Micro-XRD/XFM
Design Update – X-ray Source and Beamline Optics

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Summary

Extensive analysis and discussion of options regarding possible X-ray sources for the micro-XRD/XFM beamline have been undertaken. The preference, to date, is for a 5 T superbend as this will provide:

- Higher flux as compared to an existing 1.3 T bend magnet, which will result in greater effective depth penetration for 3D micro-XRD;
- Higher upper energy range (*e.g.* ×28 flux at 40 keV as compared to a 1.3 T bend magnet) which will enable access to a greater wealth of diffraction data, enable greater depth penetration for 3D micro-XRD and provide access to a greater range of K-edge energies to be accessed;
- Opportunities for XFM requiring a higher energy range than that provided by the Microspectroscopy beamline (usable energy range 4 to 25 keV[1]);
- Manageable heat loading on optical elements (as compared to a wiggler);
- Would not require a straight section at the Australian Synchrotron;

Moreover, the ALS superbend upgrade has been shown to have minimal impact on other beamlines and to improve orbit stability.[2]

A new simplified beamline design is also proposed consisting of:

- A single axial aperture for both 'monochromatic' and 'white' beams;
- A Pt coated toroid at 11 m from source which will deflect the beam by 4 mrad;
- Small gap monochromator (≈6 mm) with Si(111)/Si(311) and depth graded multilayers before and after the Si crystals with the second crystals tracking the beam in each case to maintain a fixed beam height;
- The toroid will provide 1:1 focus onto intermediate slits 22 m from source;
- A pair of Pt KB mirrors (elliptical cylinders) which will deflect the beam by 4 mrad (two other strips of Pd and Si will also be available to alleviate transmittance of harmonics). The first KB mirror is 7 m from the intermediate slits, deflects vertically and is 200 mm long. The second KB mirror deflects the beam horizontally and is 150 mm long. The mirror centres are 180 mm apart and their focus is 150 mm from the centre of the second KB.
- Sample stage and KB mirror pair to be mounted on a height adjustable stage, *e.g.* miCos GmbH LS-270[3] (capacity up to 150 kg and unidirectional reproducibility of 0.05 μm). This will enable the sample to be moved up and down so that white and monochromatic light are coincident and co-linear on the sample.

This design, coupled to a superbend, will lead to an improvement in performance (> 2 for the same spot size at 15 keV) and significantly simplified optics as compared to the design previously proposed. A spot size of 0.5 × 0.5 μm will be readily achievable.

1 From discussion with David Paterson, Microspectroscopy beamline.
2 Successful Completion of the ALS Superbend Project, Proceedings of EPAC 2002, Paris, France, 215-217.
3 http://www.micos.ws/web2/en/1,4,040,ls270.html.

1. Objectives

There is a clear requirement for a microdiffraction beamline (micro-XRD/XFM) at the Australian Synchrotron. To this end a Preliminary Technical Design Report[4] was prepared in 2005 which demonstrated the technical feasibility of a proposed optics design. However, with the advent of the Phase II Science Case for the Australian Synchrotron it has been timely to revisit the design of the proposed micro-XRD/XFM. In the intervening period various developments in X-ray optics and sample stages occurred and it is desirable that the beamline design takes full advantage of these. The objectives of the discussions and calculations reported herein therefore were:

1) To define the advantages/disadvantages for the micro-XRD/XFM of various X-ray sources, namely bend and superbend magnets, and wiggler and undulator insertion devices;
2) To revisit the design of the optics of the micro-XRD/XFM as described in the Preliminary Technical Design Report for the Australian Synchrotron[4] and as implemented (with minor variation) at the VESPERS beamline at the Canadian Light Source[5] with a view to simplifying the optics without comprising the central capabilities of the beamline.

This document has been prepared for the non-expert in synchrotron optics and hence many of the issues raised and comments made may appear to be obvious to those versed in beamline design. However, it is important for the potential users of the micro-XRD/XFM to understand the range of issues to be contended with when defining the beamline design and specifications.

2. Examination of the X-ray Source

During August/September 2009 there has been an on-going discussion regarding the nature of the most appropriate X-ray source for the micro-XRD/XFM beamline proposed for the Australian Synchrotron with the aim of determining the best balance between practical implementation and maximum scientific flexibility. Very useful contributions have been made to these discussions by David Cookson (Head of Scientific Operations, AS), David Patterson (Principal Scientist Microspectroscopy, AS) and Chris Glover (Senior Scientist-SXAS, AS). Further analyse and discussions have subsequently been held in conjunction with Ruben Reininger (Scientific Answers and Solutions).

