Kinoform and saw-tooth X-ray refractive lenses
development at SSRF

J. Xu, G. Liu, Q. Huang, M. Liu, X. Zhou, H. Wu, N. Li, Y. Li, X. Xu, D. Liang, H. Jiang, L. Zhang, J. Wang, J. Cao and H. Lin

School of Electronic Information and Electrical Engineering, Shanghai Jiao Tong University, 800 Dongchuan Rd., Shanghai 200240, P.R. China
National Center for Protein Science, Shanghai Institutes of Biochemistry and Cell Biology, Chinese Academy of Sciences, 333 Haike Rd., Shanghai 201210, P.R. China
Shanghai Institute of Applied Physics, Chinese Academy of Sciences, 239 Zhang Heng Rd., Shanghai 201204, P.R. China

E-mail: linhe@sinap.ac.cn

ABSTRACT: Here we describe the effort made at Shanghai Synchrotron Radiation Facility (SSRF) to develop X-ray refractive lenses for real beamline applications. Fabrication process of silicon and germanium based kinoform lenses are optimized to increase the height-width aspect ratio and decrease the roughness. Saw-tooth lenses made with a new fabrication process based on dry etching are also described. Focusing effect for both kinoform and saw-tooth lenses are tested at Shanghai Synchrotron.

KEYWORDS: X-ray transport and focusing; Inspection with x-rays

1These authors contribute to the paper equally.
2Corresponding author.
1 Introduction

For the last two decades, X-ray refractive lenses have been developing very fast. Since the breakthrough idea of compound refractive lenses (CRL) [1], people have proposed various designs and fabrication techniques for different applications. These interesting designs include (but are not limited to) parabolic lenses [2], multi-prism lenses [3], single element kinoform lens [4, 5], compound kinoform lenses [6, 7], oval lens [8], and axicon lens [9]. The refractive lenses are used for imaging [10], focusing [11], collimating [12], interfering [13], and other interesting applications [14]. When describing the focusing limit of kinoform lenses, several theoretical frameworks are used, ranging from simple geometrical optics to dynamical diffraction theory and beam propagation methods [15]. Recently, remarkable theoretical work was carried out to find the ‘ideal’ shapes of lenses that are free of geometry aberration, based on Fermat’s principle for compound lenses [16]. Such analysis on geometry aberration was done before on short focal length saw-tooth [17]. These theoretical efforts provide guides for the lens fabrication when the goal is obtained sub-micrometer or nanometer focusing.

Fabrication techniques play a crucial role in making the X-ray refractive lenses [18]. These techniques include printing, semiconductor etching, LIGA (Lithographie, Galvanoformung, and Abformung), etc. Very recently people also tried ultrafast laser micromachining [19, 20] and 3D printing method [21]. These techniques remain to be further developed and will surely bring new surprises to scientist in the field of X-ray optics.

With the ongoing and upcoming constructions of synchrotron and free electron laser facilities in China, scientists in China have made some progress on X-ray refractive lenses research [22–25].

Here we describe the effort made at Shanghai Synchrotron Radiation Facility (SSRF) to develop X-ray refractive lenses based on planar semiconductor fabrication techniques, mainly focusing on the kinoform and the saw-tooth lenses for the real applications in the upcoming beamlines construction and future update of the synchrotron. The asymmetric character of source sizes and divergences between horizontal and vertical directions of most third generation synchrotron light sources makes...
the 1D optical device like kinoform and saw-tooth quite useful. Kinoform lenses will be good candidates for XPCS (X-ray Photon Correlation Spectroscopy) experiments [26], while the saw-tooth lenses will be useful for high energy X-ray micro-focusing [27] in the new beamlines at the SSRF.

