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The new single-crystal neutron Laue diffractometer in Berlin

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Abstract. We have recently completed construction of a thermal neutron Laue diffractometer in the experimental hall of the BER-II reactor at HZB in Berlin. The Fast Acquisition Laue Camera for Neutrons (FALCON) receives a direct stream of neutrons with a low gamma radiation count. Whilst other neutron sources report that guides containing multiple instruments interfere with the intensity and quality of the neutron beam reaching end positions, FALCON will benefit from a beam that does not pass through any objects upstream. A uniquely-designed shutter and beam definer deliver a highly focused neutron beam to the instrument with <1° divergence. The instrument comprises two scintillator plate detectors coupled to four iCCD cameras each. The neutron beam passes through the detector units enabling one detector to be placed in the backscattering position and the second detector in the transmission position. The image-intensified CCDs are capable of obtaining 20-bit digitization Laue images in under ten seconds and variable sample table and detector positions allow a full range of sample environments to be utilised. Simulations using McStas and the HZB in-house software, VITESS, show the expected resolution of our diffractometer. FALCON is currently in the commissioning phase and should be available to users in 2015.

1. Introduction

The neutron instrument suite at the Helmholtz-Zentrum Berlin had never included a Laue diffractometer, however, as of 2014, the Fast Acquisition Laue Camera for Neutrons (FALCON) has completed its construction phase. In this paper we describe the design process and decisions taken to produce the instrument and present some simulations of expected performance. Neutron Laue diffraction is commonly thought of as only being useful for the orientation of single-crystal samples. However, as has been shown by instruments at reactor sources such as VIVALDI [1], LADI [2] and KOALA [3] and at pulsed sources such as SXD [4] and SENJU [5], a wealth of science has been performed using this method.

Using the method of Laue diffraction a measurement of a large area of reciprocal space can be captured in a single image. The resulting Bragg reflections can be analysed to generate information about the structure of a sample through a phase change, for example, or to show additional reflections arising from the magnetic lattice. In particular, the studies of protein crystallography [6, 7] benefit greatly from analysis of Laue diffraction data, whilst the flux and intensity requirements for high-pressure studies using restricted-view high-pressure cells also benefit from the Laue method [8, 9].

The 'white' beam necessary for the Laue technique allows samples of a very small size (relative to those normally required for neutron scattering measurements) to be measured whereby crystals typically 1mm^3 in size are sufficient for high-quality data to be obtained.

2. Instrument Concept

The FALCON project was to be realized within three years and using a budget of just 300,000€. This small budget immediately affected the choice of detectors – arguably the most critical component of the instrument – and therefore the option of ^{3}He detectors was disregarded. Photonic Science had already supplied reliable and effective scintillator detectors to ILL for instruments such as Orient Express[10] and CYCLOPS [11]. Similar instruments for the orienting of crystals are used at other neutron sources, with JOEY [12] being open for user service.

Our intention is to perform hard condensed matter studies using FALCON; for example to investigate structural phase changes of perovskites under high pressure, and so we opted for a thermal beam profile with a peak at ~1.4Å. The Laue instrument was then planned for construction in the experimental hall of the BER-II reactor where instruments have a typically short beam path to the reactor core. Many of the high-pressure cells in use within the neutron scattering community have a narrow entrance hole for neutrons to enter, such as the Paris-Edinburgh cell [13], and so we wanted to ensure as high a flux as possible.

As a Laue instrument uses a polychromatic beam, such instruments must be built at the end of a beamtube or guide. Typically, such an instrument does not have sole use of such a beamtube, and as has been shown by measurements on the H22 guide at the ILL [14] monochromators from other instruments severely hamper the quality of the beam profile arriving at the end position. It is possible to use the neutron beam that has passed through a monochromator further upstream, however, this guarantees that the wavelength associated with that monochromator will be excluded from the polychromatic beam profile and would result in the loss of sets of Bragg reflections from the Laue pattern.

