Some unconventional neutron diffractometer performances - particular properties

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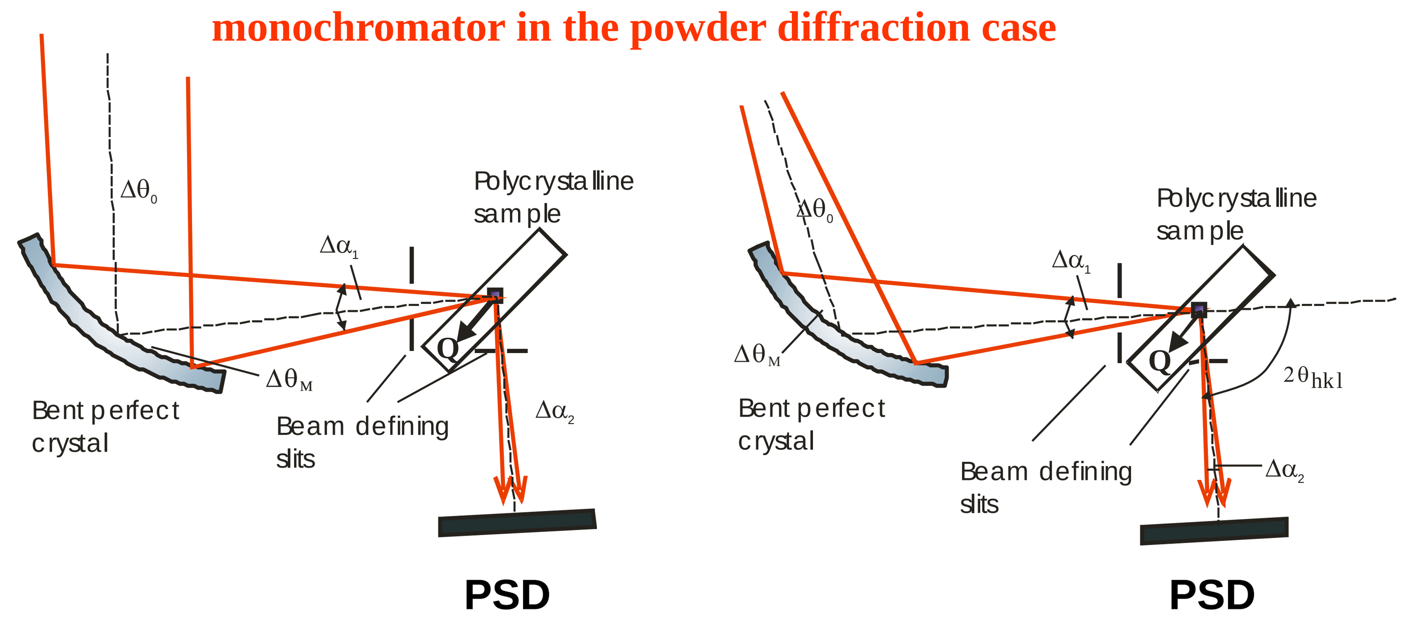
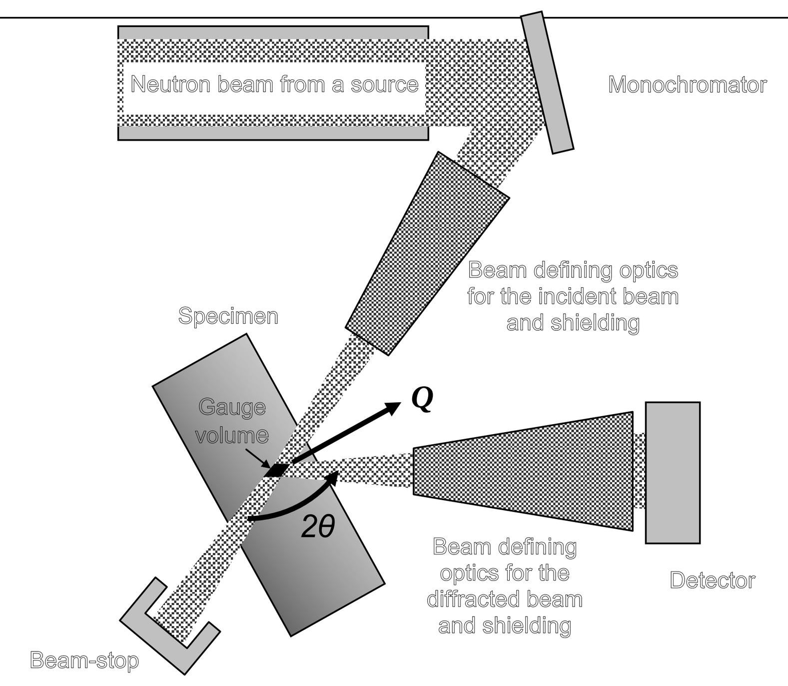
Abstract

