Neutron Optics

INSTITUT LAUE LANGEVIN

NEUTRONS
FOR SOCIETY

P. Courtois

Service for Neutron Optics

Cremlin 14-15 May 2018

THE EUROPEAN NEUTRON SOURCE

Neutron Optics

Neutrons have wave-like properties…

Basic concepts and optical components

➢ Reflecting Optics	Mirrors & Super-Mirrors
➢ Diffracting Optics	Mosaic crystals
➢ Filters	Crystals, polycrystalline materials
➢ Polarizing Optics	Super-Mirrors, crystals, ^{3}He spin filters

➢ Some examples of applications at I.L.L.

Why do we need neutron optics

Neutron Optics are key components for neutron instrumentation

- Mirror & Super-Mirror are used to construct efficient neutron guides
- Collimator determines incident and scattered neutron directions - Angular resolution
- Filter selects out unwanted neutrons
- Crystal Monochromator/Bragg Mirror determines incident wavelengths (Energies) }
- Crystal Analyzer determines scattered wavelengths (Energies) }

Wavelength resolution (Energy resolution)

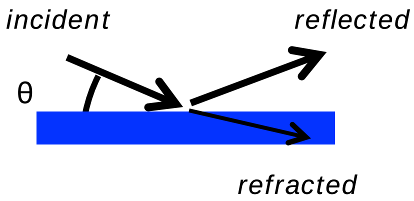
- Crystal/ Super-Mirror/ ^{3}He spin filter are used to polarize neutron beams

Orientation of the neutron spin

➢ **Instrument Performances are then determined by Neutron Optics !**

Neutron Mirrors

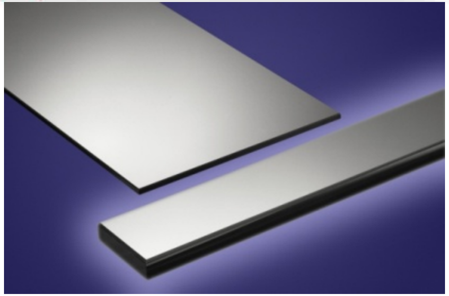
Reflection / Refraction at Surfaces



Total reflection for $\theta < \theta_c$

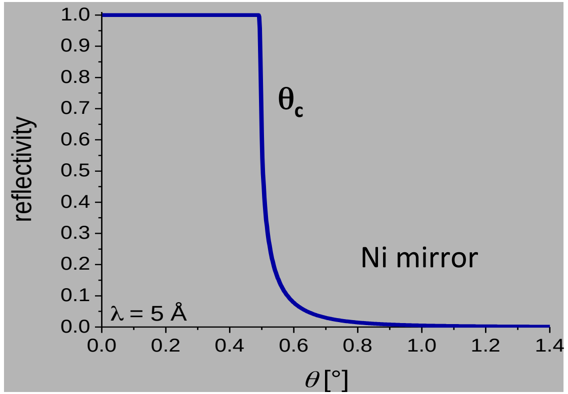
$$\theta_c = \lambda\sqrt{\frac{Nb_{coh}}{\pi}}$$

Nb_{coh} = Scattering Length Density
N = atoms/cm³
b_{coh} = coherent scattering length



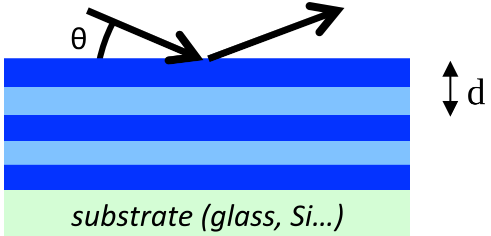
Material	Nb (x 10^{38} /m²)	θ_c (mrad)
^{58}Nickel	13.31	2.03
Nickel	9.41	1.7
Iron	8.2	1.62
Copper	6.7	1.39
Silicon	2.08	0.81
Aluminium	2.08	0.81

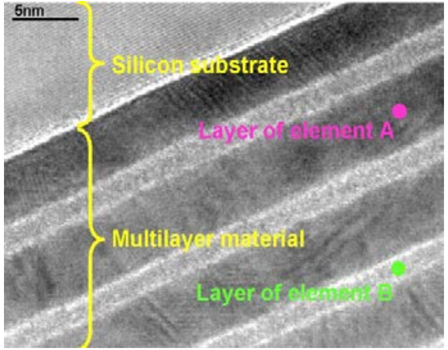
for natural Ni: $\theta_c(°) = 0.1 \times \lambda$ (Å)



Periodic Multilayers

Reflection / Refraction at Surfaces

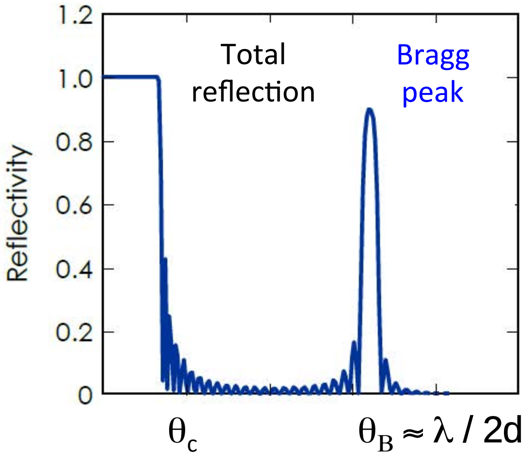




Total reflection for $\theta < \theta_c$

+

additional Bragg peak at $\theta_B \approx \lambda / 2d$



Neutron Reflectivity

$$R \propto \frac{4N^2 d^4 (N_1 b_1 - N_2 b_2)^2}{\pi^2 n^4}$$

Wavelength band

$$\frac{\Delta\lambda}{\lambda} = \frac{2d^2 |N_1 b_1 - N_2 b_2|}{\pi}$$

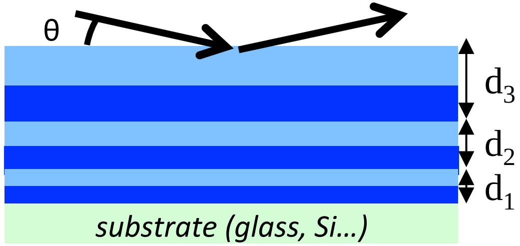
Nb_{coh} = Scattering Length Density

N : number of bilayers

n : order of the reflection

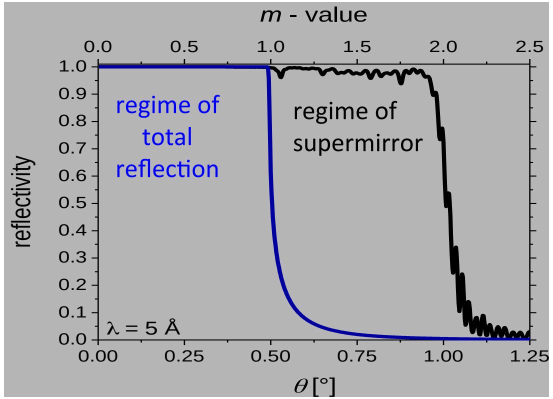
Super-Mirrors

How to increase angular acceptance of mirrors



Sequence of bi-layers of variable thicknesses d

Total reflection for $\theta < \theta_c$ + additional Bragg peaks



→ **significant increase in critical angle ($\theta_c = \lambda / 2d_{min}$)**

m Super-Mirror

$$m = \frac{\theta_c^{SM}}{\theta_c^{Ni}}$$

Gain in neutron flux

$$G = \left(\frac{\theta_c^{sm}}{\theta_c^{Ni}}\right)^2 \propto m^2$$

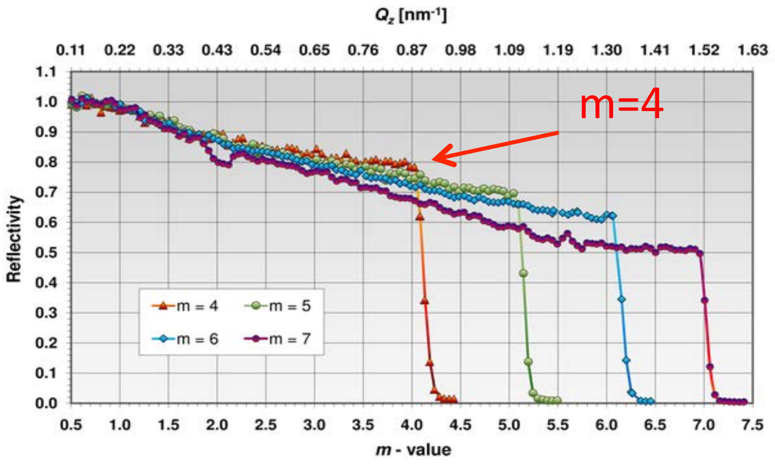
Ni/Ti Super-Mirrors

Properties

Neutron Reflectivity $R \propto (N_1b_1 - N_2b_2)^2$

- *Ni* $Nb = 9.40 \; (10^{-6}\text{Å}^{-2})$
- *Ti* $Nb = -1.95 \; (10^{-6}\text{Å}^{-2})$

High contrast → high reflectivity !



