In this contribution, we will be providing an insight into current developments of multilayer optics for X-ray analytics in the lab and at synchrotrons. We explain the manufacturing process, summarize the different types of optics and give some examples of typical applications which benefit from the new possibilities, especially in combination with modern microfocus X-ray sources, novel metal-jet anode X-ray sources, mini-synchrotrons, beamlines and FELs.

The optics consist of bent substrates with shape tolerances below 100nm. By using sputtering technology we deposit multilayers upon these substrates with several hundreds of layer pairs and single layer thicknesses in the nanometer range. To ensure high-quality X-ray optics we fabricate the multilayers with lateral thickness gradients within ± 1% deviation of the ideal shape. We use optical profilometry in order to characterize the shape and X-ray reflectometry for the characterization of the multilayer thickness distribution both laterally and as in-depth. The microstructure is investigated by transmission electron microscopy.

Modern deposition technology allows for the reproducible production of high quality multilayer mirrors with smaller d-spacing. Thus, in combination with the latest generation of microfocus sealed tubes it is possible to provide new high-performance X-ray sources for shorter wavelengths. We will be presenting selected results on the use of our new air-cooled high-brilliance X-ray source IμS for Mo-Kα and Ag-Kα radiation in small molecule and high-pressure crystallography.

For home-lab sources our so-called Montel Optics focus or collimate the beam in 2D with a very high flux density and an adequate divergence directly at the sample position. However, synchrotrons need a higher quality of the shaped substrates. We designed and produced first Montel Optics of the third generation especially for an analyzer system at inelastic scattering beamlines.

Furthermore, we developed special multi-stripe optics for Double Crystal Multilayer Monochromators (DCMM) which are used at tomography beamlines in a wide range of photon energies (10-45keV).

In special mini-synchrotrons our longest multilayers of 40 cm in length are used. We will be showing first results.

In addition we will be presenting our total reflection optics for which we developed a large variety of ceramic and metallic layers on large substrates with a length of up to 150cm.

Keywords: X-ray optics, multilayer, source

MS07.P05

Acta Cryst. (2011) A**67,** C256

The new in situ screening facility at the MX-beamlines BL14.1 at BESSY II of the Helmholtz-Zentrum Berlin (HZB)

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In spite of large scale automation and an on-going miniaturisation process in high-throughput protein crystallisation, human efforts are required to pick individual crystalline specimens from 96-well crystallization plates and to characterize them. In addition, not optimal cryogenic stabilization solutions often mask the diffraction properties of macromolecular crystals. An alternative to this conventional approach is to expose macromolecular crystals or possible crystalline material grown in crystallisation plates directly in the X-ray beam [1]. We present the implementation of an *in situ* crystal screening platform [2] using the CATS sample changer and a MD2 microdiffractometer at the HZB-MX-beamline 14.1 [3].

The hardware implementation consists of a six-axis robotic arm and a dedicated tool for gripping crystallisation plates. This mode of operation is alternative to the normally used transfer of cryo-cooled samples. Every object within the plate can be precisely positioned in front of the X-ray beam. The robot arm acts as the omega-rotation axis during diffraction data collection.

Different proteins were subjected to standard sparse-matrix crystallisation screens in three different plate types and exposed in the beam using the robot. The results show an unambiguous identification of crystalline and non-crystalline objects. The diffraction images were successfully auto-indexed and important metrics of the crystal system could be determined. Furthermore, in some cases diffraction data sets could be collected to a high completeness.

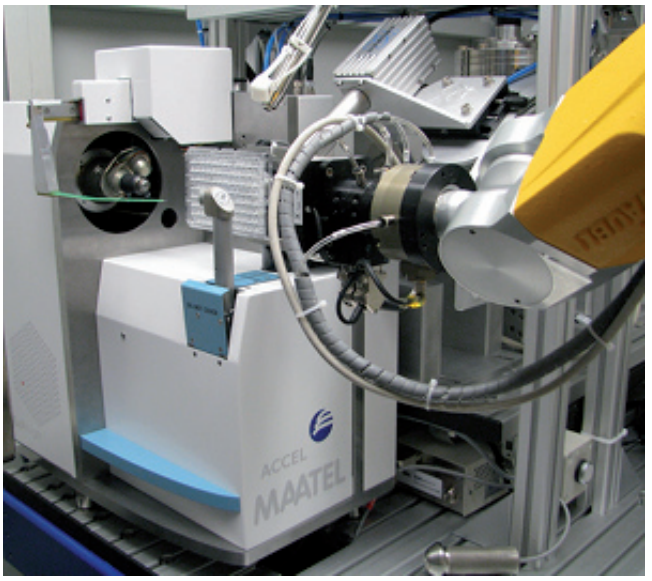

Fig. 1. A 96-well plate mounted ready for X-ray exposure

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Keywords: in-situ, bio-macromolecule, synchrotron

MS07.P06

Acta Cryst. (2011) A**67,** C256-C257

A flexible macromolecular crystallography beamline at the alba synchrotron

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ALBA is a third generation 3-GeV storage ring built near Barcelona and the Universitat Autònoma de Barcelona. Out of the seven first-phase beamlines, XALOC (BL13) is dedicated to Macromolecular Crystallography (MX). The photon source of this beamline is a 2-m long in-vacuum undulator with a period of 21.6 mm and a nominal minimum gap of 5.5 mm. This device has been optimized to deliver the highest flux at the Se K-edge while keeping full tunability in the 5-21 keV range. The optics consists in a cryogenically cooled Si(111) channel-cut crystal monochromator and a pair of mirrors in a Kirkpatrick-Baez or orthogonal configuration. The End Station

includes a high accuracy single axis diffractometer complemented with a removable mini-kappa mount and an automated mounting robot that can work with both cryogenic samples and crystallization plates. A photon-counting 6-Mpixel detector, built with parallel pixel electronics, offers outstanding capabilities like a large sensitive area (431×448 mm^2), a very fast framing rate (12 images/second), a large dynamic range (20 bits, $>10^6$), and negligible dark current noise.