Bent perfect crystals (BPC) have appeared in the last two decades as an excellent alternative of mosaic crystals in neutron diffractometry or spectrometry when employig them as monochromators or analyzers. Namely, their focusing and reflectivity properties and the possibility of their manipulation in favour of optimizing luminosity and resolution of the scattering instruments are attractive. Therefore, a permanent attention is paid to their further effective employment. In our contribution we present particular properties of a special neutron diffractometer performances employing bent perfect crystals as: A monochromator operating at small take off angle, A double-crystal (DC) monochromator employing bent perfect crystals of Si in (1,-1) and (n,-m) settings and A double-crystal (DC) monochromator employing bent perfect crystals of Si in (n,-m) settings with the second crystal in the fully asymmetric diffraction geometry. It has been found that by using quasi parallel or quasi dispersive (n,-m) settings, the related instruments provided excellent real space focusing with the spot of 1 mm or less, angular resolution $\Delta\theta \approx 10^{-3}$ rad and sufficiently high luminosity. A highly correlated monochromatic beam shows a high level of spatial coherence permitting to study refraction effects on edges often used in phase-contrast radiography.

Conventional and nonconventional focusing monochromator in the powder diffraction case



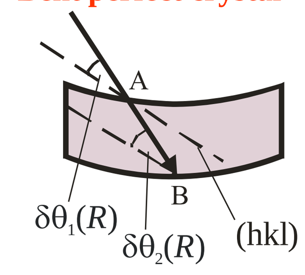
Scheme of the focusing diffractometer performance with a large take-off angle of about $2\theta_M$=90° (left) and the one tested in KAERI (right) with take-off angle $2\theta_M$=30° and $2\theta_{hkl}$ =90° .



Conventional performance of the strain/stress diffractometer equipped with mosaic monochromator. *FWHM* of the diffraction profile is strongly influenced by the mosaicity of the monochromator β being tens of minutes of arc (0.5 -1 deg).

- The larger take-off angle $2\theta_M$ at the monochromator, the better angular resolution of the instrument
- The smaller take-off angle at the monochromator, the better luminosity of the instrument when the wavelength spread of neutrons delivered at the sample is $\delta\lambda=\lambda\cdot\Delta\theta\cdot\cot\theta_M$ ($\Delta\theta$ is the angular divergence of the beam).
- The best geometrical choice corresponds to $2\theta_M=2\theta$ =90° when the gauge volume is in the form of a cube or rectangular prism. Consequently, it is required to have an intense Bragg peak at 2θ =90°

Bent perfect crystal



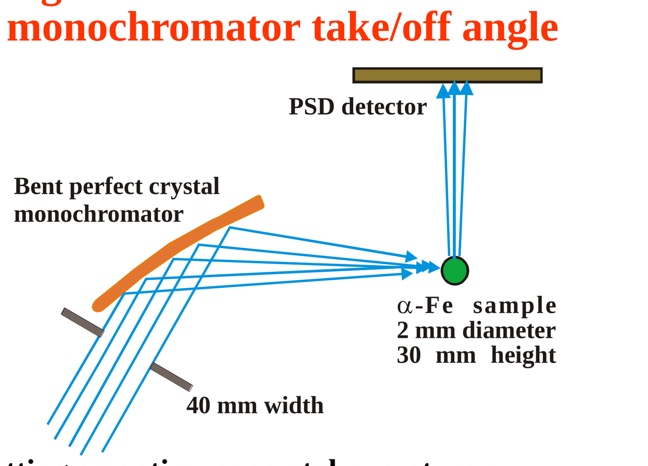
Detail of the beam passing through the BPC slab. $\delta\theta_1$ and $\delta\theta_2$ are the deviations from the mean Bragg angle due to the bending