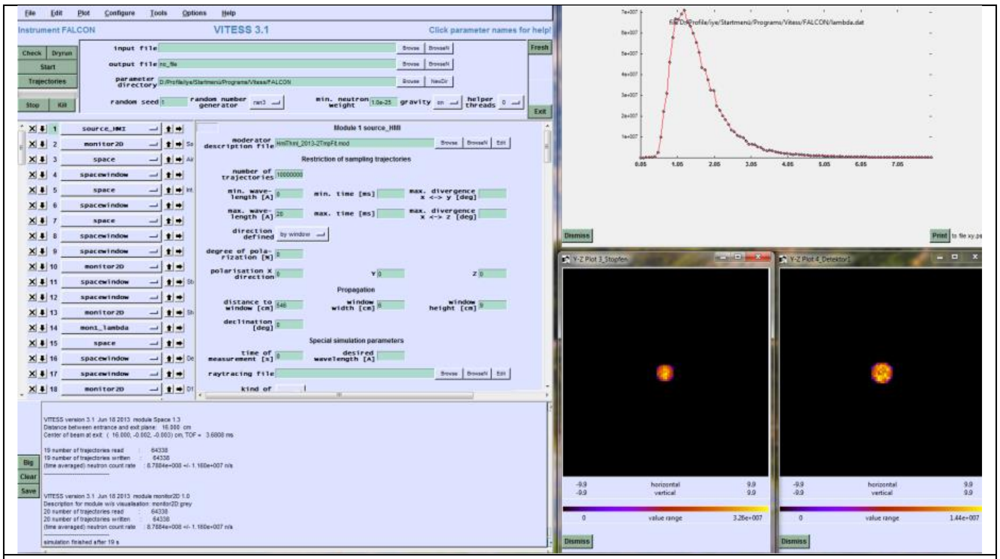
At HZB the instrument suite is being down-sized due to the imminent closure of the reactor in 2019. Therefore we were able to choose a beamtube for FALCON formerly housing two monochromator-style instruments which have been subsequently decommissioned. With both monochromators out of the beam, and an exit port just 8m from the reactor core, we have a high-flux neutron beam at the sample.

2.1. Beam profile

In order to provide a suitable beam profile for high-pressure studies, we utilised the pinhole principle. An 8m beamtube with height 90mm has a divergence of 0.64°, however, this must be collimated to provide a safe working beam of approximately 10mm in diameter. In order to match the divergence of the beamtube, we designed a well-shielded beam collimator that would simultaneously deliver a pinhole beam to the sample, and provide a shutter mechanism for opening and closing the beam. Further details of the mechanism have been reported elsewhere [15].

The exit hole using our in-house designed collimator is 16mm in diameter. This beam then travels through 5cm of air and through the centre of the first detector, impacting the sample approximately 50cm downstream diverging slightly due to the 20mm exit hole in the detector box. Simulations with VITESS [16] show a beam profile at the sample consistent with a thermal profile and is illustrated in figure 1.

Such a beam profile at the sample ensures that we can maintain our expected resolution of ~200μm whilst operating in 2x2 binning mode. This mode is most often used as it provides a good signal-noise ratio.



input file
output file no_file
parameter directory D:/Profile/iye/Startmenü/Programs/Vitess/FALCON
random seed 1
random number generator ran3
min. neutron weight 1.0e-25
gravity on
helper threads 0
moderator description file HmiThml_2013-2TmpFit.mod
number of trajectories 10000000
min. wave-length [A] 0
min. time [ms]
max. divergence x <-> y [deg]
max. wave-length [A] 20
max. time [ms]
max. divergence x <-> z [deg]
direction defined by window
degree of pola-rization [%] 0
polarisation x direction 0
y 0
z 0
distance to window [cm] 546
window width [cm] 8
window height [cm] 9
declination [deg] 0
time of measurement [s] 0
desired wavelength [A]
raytracing file
kind of

```
VITESS version 3.1  Jun 18 2013  module Space 1.3
Distance between entrance and exit plane:   16.000  cm
Center of beam at exit  (  16.000, -0.002, -0.003) cm, TOF =   3.6808 ms

19 number of trajectories read          :      64338
19 number of trajectories written       :      64338
(time averaged) neutron count rate      : 8.7884e+008 +/- 1.180e+007 n/s
----------------------------------

VITESS version 3.1  Jun 18 2013  module monitor2D 1.0
Description for module w/s visualisation: monitor2D grey
20 number of trajectories read          :      64338
20 number of trajectories written       :      64338
(time averaged) neutron count rate      : 8.7884e+008 +/- 1.180e+007 n/s
----------------------------------
simulation finished after 19 s
```

Figure 1. VITESS simulations of FALCON beam at shielding exit hole and at backscattering detector exit hole.

