Recent achievements in advanced diffractive optics for neutron monochromators

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NEUTRONS FOR SOCIETY

I N S T I T U T   L A U E   L A N G E V I N

P. Courtois

Service for Neutron Optics

Recent Achievements in advanced diffractive optics for neutron monochromators at ILL

- Basics in mosaic crystal monochromators
- New Monochromators under the scope of the modernization programme at ILL
  - Instruments on the new H24 guide : D10^{+}, IN13^{+} and XTREMED
  - PANTHER and IN20
- Development of innovative optics from perfect Si crystals

Crystal for neutron monochromator

- To select a given wavelength band according to the Bragg's Law $\mathbf{2\ d_{hkl}\ sin\theta_B = n\lambda_0}$
- To match the neutron beam divergence α which is typically 0.2°-1°

Crystal for neutron monochromator

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- Perfect crystal is not suitable since reflection range is too narrow (∽ 0.005°)

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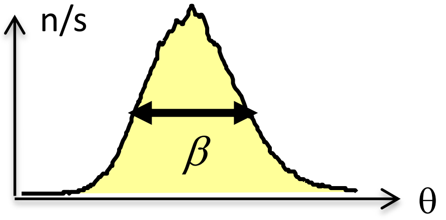
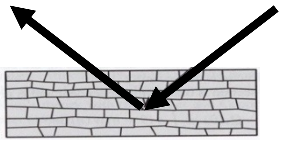
- To match the neutron beam divergence α which is typically 0.2°-1°
- Perfect crystal is not suitable since reflection range is in the order of 0.005°

Use of mosaic crystals, i.e. crystal with structural defects (dislocations …)



$$w(\theta) = \frac{1}{\sqrt{2\pi\eta}} \exp(-\Delta^2 / 2\eta^2)$$



Mosaic crystal

Bandwidth $\Delta\lambda/\lambda = \cot\theta_B \,.\, \boldsymbol{\Delta\theta}$

If Gaussian distributions we have :

$$\Delta\lambda/\lambda = (\alpha^2 + \beta^2)^{1/2} \cot\theta_B$$

α *: incoming beam divergence*

β*: neutron mosaic spread (FWHM)*

$\boldsymbol{\alpha \approx \beta}$ (flux optimization vs resolution)

- Mosaic crystals should have high neutron reflectivity, low background and small attenuation
- Large single crystals must be available ! HOPG, Cu, Si, Ge, CaF_2 and Cu_2MnAl

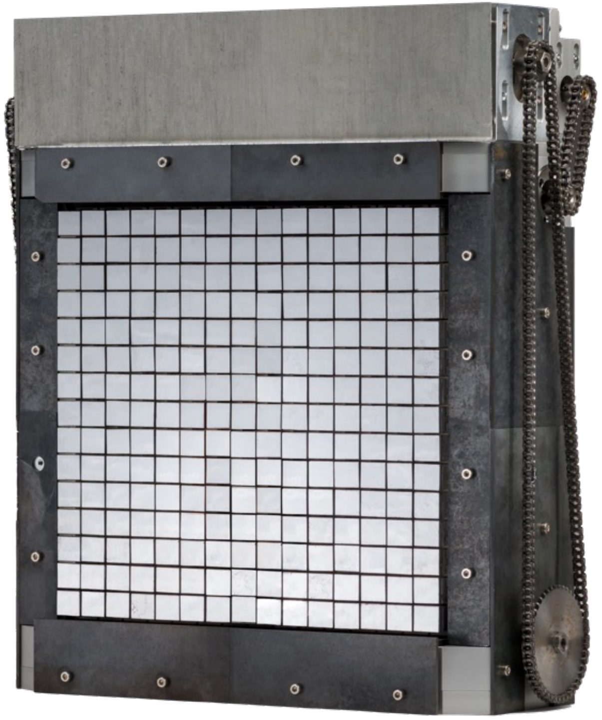
HOPG Monochromators

High neutron Flux

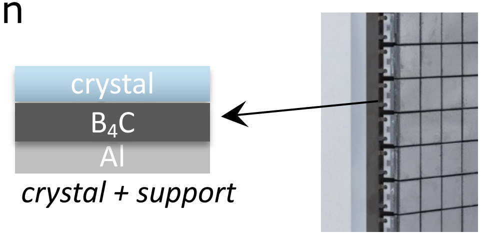
- Thermal Triple Axis Spectrometer **IN20**
- Diffractometer for extreme conditions **XTREMED**
- Single crystal diffractometer **D10+**

