CRYSTALLOGRAPHY AT MAX-LAB

Yngve Cerenius, Thomas Ursby, and Stefan Carlson

MAX-lab, University of Lund, Box 118, S-22100 Lund, Sweden

MAX-lab [1] is a synchrotron radiation source located in Lund in the south of Sweden. It consists of two operating storage rings and a third one that is under construction. The MAX I ring has been in operation since 1986 with an energy of 500 MeV, MAX II was inaugurated in 1996 and operates at 1.5 GeV, and MAX III will be another soft X-ray ring at 700 MeV. The lab has previously been focused on soft X-ray (UV) radiation applications and nuclear physics but the MAX II ring has permitted an expanding activity in the hard X-ray regime as well. This activity started with beamline I711 [2] that has been in operation since 1998.

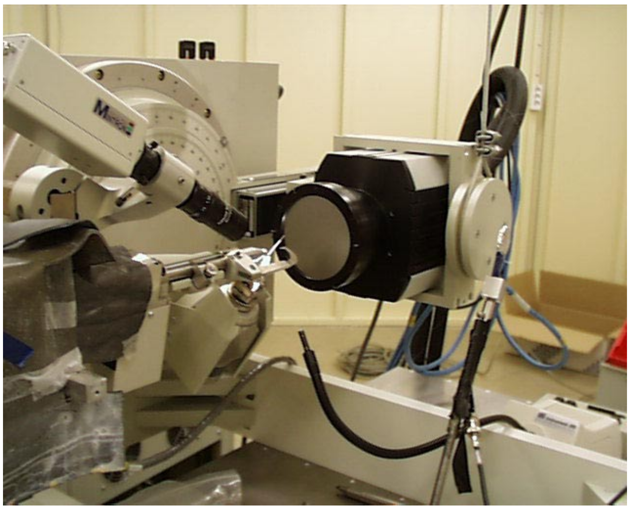

Figure 1. The set-up for small molecule crystallography at beamline I711: a Bruker SMART 1000 CCD detector on a PLATFORM 3 circle goniometer.

Beamline I711

The main optical elements of the beamline are a focusing mirror and a Si(111) single crystal monochromator. These enable a wavelength range of 0.8 -1.55 Å but with a rapidly decreasing intensity towards higher energies. The monochromator design forces the complete experimental set-up after it to be moved every time the wavelength is changed and thus sacrifices easy tunability and high energy resolution but provides high photon flux on the sample. I711 is used for macromolecular as well as small molecule crystallography and powder diffraction but it is expected that most of the protein crystallography will move to beamline I911 once it will be in full operation.

The beamline is equipped with a mar165 CCD detector from MarResearch GmbH mounted on a MarResearch Desktop Beamline ® (mardtb) goniometer system. This set-up is mainly used for protein crystallography but it has also successfully been used for powder diffraction. The area CCD detector provides the possibility to integrate over the complete diffraction rings which both seem to favor the sensitivity and of course the possibility to study preferred orientation. The beamline is also equipped with a Huber Imaging Foil Camera G670 [3] for powder diffraction work where the sample is either mounted in capillaries or on tapes. The camera covers the range from 0° to 100° with a step resolution of 0.005° in 2θ and thus enables full pattern powder data collection in one exposure. The detector is equipped with a furnace for in-situ high temperature studies up to 800°C. A Cryojet from Oxford Instruments, which is normally used for preventing radiation damage on protein crystals, has also been used in combination with this detector for expanding the temperature range down to 100 K.

The standard detector for the small molecule user community is the Bruker SMART 1000 CCD (Fig. 1). Although smaller in size than the mar165, it has the advantage that it is mounted on a “PLATFORM” goniometer. The “PLATFORM” is a 3-circle system where it is possible to rotate Φ, Ω and 2θ 360 degrees each with a reproducibility of 0.0005°. However, maximum 2θ is generally limited to 120-140° due to the position of the microscope needed for crystal alignment. As a comparison: $2\theta_{max}$ for the mar165 CCD on the mardtb is 106° and it has only a Φ axis. The SMART detector is equipped with a complete software package for data collection, integration as well as the ShelXTL package for structure determination and refinement.

Figure 2. The Newport/Micro-Controle x-ray diffractometer at I811 shown in the “kappa” configuration. The sample/UHV-chamber is aligned using a Physik Instrumente hexapod system and the detector arm is equipped with slit system, goniometer for analyzer crystal and a scintillation detector.

Beamlines I811 and I911, both have superconducting multi-pole wigglers [4] developed at MAX-lab as radiation source. This wiggler operates with a substantially higher magnetic field, 3.5 T, than the multipole wiggler of I711 which has 1.8 T permanent magnets. Therefore I811 and I911 have a critical wavelength of 2.37 Å compared to 4.60 Å for beamline I711 and slightly higher energies can be used at these beamlines.

