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The Dutch–Belgian beamline at the ESRF

M. Borsboom,[a] W. Bras,[b,c] I. Cerjak,[a] D. Detollenaere,[b] D. Glastra van Loon,[a] P. Goedtkindt,[b] M. Konijnenburg,[a] P. Lassing,[c] Y. K. Levine,[d] B. Munneke,[e] M. Oversluizen,[b,c] R. van Tol[b,c] and E. Vlieg[a]*

[a]FOM-Institute for Atomic and Molecular Physics, Kruislaan 407, 1098 SJ Amsterdam, The Netherlands, [b]DUBBLE-CRG, ESRF, BP 220, F-38043 Grenoble, France, [c]NWO, The Hague, The Netherlands, [d]Debye Institute, University of Utrecht, PO Box 80000, 3508 TA Utrecht, The Netherlands, and [e]NIKHEF, Kruislaan 409, 1098 SJ Amsterdam, The Netherlands. E-mail: dubble@esrf.fr

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A brief description is given of the design principles and layout of the Dutch–Belgian beamline at the ESRF. This beamline optimizes the use of the available bending-magnet radiation fan by splitting the beam into two branches, each accommodating two experimental techniques.

Keywords: beamline optics; monochromators; mirrors; X-ray detectors.

1. Introduction

The Dutch–Belgian beamline (DUBBLE) is a collaborative research group (CRG) beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble. DUBBLE has been designed to provide bending-magnet X-radiation for a broad scientific community and accommodates four experimental techniques: small- and wide-angle X-ray scattering (SAXS/WAXS), interface diffraction (ID), X-ray absorption spectroscopy (XAS) and high-resolution powder diffraction (HRPD). A schematic diagram of the beamline is shown in Fig. 1.

In order to provide sufficient beam time and to optimize the optics design, the available radiation fan of 9 mrad is split into two branches of 2 mrad each, with a 5 mrad gap in between. The ESRF bending magnets start with 6 mrad having a magnetic field of 0.4 T (soft edge, with a critical energy of 9.8 keV) and continue with 0.8 T (hard edge, critical energy 19.6 keV). We thus have one soft (*S*) and one hard (*H*) branch that each deliver the beam to a separate experiments hutch. XAS and HRPD are located in the *S*-hutch, while SAXS/WAXS and ID occupy the *H*-hutch.

2. Optics

2.1. *General design*

The optics are located in the optics hutch (see Fig. 1). For the *H*-branch the main optical components are a sagitally focusing double-crystal monochromator, followed by a meridionally focusing mirror. The *S*-branch has, in addition to these two components, a collimating mirror in front of the monochromator. This mirror enhances both the energy resolution for XAS and the angular resolution for HRPD. The focal length of all focusing elements is variable.

The energy spectrum of the ESRF is very hard and thus the rejection of higher-order harmonics is an important issue. In order to keep the beamline operation as simple as possible, we have chosen *not* to change the mirror angles, but rather to keep the beam path fixed and use mirrors with two coatings which can be translated sideways. The monochromators will use either Si(111) or Si(311) crystals, thus only third- or higher-order harmonics have to be considered. For the *H*-branch we have chosen Si and Pt as mirror coatings (*i.e.* uncoated for the Si side) and an angle of incidence of 2.8 mrad. This configuration leads to a calculated total flux and harmonic fraction as shown in Fig. 2. With the Pt coating the energy range from 11–30 keV is covered, while with Si the usable energy range is 5–11 keV. The plotted harmonic fraction, defined as the third-order intensity divided by the first-order intensity, assumes no detuning of the double-crystal monochromator. Since the *H*-branch will have a fixed energy during normal operation, detuning is straightforward and the harmonic fraction can be reduced by another factor of 10–100. The harmonic fraction will thus be well below 0.1% for the entire spectrum.

For the *S*-branch harmonics, suppression using detuning is a less reliable option because of the energy scanning during XAS. The two mirrors in this branch, however, more than compensate for this. The choice of mirror coatings and angle of incidence is largely dictated by the fact that the spectrum should be free of absorption edges in order not to compromise XAS. We again found Si and Pt to be a good combination, but now using an angle of incidence of 2.0 mrad. This small angle extends the useful energy range of the Si coating to beyond the Pt *L*-edges.

