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Elliptical X-ray Microprobe Mirrors by Differential Deposition

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Abstract
A differential coating method is described for fabricating high-performance x-ray microfocusing mirrors. With this method, the figure of ultra-smooth spherical mirrors can be modified to produce elliptical surfaces with low roughness and low figure errors. Submicron focusing is demonstrated with prototype mirrors. The differential deposition method creates stiff monolithic mirrors which are compact, robust and easy to cool and align. Prototype mirrors have demonstrated gains of more than 10^4 in beam intensity while maintaining submilliradian divergence on the sample. This method of producing elliptical mirrors is well matched to the requirements of an x-ray microdiffraction Kirkpatrick-Baez focusing system.

Introduction
With the recent availability of high-brilliance third-generation synchrotron sources, there is an immediate need for efficient x-ray microfocusing optics. Efforts are currently underway to produce microfocusing optics by a variety of means including tapered capillaries, Bragg-Fresnel Optics, Fresnel Zone plates, compound refractive lenses and Kirkpatrick Baez (K-B) mirrors.[1] Total external specular-reflecting Kirkpatrick-Baez mirrors are particularly attractive for microfocusing broad-bandpass or tunable radiation since they are inherently non-dispersive. Kirkpatrick-Baez optics utilize two concave mirrors at glancing angle to collect and focus x-rays in both vertical and horizontal axis (Fig. 1). K-B mirrors were pioneered in the late 40's,[2] but are being revolutionized by modern materials and processing methods.

In the x-ray regime, K-B optics can be made from total-external-reflection mirrors, multilayer mirrors or a combination of total-external and multilayer mirrors.[3] Total external reflection x-ray optics are efficient x-ray analogs to total internal reflection optics commonly used with visible light. However, at x-ray wavelengths the index of refraction of materials is less than 1 and the deviation from unity is roughly proportional to the electron density.

$$n \sim 1-\delta+i\beta, \qquad (1)$$

Here $1-\delta$ is the real and $i\beta$ is the imaginary index of refraction. In the hard x-ray regime, $\delta \sim 10^{-5}$-10^{-6}. Because $n<1$ x-rays bend away from the normal when entering dense materials whereas visible light bends toward the normal (Fig 2). Nearly 100% specular reflectivity occurs when the glancing angle, θ, is below a so called critical angle θ_c. For a dense metal like palladium, the critical angle depends on x-ray energy as $\theta_c \sim 0.06/E_{keV}$ and the evanescent wave penetrates ~15 to 60 angstroms for θ=3 milliradians and $h\nu$=5 to 20 keV (Fig. 3). High reflectivity is therefore achieved if E<20 keV and θ < 0.003 rad. For x-ray microdiffraction ~25 keV is a useful upper energy and therefore Pd with a K-absorption edge at 24.35 keV is an excellent surface material.

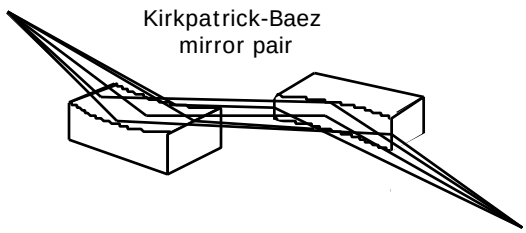


Fig. 1 Kirkpatrick-Baez focusing scheme. The crossed mirrors focus the x-ray beamin orthogonal directions.

