Practical Aspects of Neutron Reflection
How to Collect Data

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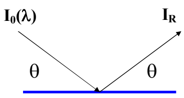
Reflection – measured quantities

Reflection



Reflected beam deflected:

2 θ

Reflectivity

$R(\theta, \lambda) = I_R/I_0(\lambda)$

Momentum transfer

$Q = (4\pi/\lambda) \sin \theta$

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Best Sources of Neutrons

ILL reactor continuous

Thermal Flux 1.5 x 10^{15} n cm^{-2} s^{-1}

SNS, ORNL

60 Hz, 300 μs

5 x 10^{17} n cm^{-2} s^{-1} (Peak)

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Neutrons: Speed & Wavelength

Velocity, v, from de Broglie relation

$v \lambda = 3956$ m s^{-1} Å

i.e. 10 Å has 400 m s^{-1}

Gravity is significant, separate wavelengths mechanically

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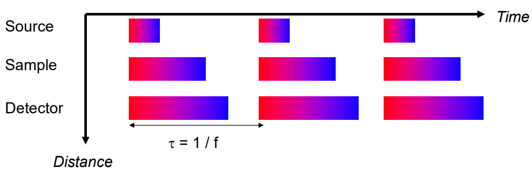
Using a Pulsed Source



Detection time (after source pulse) gives wavelength

Choppers can select a wavelength

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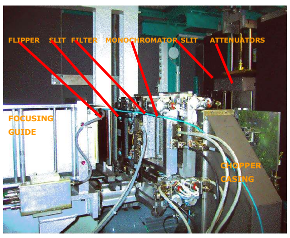
D17 Reflectometer



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Practical Issues

Reflectivity drops quickly with increasing Q (or angle). Signal is easily ‘lost’ in background.

To observe fringes it will be necessary to measure over an appropriate range of Q and to have sufficient resolution (ΔQ small enough).

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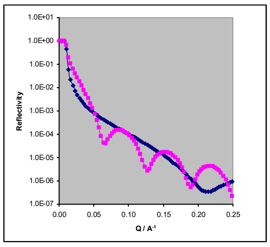
Reflection from a Thin Film

Model calculation on smooth surface.

Fringe spacing depends on thickness

Fringe spacing ~ $2\pi/d$



Model layer with ρ = 5 x 10^{-6} Å^2 on Si (2.07 x 10^{-6} Å $^{-2}$) Blue 30 Å, Pink 100 Å. No roughness.

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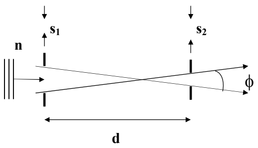
Resolution in Q

$Q = (4\pi/\lambda) \sin\theta$

Depends on $\Delta\lambda$ and $\Delta\theta$

Angle resolution, $\Delta\theta$, depends on collimation (slits)

Wavelength resolution depends on monochromator or time resolution in measuring neutron pulse



Higher Resolution = Lower Flux

$(\Delta Q/Q)^2 = (\Delta\lambda/\lambda)^2 + (\Delta\theta/\theta)^2$

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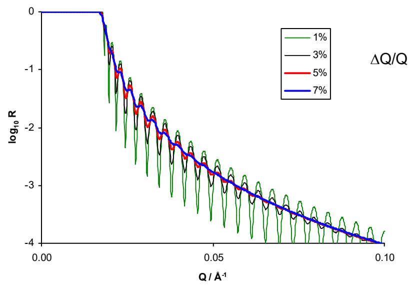
Effects of Resolution



Silicon substrate: film thickness 1500 Å (150 nm) scattering length density 6.3 × 10^{-6} Å^{-2}

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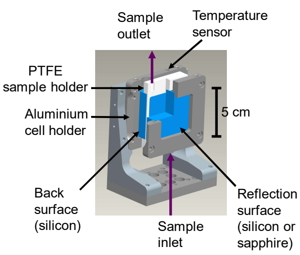
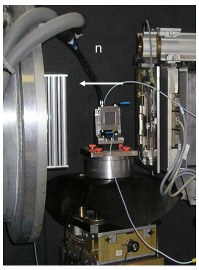
Sample Holder



