The challenging optics of XtremeD – a neutron diffractometer for high pressures and magnetic fields at ILL developed by Spain

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Abstract. The Spanish community of neutron scatterers and the ILL are considering the construction of a CRG “eXtreme conditions Diffractometer (XtremeD)” for both single crystals and powders, operating at high pressures (up to 50 GPa) and high magnetic fields (up to 15 Tesla). High pressure studies require reduced sample volumes. This makes the focusing optics a crucial part of the instrument, in order to have increased flux and to avoid the scattering from the sample environment. The different solutions at different levels of the instrument, which are being studied, will be discussed in this paper.

1. Introduction
Over the last few years, there has been a regain in the interest on scientific problems related to the behaviour of matter under extreme conditions of pressure, magnetic field and temperature [1]. Neutron diffraction has unique capabilities for this kind of research and the interest for this technique, in particular in the Spanish scientific community, has led to the proposal for the construction at ILL of a new neutron diffractometer optimized for high pressure and high magnetic field studies for both single crystals and powders, which will be developed by Spain. At the present time we are finalizing the technical project, and the construction phase is expected to start in 2011.

There are fundamental differences between synchrotron X-ray beams, which are intrinsically small and well collimated, and neutron sources, which are both large in size and have a large divergence [2]. The neutron phase space is approximately 1000 times less dense than X-ray phase space. On the other hand, the lower fluxes of neutrons and the lower interaction of neutrons with matter compared with X-rays makes necessary the use of much bigger samples than in synchrotron X-ray experimentation. For these reasons, the experimentation with very high pressures is a challenging field for neutrons. The small sample size -around 1mm³- required in order to reach pressures above 10GPa implies that the focalization of the beam is absolutely necessary in order to have increased flux to compensate for the small sample and to avoid the spurious scattering from the sample environment. Different solutions at different levels of the instrument are being studied and will be discussed in this contribution, such as a focusing guide, different types of focusing monochromators, collimators, or the possibility of...
installing a focusing device between the monochromator and the sample integrated in the dedicated magnet.

2. Previous considerations for the projected instrument
The techniques for focusing neutrons are numerous and their application fields differ from one to the other. Each instrument, depending of its scientific purpose, has thus its specific solution for the focalization. Before choosing any of them, one must first know well some aspects of the instrument to be built. Hereafter, the specifications of the focusing system for the case of XtremeD are presented in order to define the objectives for the future designs:

(i) The sample size: In our case, it will finally depend on the pressure cell. The projected instrument will operate with a variety of pressure cells. With the present pressure cells, the sample volume varies from 30 to 100mm$^3$, but it is to be considered that future developments for very high pressure (∼50GPa) will imply a reduction to 1mm$^3$ samples or even smaller. For the moment, this size -1mm$^3$- is due to be taken as reference.

(ii) Focal length: In our case, it can not be higher than 1-3 meters for a focusing monochromator, due to the available space for the instrument.

(iii) Dimensionality: XtremeD’s requirements of flux will be very ambitious and probably it will be necessary 2D focusing in order to reach the required levels. The final limitation will be the degradation of the resolution.

(iv) Wavelength band: The range of wavelengths will be in principle between 0.8Å and 4.1Å

The following lines describe the different alternatives that have been studied for the case of XtremeD.

3. Studied solutions

3.1. Passive focusing: Collimating focusing
The principle of these systems is to reduce the phase space distribution instead of modifying it, being the simplest device of this type a pair of slits or pinholes. The collimators allow shaping the neutron beam, tuning its size with respect to the sample, in order to minimize the background noise, and correcting its divergence. Despite of the simplicity of this method, it has disadvantages: all the neutrons that are not inside the area of collimation are lost. This reduces largely the efficiency of the system. In the case of XtremeD, this device is to be foreseen in the closest area to the sample and only if the beam size at that point is still too big for maintaining an acceptable level of background.

3.2. Crystal focusing
This technique has been frequently used and combines the monochromatization process with the focusing. We distinguish two types: mosaic monochromators and bent perfect crystal monochromators.