Effective mosaicity

$\delta\theta = \delta\theta_1 - \delta\theta_2 = \Delta s / R$

$\Delta s = D/\sin\theta$ is the distance between the points A and B

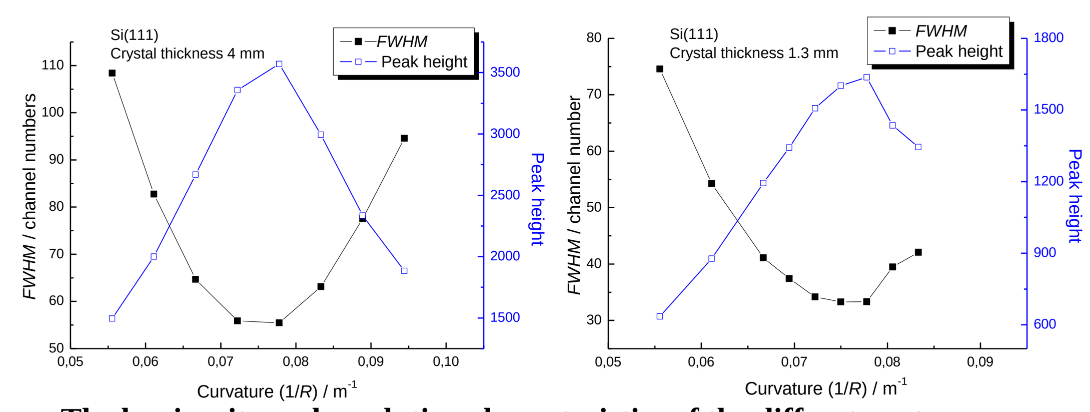
Focusing strain/stress diffractometer at a small monochromator take/off angle



By setting an optimum crystal curvature a quasiparallel diffracted beam can be obtained.

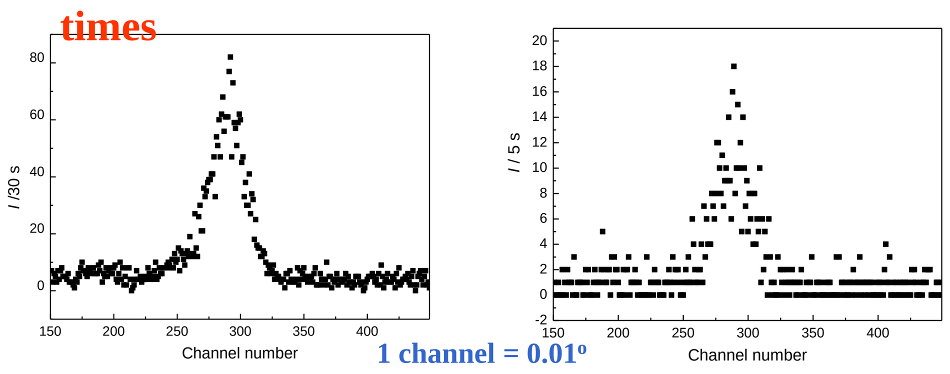
- Thanks to a small take/off angle the wavelength spread of neutrons delivered at the sample given by $\delta\lambda=\lambda\cdot\Delta\theta\cdot\cot\theta_M$ ($\Delta\theta$ is the angular divergence of the beam incident on the samle) is much lager but generally, the resolution of the instrument is worse.
- The resolution is strongly influenced by the mosaicity of the monochromator
- In the case of the BPC monochromator its effective mosaicity is strongly depended on the radius of curvature as well as the thickness of the crystal and therefore, can be easily manipulated
- Some compromise between the required luminosity and the resolution can be found

Experimental results: Optimization of curvature



The luminosity and resolution characteristics of the diffractometer performance for the focusing Si(111) monochromator of the thickness of 3.9 mm and 1.3 mm, α-Fe(211) steel pin of 2 mm diameter and 40 mm height, $2\theta_M$ =30°, for λ=1.62 Å the scattering angle on the sample was $2\theta_S$ = 87.8° .

