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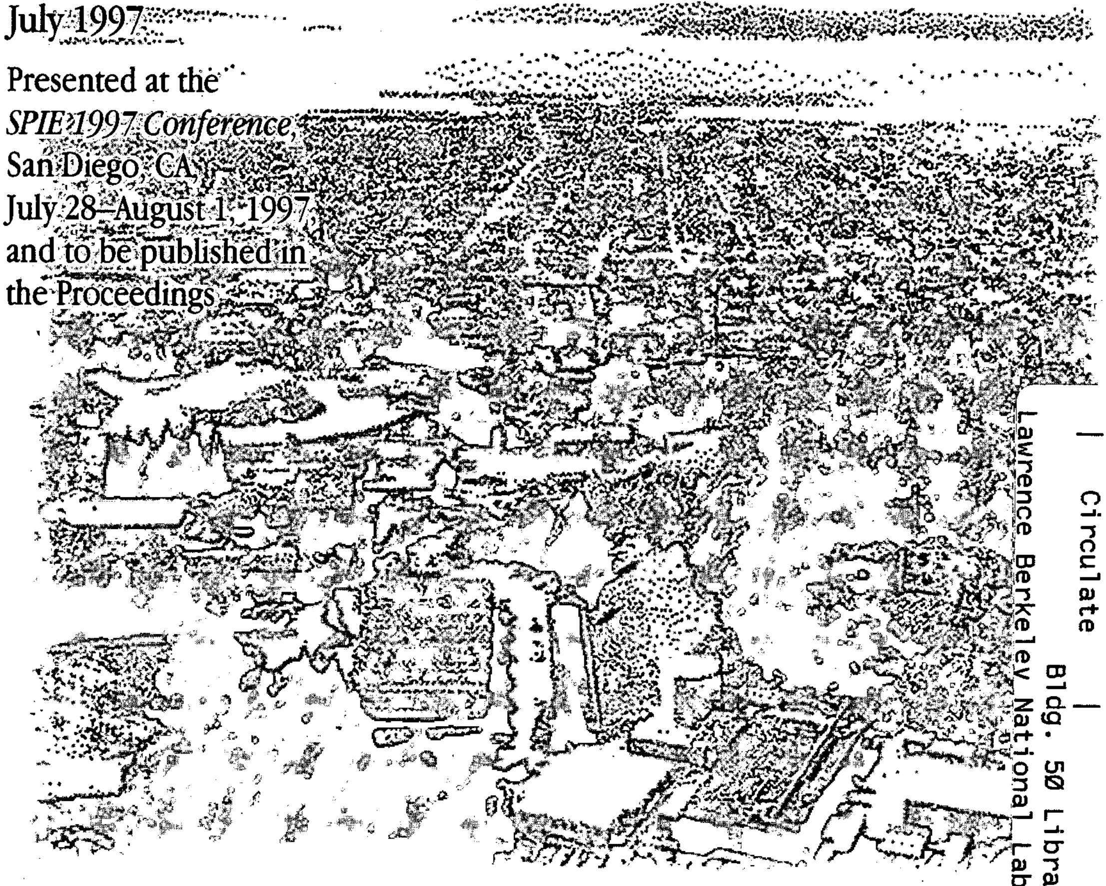

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ABSTRACT

Of the many methods used to focus x-rays, the use of mirrors with an elliptical curvature shows the most promise of providing a sub-micron white light focus. Our group has been developing the techniques of controlled bending of mirror substrates in order to produce the desired elliptical shape. We have been successful in producing surfaces with the required microradian slope error tolerances. Details of the bending techniques used, results from laboratory slope error measurements using a Long Trace Profiler (LTP) and data from the measurement of focus shape using knife edge and imaging methods using x-rays in the 5 - 12 KeV energy range are presented. The development of a white light focusing opens many possibilities in diffraction and spectroscopic studies.

