The MCX project: a Powder Diffraction beamline at ELETTRA

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In order to meet the diffuse need of non-single crystal diffraction experiments using SR, a new bending magnet beamline for Material Characterization by X-rays – MCX – is going to be implemented at ELETTRA, the Italian national Synchrotron Radiation facility in Trieste. The beamline has been designed to work in the range 3-12 keV, exploiting the high brilliance of ELETTRA bending magnets in this spectral region. Photon flux values of interest for specific experiments will be also available in the ranges 2.3-3 keV and 12-23 keV. The main target of the new station is research and development in the area of physics, chemistry and engineering of materials surfaces, thin films and coating technology. The intrinsic flexibility of the proposed geometry makes MCX an ideal tool for diffraction measurements from polycrystalline materials in general (thin films, powder and bulk materials). In addition to the scientific heritage, a valuable activity will be the support to technology and industrial production, for specific tasks of non-destructive control as well as for the development of new products.

1. Introduction

The bending magnet beamline MCX has been designed to meet the maximum flexibility and user-friendliness, so to be of real use as a tool aimed at the characterisation of various materials, with an experimental station allowing the structural analysis of: organic and inorganic thin films, thermally and/or mechanically modified surfaces of mechanic components, polymers, catalysts, highly disordered materials in the form of films, powders and fibres. As shown in Figure 1, the expected flux (calculated with the code Shadow) at the sample is of about 10^{12} photon/s.

The spectrum of possible applications is very broad and covers all the characteristics of X-ray diffraction applied to the study of materials (*e.g.* phase identification, structural solution and refinement, reticular defects and domains, texture analysis).

The planned flexibility of the line and the features of its optics, will allow anomalous scattering and DAFS studies from powders and thin films from the K edge of sulphur to that of platinum and using the L edge of the heavier elements. Given the output spectrum of the Elettra bending magnet, absorption edge studies on the elements from calcium to vanadium will be particularly efficient.

2. Source and front-end

Twelve bending magnet (BM) source points are available at the Elettra electron storage ring. The BM field intensity is 1.2 T for the ring operating at 2.0 GeV, and is raised to 1.45 T at 2.4 GeV. The corresponding critical energy values are E_c = 3.2 keV and E_c = 5.5 keV, respectively. At 2.0 GeV the source dimensions are 371 μm × 43 μm (horiz. × vert.), 698 μrad × 14 μrad (horiz × vert) [1].

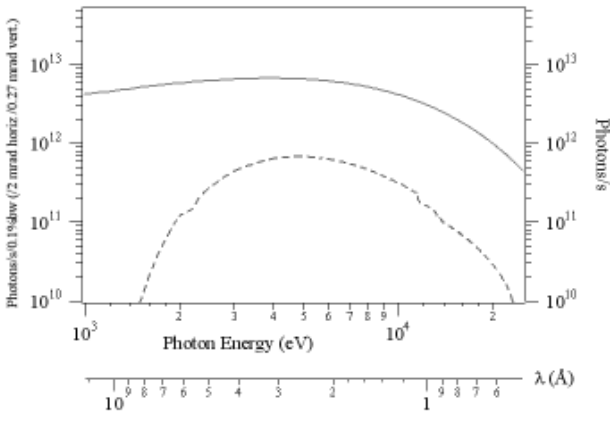

Figure 1. Calculated source brilliance integrated over the beamline acceptance (continuous line, left scale) and photon flux expected at the sample on 1x1 square mm spot (dashed line, right scale) as a function of the energy selected at the monochromator. Data are given for the ring operated at 2.4 GeV, 150 mA.

Components positioned in the front-end of the beamline consist of a water-cooled photon shutter-stopper followed by a fast closing valve (closing time < 5 ms) and a set of apertures limiting the horizontal and vertical aperture of the beam to 6 mrad, for a total power of about 70 W, for both 2.0 and 2.4 ring operating conditions (the value is given for the injection current 330 mA and 150 mA, respectively). The two spectra cross at an energy of 5.7 keV (λ = 2.17 Å) at which energy the flux per mrad in the horizontal is about 3×10^{13} photons/s/0.1%bw emitted into an approximately Gaussian profile with vertical FWHM of about 0.25 mrad. The front-end will also integrate two x-ray beam position monitors separated by about 2 m distance, which will allow the vertical beam position and direction within 1 μm and 1 μrad respectively.

3. Beamline optics

The beamline optics is shown in Figure 2. The first mirror (MIRR1) is positioned face-up and will provide vertical collimation. The mirror, manufactured by SESO (France), consists of a 80 mm wide, 1.2 m long flat Si blank, Pt-coated, tilted to a pitch of 3.5 mrad. In order to obtain the required cylindrical shape, it is positioned on a bending mechanism which allows changing the tangential radius between 6 and 15 km. This will also allow re-positioning of the beamline on the alternative multipole source, when available. The surface of the mirror is polished to a rms roughness less than 3 Å. The rms residual tangential (sagittal) slope error for the mirror mounted on the bending mechanism is less than 0.5 arcsec (3 arcsec). The mirror will be cooled by water-cooled blades immersed in Ga-filled grooves machined sideways in the mirror surface along the beam footprint.