Diffraction profiles for different measurement times



1 channel = 0.01°

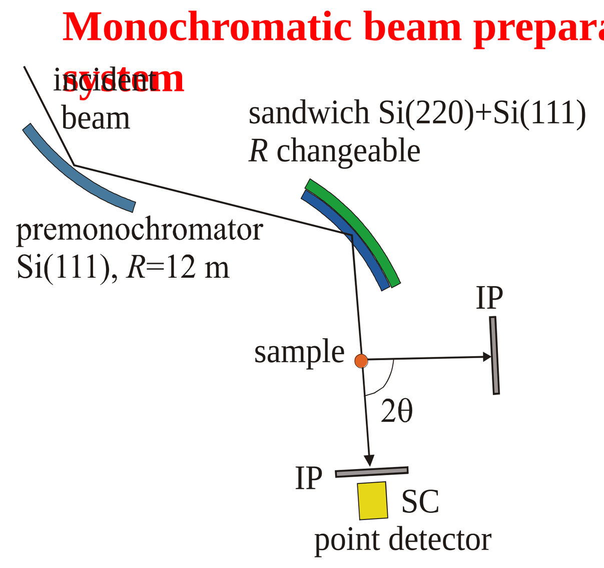
Summary

- Very good luminosity and resolution, possibility of studies of micro-strain/stress and even some kinetic processes in polycrystalline materials running within a few seconds.
- Possible improvements: Horizontally and vertically focusing monochromator and PSD with a better spatial resolution.

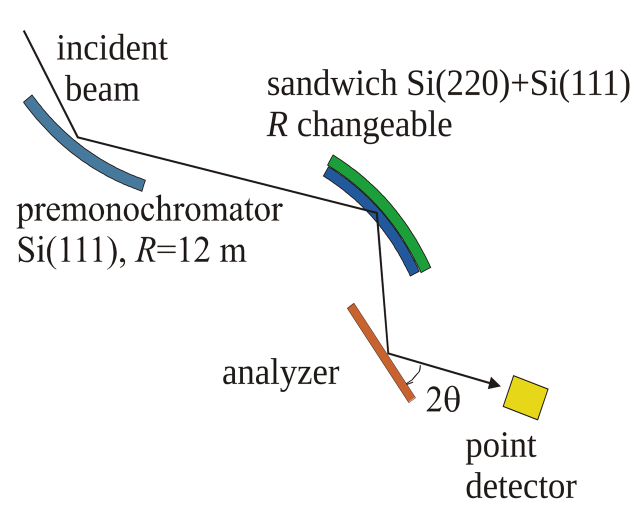
Versatile diffractometer with double/crystal monochromator

Following our previous investigations, properties of a special neutron double-crystal (DC) monochromator emloying BPC crystals of Si in parallel (1,-1) and (n,-m) settings are presented. The first premonochromator was the bent Si(111) crystal (4 mm thickness) and the second one was in the form of the sandwich consisting of two bent Si(111) and Si(220) slabs (2 mm and 1.3 mm thickness, respectively). It has been found that by a simple exchange of diffraction conditions of the second monochromator one can use either Si(111) + Si(111) double crystal (1,-1) setting providing good luminosity and worse diffractometer resolution or Si(111)+Si(220) quasi-dispersive (n,-m) setting providing very good diffractometer resolution and correspondingly worse luminosity. Besides excellent focusing and reflectivity properties of the quasi-dispersive double bent-crystal settings, the obtained monochromatic neutron current is sufficiently high for diffraction experiments even at the medium power research reactor. Such monochromator systems can at least be succesfully exploited in diffractometers dedicated to residual or *in-situ* strain/stress measurements when permitting strain/stress scanning (e.g. in the vicinity of welds) with good luminosity and worse resolution performance or microstrain studies (e.g. micro-strain behaviour in samples under the external load) with the other high resolution performance. Furthermore, in the case of different lattice spacings of crystals in the (n,-m) setting, the double diffraction provides a monochromatic beam for high resolution in both $\Delta(2\theta)$ (2θ is the scattering angle) and $\Delta\lambda/\lambda$.

Monochromatic beam preparation by a double crystal system

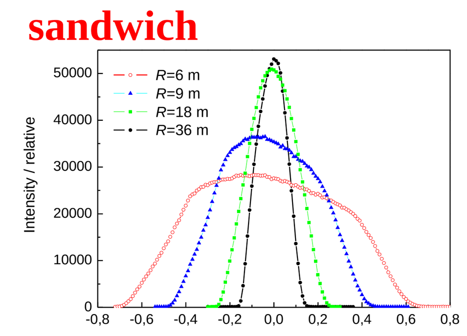


Sketch of the diffractometer performance with the sample imaging plate, scintillation camera, or a point detector