2.2. Detectors

Essential for the Laue technique is to have a detector to collect backscattering reflections as these are the strongest and most easily treatable reflections. Neutron image plate detectors such as VIVALDI and KOALA consist of almost 360° detector plates along the vertical axis whilst SXD and SENJU have a configuration of several small detectors placed in a spherical arrangement around the sample to capture as many reflections as possible. With our limited budget we opted for two scintillator plates; one to be fixed in the backscattering position, with another free to rotate about ω. In this way, a full map of reciprocal space can be generated with a few 'snapshots' at pre-determined positions around the sample.

Photonic Science [17] provided us with two detector boxes containing a 40x40cm scintillator plate of 6Li:ZnS:Ag coupled to four image-intensified CCDs (iCCD) each. In order to preserve the integrated chips within the cameras, each camera is placed at 90° with respect to the incoming beam. A mirror then placed at 45° to the beam direction, directs the created photons towards the image intensifier in order to protect the cameras from further degradation.

In keeping with the direct geometry of the instrument, the detector boxes designed by Photonic Science incorporate a steel tube which passes through the exact centre of the box to transport the neutrons directly to the sample. This then appears as a black hole in the centre of a FALCON image [18].

3. KOALA Data

During beamtime at the OPAL reactor operated by ANSTO, we measured Cu_2ZnSnS_4 samples [19] to investigate the site defects in these solar cell absorber materials [20]. Data from Laue diffractometer image plates has a resolution of ~100μm showing clear and distinguishable Bragg reflections, as shown in figure 2.

Whilst the scientific findings of this experiment are reported elsewhere [21], this data serves as a reference dataset for the commissioning of FALCON. The exact same samples measured using KOALA will be measured on FALCON, and in this manner we can calibrate our detectors and geometry algorithms correctly.

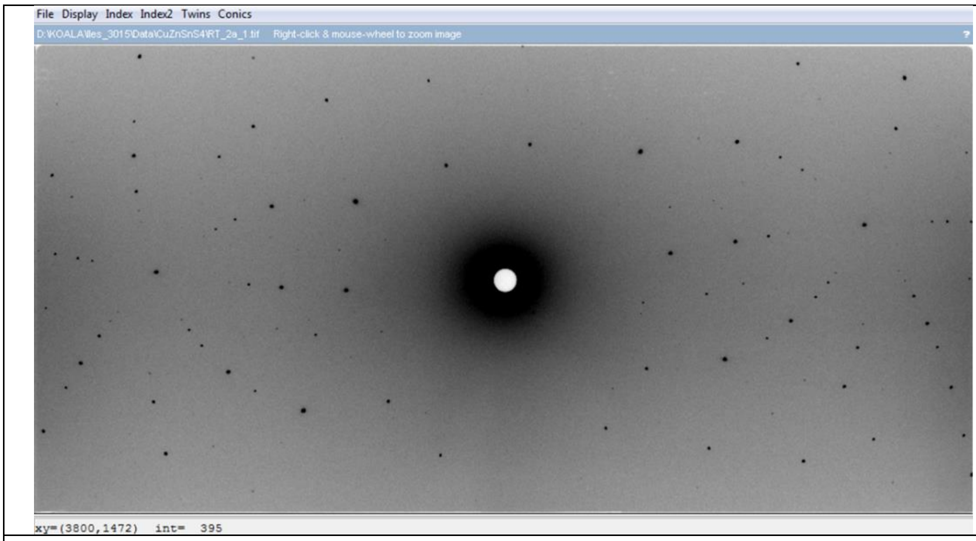


Figure 2. Example of Laue data obtained using KOALA (ANSTO) on a single-crystal sample of Cu_2ZnSnS_4.

4. Simulation results

The European Spallation Source (ESS) [22] is currently in the design phase and therefore extensive use is made of the simulation programme McStas, a monte carlo ray-tracing software for simulating neutron beams and scattering of particles through interactions [23]. One of the particularly useful features of this programme is a library of software for simulating existing neutron instruments at neutron sources around the world. With a minimum of experience a researcher can use McStas to simulate the results of a neutron scattering event with a sample of their choice on any instrument. To this end we have generated the code for simulating FALCON in McStas.

