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Citation: *Appl. Phys. Lett.* **110**, 101103 (2017); doi: 10.1063/1.4977882

View online: <http://dx.doi.org/10.1063/1.4977882>

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Focusing hard x rays beyond the critical angle of total reflection by adiabatically focusing lenses

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(Received 3 January 2017; accepted 20 February 2017; published online 7 March 2017)

In response to the conjecture that the numerical aperture of x-ray optics is fundamentally limited by the critical angle of total reflection [Bergemann *et al.*, Phys. Rev. Lett. **91**, 204801 (2003)], the concept of adiabatically focusing refractive lenses was proposed to overcome this limit [Schroer and Lengeler, Phys. Rev. Lett. **94**, 054802 (2005)]. We present an experimental realization of these optics made of silicon and demonstrate that they indeed focus 20 keV x rays to a 18.4 nm focus with a numerical aperture of $1.73(9) \times 10^{-3}$ that clearly exceeds the critical angle of total reflection of 1.55 mrad. Published by AIP Publishing. [<http://dx.doi.org/10.1063/1.4977882>]

While the wavelength of hard x rays lies in the Ångström range, focusing x rays down to atomic dimensions has not been achieved, so far. The main reason is the weak interaction of x rays with matter that makes it difficult to fabricate x-ray optics with high numerical aperture. When analyzing the spatial confinement of x rays inside a waveguide, Bergemann *et al.*¹ conjectured that the numerical aperture of x-ray optics was physically limited to the critical angle of total reflection. Indeed, besides for waveguides,¹ this assumption holds for mirror optics based on total external reflection² and refractive x-ray lenses,^{3–5} limiting x-ray nanobeams to tens of nanometers.^{1,4} For x-ray optics based on diffraction, such as Fresnel zone plates, multilayer Laue lenses, or multilayer mirrors, it was shown^{6–8} that their numerical aperture is not bounded from above by the critical angle of total external reflection. Also kinoform lenses can in principle overcome this limitation.⁹ Their fabrication is extremely challenging, and their performance so far largely dominated by aberrations.^{9–11}

Conceptually, adiabatically focusing lenses (AFLs) were shown to be able to overcome the limited numerical aperture of conventional refractive optics.¹² The main idea is to make an optically thick compound refractive lens, in which the aperture of individual lenses is gradually (adiabatically) adapted to the size of the converging beam inside the optic [cf. Fig. 1(a)]. In this way, the refractive power and with it the numerical aperture increases towards the exit of the lens, making in principle single-digit nanometer focusing possible.¹² Realizing such an optic is technologically very challenging, as individual refractive lenses become smaller and smaller towards the exit of the lens.

In this letter, we report on the first experimental realization of an adiabatically focusing lens made of silicon,

demonstrating nanofocusing down to 18.4 nm full width at half maximum (FWHM) at an x-ray energy of $E = 20$ keV. The nominal numerical aperture $NA = 1.73 \times 10^{-3}$ of this optic¹² exceeds the critical angle of total reflection $\theta_C = \sqrt{2\delta} = 1.55$ mrad, where δ is the index of refraction decrement for x rays in silicon. For hard x rays, the refractive index of a medium is typically written as $n = 1 - \delta + i\beta$, where $\delta > 0$ is the refractive index decrement, and $\beta = \frac{\mu}{4\pi}$ describes the attenuation in the medium. These values are compared to those determined experimentally using scanning coherent x-ray diffraction microscopy, also known as ptychography.^{13,14} Based on the reconstructed wave field from ptychography, we extract the numerical aperture by two methods, measuring the speckle size in the near field of the focus and evaluating the divergence of the beam in the far field.

Following the general design presented in Ref. 12, we fabricated one-dimensional adiabatically focusing lenses by deep reactive ion etching into silicon. Silicon was chosen primarily for the advanced microfabrication techniques that are available for this material. Diamond and alumina would be even better suited lens materials, due to their high density and low absorption compared to silicon.¹² However, it is currently not possible to microstructure these materials with sufficient accuracy. AFLs are designed for a given energy. Their optical properties are determined¹² by the lens material and their entrance and exit aperture radius R_{0i} and R_{0f} , respectively (cf. Fig. 1).

