Hard X-ray nanobeam scanning using advanced Kirkpatrick-Baez mirrors and prisms

Scanning X-ray microscopy (SXM) is one of the major X-ray microscopy techniques. In SXM, a focused X-ray beam is irradiated onto a sample and images are acquired by scanning the relative positions of the beam and the sample. The spatial resolution of SXM is basically determined by the focused spot size. However, the resolution is practically limited by the scanning accuracy. Recent advances in synchrotron radiation sources and X-ray nanofocusing optics have markedly reduced the focused beam size to the single-nanometer scale [1]. The SXM performance has eventually become no longer limited by the size of the X-ray beam, but by the scanning system. In scanning electron microscopies that have achieved an outstanding resolution, the beam is precisely controlled by an electromagnetic deflector. In contrast, X-ray beams are rarely steered in SXM in the same way as in electron microscopy owing to the lack of appropriate X-ray optical components, and hence, a mechanical scanning device for the sample, not for the X-ray beam, has been explored for over half a century. In recent years, scanning systems with an accuracy of ~20 nm have been constructed, but complex equipment such as positioning stages with closed-loop feedback using additional measurement devices, heat-insulated mechanics, and massive granite bases have led to difficulties in achieving further precision without constraining the sample environment. Although fourth-generation high-brightness SR sources can significantly enhance the SXM performance, sample scanning restrictions have been a barrier to the widespread use of ultrahigh resolution SXM for diverse samples.

In this work, we propose an optical scheme of the hard X-ray nanobeam scanner for ultrahigh resolution SXM [2]. The optics consists of an X-ray prism to deflect the incident X-ray beam and an advanced Kirkpatrick-Baez (AKB) mirror to generate nanobeams (see Fig. 1). The AKB mirror approximately satisfies Abbe's sine condition and thus has an excellent imaging property with a wide angle of view. Reciprocally, when AKB mirrors are used in a focusing configuration, one can keep a wide range in which the focus size will not change regardless of the incident angle. Taking advantage of this characteristic, the focused beam with AKB mirrors can be scanned with a nanometer-level accuracy by changing the incident angle by adjusting the X-ray prism. The deflection angle of the incident beam induced by prism rotation is very small and can be precisely controlled with a nanoradian accuracy by degree-order rotation of the prism. We note that the total reflection focusing mirror can generate high-intensity X-ray nanobeams owing to its larger spatial acceptance and higher reflectivity than the focusing zone plate.

Using a prism with an apex angle of 90°, the deflection angle of the X-ray prism $\Delta\theta$ is approximately given by [3]

$$\Delta\theta \approx \frac{2\delta}{\sin 2\theta} \,. \qquad (1)$$

where δ denotes the phase-shifting part of the refractive index and θ denotes the glancing incident angle, as illustrated in Fig. 2(a). In Fig. 2(b), $\Delta\theta$-*versus*-θ relationships are shown as solid lines, which were calculated for the rotation of the prism made of glassy carbon (density ρ = 1.51 g/cm^3) and photon energies of 10 and 12 keV. Note here that $\Delta\theta$ is in units of μrad, whereas θ is in degrees, which leads to high-accuracy scanning by prism rotational scanning. The transmittance of the prism is acceptable for SXM when one chooses a higher photon energy above ~10 keV and low-atomic-weight materials for the prism.

A nanobeam scan experiment was performed at SPring-8 **BL29XU** with a photon energy of 10 keV. X-ray prisms with apex angles of 90° made of glassy carbon were utilized. The incident and exit surfaces of the prism were polished. First, the deflection property was tested. An X-ray beam from a slit with a 15-μm-square aperture irradiated the prism arranged in the horizontal direction. The deflected beam was monitored using a high-resolution X-ray camera (Fig. 2(c)) placed 0.988 m downstream from the prism. The incident angle was varied by rotating the prism,

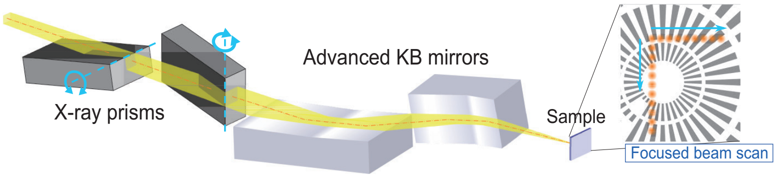

Fig. 1. Conceptual schematic of the hard X-ray nanobeam scanner.