The double focusing HOPG Monochromator for IN20

Thermal neutron three-axis spectrometer



- Effective Area 20 x 22.3 cm^2
- 225 HOPG mosaic crystals
- Neutron mosaic FWHM = 0.5 ° - 0.6°
- $^{10}B_4C$ plate is used to reduce background and activation



crystal + support

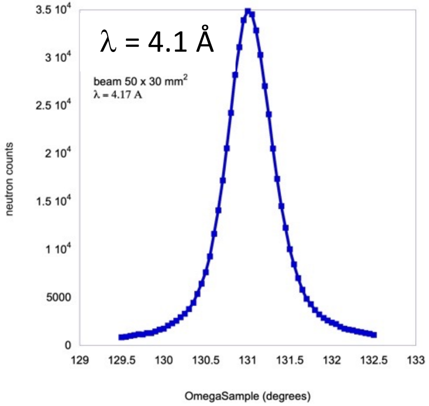
- **SMALL Crystal size = 13.4 x 14.9 mm^2**

-> Size of HOPG crystals has been chosen to optimize focusing efficiency in both direction

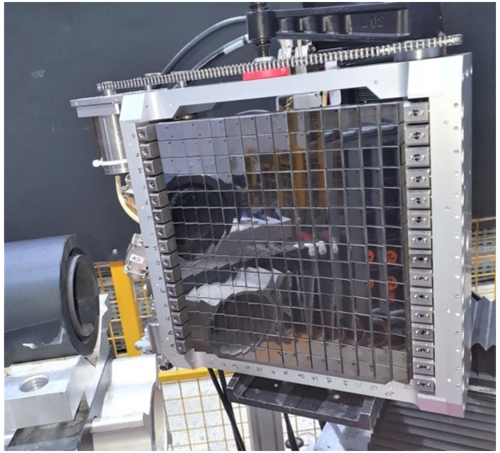
-> Crystal size ~ average sample size

The double focusing HOPG Monochromator for IN20

Crystal alignment using neutron diffraction on T13C - First results on IN20

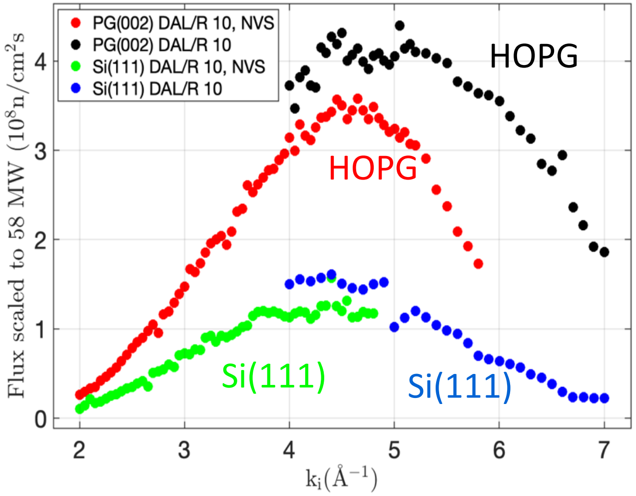


Neutron Rocking curve from HOPG(002) crystals



Monochromator alignment on T13C

Courtesy M. Enderle



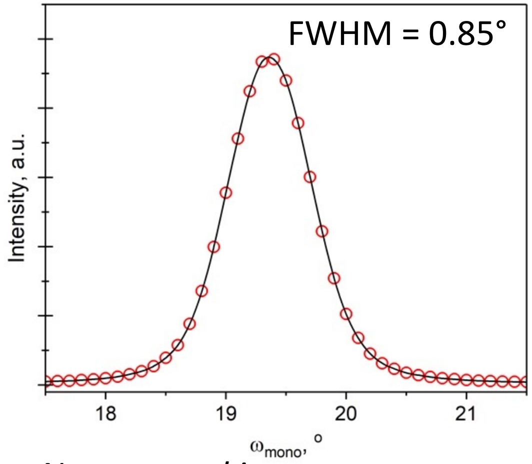
HOPG(002) vs Si(111)

- The final alignment of the monochromator was performed by neutron diffraction on T13C (450 rocking curves!)
- ➢ Orientation accuracy of each crystal: ± 0.1° (// diffracting plane). ± 0.2° (perp diffracting plane)
- First tests on IN20 : Direct comparison of performance between HOPG(002) & Si(111) monochromators
- ➢ Flux Gain up to a factor of 3 (at k_i = 4 Å^{-1} / λ = 1.5 Å)

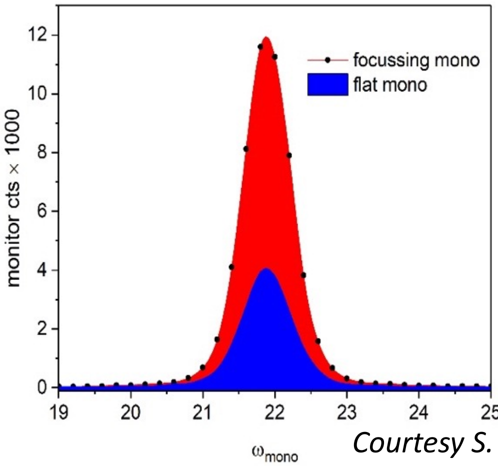
The double focusing HOPG Monochromator for XTREMED