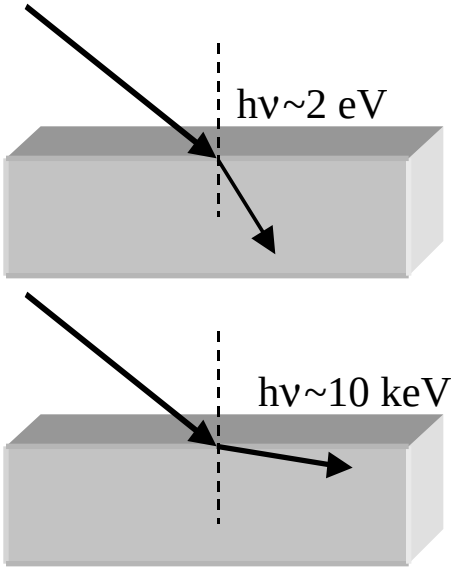


Fig. 2 (a) Visible light is refracted toward the normal on entering materials (b) x-rays are refracted away from the normal on entering materials.

A major obstacle to the use of Kirkpatrick-Baez mirrors is the need to create elliptical surfaces with x-ray quality figure and roughness. Surface figure perfection is required to focus x-rays to a small focal spot. Surface roughness perfection is required to prevent diffuse scattering of x-rays. For microfocusing, mirrors with submicroradian figure perfection and surface roughness of less than ~3 Å is desirable.[4]

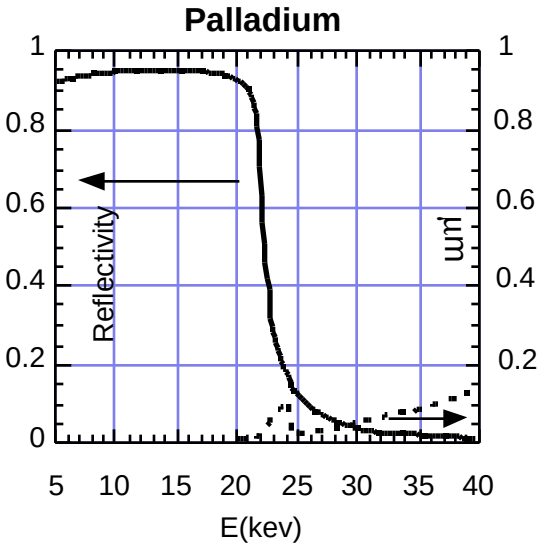


Fig. 3. Reflectivity (solid line) and penetration depth (dashed line) of x-rays from Pd as a function of energy at 3 milliradian glancing angle. The beam penetrates only about 15-50 Å into the Pd surface below 20keV.

Advances in mirror manufacturing have recently made it possible to fabricate x-ray mirrors with a few angstroms root-mean-square (RMS) roughness and with sub-microradian deviations from ideal spherical or flat figure. However no known technique exists for polishing elliptical mirrors to x-ray quality surface roughness. As a result, sophisticated bending techniques are used to shape x-ray quality flats to ellipses.[5,6] These methods have successfully produced submicron x-ray beams, but are sensitive to the bender adjustment, are difficult to cool, and are bulky compared to monolithic mirrors. Because of their additional bulk, benders become increasingly less attractive as the demagnification and hence the focal distance decreases.

In this paper we describe a new approach for the production of elliptical K-B mirrors. With this approach, an ultra-smooth Au layer is differentially deposited on a cylindrical substrate to modify the cylinder to an ellipse. This approach was first suggested by Cai et al.[7] and is similar to ion milling modification of the mirror figure which has been used to perfect figure on high-performance astronomical laser and x-ray mirrors.[8]

Figure modification from spherical to Elliptical.

At large demagnifications elliptical optics are essential for efficient focusing and preservation of beam brilliance (photons/s/eV/μrad^2/μm^2). For example, Howells and Hastings[9] have considered the case of microfocusing with cylindrical optics. They show that spherical aberrations cause a point source to be imaged into a blur in the scattering plane with dimensions determined by the distance to the source, F_1, the opening angle,2γ, of the intercepted radiation and the magnification, M,