In addition to the mirrors and monochromators, the beamlines have the usual slits, filters and beam-position monitors (using scanning wires). The slits are equipped with phosphor screens that can be used as quick diagnostic tools.

The beamline has been designed such that both branches have as little interference as possible and can operate completely independently. Each branch has a shutter in the optics hutch, allowing entry into the corresponding experiments hutch without the need to stop the other beam (while keeping the optics

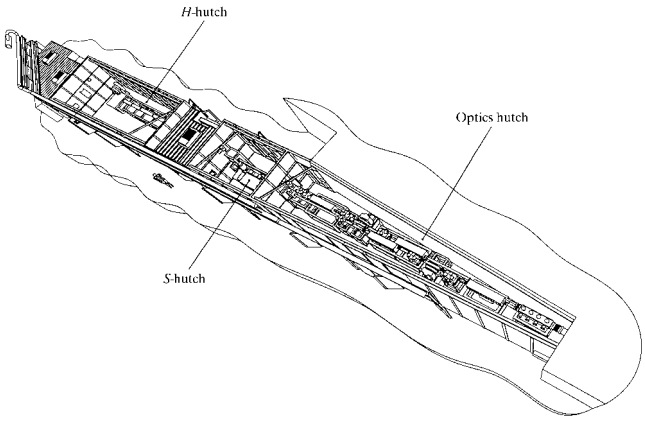

Figure 1
Overall layout of DUBBLE. The optics are located in the optics hutch, while the two branches each deliver the beam to a separate experiments hutch. XAS and HRPD are located in the *S*-hutch, while SAXS/WAXS and ID are installed in the *H*-hutch. A total of three control cabins are available for beamline control and sample preparation. A small distance away, additional laboratory and office space is available.

exposed to the beam). In order to minimize possible interference, the vacuum of the two branches is separated by two fixed Be windows immediately after the common splitter vessel.

2.2. *Monochromators*

The monochromators are based on a commercial platform (Fisons) that delivers an accurate and stable rotating disc inside a vacuum vessel. We use an in-house-designed crystal cage (see Fig. 3). The power load and densities from an ESRF bending magnet are such that water cooling is sufficient. The first crystal (provided by Crystal Scientific Limited) is placed in the centre of the monochromator. The water-cooling is of a jet-type design in which several slots are machined in the back of the crystal that each have their own jet. The ribs between the slots prevent bending of the crystal (85 × 65 mm) caused by the vacuum forces.

The sagittally focusing second crystal and its bender are based on an ESRF design. For the large crystals required at a bending-magnet source (in our case 90 × 70 mm), usually many ribs are machined on the back of the crystal in order to minimize anticlastic bending (Sparks *et al.*, 1982). Since the ribs lead to a deviation of the crystal surface away from the ideal focusing shape, one usually minimizes this effect by making the ribs as thin as possible. We have chosen to use a different route, namely to use only two ribs (Fig. 3). Extensive finite-element analysis has shown that this gives sufficient suppression of the anticlastic bending. Advantages of this 'few-rib' design compared with the many-rib design are (i) more crystal area with ideal shape, (ii) the possibility to slit down the beam to only the central ribless part, and (iii) easier manufacture. The monochromator will be operated in a constant-offset mode (20 mm beam offset).

