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Focusing monochromator with extreme energy resolution

Principal publication and authors
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High energy resolution is a figure of merit for many synchrotron radiation techniques. The most extreme requests come from inelastic X-ray scattering and nuclear inelastic scattering, where ~1 meV and sub-meV resolution is reached. An even higher energy resolution is needed for demanding scientific cases such as the novel high-T_C superconductors, liquids, and biological materials. Therefore, the improvement of the energy resolution down to 0.1 meV is a key project at several synchrotron radiation facilities.

Attempts to reach an energy resolution of 0.1 meV with conventional optical schemes encounter severe losses of intensity [1,2]. Analysing alternative schemes with higher throughput, we propose an approach with a 'focusing monochromator', whereby the X-ray energy spectrum is swept along the spatial coordinate rather than along the angular coordinate.

The operation of the focusing monochromator is based on a combined action of a focusing lens and a dispersive crystal (Figure 146). The lens alone would focus X-rays of all relevant energies towards a single spot. The crystal in a highly asymmetric reflection works as an optical prism [3], sorting the X-rays of different energies into different directions. In the combined action, the lens focuses X-rays *through* the dispersive crystal. Deflecting the X-ray components of different energies by different angles, the crystal re-directs them to different focal spots. The selection of the narrow energy band is provided by a slit located in the focal plane. The more convenient *in-line* geometry of the monochromator is achieved by adding another crystal with the same reflection, but in a symmetrical scattering geometry (Figure 147).

The ultimate relative energy resolution $\Delta E/E$ of the focusing monochromator is determined by the angular size of the radiation source as

$$\frac{\Delta E}{E} = \frac{\Delta\theta}{\tan\theta_B} \quad \frac{b}{(b-1)} \ ,$$

where $\Delta\theta = S/L$ is the angular size of the radiation source as seen from the lens location; S is the spatial source size; L is the source-lens distance; b is the module of the reflection asymmetry parameter; and θ_B is the Bragg angle. This resolution is reached with the slit size equal to the focal spot of monochromatic radiation. Opening the slit, one can gradually increase the bandwidth of the selected radiation up to the entire energy band reflected by the crystal. This offers a useful possibility to perform fast measurements with coarse resolution but high count rate before studying selected features with the ultimate energy resolution.

For a moderately high asymmetry parameter b, the relative energy resolution $\Delta E/E$ is determined only by the angular size $\Delta\theta$ and the Bragg angle θ_B. The smallest angular size of the radiation source available at the ESRF (for a 200 m-long beamline) is 0.1 μrad. Then, the 0.1 meV bandwidth can be

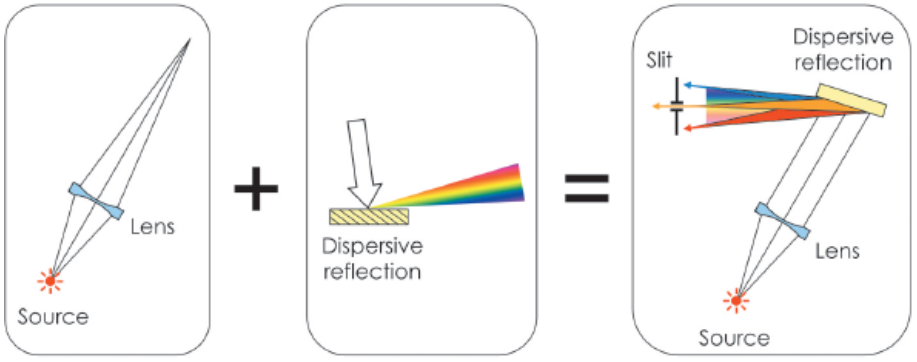

Fig. 146: The concept of a 'focusing monochromator': By the combined action of the focusing lens and the dispersive crystal, X-rays of different energies are focused to different focal spots. Selection of the narrow energy band is achieved by the slit.