Diffractometer for extreme conditions of pressure and magnetic field

HOPG Monochromator
81 crystals (19 x 15 x 2 mm^3)



Neutron rocking curve from the entire monochromator



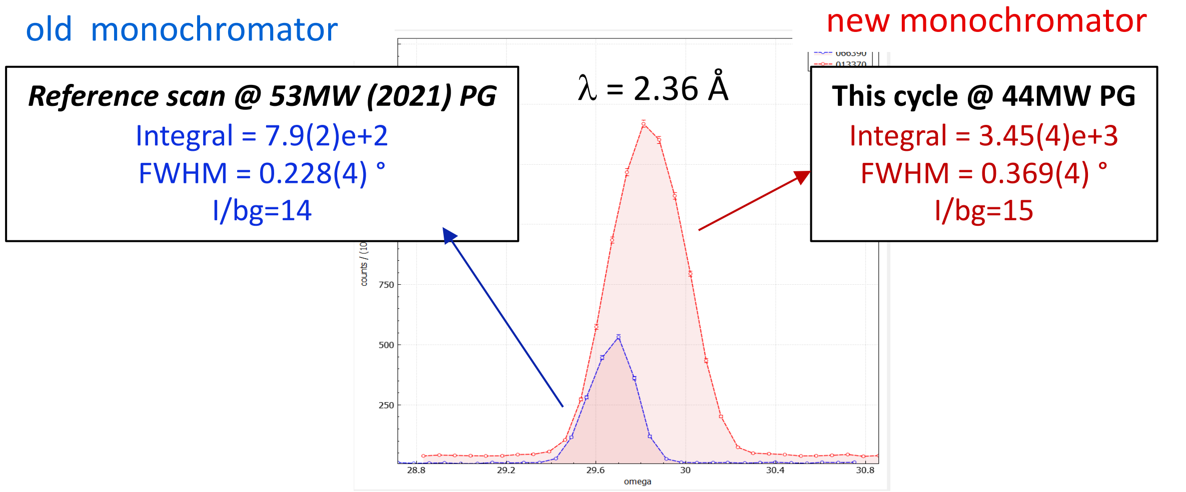
Courtesy S. Savvin

Vertical focusing vs neutron flux

- Neutron rocking curve from the monochromator shows the good alignment of HOPG single pieces
- **Vertical Focusing leads to a gain in neutron flux of a factor of 3**

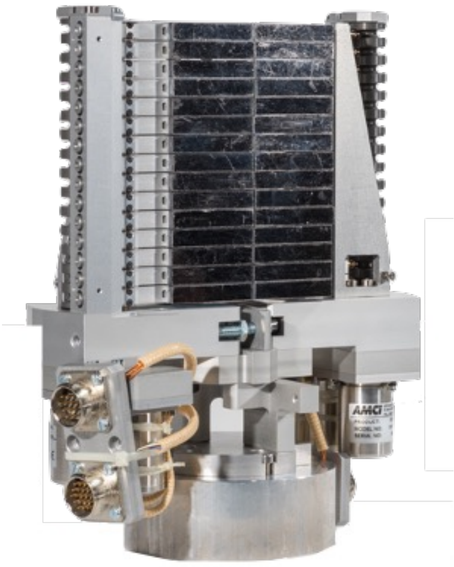
The vertical focusing HOPG Monochromator for D10+

Single crystal four-circle diffractometer with 3-axis energy analysis



Peak profile of 110 reflection from a ruby crystal

(Courtesy : B. Ouladdiaf)



HOPG Monochromator

30 HOPG crystals (42 x 8 x 2 mm^3)

- High neutrons flux Monochromator at λ = 2.36 Å
- **Gain in neutron flux of a factor of 5.3 !**
- But degradation of the resolution due to the higher m value of the new H24 guide (m=2)

Copper Monochromators

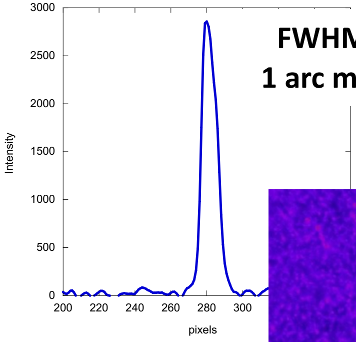
High neutron Flux or High Resolution

- Production of mosaic Cu single crystals
- New Monochromators for **PANTHER** and **D10^{+}**
- First Results on **D10^{+}**