Reflectivity profiles of Ni/Ti super-mirrors for 4 ≤m ≤ 7
(sources : www.swissneutronics.ch)

Performances

- R > 80% for m = 4 Ni/Ti

➢ Gain factor / Ni mirror = 16 (2D)

➢ but transmission $T \propto R^n$!!

eg : for a 100 m long guide , at least ten reflections → Transmission < 10% ...

Ni/Ti Super-Mirrors

Deposition : Reactive DC Magnetron Sputtering

Sputtering machine (ILL) - Production 0.8 m² / day

Production

- m= 4 : 1600 layers !
- Substrate : 0.5 cm thick Si wafers or 0.2 cm thick Glass/Si/Sapphire

Applications

- Neutron guides (previous presentation)
- Collimation
- **Filters** (SANS instruments)
- **Large Band monochromators** (Laue Diffraction)

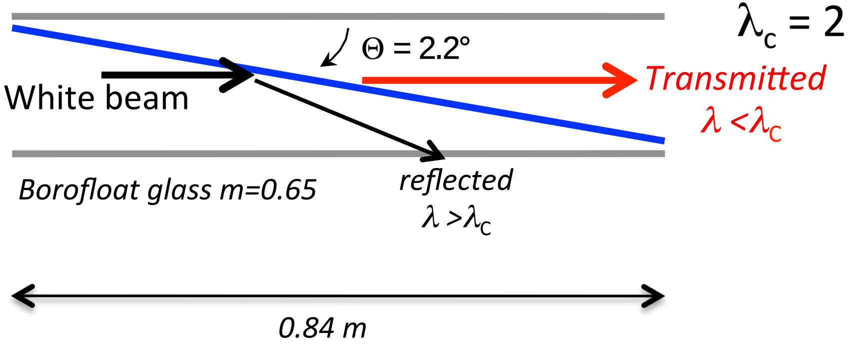
m Super-Mirrors

Long wavelength cut-off filters on D33 at I.L.L. (SANS instrument)

- **to select out unwanted neutrons of wavelengths >** λ_c (3 available wavelength bands)

m=1.1 SM NiV/Ti on 0.5 cm thick Si substrate mounted at an angle θ in a neutron guide

$$\lambda_c = \frac{\theta}{m\theta_c^{Ni}}$$



$\Theta = 2.2°$

White beam

Transmitted

$\lambda < \lambda_C$

Borofloat glass m=0.65

reflected

$\lambda > \lambda_C$

0.84 m

λ_c = 20 Å

m Super-Mirrors

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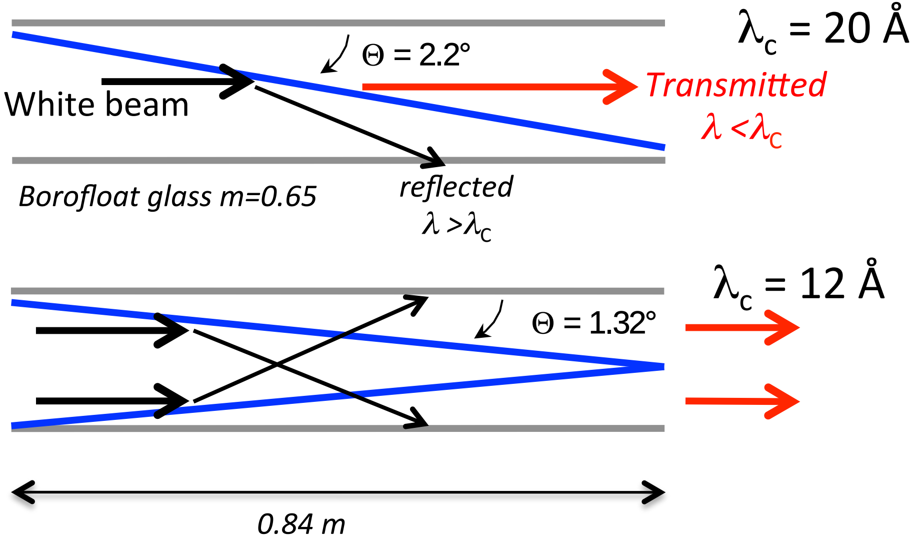

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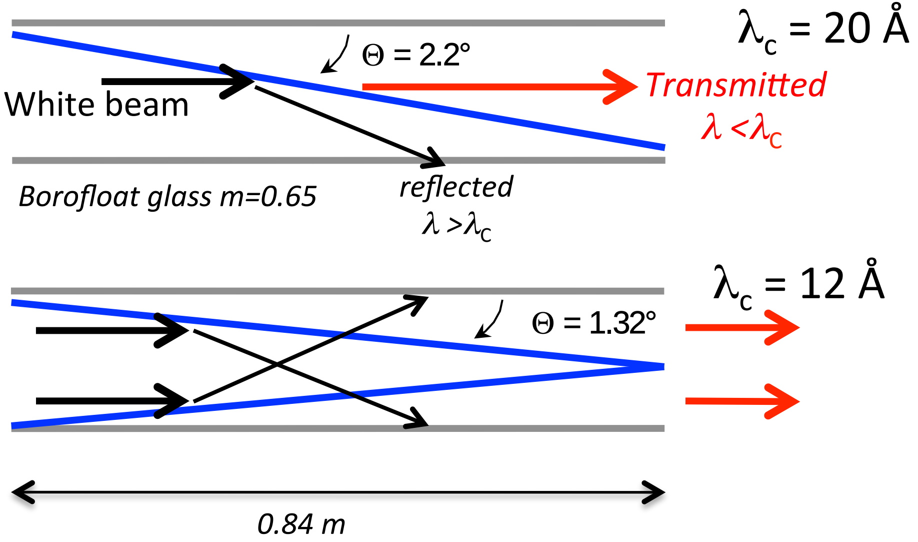
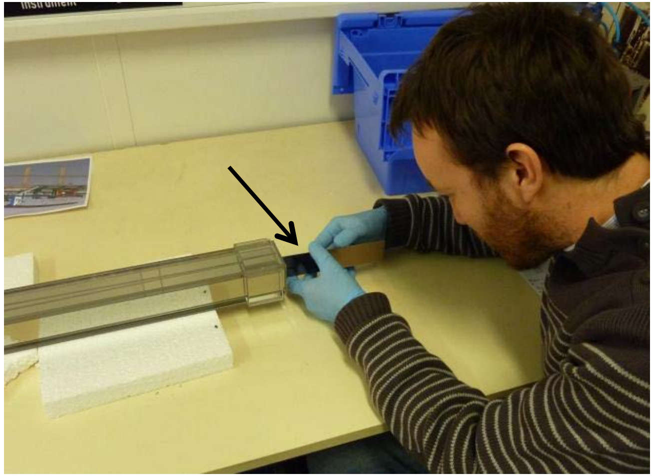
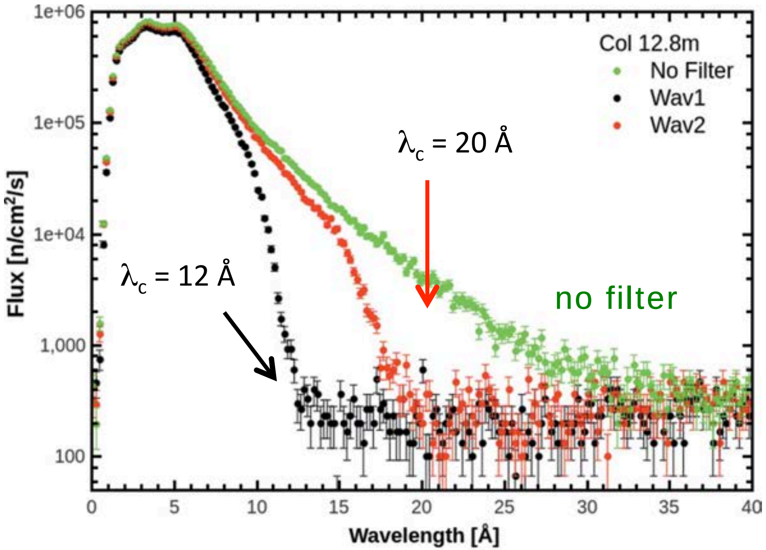




m Super-Mirrors

Long wavelength cut-off filters on D33 at I.L.L. (SANS instrument)