The central objective of this beamline is to carry out micro-XRD using both pink and monochromatic light, while also providing XRF and XAS. It should be remembered that 3D micro-XRD cannot viably be carried out with white light and hence sample penetration for white light applications is not a central priority. While usable flux obviously plays a central role in beamline viability this is generally not an issue for pink light applications, for instance

4 Preliminary Technical Design Report, Beamline 11, The Microdiffraction Fluorescence Probe, Australian Synchrotron; Ruben Reininger and Andrea Gerson, June 2005.

5 Preliminary Design Report for the VESPERS, Beamline at the Canadian Light Source Inc., CLS Design Note 28.2.69.1- Rev B, Date: 2005-09-22

a 10% band pass will provide, by definition, a factor of ≈×250 flux (reflectivity of 50% from one bounce) over the light transmitted by Si(111). Our requirements are therefore to maintain a reasonable X-ray flux while maximising the accessible energy range.

Three near analogues of the proposed beamlines, which are relatively well known to the authors, are 34-ID-E which is on an undulator beamline at the APS, end-station 12.3.2 at the ALS which is on a superbend and VESPERS at the CLS which is on a bend magnet. We have attempted to list below the advantages and disadvantages of each possible X-ray source; undulator, wiggler, superbend and bend.

Undulator

Advantages

- High flux will result in greater effective depth penetration for 3D micro-XRD;
- High flux will enable greater XAS and XRF sensitivity to low concentration elements;

Disadvantage

- Limited upper X-ray energy range. An effective energy range of 7 to 25 keV is available on 34-ID-E at the APS only by tapering the beamline undulator;
- Very high harmonics of an in-vacuum insertion device will be needed at the Australian Synchrotron to achieve energies above 10 keV;
- Would require allocation of a straight section at the Australian Synchrotron.

Wiggler

Advantages

- High flux will result in greater effective depth penetration for 3D micro-XRD;
- Higher energy upper range (dependent on magnetic field strength) of usable X-ray flux for a wiggler source as compared to a bend magnet (estimated as 31 keV[6]), will enable access to a greater wealth of diffraction data and enable greater depth penetration for 3D micro-XRD;
- Higher flux (proportional to the number of poles) will enable greater XAS and XRF sensitivity to low concentration elements;
- Higher energies would enable a greater range of K-edge energies to be accessed;
- The provision of a wiggler would enable the micro-XRD/XFM to cater to those users requiring a higher energy range than that provided by the XFM beamline (usable energy range 4 to 25 keV[1]).

Disadvantages

- High flux at high energies (*e.g.* a multipole 5 T wiggler) leads to very high heat loading on the monochromator elements (reducing the wiggler magnetic field strength reduces the accessible energy);
- The resulting requirement for cryogenic cooling leading to vibrational instability;
- Strong likelihood of detector saturation when using pink/white light thus limiting the usefulness of the extra flux;
- Increased OHS&W requirements to cope with white light radiation in the two hutches (optics hutch and experimental station) and bremsstrahlung radiation particularly in the first optics hutch;

6 From discussion with Kia Wallwork, Powder diffraction beamline.

- Long source length may lead to defocus of incident X-ray spot size and/or loss of flux. However, due to the small angular aperture acceptance of X-rays into the beamline (0.1 mrad) this has been found to have only a very marginal effect.
- Would require allocation of a straight section at the Australian Synchrotron.

Superbend

Advantages

- High flux will result in greater effective depth penetration for 3D micro-XRD;
- Higher energy upper range (dependent on magnet field strength) of usable X-ray flux for a superbend as compared to a bend magnet (estimated as 31 keV[6]), will enable access to a greater wealth of diffraction data and enable greater depth penetration for 3D micro-XRD;
- Higher energies would enable a greater range of K-edge energies to be accessed;
- The provision of a superbend would enable the micro-XRD/XFM to cater to those users requiring a higher energy range than that provided by the XFM beamline (usable energy range 4 to 25 keV[1]).
- Manageable heat loading on optical elements;
- Would require bend magnet section of the Australian Synchrotron.
- The ALS superbend upgrade has been shown to have minimal impact on other beamlines and to improve orbit stability.[2]

Bend

Advantages

- Readily manageable heat loading;
- Would require bend magnet section of the Australian Synchrotron.