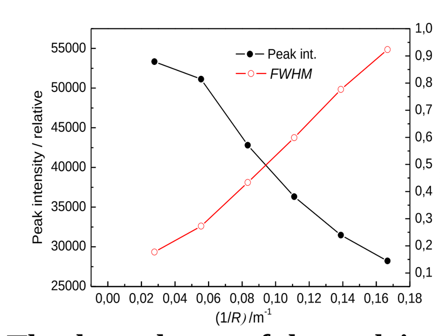


Sketch of the diffractometer performance with the flat Si(400) analyzer

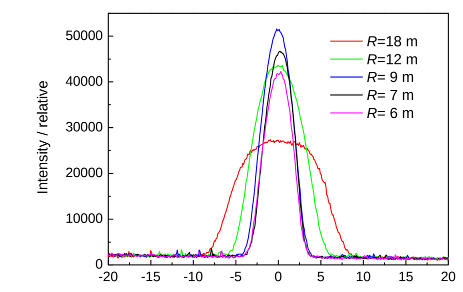
Experimental results with the active Si(111) slab from the sandwich



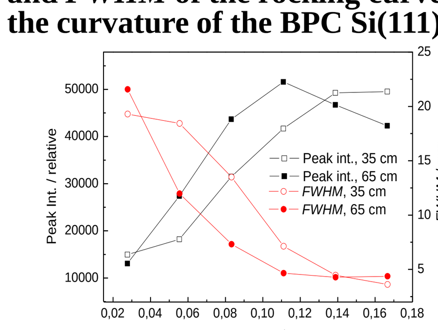
Rocking curves of the BPC Si(111) (different R_2) with respect to PM.



The dependences of the peak intensity and *FWHM* of the rocking curves on the curvature of the BPC Si(111) .



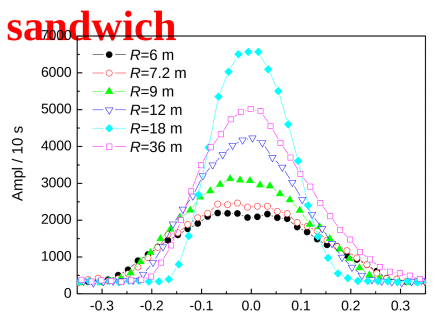
Diffraction profiles registered by a SC at 65 cm from the sandwich



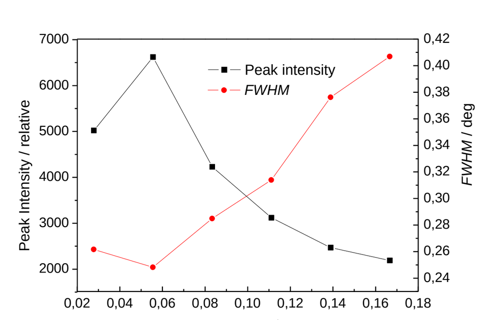
Parameters of the diffraction profiles from the Si(111) slab registered by SC at 35 cm and 65 cm vs curvature

Details: Premonochromator (PM): BPC Si(111), t=4 mm, constant radius of about 12 mm. Fixed premonochromator take-off angle (λ=0.162 nm). Second monochromator: Sandwich of the Si(111) and Si(220) slabs of the thickness of: 1x2 mm and 1x1.3 mm, respectively. Sample and detector positions: 50 cm and 75 cm from the sandwich, respectively. Analyzer: flat Si(004) slab at the position of the sample IP 40 cm from the sample pin, width of the incident beam: 20 mm.

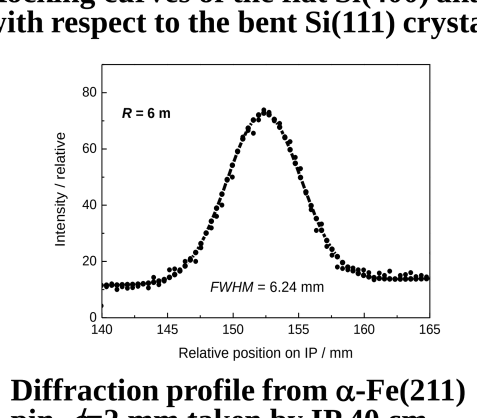
Experimental results with the active Si(111) slab from the sandwich