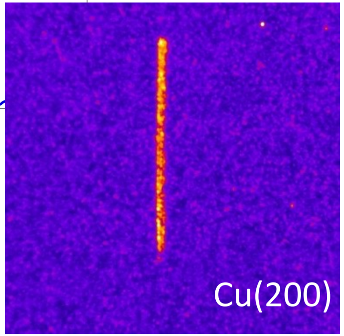
Production of mosaic Cu single crystals
Crystal growth, Characterization & Cutting

Bridgman furnace

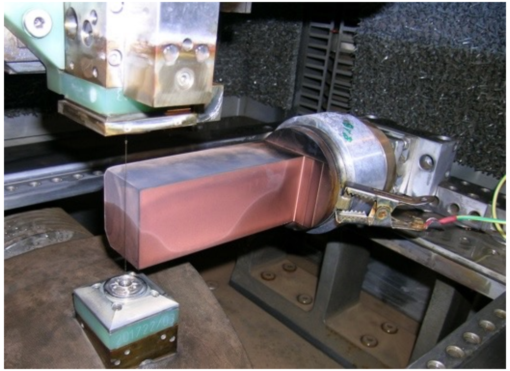
Cu single crystals
(8 Kg)



Peak profile



Hard X-Ray image



Crystal cutting
by spark erosion

- Growth of large Cu single crystals of high Quality well established at ILL, in our laboratory
- Non-destructive characterization of the as-grown crystal by Hard X-Ray diffraction (100-450 keV)

➢ The neutron mosaic spread is too narrow for neutron applications !

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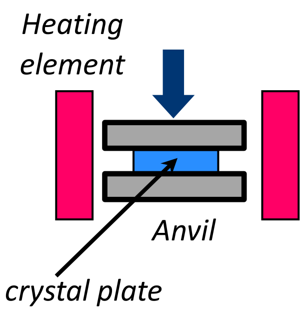
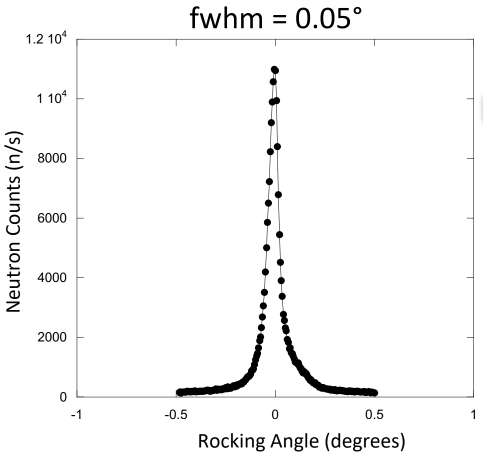
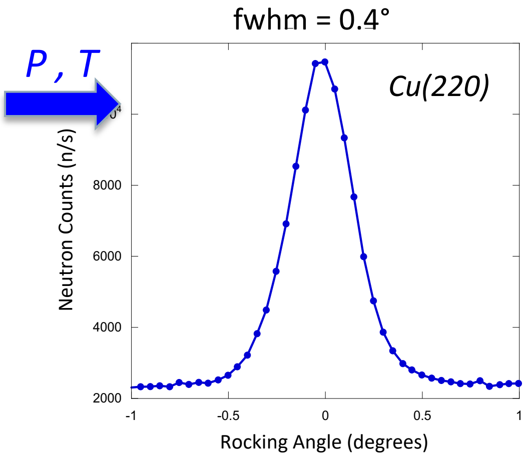
Production of mosaic Cu single crystals

Control of the mosaic distribution by plastic deformation

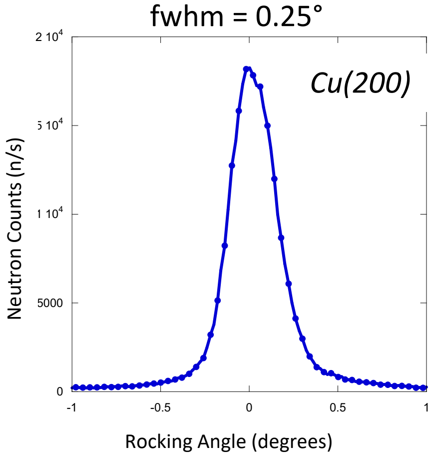




Neutron rocking curve from the as-grown crystal



PANTHER
Peak reflectivity
R_{exp} = 35-40 % (at λ = 1.1 Å)