Keywords: x-ray, micro-focusing, x-ray diffraction, bent mirrors, adaptive optics

1. INTRODUCTION

The advent of third generation synchrotron sources has required the continued development of x-ray focussing optics in order to utilize the available new source brightness. Many new types of microscopy and microanalysis can be performed if the brightness of the synchrotron source is utilized. All of these techniques require the focussing of x-rays by means of the various x-ray focussing optics such as capillary optics, [1] zone plate optics, [2] Kumakov whispering galleries, [3] Bragg Fresnel optics [4] and grazing incidence reflection mirrors.[5,6] All have their advantages and disadvantages dependant on the particular application. For the various microprobe techniques under development at the Advanced Light Source (micro-X-ray Photoelectron spectroscopy, x-ray micro-diffraction, micro-XAFS), we have opted to develop grazing incidence mirrors - in particular the Kirkpatrick-Baez (KB) focussing geometry [5] with a single metal film as the reflector. The desired elliptical shape is produced by the controlled bending of a flat mirror. Such optics offer broad bandpass and can be inexpensive. The origin of this technique and its development to date has been described earlier [7].

2. X-RAY MICRO-DIFFRACTION - X-RAY OPTICAL REQUIREMENTS

Our requirement is to perform x-ray micro-diffraction in the photon energy range of 5-12 keV. Sample sizes are intended to be on the micron scale. The customary way of performing x-ray diffraction is to fix the photon energy and map the Bragg reflection peaks by rotating the sample and detecting the diffracted x-rays by means of tracking the detector in a an angle doubling mode (the so called theta - 2 theta scan). Such a scheme is appropriate for macro sized samples (>100 μm), but for micron sized samples, the mechanical difficulty in rotating such a small sample while ensuring the focussed beam does not move from it, suggests that alternative ways of carrying out x-ray diffraction on micron sized samples should be considered. Our scheme is to scan the photon energy whilst keeping the sample and detector fixed. One problem that arises with all microscopy techniques is the ability to find the sample by means of some detectable signal - preferably the signal should be that of the technique employed - in our case, x-ray diffraction. Simply irradiating the micron sized sample with monochromatic x-rays is unlikely to generate any signal unless the photon energy, sample crystal planes and detector all happen to satisfy Bragg's Law. This is unlikely without an elaborate setup procedure. As an alternative, a simple setup procedure could be to irradiate the sample with a focussed beam of white radiation. This will result in at least some diffracted x-rays in the form of a Laue x-ray pattern. This will enable the determination of sample angle, detector position and the relevant photon energy range to be scanned. Thus the x-ray optics are required to be able to switch between white and monochromatic x-rays without changing the point on the sample being irradiated. Fig.1 shows the schematic layout of such an x-ray micro-diffraction beamline.

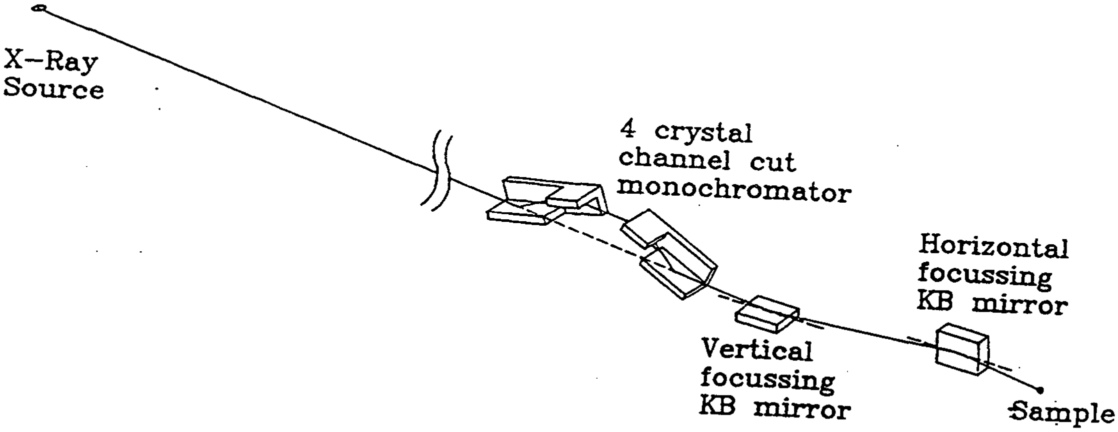

Fig.1. Schematic layout of the 4 crystal monochromator and KB mirrors.