Neutron Flux at the sample position on D33

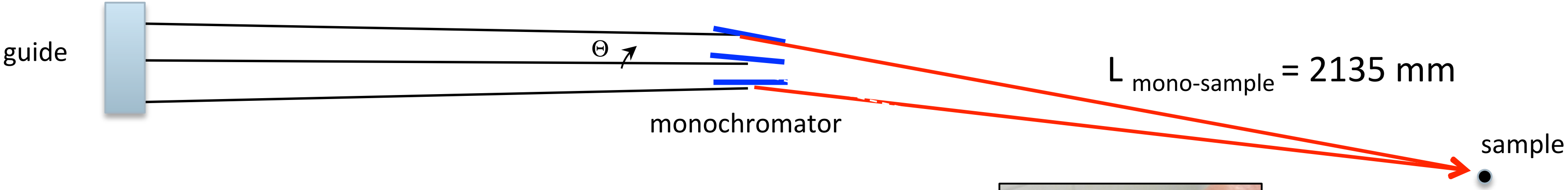


C. D. Dewhurst et al. , J. Appl. Cryst. (2016). 49, 1-14

Bragg Mirrors

Large wavelength band Monochromator on LADI (Laue diffractometer for Protein crystallography)

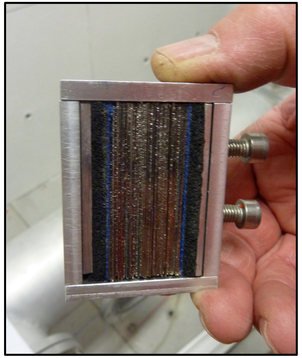
- **to produce a « monochromatic » beam at 3.5 Å with a bandwidth Δλ/λ = 20%**



Stacked Multilayer monochromator

- Beam width = 20 mm
- $\theta_B = 1.25°$ -> $d_0 = 80$ Å
- Δλ/λ = 20 % -> **graded d-spacing 74-90 Å**
- Beam size >> Sample size (1 mm)

  -> **Focusing device** (fan geometry)

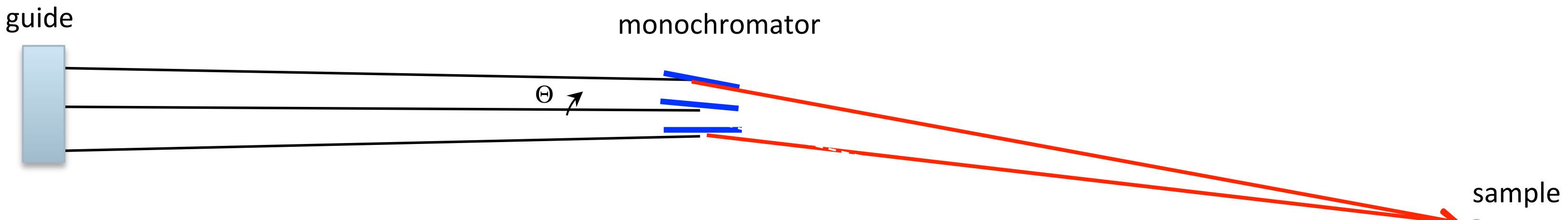


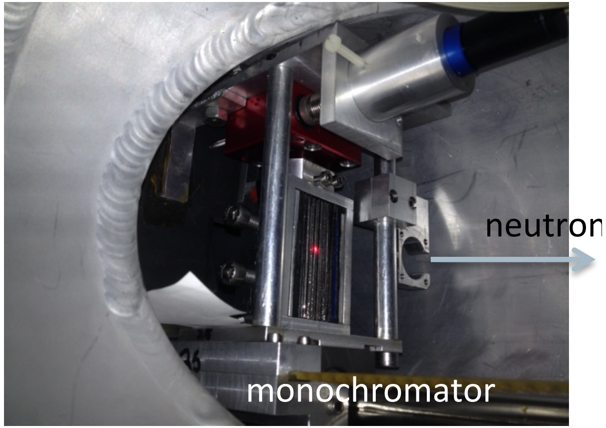
- *mirror length = 25 mm*
- *thickness 0.5 mm*
- *40 mirrors Ni/Ti*
- *Si substrate*

  (low attenuation factor)

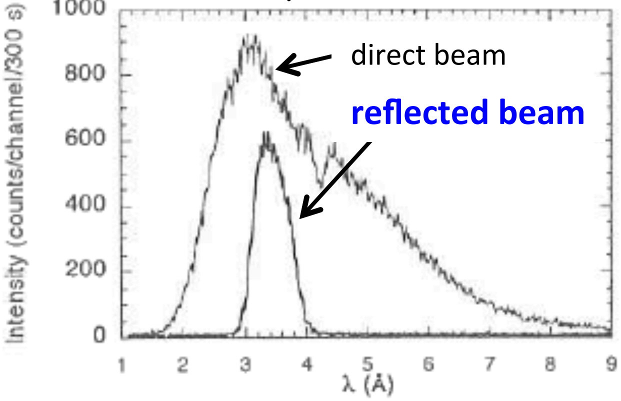
Bragg Mirrors

Large wavelength band Monochromator on LADI
(Laue diffractometer for Protein crystallography)





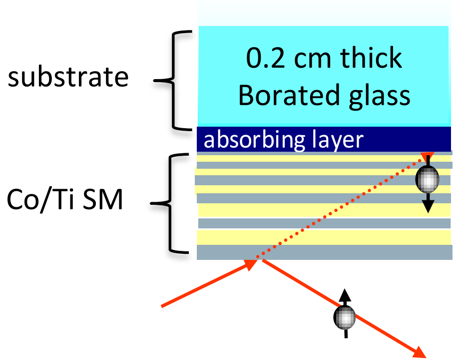
TOF spectra on LADI



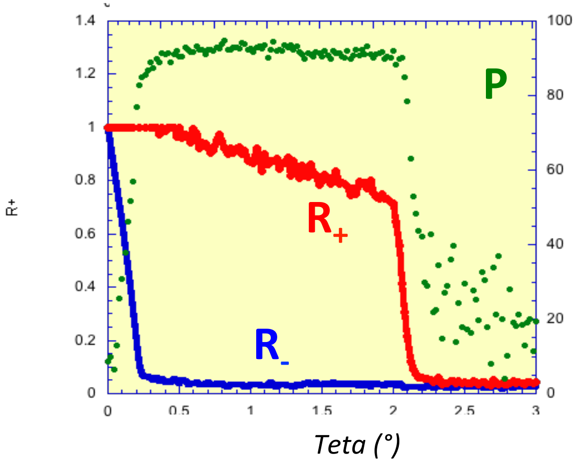
- $\lambda_0 \approx 3.5$ Å
- $\Delta\lambda/\lambda \approx 20\%$
- Reflectivity ≈ 75%

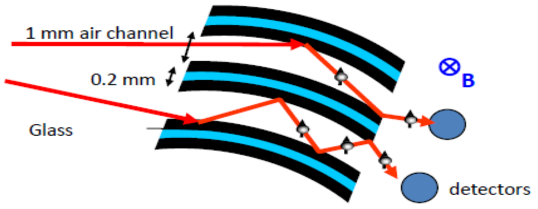
Polarizing Co/Ti Super-Mirrors

Analyzer for wide angle spin echo spectrometer (WASP)