2 Fabrication method and focus test

2.1 Kinoform lenses

The kinoform lens is an interesting X-ray optical component due to the high transmission rate it provides. In some sense, the absorption by the lens material is minimized when X-ray pass through the lens [4, 5]. Very high transmission (~90% theoretical transmission efficiencies) can therefore be reached [28]. The index of refraction for X-rays is

\[
n = 1 - \delta + i \times \beta, \tag{2.1}
\]

where \(\delta\) is related to the phase changes and \(\beta\) is related to the energy absorption in the materials. The focal length \(f\) can be expressed as

\[
f = R/\delta, \tag{2.2}
\]

where \(R\) is the lens radius of curvature at the lens apex. The typical value for \(\delta\) is of the order \(10^{-6}–10^{-7}\) for hard X-ray. It suggests the radius of curvature for a kinoform optics should be small enough to produce an acceptable X-ray focus length. The process for making single crystal silicon based planar kinoform lens is compatible with that of the existing CMOS fabricating techniques. Lens structures with very small radius of curvature (typical value of several micro-meters for photon energy ranging from 6 keV to 20 keV) are thus achievable.

In recent years, extensive research on the Si-based kinoform lens has been carried out [29–31]. Stein et al. fabricated the Si kinoform lens with a measured resolution of about 0.3 \(\mu\)m full width at half maximum (FWHM) at an energy of 11.3 keV [32]. Shastri et al. reported vertical focusing of 50–100 keV hard X-rays to 0.2–1.5 \(\mu\)m beam sizes at 0.25–2 m focal length by using a planar kinoform lens [7]. By introducing some special designed sacrificial structures at the periphery of target structures, Stohr et al. realized accurate control of the etch profile and fabricated the compound kinoform lens allowing focusing an energy of 17 keV X-ray into a spot with the size of 0.4 \(\mu\)m [33].

In this work, the research progress of kinoform lenses at SSRF is reported. The silicon planar kinoform lens was manufactured based on the electron-beam lithography (EBL) and the deep reactive-ion etching (DRIE) techniques. With the optimized etch process, the lens with the outmost sidewall thickness of 400 nm and etch depth of 30 \(\mu\)m was fabricated, with an aspect-ratio over 70:1. The measured focal size was 0.87 \(\mu\)m (FWHM from Lorentz fit) at the photon energy of 12 keV with a focal length of 8 cm. For high energy (50–100 keV) X-ray focusing application, the single crystal Germanium (Ge) based kinoform lens is preferred, because for the same photon energy and focal length, the Ge kinoform lens provides larger radius at the lens apex compared with the Si based one, and easier fabrication process [34]. The design specifications for Si-based and Ge-based kinoform lenses with different focus lengths and energies are listed in table 1.

| Parameter | Description | Specification |
|-----------|-------------|---------------|
| \(R\)     | Radius of curvature | 400 nm |
| \(f\)     | Designed focal length | 8 cm |
| \(m\)     | Order of the kinoform | 0–2 |

Parameter \(R\) is the radius of curvature at the lens apex, \(f\) is the designed focal length, and \(m\) is the order of the kinoform (\(m\times2\pi\) is the relative phase shift along each step length of the lens compared to the normal un-etched lens). The parameter \(m\) is crucial in kinoform design. The
Table 1. Design specifications for kinoform lens with different focus lengths and energies.

| Energy (keV) | Material | R (µm) | f (m) | m |
|--------------|----------|--------|-------|---|
| 12 Si        | 0.270    | 0.08   | 1     |
| 12 Si        | 3.370    | 1      | 4     |
| 50 Ge        | 1.177    | 3      | 1     |

Table 2. The optimized etching characteristics for the test line.

| Etch rate [nm/sec] | Etch bias [nm]/Sidewall roughness (µm) | Etch depth [µm] | Feature size [nm] | Aspect ratio |
|--------------------|----------------------------------------|-----------------|-------------------|-------------|
| 33.01 ~100         | 29.13 (p-v)                           | 30.20           | 421.1             | >70         |

The smallest $m$ (=1) minimizes the absorption effect [4, 5] and gives the smallest distortion of wave field from the lens edge diffraction effect [15]. However, the smaller the value of $m$, the smaller the lens feature is. As a result, the fabrication is even more challenging.