The optical arrangement is to use the electron beam within the bending magnet of the storage ring as the x-ray source and image it with demagnification by means of the KB mirror pair onto the sample. Just prior to the KB mirror pair is a 4 crystal monochromator in the +--+ configuration,[8] which has the property of directing the monochromatic x-rays along the same axis as the incoming white light. The monochromator crystals to be used are 2 identical channel cut crystals mounted such that the rotation axis passes through the surface of crystals 2 and 3. In this manner a Bragg angular range of 7-70 degrees is allowed whilst the channel cut crystals themselves remain compact having a total length of only 59 mm. For Si(III) monochromator crystals the angular range corresponds to an energy range of 16222 - 2104 eV which is more than adequate to cover the energy range required for x-ray diffraction. The off axis rotation of the crystals allows for them to be rotated out of the way and thus allow white radiation to continue to the KB mirrors such that Laue x-ray patterns can be recorded. Diffracted x-rays can be detected by either a x-ray image plate that mounts between the sample and the last KB mirror, or a x-ray area CCD camera that mounts on a 2 circle goniometer and can be positioned over most of the surface of a sphere centered on the sample.

The mirror dimensions are defined by the source size, operating wavelength, demagnification and the beamline length. The source size at the ALS is around 30 µm x 300 µm (vertical x horizontal FWHM) and the experiment location (beamline 10.3.2) defines the source to mirror distance to be 30m. With a platinum coating, adequate reflectivity is achieved for up to 12 KeV photons with a mirror grazing angle of 5.8 mrad (0.33°). In the vertical plane a demagnification of 60 should achieve a 0.5 µm focus with a mirror to focus distance = 500 mm. The maximum convergence of the light onto the sample is limited by the critical angle of reflection of the highest energy required. If a higher convergence angle is used it means that at one end of the mirror light will be traveling almost parallel to the surface and at the other it will be exceeding the critical angle and will not be reflected. For 12 KeV photons the critical energy reflected from a platinum mirror is about 6 mrad. Given the demagnification the maximum acceptance could be up to 100 µrad which in turn defines the maximum mirror length of 516 mm. As this is a prototype device, we conservatively opted for a mirror length = 163 mm - about 1/3 of the maximum value. The vertical acceptance aperture is 0.93 mm and the radiation convergence angle onto the sample is 1.9 mrad.

Similar arguments apply to the horizontally focussing mirror. The horizontal beam size is approximately 300 µm and thus a demagnification of 300 will result in a 1 micron sized image with a mirror to focus distance of 100 mm, this being considered a reasonable working distance between this mirror and sample. With this demagnification the maximum acceptance could be 20 µrad requiring a mirror length of 103 mm. To allow for a conservative design and a reasonable sample clearance we opted for a mirror length of 40 mm. The horizontal acceptance aperture is 0.23 mm and the radiation convergence on the sample is 2.3 mrad.

Progress to date since the last work [7] has included the assembly of all the components and the testing of the KB mirror system. The 4 crystal monochromator is of a design similar to that of Tolentino et al [9] consisting of two rotational stages onto which two channel cut crystals mount. For Bragg angle changes the two stages rotate in opposite directions by means of a tape drive, which is driven by a linear slide. The monochromator has yet to be fully commissioned, but its mechanical performance indicates a measured tracking deviation of the two rotation axes of <0.001 degrees over several tens of degrees of crystal rotation. The remainder of the paper discusses the design and performance of the KB mirror performance.

3. KIRKPATRICK BAEZ FOCUSING MIRRORS

The ideal mirror shape required for imaging the synchrotron source in Fig. 1 is that of the surface of an off axis plane ellipse. Such a surface is able to perform true point to point imaging. The desired elliptical surface is produced by the controlled bending of a flat mirror produced by holding the ends of the mirror and applying opposite couples. If the mirror is treated as a parallel beam (uniform rectangular section) and the couples are equal and opposite, the mirror bends to the shape of a cylinder (a second order polynomial shape). If the two end couples are different the mirror adopts a third order polynomial surface that is a much closer approximation to the required ellipse. To eliminate the remaining figure errors, the mirror can be made not as a uniform rectangular cross section parallel beam, but with its width varying along its length. The moment of inertia of cross section of the mirror thus changes along its length and this in principle allows the mirror to be bent to the exact plane ellipse shape. In practice, the starting mirror is not exactly flat, and thickness variations along the mirror length and anticlastic effects will take their toll in how close an elliptical shape one can bend to. We have made the two KB mirrors each with the bending mechanism based on the leaf spring principle. Each mirror has a slightly different bending mechanism and will be described. The mirror flats used in the benders were flat to sub-microradian slope errors as measured with an LTP and had around 0.2 nm roughness as measured with a micromap optical profiler.