$$Y_{Spherical} = 3F_1(1+M)\gamma^2 / 4M\theta . \quad (2)$$

At the Advanced Photon Source (APS), the type A undulators have typical full-width-half-maximum horizontal and vertical source sizes of ~720 x 65 μm^2.[10] Based on the required geometrical demagnification with an ~30 m object distance (F_1), Eq. 2 places a limit on the vertical and horizontal acceptance $2\gamma_V$ and $2\gamma_H$ that can be focused by meridional-focusing cylindrical mirrors to submicron dimensions. In the horizontal axis $2\gamma_H$ =0.86 μrad (26 μm). In the vertical direction $2\gamma_V$=1.6 μrad (49μm). Hence with cylindrical or spherical optics, submicron focusing at 3 milliradian glancing angle is only practical with an ~1.3x10^3 μm^2 beam (gain ~1.3x10^3). In contrast, with elliptical mirrors and the same object distance and magnification, a practical K-B mirror system with less than a 0.5x0.5 μm^2 beam can be fabricated with a collection of ~2.5x10^4 μm^2 (gain~10^5).

The deposition profile required to modify a cylindrical mirror into an elliptical mirror is easily determined from the ideal surface figure of a focusing ellipse and the figure of the cylindrical approximation. For example, in Fig. 4 we plot the surface profile of an ideal ellipse with F_1=30 m, F_2=0.06 m and θ=0.003. This profile is compared with the profile for its cylindrical approximation. The difference between the two profiles is plotted in Fig. 5. This deposition will modify the cylindrical approximation and turn it into an ideal elliptical surface.

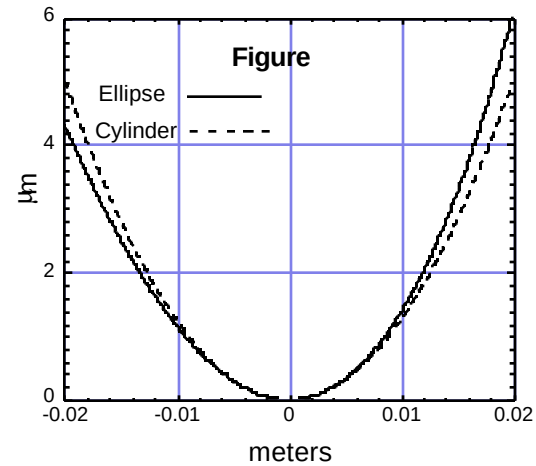


Fig. 4 Figure for an ideal ellipse and for an ideal cylinder.

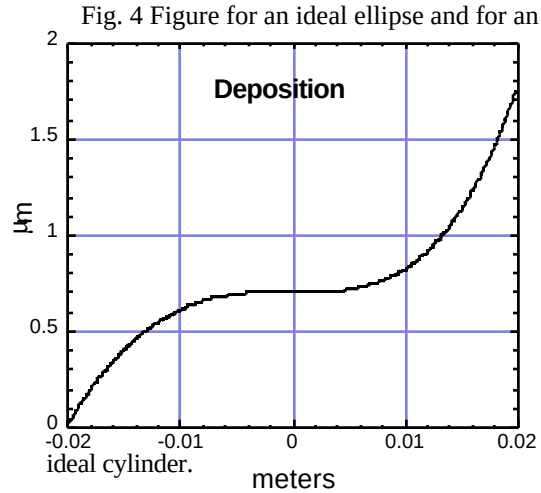


Fig. 5 Deposition to turn an cylinder into an ellipse for object distance F2~35m, and mirror angle θ=0.003 rad.

For a real mirror, the deposition process can also be used to correct small deviations from the nominal cylindrical figure. For example as shown in Fig. 6, the deviations of a 90 mm long substrate from its design cylindrical approximation are on the order of ±3 μrad with an RMS slope error of 1.7 μrad. The long period deviations in slope can be corrected with the potential of reducing the residual slope errors to less than 0.5 μrad as shown.