Beamline I811

The material science beamline, I811 [5], has an energy range 2.4 - 12 keV. The design is based on adaptive optics where the beam is collimated and focused vertically by cylindrical bendable first and second mirrors. Horizontal focusing is obtained by sagittal bending of the second crystal in the double crystal monochromator. An experimental station for XAFS experiments has been implemented. A large multiple-axis diffractometer (Fig. 2), capable of handling heavy equipment such as UHV-chambers, has been installed and will be commissioned in autumn 2004. User experiments on surfaces, interfaces and thin films will start in spring 2005.

Beamline I911, Cassiopeia

The Cassiopeia beamline is designed for macromolecular crystallography and will have five independent stations. Four of these are fixed-wavelength stations (with a small wavelength tunability of 1%) at 0.91, 0.97, 1.03 and 1.25 Å. The central station is tunable between 0.7-1.8 Å. The optics used for splitting up the wide fan of radiation from the wiggler is further described in [6].

As of March 2004, two of the side stations (0.97 and 1.03 Å) have had users though so far only the 0.97 Å station is regularly taking users. The measured flux at Station 5 is 1×10^{11} photons/s in 0.15 x 0.3 mm^2 ($v\times h$), but the flux will increase when the wiggler will be operated at full field. The central station will be commissioned during spring 2004 and is expected to deliver 10^{12} photons/s in 0.3×0.3 mm^2 ($v\times h$). It is designed for optimised use of anomalous scattering in data collected with the MAD method.

Figure 3. The experiment hutch of the MAD station at I911. The kappa goniostat can be seen through the open hutch window. The setup with sample microscope (to the right), the fast and easy access to the goniostat through the hutch window, the sample alignment interface (the touch screen on the left) and the close access to the control room (2-3 m) forms a very convenient environment for the user.

The first two side stations are equipped with single-axis goniostats while the MAD station has a kappa goniostat (Fig. 3). One of the side stations will be equipped with an automatic sample changer. Presently there are two CCD-based detectors at the beamline, one with 225 mm diameter active area and one with 165 mm. The smaller CCD detector is on-loan and will be replaced by two larger solid-state detectors. There are also image-plate detectors available as back-up. The experimental hutches are small enclosures (2.5×1.25 m^2) with the sample being mounted through a sliding window.

Experimental results

Five years of crystallographic experiments at MAX-lab have resulted in some 250 articles that are based on or partly based on experimental results obtained at MAX-lab. Here are two examples of such experiments.

In Figure 4, a diffraction image from the mar165 CCD detector on beamline I711 of a 50 μm thick FeCrAl steel foil is shown. The foil has been oxidized in pure oxygen for 168 h at 900°C. It could be concluded from the image that grain size of the bulk material has increased during the heat treatment while the aluminium in the alloy has diffused and formed a small grained thin layer of aluminium oxide without any visible affects of preferred orientation. The well separated spots on the image are from the bulk material while the diffraction rings are from the alumina scale. Inserted are the integrated intensities of the raw image (data kindly provided by Dr. Mikael Grehk, Dalarna University, Sweden).

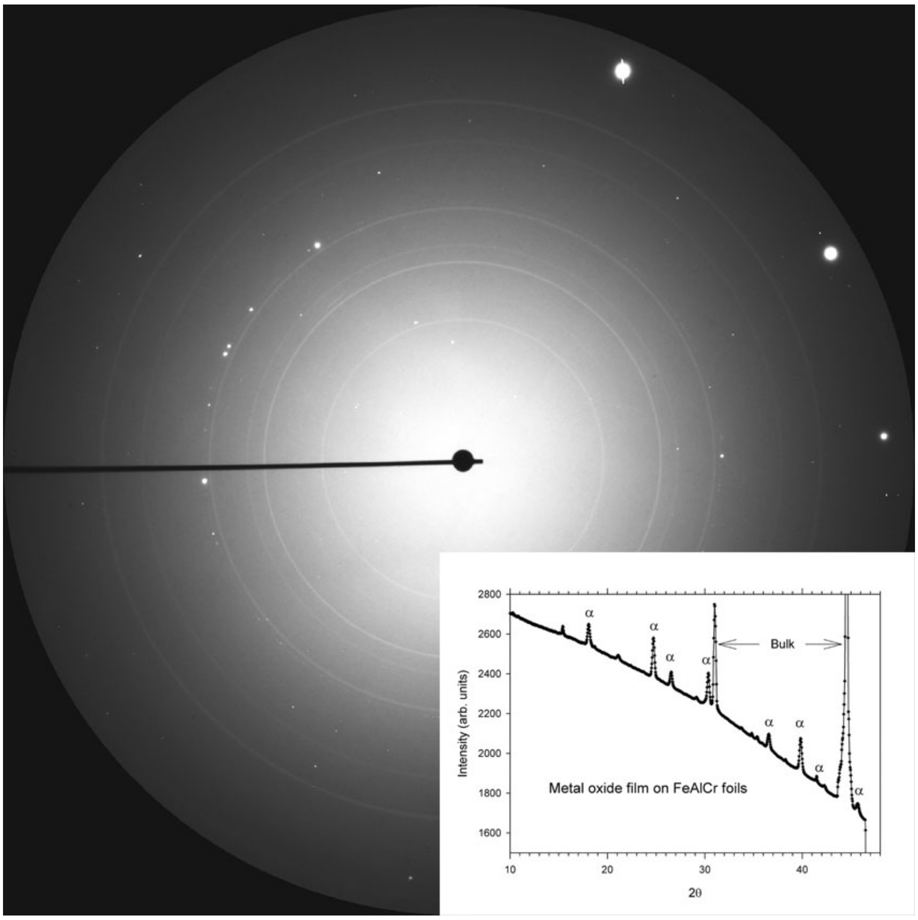

Figure 4.