Fig.2 show the schematic diagram of the vertical focussing mirror. The system works in the following way. The ends of the 9.525 mm thick fused quartz mirror beam are glued [10] to invar blocks which are bolted to two thin leaf springs. It was found necessary to bond the blocks to the end of the mirror rather than the underside as the latter was found to cause mirror distortion due to glue shrinkage. Bonding to the ends of the mirror allows the compressive stress caused by glue shrinkage to be is a direction perpendicular to the mirror length and thus have a reduced effect on the mirror shape. The other ends of the springs are at one end attached to a slide way, and at the other to a rigid base. If the slide way is moved to the left, the springs are bent into equal and opposite S shapes, and exert equal and opposite couples. If the mirror has parallel sides and of constant width, it would bend into a

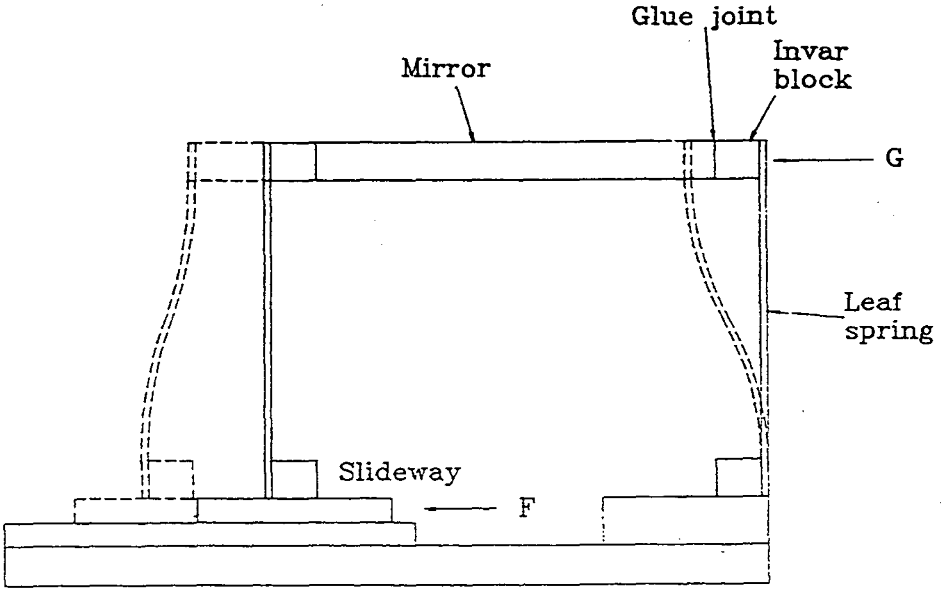

Fig.2. Vertical focussing 'S' spring mirror bender. Equal couples are applied by moving the slide way to the left (F). These couples are made unequal by applied the whole mirror to the left (G).

circle. If the beam is deflected by a pushing on the neutral axis of the mirror (G), then it can be seen that the extension of the spring at the right gets larger and at the left gets smaller. This increases the couple at the right, and decreases the couple at the left, and gives a linear change in moment with position along the beam. For a parallel sided mirror the shape would be that of third order polynomial, however the mirror is designed with a smooth width variation along its length[6]. The upstream width used was 40.6 mm, the mid point width is 42.5 mm and the downstream width is 40.8 mm. This mirror shape was achieved by CNC milling two pieces of steel to the required shape and then clamping the mirror between them in a temporary wax fixture. The plates were used as a template for careful hand grinding of the mirror. This gave a width accuracy with extreme errors of 25 μm, and a mean of < 10 μm. The computed slope error at these values (in this case) is negligible. The length of the leaf springs used was 40mm, and thus the slide extension is large, typically 10 mm. This means that the device is easily tunable, and is immune to temperature differences between the mirror and the bending mechanism.

For the horizontal focussing mirror the bender (shown schematically in Fig.3) operates in a similar way to the vertical focus mirror bender, but space constraints to allow for sample clearance dictate the use of compact picomotors[11] with consequent low driving force. To address this weak motor problem, the gross couples (C1+C2) are fixed (giving the central radius) and are set by extending the two springs and simply bolting the end blocks to the base. The difference in couples is produced by a static deflection of one end of the beam, achieved by pressing on the neutral axis. Fine tuning is provided by two picomotors acting on cantilever leaf springs. In this way the force required from the motor can be kept below 22 N which is within the specification of the picomotor. The fused quartz mirror is required to have a non uniform cross section along its 40mm length. Calculations indicated that the smoothly varying mirror width could be approximated by 3 equal length straight sections without significantly affecting its predicted performance. The upstream mirror width starts as 9.4 mm wide, widens to 10.15 mm over the central 1/3 of the mirror length and then linearly reduces to a 9.5 mm width at the upstream end. The mirror thickness is 4 mm.