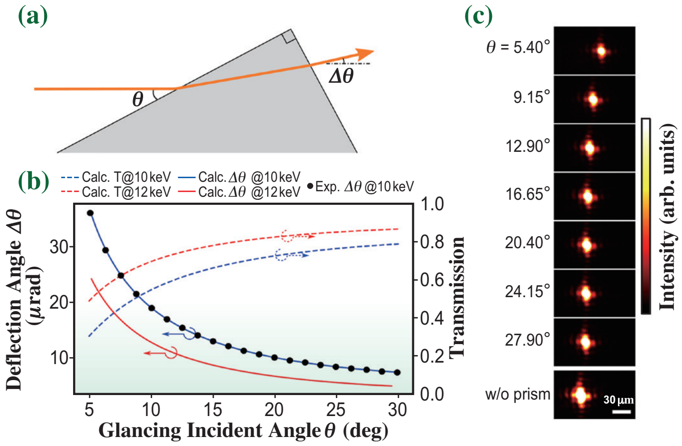

Fig. 2. **(a)** Schematic of an X-ray prism and trajectory of the deflected X-ray beam. **(b)** Incident angle dependence of the deflection angle and transmission of the prism. The solid blue (red) line indicates the calculated deflection angle at a photon energy of 10 keV (12 keV). The dashed blue (red) line indicates the calculated transmission at 10 keV (12 keV) when the incident beam width is 0.6 mm (see Suppl.). Black dots indicate the deflection angles examined by experiment. **(c)** Images of the deflected X-ray beams while the incident angle to the prism was varied from 5.4 to 27.9 degrees. The bottom image is that without the prism.

whose rotation center was at the apex. The obtained relationship between the deflection angle and the glancing incident angle was in good agreement with the calculation, as shown by the black dots in Fig. 2(b).

The estimated deflection accuracy was 17 nrad root mean square (rms), including the uncertainty of the deflection angle measurement, which guaranteed a scanning accuracy of at least 2.9 nm rms on the focal plane.

Subsequently, a vertical prism and AKB focusing mirrors were added and aligned. The mirrors were developed in a previous study [4] and achieved nearly diffraction-limited performance. From the results of knife-edge scanning measurements with 50 μmϕ gold wires, the focus size was measured to be 52.5 (vertical) × 53.2 (horizontal) nm^2 full width at half-maximum (FWHM) (see Fig. 3(a)). An SXM image was obtained using the nanobeam scanner. X-ray transmission through a radial test pattern was acquired using two intensity monitors placed immediately upstream of the mirrors and downstream of the pattern. The result is shown in Fig. 3(b). A clear image, in which the innermost structures were 50 nm lines and spaces, could be obtained. The high-resolution image without notable distortion represents the accuracy and validity of the proposed scheme.

The hard X-ray nanobeam scanner demonstrated here is based on a simple principle and can readily achieve single-nanometer accuracy that is nearly free from scanning error and the thermal instability of mechanical stages. It will pave the way toward the use of ultimate-resolution SXM for various scientific investigations, especially in 4th-generation SR sources.

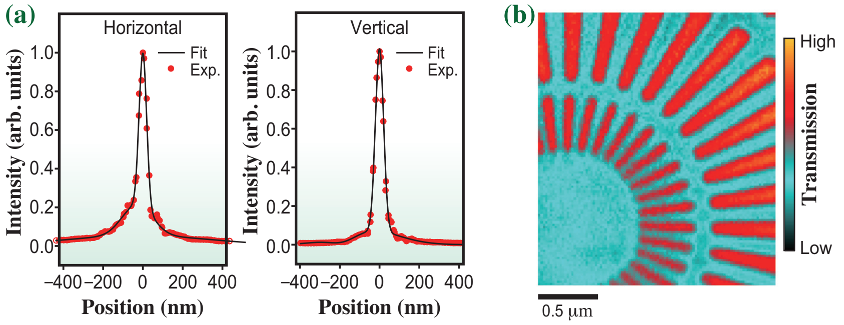

Fig. 3. **(a)** Focusing profiles in the horizontal (left) and vertical (right) directions. Red dots indicate the experimental data. Black solid lines represent the fitting results with the sum of three Gaussian functions. The focus size was 53.2 nm (horizontal) × 52.5 nm (vertical) FWHM. **(b)** SXM image obtained using the nanobeam scanner. Transmission image of the radial test pattern made of 500-nm-thick tantalum.

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