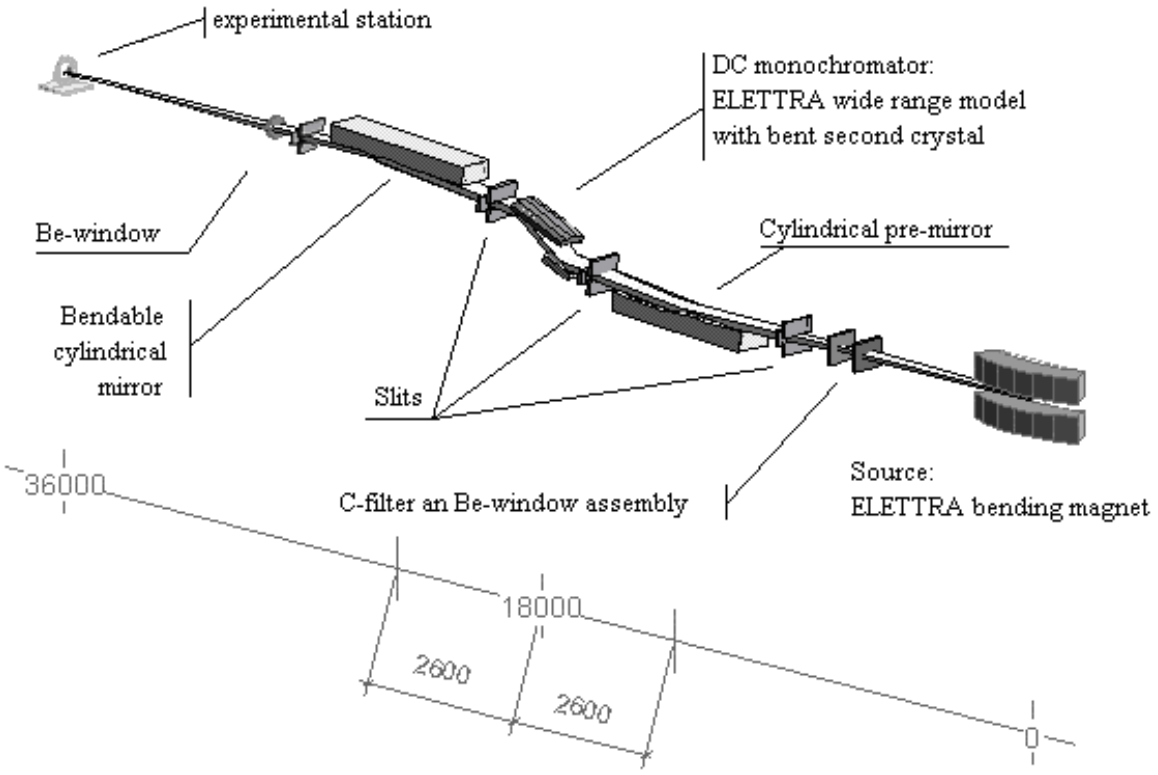

Figure 2. Schematic of the Materials Science Beamline optical system, showing, from left to right, the first (vertical collimating) mirror, the first (flat) crystal, the second (horizontal focusing) crystal and the second (vertical focusing) mirror. Distances from the source are given in millimetres.

The double crystal monochromator (DCM) consists of two Si crystals (active area 50×50 mm^2, manufactured by Polovodice, Czech Republic) cut along the [111] direction, which can be precisely positioned and oriented in the X-ray beam. Two successive Bragg reflections with an inherent energy resolution of 0.014% (given by the Darwin angular width of the Si 111 reflection) will direct photons of the desired energy parallel to the incoming beam direction, but offset upward (out of the direct Bremsstrahlung beam). This "fixed exit" operation is achieved by placing the crystals on two independent rotation stages, and translating crystal 2 along the beam direction. Additional degrees of freedom allow corrections for crystal yaw, roll and pitch.

The heat load is removed from the first crystal by indirect water cooling. The second crystal provides sagittal focusing; it is a ribbed crystal, cylindrically bent to a variable curvature radius.

Higher harmonics rejection will be obtained by de-tuning the 2nd crystal. The DCM, designed and patented by ELETTRA, will be described in detail elsewhere [2].

