Studies on phase Ronchi gratings and the fabrication of soft x-ray condenser zone plates

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ABSTRACT

Ronchi gratings are widely used as beam splitters in interferometry. The phase Ronchi gratings are fabricated in K9 (or BK7) substrates using a photolithographic process and ion beam etching process. The geometry of the gratings was determined within the approximations of the scalar diffraction theory to minimize their diffraction intensities in 0 and 2 orders at 514.5nm. Their diffractive characteristics are measured using Ar+ laser at 514.5 and 496.5nm. The phase Ronchi gratings exhibit the diffraction intensity in 0 or 2 order to be three orders of magnitude less than that in 1 order. Soft x-ray condenser zone plates are one of the most essential condensing and dispersing elements in the field of soft x-ray microscopy. The zone-plates are used at 3.2nm, whose diameters are 2.8mm. The widths of their outermost zone are 647nm. The zone plate is made of Au. Self-supporting zone plates are fabricated by holography-ion beam etching process to obtain x-ray lithography mask, the x-ray lithography, photolithography and micro electroplating process were used to obtain their self-supporting structure. The self-supporting zone plates are free of membrane absorption and degradation.

Keywords: phase Ronchi grating, soft x-ray, condenser zone plate, x-ray lithography, self-supporting

1. PHASE RONCHI GRATINGS

1.1. Introduction

Ronchi gratings\(^1\) have low spatial frequencies and long periods, whose periods are between 2 and 100 \(\mu\)m usually\(^2\). At present, Ronchi gratings are still used in many kinds of interferometers to measure the long focal length\(^3\), the linear thermal expansion coefficient of the material of metallic bars\(^4\), displacement\(^5\), and so on.

One dimension and orthogonal phase Ronchi gratings were fabricated using a lithography and ion beam etching process by optical element group at National Synchrotron Radiation Laboratory (NSRL), USTC. They were used for Fourier optical transform and experimental mechanics\(^6,7\). In this paper, we report recent research progress of phase Ronchi gratings at NSRL. First we analyse the diffraction characteristic and fabrication errors within the approximations of the scalar diffraction theory. Then the fabrication process of Ronchi gratings is described and the experimental results are shown. Finally we characterize the performance of the gratings designed for normal incidence operation at 514.5nm using Ar\(^+\) laser.

1.2. Ronchi grating theory

In this section, we briefly outline the theory of phase Ronchi gratings. A schematic diagram of a phase Ronchi grating is shown in Figure 1. The height of each grating line or the depth of each grating groove is \(h\). The width of the grating groove is \(g\). The width of the grating line is \(l\).

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line is $a$ and the width of the grating groove is $b$. Then the period of the grating is $d = a + b$. The duty cycle of the grating is $r = a/d$.

When light is incident normal to the Ronchi grating, the grating equation is $d \sin \theta_m = m\lambda$, where $\theta_m$ is the angle of diffraction for the $m$th order, $\lambda$ is wavelength. And a relative phase delay $\Phi_0$ is introduced, where

$$\Phi_0 = \frac{2\pi(n-1)h}{\lambda}.$$ 

Because the period of each Ronchi grating is very much greater than the height of grating line, their diffraction characterization can be estimated within the approximations of the scalar diffraction theory. So, the complex amplitude in various diffraction orders are given by the following equation

$$U_m(\theta) \propto (1 + e^{i\Phi_0} e^{i\beta}) \sin \alpha \sin(N\beta) \sin \beta$$

(1)

where $\beta = \frac{\pi l \sin \theta}{\lambda}$, $\alpha = \frac{\pi a \sin \theta}{\lambda} = \frac{\pi (r d) \sin \theta}{\lambda}$, $N$ is the slit number of a grating and approaches infinite.

$$U_m(\theta) \propto (1 + e^{i\frac{2\pi(n-1)h}{\lambda}} e^{im\pi}) \cdot \sin(m) \cdot N$$

(2)

then the diffraction intensities in various orders are given by the following equation

$$I_m(\theta) \propto |U_m(\theta)|^2 = \left(2 + 2 \cos \left(\frac{2\pi(n-1)h}{\lambda} + m\pi\right)\right) \sin^2(rm) N^2$$

(3)

From equation (3) we know that the intensity of each phase Ronchi grating diffraction order are determined by their geometry and independent of grating period. $\eta_m$ denotes the relative diffraction efficiency of the $m$th order and defined as the ratio of the diffraction intensity of $m$th order to that of the 1st order.

$$\eta_m = \frac{I_m}{I_1} = \frac{\left[2 + 2 \cos \left(\frac{2\pi(n-1)h}{\lambda} + m\pi\right)\right] \sin^2(rm)}{\left[2 - 2 \cos \left(\frac{2\pi(n-1)h}{\lambda}\right)\right] \sin^2(r)}$$

(4)

We design a phase Ronchi grating to vanish or reduce the diffraction intensity of the 0th and 2nd orders. From equation (3), we obtained that

if $m = 0$, $h = \lambda[2(n-1)]$ (or $\Phi_0 = \pi$) and $r$ is optional, $I_0 = 0$, and

if $m = 2$, $h$ is optional and $r = 0.5$, $I_2 = 0$.

