Fabrication of fused-silica sub-micron gratings with high aspect ratio by transfer holographic resist masks with ICP dry etching

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Sub-micron gratings have novel diffraction properties, including high-efficiency and polarization-dependent diffraction. The detailed fabrication process of fused-silica sub-micron gratings especially the grating mask was described, which can be fabricated using holographic interference recording and inductively coupled plasma (ICP) dry etching technology. Deep-etched fused-silica sub-micron gratings were obtained for period 890 nm with different depths, whose aspect ratios can reach the maximum 6.7 for an etched depth of 3.0 μm. It demonstrates that the holographic recording together ICP etching is an effective method for fabricating fused-silica gratings, which are useful elements in a variety of optical systems.

1. Introduction

Diffraction gratings are widely used in numerous optical information processing systems. With the development of microfabrication, a grating with higher density seems to show more and more interesting and useful optical properties [1-3]. Especially, fused silica is an ideal grating material with high optical quality: high damage threshold, stable performance and wide transmitting spectrum. So, it is important to etch high-density gratings in fused silica as novel diffractive optical elements. A few fused-silica transmission gratings with high efficiency have been fabricated for UV high-power laser [4], chirped-pulse amplification [5], dense wavelength division multiplexing [6], wideband polarizer [7], and beam splitter [8].

The fabrication of a fused-silica grating mainly involves forming a grating mask and etching in fused silica. Laser direct writing, electron beam and holography technology can be used to obtain grating patterns. However, laser direct writing system is suitable for fabricating low-density grating. Although sub-micron grating patterns can be obtained by electron beam writing system easily, the grating area needs to be improved further. Generally speaking, holographic interference can generate sub-micron grating patterns with large area effectively. The etching process is also an important step to etch a sub-micron grating in fused silica. Although conventional wet chemical etching is cheap and fast, it is difficult to obtain a rectangular high-density grating with vertical sidewalls because of undercut effect. For it's high etching rate and controllable energies of bombarding ions,

the ICP dry etching have more merits than other dry etching methods such as reactive ion etching, ion milling, and electron cyclotron resonance [9]. However, it is still a difficult problem to increase the grating density and etched depth due to rigorous restrictions for grating masks and the polymer deposition produced during the etching process. Fabrication conditions for such fused-silica gratings need to be further investigated.

In this paper, we described the fabrication process of sub-micron deep-etched fused-silica gratings. Especially, fabrication techniques of grating masks were discussed in detail. Fused-silica gratings with a period of 890 nm and aspect ratio (the ratio of grating depth to the ridge width) approaching 6.7 can be obtained using holographic interference recording and ICP dry etching technology.

2. Experimental set-up

Fig. 1 shows the process flow for fabrication of a deep-etched sub-micron fused-silica grating, which includes: (1) depositing a chromium layer on the dry and clean fused-silica substrate; (2) coating a photoresist (PR) film on the chromium layer; (3) holographic exposure and developing to generate a photoresist sub-micron grating; (4) transferring the grating pattern to the chromium layer by dechromisated solution; (5) etching the sub-micron grating in fused silica with the ICP facility; (6) cleaning the remaining grating mask by chemical solution. The photoresist (Shipley, Model s1805, USA) may be diluted for recording high-density gratings.
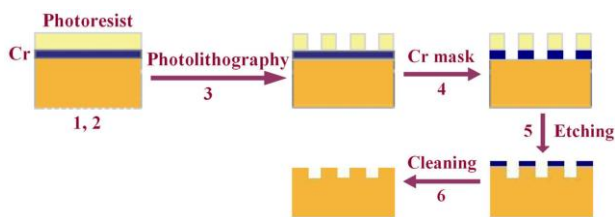
Fig. 1. (Color online) The process flow for fabricating fused-silica sub-micron gratings.

In experiments, the fused-silica substrate size is 18×18 mm^2. Thicknesses of fused-silica substrate, chromium layer are 1.67 mm, 145 nm, respectively. Photoresist film diluted with 1:2 is spin coated at the speed of 3000 rpm. Dechromisated solution can be confected with $Ce(NH_4)_2(NO3)_6$ 200 g, 98% CH_3COOH 35 ml, and deioned water 1000 ml.

Fig. 2 shows the experimental set-up for recording holographic gratings. In order to obtain a sub-micron grating with large area, the collimating lens is designed with diameter ϕ 100 mm. The beam from a He-Cd laser (λ = 441.6 nm) is divided into two. After expanded and collimated, two plane waves interfere to form holographic grating pattern. The grating period d can be described as $d = \lambda /2/\sin\theta$, where θ is the half of angles between two recording beams. With the increase of the angle θ, higher-densities photoresist sub-micron gratings can be obtained. By chemical solution, recorded photoresist sub-micron grating patterns can be transferred to the chromium layer to form a metal grating mask for deep-etching.
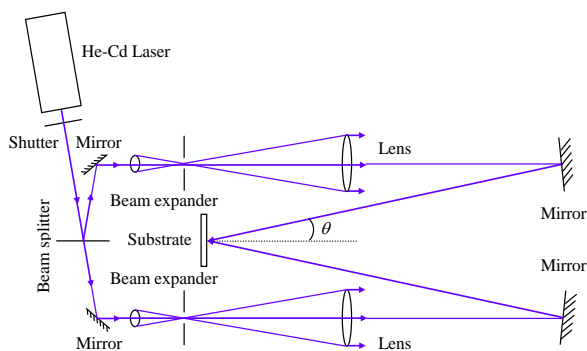

Fig. 2. (Color online) The schematic diagram of the experimental set-up for recording holographic sub-micron gratings.