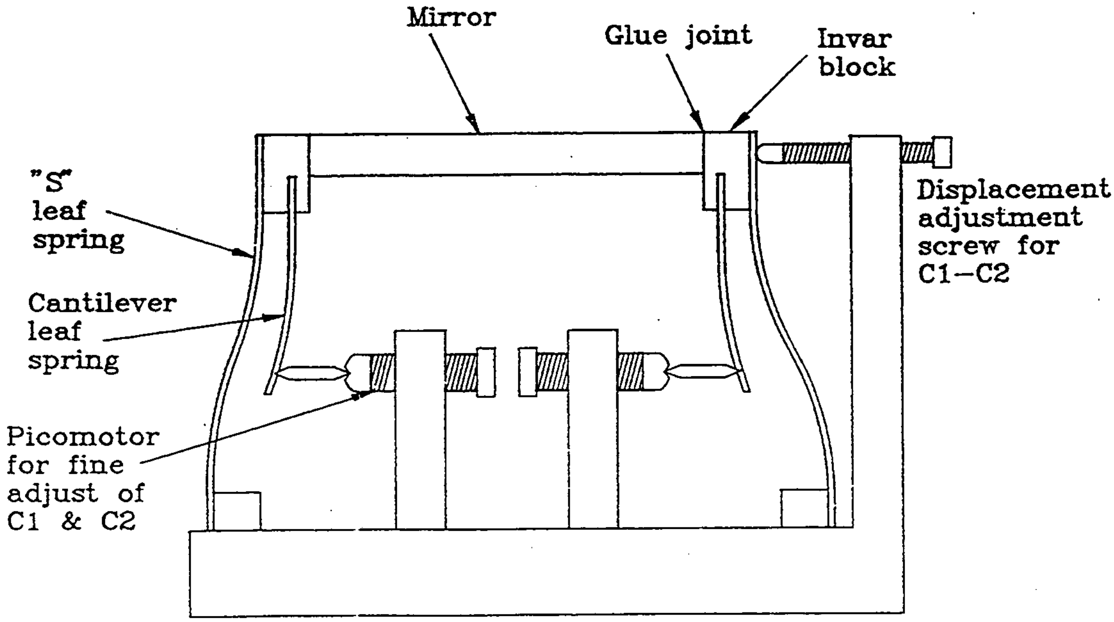

Fig.3. Horizontal focussing mirror bender.

The mirrors have been tested extensively in the ALS optical metrology laboratory, primarily using a long trace profiler[12] to measure slope as a function of position. Typical rms slope errors from the required ellipse over the middle 140 mm of the vertical focussing mirror are around 1.5 μrad. This would be expected to degrade the expected vertical focus to around 1.6 μm FWHM if the 0.5 μm focus size and blurr due to mirror figure error are added in a root mean square manner. For the shorter horizontal focussing mirror we measure an rms slope error from the required ellipse over the central 32 mm of 0.6 μrad as shown in Fig.4. Due to the short distance between the horizontal focussing mirror and the focus, this small slope error is expected to have minimal effect on the

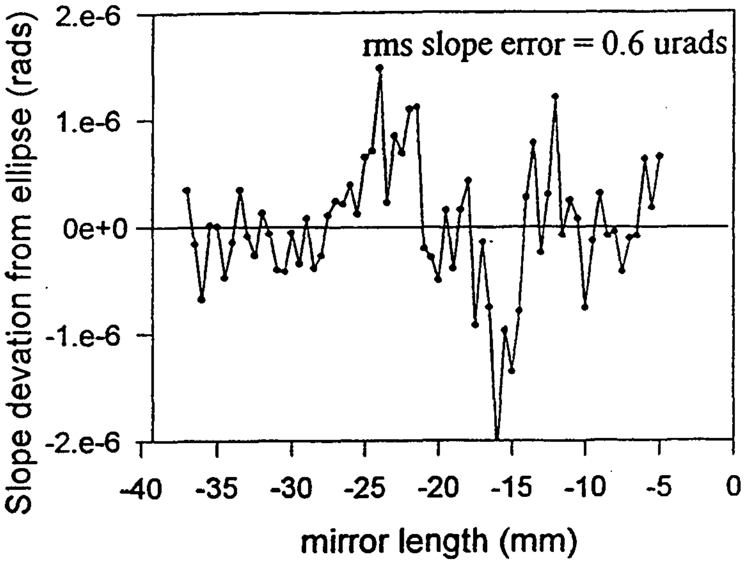

Fig.4. Slope error versus position along the horizontal focussing mirror as measured by the LTP.