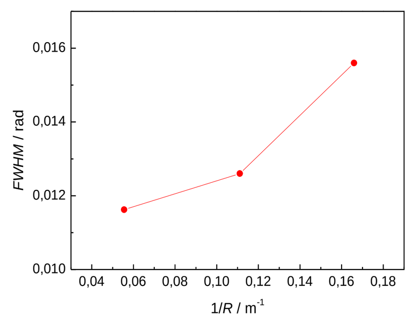
Rocking curves of the flat Si(400) analyzer with respect to the bent Si(111) crystal



Parameters of the rocking curves

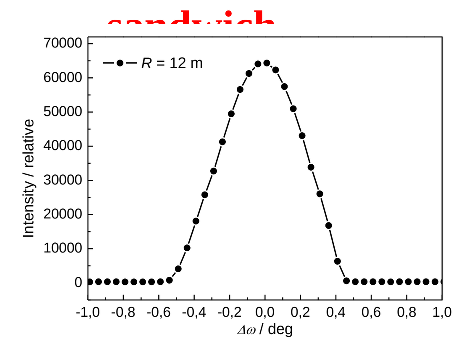


Diffraction profile from α-Fe(211) pin, ϕ=2 mm taken by IP 40 cm from the pin; 2θ=88°.

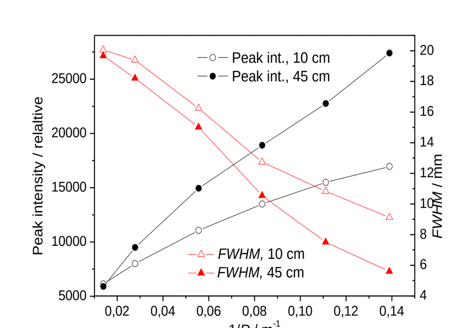


FWHM of the profiles from the α-Fe(211) pin for three curvatures of the Si(111) slab.

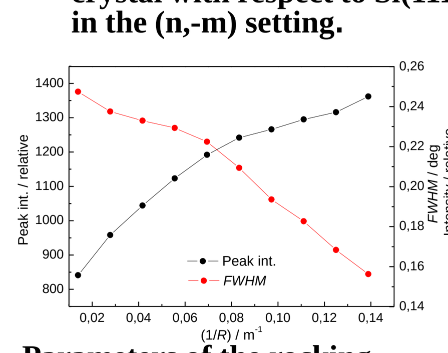
Experimental results with the active Si(220) slab from the sandwich



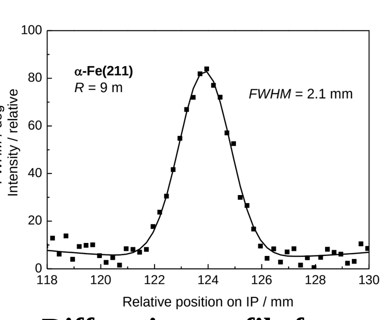
Rocking curve of the Si(220) bent crystal with respect to Si(111)–PM in the (n,-m) setting.



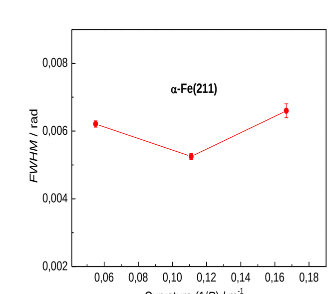
Parameters of the diffraction profiles from the Si(220) slab registered by SC at 10 cm and 45 cm vs the curvature.



Parameters of the rocking curves of the Si(400) analyzer with respect to Si(220) slab.

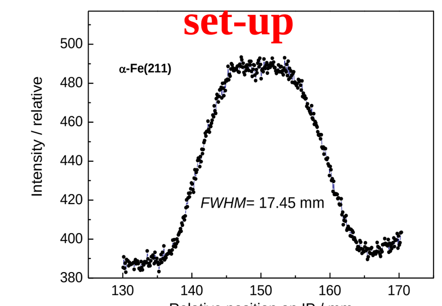


Diffraction profile from the pin of ϕ=2 mm.

