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ScienceDirect
Nuclear Instruments and Methods in Physics Research A 582 (2007) 129–131
NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH Section A
www.elsevier.com/locate/nima

Reflective optics for microdiffraction

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Available online 12 August 2007

Abstract

Nondispersive optics are essential for emerging microdiffraction and nanobeam research. Here we describe extensions to traditional Kirkpatrick–Baez optics required to develop nondispersive microdiffraction and nanoprobe optics with 1–10 nm spatial resolution.
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PACS: 07.85.QE; 07.85.FV; 41.50.+h; 83.85.HF; 82.80.Ej

Keywords: Microdiffraction; Kirkpatrick–Baez mirrors; Microbeams; Nanobeams

1. Introduction

Reflective microfocussing optics are undergoing a performance revolution that together with intense synchrotron sources, will enable important new diffraction opportunities for a wide range of science including fundamental materials physics, geology, high-pressure research, and energy-materials research. Although most X-ray diffraction is performed with monochromatic beams and precision sample rotations, *spatially resolved diffraction* requires new approaches based on wide-bandpass or energy-tunable X-ray beams. Nondispersive focusing is essential for both these approaches and Kirkpatrick–Baez mirrors have emerged as the focusing choice for high-resolution microdiffraction beamlines. The potential of advanced reflective optics for microdiffraction and for other synchrotron science has led to a world-wide race to produce ever smaller X-ray beams with pioneering mirror-fabrication efforts centered in Osaka University, Japan, the European Synchrotron Radiation Facility, France and the Advanced Photon Source, USA.

Three-dimensionally resolved microdiffraction is emerging as a particularly important tool for understanding materials. The field is being driven by the ability to resolve small volumes in inhomogeneous "real" materials and by the ability to map local structure and defect distributions. Unlike surface-sensitive probes, microdiffraction can non-destructively study materials below the sample surface. The impact of microdiffraction methods is increasing both due to the development of advanced optics and due to the development of new techniques and software. Interest in microdiffraction is evidenced by the growing number of international conferences addressing this area, and by the development of dedicated—particularly polychromatic—microdiffraction facilities. For example, dedicated beamlines at the Advanced Light Source and Advanced Photon Source have recently been joined by beamlines at the National Synchrotron Light Source and the Swiss Light Source with beamlines on the Canadian Light Source, Australian Light Source and Soleil under development.

Achromatic optics are particularly important for microdiffraction, but are important for ultra-small microprobes of all kinds. Indeed the smallest doubly focused hard X-ray beams have been reported by scientists from Osaka University using total-external-reflection Kirkpatrick–Baez mirrors that have been polished using specially developed methods [1]. More fundamentally, diffraction sets a limit to the product of bandpass and focal length of *chromatic optics.* For optics where the focal length is proportional to the energy, the bandpass that can be focused scales linearly with focal length and scales quadratically with the focal-spot diameter; a 1 nm probe with a 1–10 mm focal length has a theoretical maximum bandpass of less than 10^{-5}–10^{-6} for chromatic optics. This extremely small bandpass sacrifices three to four orders of magnitude from

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0168-9002/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.nima.2007.08.090

a 1%- bandpass undulator spectrum. As a result, achromatic optics are *probably required* for single atom detection with an X-ray nanoprobe. Because of the uncertainty relationship between energy and time, achromatic optics are *definitely required* for femtosecond science with ultra-small beams below ~10–30 nm in size.

For microdiffraction the need for achromatic optics is clear even for larger beams. Polychromatic microdiffraction using Laue diffraction delivers two-dimensional (2D) spatial resolution determined by the focal-spot size of the probe beam, whereas monochromatic diffraction requires sample rotations that introduce sphere-of-confusion and parallax errors that smear the spatial resolution [2]. In addition, polychromatic microdiffraction allows for differential-aperture microscopy, [3] which provides a powerful new way to achieve tomographic diffraction information from crystalline samples. For these reasons, high-performance achromatic optics are essential for emerging micro- and nanodiffraction applications.