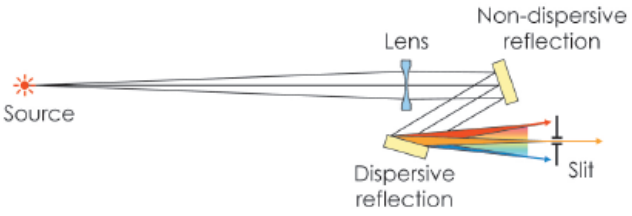

Fig. 147: In-line geometry of the focusing monochromator with an additional crystal in symmetric reflection.

obtained with a Bragg angle of 84 or 87 degrees for X-rays with an energy of 10 keV or 20 keV, respectively.

The proposed optical scheme is expected to provide very high throughput of radiation within the selected energy band. According to the operation requirements (see principle publication), the lens delivers a highly collimated beam well within the angular acceptances of both crystals. In addition, the moderate asymmetry parameter allows one to keep the high reflection coefficient.

From a practical point of view, the proposed design is simple and includes a minimal number of optical elements. Relative to conventional high-resolution optics, the X-ray spot on the crystals of the focusing monochromator is much smaller, well below 10 mm. Therefore, the proposed design should be relatively insensitive to possible imperfections of the crystal quality.

References
[1] M. Yabashi, K. Tamasaku, S. Kikuta and T. Ishikawa, *Rev. Sci. Instrum.* **72**, 4080 (2001).
[2] T. S. Toellner, M. Y. Hu, W. Sturhahn, G. Borel, E. E. Alp and J. Zhao, *J. Synchrotron Rad.* **8**, 1082 (2001).
[3] Yu. Shvyd'ko, *X-Ray Optics*, Springer-Verlag Berlin Heidelberg, 2004.

Controlled dehydration of macromolecular crystals to improve diffraction properties

Principal publication and authors
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The weakly diffracting nature of protein crystals often leads to data of insufficient quality to answer the biological question being asked, either due to a failure to solve the structure or to other factors, such as insufficient resolution to accurately determine modes of ligand binding. Methods exist to improve the diffraction properties of macromolecular crystals, dehydration often being found the most effective. Dehydration of protein crystals often has positive effects: reductions in the length of one or more unit cell dimensions are often accompanied by an increase in diffraction data resolution; a decrease in the mosaic spread and improvement in the profile of Bragg peaks. However, despite many descriptions of successful dehydration experiments and the availability of dedicated systems, the technique remains little used as conditions can be difficult to reproduce and the method can be problematic to implement. To standardise the technique, the EMBL and ESRF developed a humidity control device (HC1, **Figure 148**). Based on a modified cryostream nozzle, it produces an air stream at the sample position and allows precise control of the relative humidity (RH) between 50 and 99%. The device is fully compatible with the standard experimental environment at the ESRF and coupled with a synchrotron beamline, dehydration experiments have now become a practical way to improve the diffraction properties of some protein crystals.

The device was initially tested using bovine mitochondrial F_1-ATPase. This complex crystallises in the orthorhombic space group $P\ 2_12_12_1$ with unit cell dimensions of a = 108, b = 140 and c = 285 Å. Crystals of F_1-ATPase are known to react well to dehydration. Indeed, they are inclined to undergo spontaneous dehydration events, where the *c* cell dimension is reduced to *ca* 268 Å, after increasing the precipitant concentration or during prolonged crystal handling. This change in unit cell dimensions occurs rapidly and is favourable as it leads to an

Cover

Design by M. Collignon. Featured images: a) Structure of the η-phase of oxygen, L.F. Lundegaard *et al.*, p 10; b) Structure of A-amylose by synchrotron microdiffraction of crystals, D. Popov *et al.*, p 64; c) Dendritic solidification in an aluminium alloy observed by 3D microtomography, N. Limodin *et al.*, p 109; d) DNA binding to a protein drUvrA involved in DNA damage recognition, J. Timmins *et al.*, p 99.

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