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Citation: AIP Conf. Proc. **1365**, 231 (2011); doi: 10.1063/1.3625346
View online: http://dx.doi.org/10.1063/1.3625346
View Table of Contents: http://proceedings.aip.org/dbt/dbt.jsp?KEY=APCPCS&Volume=1365&Issue=1
Published by the American Institute of Physics.

Development of Coherent X-ray Diffraction Apparatus with Kirkpatrick-Baez Mirror Optics

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Abstract. To realize coherent x-ray diffraction microscopy with higher spatial resolution, it is necessary to increase the density of x-ray photons illuminated onto the sample. In this study, we developed a coherent x-ray diffraction apparatus with Kirkpatrick-Baez mirror optics. By using mirrors fabricated by elastic emission machining, a high-density coherent x-ray beam was produced. In a demonstration experiment using a silver nanocube as a sample, a high-contrast coherent x-ray diffraction pattern was observed over a wide-q range. This proves that both the density and the degree of coherence of the focused beam were high.

Keywords: Coherent x-ray diffraction microscopy, Kirkpatrick-Baez mirrors
PACS: 61.05.cf, 07.85.-m, 68.37.Yz

INTRODUCTION

Coherent x-ray diffraction microscopy (CXDM) [1], which combines coherent x-ray scattering and the iterative phasing method, is highly attractive as a method of lensless x-ray microscopy. In principle, CXDM provides a spatial resolution close to the x-ray wavelength. In principle, the resolution of this microscopy depends on both the quality of x-ray diffraction data over a wide-q region and the uniqueness of phase-retrieval calculations, where q is the magnitude of the scattering vector. Since the diffraction intensity decays with the power law $q^{-\alpha}$ (α~4), the sample must be illuminated with high-flux coherent x-rays to collect high-q diffraction data. Kirkpatrick-Baez (KB) mirrors [2], which consist of two elliptical mirrors, are a promising x-ray focusing device. Robinson *et al.* have proposed the use of KB mirrors in CXDM and have shown the enhancement of the available signal for coherent x-ray diffraction at Advanced Photon Source [3]. Recently, we have realized high-resolution CXDM using a synchrotron x-ray beam focused by KB mirrors with a high-accuracy surface at SPring-8 [4]. The spatial resolution has reached the sub-5-nm scale in two dimensions [4] and the sub-10-nm scale in three dimensions [5]. In this paper, the high-resolution coherent x-ray diffraction apparatus with KB mirrors and its performance are described.

DESIGN AND FABRICATION OF ELLIPTICAL MIRRORS

KB mirrors were designed to produce diffraction-limited focused x-rays of ~1-μm spot size at an incident x-ray energy of ~10 keV. In addition, to obtain a high photon density at the focus, a large geometrical demagnification of the source was considered. Table 1 shows a summary of the parameters of the elliptical mirrors designed for the CXDM experiments at BL29XUL in SPring-8. The material of the mirrors is fused silica. The maximum glancing

The 10th International Conference on X-ray Microscopy
AIP Conf. Proc. 1365, 231-234 (2011); doi: 10.1063/1.3625346

angles of the first and second mirrors are 1.30 and 1.05 mrad, respectively. More than 99% of the x rays at ~10 keV are reflected at the mirror surface when the surface is ideal. The mirror length and width are 90 and 4 mm, respectively. The synchrotron x-rays are vertically focused by the first mirror and horizontally focused by the second mirror. According to the result of wave optical simulation, the designed KB mirrors can produce nearly diffraction-limited two-dimensional focused x-rays of ~1-μm spot size at ~10 keV [6]. To realize nearly ideal x-ray focusing, mirrors with a peak-to-valley (PV) figure error of less than ~5 nm had to be fabricated. To meet this requirement, KB mirrors were fabricated by elastic emission machining [7] (EEM). Figure 1(a) shows the appearance of the KB mirrors. Figures 1(b) and 1(c) show the profiles of the surface figures of the fabricated mirrors, which were obtained by combining microstitching interferometry and relative-angle determinable stitching interferometry. Figures 1(d) and 1(e) show the figure error profiles for both mirrors. The PV error was less than 1.0 nm.