From above results, we determine the geometry of this kind of Ronchi grating is that its groove depth is $h_0 = \lambda/[2(n-1)]$, corresponding to the phase delay is $\pi$ and its duty cycle $r_0 = 0.5$. The grating is operated at 514.5nm and $\lambda_0 = 514.5nm$.

We simulate the relative diffraction efficiencies of the 0th and 2nd orders according to equation (4). The geometry of grating is near its perfect parameters $(h_0, r_0)$. The refractive index of glass K9 is 1.5201. The results of the simulation are shown in figure 2.

From figure 2(a), we know that if the groove depth $h$ is $h_0$, $\eta_0$ is independent of grating duty cycle from equation (4) and the diffraction of the 0th order vanish with optional $r$. If the duty cycle of each grating with the same groove depth is not exactly 0.5, then the $\eta_0$ will increase with the increase of its duty cycle.

From figure 2(b), we know that if the duty cycle is 0.5, the relative diffraction efficiencies of order even are independent of the depths of gratings including that the diffraction of the 2nd order vanish. $\Delta r$ denotes the deviation of ideal duty
cycle \( r_0 = 0.5 \) and the fabricated duty cycle \( r, \Delta r = r - r_0 \). For phase Ronchi gratings with same groove depths, the \( \eta_2 \) tends to vanish with the reduction of \( \Delta r \).

![Graph](image)

Fig. 2. Relative diffraction efficiencies of the 0th (a) and 2nd (b) orders of gratings with different duty cycles as a function of groove depths at 514.5 nm.

### 1.3. Ronchi grating sensitivity to fabrication errors

In this section we discuss the sensitivity of the RG efficiency to various fabrication parameters according to the Ronchi grating theory analysis above. The Ronchi grating profile has to be fabricated using a lithography process with a dry etch into K9(or BK7) glass. The fabrication errors consist of errors in the etch depths and duty cycles. Although the theoretical calculations above predict the 0th and 2nd orders diffraction efficiencies would be nearly zero, but the inevitable fabrication errors in a practical application will raise the 0th and 2nd orders diffraction light more or less.

![Graph](image)

Fig. 3. A contour map of the relative efficiency of the 0th (a) and the 2nd (b) orders of a Ronchi grating as a function of the etch depth error and the duty cycle errors.

Figure 3(a) and 3(b) display the variation of the relative diffraction efficiencies in the 0th and 2nd orders with the etch depth and duty cycle errors in the fabrication at 514.5 nm.

From figure 3(a), we know that to keep the \( \eta_0 \) be less than 1%, the greater the grating duty cycle is, the less the permissible etched depth range is. To keep \( \eta_0 \) less than 1%, when the grating duty cycle is 0.40, its depth deviation is less than \( \pm 7 \) nm and when the grating duty cycle is 0.60, its depth deviation is less than \( \pm 5 \) nm.

From figure 3(b), we know that if the etched depth deviation between the ideal and practical depths is less than \( \pm 25 \) nm and the deviation of duty cycle is less than \( \pm 0.1 \), the diffraction of order 2 can be controlled very less.

We know the variation tendency of diffraction efficiencies of a phase Ronchi grating as a function of its geometry according the simulations and analyses above. In practical process, many factors, such as material, fabrication, even environment and so on can have effect on the characteristic of gratings. By our experience, it is not easy as the analyses...
above to fabricate a phase Ronchi grating whose $\eta_0$ and $\eta_2$ are less than 1‰. The precision control of the fabrication process is very vital.

1.4. Fabrication of Ronchi gratings

We fabricated Ronchi gratings using a lithographic progress and ion beam etching process. The Ronchi grating fabrication method is schematically illustrated in Figure 4. The Ronchi grating substrate is first spin-coated with a layer of photoresist. The photoresist layer is then exposed by UV light through a photomask. The exposed photoresist is developed away and the substrate in these areas is removed using an ion beam etching. After lithography and development process, a photoresist grating on K9 substrate is obtained as etch mask. This photoresis grating is subsequently etched by ion beam. Through the above process, a phase Ronchi grating is fabricated.

![Schematic illustration of the fabrication of a Ronchi grating by lithographic method](image)

Figure 5 illustrates the micrograph of a photoresist grating after UV exposure and development. Some short breaking photoresist lines are shown in figure 5(a) caused by edge diffraction in the exposure. It happens when the mask and the substrate covered with photoresist are not contacted closely. Figure 5(b) shows a good photoresist grating whose duty cycle is almost 0.5, which is suitable to the subsequent ion beam etch. The etch depth is controlled by the time duration of the etch. The etch rate of glass K9 varies from 19.8 to 21.3nm in our experiment though we control the same operation parameter in each etch process. We tried to achieve the desired etch depths of 490nm by first etching to 80% of the desired value, measuring the step height in a few selected places on the substrate, and completing the remaining etch.