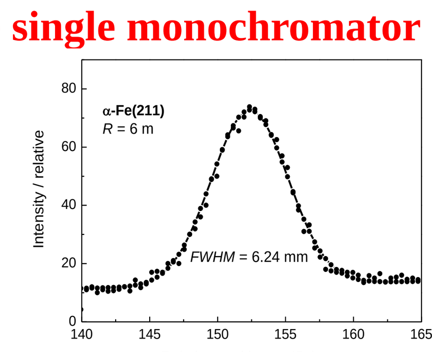


FWHM of the α-Fe(211) profiles vs the curvature of Si(220)

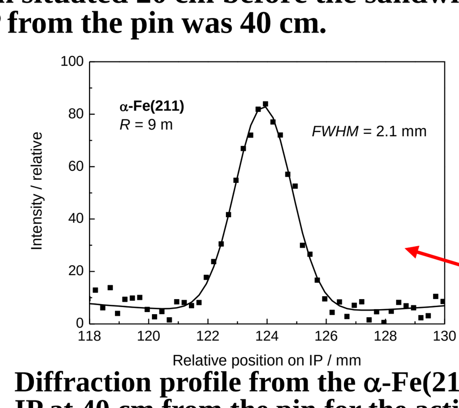
Comparison with the single monochromator set-up



Diffraction profile from the α-Fe(211) pin situated 20 cm before the sandwich. IP from the pin was 40 cm.



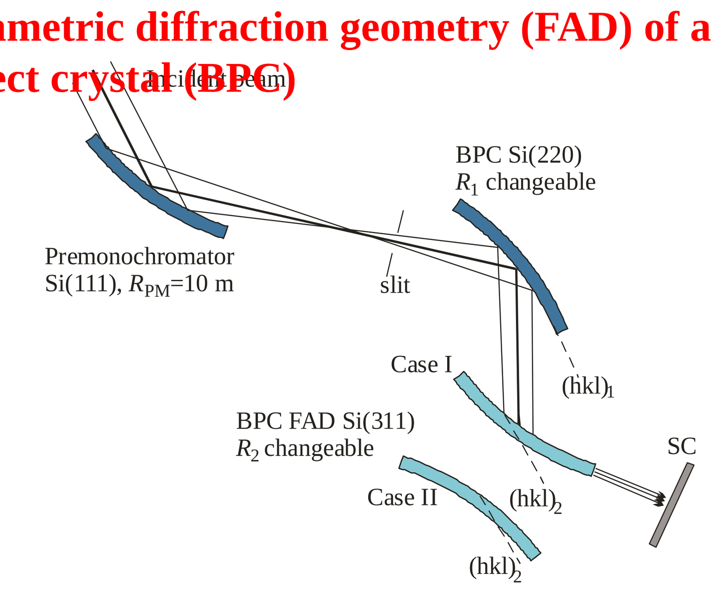
Diffraction profile from the α-Fe(211) pin taken by IP at 40 cm from the pin for the active Si(111) slab.



The parallelism of the diffracted beam is about 1.5×10^{-3} rad.

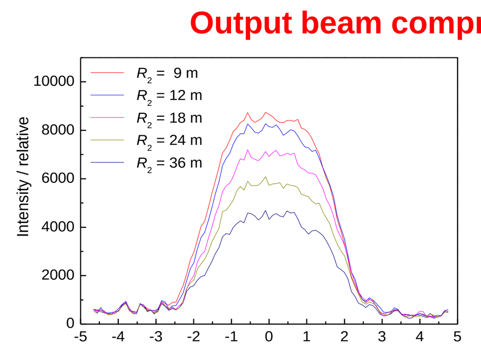
Diffraction profile from the α-Fe(211) pin taken by IP at 40 cm from the pin for the active Si(220) slab.

Diffractometer performance with the fully asymmetric diffraction geometry (FAD) of a bent perfect crystal (BPC)