The second mirror (MIRR2, also manufactured by SESO) is similar to MIRR1, without the cooling system. MIRR2 also shares the same figures for dimensions, pitch, surface roughness and slope errors as MIRR1, but is positioned with the optical surface down, so that the beam returns horizontal at the end of the entire optical system. The variable curvature (6 km to flat) will permit vertical focusing at the experimental station position. Since this focusing introduces a vertical divergence, which is undesirable, for example, in high-resolution powder diffraction, the user may choose to set the radius of curvature to infinity. In this way the vertical spot dimension can be varied from 0.3 mm (focussed) to 4 mm (unfocussed).

4. Additional beamline elements

The beam definition system is the first element of the beamline, after the front-end and consists of a water-cooled copper mask (2.0×0.35 $mrad^2$). A slotted holder for a set of pyrolytic graphite multi-foil filter is integrated in the same copper block, immediately after the beam-defining aperture. The pyrolytic graphite filter will remove low-energy photons thus protecting the Be window. There is also provision for later inclusion of a beam profile monitor.

Two bent Be-windows are used along the beam-path, the first (BeW1) to separate the ring and front-end UHV environment from the beamline HV, and the second (BeW2) at the HV-air interface. BeW1, which is positioned immediately after the pyrolytic graphite filter, has a thickness of 25 μm thick, and is actively cooled, via the insertion of Indium strips side-by-side of the Viton seal ensuring UHV integrity. BeW2, that will ensure protection against pressure surges, has a thickness of 50 μm and is DURACOAT ™ coated on the air side to prevent oxidation. The absorption of the total thickness of 75 μm of beryllium is less then 0.1 at energies above 4 keV, rising to about 0.5 at 2.1 keV.

Three 4-blade slit systems will be mounted before each of the three optical elements (MIRR1, DCM and MIRR2), and a fourth slit systems is foreseen at the end of the optical path, before the last Be-window. The blades of all slit systems positioned before the monochromator are made of water-cooled Copper, while those after the monochromator are of Tungsten, without cooling.

As already mentioned a synchrotron shutter and Bremsstrahlung stopper, and a fast valve, are integral part of the safety system in the front end. An additional fast shutter will be included after MIRR2 and will trip in case of air inrush from the end-window, to provide protection of the upstream beamline vacuum sections. A safety shutter, positioned at the end of the Optics Hutch will allow experimenters to access experimental hutch (EH) with full power on the optics, thus maintaining stable operation of the optical elements.

Accurate measure of the incident flux at the experimental station, required for data normalization and for the optics feedback procedures will be provided by ionization chambers integrated in the collimation system of the diffractometer. This collimation system will be coupled to the alignment system thus allowing the accurate determination of the position of the beam and for rapid alignment of the whole instrument with the beam. A long nipple will be used to bridge the distance between the OH and the EH. This same vacuum section contains also an acoustic delay line which combines with the fast closing valve in protecting the beamline vacuum from failure of the Be-window [3].

5. Experimental Hutch

The station has been designed to provide the maximum flexibility and easy use with reasonable precision and performance and is based on a 4-circle Huber goniometer (2θ precision better than 0.0001°) in full circle configuration with a flat sample-holder plate (Ø 100 mm) controlled by a precision (1 μm) x-y-z motor system, 360° phi-rotation and -90 ÷ +90° chi-tilting. The 2*theta* rotation is provided with a shaft optical encoding system for accurate control and real-time feedback on the actual angular positions.

The instrument will be supported by a high-precision positioning station by ADC for accurate and fast alignment along the beam path.

The diffracted arm will carry, as a default, a crystal analyser/scintillation detector system. Changes in photon energy will require a (remote-controlled) realignment of these components. Provision will be made for later extension of the analyzer-detector system to a multi-analyser-detector system. A second detector system will

be based on a photo-diode counter, for high counting rates operating mode (*e.g.*, in reflectivity measurements, single crystal studies, epitaxial layers). Remote control attenuators, placed before the diffractometer, will be used to automatically reduce the incoming beam intensity, adapting it to the time characteristics of the detectors. In addition to the flat-plate mounting, capillary measurements will be possible with a simple spinner tool to be installed on the sample-holder. Provision of a removable He-enclosure allowing data collection at low energy is also foreseen. In addition chambers will be mounted on the standard sample-holder plate, for specific atmosphere control and reactive (kinetic) studies. Special attachments to be installed on request will include mechanical testing machines (4-point bending and tensile testing) as well as a high temperature stage. The whole system will be handled by means of the Portable INstrument Control interpretER (PINCER) program and associated macro sets, which is the data acquisition software in use on all XRD stations at SRS, Daresbury. Localization of the program will be carried out in the framework of collaborative joint effort of SRS, ELETTRA and the University of Trento.

6. Research opportunities of the new station

Various experiments will be possible at MCX, covering the widest range of user's needs: powder diffraction, grazing angle diffraction and reflectivity, residual stress and texture analysis, phase identification and structural studies, kinetic studies, anomalous scattering and diffraction anomalous fine structure (dafs), briefly described and illustrated in the following.