Disadvantages

- Reduced scientific flexibility as compare to, particularly, a superbend or wiggler.

For wiggler, superbend and bend magnet sources only a small component of the horizontal fan can be collected (in the current design this is approximately 0.04 mrad) due to the small angle of incidence on the mirrors required to have good reflectivity and the need for high demagnification. Also if energies over 20 keV are to be measured for the purposes of XFM or XAS a Ge solid state detector will be required.

From the list given above it is clear that an undulator source would not provide significant scientific advantage to micro-XRD as would either a wiggler or superbend. However, a wiggler source also provides significant disadvantages if combining a significant number of poles and a high magnetic field strength, as would be required for high flux at high energies. To quantify the specific differences in X-ray sources we have carried out a number of comparative calculations on various bend and superbend X-ray sources.

Fig. 1 shows a comparison of the estimated flux (per horizontal mrad) of three possible X-ray sources consisting of an existing end magnet at the Australian Synchrotron (1.3 T), a 1.9 Tesla bend magnet and a 5 T superbend (as implemented at the ALS). The equivalent traces for wigglers of the same magnetic field can be approximated by multiplying the traces in Fig. 1 by the proposed number of poles. Thus flux may be increased by the application of a wiggler but the accessible energy range is not changed in comparison to the same magnetic field strength bend magnet.

At 30 keV the flux from an existing bend magnet is in the order of 9×10^{11} photons/s/0.1% BW/mrad. The 1.9 Tesla bend magnet would clearly extend this energy range so that for the same flux an energy of over 40 keV is achievable. In contrast for the 5 T superbend this flux is still achievable at energies higher than 100 keV. However, this analysis does not take into account the effect of downstream optics on the accessible energy range.