D17 reflectometer ILL, France



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Alignment

Rotation table must have centre on beam axis

Sample must be centred on rotation (half obscure the direct beam) – eucentric mount

Determine θ from the position of beam on a detector

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Aligning a Sample

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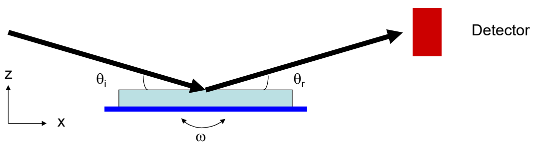

Design mount with surface at centre of rotation of ω. Eucentric mount.

Put centre of surface on the line through axis of rotation (x direction)

The rotation ω stage must be centred on the incident beam.

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Aligning a Sample

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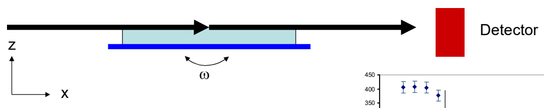
Set sample and detector to nominal zero

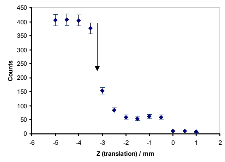
Choose fine slits to give collimated beam



Scan z

Look at intensity on detector

Identify z = -3.2 (~230 cts) as position interface intersects direct beam



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Aligning a Sample

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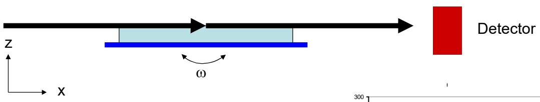
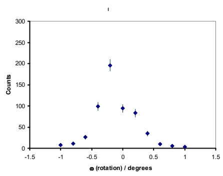
Move z to approximate sample in beam position



Scan ω

Look at intensity on detector

Identify ω = -0.22 (~190 cts) as approximate sample offset angle



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Aligning a Sample

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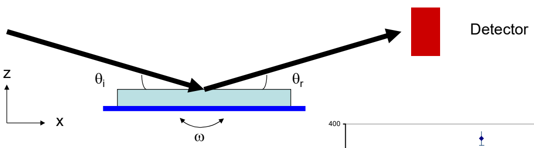
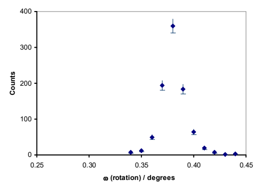


Use approximate ω and z offset from alignment on direct beam

Set detector to small angle of reflection (e.g. 0.5°) and align more precisely.

Scan ω and look for peak. Position is 0.378° and so offset is -0.122°.



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Aligning a Sample

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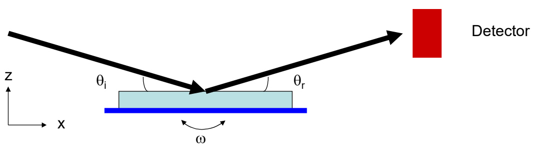
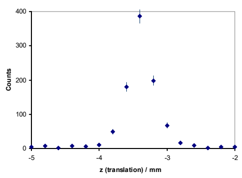


Use new ω offset and z offset from alignment on direct beam

Check translation (z) offset in reflection mode.

Scan z and look for peak. Position is -3.38 mm.



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Comments on Alignment

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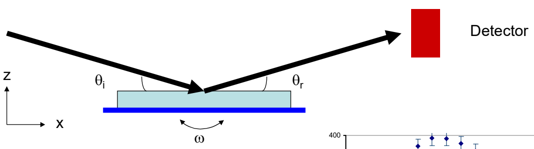
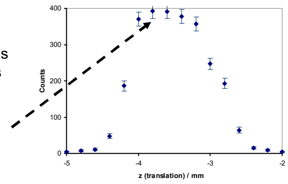


Angular (ω) width can depend on flatness of sample as well as resolution from slits and wavelength spread