TABLE 1. Parameters of Designed Elliptical Mirrors

	First mirror	**Second mirror**
Maximum glancing angle (mrad)	1.30	1.05
Acceptance width (μm)	113	90
Focal length (mm)	600	495
Length of major axis (m)	48.600	48.600
Length of minor axis (mm)	6.708	4.880

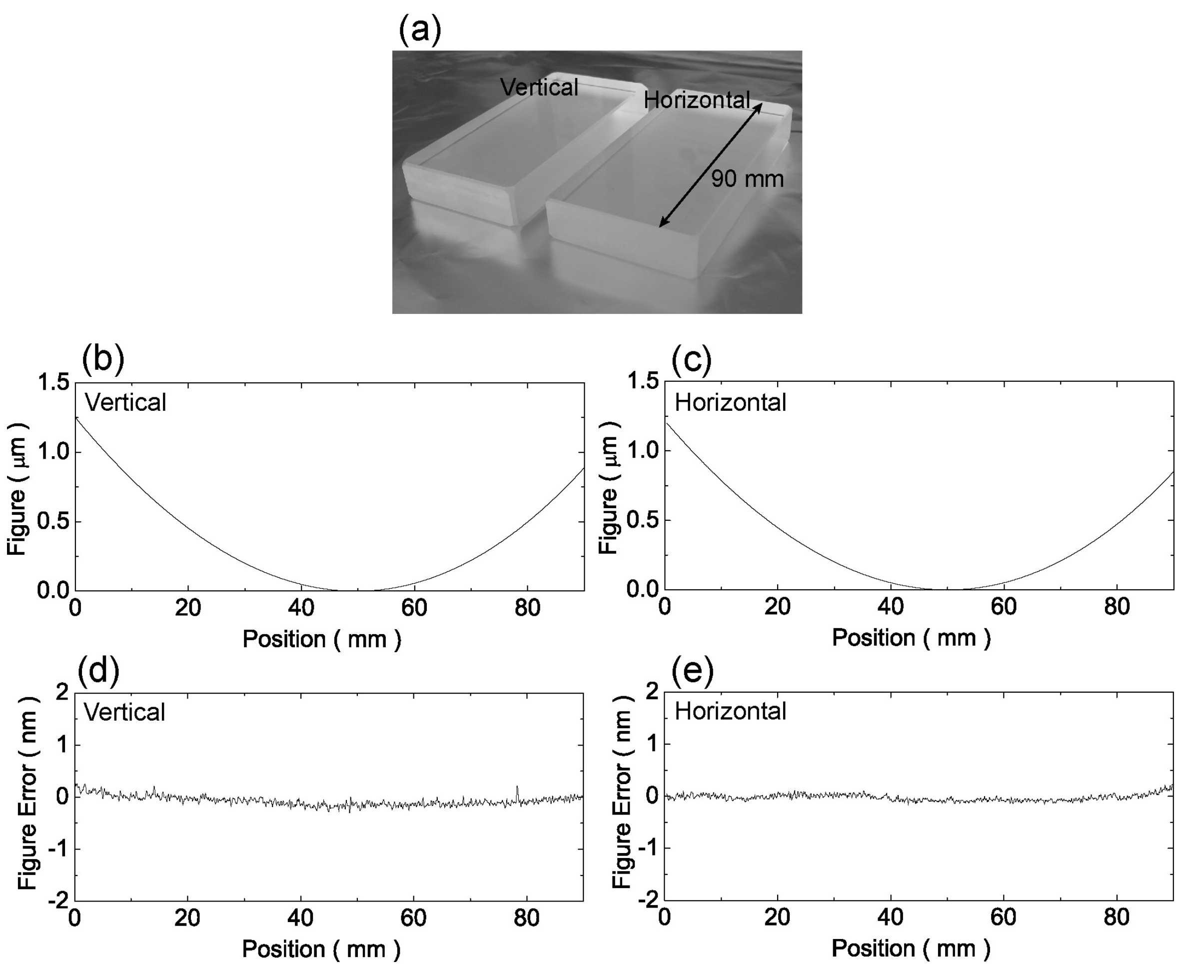

FIGURE 1. (a) Appearance of elliptical mirrors fabricated by EEM technique. (b, c) Profiles of the surface figures of fabricated (b) vertical and (c) horizontal mirrors. (d, e) Figure errors of the (d) vertical and (e) horizontal mirrors.

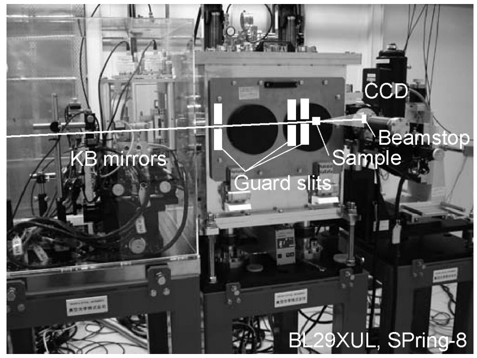

FIGURE 2. Experimental setup for coherent x-ray diffraction microscopy with KB mirrors at BL29XUL in SPring-8.