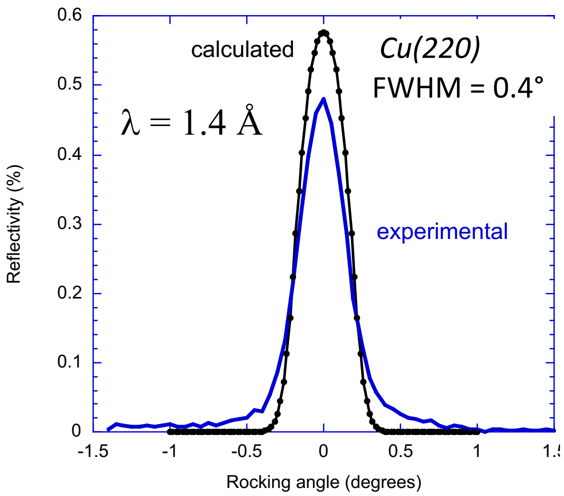


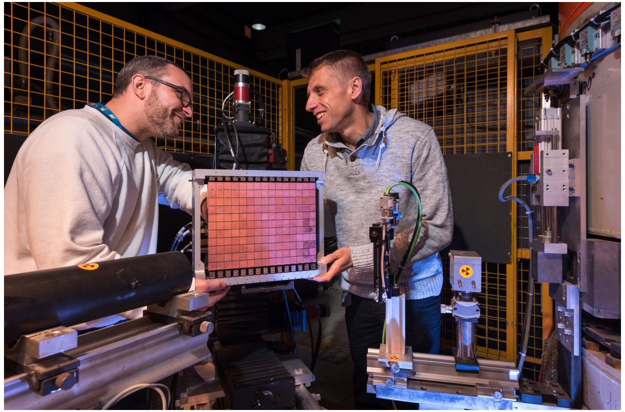
D10+
Peak reflectivity
R_{exp} = 40-45% (at λ = 1.1 Å)

- Production of high quality Cu(220) and Cu(200) single crystals with a controlled mosaic distribution
- Peak reflectivity at λ = 1.1 Å $R_{exp} \approx$ 80-90 % of R_{th}
- Construction of Cu monochromators for D10+ and PANTHER

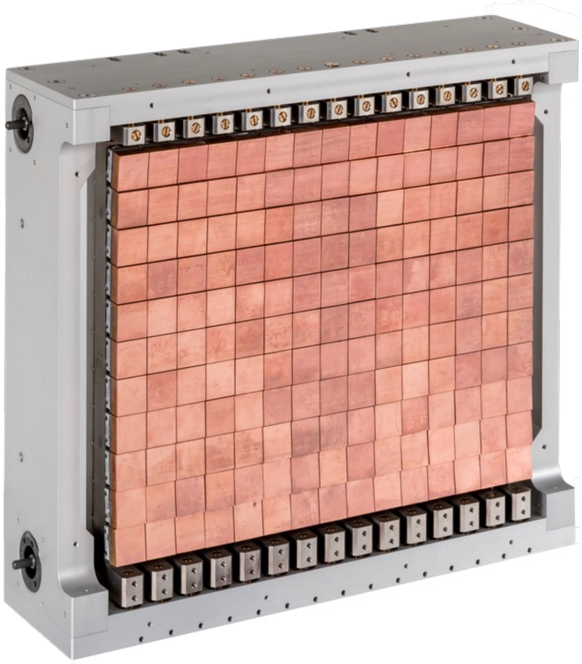
The double Focusing Cu(220)&Cu(331) Monochromator for PANTHER

Thermal neutron time-of-flight spectrometer





Crystal alignment on T13C



- Hot and Thermal Neutrons (λ = 0.5 - 2 Å)
- (hhl) reflections available, especially the Cu(331) reflection

➢ Extend the energy range to short wavelengths up to λ = 0.5 Å

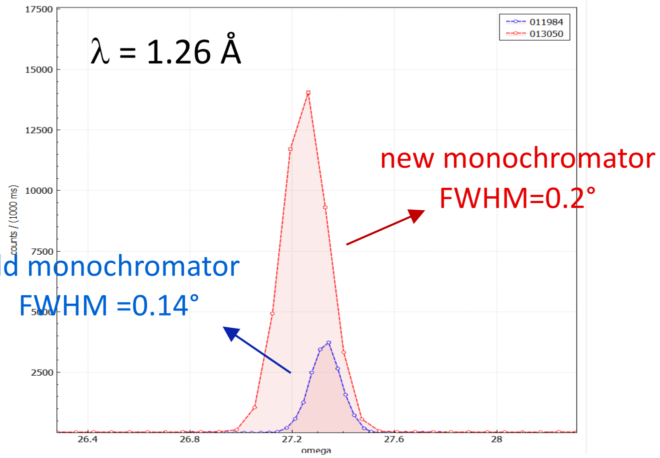
➢ Better resolution compared to the old set-up

- 165 Cu(220) crystal (20x 20 x 7mm^3)
- FWHM = 0.4°- 0.5°
- Alignment accuracy **± 0.03°**