The optic presented here was designed for $E = 20$ keV and has a $R_{0i} = 4.7 \mu\text{m}$ and a $R_{0f} = 1.0 \mu\text{m}$ and is made of $N = 508$ individual lenses. Fig. 1 shows the scanning electron micrographs of the AFL, showing in detail the entrance [Fig. 1(b)] and exit [Fig. 1(c)] of the lens. The main technological challenge to make these optics lies in the fabrication of the deeply etched structures with steep and smooth side walls and with very different lateral sizes within the optic.

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FIG. 1. (a) Scheme of the adiabatically focusing lens (AFL, adapted from Ref. 12). (b) and (c) Scanning electron micrographs of the AFL. (b) Entrance and (c) exit of the optic on the same scale (magn. 750 $\times$). The insets show enlarged details of the very first (magn. 3000 $\times$) and last lenses (magn. 12 000 $\times$) in the stack, respectively.

The aperture radius R_{0j} and with it the radius of curvature R_j of the j -th individual lens in the stack are adapted to the converging x-ray beam at the design energy (Fig. 1). For practical reasons, the minimal thickness of the individual lenses was kept constant at $d_j = 1.0 \mu\text{m}$ (Fig. 1). As a result, the analytical expressions for the optical properties of AFLs given in Ref. 12 do not properly describe the optic considered here. Instead, here, we calculate the properties numerically by wave propagation. The AFL has an overall length of $L = 3.714 \text{ mm}$ and a focal length of $f = 1.915 \text{ mm}$ as measured from the second principal plane that lies $H = 1.510 \text{ mm}$ before the exit of the lens [cf. Fig. 1(a)]. Table I summarizes the parameters for this optic.

TABLE I. Summary of geometric and optical parameters of both AFL and the nanofocusing lens (NFL). Optical parameters are given for 20 keV.

Optic	AFL	NFL
Material	Si	Si
Length L (mm)	3.714	9.0
Number of lenses N	508	300
Entrance aperture $2R_{0i}$ (μm)	9.4	38
Entrance curvature radius R_i (μm)	2.45	12.4
Exit aperture $2R_{0f}$ (μm)	2.0	38
Exit curvature radius R_f (μm)	0.5	12.4
Minimal lens thickness d (μm)	1.0	1.0
Transmission	0.27	0.29
Focal length f (mm)	1.915	18.7
Working distance z_f (mm)	0.405	14.0
Numerical aperture NA	1.73×10^{-3}	0.48×10^{-3}

The lens is made of silicon by a plasma-based anisotropic etching process developed at the Institute of Semiconductor- and Microsystems Technology (IHM) of Technische Universität Dresden, Germany. The resist mask pattern was fabricated by optical lithography using an i-line wafer stepper at SAW Components, Dresden. In order to achieve small and precise deeply etched structures with steep side walls and low surface roughness, a semi-continuous deep reactive-ion etching technique (DRIE) was developed. It combines the classical discontinuous DRIE process (also known as Bosch process)¹⁵ with a continuous one. The main idea of DRIE is to apply the surface passivation in addition to the reactive ion etching. In discontinuous DRIE, passivation and etching are performed alternatively, while in continuous DRIE, passivation and etching are carried out simultaneously without interruption. The first (discontinuous) process is characterized by very high etch rates in the range of several $\mu\text{m min}^{-1}$. Furthermore, a high etching selectivity between the mask material (photoresist) and the substrate material (silicon) of $\approx 50 : 1$ can be achieved. Passivation by polymer deposition and etching using SF_6 is adjusted in such a way, that steep side walls are created. But the surface roughness is quite high (about 150 nm rms), due to the cyclic changing plasma conditions. In the second (continuous) case, the surface roughness is much reduced (to less than 60 nm rms), but the etch rate is too low ($< 1 \mu\text{m min}^{-1}$), the selectivity only amounts to ($\approx 25 : 1$), and the tilt angle of the sidewalls lies between 85° and 90° . In the semi-continuous DRIE process, etching and non-etching periods take turns, while passivation is performed continuously during the whole process. In this way, the advantages of the continuous and the cyclic DRIE (Bosch) process are combined. In addition, the so-called underetching effect (etching beyond the margin of the mask) is reduced significantly.