The idea of mosaic type monochromators is to obtain an artificial curvature dividing the surface into a number of sub-elements with a size of the order of 10-20 mm. The curvature can be tuned with monitored movements. The main limitation is the size of the focal spot, which depends directly on the size of each of the elements. Consequently, it can not be smaller than 10x10mm$^2$ with this technology (figure 1). Though it is quite expensive, it has proven to be very efficient and reliable over the years. It can be designed both for vertical and horizontal focalization.
Bent perfect crystal (BPC) monochromators consist of an elastically bent perfect crystal – usually silicon – without mosaic structure. The bending radius can be modified, changing the focal distances of the system. Comparing it with a mosaic monochromator, we can achieve higher intensities with an excellent resolution [3] (this does not consider the higher efficiency of graphite monochromators, which for high wavelengths – higher than 2.3 Å – would imply a higher flux). Unfortunately, BPC monochromators cannot be bent in two different directions. If a second focusing direction is desired, differently oriented curved plates are used in the same way as mosaic monochromators. As a consequence, there is a limitation at the spot size, which can not be smaller than the plate’s width (~10 mm) in this direction. There is an important characteristic that makes silicon BPC monochromators particularly suitable for our instrument: they are almost transparent devices and do not affect the flux which is not diffracted by the crystal. XtremeD will be placed in the first position of a shared guide which is projected to be refurbished. The transparency of the monochromator gives different opportunities for the flux optimization of the guide without having the drawbacks of being placed at a first position site. Following this idea, there is a conceptual development which could be considered for the case of XtremeD: the combination of two monochromators for the focalization. The first monochromator is a focusing elliptical or parabolic monochromator that will generate a small virtual source for the second monochromator. The second one will then further reduce the beam size to obtain a small spot at the sample position. Another option is to use each monochromator for the focusing in a different direction.

3.3. Reflective optics

Two different applications are being considered for this kind of systems to our instrument: a convergent guide between the monochromator and the sample, and a focusing neutron delivery guide. The design of a new instrument should comprise the whole setup between the source and the sample, not only the components downstream of the monochromator [2].
For the delivery guide, the interest of a focusing module is at the creation of a small virtual source for the focusing monochromator. In order to maximize the flux, the implementation of the so-called “ballistic guides” has also been proposed [4]. The scheme in figure 2 shows the operation of this system combined with a bent monochromator.

The use of focusing guides between the monochromator and the sample has to be treated carefully. There is a positive effect at horizontally focusing monochromators called “Q-space focusing effect”, which means that the gain in divergence (loss of resolution) due to the horizontal focusing of the beam is compensated by the diffracted output at the sample, maintaining the resolution at good values for a certain position in 2θ. The introduction of any optical device that changes the beam’s horizontal divergence is not desirable. However, the use of a guide for vertical focusing would be possible. In scattering experiments like powder diffraction, a downgraded resolution in the vertical axis can be accepted, as the intensity is integrated in this direction. In the case of single crystal experimentation - less demanding in terms of flux - the possibility of replacing this device with a Soller collimator is an option.

The easiest way to collect more neutrons from a large source is to use a tapered guide consisting of a standard flat super-mirror substrate on a tube structure whose entrance is reduced in size. For a guide with an exit two times smaller than the entrance, a flux density four times bigger is to be expected at the exit in relation to the initial value. Meanwhile, the divergence of the beam increases in a proportional way (Liouville’s law). A very important requirement of this focusing device is that the sample has to be positioned very close to the guide exit to benefit from the flux increase. This can be a problem at XtremeD, as the sample environment will be large. An analysis of the implantation of the focusing guide inside of the sample environment (high field magnet) has been done (figure 3). The main limitation could be the response of the optical materials to the thermal changes that are generated inside the magnet.

**Figure 2.** Combination of a ballistic guide and an elliptically bent monochromator

**Figure 3.** Implantation of an optical guide inside of a magnet.
4. Conclusions
After considering the different options for the optical system of the new instrument XtremeD, the solution considered to be the best is the use of a BPC monochromator (preferably after an elliptical ballistic guide). The monochromator will re-focus the beam on to a spot down to 1mm³ at the sample position, obtaining gains of 10 to 50 in flux [2]. A vertically focusing trumpet or a Soller collimator combined with diaphragms depending on the experiment to be carried out will achieve the final conditioning of the beam in order to reduce the size of the spot to the desired value.

References
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