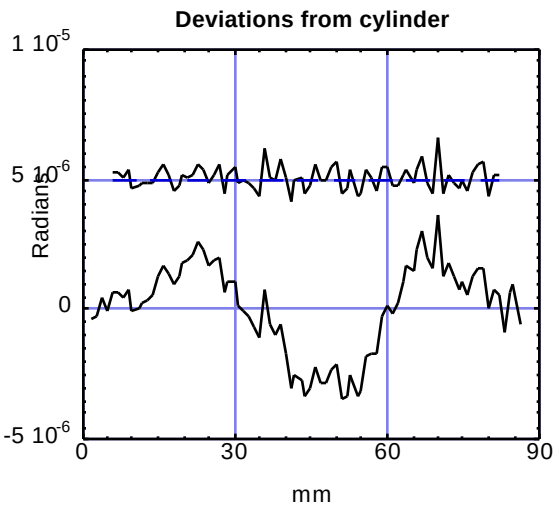


Fig. 6 Bottom curve shows residual slope errors in a nominal 90 mm long cylindrical mirror. If the low frequency slope errors are removed, the high frequency slope errors have a much lower RMS deviation from an ideal cylinder (top curve displaced by 5 microradians).

Fabrication and alignment tolerances

Surface roughness

The surface roughness required for efficient mirrors can be estimated from the fraction of the reflected power contained in the geometrical image. The total intensity into the geometrically demagnified beam is approximately by,

$$I = I_0 \exp\left(\frac{-4\pi\theta\sigma_s}{\lambda}\right)^2. \qquad (3)$$

Here σ_s is the RMS surface roughness below spatial frequencies of ~1 cm and θ is the mirror angle.[4] For 90% power into the geometrical image and with θ=3 mrad, the surface roughness below 1 cm must satisfy σ_S<8.6λ (Å). With a typical operational energy of 20 keV this corresponds to σ_S< 5 (Å). In the horizontal direction, the mirror surface tolerance is less stringent because the focal length is much shorter and the source size larger.

Surface figure

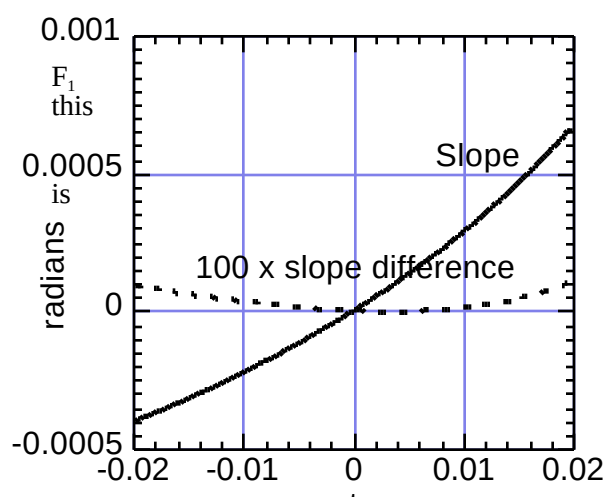
The required surface figure precision can also be set by a simple estimate. Image blur due to slope errors depends on the focal length of the mirror and the root-mean-square (RMS) slope errors; $b=2F_2\sigma_s$ where σ_s is the RMS slope error. For a focal length of 0.13 m, and an RMS desired focal spot of less than 0.5μm, this corresponds to an RMS slope error of ~2μrad. Note to achieve a full-width-at-half-maximum FWHM Gaussian focus below 0.5μm requires a sub-microradian RMS slope errors for this focal length.

Object distance

One concern with a monolithic focusing system is that the device may only be useable on a particular beamline. We note that an ideal microfocusing elliptical mirror works well over a wide range of object distances. Locally the radius of curvature is given by,

$$R = \frac{2F_1F_2}{(F_1 + F_2)}\sin\theta \qquad (4)$$

Here F_1 is the object distance, F_2 is the image distance and θ is the mirror angle. As shown in Fig. 4, the cylindrical approximation matches the slope of the ideal ellipse over a small region, but deviates as the useful aperture of the mirror increases. For F_2 large compared to



approximated by $R=2F_1\sin\theta$; hence the object distance has only a secondary impact on the figure. For example, the ideal slopes for two elliptical mirrors with identical properties except for their object distances (10 and 30 m) are compared in Fig. 7.