EXPERIMENTAL SETUP

We carried out coherent x-ray diffraction measurement of a silver nanocube with an edge length of ~100 nm to test the performance of the diffraction microscope with KB mirrors. The silver nanocube was prepared by a modified version of the polyol synthesis technique developed by Xia *et al.* [8]. The x-ray energy was tuned to 11.8 keV using an undulator gap and a Si (111) double-crystal monochromator. The monochromatic x-rays passed through the first cross slit with a 100-μm opening in both directions, which was placed ~50 m downstream of the source. The KB mirrors were placed in air ~100 m downstream of the source in the second experimental hutch (EH2). Figure 2 shows the experimental setup in EH2, which consists of KB mirror units, a vacuum chamber, and detectors. The nanocube and guard slits were placed in the vacuum chamber. The second cross slit was placed immediately before the KB mirrors to control the area of x-ray illumination on the mirrors. The size of the second slit was adjusted to 100 μm in both directions. The x-ray beam was two-dimensionally focused in the vacuum chamber 445 mm downstream of the second mirror by the KB mirrors, which were controlled by a specially designed manipulation system. The higher-order harmonics of the undulator radiation were cut to 1% by double reflections. The focal profile was measured by the wire-scanning method using a gold wire of 200-μm diameter. To interrupt x-rays scattered from the mirrors, three guard slits were placed between the mirrors and the focal point. The nanocubes were mounted on a 100-nm-thick SiN membrane. To prevent x-ray-induced charging, a few-nm-thick carbon film was deposited beforehand on the SiN membrane chip. An isolated nanocube was selected and illuminated using the focused x-ray beam. The diffracted x-ray photons were detected by an in-vacuum front-illuminated CCD detector with 1300 × 1340 pixels and a pixel size of 20 × 20 μm^2 placed 306 mm downstream of the sample. A beam stop was placed in front of the CCD detector. The exposure time of x-rays was 800 s.

EXPERIMENTAL RESULTS

The intensity of the reflected x rays was ~2 × 10^{11} photons/s, which was almost equal to that immediately after the second slit. The x-ray reflectivity was close to 100%. The full width at half maximum of the focal profiles was ~1 μm along both directions. The estimated x-ray photon density at the focus was ~1 × 10^4 photons/nm^2/s, which was more than 100 times that of the unfocused x-rays. Figure 3 shows the measured coherent diffraction pattern of the silver nanocube. The black square in the upper right is an unmeasured region due to the beam stop. The clear speckle patterns extending crosswise reflect the cubic shape of the particle. Interference fringes with high visibility are observed over a wide-*q* range, which means that both the density and the degree of coherence of the focused beam were high. The highest-*q* diffraction intensities were observed at 500 μm^{-1}, which means that the structure was resolved with 1-nm resolution by x-ray diffraction. In the present experiment, the observable highest-*q* diffraction was limited by the detector size.

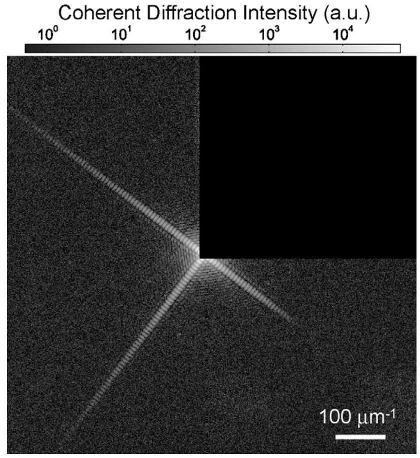

FIGURE 3. Coherent diffraction pattern of a silver nanocube in 1340 × 1300 pixels.

SUMMARY AND OUTLOOK

We have developed a coherent x-ray diffraction apparatus with KB mirror optics to realize high-resolution CXDM. By using the mirrors fabricated by EEM, a high-density coherent x-ray beam, which is suitable for CXDM, was successfully produced. In a demonstration experiment using a silver nanocube, a high-contrast diffraction pattern was observed. The present apparatus is expected to not only be useful for high-resolution CXDM experiments at synchrotron radiation facilities but also contribute to single-molecule analysis using x-ray free-electron lasers in the near future.

ACKNOWLEDGMENTS

This research has been carried out at the Frontier Research Base for Global Young Researchers, Osaka University, in the Program of Promotion of Environmental Improvement to Enhance Young Researcher's Independence, the Special Coordination Funds for Promoting Science and Technology, from the Ministry of Education, Culture, Sports, Science and Technology (MEXT). This work was also partly supported by funds from a Grant-in-Aid for the "Promotion of X-ray Free Electron Laser Research", Specially Promoted Research (No. 18002009), Young Scientists (No. 21686060), Challenging Exploratory Research (Grant No. 22651040), the Global COE Program "Center of Excellence for Atomically Controlled Fabrication Technology" from MEXT, and Shimadzu Science Foundation. The authors would like to acknowledge JTEC Corporation for fabrication of the mirrors, and N. Zettsu for fabrication of the silver nanocube.

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