Co/Ti m= 2.8





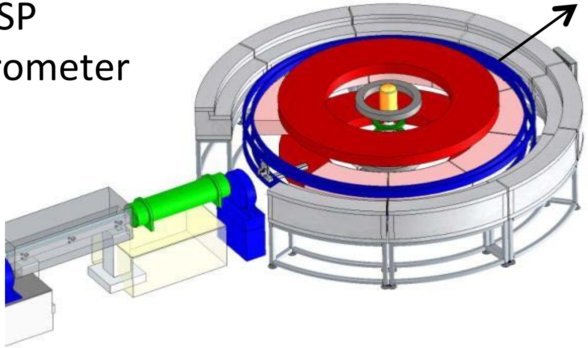
C *bender*

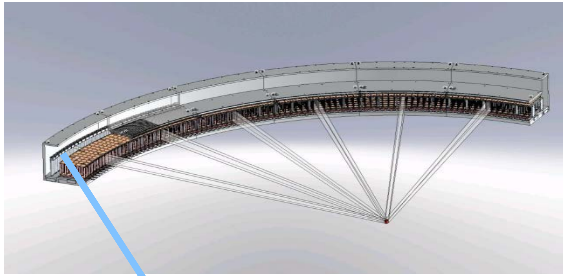
- **C bender - optimized for λ = [4 -12 Å]**
- Neutrons propagate into air
- Curvature R = 7m to avoid direct line of sight : at least on reflection !
- Double sided mirrors (h 141 x w 254 mm^2)

Polarizing Co/Ti Super-Mirrors

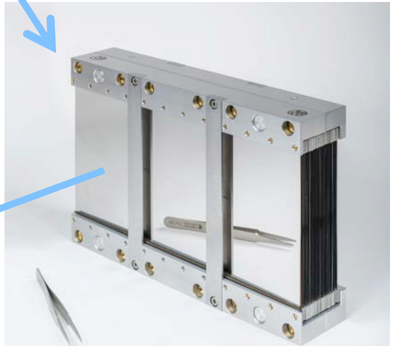
Analyzer for wide angle spin echo spectrometer (WASP)

WASP spectrometer





90° analyzer



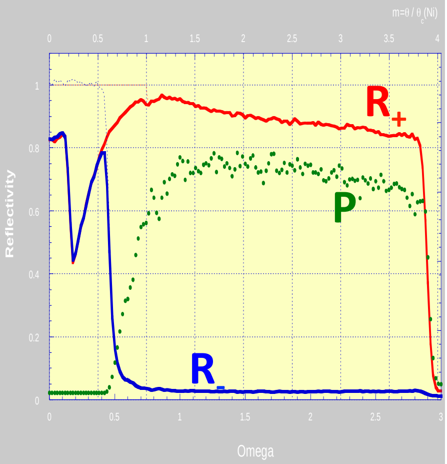
Analyzer 1° = 37 CoTi SMs

CoTi Super Mirror

Polarizing Fe/Si Super-Mirrors at I.L.L.

S bender Polarizer for neutron reflectometer D17

- **to produce a polarized cold neutron beam**



m = 3.8 Fe/Si

R+ > 80%

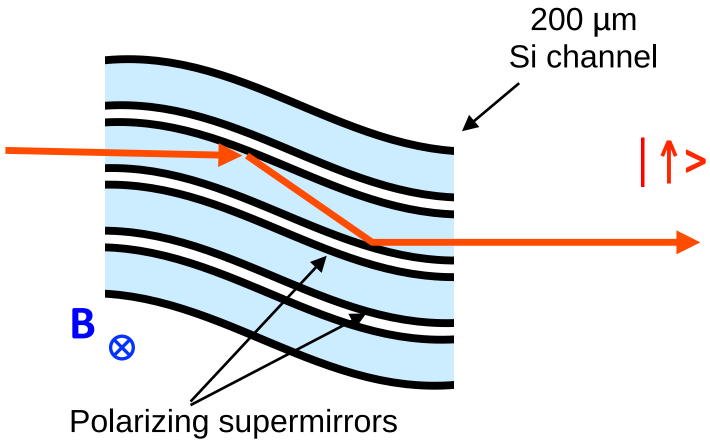
m=3.8 Fe/Si SMs

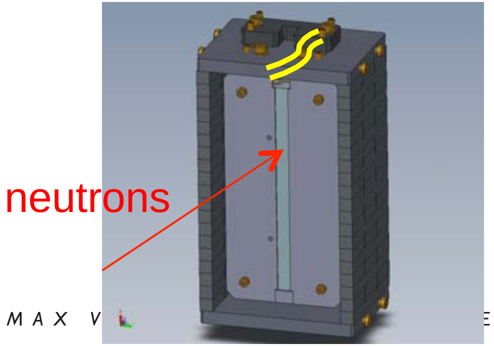
on 0.2 cm thick Si substrate

- Spin |↑> transmitted
- Stack of 60 blades

(l 50 mm; h 160 mm; w 10 mm)

- $P_{neutrons}$ > 95% (5.5 Å)
- T ~ 40% of the good neutrons

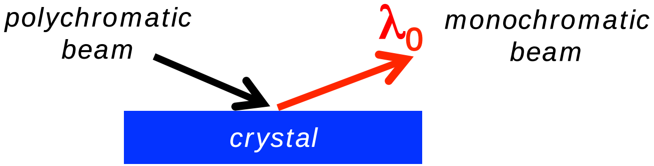




Mosaic Crystals

Bragg Diffraction – Monochromators & Analyzers

Use of single crystals to select a given wavelength band according to the Bragg's Law



$$2\, d_{hkl}\, \sin\theta_B = n\lambda_0$$

d= distance of the lattice (hkl) planes

θ_B = Bragg angle

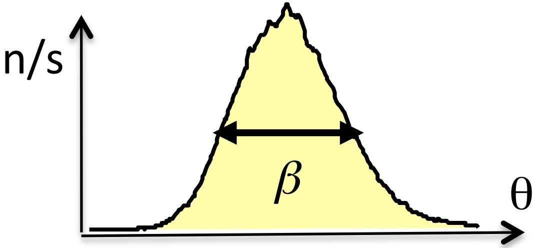
- The relative bandwidth $\Delta\lambda/\lambda$ is given for Gaussian distributions by $\Delta\lambda/\lambda = (\alpha^2 + \beta^2)^{1/2}\cot\theta_B$

  α : *divergence of the primary beam*

  β : *full width at half maximum of the neutron "rocking curve" or neutron mosaic spread*

If $\alpha \sim \beta$, the resolution and intensity are said to be optimised

- **Mosaic Crystal**, i.e. crystal with defects such as dislocations, must be used **to match the divergence** α of primary beam which is typically 0.2°- 0.8°



Mosaic Crystals

Good Materials should have

- ☐ ***High neutron reflectivity*** *$\propto$ scattering power Q* $Q = (F_{hkl}e^{-W}/V)^2 \lambda^3/\sin 2\theta_B$
  - ➢ Large structure factor F_{hkl}
  - ➢ High coherent scattering length b_{coh} ($F_{hkl} \propto b_{coh}$)
  - ➢ Small unit cell Volume & compact structures = Cubic, Diamond
  - ➢ d-spacing optimized for a given wavelength (d ~ λ)

- ☐ low incoherent scattering length → *low background*
- ☐ low absorption cross sections → *small attenuation*
- ☐ *No higher orders*

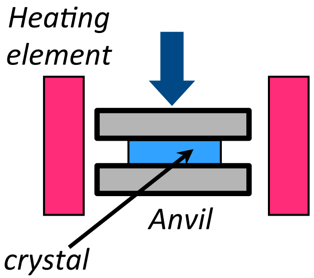
➢ **Availability of large single crystals** *with suitable width and uniform mosaic block distribution !*

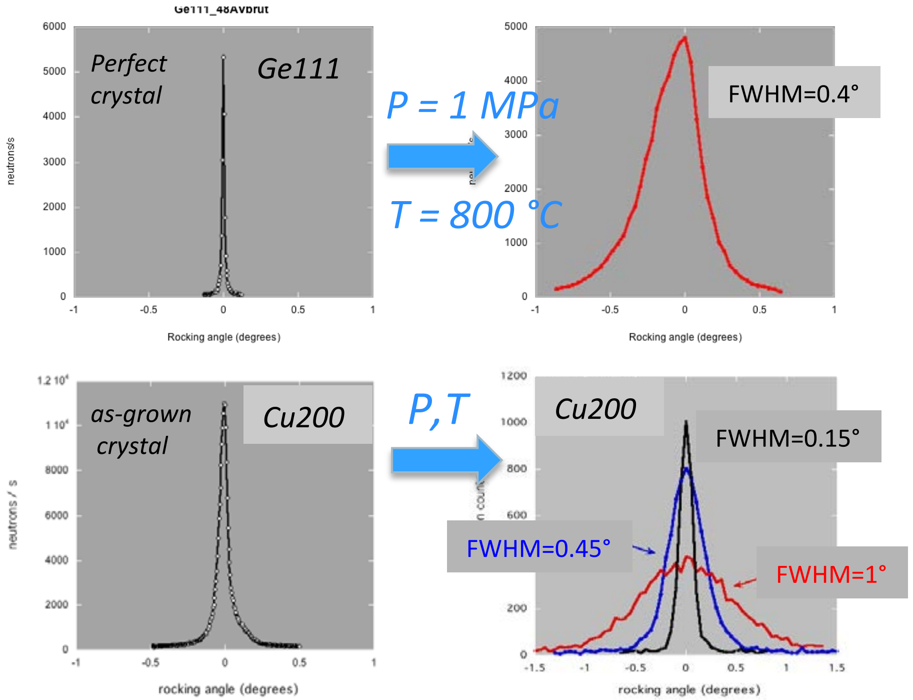
Mosaic crystals for neutron monochromators

