**XtremeD** – a new neutron diffractometer for high pressures and magnetic fields at ILL developed by Spain

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**Abstract.** Neutron diffraction has unique capabilities for scientific research under extreme conditions, mainly in two large areas: crystallography/geosciences and magnetism/solid-state physics. The growing interest in these fields is attested by the quantity and quality of publications, by the number of experiments proposed at the different neutron sources and by the new instrumentation projects under development all around the world. Therefore, the Spanish scientific community 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). At the present time we are working on the finalization of the technical project, and the construction phase is expected to start in 2011. The scientific areas in which the projected instrument can make significant contributions and the main technical characteristics of the project are discussed in this paper.

1. **Introduction**

Science under extreme conditions generally implies a (very) high pressure applied to the sample, possibly combined with other extreme parameters such as high or low temperatures, high magnetic fields, extreme chemical environments, among others. The interest on scientific problems related to the behaviour of matter under such extreme conditions has experienced an important increase over the last few years, and the scientists dedicated to such studies constitute one of the most active communities nowadays [1]. Neutron diffraction has unique capabilities for this kind of research and the interest for this technique is attested by the number of experiments proposed at the different neutron sources, by the new instrumentation projects under development all around the world and by the quantity and quality of the publications issued from this field.

Studies under high pressure are easier with X-ray diffraction because very small samples can be used due to the high fluxes from synchrotrons. But, for the same reasons that neutrons are indispensable for studies at ordinary conditions, there are many problems where neutron diffraction is complementary to X-ray diffraction, and other cases, like magnetism, in which neutron diffraction has unique capabilities. With the appropriate design of an “eXtreme conditions Diffractometer (XtremeD)” at ILL, the expected performances can be largely superior to those of the spallation...
neutron sources for magnetism and only slightly worse (or comparable for single crystals) in the case of crystallographic applications.

The problems where neutron diffraction can contribute can be grouped mainly in two large areas: crystallography/geosciences and magnetism/solid-state physics. In Spain, there are strong communities in both areas with great interest in neutron diffraction at extreme conditions. This interest has led to the proposal for the construction at ILL of a new neutron “Xtreme conditions Diffractometer (XtremeD)”, optimized for high pressure and high magnetic field studies for both single crystals and powders, which will operate as a CRG (Collaborative Research Group) managed by the Spanish CRG’s team. Other major groups in Europe dedicated to high pressure investigations have also shown their interest for the XtremeD project by providing representatives to participate as scientific advisors. The project was positively evaluated by the end of 2009 both by the Scientific Council and the Steering Committee of the ILL. At the present time we are working on the finalization of the technical project, and the construction phase is expected to start in 2011. The main technical characteristics of the projected instrument and the range of scientific topics that could by tackled in such a facility are discussed in this contribution.

2. Technical characteristics and management framework
The main idea of the instrument XtremeD is to combine a large solid-angle detector with an optional highly focused beam on the sample, thus providing high flux while maintaining low background. The principal features of XtremeD will be:

(i) Large 2D position-sensitive detector for powders and single crystals. The detector, most probably of He3, will cover an angular area of 153.6x19.2 degrees (solid angle of 0.9sr). It will have a definition of 0.15 degrees horizontally and 0.2 degrees vertically. This means a pixel size of 2.1mm horizontally and 2.8mm vertically. A local counting rate of 10 kHz will permit to take profit of the high flux onto the sample.

(ii) Optimization for small samples: optional and variable focusing optics, which will produce a maximum flux on the sample of $10^8$ n·cm$^{-2}$·s$^{-1}$ at the sample (equivalent to D20 instrument at ILL).

(iii) Radial oscillating collimator and neutron shielding to suppress background

(iv) Sample environment adapted for high pressure (variety of pressure cells, up to 30 GPa for the moment, but with a goal of 50 GPa after the envisaged developments) and high magnetic fields (up to 15 Tesla in continuous mode, with the possibility of higher pulsed fields under study).