If sample is very under-illuminated, translation (z) scan will have a flat top



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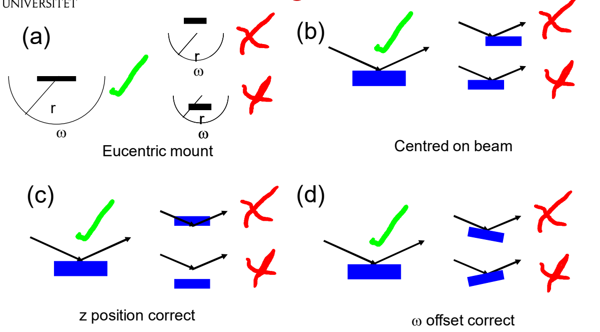
Summary - Mounting and Alignment



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Comments on Alignment

Using the results of alignment scans needs offsets or new zero positions to be set on the instrument. Warning: there is no general convention of signs on different instruments

Linear thermal expansion can be ~2 x 10^{-5} K^{-1}. 4 cm of aluminium changed by 50 C gives a shift of 0.04 mm.

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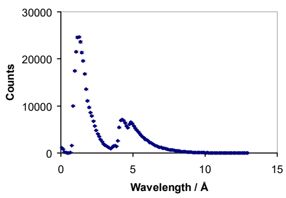
Calibrations

Scan angle, measure different λ or a combination of λ and angle

Measure direct beam (through sample environment if needed)



Incident beam spectrum, LARMOR

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Samples

Low incident angle requires large uniform surface area. Footprint ~ s / tan θ.

Areas often several cm^2.

Smooth surface. 10 Å roughness will reduce the reflectivity at q=0.1 $Å^{-1}$ by 2.7. 15 Å reduces reflectivity by a factor of 10.

Liquids will have surface oscillations (capillary waves). Need to avoid other, induced waves.

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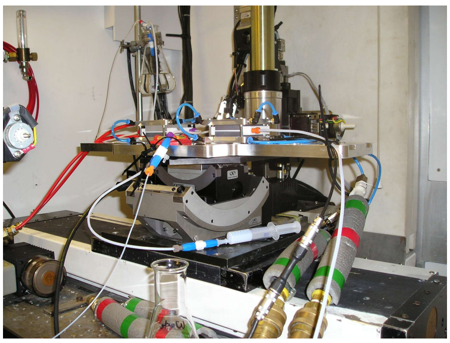
Sample Cell



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What is measured?

Reflected signal may have a large background

For hydrogenous substrate ~ 5 x 10^{-6} incident beam

Attenuation by reduced transmission (caused by scattering or absorption) may be significant

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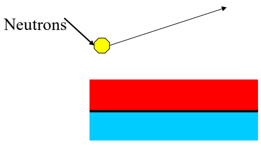
Fate of a Neutron at an Interface

- Reflected
- Scattered/Diffracted from surface
- Absorbed
- Scattered from bulk (either side of surface)
- Other accidents



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Contrast Matching

H_2O $\rho = -0.56 \times 10^{-6}$ Å^{-2}
D_2O $\rho = +6.35 \times 10^{-6}$ Å^{-2}

$y \times 6.35 + (1-y) \times (-0.56) = 0$
$6.91\ y = 0.56$ or $y = 0.56 / 6.91 = 0.081$

i.e. 8% by volume of D_2O in H_2O has n = 1

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Comments on Calculations

Programs that lose data

It is common to use logaritmic scales but background subtraction can give negative data points. R Q^4 is useful.

Experimental issues

Resolution – often needs to be included

Illumination

Small samples are often not able to reflect all the beam and a geometrical correction is applied.

Absolute reflectivity

Data is constrained if it is on an an absolute scale

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What has not (yet) been covered?

Ellipsometry and X-rays
Needs more calculations for *s* and *p* waves

How to write a minimisation routine?

How to install your favourite program?

Specific examples of real samples etc.

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Do's and Don'ts

- Do not bend samples – care with mounts
- Use anti-vibration mounts for liquids – air borne noise causes vibrations
- Capillary waves cause scattering

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Questions?

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