Crystal	orientation	Crystal Mosaic	Neutron Energy	Application	*Supplier*
HOPG (C) d_{002}= 3.35 Å	HOPG(002)	0.5°- 3°	Cold Thermal	High flux	*Panasonic, Optigraph, Momentive*
Cu d_{111}= 2.08 Å	(111) (220) (200)…	0.01° - 3°	Hot Thermal	High resolution or high flux	*I.L.L. (or ?)*
Si d_{111}= 3.13 Å	(111) (113)…	Perfect	Cold Thermal	High resolution	*many !*
Ge d_{111}= 3.26 Å	(111) (113)…	< 0.4°	Cold Thermal	High resolution	*I.L.L. (or ?)*
KC_8 d_{002}= 5.3 Å	(002)	2°- 5°	Cold	High flux	*I.L.L.*
Heusler Cu_2MnAl	(111)	0.2 – 0.6 °	*Hot* *Thermal*	*Polarized neutrons*	*I.L.L.*

Mosaic Crystals

Control of the mosaic distribution by plastic deformation (I.L.L.)





Peak reflectivity at λ = 3.4 Å

R_{exp} = 50-55% (FWHM=0.4°)

$R_{exp} \approx 70$ % of R_{th}

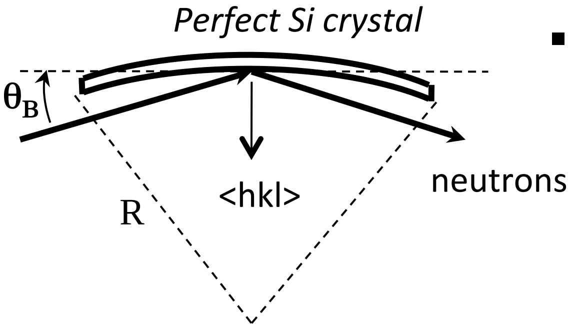
Peak reflectivity at λ = 1.1 Å

R_{exp} = 40-45% (FWHM=0.3°)

$R_{exp} \approx 80\text{-}90$ % of R_{th}

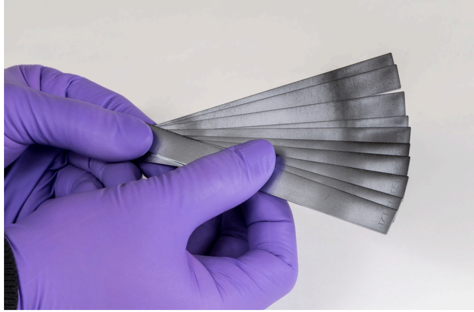
Mosaic Crystals

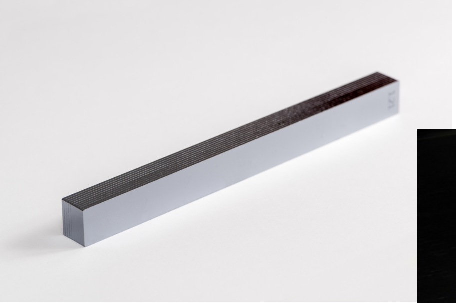
Bent perfect Silicon crystals (I.L.L.)

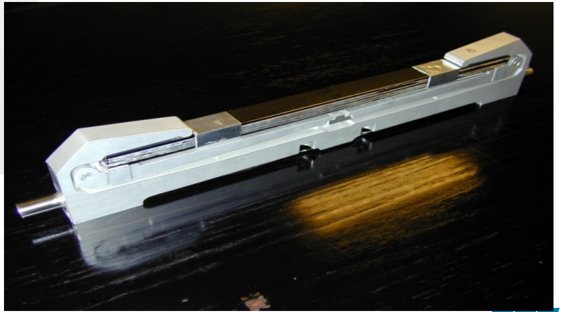


Stack of thin wafers to allow bending

- wafer thickness = 1 mm
- 10 wafers to get t = 10 mm (or more)
- Curvature : flat to $R_H \approx 2$ m



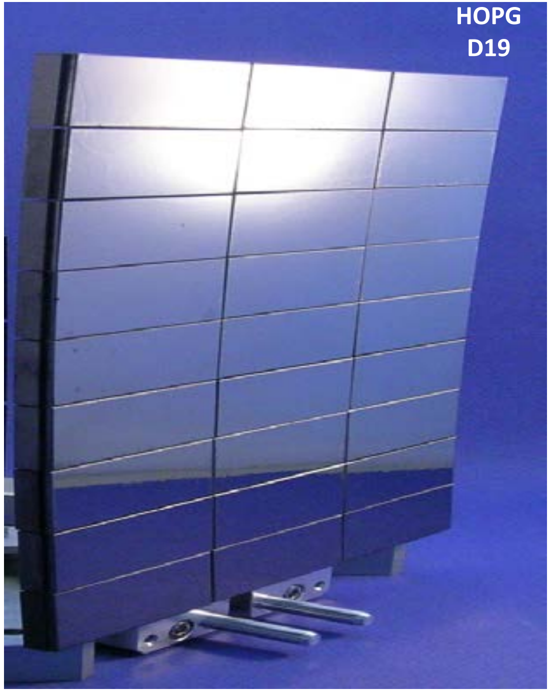




Effective mosaic δ (rad) $\delta = \cot(\theta_B)\, t / R$

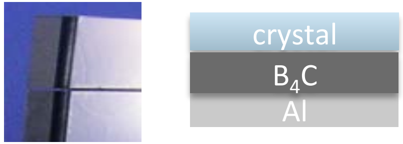
t = total crystal thickness
R = radius of curvature
θ_B = Bragg angle

Monochromators



- Large effective area to cover the direct beam

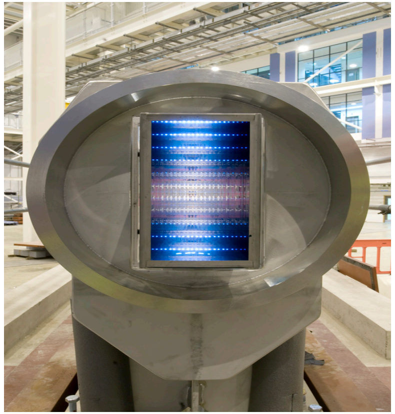
  Assembly of mosaic crystals
- **FWHM ~ beam divergence** 0.2 < FWHM < 0.6 °
- **Crystal size ~ Sample size** (1 to 10 cm^2)
- Crystals fixed onto specific mechanics
- $^{10}B_4C$ is used to reduce background and activation
- Orientation accuracy ± 0.03°

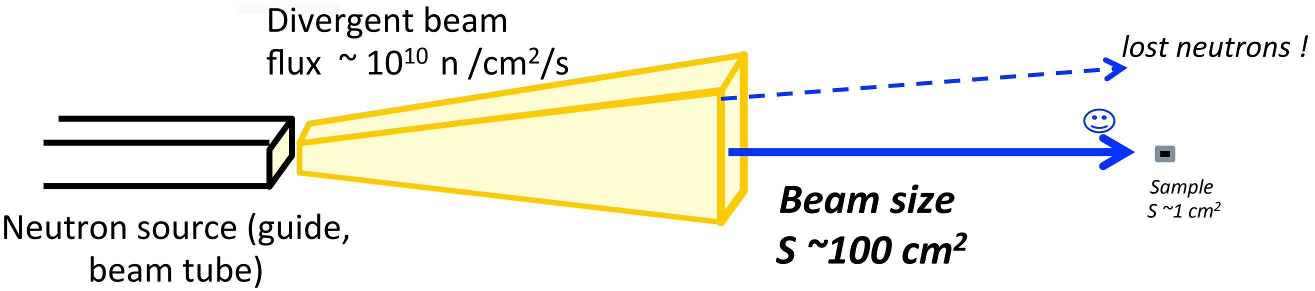


crystal + support

- Vertical Focusing
- Horizontal Focusing (Triple axis spectrometers)

Focusing devices





Divergent beam
flux ~ 10^{10} n /cm^2/s

lost neutrons !