A diffraction image of a FeCrAl steel foil with the integrated intensities of the raw image inserted (courtesy of Dr. Mikael Grehk, Dalarna University, Sweden).

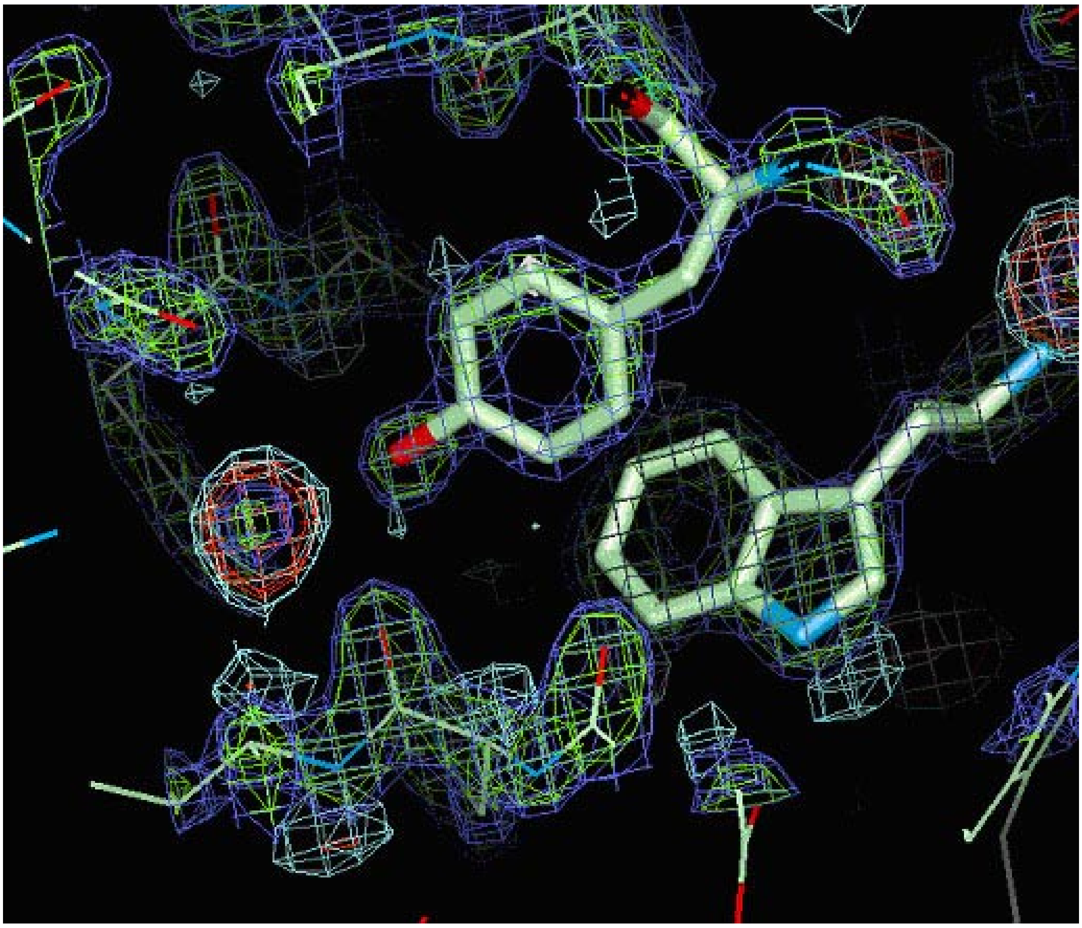

Figure 5. Electron density map calculated with data from I911-2 (courtesy of Novo Nordisk A/S).

Beamline I911 is now regularly taking users. An example of measured data is shown in Figure 5. The image shows a detail of DPPIV, which is an enzyme that is playing an important role in the immune system and in the metabolism. Understanding of DPPIV is important for the development of new diabetes drugs. The data have been measured on station 911:2 in December 2003.

Access to MAX-lab

Access to all three beamlines is through proposals addressed to MAX-lab. The Program Advisory Committee (PAC) has so far evaluated proposals once a year (in spring) and there has been a deadline for proposals in late February. However, the evaluation process will most likely change, at least for the protein crystallography user community, so potential users are advised to regularly check the MAX-lab homepage [7] for updated information. MAX-lab provides researchers from the European Union and Associated States access to the facility through the project "Integrating Activity on Synchrotron and Free Electron Laser Science" within the Sixth Framework Programme. A minor part of the beamtime is also available for commercial users.

References

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[7] http://www.maxlab.lu.se1