horizontal focus size. It is apparent that the closer the KB mirrors are placed to the sample, the less figure errors will degrade the focussed spot size, but as the demagnification will be higher the less aperture can be accepted.

3. X-RAY PERFORMANCE

We have performed knife edge tests on the KB focus with white light radiation. The knife edge used was a cleaved GaAs wafer. By reducing the mirror acceptance aperture to about 60 - 70 %, the best horizontal and vertical foci achieved were 1.6 µm and 2.0 µm FWHM respectively (see Fig.5). These spot sizes are a little larger than one might expect from the consideration of figure errors detailed in the previous section. Fig. 5 shows that there are wings associated with the focussed spot. Fig.6. shows a Laue diffraction spots from a silicon (100) wafer recorded on an x-ray image plate located 40 mm infront the sample. It is clear that the wings are in orthogonal directions consistent with the scattering being caused by the KB mirrors. Such scattering is generally considered[13] to be due to mid spatial frequency errors in the mirror (1mm - 1µm spatial range) that the LTP is unable to resolve. The next iteration of the micro focus mirrors will require special attention to this.

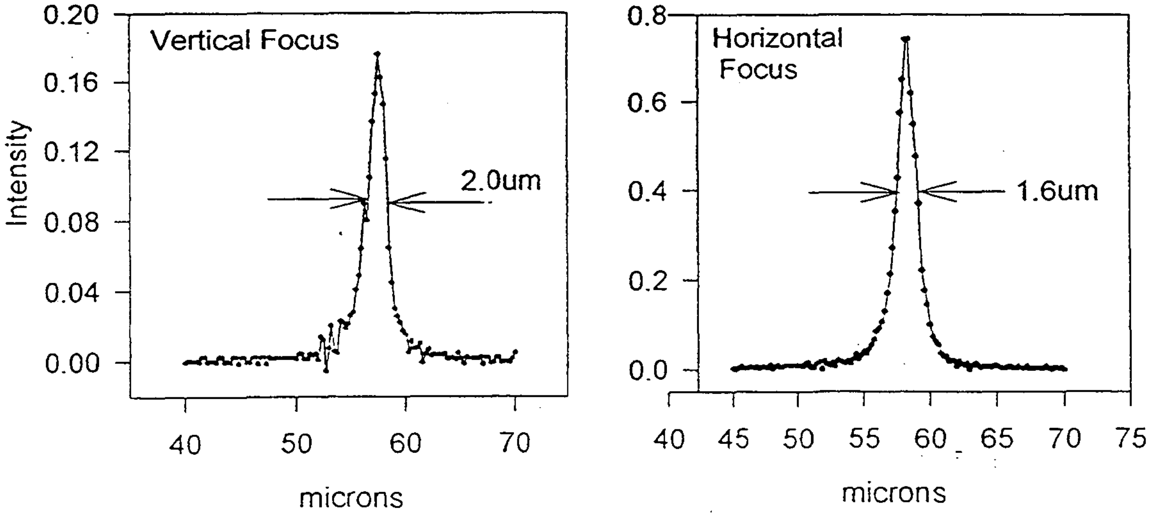

Fig.5. Differentiated knife edge scans in both vertical and horizontal planes, showing the best focus achieved to date.

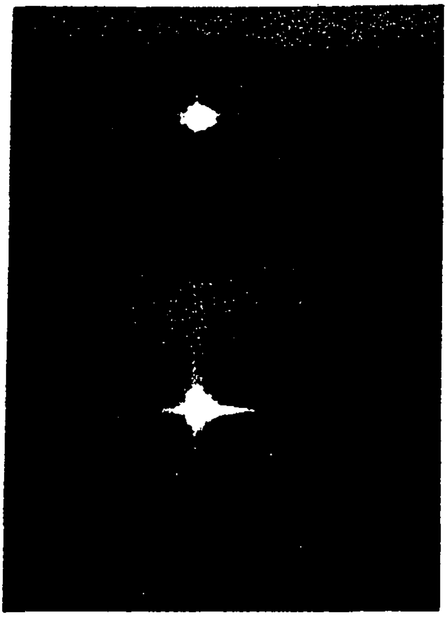

Fig.6. Laue spots (106 - upper, 105 - lower) from a silicon (100) wafer recorded on an x-ray image plate located 40 mm from the sample. The cross shape is considered due to scattering from the KB mirrors.