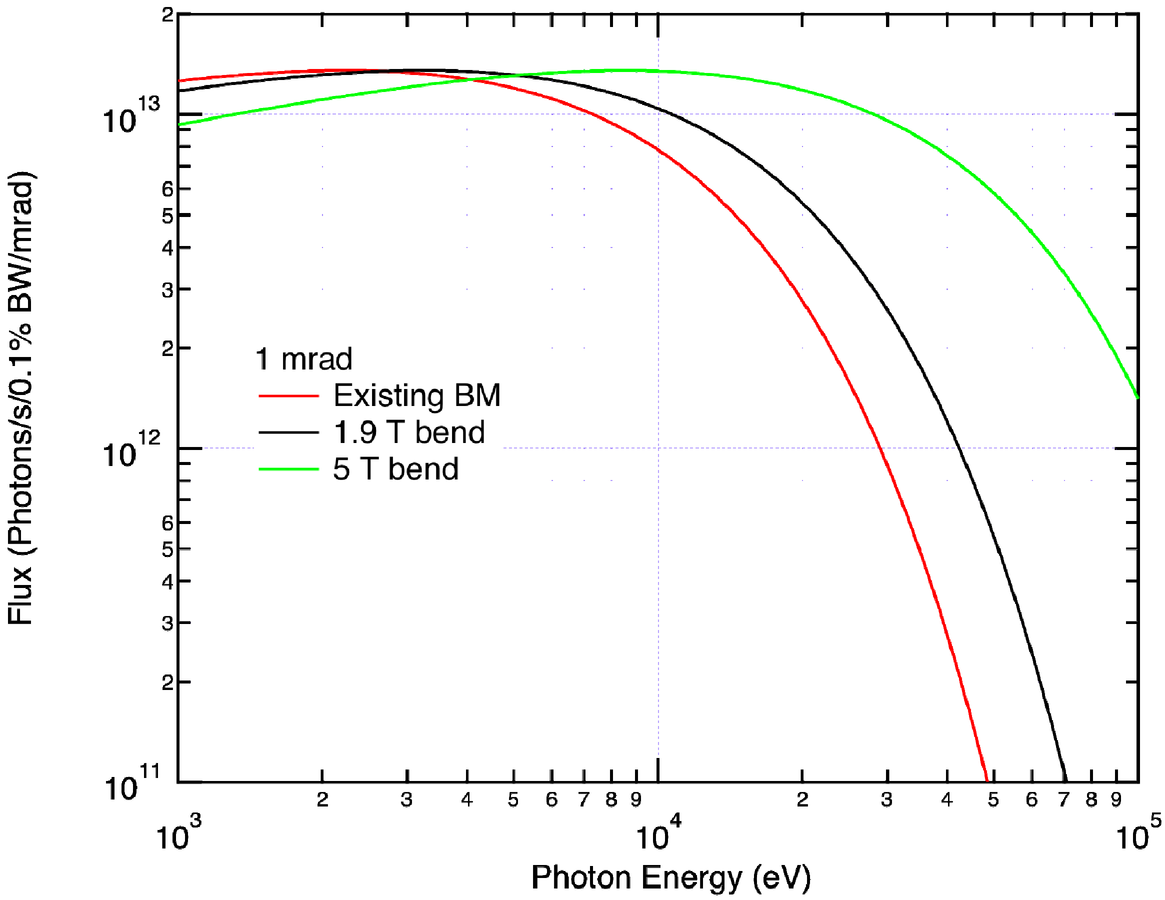

Fig 1 A comparison of the estimated flux from an existing bend magnet at the Australian Synchrotron, a 1.9 Tesla bend magnet and a 5 Tesla superbend. The stored current is 0.2 A.

There are only a limited number of different mirror coatings available for high x-ray energies and Pt coatings enable the broadest range of X-ray energies to be accessible. Higher energies are accessible with decreased glancing angle onto X-ray optics, however with decreasing angle of incidence either the mirror(s) must either be lengthened (with commensurate increase of cost and demagnification loss) or the transmitted flux reduced. Hence the accessible energy range is not only a function of the X-ray source but also the acceptable loss of flux as the X-ray beam progresses down the beamline. In Fig. 2, we show the reflectivity of a Pt coated surface at various incident angles assuming 0.2 nm RMS roughness. The incident angle of 2.6 mrad was adopted in the preceding micro-XRD/XFM beamline to enable energies of up to 30keV to be accessed. It is clear that energies exceeding 50 keV are accessible at a glancing angle of 1.5 mrad. However the importance of this needs to be offset by the resulting loss of flux which in comparison with 2.6 mrad would be a factor of approximately $(1.5/2.6)^2$ or a loss of flux of 67%.

The design of the beamline predicates an upstream focus onto a set of slits which act as a secondary source. Regardless of the nature of the optics of the upstream focus the upper energy range is essentially a fixed value and must be defined at the time of design of the beamline. The reason for this is simply that a change of incident angle for a specific optic element radius of curvature will result in a different X-ray beam path so that X-ray beam will not track correctly through subsequent optics. A difference of 0.6 mrad will result in a shift of 12 mm in 20 m of beamline. Furthermore, the foci distance depends on the angle of incidence making a change in angle impractical.