4.1 McStas simulations

Incorporating all the elements of the instrument, including the correct dimensions of the upstream elements e.g. shutter, collimators, FALCON has been simulated using McStas as illustrated in figure 3.

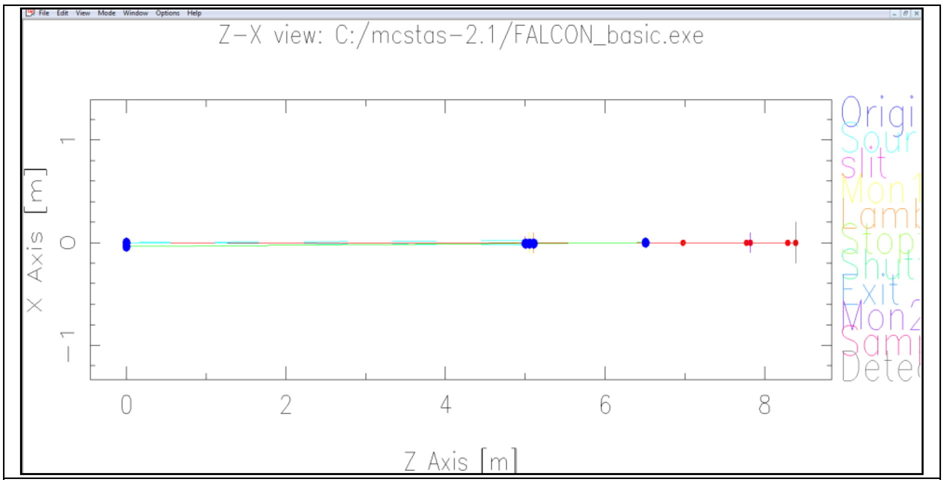
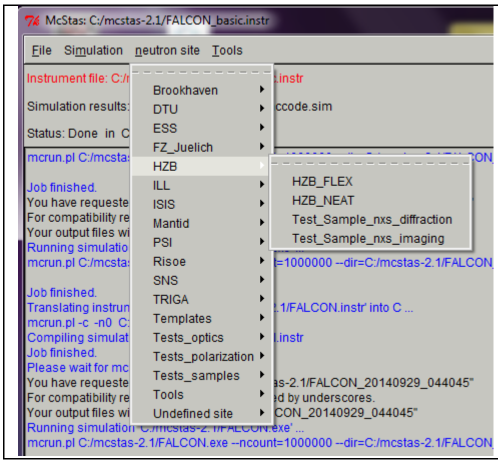


Figure 3. McStas ray-tracing simulation of FALCON from reactor core to sample.

In order to perform a simulation using the FALCON code, one must first download the McStas software, and then select FALCON within the drop-down menu as shown in figure 4.



```
Instrument file: C:/r          .instr
Simulation results:            ccode.sim
Status: Done  in  C
mcrun.pl C:/mcsta:

Job finished.
You have requeste
For compatibility re
Your output files wi
Running simulatio
mcrun.pl C:/mcsta:             t=1000000 --dir=C:/mcstas-2.1/FALCON

Job finished.
Translating instrun            .1/FALCON.instr' into C ...
mcrun.pl -c  -n0  C:
Compiling simulat              .instr
Job finished.
Please wait for mc
You have requeste              as-2.1/FALCON_20140929_044045"
For compatibility re           ed by underscores.
Your output files wi           CON_20140929_044045"
Running simulation 'C:/mcstas-2.1/FALCON.exe' ...
mcrun.pl C:/mcstas-2.1/FALCON.exe --ncount=1000000 --dir=C:/mcstas-2.1/FALCON
```

Figure 4. McStas drop-down menu showing location of FALCON simulation code.

5. Conclusion

Construction of FALCON is complete and in 2015 this Laue diffractometer will enter the commissioning phase. Simulations of the beam profile have been performed using VITESS whilst the instrument code for simulating neutron scattering from a given sample has been generated in McStas. FALCON will enter user service once commissioning is complete.

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