Fig. 3 shows schematic diagrams of sub-micron photoresist grating patterns with different developing time. As shown in Fig. 3 (a), it is important to control the time of exposure and developing appropriately in order to obtain a homogeneous grating pattern mask, which plays a primary role for etching in fused silica to form a sub-micron grating with good quality. In Fig. 3 (b), if the developing time is insufficient, photoresist will still exist on the chromium layer for some grating grooves. The

dechromisated solution can't reach the chromium layer for reaction to form a chromium metal grating mask. On the contrary, with excessive developing time, there will be little even no photoresist for some grating ridges, which will lead to an inhomogeneous chromium grating mask or undesired duty cycle (the ratio of the grating ridge width to period), as shown in Fig. 3 (c). In order to obtain ideal grating pattern masks, diffraction efficiency should reach the maximum and uniformity when developing. In experiments, angle between two recording beams is 28.73° for a grating period of 890 nm. Proper exposure and developing time are 110 s and 9 s, respectively. The He-Cd laser power is 28.8 mW. Exposure dose and power density are 40.34 mJ/cm^2 and 0.183 mW/cm^2, respectively.
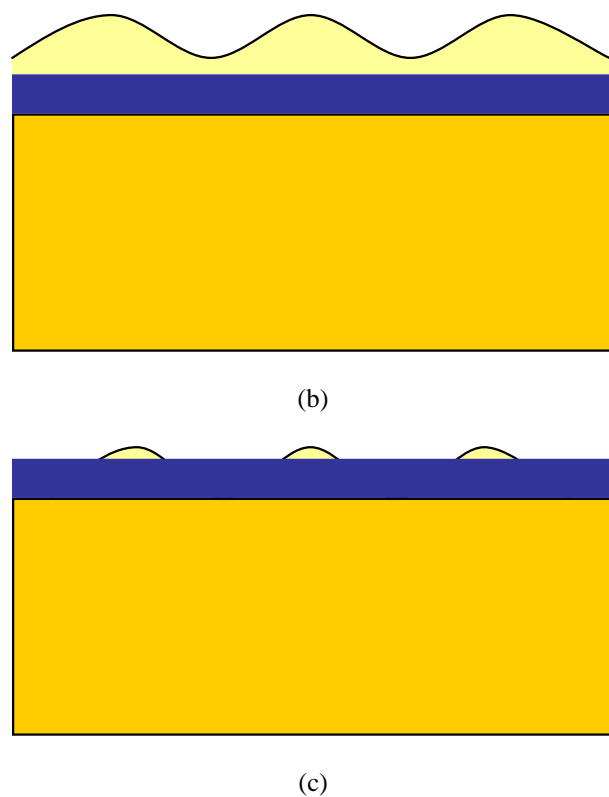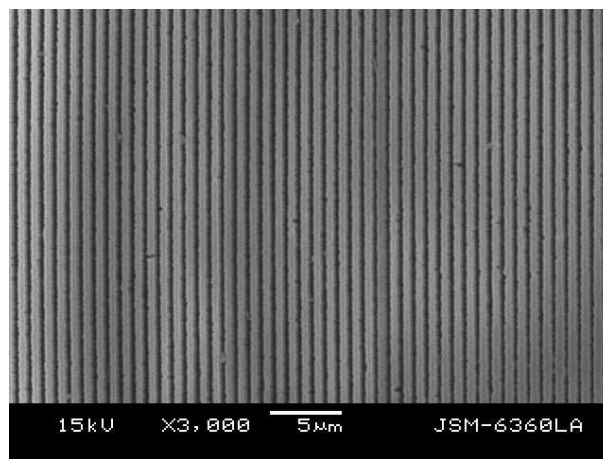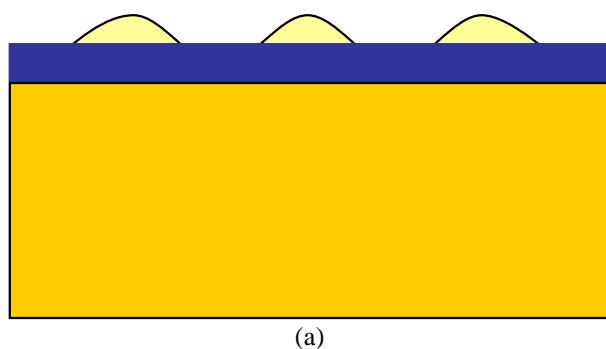

(b)

(c)

Fig. 3. (Color online) Photoresist sub-micron grating patterns with (a) appropriate, (b) insufficient, and (c) excessive developing time.