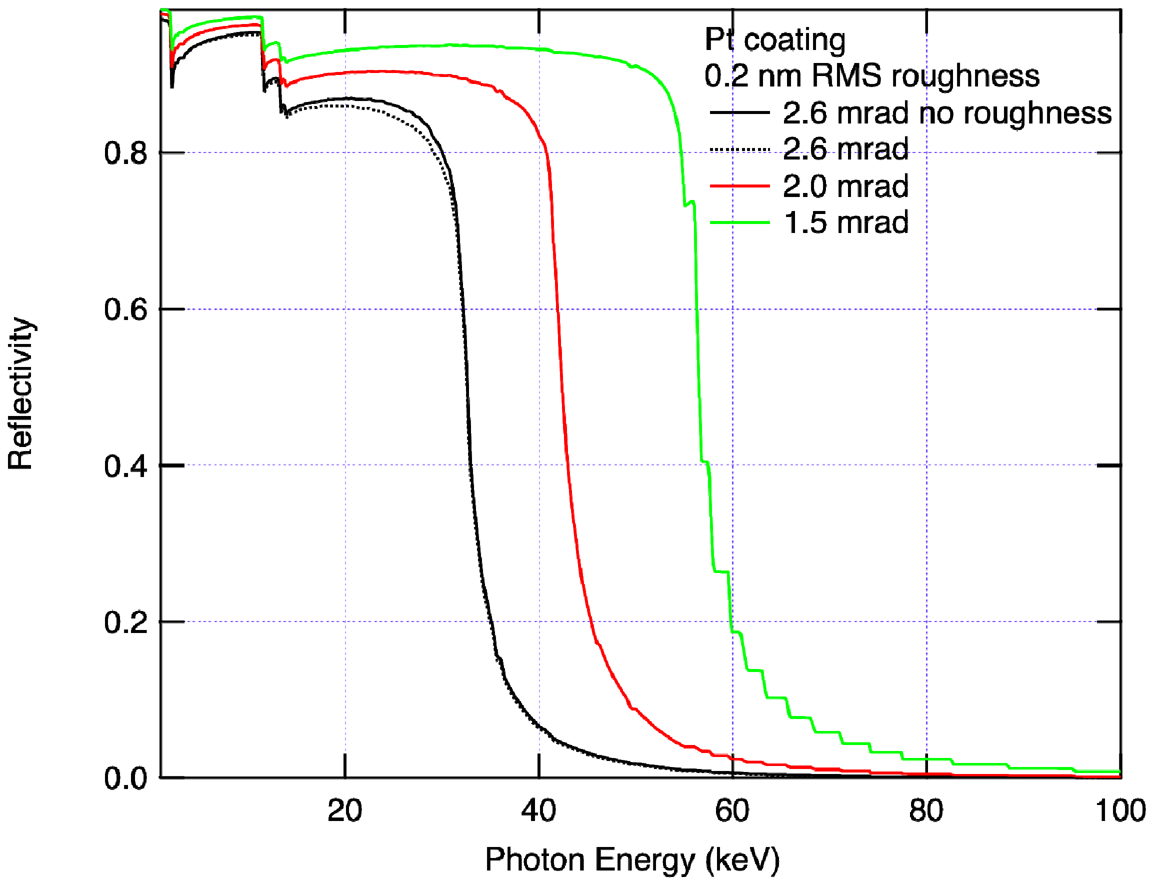

Fig. 2 Reflectivity of Pt coated mirrors at various angles of X-ray incidence.

Fig 3 shows the convolution of the results of Fig. 1 and Fig. 2 assuming the beamline has three mirrors (1 toroidal and 1 KB mirror pair) and taking into account the mirror acceptances. The price paid for a higher flux at high energies is a decreased flux at low energies. This begins to become significant for instance for the 5 Tesla superbend at an incident *X-ray angle of 1.5 mrad*. It is clear that the superbend (5 T) at an incident angle of 2.0 mrad provides a superior performance to 40 keV with an acceptable reduction in flux at < 6 keV. At 40 keV the flux achieved by a superbend is approximately 28 times that from an existing Australian Synchrotron bend magnet (2.5×10^{12} *cf* 8.6×10^{10}).

