2020**, *8*, 121103

PNR/PA Practical points for experiments:



- **Null Experiments:**
  - Often results are not clear, so think carefully about the null measurements you need to make to demonstrate an effect is present or not!
    - E.g. saturate the sample
    - E.g. change the temperature to be above or below a transition temperature.
  - Experiment time is limited so pick carefully!
  - Same idea applies to analysis of the data!

- **Setup time and correction/calibration measurements:**
  - Remember to factor in any transmission measurements. **This is very important for SM experiments as not all Si blocks, etc are the same and may have different transmissions!**
  - Remember to factor in sample environment changes like sample changes, sample alignment warm up and cool down times for cryostats
  - **TALK TO THE LOCAL CONTACT ABOUT THE ABOVE!!!!!!!**

N Satchell *et. al.* Journal of Physics: Condensed Matter, Volume 29, Number 5 2016

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PNR/PA Practical points for experiments:

- **Pre characterise samples:**
  - Please ***Please*** pre characterise your samples if at all possible! We have the R53 facility at ISIS to help with this!
    - XRR at a bar minimum along with SQUID (HvsT or MvsT)
    - XRD/Transport/AFM/MOKE/TEM if you have time.
- Why? PNR/PA takes a long time to count out so you have to count in the right place on the right sample!
  - Does the sample display the effect/phenomena?
  - It is the worst idea in the world to use a PNR beamline like a SQUID because you don't know where the transition is. You could have just used a squid and be counting out the magnetic depth profile or vector profile which is what PNR/PA does best!
- The Mark 1 Eyeball is a good technique to use to check a sample is optically flat. I.e. if the sample is rough visibly! Its unlikely to work on a reflectometer!

- **Pre characterise samples:**
  - Anisotropies! Do you know the sample anisotropies, easy and hard axes?
    - SQUID/MOKE/crystallography
    - This is so the sample can be mounted in the right direction in the beamline.
  - Silicon block roughness/substrate roughness! THIS IS VERY IMPORTANT FOR SM experiments! A quick XRR scan can show if you have a smooth block or not! If its too rough it will not work well.

In science fortune favours the prepared!

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Thank you

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