Fig.7. Far field x-ray image of a Foucault knife edge test with the knife edge in the middle of the best focus. The knife edge is horizontal in the figure.

We also carried out Foucault Knife edge tests [14] on the mirror, where we view the far field x-ray image after the knife by visible light conversion using a single crystal YAG crystal, a visible light microscope and a CCD camera.[15] Ideally a Foucault test on a perfectly focussing optic results in a gradual darkening of the entire far field image as the knife edge is moved through the focus point. Fig .7. shows the Foucault knife edge image recorded at the mid point of the vertical knife edge scan. The overall illumination shows a gradual intensity variation that is consistent with not having a true point focus. There is also some fine structure present in the image which is indicative of possible mirror surface problems, however the small spatial scale of them does not allow us to do anything about them with the bending mechanism.

4. FUTURE DEVELOPMENTS

This development to date is a precursor to the construction of a dedicated beamline for micro-diffraction and x-ray absorption spectroscopy (beamline 7.3.3). This beamline differs in one important respect from the present system. We will use an intermediate toroidal optic to produce a 1:1 focus of the source in the end station hutch at an adjustable slit. The mirror is platinum coated, and will be operated at a 0.33 degree grazing angle, 16 m from the bending magnet source. The adjustable slit will then act as the source for the micro-diffraction setup shown schematically in Fig.1. We note that the current setup uses a large source with gaussian tails (300 x 30 μm FWHM) and a long 30 m beamline that only accepts 7.6 x 31 μrad (horizontal x vertical), whereas the proposed new system will use a smaller adjustable hard edged source (typically 10 x 20 μm), which will image to a 0.5 μm x-ray spot with a 2 mrad convergence angle with KB mirrors only 4 m from the source slits and accepting 100 x 50 μrad of light. Such a setup allows for both KB mirrors to have short mirror to sample distances of 20 and 10 cm which relaxes figure error tolerance. KB mirror lengths will be 35 and 70 mm. As the adjustable slits are overfilled they can be opened to allow more flux through - trading spatial resolution for flux. Fig. 8 shows the calculated throughput for the proposed new setup with Ge(111) monochromator crystals which offer a larger bandpass and are better matched to the KB angular acceptance than Si(111) crystals.

The above discussion presumes that the 1:1 toroidal mirror does not degrade the brightness of the source by imaging the source poorly onto the adjustable slits. The optical surface of the toroidal mirror is of dimensions 700 x 75 mm with a specification of slope errors of < 5 μrad rms over its entire surface. By swapping the order of the

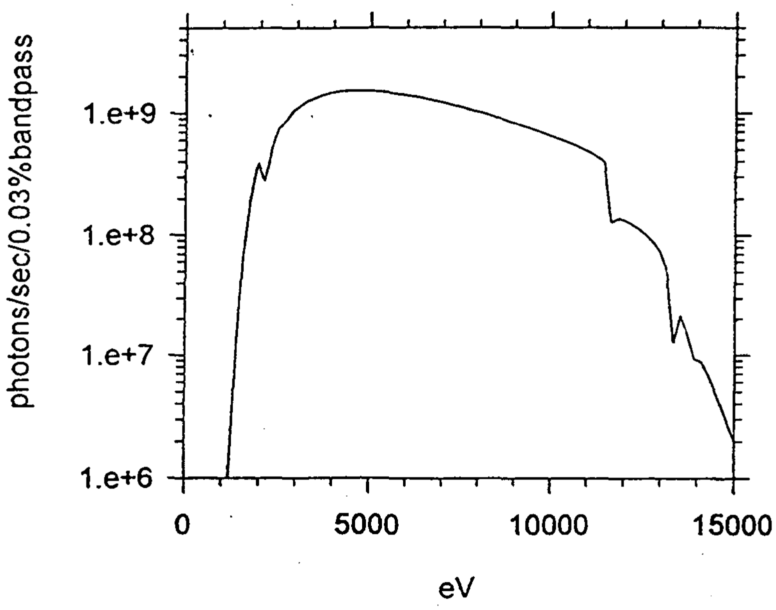

Fig.8. Calculated flux into a 1 μm spot with the proposed micro diffraction beamline with Ge(111) monochromator crystals (0.03% bandpass). The low energy cut off is due to a 125 μm beryllium window, the high energy cut off is due to reduced reflectivity of the mirrors and the fine structure is that of the platinum edges of the mirror coatings. Advanced Light Source operating at 1.9 GeV, 400 mA.