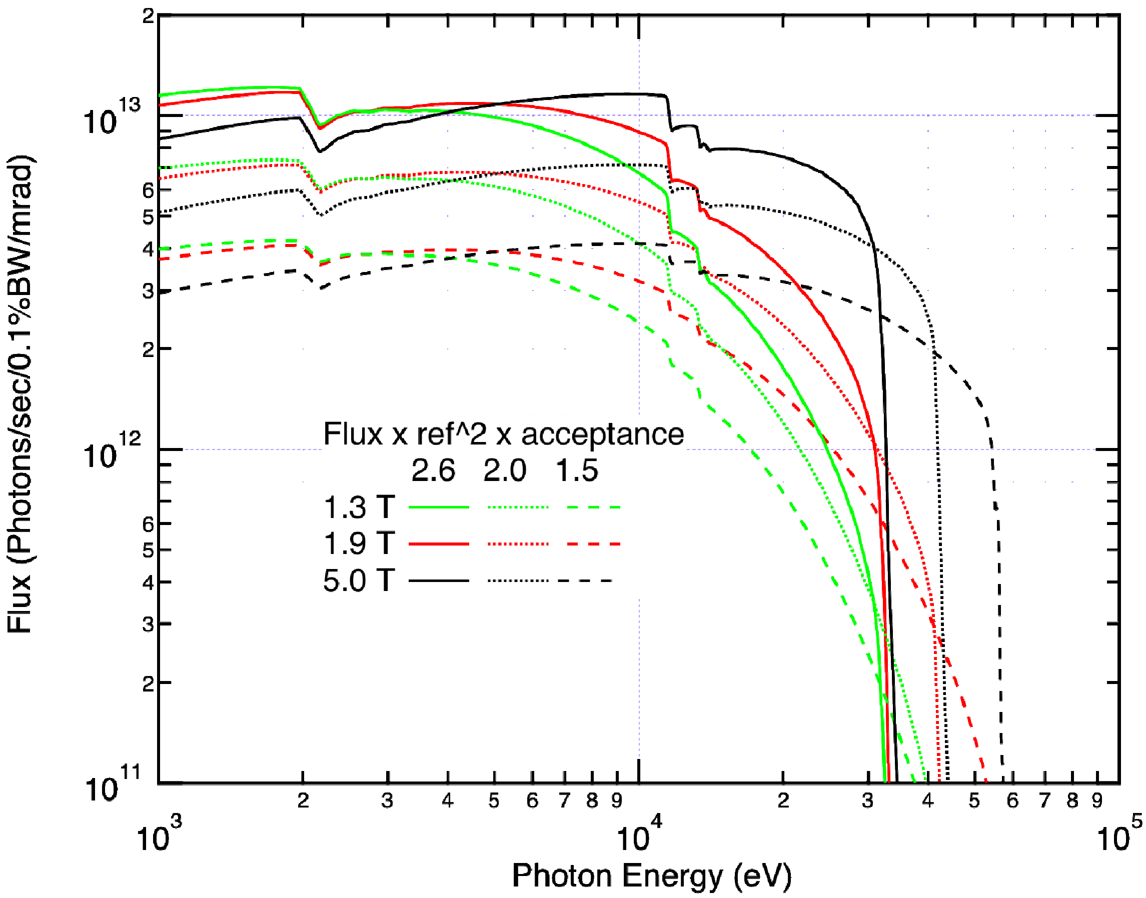

Fig. 3 Convolution of X-ray source and optics incident X-ray angle for 3 consecutive Pt coated optics devices (1 mrad acceptance, same length mirrors).

3. Re-examination of the Optics Design of the micro-XRD/XFM

The design of the beamline requires that the various band pass options adopted (white, depth graded multilayers and monochromatic) must be easily interchangeable and that on changing between these options the same sample volume must be accessed. For clarity the description of the previous design is provided in italics below.

The original proposal was for the micro-XRD/XFM beamline to be closely similar to the design adopted by the CLS VESPERS beamline. Beam co-linearity was achieved in the previous design by having two front end apertures above and below the axial position. The upper aperture transmits the beam that will go through the monochromator ('monochromatic') and the lower aperture transmits the beams bypassing the monochromator ('white'). Downstream of the apertures there is a horizontally deflecting mirror (at 13.5 m) which has on it three strips of Pt, Pd and Si. The reflectivity characteristics of these strips will ensure that harmonics are not transmitted by the monochromator. Following the horizontally focusing mirror there are two vertically focusing mirrors, ***A*** *and* ***B****.* ***A*** *is designed to focus the white beam and* ***B*** *focuses the monochromatic beam both at the intermediates slit.* ***A*** *and* ***B*** *deflect the beams upwards by 5.2 and 5.1 mrad, respectively.*

*The distance between the poles of **A** and **B** is 480.39 mm and the pole of **B** is 1.48 mm higher than that of **A**. After these two mirrors the beams collected above and below the ring plane are parallel, make an angle of 5.15 mrad with the horizontal plane, and are separated along the vertical direction by 1 mm.*

This design is rather complicated and while achievable it would benefit from simplification if the same flexibility and capabilities can be maintained. Moreover the selection of X-ray beams from above and below the equatorial axis has two negative side effects: partial polarisation which may affect XAS measurements and loss of flux. We propose to replace the three upstream focussing mirrors (1 horizontal and 2 vertical) with a toroidal mirror at 11 m. The toroid deflects the beam horizontally by 4 mrad (instead of the 5.2 mrad we had before). The toroidal makes a 1:1 image of the source. A double slit (horizontal and vertical) is located at the toroidal focus. The 1:1 focus will minimise the effect of any mirror aberrations and the reduction of 1 mirror will also serve to increase the flux (*i.e.* from one horizontal and one vertical focus mirror for each beam to the toroidal mirror).

The double monochromator is placed 1.5 m upstream of the intermediate slits. It will produce a vertical shift upwards of 1 mm to the monochromatic beam going through it, making it collinear with the white beam. The fact that the second crystal in the current design does not track results in a shift in the vertical of the monochromated beam depending on the energy chosen. For the VESPERS beamline over the entire accessible angular range (from 0.3 to 17°) this results in a vertical motion of 44 µm when using Si(111). It is proposed that this can be corrected for by altering the tilt of the first crystal.