6.1. Grazing angle: diffraction

X-ray Diffraction at grazing angle [4] (both in the asymmetric (AGID) and in the symmetric (GID) configuration) give access to several applications of considerable interest, both for scientific studies and for technological development. Practical examples are: identification and study of crystalline monolayers, differences in crystallinity between surface and bulk, surface residual stresses and texture, with the possibility to observe gradients of both these parameters and of phase composition (composition depth profiling). A further interesting opportunity can be offered by fluorescence measurements in glancing incidence (TXRF), to integrate structural information with a chemical analysis down to *ppb* on most technologically relevant elements, profiting also from the tunability of the wavelength.

6.2. Grazing angle: reflectivity

This type of interference measurements is gaining a growing interest in the electronic industry, which is mostly concerned with thin films and multilayer devices made of both inorganic and, more recently, organic materials. Surface morphology (in terms or roughness, waviness, *etc*.), composition (thin oxide films, adsorbed layers) and structure of multilayer thin films can be studied in great detail. The station permits one to take advantage from the unique features of the SR, including the high brilliance, the precision in beam/sample positioning and definition of the glancing incidence condition and possibility of selecting the wavelength, in particular by using relatively low energies offering low penetration, large critical angle and better resolution. High-quality data can be collected with an extension of the dynamical measurement range of 3 to 4 order of magnitude with respect to conventional-laboratory instruments, making it possible the study of complex layer sequences and evaluate details in their structure.

6.3. Residual stress and texture analysis

The non-destructive character of XRD can be limited or negatively counterbalanced by the shallow beam penetration and sample fluorescence, which can result in a low statistical quality of the data. These limitations frequently discourage the use of this valuable analytical tool. The new station easy tunability, besides offering a much higher brilliance than in conventional sealed tube lab instruments, will permit to investigate different depth inside materials, up to a few millimetres for some cases of concrete technological interest (*e.g.*, aluminium alloys), and in the region of the tens of microns for most materials, which is perfect for thin film studies. Problems in the reliability of the results are easily solved by the high beam intensity and the parallel-beam condition achievable. The use of a specifically designed stress/texture diffractometer allows the measurement of residual strain and texture in materials and real components of various shape and sizes within a sample holding volume ($80\times80\times80$ mm^3), which is often a problem at other facilities. The stress/texture goniometer can handle samples up to 5 kg and has seven motors to allow any rotation. This instrument is ideally suited for thin film studies as well for the stress and texture characterisation of bulk mechanical components and fibres (inorganic and organic).

6.4. Phase identification, profile analysis, kinetic and structural studies

Given the high signal to noise ratio, the narrow instrumental resolution and the possibility to use a focalized beam, phase identification can be extended to cases of practical interest which cannot be treated by conventional-laboratory instruments. This involves low amounts of phases in mixtures and, interestingly, samples available in very low amounts; qualitative and quantitative phase analysis can thus be performed under very special conditions. Like phase identification, line profile analysis (LPA) is also a historical topic of XRD: from the XRD profiles it is possible to study the distribution of size and shape of the coherently

diffracting domains, together with the effect of lattice defects, which introduce a local lattice distortion, as well as paracrystallinity in polymers. The station can allow LPA on both powder-bulk polycrystalline and thin film samples with considerable advantages with respect to lab instruments; again these advantages are due to the high signal to noise ratio obtainable, narrow instrumental resolution function and monochromaticity. The limits of the LPA technique can be considerably extended, in such a way allowing the analysis of very small amount of materials and the extension of a typical powder technique (*e.g.* LPA of powder catalysts) to thin films and surfaces modified by thermal and mechanical treatments (epitaxial thin films, surface diffusion, hard coating deposition, polishing, grinding burns in mechanical components, *etc*.).

The high temperature facility will also permit the study of transformation kinetics and phase transitions; in addition to the high temperature induction furnace (T_{max} ca 2000 K), a cryostat could be also easily installed. These non-ambient conditions can also be realised in a vacuum (up to the limit of high vacuum) or in conditioned atmospheres, in order to permit the study of phase transformation and equilibrium diagrams, as well as oxidation and corrosion kinetics in general. The latter features have considerable relevance for industrial users concerning surface kinetics.

7. Conclusions

The intrinsic flexibility of the proposed geometry of both beamline and experimental station makes MCX an ideally suitable tool for diffraction measurements of polycrystalline materials in general (powder and bulk materials). The main target of the new station is research and development in thin film and coating technology. The key features, the design and the target of the station is toward high intensity at relative low energy. In this way it will be possible to benefit from resonant scattering conditions for studies of materials containing elements of great technological relevance like Ca, Sc, Ti, V and Cr, whose absorption edges (K) are well within the range of optimal performance of the station. MCX is expected to complete the commissioning in 2007.

Acknowledgments:

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