A High-Resolution Powder Diffraction and Small/Wide Angle X-ray Scattering (SAXS/WAXS) Beamline on MAX IV

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A combined powder diffraction and small/wide angle X-ray scattering beamline is described. This beamline is one of the proposed first-phase beamlines to be placed on the 3 GeV ring at the suggested new MAX IV facility. The proposal is designed to take full advantage of the unique properties of the MAX IV accelerators and at the same time to reflect the demands of potential users.

1. Background

MAX IV, a new synchrotron light source to be placed in Lund, Sweden, has been proposed. It would be a successor to the existing MAX I and MAX II storage rings that are today in operation. The history of synchrotrons in Lund began in 1986 with the MAX I ring. This is a soft X-ray ring operating at 550 MeV, partly used for synchrotron radiation production and partly as a source of energetic electrons for nuclear physics experiments. Today, there are 5 beamlines in operation on the MAX I ring, working at photon energies up to 200 eV. In 1996, His Majesty King Carl XVI Gustav visited MAX-lab for the inauguration of the MAX II ring. This ring is operating at 1.5 GeV, and with high magnetic field insertion devices it is possible to reach photon energies in the hard X-ray (>10 keV) regime. Presently, there are four soft X-ray beamlines equipped with undulators for the VUV and soft X-ray energies as well as three hard X-ray beamlines with multipole wigglers in operation on MAX II. Recently there was the first stored beam in the MAX III ring. This is another soft X-ray ring operating at 700 MeV with performance that is superior to that of the old MAX I ring. In some respects, it has also served as a prototype for the magnetic lattices of the MAX IV rings.

2. MAX IV

The design of the MAX IV facility (Fig. 1) is unique in the sense that it will consist of two large storage rings operating at different energies and placed on top of each other. There will also be a third, much smaller low-energy ring in the same building. The idea behind this design is to offer electromagnetic radiation of a very high quality over a broad spectral range, from IR radiation, VUV radiation and soft X-rays to hard X-rays with dedicated storage rings for the different energy regimes without compromising on the performance. By putting the two rings upon each other, a lot of the infrastructure can be shared and the total building size be minimized. The two larger rings will have a circumference of 287 m and operating energies of 3 and 1.5 GeV, respectively. The third ring would simply be the MAX III ring transferred to the new lab. Some of the basic machine parameters of the three rings are listed in Table 1. The key parameter of the two MAX IV rings is the extremely small emittance. This emittance, combined with the new generation of small-gap insertion devices, will result in an extremely competitive brilliance of the photon beam.

Table 1. Basic machine parameters of the storage rings.

	MAX III	MAX IV soft	MAX IV hard
Circumference [m]	36	287.2	287.2
Electron Energy [GeV]	0.7	1.5	3
Current [A]	0.5	0.5	0.5
Horizontal emittance [nm rad]	14	0.34	0.8