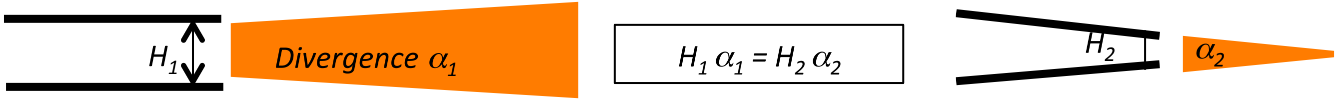
☺

Neutron source (guide,
beam tube)

Beam size
S ~100 cm^2

Sample
S ~1 cm^2

- Focusing devices are used to increase the neutron flux at the sample position. However, the increase of neutron flux implies a degradation of the angular resolution. (Liouville's theorem !)



H_1

Divergence α_1

$H_1 \alpha_1 = H_2 \alpha_2$

H_2

α_2

Focusing Devices

Principles

Vertical Focusing

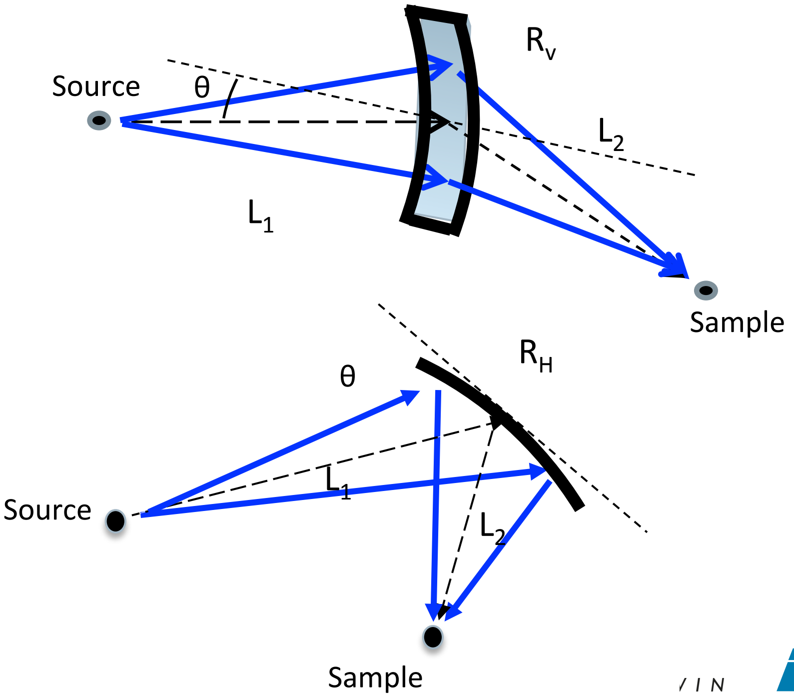
$$\frac{1}{R_v}=\frac{1}{2\sin\theta}\left(\frac{1}{L_1}+\frac{1}{L_2}\right)$$

- Image size ~ crystal size (height)
- **Gain in flux ∝ compression factor**
- **Increase of vertical angular divergence**
- $R_v \propto \lambda$

Horizontal Focusing

$$\frac{1}{R_H}=\frac{\sin\theta}{2}\left(\frac{1}{L_1}+\frac{1}{L_2}\right)$$

- **Horizontal focusing affects Q resolution**
- $R_h \propto 1/\lambda$



Focusing Devices

Monochromators for neutron Diffractometers

Production of a fixed neutron wavelength at a given take-off angle

Ge (335) - λ = 1.6 Å
crystal mosaic = 0.2 °
High Resolution

Ge (111)- λ = 3.15 Å
crystal mosaic = 0.4 °
High Resolution

Ge monochromator is well suited for high resolution neutron diffractometers

D20 (high intensity difffratometer with variable resolution) - D2B & D1B (powder diffractometers)

- Large dimensions Heigth 300 mm
  Width 50-120 mm
- **no λ/2 contamination (odd reflections)**
- Fixed vertical focusing $R_v = 2 L_{MS} \sin\theta_B$
  D20 : R_v = 3200 mm
  D2B : R_v = 4600 mm

Focusing Devices

Monochromators for neutron Diffractometers

Single crystal diffractometer
HOPG (002) - λ = 2.4 Å
crystal mosaic = 0.5 °
High Flux

Single crystal diffractometer
Cu(200) - λ = 1.2 Å
crystal mosaic = 0.2 °
High Resolution

HOPG monochromator

- Provides high neutron flux (thermal & cold neutrons)
- ▪ λ/2 contamination (002 reflections)
- ➢ Use of HOPG Filter !
- ▪ Fixed vertical focusing

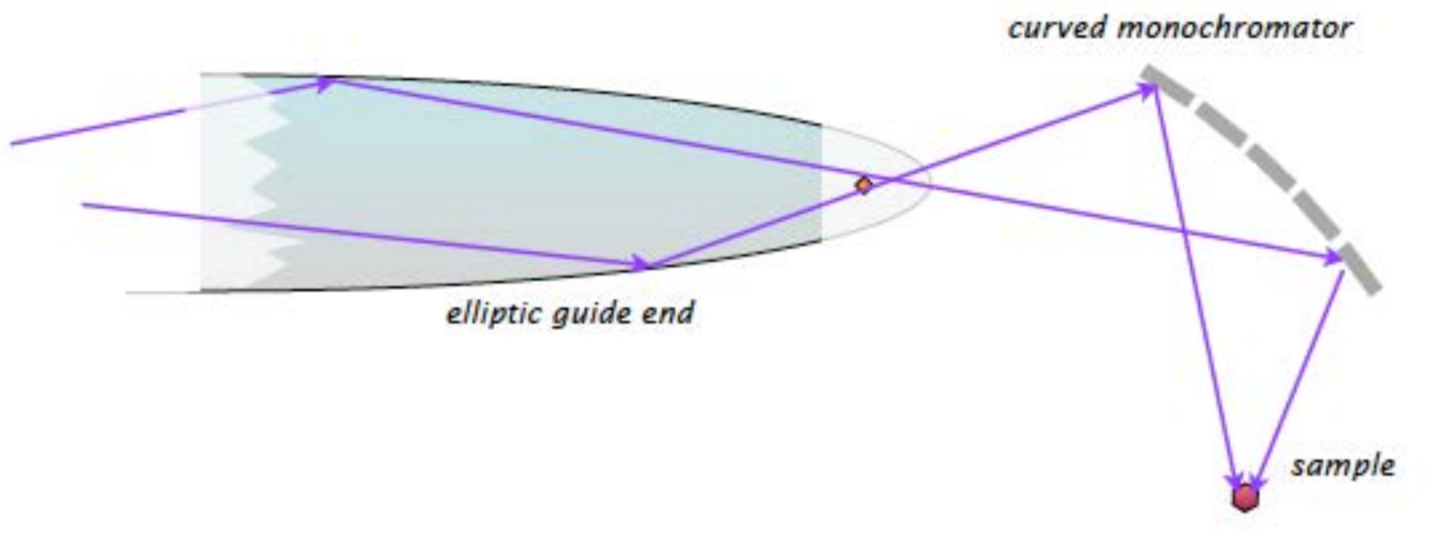
Cu monochromator

- ▪ provides high neutron flux or high resolution (hot & thermal neutrons)
- ▪ λ/2 contamination
- ➢ use of HOPG Filter !
- ➢ thermal neutron guide cut-off

Focusing Devices

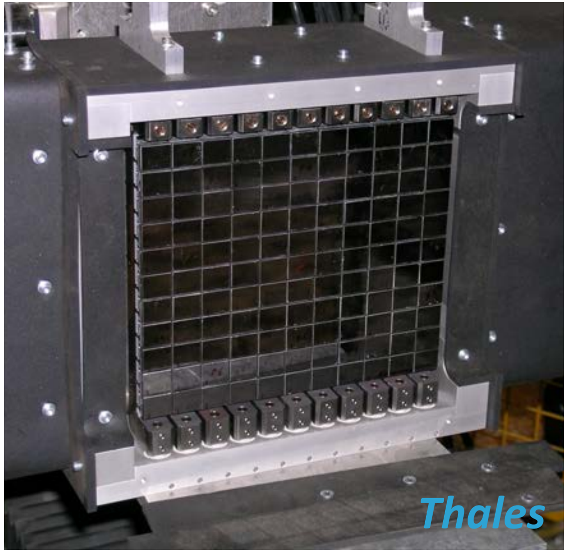
Monochromators for Triple Axis Spectrometers

- **Optimization of instrument performances for a wide energy range**
- Variable horizontal and vertical curvature
- Focusing monochromator is used in combination with focusing guide (virtual source) see previous presentation from J. Kulda !

