19. ALADIN - Advanced LAue Diffraction Instruments using Neutrons

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Abstract

Laue diffraction techniques have proven to be very attractive to a broad user community interested in obtaining detailed structural information on very small single-crystal samples or needing data collection speeds comparable to those available with the powder diffraction technique. However our experience has clearly demonstrated the negative effect of up-stream monochromatic instruments on the quality of Laue data. In order to obtain Laue diffraction data with a statistical accuracy similar to that achieved on a monochromatic instrument (neutron or X-rays), the project ALADIN (for Advanced LAue Diffraction Instruments using Neutrons) aims to:

- construct a Laue-dedicated thermal neutron guide, with m=2 super-mirror coating, providing access to the desirable wavelength bandwidth
- installation one of the ILL Laue diffractometers (VIVALDI or CYCLOPS) on this new guide.

Motivation

Chemists and physicists are interested in solids exhibiting a wide spectrum of optical, dielectric and magnetic properties, often resulting from a delicate balance between various competing interactions. In many cases, this balance can be tuned through direct chemical substitution, as well as by varying external parameters such as temperature, pressure, electric or magnetic fields, or light irradiation. In all cases, a proper interpretation of the experimental results cannot be performed without the help of detailed and systematic structural studies. However because of their complex nature, novel and interesting materials can rarely be obtained in the form of large single crystals, a situation which limits considerably the usefulness of the neutron scattering technique. Indeed, while neutron diffraction is an extremely powerful tool, capable of providing precise structural information, often inaccessible with other techniques such as X-ray diffraction or multi-quanta NMR, in practice it is frequently disregarded because of the large-size single-crystal samples usually required. With the recent renaissance of Laue techniques, implemented at high-flux thermal neutron sources, structural and/or magnetic information can now be routinely extracted from crystals of volume 0.1 mm^3 or smaller, with possibilities to tune external control parameters such as pressure, temperature and/or high magnetic field. Beside its unique capability concerning crystal size, the Laue technique (especially with the newly developed CCD-based neutron cameras) also offers a possibility to study kinetics or the influence of control parameters in a systematic way, as is done in the fast data collection mode on powder diffraction machines, but with the added capability of visualizing details that only single crystal diffraction can afford.

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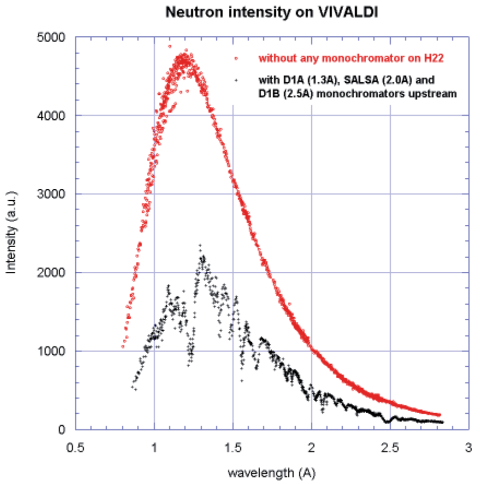
At the present stage, the Laue technique has demonstrated its potential but the accuracy of the data obtained from Laue instruments at ILL is still far from that obtained on monochromatic diffractometers, making it extremely difficult to perform studies requiring a high spatial resolution. A recent study conducted on VIVALDI, the thermal Laue diffractometer installed on the thermal H22 guide, has directly demonstrated the dramatic effect of up-stream instruments (in that particular case, two powder diffractometers –D1A and D1B- and the strain imager SALSA; see Figure below):



a flux reduction by a factor of ~2 is observed and numerous sharp dips are present in the wavelength distribution, depending on the monochromator settings. As a direct consequence, the normalisation to a common wavelength on VIVALDI is extremely delicate, even when the up-stream monochromators remain fixed during the Laue data collection. A similar situation is foreseen in the case of CYCLOPS, the CCD-based Laue instrument, presently installed on H24 and still being commissioned.

In order to address topical structural problems with sample sizes in the range of ~0.001mm^3 or less, a domain accessible so far only by means of X-ray diffraction, we propose the re-location of one of the Laue instruments on a dedicated thermal white beam with a wavelength bandwidth from ~0.6-0.7 to 5 Å, a condition sine qua non for a complete data collection in view of precise structural investigations.