Fig. 7 Comparison of the slopes for F_2=0.06 m, θ=0.003, and F_1=10 or 30 m. The difference is shown multiplied by a factor of 100 for clarity.

Image distance

Whereas the object distance can be easily adjusted, the image distance must be held to tolerances of less than about 2%. As shown in Fig. 8, an error of 2 mm in the image distance for a nominal 60 mm focal length mirror (3%)restricts the useful aperture of the mirror to about 75% of the range necessary to collect a 1 milliradian focused beam.

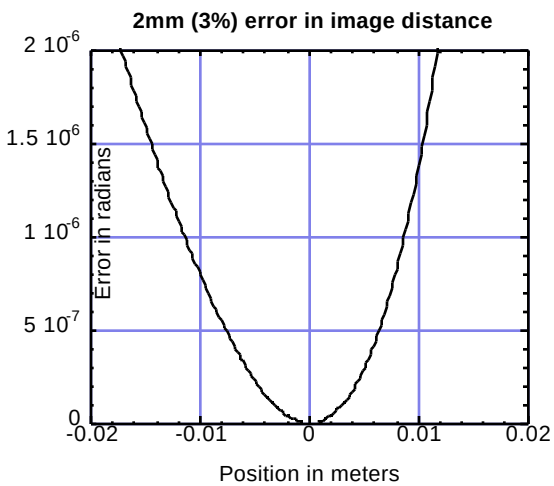


Fig. 8 deviation between an ideal elliptical surface with 62 and 60 mm focal length and a nominal 3 milliradian glancing angle. Note that the angle is adjusted slightly to minimize the deviations.

Scattering angle

The angular precision with which the mirrors must be adjusted can be estimated from the mirror aperture and the desired spot size. For example, a typical microfocusing mirror with 100 µm aperture and a goal of less than 0.5 µm image, must control the focal distance to 0.5%. For a 3 milliradian glancing angle this corresponds to ~15 µrad. If the aperture is smaller, than the angular precision can be relaxed whereas larger aperture increases the required angular precision.

Mirror substrate, measurement and deposition methods

The mirror substrates were prepared at Beamline Technology Corporation (BTC) and differential depositions were performed in a dedicated differential deposition chamber. Small ultra-smooth spherical mirrors were prepared by polishing a large pre-diced Si plate. The large Si plate was divided into smaller mirror blanks by nearly cutting through the plate. The cuts were filled with hard wax and then the entire plate was optically- and super-polished to achieve the desired spherical radius and surface roughness. By polishing a large plate, round-off errors at the edges of the individual mirrors were minimized. After super-polishing, the mirrors were separated and measured with a long-trace profiler (see e.g. Fig. 6). The long-trace profiler measurements given in this paper were performed at the Advanced Photon Source (APS)metrology laboratory and have an uncertainty of less than 0.25 µrad. The design and performance of the APS long-trace profiler has been described earlier.[11]

The differential deposition required to turn the mirror into an ellipse was calculated from the theoretical and measured slopes. Differential deposition was performed at BTC in a specially designed 1.5 m linear UHV coater. Studies at BTC indicate that with proper care, and for ~1 Å RMS roughness substrates, films as thick as 3 µm can be deposited before a measurable increase in surface roughness can be detected. An initial binder coat of Cr is deposited. The mirror is then passed under a sputter source and the sputter source power is varied depending on the position of the mirror. The sputter power is limited to a small fraction of its range to ensure a linear deposition versus power dependence. For the K-B mirrors as many

as 300 passes are required to achieve the desired elliptical figure. A linear gradient in the deposition can be introduced by tilting the mirror. This can compensate for some smooth deviations in the profile and allows the freedom to either choose a deposition profile where the deposition gradient is minimized at the center of the mirror, or a deposition profile where the maximum deposition is minimized. In general we have adopted the approach of minimizing the maximum total deposition at any one point.