Rather than having two beams we now propose to have only one beam which may or may not pass through the monochromator as desired. The result will be two beam paths offset in the vertical but parallel to each other. By employing a monochromator where the second crystals track it will be possible to eradicate beam displacement as a function of energy. The design is still likely to utilise a relatively small vertical offset of perhaps 6 mm. This enables the depth graded multilayers to be placed before and after the Si(111) crystals (and Si(311)) crystals for higher energy monochromated light) but still remain within the same chamber. For instance for an effective depth graded multilayer *d*-spacing of 2.6 nm (used to provide a band pass of 10%) the longitudinal offset at 6 keV is 75 mm while at 40 keV this offset is increased to 503 mm assuming a constant 6 mm vertical offset between the two depth graded multilayers.

In order to ensure that the 'monochromated' and 'white' beams are readily coincident and collinear on the sample, *i.e.* are sampling the same volume, the KB mirror pair and sample stage will be placed on a vertically adjustable mounting. The miCos GmbH LS-270[7] stage, for instance, would be well suited to this task with a load capacity up to 150 kg and unidirectional reproducibility of 0.05 µm. This will enable ready interchange between 'monochromatic' and 'white' light as well as any secondary corrections required for shifts in vertical offset on changes in 'monochromatic' crystal selection.

7 http://www.micos.ws/web2/en/1,4,040,ls270.html

Preliminary X-ray tracing calculations have been undertaken to explore this proposed design. The source RMS sizes (87 × 60 µm, H×V) are taken from the Australian Synchrotron website.[8] The divergences used in the ray tracings are 80 µrad horizontal and 70 µrad vertical (as for [4]). Slope errors of 2 µrad were assumed for the toroid and 0.8 µrad for the KB mirror pair. The values determined by the acceptance of the KB pair are 42 × 57 µrad (H × V). The only difference between the existing KB pair and the one modelled in the Preliminary Technical Design Report[4] is the angle of incidence. Both KB mirrors (Pt coated) are elliptical cylinders and deflect the beam by 4 mrad. The first KB mirror is 7 m from the intermediate slits, deflects vertically and is 200 mm long. The second KB mirror deflects the beam horizontally and is 150 mm long. The mirror centers are 180 mm apart and their focus is 150 mm from the center of the second KB. One of these KB mirrors will also have Pd and Si strips deposited on it for the reduction of harmonic transmittance (and requisite motors to access these strips).

Fig. 4 shows the spot size at the intermediate slits when they are wide open and the resulting incident sample spot size. The following pairs of figures show the spot size at the intermediate slit and its image after the KB pair. Table 1 provides the slit widths, resulting incident beam size and the fraction of X-rays remaining at the sample as between the intermediate slits and the sample. The simulated performance of the new design compares very favourably of the previous design. For instance at 15 keV the previous design was estimated to give rise to a performance of 5.8×10^{8} photon/s for a 0.01% BW (Si(111)) for a 2 × 2 µm spot size. The estimated flux for the 1.5 × 3.1 µm spot size (Table 1) at 15 keV is a factor of ×3 improvement on this.

Table 1 Spot size (H × V, µm), estimated flux (photon/s) at sample at 15 keV and 40 keV (0.01% BW, *e.g.* Si(111), 1% BW and 10% BW, 200 mA, 2 nm RMS roughness) and required intermediate slit widths (H × V, µm). For 1% BW and 10% BW multiply by 56 and 250 respectively (this takes into account the reflectivity from two multilayers).

Final spot size (FWHM*)	**3.7 × 5.6**	**2.7 × 5.0**	**1.5 × 3.1**	**1.0 × 1.0**
0.01% BW @ 15 keV	5.5×10^{9}	4.3×10^{9}	1.8×10^{9}	3.0×10^{8}
1% BW @ 15 keV	3.1×10^{11}	2.4×10^{11}	1.0×10^{11}	1.7×10^{10}
10% BW @ 15 keV	1.4×10^{12}	1.1×10^{12}	4.5×10^{11}	7.4×10^{10}
0.01% BW @ 40 keV	4.1×10^{9}	3.3×10^{9}	1.4×10^{9}	2.2×10^{8}
1% BW BW @ 40 keV	2.3×10^{11}	1.8×10^{11}	7.7×10^{10}	1.2×10^{10}
10% BW BW @ 40 keV	1.0×10^{12}	8.1×10^{11}	3.4×10^{11}	5.5×10^{10}
Intermediate slit size	No Slit	200 × 200	100 × 100	60 × 20