(v) Modularity: Choice of wavelengths (0.9 Å – 4 Å) and resolutions. The primary spectrometer will consist of a system of two interchangeable double variable focusing monochromators: a graphite mosaic monochromator (002) and a silicon bent perfect crystal monochromator (hhl odd). With all the elements of the instrument mounted in air cushions, the take-off angle could be set in a continuous range from 40º to 120º. This means that the wavelength range covered will be 2.29Å – 4.1 Å (long wavelengths, medium resolution) for the graphite monochromator and 0.81 Å – 4.1Å (wide range of wavelengths, high resolution) for the silicon monochromator, with a best resolution of 0.3º (FWHM).

XtremeD will be installed at the position presently occupied by IN3 (a triple-axis spectrometer on the thermal side of the ILL7 guide hall, on guide H24), taking advantage of the variable wavelength capacity of its monochromator and casemate and its large experimental zone that would be needed for operating a flexible and versatile diffractometer incorporating a removable focusing system, variable monochromator-sample distance, different take-off angles, etc (figure 1). A neutron guide position would also help to provide low background, which is very important for small samples. Finally, the project of having a new supermirror guide in order to expand the wavelength range and the flux is crucial for the project.

XtremeD will be a CRG managed, like D1B and D15, by the Materials Science Institute of Aragón, most probably with other scientific partners. The proposed CRG framework will provide the necessary flexibility to test new instrumentation developments and to perform difficult and uncertain experiments.
3. Scientific case
Progress in condensed matter physics, solid state chemistry and geosciences depends on the understanding of fundamental properties of materials under extreme conditions. Furthermore, in the study of compounds relevant to molecular biology the use of moderate pressures is achieving great interest. Among the different topics in which an extreme conditions diffractometer could contribute, three representative areas are presented here: water-related systems, molecular chemistry, and magnetism under extreme conditions.

3.1. Water-related systems
The study of ices and some other water-related systems under extreme pressure conditions implies systematic studies of hydrogen bonding and clustering, host-guest interactions in clathrate hydrates and the role of water in crystals. Understanding such systems has far ranging implications in physics, chemistry, planetary sciences and geochemistry. [2,3] As an example, current interest in clathrates is motivated by the recognition of their abundance in Earth’s subsurface and in icy bodies of the solar system, their potential role in climate changes, and their possible economic importance as a source of fuel.

3.2. Molecular chemistry
In this field, research under extreme conditions tackles a variety of exciting physical phenomena and a number of problems with high social impact, like the improvement of the efficiency in hydrogen storage materials (for example, ammonia borane, NH₃BH₃ in the range of 6 GPa [4]), the production and characterization of new phases for pharmaceutical applications (polymorphism) or the magnetic and conducting properties of O₂, the only elementary molecular magnet [5,6].

3.3. Magnetism under extreme conditions
A great variety of materials exhibit drastic changes in their magnetic behaviour under high pressures and/or applied magnetic fields: Transition-metal oxides, multiferroics and magnetoelectric materials,
frustrated magnetic systems with magnetoelectric properties, magnetic molecular materials, etc. Special attention should be paid to magnetic oxides. The search of novel properties in magnetic oxides is currently a very active area of extraordinary technological importance that covers a great variety of activity domains in which particular magnetic orders and lattice effects are key: from superconductivity to spin-electronics and magnetoelectricity. As an example, among transition-metal oxides there is an ample variety of multiferroic materials, which is one of the areas of higher interest nowadays. These are materials of great interest in high pressure research due firstly to the synthesis of new multiferroics, and secondly, as a way for material transformation under high pressure conditions. The spin state and its pressure and temperature dependences in (La,Sr)CoO$_3$ [7] and Co$^{3+}$-analogues [8] or the strong change in electronic structure by pressure in the multiferroic BiFeO$_3$, [9] are selected examples of current interest deserving clarification. Neutron diffraction under extreme conditions has unique capabilities for this type of research and can make crucial contributions in this field.

4. Conclusions
The interest for the problems related with the behaviour of matter under extreme conditions, where neutron diffraction can make decisive contributions, has led the Spanish community of neutron scatterers and the ILL to propose 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). The characteristics of XtremeD together with its emplacement at the highest neutron flux source, ILL, will make XtremeD an unique instrument in the world. It is expected that the new sample environment developments, combined with advances in neutron science and technology boosted by the construction of such a diffractometer, would open opportunities for new scientific breakthroughs.

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