2. Future of microdiffraction optics

The spatial resolution of microdiffraction has rapidly improved over the last 10 years with the development of ultra-perfect Kirkpatrick–Baez mirror optics [1,4–6]. The current world's record for smallest doubly focused hard X-ray beam is about $25 \times 30\,\mathrm{nm}^2$. By pushing the design of KB mirrors, it is possible to develop mirrors with beams approaching possibly 17 nm, but current approaches based on total-external-reflection optics cannot go beyond this limit set by diffraction and the critical angle of high-Z metal-coated mirrors.

Several strategies have recently emerged to push microbeam resolution below 10 nm. For example, kinoform refractive optics [7] and Laue Zone-Plate lenses [8] have been suggested as paths to sub-10 nm focusing. However as described above, these chromatic options are not generally suitable for micro/nanodiffraction and are limiting for spectroscopy.

Achromatic mirror optics can also achieve ultra-small spots, but with geometries that go beyond the simple KB design or which rely on multilayer coatings to increase the scattering angle of the mirrors. For example, efforts at Osaka and the ESRF to achieve sub-10 nm beams are based on the use of multilayer mirrors that increase beam divergence (numerical aperture) by a factor of ~4. These strategies provide modest bandpass (~1%) suitable for spectroscopy. Scientists at the APS have proposed mirror optics based on wide-bandpass (~10%) multilayers more suitable for polychromatic microdiffraction. These optics are however somewhat inefficient due to low reflectivity.

Another approach is to use total-external-reflection with geometrical methods slightly more complicated than with traditional KB optics. We propose four methods to achieve high-performance ultra-small beams suitable for polychromatic microdiffraction. These approaches are illustrated in Fig. 1 and are based on challenging but possible extensions of current technology. We model the focal performance with a simple phase-based calculation in Fig. 2. In this calculation the mirror reflectivity was assumed to be nearly uniform. The most straightforward extension of KB mirror optics is with a nested design where the X-ray beam reflects off a small region near the corner of the vertically and horizontally deflecting mirrors (Fig. 1a). This geometry improves the numerical aperture (diffraction limit) by a factor of about ~1.5, and can potentially achieve a beam of ~11 nm with a focal-plane profile very similar—but smaller—than beams currently in use. For example, intensity contour maps in the focal plane for a theoretical KB mirror system working at the critical angle, is compared in Fig. 2a, to an equivalent "nested" design also working near the critical angle (Fig. 2b). An extension of this approach uses four corners to collect an even larger convergence (Figs. 1b and 2c). This geometry leads to much better resolution with a slightly more complicated beam profile at the focus. However, the precision control of 6 degrees of freedom for each of 8 mirror surfaces is a daunting challenge.