vertical and horizontal KB mirrors in Fig.1 the area of the toroid that the KB mirrors use is only 1.4 x 140 mm. It is anticipated there will be small areas on the toroidal mirror that will have the sub micro radian slope errors such that source brightness is not degraded.

5. CONCLUSIONS

We have demonstrated that grazing incidence K-B mirror systems are capable of high spatial resolution, and large collection aperture. They offer a number of attractive features in comparison to diffractive focusing elements, and it is likely that with further development we should reach 0.5 μm resolution. This will be a matter of procuring better quality substrates. An optical arrangement is under construction that will allow for a hard x-ray microprobe to have high throughput, lower bandpass and variable spot size.

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REFERENCES

1. S.M.Heald, D.L.Brewe, K.H.Kim, C.Brown, B.Barg, E.A.Stern, "Capillary concentrators for synchrotron radiation beamlines", *Proc. SPIE,* **2856**, pp.36-47, 1996.
2. A.A.Krasnoperova, J.Xiao, F.Cerrina, E.Di Fabrizio, L.Luciani, M.Figliomeni, M.Gentili, W.Yun, B.Lai, E.Gluskin, "Fabrication of hard x-ray zone plates by x-ray lithography", *J.Vac.Sci.Technol.B.*, **11**, pp.2588-2591, 1993.
3. M.A.Kumakhov, "Channeling of photons and new x-ray optics", *Nuc.Insrum.Meth.* **B48**, pp.283-286, 1990.
4. A.Snigirev, "The recent development of Bragg Fresnel crystal optics. Experiments and applications at the ESRF", *Rev. Sci. Instrum.*, 66, pp.2053-2058, (1995).
5. P.Kirkpatrick, A.V.Bacz, "Formation of optical images by x-rays", *J.Opt.Soc.Am.*, **38**, pp.766-774, 1948.

6. Underwood, J. H., "Generation of a Parallel X-ray Beam and its use in Testing Collimators", *Space Sci. Instrum.*, **3**, pp.259-270, (1977).
7. H. A. Padmore, M. R. Howells, S. Irick, T. Renner, R. Sandler, and Y-M Koo, " Some new schemes for achieving high-accuracy elliptical x-ray mirrors by elastic bending.", *Proc. SPIE,* **2856**, pp.145-156, 1996.
8. J.H.Beaumont, M.Hart, "Multiple Bragg reflection monochromators for synchrotron radiation", *J.Phys.E,* **7**, pp.823-829, 1974
9. H.Tolentino, A.R.D.Rodrigues, "High resolution monochromator for soft and hard x-rays", *Rev.Sci.Instrum.* **63**, pp.946-949 (1992)
10. Epoxy glue; Epibond 1210A/9615-10 Ciba Geigy, 5121 San Fernando Rd. West, Los Angeles, CA 90039
11. Picomotor, New Focus Inc, 2630, Santa Clara, CA 95051
12. S. Irick, "Improved measurement accuracy in a long trace profiler", *Nucl. Inst. Meth.*, **A347**, 226-30 (1994).
13. J.E.Harvey, K.L.Lewotsky, A.Kotha, "Effects of surface scatter on the optical performance of x-ray synchrotron beamline mirrors", *App.Opt.* **34**, pp.3024-3032, 1995
14. J.Ojeda-Castenada, "Foucault, Wire and Phase Modulation Tests", Chap. 8, Optical Shop Testing, J.Wiley & Sons (pub.), D.Malacara (ed.), 1992
15. B.La Fontaine, A.A.MacDowell, Z.Tan, D.L.White, G.N.Taylor, O.R.Wood II, J.E.Bjorkholm, D.M.Tennant, S.L.Hulbert, "Submicron, soft x-ray fluorescence imaging", *Appl.Phys.Lett.*, **66**, pp.282-284, 1995

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