To estimate the etch depth of K9 substrate, we combine the depth measurement by alpha-step 500 Surface Profiler with the relative diffraction efficiency measurement. Figure 6 illustrates the relative diffraction efficiency of the 0th order (a) and 2nd order (b) at 514.5nm and 496.5nm. The geometry of gratings is near their designed parameters. If the etch depth is a little deeper, the relative diffraction efficiency of the 0th order at 496.5nm is more than that at 514.5nm. If the etch depth is a little shallower, the relative diffraction efficiency of the 0th order at 514.5nm is more than that at 496.5nm. Figure 7 illustrates a SEM photograph of a phase Ronchi grating. It is fabricated on K9/BK7 substrate.
1.5. Diffraction performance

The diffraction performance of the Ronchi grating was characterized by measuring the 0th and 2nd transmission efficiencies at 514.5 and 496.5 nm of Ar\textsuperscript{+} laser. The Ronchi grating is worked at 514.5nm. Its diffraction intensity at 496.5nm is referenced to the fabrication of the grating. The Ar\textsuperscript{+} laser is vertical incident upon the Ronchi grating after reflected by an Al mirror which is applied to change the optical path and reduce the power of the laser in some degree. The diffraction intensity of each order is collected by one power meter at different positions.
In the grating fabrication, we combined the measurement of etch depth by alpha-step 500 Surface Profiler with the measurement of grating diffraction intensity in the 0th and 1st order to determine the end point of the etch commonly. The measurement results are shown in table 1. From the results we know that their diffraction intensities in 0th or 2nd order is three orders of magnitude less than the diffraction intensity in the 1st order.

<table>
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<tr>
<th>No.</th>
<th>Wavelength (nm)</th>
<th>The relative diffraction efficiency of the 0th order (%)</th>
<th>The relative diffraction efficiency of the 2nd order (%)</th>
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<td></td>
<td>496.5</td>
<td>16</td>
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</tr>
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1.6. Conclusions
In summary, we designed and fabricated phase Ronchi gratings using a photolithography and ion beam etching process. Their diffraction intensities in the 0th and 2nd orders are three orders of magnitude less than the diffraction intensity in the 1st order. They have been applied in interferometer to measure the deformation of the great buildings.

2. THE FABRICATION OF SOFT X-RAY CONDENSER ZONE PLATES

2.1. Introduction
Soft x-ray condenser zone plates (CZPs) play a key role as a focusing and dispersion element in soft x-ray microscopy operated in many synchrotron radiation facilities. In National Synchrotron Radiation Laboratory, Hefei, China, CZPs with substrate were fabricated and used at soft x-ray microscopy experiment station. Compared with CZPs with substrate, self-supporting zone plates are free of membrane absorption and degradation. In this section, we briefly introduce our recent progress in fabrication of free-standing CZPs.

2.2. Fabrication and results
The fabrication of free-standing CZPs contains two steps. The first step is to fabricate a CZP with substrate as a free-standing CZP mask using a holography-ion beam etch process as shown in figure 8. The optical beam splitter used in holography optical layout was designed by Thime. The second step is to fabricate a free-standing CZP with synchrotron radiation x-ray lithography, lithography and microelectroplating process as illustrated in figure 9. The first step is the base of all the process and is time-consuming. We have designed the self-supporting structure of CZPs. Figure 8 is the micrograph of a CZP with substrate. The self-supporting structure was designed and fabricated in Au substrate to try the lithography and microelectroplating process. This experiment results is shown in figure 9.
Fig. 8. Schematic illustration of the various steps in the fabrication of a CZP with substrate using holography-ion beam etch process:

(a) Exposure

(b) Development

(c) Ion Beam Etch and Removal of Remaining Photoresist

(d) Pasting the support ring and removal glass substrate using HF solution

Fig. 9. Schematic illustration of the various steps in the fabrication of a self-supporting CZP using a SR lithography-ion beam etching and lithography-microelectroplating process:

(a) x-ray lithography-ion beam etch for zone plate pattern

(b) Lithography for self-supporting structure

(c) Gold microelectroplating for self-supporting structure

(d) Removal of the glass substrate and zone plate mount
2.3. Conclusions
A synchrotron radiation lithography, ion beam etching, lithography and microelectroplating process proofs feasible to fabricate free-standing CZPs. The main fabrication steps were performed successfully. Our next work is to fabricate free-standing CZPs. Our future work will focus on the fabrication of soft x-ray phase zone plate with finer zones to improve their efficiency and optical resolution.

3. CONCLUSIONS
Phase Ronchi gratings were fabricated successfully using a lithography-ion beam etch process. Their diffraction intensities in the $0^{th}$ and $2^{nd}$ orders are three orders of magnitude less than the diffraction intensity in the $1^{st}$ order. The fabrication of soft x-ray self-supporting CZPs was explored. Based on these experiments, we will extend our fabrication process to some new kinds of high-efficiency zone plates with finer zones.

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