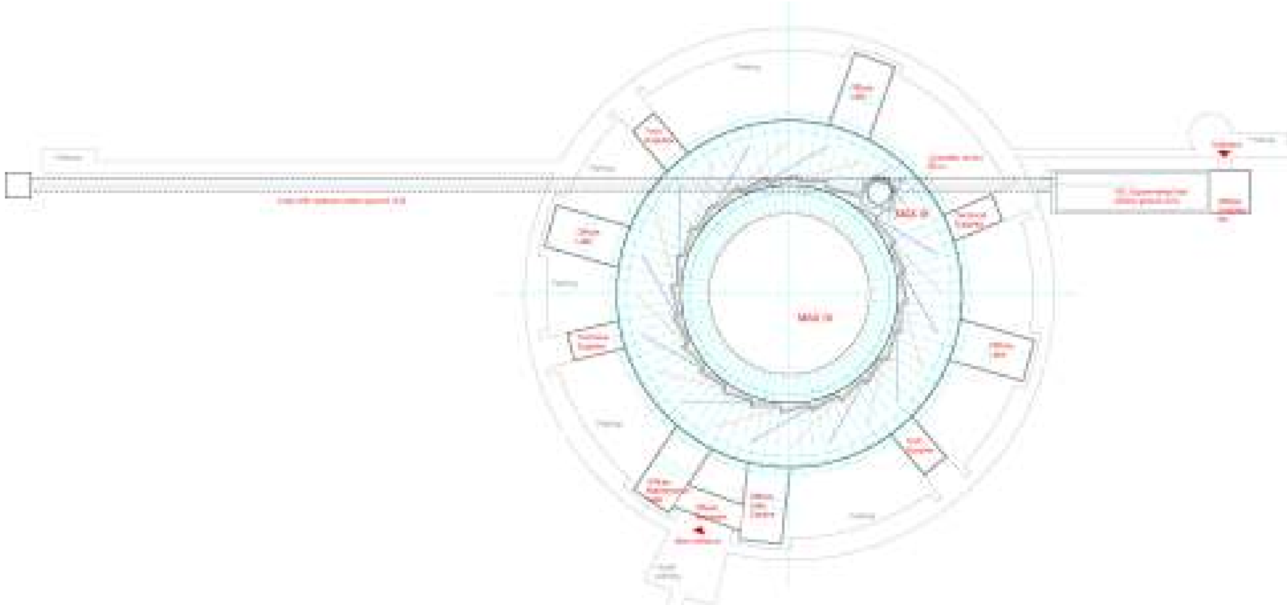

Figure 1. The proposed MAX IV facility with a several hundred meters long linac for injecting the three storage rings and for feeding a FEL (Illustration from the Conceptual Design Report of MAX IV).

Figure 2. Andy Fitch (ESRF) at the Powder Diffraction workshop held at MAX-lab in January 2006 (Photo by W. Paszkowicz).

A 3 GeV linear accelerator will be used for full energy injection of all three storage rings, which allows the rings to be run in top-up mode. The linac will also be used for generating short X-ray pulses and, at a later stage, for feeding a Free Electron Laser (FEL).

3. First-phase beamlines

There will be twelve straight sections on each of the two large storage rings, eleven of which can be used for insertion devices for beamlines. In principle, the beamlines from the straight sections will then be oriented 30 degrees apart. However, because the two rings are rotated 15 degrees relative to each other, there will be only 15 degrees between them. In addition some of the bending magnets could be considered to be used, for instance for relocated beamlines from the MAX II ring. Thus, the floor space will be somewhat limited. In any case physical and budget limitations will also limit the number of possible beamlines that can be built at the MAX IV facility. Together with funding of the machine there is a proposal for 15-20 first-phase beamlines. The proposed designs and scientific areas of these beamlines are based upon an analysis of the user demand as, for instance, formulated at a number of workshops. They should take full advantage of the unique performances of the MAX IV rings. Much of the beamline work started with the “Our Future Light Source” workshop, held in Lund 27-29 September 2004 with more than 400 persons attending, to identify the areas of interest for the MAX IV facility. The work has then continued in smaller groups, going into more detail on the different beamlines and experimental techniques. In order to host a wide range of highly demanded experimental techniques it was early realized that some of the beamlines should combine two or more techniques. One of these suggested combined beamlines is a high-resolution powder diffraction and small/wide angle X-ray scattering (SAXS/WAXS) beamline.

4. The design work

In January 2006 there was a workshop in Lund covering powder diffraction at MAX IV (Fig. 2) and another one in Uppsala about small angle X-ray scattering. These workshops were attended by academic and commercial expertise, and by present beamline users as well as potential future users. On the agenda there were talks about the required performance and instrumentation of the beamline(s) serving these user communities, as well as discussions of scientific opportunities and attempts to identify future trends. The beamline presented below is to a large extent the result from these workshops. However, some of the outcome deserves to be highlighted: It was realized that the performance of the high energy ring of MAX IV would permit an X-ray beam with very suitable properties for both powder diffraction and SAXS/WAXS and that there are possibilities to combine these techniques onto the same beamline. There must be special precautions taken, such as the use of extensive beam diagnosis equipment and high accuracy optics, to ensure easy exchangeability. A wide variety of different sample environments must be offered to provide extensive possibilities for in-situ studies, and these environments must be well incorporated in both the hard and soft-ware of the beamline. There was a consensus at the powder diffraction workshop that the beamline must host possibilities for high resolution data acquisition suitable for Rietveld refinement of complex substances. At the same time it should be possible to perform kinetic experiments, preferably down to the millisecond regime. There are several examples of beamlines with diffractometers that can cope with these requirements by combining a high-resolution scanning point detector and a rapid position-sensitive detector in the same instrument [1, 2]. At the moment there is a rapid development within the area of linear detectors. As an example of a present state of the art detector several participants mentioned the Swiss microstrip detector [3], where 15,000 channels covering 60 degrees can be read out in just a few tenths of a millisecond. Automated sample handling was also considered to be important. This would enable parametric studies of samples with, for instance, varying composition.