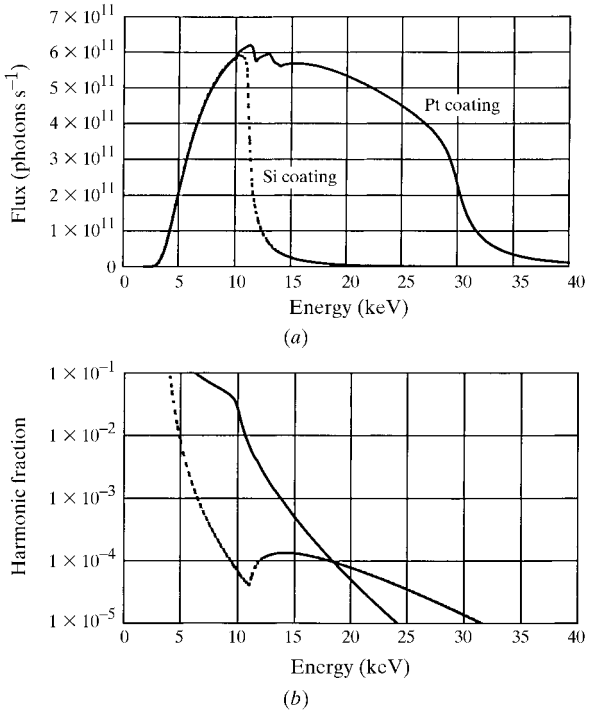

Figure 2
Calculated flux (*a*) and harmonic contents (*b*) of the *H*-branch assuming a 100 mA beam current and an Si(111) monochromator. The two choices for the mirror coating are shown.

2.3. *Mirrors*

The mirrors and benders were purchased (REOSC and IRELEC). The mirrors are made from Si boules and have a surface area of 1200 × 130 mm. The large width is required to have adjacent strips of bare Si and with Pt coating that each accept 2 mrad of radiation. The bender uses transducers to convert the displacements of stepper motors into a constant bending force (ESRF design).

The overall layout of the complete mirror set-up is shown in Fig. 4. The mirror and bender are kinematically mounted onto a heavy granite block that decouples the mirror from the vessel. Bellows plus linear translation stages allow a complete alignment of the mirror, including the 60 mm sideways translation to put either the Si or Pt coating into the beam. The mirror tilt angle is read using a commercial sensor (Applied Geometrics) (Knapp *et al.*, 1995).

3. Experimental techniques

3.1. *SAXS/WAXS*

Fig. 5 shows the SAXS/WAXS equipment that is located in the *H*-hutch. It is designed with a maximum of flexibility in mind and consists essentially of two frames. The first one contains the sample environment and the WAXS detector, while the SAXS detector is mounted on the second frame. The two frames are connected by a vacuum pipe of variable length. Changing of the SAXS camera length is straightforward, since both frames are accurately positioned using an alignment rail that covers the entire range (1–7 m) of the camera. The sample position remains fixed, but also the sample frame can be quickly exchanged. This is useful when complicated sample environments are used.

The mounting of the SAXS and WAXS detectors is such that they can be used simultaneously and that they can have an

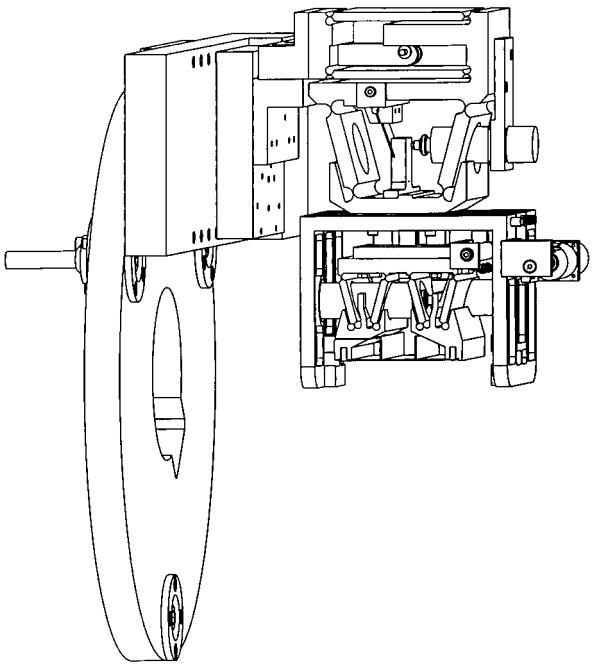

Figure 3
Design of the second crystal bender and manipulator. The second Si(111) crystal (bottom) has two ribs in order to suppress the anticlastic bending.

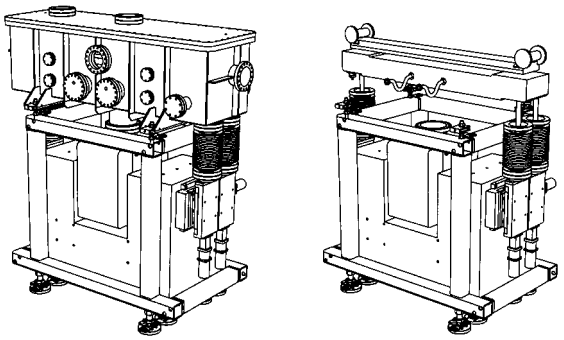

Figure 4
Overall layout of the mirror vessel. The actual mirror with bender is kinematically mounted on a solid granite block and is thus vibrationally isolated from the vacuum vessel.