The required perfection of the deposition can be estimated from Eq. 2; the actual blur correction scales linearly with fractional deposition scale error times the uncorrected blur predicted by Eq. 2. Typically a deposition perfection of better than 5% is essential for high-performance. A perfection of 1% is required to achieve a 100-200 nm image at the design focal length. Small errors in deposition however can be compensated by fitting the measured slopes to a general elliptical figure to determine the best fit mirror angle and focal length. The mirrors can then be mounted at their best fit positions.

We note that repeated depositions can be used to refine the surface figure and remove residual errors. For example, two depositions within ~10% of the design deposition can be used to achieve the desired 1% figure deposition accuracy.

The slopes from a typical deposition are compared in Fig. 9 to an ideal elliptical surface. Note that the edge effect over the first few mm's of the mirror can be corrected by more precise control of the sputter gun and the carriage.

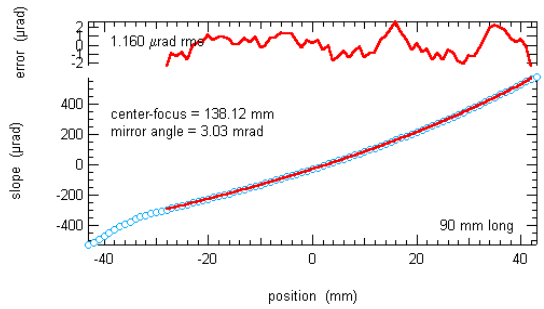


Fig. 9 Comparison of the ideal and actual figure for an elliptical surface. The RMS deviation from the ideal elliptical fit ignoring the first 10 mm of the mirror is about 1µrad. The deviation from the ideal ellipse is even less over smaller regions of the mirror.

Other considerations

<u>Geometrical demagnification</u>
Slits can be used to relax the required geometrical demagnification. For example, as shown in Fig 10, a slit 27 m from the source restricts primarily the beam divergence of the source. The mirror aperture further restricts the effective phase space. Ray tracing indicates that an ideal elliptical surface, optimized for focusing at the undulator source can image to ~0.25µm if the focusing optics is preceded by a 100µm slit 10 m upstream. This can be understood in terms of focusing from the slit itself. Although the object distance is shorter, the effective object size is much smaller which allows for smaller final image size.

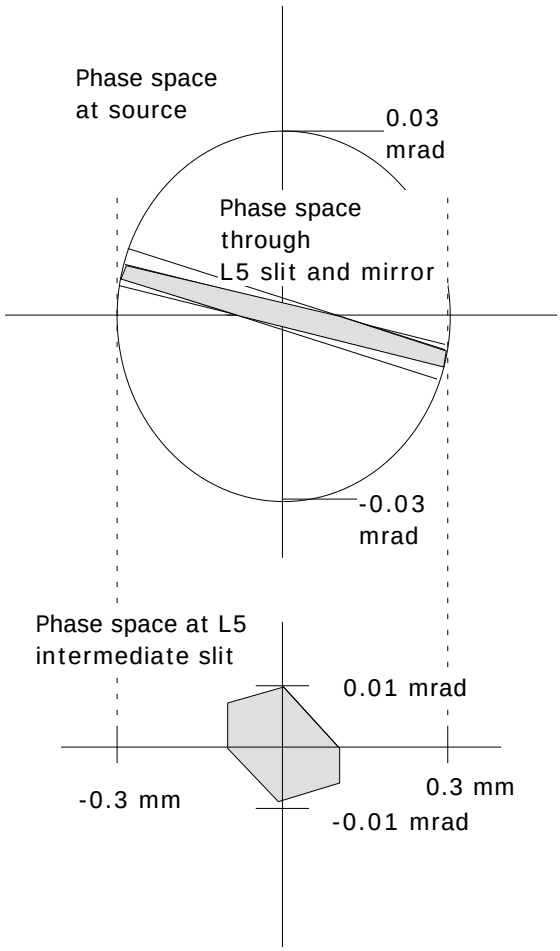


Fig. 10 Horizontal phase space at the source showing the effect of a 100 μm slit at 27 m and a 100 μm mirror aperture at 37 m.