The main conclusion from the SAXS workshop was the importance of offering a flexible set-up permitting a wide variety of different sample chambers, including the possibility to do windowless measurements for weakly scattering samples. A pinhole based set-up is giving the best flexibility, and with a low divergence beam it is still possible to reach very small scattering angles.

5. A beamline for High-Resolution Powder Diffraction and Small/Wide Angle X-ray Scattering (SAXS/WAXS)

5.1. General Layout

The new beamline would consist of separate experimental hutches for the powder diffraction and SAXS experiments but with a common front end and optics set up (Fig. 3). The properties of the MAX IV beam coupled with the use of bimorph mirrors would permit close to ideal beam properties for both applications although sharing the optics. The two applications can not run independently of each other but the switching between techniques would be a reasonably fast process since both experimental set-ups would be permanently mounted on the beamline.

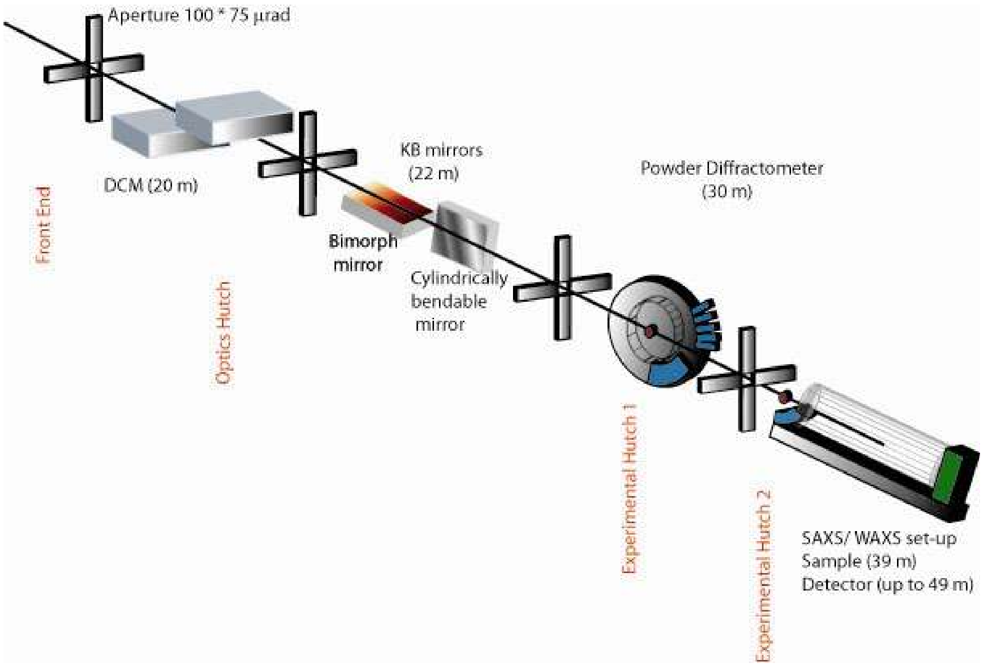

Figure 3. The general layout of the proposed combined powder diffraction and SAXS beamline.