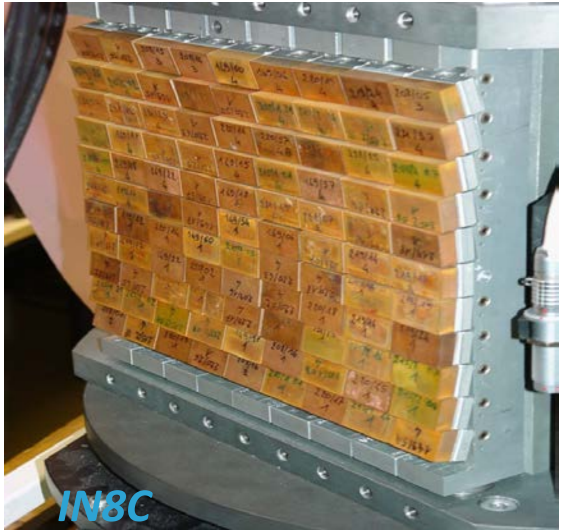


Focusing Devices

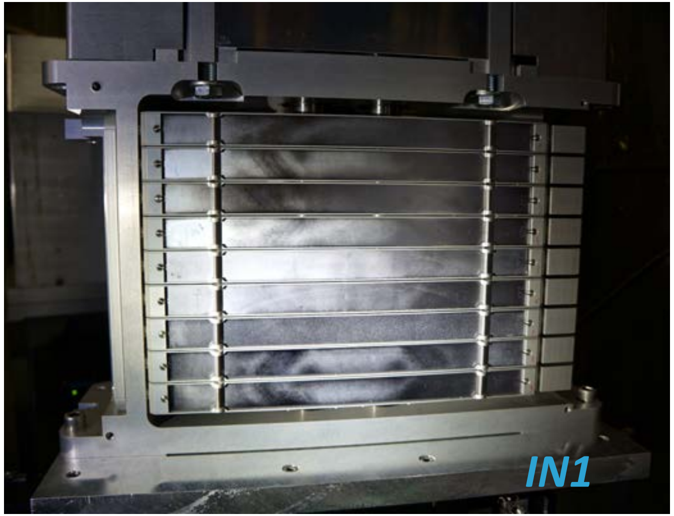
Monochromators for Triple Axis Spectrometers



HOPG(002) monochromator
crystal mosaic = 0.5 °
cold neutrons



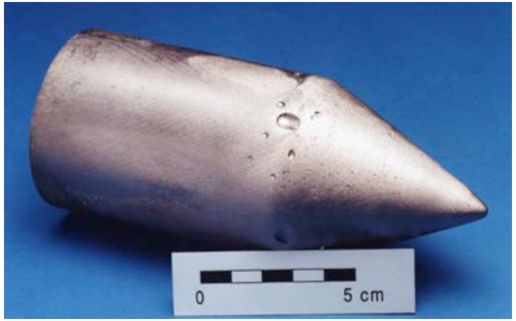
Cu(200) monochromator
crystal mosaic = 0.3 °
High Flux – Thermal neutrons



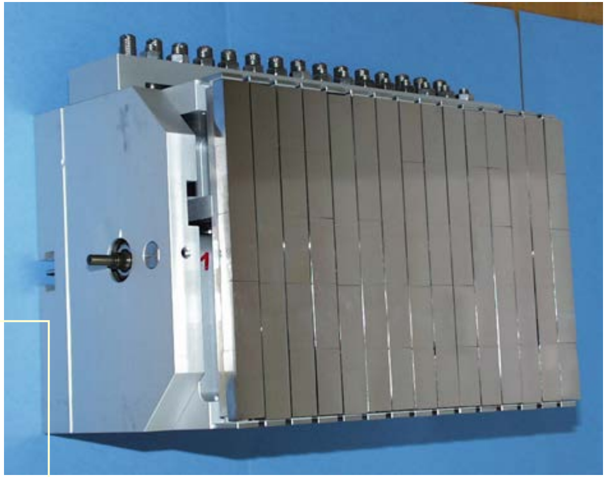
Si(113) monochromator
bent perfect crystals
Hot neutrons

Monochromator for polarized neutrons

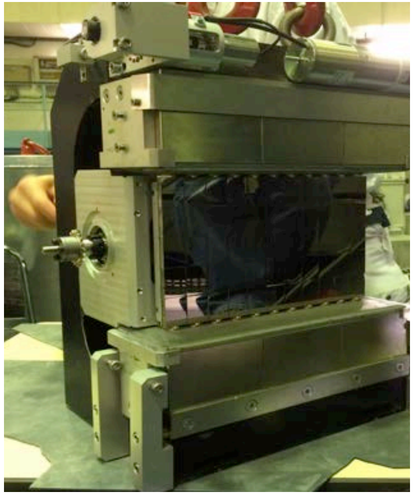
Heusler Cu_2MnAl mosaic crystals



Fixed Vertical Curvature – Variable Horizontal Curvature



IN20
(Thermal neutron 3 axis spectrometer)

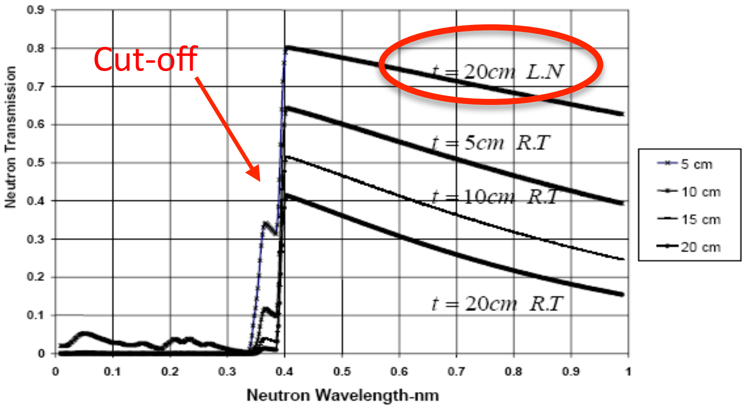


THALES
(3-Axis for low energy spectroscopy)

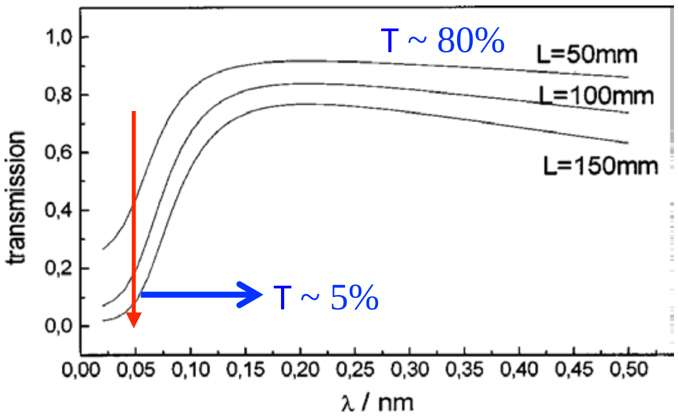
• (111) reflection	$F_{111N} = -F_{111M}$
• Mosaic	$0.2° < \text{fwhm} < 0.6°$
• Reflectivity	$R_{exp} \approx R_{theor}$
• Polarization	$P > 92\ \%$

Neutron Filters

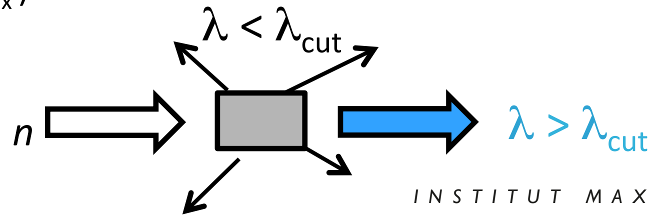
Neutron filters are used to select out unwanted neutrons