Windows and scattering sources

Windows and other scattering sources increase the beam emittance and degrade focusing performance by shortening the object distance and by increasing the effective object size. The number of windows should therefore be restricted, and they should be polished to minimize scattering.[1] For example, experience on UNICAT beamline 33-ID and other beamlines has shown that the removal of graphite filters can greatly improved x-ray imaging with microfocusing optics.[12] The measurements reported in this paper were preformed with a standard APS graphite thermal filter.

Scattering angle variation

Strongly demagnifying K-B mirrors include a significant variation in the x-ray scattering angle from one end of the mirror to the other. For x-ray microdiffraction it is useful to limit the beam divergence to less than ~1 mrad. This sets a limit on the scattering angle variation to about 0.5 mrad from one end of the mirror to the other. Hence a mirror with a nominal scattering angle of 3 milliradians, has a scattering angle of 2.75 milliradians on one end of the mirror and a scattering angle of 3.25 milliradians on the other end of the mirror. This small variation in the scattering angle causes a gradient in the critical energy from one end of the mirror to the other of ~18-21 keV.

Experimental Results

An x-ray microfocusing system based on differentially deposited mirrors has been fabricated and tested on the MHATT-Cat

beamline 7-ID at the Advanced Photon Source. The source is an APS type A undulator with source parameters described previously. The system used a 90 mm long primary mirror for focusing in the vertical plane and a 40 mm long secondary mirror for focusing in the horizontal (ring) plane. An L5 slit at 27 m was used to control the total power in the beam and to restrict the source size. The geometrically demagnified beams should have a full-width-at-half-maximum (FWHM) of 0.23 x 0.22 in the vertical and horizontal directions. However, slope errors add an additional blurring of about 0.63 (vertical) x 0.3 (horizontal) for 1 μrad slope errors. Adding in quadrature the slope error blurring to the geometrical image we predict an image size of 0.67 vertical x 0.38 horizontal for 1 μrad slope errors. The measured image size is in good agreement with this estimate and indicates that somewhat better than 1 μrad slope errors are present for these mirrors. For best performance, only a fraction of the total mirror acceptance can be used because the deposition process is still not yet precise enough over the entire mirror. With a 50x50 μm^2 entrance aperture, beams as small as 0.4x0.5 μm^2 have been measured. Example beam profiles are shown in Figs. 11a and 11b.

Conclusion

Differential deposition has been used to produce small monolithic elliptical mirrors for x-ray microfocusing. Prototype mirrors already work well and the performance of future versions will continue to improve as the accuracy and precision of the deposition process improves.

The implications of the differential deposition techniques illustrated in this paper go beyond microfocusing mirrors. In general, differential deposition offers a new direction for improving figure errors in virtually all x-ray mirrors. Although differential deposition was used to correct spherical errors in K-B mirrors, it can also be used to remove polishing errors for flats, spheres and aspherical surfaces. Mirrors up to 1.5 m can be handled in the differential deposition chamber used for these studies. BTC has demonstrated the use of differential deposition for refiguring large mirrors where figure errors approaching 1 μrad have been achieved. This new direction offers greater freedom in mirror design and fabrication.