We used this AFL for nanofocusing in the hard x-ray nanoprobe instrument at beamline P06 of the synchrotron radiation source PETRA III at DESY in Hamburg, Germany.¹⁶ At P06, the nanoprobe instrument is located approximately 98 m from the undulator source. On the way to the instrument, the x-ray beam is monochromatized ($E = 20 \text{ keV}$) by a Si (111) double-crystal monochromator. In order to focus the x-rays in two dimensions, the AFL was combined with a conventional nanofocusing refractive x-ray lens^{4,17} ($N = 300$, $R = 12.4 \mu\text{m}$, $R_0 = 19 \mu\text{m}$) in a crossed geometry as shown in Figure 2. At the entrance of the nanoprobe, the aperture of the optic is defined by a pair of high-precision slits that were set to $10 \mu\text{m} \times 50 \mu\text{m}$ (horiz. $\times$ vert.). The beam was vertically focused by the nanofocusing lens (NFL) with a focal length of $f_{\text{NFL}} = 18.7 \text{ mm}$ and then horizontally by the adiabatically

FIG. 2. Schematic experimental setup.

focusing lens. Both lenses were adjusted such that they focus the x rays to a common focal plane about $z_f = f - H = 0.405$ mm behind the exit of the AFL. The overall transmission of the two focusing optics together was about 8% (cf. Table I), resulting in a focused flux of about $2 \times 10^7 \text{ s}^{-1}$.

We used ptychography to characterize the nanobeam created in this way.¹⁴ In this technique, an object is scanned through the focused x-ray nanobeam, recording a far-field diffraction pattern at each position of the scan.^{13,14} From these data, the object's complex transmission function and the complex wave field of the illumination in the object plane is reconstructed using iterative phase-retrieval techniques.^{18,19} The complex wave field in the object plane can be propagated numerically along the optical axis, yielding a faithful representation of the full caustic of the beam.²⁰ In this way, ptychography has revolutionized the characterization of x-ray nanofocusing optics and is by now well established.^{11,21}

We recorded a ptychogram using a NTT-AT resolution chart (model: ATN/XRESO-50HC) as a test object placed near the focal plane. It was raster scanned perpendicularly to the beam in 100×100 steps, covering an area of $2 \mu\text{m} \times 2 \mu\text{m}$. At each position of the scan, a far-field diffraction pattern was recorded by a Pilatus 300k photon counting pixel detector by DECTRIS (pixel size: $172 \mu\text{m}$) placed 2.22 m behind the sample (exposure time 0.5 s). From these data, both the complex incident wave field and the transmission function of the test object were reconstructed following Ref. 19.

By propagating the complex illumination function along the optical axis, the full caustic of the beam can be reconstructed.²⁰ Fig. 3(a) shows the vertical and Fig. 3(b) horizontal beam profile. The vertical beam profile [Fig. 3(a)] generated by the nanofocusing lens is nearly gaussian and free of aberrations. The minimal lateral beam size in the vertical direction is $47.3(2)$ nm (FWHM).

FIG. 3. Caustic of the nanofocused beam: amplitude of the wave field projected onto (a) the vertical and (b) horizontal plane. The dashed line in (a) and (b) depicts the sample plane, in which the ptychogram was recorded. The semi-transparent lines in (b) delineate the numerical aperture. (c) Intensity distribution around the focus [dashed area in (b)] projected onto the horizontal plane. Line (i) in (c) is the section with the highest peak intensity (focus), line (ii) is a section through the laterally smallest speckle.

A parabolic lens—both nanofocusing^{4,17} or adiabatically focusing¹²—has a truncated gaussian aperture function (transmission profile), damping the peripheral amplitudes compared to those on the optical axis. The FWHM size of the Airy disc d_t (diffraction limited focus) can be associated to an effective aperture D_{eff} and with it a numerical aperture NA by²²

$$d_t = \alpha \frac{\lambda f}{D_{\text{eff}}} = \alpha \frac{\lambda}{2NA}, \quad (1)$$

where λ is the wavelength of the x rays and α depends on the aperture function and lies in the interval of $0.75 \leq \alpha \leq 0.85$ for rectangular truncated gaussian apertures. The effective aperture D_{eff} of a truncated gaussian aperture is smaller than the geometric aperture.²² The amplitude of the wavefield $|\psi(D_{\text{eff}}/2)|$ at the edge of the effective aperture (in the principal plane) is

$$\gamma := |\psi(D_{\text{eff}}/2)|/|\psi(0)| = \exp \left[1 - \exp \left(-\frac{\mu L'}{2} \right) \right], \quad (2)$$

times smaller than the amplitude $|\psi(0)|$ on the optical axis. Here, μ is the mass attenuation coefficient of the lens material, and L' is the length of the lens without counting the lens material of thickness d_j at the apexes of the individual lenses (cf. Fig. 1). As the beam propagates to the focus, the ray with the relative amplitude γ as in Eq. (2) follows the numerical aperture except in the near-field of the focus.