* 2.53×σ

8 http://www2.synchrotron.org.au/files/documents/Machine-fact-sheet_23Oct08_final.pdf

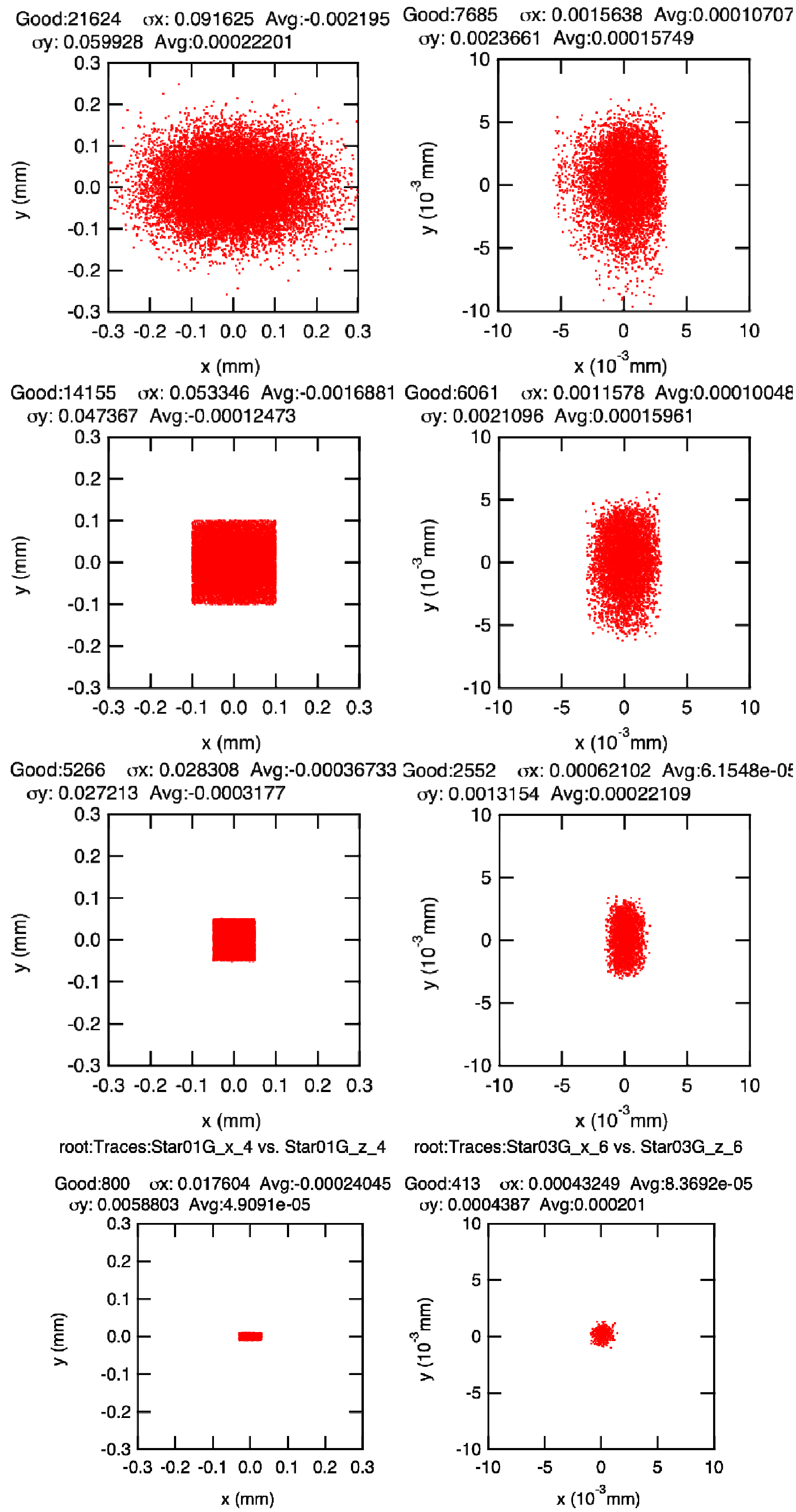

Fig. 4 The pairs of figures show the spot size at the intermediate slit and its image after the KB pair. In the captions above the figures are RMS values.