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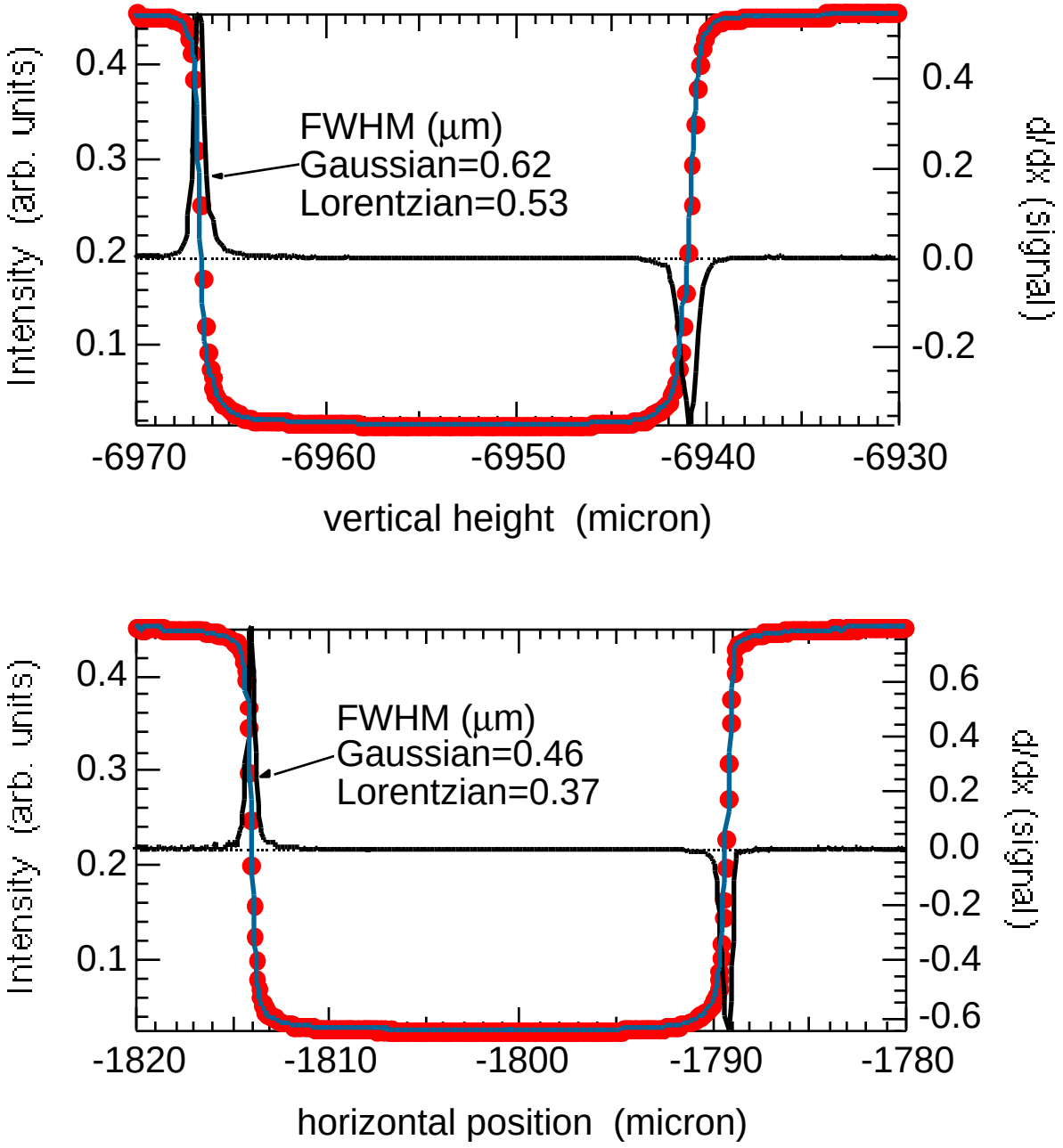

Fig. 11 Measured upper limit to the horizontal and vertical focual spot size. In these measurements the spot size is determined by taking the derivative of the transmitted intensity recorded as a 22 micron diameter Au coated Pt wire is passed in front of the beam. The actual focus is in fact slightly smaller than measured due to edge effects at the wire.

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