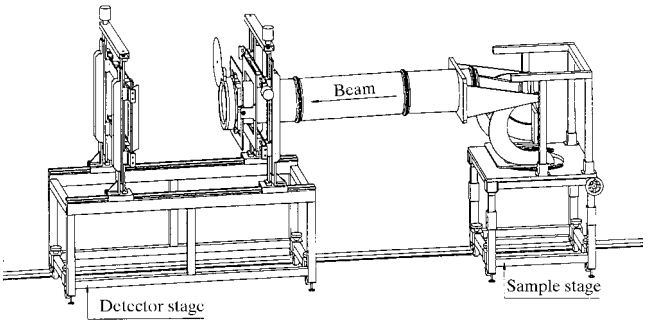

Figure 5
The SAXS/WAXS equipment. The WAXS detector can be mounted on the sample stage, while the SAXS detector is mounted on a separate frame that can be moved up to 7 m away from the sample.

overlapping angular range. On the sample stage additional equipment can be mounted for sample environment control.

To reduce the parasitic scatter which determines the minimum observable scattering angle (and thus the maximum distance), three slit sets are used with a total separation of 12 m. Due to the large difference in beam divergence and beam size between the horizontal and vertical directions, there is a large difference in the resolution along those directions. Using the full beam, the maximum observable distances are 100 and 1000 Å along the horizontal and vertical directions, respectively. With reduced flux, these values can be improved considerably.

As Fig. 2 shows, the SAXS/WAXS equipment can make use of a wide spectral range. This is useful when tuning the optimum penetration depth of a sample and its environment (which cannot always be made of the desired thickness) and for anomalous scattering.

For the SAXS detector, wire chambers will be used (Hall & Lewis, 1994), both of two-dimensional and quadrant type. Detectors are often the limiting element in a SAXS/WAXS experiment. For this reason we are developing a curved WAXS detector based on microstrip technology and with a 60° angular range (Zhukov *et al.*, 1997). This detector has a maximum count rate of ~100 MHz and a readout time of ~1 ms. In combination with the high available flux, time-resolved scattering experiments with high data quality down to the millisecond timescale are thus possible.

3.2. *ID*

The interface diffraction equipment is located in the same hutch as the SAXS/WAXS equipment. The diffractometer (Micro-Controle/Newport) is of the '2+3' type (Vlieg, 1998) which is suitable for a set-up combining both horizontal and vertical scattering geometries. For the horizontal scattering geometry, an extra tilt stage (for the angle of incidence) plus rotation axis are used.

A novelty is the goniometer head used to align the samples with respect to the diffractometer axes. Rather than a conventional design using translation stages and cradles, a compact hexapod (Physik Instrumente) will be used for this. The hexapod has all the necessary degrees of freedom and is sufficiently light to allow its use in both the vertical and horizontal geometries. Since the rotation centre can be arbitrarily defined, the hexapod facilitates the mounting of sample chambers with different dimensions.

3.3. *XAS*

XAS equipment is located in the *S*-hutch. With XAS one ideally wants a constant flux over an entire energy scan. This requires dynamical focusing of the second monochromator crystal. Tests need to be performed in order to find out how reliable and fast this can be performed with our equipment.

The XAS set-up will have facilities for both transmission and fluorescence yield measurements. For XANES the Si(111) monochromator crystals can be replaced by Si(311) ones. The high flux and low beam divergence make the beamline suitable for REFLEXAFS.

3.4. *HRPD*

HRPD shares the *S*-hutch with XAS. Since the optics are especially designed for a high-energy resolution and for energy scanning, this makes the equipment suitable for anomalous scattering and diffraction anomalous fine structure (DAFS). The variable focal lengths of the optics makes it possible to trade 2θ resolution for flux in the vertical direction and to tune the beam to the sample dimensions.

For high-resolution work, analyser crystals will be used, while for time-resolved measurements the curved WAXS detector is available.

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