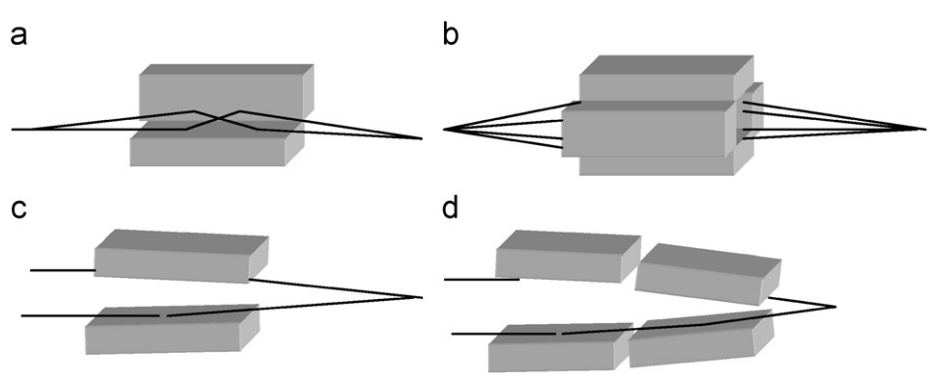

Fig. 1. (a) With nested mirrors the diffraction limit is improved by about a factor of 1.5 over standard KB mirrors (see Fig. 2(b) as compared to Fig. 2(a). (b) With a four corner geometry the diffraction limit is further improved, but with added structure (Fig. 2(c)). (c) Opposing mirrors in each focusing plane improve the diffraction limit by about a factor of 5, but with structured illumination (Fig. 2(d)). In the figure, only the two mirrors collecting the vertical divergence are shown. In a complete system a pair of horizontally focusing mirrors are also needed. (d) Four (or more) mirrors in each focusing plane can be used to further improve the diffraction limit, but with vastly increased complexity and structure in the focused beam. Four of eight mirrors are shown.

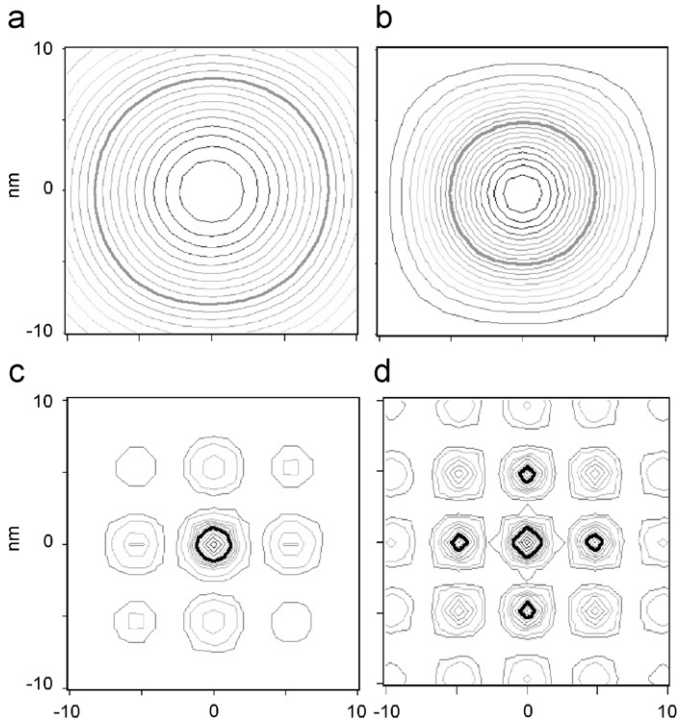

Fig. 2. Model beam intensities at the focal plane for ideal mirror focusing optics are compared. A dark contour is plotted in all four cases at the half intensity contour: (a) this insert illustrates the achievable focal spot for a standard KB mirror system with a diffraction-limited FWHM of about 17 nm; (b) illustrates the improvement possible (~11 nm) with a nested geometry (a in Fig. 1); (c) illustrates the slightly structured illumination that will result from a 4-corner geometry (b in Fig. 1), note the very large improvement in the FWHM at the expense of a complicated beam profile; (d) illustrates the highly structured imaging that results with opposing mirrors in each focal plane (c in Fig. 1). Here the structure is very strong.

A third approach uses opposing sequential KB mirrors to also increase the numerical aperture. This approach creates a highly structured intensity distribution at the focus. Similar approaches are used in optical microscopy and are referred to as "structured illumination microscopy". Once a structured illumination is accepted, the resolution can be further improved by using additional mirrors to collect a larger divergence onto the sample (Fig. 1d). With an 8 mirror system as illustrated in Fig. 1d, the diffraction limit can, in principle, be below 2 nm even with total-external-reflection optics. The ultimate limit, achievable with these optical strategies, may be set by the evanescent wave field in total external reflection.

Acknowledgement

This research was supported by the Division of Materials Science and Engineering, US Department of Energy through Contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory. Experimental measurements were performed in part on beamline 34-ID at the Advanced Photon Source, which is supported by the US DOE Office of Basic Energy Sciences, Office of Science under Contract no. W-31-09-ENG-38.

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