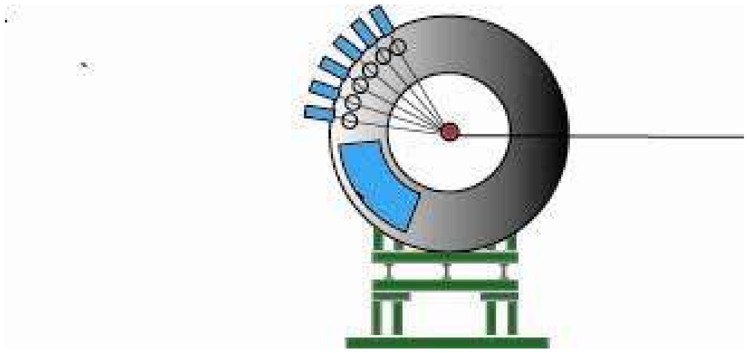

Figure 4. The high-resolution powder diffractometer with several analyser crystals/ scintillation counters and a rapid PSD detector at negative 2θ for kinetic studies.

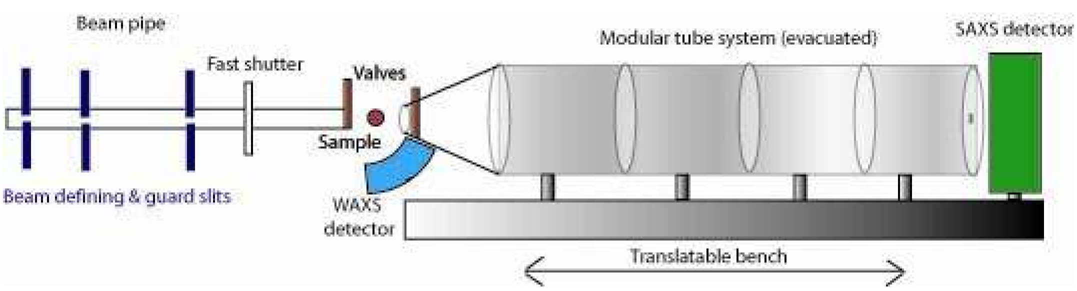

Figure 5. The SAXS/ WAXS set-up in experimental hutch 2.

5.2. Beamline optics and beam properties

The beamline design has been based on a 3 meter long, cryogenically cooled, in-vacuum undulator with permanent magnets. It has an energy range at the MAX IV ring from 5 to 30 KeV when utilizing up to the 9'th harmonics. The software Spectra 7.2 [4] has been used for the initial design calculations, and the ray tracing has been done with Shadow VUI 1.0 [5]. In the front end there is an aperture accepting 100×75 μrad that will significantly reduce the heat load on the optics downstream. It is followed by a 0.2 mm thick water-cooled diamond window separating the beamline from the storage ring vacuum. The diamond also works as a low-energy filter, absorbing most of the radiation below 5 keV. At 20 m from the source there is a fixed-exit, cooled Si(111) double crystal monochromator. The heat load on the first crystal of the monochromator will be of the order of 20 W/mm^2 at K = 1.874 (λ = 1 Å). This must be cooled with liquid N_2 to achieve thermal stability. The monochromator is immediately followed by a guard slit and thereafter, at 22 m, by a pair of Kirkpatrick-Baez (KB) mirrors. The first is a cylindrical bimorph mirror that will either collimate the beam in the vertical direction for the powder diffraction experiments or focus it on the SAXS/WAXS set up. The second one could either be a flat and cylindrically bendable mirror or a bimorph mirror. In the first case it would focus the beam in the horizontal direction for the SAXS/WAXS set up and would be moved out of the beam for powder diffraction measurements. In the latter case, it would either focus the beam onto the powder or the SAXS set up. The resulting beam size for powder diffraction would be 1×0.6 mm^2 in the unfocused case, with an extremely low divergence of 29×3 μrad^2. Alternatively, with a second focusing bimorph mirror, the horizontal beamsize would be slightly smaller but with a higher divergence. With the same optics it is possible to obtain a 1:1 focus at 44 m with a beam size of 190×30 μm^2 and slightly detuned from the 1:1 focus a beam size of 200×100 μm^2 with a divergence of 30×20 μrad. The calculated flux on the sample will be of the order of 6×10^{13} photons/s at 1 Å and with a bandpass of 10^{-4}.