The optical design foresees two main operation modes: an unfocused configuration, where one or both mirrors are removed from the photon beam path, resulting in a very small beam divergence of less than 0.03 mrad vertically, a mode that can be especially useful for large macromolecular complexes with large unit cell parameters; and a focused configuration, where both mirrors can focus the beam to 50×7 μm^2 FWHM (H×V) on small or microcrystals, while at the same time keeping a small and useful vertical divergence (0.1 mrad). In addition, the mirrors allow variable focusing if matching the size of the x-ray beam to the dimensions of the crystals or if focusing at the detector (which can be placed at any distance between 80 mm to 1300 mm from sample) are required. In this case, the beam size at sample position can range from 50×7 μm^2 to 300×300 μm^2 (H×V). In order to avoid x-ray beam deformations caused by the optics when defocusing, slope errors of the mounted mirrors have been reduced to 70 nrad rms and the monochromator crystal can work near the zero expansion temperature of Silicon (124 K).

To fulfil the needs of standard multiple wavelength anomalous diffraction experiments, the beamline will deliver over $3 \cdot 10^{12}$ ph/s in the 5-21 keV energy range, which covers all the common K and L_3 absorption edges, and an energy resolution of $\Delta E/E \sim 2 \cdot 10^{-4}$. Finally, a major design objective has been to optimize the beam stability at sample position to improve successful data collection. In this respect, the beamline is equipped with an exhaustive diagnostics system that includes 4-diode and diamond X-ray monitors and fluorescent screens, a feedback system on the pitch of the second crystal surface of the monochromator, and seismic accelerometers near the critical optical surfaces to monitor vibrations in real time. Moreover, the cooling system of the monochromator has been designed to work at low liquid N2 flows, close to the laminar regime.

Currently being the only MX beamline at ALBA, XALOC has been designed to deal not only with heavily automated x-ray diffraction experiments but also non-standard and trickier ones as well as a myriad of crystal sizes and unit cell parameters. XALOC is now finalizing the control system and is ready to start x-ray beam commissioning. It is expected to be opened to users at the beginning of 2012.

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Keywords: beam line, synchrotron, macromolecular crystallography

MS07.P07

Acta Cryst. (2011) A**67,** C257

High-brightness liquid-metal-jet x-ray tube

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High-end x-ray diffraction and scattering techniques such as high-resolution XRD, protein crystallography, and SAXS rely heavily on the x-ray source brightness for resolution and exposure time. Thus, synchrotron radiation sources are the obvious choice. However, many users and applications benefit from laboratory systems based on compacts sources. Unfortunately, the brightness of present electron-impact x-ray sources is fundamentally limited by thermal constraints in the anode technology and this limits their applicability.

We have demonstrated a new anode concept, the liquid metal jet [1]. This regenerative anode allows operation of micro focus tubes with an electron beam power density orders of magnitude higher than present solid or rotating anodes. The source has been demonstrated for a wide range of liquid anodes and x-ray emission energies, e.g., [1,2]. Our current liquid-metal-jet prototype x-ray source systems rely on room-temperature liquid-metal alloys as anode and typically operate at approximately one order of magnitude higher brightness than present state-of-the-art x-ray tubes. This unprecedented brightness makes the liquid-metal-jet-x-ray source suitable for a wide range of diffraction, scattering, and imaging applications.

In this contribution we will present a new version of the source which is optimized for diffraction and scattering applications. It runs with an almost pure Ga alloy and a 70 kV magnetically focussed LaB_6-based electron gun to generate intense emission of the 9.25 kV Ga-K_α line. The source typically operates with a 20 µm diameter focus and 200 W of electron-beam power. We will present a detailed characterization of the source including spot size, stability, lifetime, flux and brightness. In addition, we demonstrate the use of the liquid-metal-jet source for important applications, including SAXS and phase imaging [3], clearly showing the benefits of increased brightness.

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Keywords: X-ray, source, tube

MS07.P08

Acta Cryst. (2011) A**67,** C257

On the purirty of micro-source X-ray radiation

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Multilayer optics are currently adopted in many laboratories for single crystal diffraction, using very low power X-ray generated by micro-sources. The high-brilliance and the micro-focusing are the main advantages of these radiation sources, that allow high performance experiments also on a laboratory scale.

However, we recently discovered [1] a fundamental defect of this technology, namely the significant contamination of the characteristic radiation by low energy photons which are reflected by the mirrors because of the small incidence angle. Simple experiments show that the contamination can significantly reduce the accuracy of measured intensities, especially when Mo Kα radiation is used.

We have therefore proposed a simple and economic solution to the problem [1]: an aluminium filter of adequate thickness efficiently removes the low energy contaminant photons. Performances of Al-filtered data collections are reported and alternative solutions are discussed.

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Keywords: microsource; optics; X-ray diffraction