Description of instrument/infrastructure

With ALADIN, our team wishes to create at ILL an advanced Laue Diffraction instrument suite, based on the existing Laue diffractometers VIVALDI (Image Plate detection technology when studies on ultra-small crystals are envisaged) and the CYCLOPS (intensified CCD-scintillator technology when fast acquisition or sample environment aspects are dominant). In order to outperform existing instruments worldwide, it is essential to be able to use a clean, white, thermal neutron beam, with a bandwidth from ~0.6-0.7 to 5 Å.

The envisaged configuration is the installation of one of the Laue machines CYCLOPS or VIVALDI in the new guide depending on the final results we will obtain on the benchmarking of both. This option is open and the details of the final configurations may evolve during the study phase. In the present situation at ILL it is difficult to find end-of-guide positions as required for Laue instruments. Only three options can be foreseen:

Option 1: Relocate CYCLOPS or VIVALDI on the guide H23.

This option implies finding new positions for the service diffractometers (the present VIVALDI or CYCLOPS position may accommodate one of them), but at the cost of a lower flux in particular in the short wavelength range if the existing guide is kept.

Option 2: Extract a small guide from the position of the present tomography and install the Laue machines within the reactor hall.

This option has the advantage of getting a huge flux on the sample (a factor 10 with respect to the present situation on VIVALDI), but needs still a detailed study for constructing the appropriate shielding for reducing the background.

Option 3: Refurbishment of H24

Other projects at ILL, as the extreme conditions diffractometer (XtremeD) and the demand of the IN13 CRG, envisage the refurbishment of the thermal guide H24. This will be a unique opportunity to create an additional small thermal guide above H24, in order to feed the ALADIN Laue machine. This small section guide will start, together with the main refurbished H24, from the OT2 H24, and would have a section of 50x50mm^2, a total length of ~28m and an m=2 supermirror coating.

With the construction of such a dedicated neutron guide we foresee an improvement of the quality of data by a factor of ~5. In particular the internal reliability factor (R_{int}), measuring the equality of equivalent reflections, will improve from presently Rint ~0.15 at best to R_{int} ~0.05 with the new guide. The aim is to get values of R_{int} comparable to those obtained on monochromatic instruments even for samples in the range of 0.001mm^3.

We have not yet evaluated the cost of the changes but we think that the additional cost, of adding a small section guide, to the refurbishment of H24 will amount to around 500k€.

20. NESSE: NEw Standards in Sample Environment

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Abstract

The actual sample environment equipments suite has been designed for instruments built a few decades ago. Since then, we have considerably improved the ILL instruments and the experiments are slowed down by the performance of the sample environment. We propose to establish a new standard that will permit to exploit the new-generation instruments and to develop the environments not presently available for investigating novel scientific challenges.

Motivation

The sample environment equipments suite does not fit the latest generation of ILL instruments and the novel scientific challenges. For example, the powder diffractometers and the time-of-flight spectrometers have seen a flux increase of several orders of magnitude leading to a new quality of experiments. On IN5, reasonable statistics over the full accessible Q-range have been obtained on a single Ø3 mm capillary of water (approximately 4 mg) in not more than 10 minutes of counting time. Under these conditions the sample environment becomes even more crucial than in the past. Not only we need reliable equipments covering very large ranges of temperature, pressure, magnetic and electric fields, we must also be able to change the physical parameters much more rapidly as well as exchanging samples automatically. Further to these needs, there are many scientific challenges that cannot be investigated without specific equipment presently unavailable at ILL. For example, among many subjects, high-pressure gas-handling systems are required to improve the storage of hydrogen; specific pressure cells would contribute to the optimization of the food distribution and ultra-high temperature furnaces would allow us to investigate the preparation process of ceramics, cement and refracting materials.

Description

ILL has a strong track record in producing standards in sample environment for the whole neutron community. This proposal aims at renewing with this tradition by putting considerable efforts in developing the environment adapted to the latest generation of ILL instruments and new scientific challenges.

To fully profit from the new instrumental capacities we would like to have:

- fast cooling cryostats with modular tail designs
- cryogen-free cryostats and cryofurnaces featuring a sample changer using standardized sample containers
- temperature controlled humidity chambers, rheometers and acoustic levitators