The vertical Focusing Cu(200) Monochromator for D10+

Single crystal Diffractometer D10⁺

First results on D10+



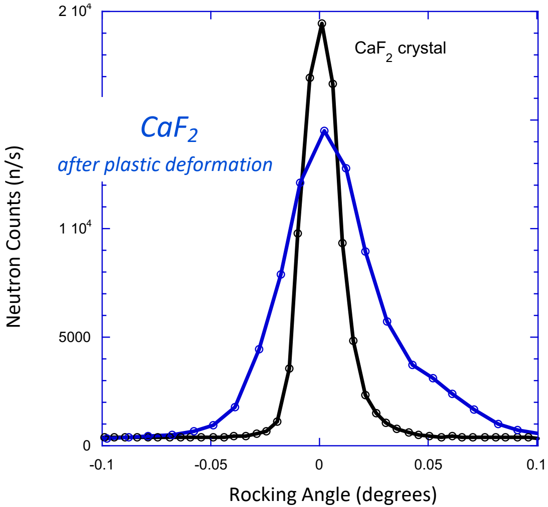
Peak profile of 110 reflection from a ruby crystal
(Courtesy : B. Ouladdiaf)

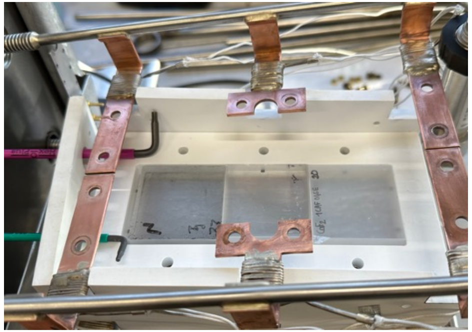
- 30 Cu(220) crystals (42 x 8 x 7 mm^3)
- FWHM = 0.25°- 0.3°
- Alignment accuracy **± 0.03°**

- High Resolution Diffractometer at λ = 1.26 Å
  - ➢ Use of Cu single crystals with a neutron mosaic spread of 0.25°
  - ➢ Intensity gain of a factor of 6.6 at λ = 1.26 Å

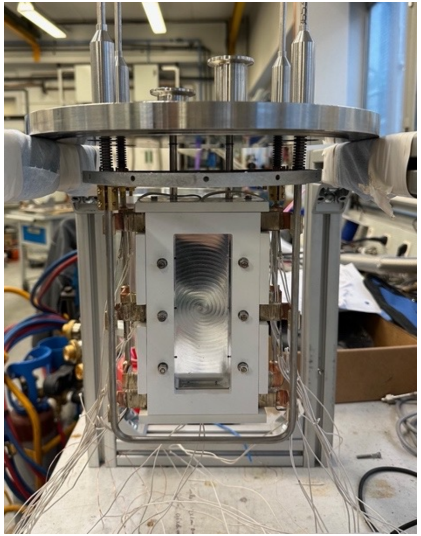
New CaF_2(224) mosaic crystals for the monochromator of IN13+

Thermal neutron backscattering spectrometer





CaF2 crystals
(45 x 45 x 10 mm³)



IN13 Cryo-furnace (100K - 450K)
(SANE - Eddy Lelièvre)

- Production of mosaic CaF_2 crystals (FWHM = 0.05°) by plastic deformation
- Cryo-furnace: variation of the temperature of the monochromator at a fixed Bragg angle

➢ Energy resolution 2 μeV at 16.5 meV

➢ Commissioning in progress !

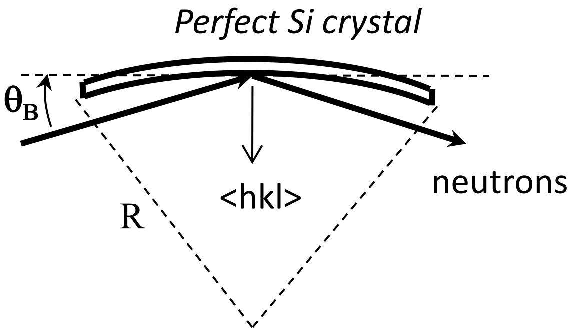
Development of Innovative neutron optics from perfect Silicon crystals

- Bent perfect Si crystals
- An Innovative Analyzer concept
- Mosaic Si crystals

Silicon Monochromator

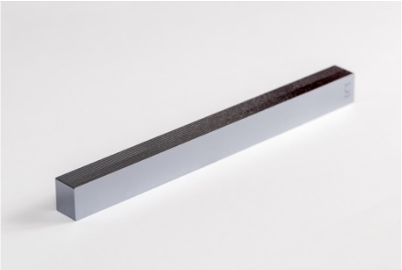
Si perfect crystal exhibits excellent properties for neutrons applications