Polycristalline Beryllium at 77K
is used to select out neutrons of $\lambda < 4$ Å
(Cut-off at $\lambda = 2d_{\text{Be max}}$)



Perfect Saphirre crystal
is used to select out fast neutrons of $\lambda < 0.5$ Å
➢ Reduction of background



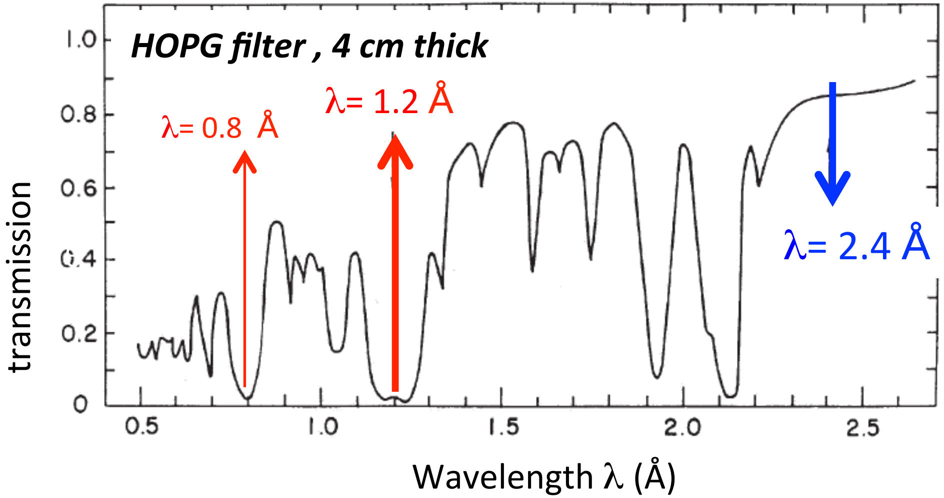
$\lambda < \lambda_{cut}$

n

$\lambda > \lambda_{cut}$

Neutron Filters

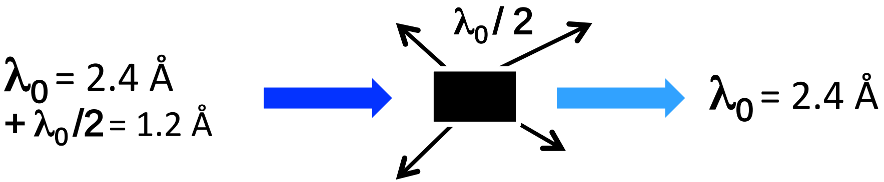
Neutron filters are used to select out unwanted neutrons



HOPG crystal (Highly Oriented Pyrolitic Graphite) is commonly used for eliminating higher-order contamination of a monochromated neutron beam

PG filter has strong attenuation at 1.2 Å but passes 2.4 Å

- T > 80 % at 2.4 Å
- T < 1% at 1.2 Å



λ_0 = 2.4 Å
+ $\lambda_0/2$ = 1.2 Å

$\lambda_0/2$

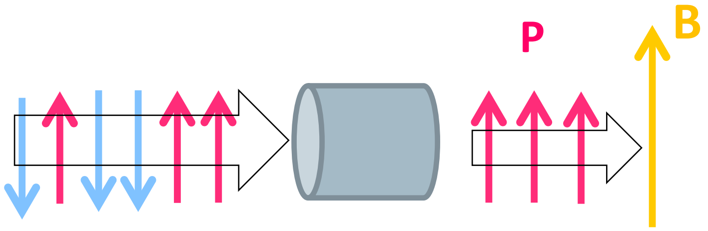
λ_0 = 2.4 Å

Neutron Filters – Polarized Neutrons

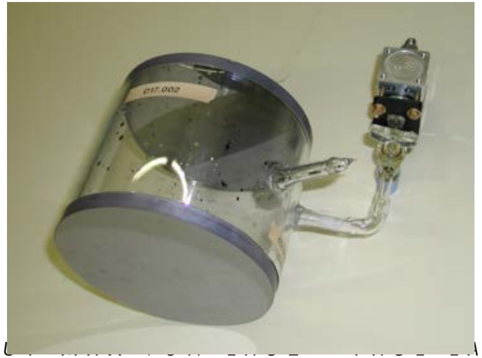
^{3}He spin filters at I.L.L.

- **Absorption cross section of ^{3}Helium nuclei**
  - If the nuclear spin of He and the neutron spin are parallel, $\sigma_{a\uparrow\uparrow} \approx \mathbf{0}$
  - If the nuclear spin of He and the neutron spin are anti-parallel, $\sigma_{a\uparrow\downarrow} \approx 6000$ barns

- For fully polarized ^{3}He (*$P_{He} = 1$), one spin state goes through the filter with zero* absorption. The other spin state is almost fully absorbed since σ = *6000 barns → polarized neutron beam*



^{3}He spin filter cell



Neutron Filters – Polarized Neutrons

^{3}He spin filters at I.L.L.

- ➢ Polarization of neutron beams (hot, thermal, cold)
- ➢ ^{3}He spin filters provide an unique tool to perform XYZ polarization analysis

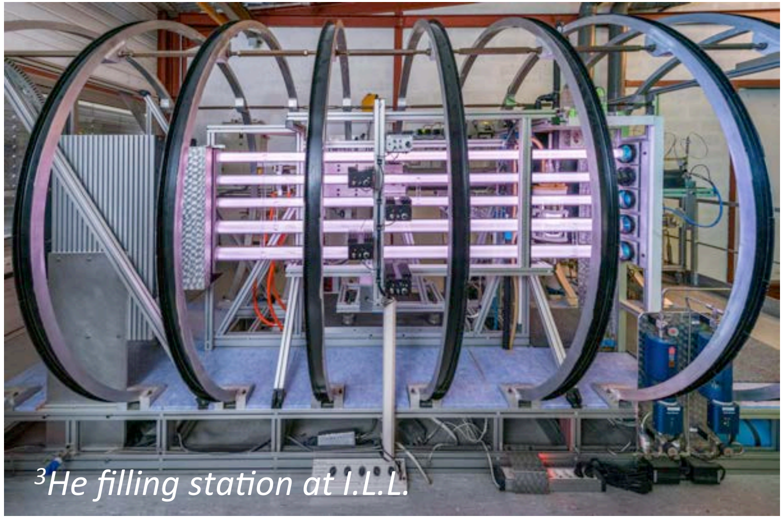
Polarization of ^{3}He: $P(t)=\exp(-t/T_1)$ and $$\frac{1}{T_1}=\frac{1}{T_d}+\frac{1}{T_w}+\frac{1}{T_m}$$

$$T_d[\mathrm{h}]=\frac{750}{P[\mathrm{b}]} \quad T_w \approx 200-400\,\mathrm{h} \quad T_m[\mathrm{h}]=\frac{P[\mathrm{b}]}{7000}\left(\frac{\partial B/\partial r[\mathrm{cm}]}{B}\right)^{-2}$$

- ▪ To perform polarized neutrons experiments : $T_1 > 100\mathrm{h}$
- ➢ High quality ^{3}He spin filters (Polarization, T_w)
- ➢ Homogeneous magnetic field

Production of ^{3}He spin filters at I.L.L.

Metastability Exchange Optical Pumping (MEOP)



^{3}He filling station at I.L.L.

- Production rate > 1 bar-litre/h
- Final pressure up to 4 bar
- Polarization of ^{3}He ≈ 80 %

Development of ^{3}He cells

- Banana shaped
- Coverage angle up to 125 °
- Si windows : Reduction of background
- Cs coating
- **T_w > 200 h**

Conclusion

- **Neutron Optics define beam properties**
  - Direction, Divergence, Wavelength, Energy, Polarization
  - Angular Resolution, Wavelength resolution, Energy resolution
- Vertical focusing devices allow the optimization of the neutron flux at the sample position
- Double variable focusing devices allow the optimization of instrument performances for a wide energy range

➢ Since the power of the source is low, **neutron optical components must be of high quality** and **properly designed**

➢ Neutron Optics obey to Liouville's Theorem **:** It costs flux to increase resolution and it costs resolution to increase flux

The optimization of instrument performances is always

a compromise between flux and resolution

Thank You for your attention