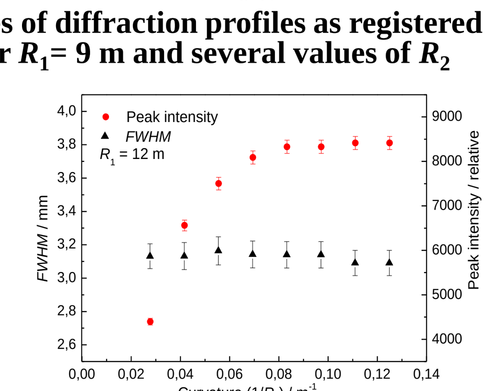
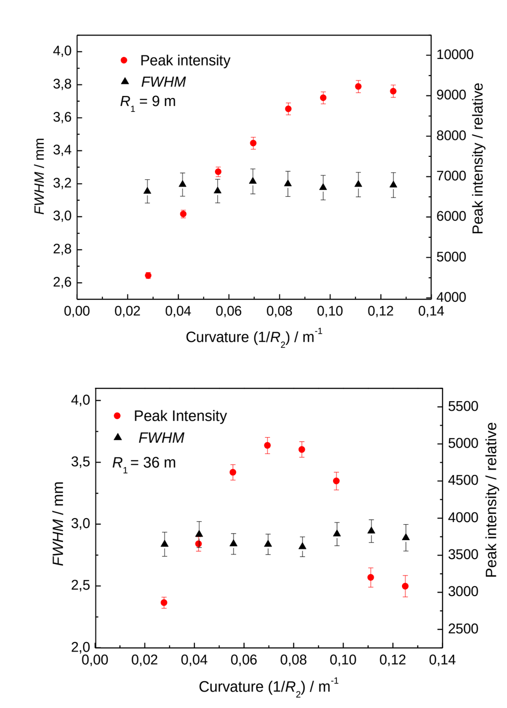


Scheme of the experimental arrangement as used for the studies of the FAD-OBC (wide beam incident on the largest face of the FAD crystal and the output beam compression – OBC) properties of the BPC Si (311) crystal in the double crystal setting with respect to the Si(220) one. SC - scintillation camera was at 56 cm from the FAD crystal. Dimensions of the crystal slabs: 200x40x4 mm³ (length x width x thickness).

Output beam compression (OBC) – Case I

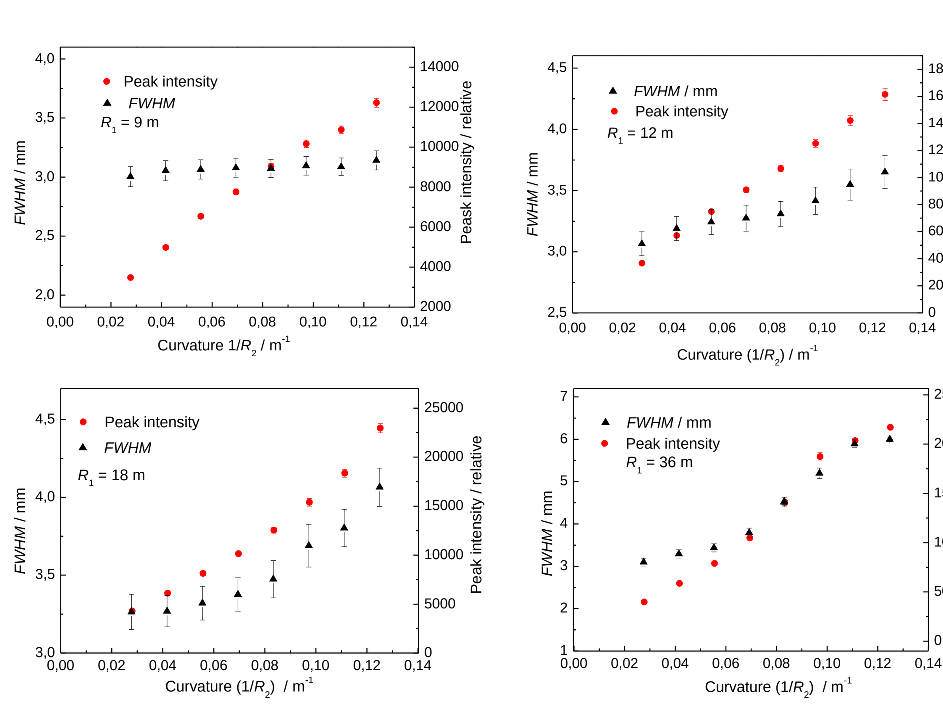


Examples of diffraction profiles as registered by SC for R_1= 9 m and several values of R_2

Peak intensity and *FWHM* of the diffraction profile dependence on the FAD crystal curvature for a fixed curvature of the Si(220) one of 0.11 m⁻¹, 0.083 m⁻¹ and 0.028 m⁻¹. Output beam is quasiparallel !!!!

Output beam compression (OBC) – Case II



Peak intensity and *FWHM* of the diffraction profile dependence on the FAD crystal curvature for a fixed curvature of the Si(220) one of 0.11 m⁻¹, 0.08 m⁻¹ , 0.056 m⁻¹ and 0.028 m⁻¹. The output beam is quasiparallel only for R_1=9 m.