5.3. Instrumentation

Powder Diffraction

At 30 m from the source in the first experimental hutch a high resolution diffractometer (Fig. 4) will be mounted. The detection system will consist of several Si(111) analyser crystals coupled with scintillation counters that will ensure the best possible angular resolution matching the very small divergence (29×3 μrad^2) of the X-ray beam. This in combination with good count rates and a wide range of different sample environments (varying temperature, pressure, electric field, and humidity as well as mechanical stress and strain) will make it possible to perform detailed structural analysis of complex materials at an array of different conditions. The sample handling will be fully automated to enable high-throughput investigations. In combination with the high resolution instrument, there will also be a position sensitive linear detector with a fast read-out mounted at a negative 2θ angle on the diffractometer. This detector will provide a possibility to make kinetic studies down to the 0.05 seconds regime. The two types of detectors could be mounted simultaneously on the goniostat for a simple switch between the kinetic and high resolution measurements. There will also be a possibility to let the X-ray beam bypass the diffractometer onto an experimental table with user supplied temporary set ups in combination with an area detector. When the powder diffraction station is not in use the diffractometer is rotated so that an additional beam flight tube can be mounted and the X-ray beam can bypass into the next experimental hutch.

Small Angle X-ray Scattering

The SAXS/WAXS sample will be situated 39 meters from the source and in front of it the beam defining and guard slits to minimize the parasitic scattering cone as well as a fast shutter and a beam intensity monitor are placed. The sample to the SAXS detector distance can be varied up to 10 meters with an evacuated modular tube system. The KB mirrors will have the possibility to provide a one to one focus at 44 meters from the source with a beam size as small as 190×30 μm^2 but if slightly detuned from the 1:1 focus a beam size of 200×100 μm^2 with a divergence as low as 30×20 μrad^2 can be reached. A low divergence is important for all SAXS experiments and becomes increasingly important as the scattering angle gets smaller. The complete tube and detector system can be translated along the beam to permit large variations in the sizes and types of sample environment cells and to minimize the air path once the sample is in place (Fig. 5). When special precaution must be taken to minimize the background signal, for example for weakly scattering samples such as proteins or other macromolecular substances in solution, the tube system can be attached via the sample chamber to the beam pipe to permit windowless measurements.

6. Timeline

At the time of writing the conceptual design report (CDR) is being evaluated by the Swedish Research Council. This design report is available to download through the MAX-lab homepage (www.maxlab.lu.se). It consists of both the machine design and the preliminary designs of the suggested first phase of beamlines. The result from this evaluation will of course to a very large extent decide if and how this project can proceed. Below is a potential timeline for MAX IV if a positive evaluation will result in funding of the project within this year.

2002 Start of the MAX IV planning,
Project discussed at the MAX-lab Users Meeting,
Presented in connection with the Swedish Research Council evaluation of the National Facilities,
CDR funding from the Wallenberg foundation.

2003 Research Council funding of a FEL research program.

2004 Workshop "Our Future Light Source" - Large attendance.

2005 The Lund University requests the City of Lund to plan for the location of MAX IV,
The Research Council defines the evaluation procedure,
The machine part of the CDR is delivered,
Project discussed at the MAX-lab Users Meeting,
Funding of further design studies.

2006 The scientific case and a proposal for the first phase of beamlines,

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Detailed design – requires additional funding.

2007 Preferred start of construction.

2011 First operation.

2012 Start of phasing out of MAX II.

Conclusion

At the time of writing the funding of the MAX IV project is by no means decided and it represents a major investment. It would, however, be a world-class facility that would enable a large number of user groups from mainly northern Europe to perform their synchrotron radiation based research at the best possible experimental conditions. The high-resolution powder diffraction and small/wide angle X-ray scattering beamline is one of the first-phase beamlines that are described in the conceptual design report. The design takes full advantage of the unique properties of the MAX IV storage ring and offers very suitable conditions for both experimental techniques. It would be a very much superior successor to the present beamline I711 at the MAX II ring for both powder diffraction and SAXS/ WAXS measurements.

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