In order to simplify the fabrication process, the lens pattern was firstly defined on a layer of Chrome (Cr) by the EBL and the lift-off process. The Cr pattern was used as the hardmask during the Bosch™ process afterwards for the deep Si (or Ge) etching. The test line pattern, which was employed to find out the smallest line width, or critical dimension (CD) for a given structure height (30 µm in this work), has an etch profile as illustrated in figure 1. It was shown that for a line width of 421.1 nm, the etch depth of 30.20 µm, and the sidewall roughness (peak to valley distance of a wave) below 30 nm can be achieved. The CD line parameters resulted from the optimized etching were summarized in table 2.

The CD was factored in the kinoform design by adding a line bias of CD to the smallest line width in the lens. The optimized recipe was then applied for the kinoform lens etching. The obtained kinoform lens profile was illustrated in figure 2(a). The focusing test of the Si-based kinoform lens was performed at the U15 beamline at SSRF [35]. A Kohzu sample stage was used for lens positioning and alignment. The knife-scan method was used for measuring the focused beam profile. Detailed optical setup and measurement will be described elsewhere [36]. The measurement result is shown in figure 2(b). As illustrated in the curve, the focal size of 0.87 µm was obtained, which is not much bigger than the diffraction limit value of 0.589 µm considering the absorption and scattering effects in the real material (supplementary material containing the detailed calculation is attached at the end of this article). The gain calculated from the scan data is 6.0. The theoretical calculation showed that the outer steps of the lens contributes to the focusing, since the diffraction limited focal size from only the first step (the $h_1$ in the supplementary material) will give bigger focal size ($\approx$1.01 µm), even not taking the real absorption and scattering effects into accounts.

Ge-based kinoform lens is suitable for high energy applications. The design specification for Ge-based kinoform lens is listed in table 1. The fabrication result was observed using SEM, as photographed in figure 3. The focusing effect of the Ge kinoform will be tested in high energy beamline at SSRF later.
Figure 1. The etching profile of a 400 nm width test line pattern, the pattern was cleaved for cross sectional inspection. The measured width was 421 nm, the etch depth was 30 µm and the sidewall roughness was 29 nm.

Figure 2. (a) Tilted SEM image of silicon based kinoform lens. (b) Knife edge scan measurement for the Si kinoform lens focused beam at the photon energy of 12 keV. The focal size was 0.87 µm (Lorentz fit FWHM) by fitting (magenta line) the derivative intensity data (red circles), with the raw intensity data (blue line and dots) also plotted on the same figure.

2.2 Vertical saw-tooth refractive lens (vSRL)

Saw-tooth refractive lenses (SRL) have been used with success in high energy undulator beamlines [15]. Since the fabrication technique of SRL based on the anisotropic wet etching of silicon cannot be easily duplicated with success [16], a diamond saw was used for the fabrication of silicon SRL in the case when the fabrication precision can be somewhat relaxed [17]. Here we describe an alternative design based on a silicon dry etching technique, called vertical saw-tooth X-ray refractive lens (vSRL), as illustrated in figure 4. A conventional planar SRL is implemented on a silicon substrate using microfabrication techniques such as LPCVD (Low Pressure Chemical Vapor Deposition), high temperature thermal
oxidation, and wet etching [37, 38]. The processes are complex, and as a result uniform and high quality lens are difficult to produce. Since a complete lens is composed of two individual jaws, the connection and alignment of the two jaws can be challenging. The planar SRLs fabrication relied heavily on the anisotropic etching of silicon planes with different orientation in alkaline solution to form V-groove. This wet etching method can therefore be applied only on a very limited number of materials. Furthermore, every prism of silicon SRL has a fixed base angle, which limits its possibility on applicable SRL designs.

vSRL, however, has the two jaws manufactured at the same time. The structure of the whole lens was defined by the EBL and the DRIE processes. It has several distinct advantages over the conventional SRL manufacture process:

1. Unlike the conventional SRL, where it is necessary to align and link two jaws mechanically when focusing X-ray beam, the optical path of vSRL is already fixed and aligned at the pattern design stage, and is stable as a result. The possible displacement problem is therefore effectively avoided when in use.