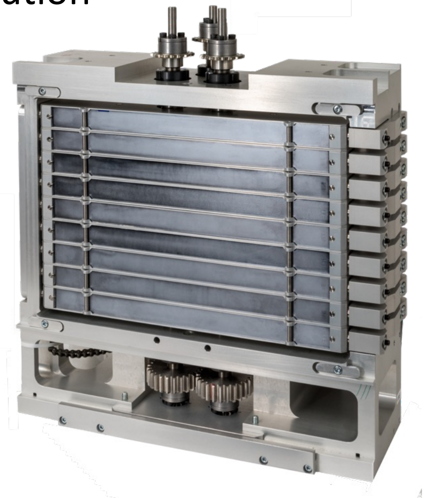
- No $\lambda/2$ contamination, low attenuation factor, no parasitic scattering
- Use of elastically bent perfect crystals to produce effective mosaic distribution



Stack of thin Si blades to allow bending

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





THALES

Si(111) Monochromator

Si blade dimensions : 270 x 19 x 1mm^3

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

t = total crystal thickness

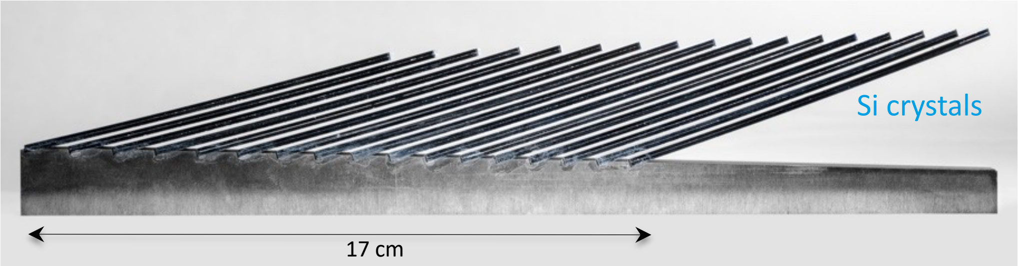
R = radius of curvature

θ_B = Bragg angle

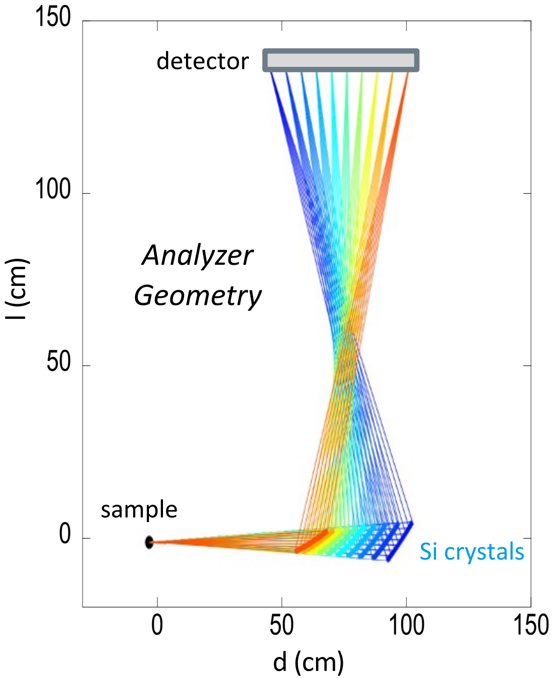
(*ex:* θ_B= 30°, t=10mm, R=2m -> $\delta = 0.5°$)

Validation of an innovative multi-analyzer concept

Multiplexed Array for Mapping on ThALES (Three Axis Low Energy Spectroscopy)



MARMOT Prototype
Si blades: length 100 mm - R = 2 m



(P. Steffens, M. Boehm)

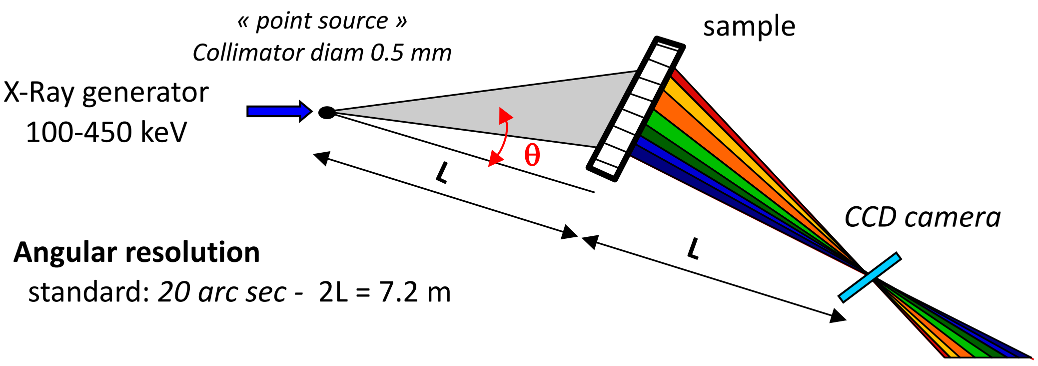
Aims to provide a continuous energy analysis from 3 to 6 meV