As the amplitude of the complex wave field of the nanofocused beam is accessible by ptychography, we can follow the ray with the relative amplitude γ along the caustic and by that measure the numerical aperture NA associated with D_{eff} . From the data in Fig. 3(a) $NA_v = 0.48(1) \times 10^{-3}$ is obtained. From Eq. (1) follows a diffraction limit $d_t = 48.4(10)$ nm in good agreement with the measured beam size of $47.3(2)$ nm.

The horizontal focus that is generated by the AFL is shown in Figs. 3(b) and 3(c). Ideally, the beam would be similarly gaussian as in Fig. 3(a) but with a significantly higher numerical aperture. From the reconstruction of the caustic, it becomes apparent that the AFL suffers from aberrations, generating a far from ideal focus. The brightest speckle [Fig. 3(c)] can be considered the focus and lies in the plane marked by (i) in Fig. 3(c). The horizontal intensity profile in this plane is shown in Fig. 4(i) and shows a focal spot size of 18.4 nm (FWHM). About 65% of the radiation fall into this

FIG. 4. (i) Horizontal intensity distribution in the focal plane of the AFL [line (i) in Fig. 3(c)]. The central speckle has a lateral size of 18.4 nm (FWHM). (ii) horizontal intensity distribution at the section with the laterally smallest speckle [line (ii) in Fig. 3(c)]. The smallest speckle has a lateral size of 14.0 nm (FWHM).

focus (central speckle). The rest of the radiation is mainly concentrated in the two pronounced side maxima [Fig. 4(i)].

We can measure the numerical aperture by determining the divergence of the beam that is clearly visible in Fig. 3(b). As for the vertically focusing lens, the numerical aperture of the AFL is associated with the effective aperture [cf. Eq. (1)] and a relative lateral drop of the amplitude by a factor $\gamma = 0.45(1)$ [Eq. (2)]. The semi-transparent lines in Fig. 3(b) delineate the wedge, on which the amplitude of the wave field drops laterally by this factor γ . The opening half angle of this wedge is $1.75(7)$ mrad in good agreement with the nominal numerical aperture of $NA_h = 1.73 \times 10^{-3}$. The measured numerical aperture thus well exceeds the critical angle of total reflection $\theta_C = \sqrt{2\delta} = 1.55$ mrad ($\delta = 1.21 \times 10^{-6}$).

The numerical aperture of $NA_h = 1.73 \times 10^{-3}$ would ideally lead to a lateral focal spot size of 13.9 nm. This value was determined numerically by wave propagation through the ideal AFL. For the given truncated gaussian aperture $\alpha = 0.77$. In the present case, the AFL has significant aberrations and the paraxial and peripheral rays do not meet in a common focal plane. The focus lies in plane (i) [cf. Fig. 3(c)] slightly behind the plane (ii), where the peripheral rays cross the optical axis. Thus, it is not the full aperture that contributes to the focus making it effectively larger [cf. Fig. 4(i)]. In the plane (ii) in Fig. 3(c), amplitudes from larger aperture angles contribute to the interference pattern, generating laterally small speckles about 14.0(5) nm (FWHM) in size. This speckle size is nearly optimal and corresponds to a numerical aperture of $NA = 1.72(6) \times 10^{-3}$ in excellent agreement with that independently measured by the beam divergence. In the future and with improved fabrication techniques, these optics can generate nearly gaussian sub-10 nm x-ray beams for x-ray microscopy applications.

This work was supported by the German Ministry of Education and Research (BMBF) under Grant Nos. 05K10OD1 and 05K13OD4 and by VH-VI-403 of the Impuls- und Vernetzungsfonds (IVF) of the Helmholtz Association of German Research Centres. Beamtime at beamline P06 at PETRA III was granted within the user program of DESY, a member of the Helmholtz-Association.

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