2. vSRL eliminates the constraint of the fixed base angles of every prism. Lenses with various structure parameters can be designed as a lens array with the same substrate for different focus lengths and energies, which greatly improves the flexibility of the lens application.

3. The deep etching of silicon or Ge can be performed by using a common Bosch™ process or metal assisted chemical etch [39, 40]. Structures with high aspect ratios can also be obtained on PMMA by soft X-ray exposure method [24] and the LIGA process [41, 42]. These are all precision machining techniques with good reproducibility and high fidelity, and therefore are suitable for mass production.

The diagram of a vSRL is shown in figure 4(a), top view of which is illustrated in figure 4(b). The design specifications are listed in table 3 and the fabrication result is shown in figures 4(c) and 4(d).
Table 3. Design specifications for vSRL.

| Energy (keV) | Material | f (m) | H (µm) | y_g (µm) | D (µm) | N  |
|--------------|----------|-------|--------|----------|--------|----|
| 12           | Si       | 0.21  | 100    | 100      | 140    | 100|
| 12           | Si       | 0.08  | 70     | 70       | 90     | 200|

Figure 4. a) Diagram of a vertical saw-tooth refractive lens (vSRL); b) top view of a vSRL; c) SEM top-view image of a fabricated vSRL; d) tilted SEM image of a fabricated vSRL. This lens has an etching depth of about 75 µm.

The measurement on the focusing effect of vSRL was also performed on the U15 beamline at SSRF using the same experimental setup [35]. Detailed optical setup, measurement and discussions will be presented in another paper [36]. The etching depths of both of the two lenses can be increased further.

Figure 5 showed the tilted view of a fabricated half saw-tooth vSRL and an integrate vSRL, both of which have a focal length of 8 cm at the photon energy of 12 keV. Some etch defects in the sample surface induced by particle contaminants can be observed. These lenses have an etching depth of 175 µm. The side grooves are for easy alignment. Preliminary focal test showed a focal size around 3 µm with a gain around 7 for the integrate vSRL. Further experiments with different X-ray energies will be carried out [36].
3 Conclusions

The efforts for making the X-ray refractive lenses (kinoform and saw-tooth) at SSRF are reported. By using the microfabrication techniques, the Si and Ge kinoform lens and saw-tooth lens were manufactured. The focus test results showed that at the energy 12 keV, a focal size of 0.87 \( \mu \text{m} \) and optical gain of 6.0 were achieved for the Si refractive lens with kinoform structure. A novel vertical saw-tooth X-ray refractive lens (vSRL) was also proposed and fabricated. Future work involves designing compound kinoform to further reduce the focal size, developing techniques to make 2D kinoform lens, and perfecting the saw-tooth structure by taking geometrical aberration correction into account. Fabrication techniques based on other materials (e.g. diamond) suitable for making refractive lenses will also be tried.

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A Supplementary material

Theoretically, the diffraction limited focal size should be:

\[
D = \frac{\lambda}{2 \cdot n \cdot \sin(\theta)} \quad (A.1)
\]

In our case, \( \lambda = 1.03 \text{Å}, n \approx 1, f = 8.0 \text{ cm}, \) half angle \( \theta = \tan^{-1} \left( \frac{h}{f} \right), h \) is the half of the aperture size.
a. The incoming X-ray aperture set to be 14.0 µm, the calculated $D=0.589$ µm.

b. In our design, the phase shifting of the steps is of the maximum $1 * 2\pi (m=1)$. The height of the inner steps are $h_1 = 4.065$ µm, $h_2 = 1.683$ µm, $h_3 = 1.290$ µm.

The corresponding diffraction limited focal size would be 1.01 µm (from $h_1$ only), 0.717 µm (from $h_1 + h_2$) and 0.583 µm (from $h_1 + h_2 + h_3$).

Obviously there has to be contribution from other outer steps to the total effective aperture rather than just the innermost step.

Given the step length $L=30.660$ µm and the absorption length of the silicon at 12kev being 215.8 µm, the defect scattering probably gives more broadening effect than absorption.

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