- Use of plastically bent perfect Si crystals (R=2m)
- Each Si crystal diffracts neutrons having a given energy range
- It requires precise alignment & positioning of each crystal

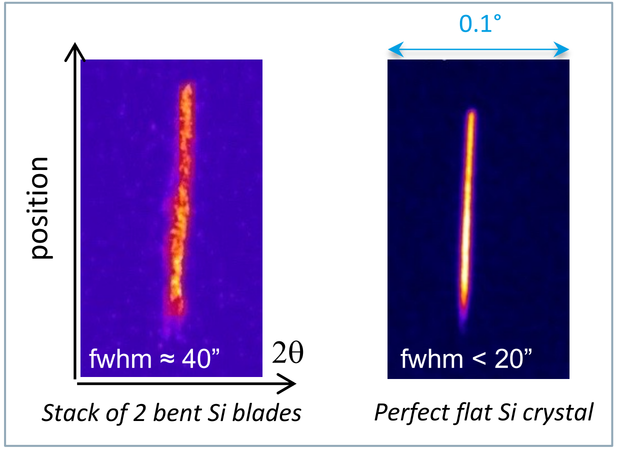
➢ Construction of a full-scale prototype

Construction of a Prototype

A key facility : the Hard X-Ray diffractometer



Principle of the Hard X-Ray Diffractometer



Hard X-Ray diffraction images

- Production of high quality bent perfect Si crystals using plastic deformation at high temperature

  Detailed studies using Hard X-Ray diffraction have shown that crystal quality is not affected by bending

- Accurate alignment using Hard X-Ray diffraction (+/- 1 arc minute)

➢ **Prototype successfully tested on ThALES !!**

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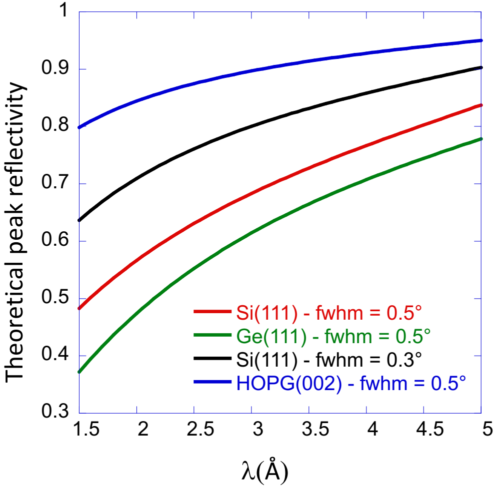
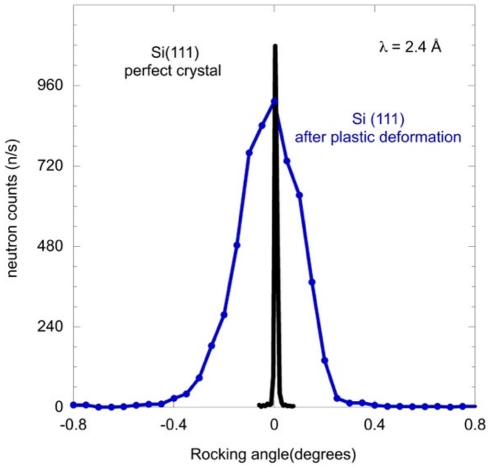
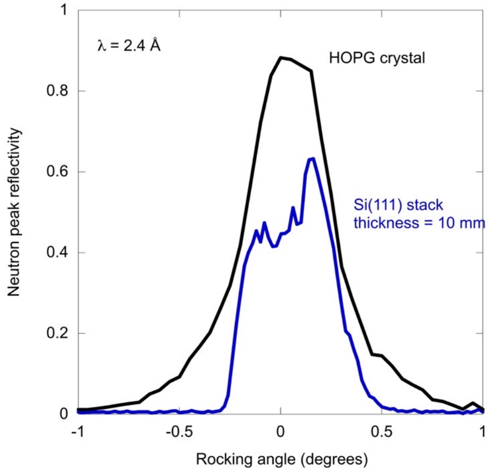
Mosaic Silicon crystal

A new Optical component for neutron monochromator ?







A mosaic Si crystal would theoretically outperform a mosaic Ge crystal: Si could replace Ge and why not… HOPG !

- Production of mosaic Si crystals (FWHM = 0.2°) using plastic deformation at high temperature
- Construction of stacks to improve diffraction efficiency and increase mosaic distribution up to 0.5°

➢ Promising results !

MANY THANKS TO :

The monochromator Group – Service for Neutron Optics
(for the huge work on the construction of monochromators)

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Florian Philit

Gilles Pastrello (Drawing Office - mechanics)

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Mechtild Enderle (IN20)
Bachir Ouladdiaf (D10+)
Stanislav Savvin (XTREMED)

Thank you for your attention