After forming a chromium grating patterns, the fused-silica substrate with the grating mask can be put in to the ICP facility for etching to achieve a fused-silica sub-micron grating. In the ICP system, gases flow rates are CHF_3 200 SCCM, Ar 20 SCCM, and O_2 5 SCCM. CHF_3 is the main etching gas to produce most of the ions and erosive neutrals. Ar can help to keep the plasma stable. O_2 is used to decompose the polymer to prevent polymer deposition during etching process. High-density plasma is generated by one radio frequency induction source RF_1. And the other source RF_2 can introduce a self-bias voltage in order that the ion can bombard the sample in direction
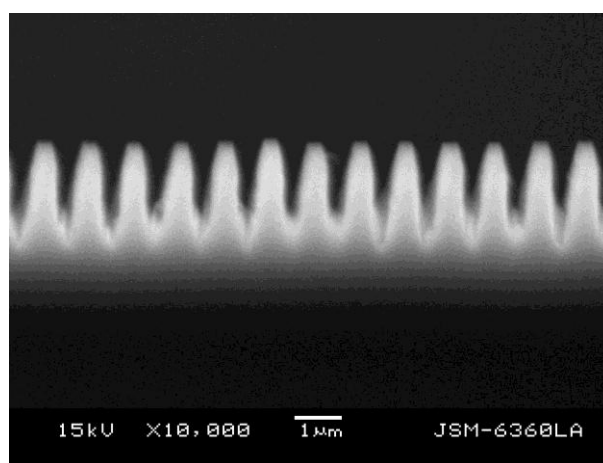
toward the ICP facility substrate, by which energies of ions can be adjusted independently to control the etching rate. In experiment, radio frequency sources of RF_1 and RF_2 are 600 W and 300 W, respectively. The intensity of pressure in the etching chamber is 2.0 Pa. The measured self-bias voltage is 500 V. The etching rate of fused-silica is 0.45 μm/10 mins.
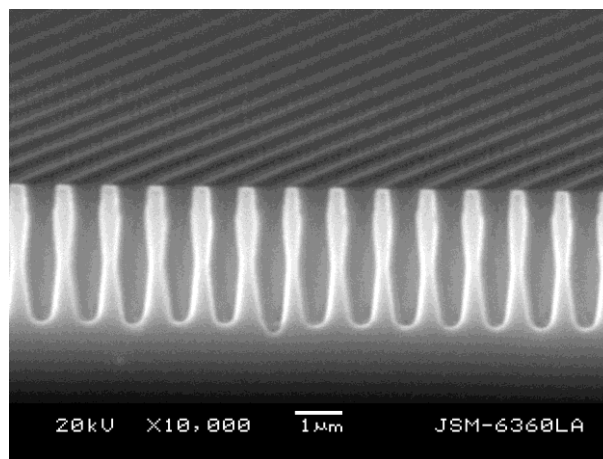
3. Fabrication results

Using holographic technology and dry etching of ICP, sub-micron gratings can be etched in fused silica with good quality. For the case of a Gaussian beam, the intensity distribution must be considered in the exposure process, which will affect the uniformity of the grating to some extent. Fig. 4 shows fabricated deep-etched fused-silica sub-micron grating with different etched depths for period 890 nm. The aspect ratio is 4.9 for depth 2.2 μm and 6.7 for depth 3.0 μm. One can see that the grating has the rather curved side wall and deep depth from the scanning electron micrograph. For deep etching, lateral etching may affect the grating groove shapes that deviate from perfect rectangles. The surface roughness of gratings is mainly determined by homogeneity of generated high-density plasma. The etched grating surfaces are rather even. It indicates that sub-micron gratings can be achieved with large areas homogeneously. ICP dry etching method is especially suitable for deep-etching of high-density gratings. For wet etching, the grating surface may be wavy for small grating constant due to low frequency noise. It is still a novel method to fabricated high-density fused-silica gratings. Illuminated by an incident wavelength of 1550 nm for TM polarization near Bragg angles, efficiencies in the 0th order for the etched depth 2.2 μm and 3.0 μm can approach 87.01% and 87.33%, respectively. Such fabricated fused-silica gratings may be useful polarization-selective optical elements for TM polarization.

(a)

(a)

(b)

(c)

Fig. 4. Scanning electron micrographs of fabricated fused-silica sub-micron gratings with period 890 nm: (a) Top view, (b) cross section with depth 2.2 μm, (c) depth 3.0 μm.

4. Conclusions

Photoresist gratings were obtained by holographic technology with exposure dose 40.34 mJ/cm^2 and power

density 0.183 mW/cm^2 for period 890 nm. Recorded gratings were etched in fused silica with aspect ratios reaching 6.7 using ICP etching with radio frequency sources of RF_1 600W, RF_2 300W, and self-bias voltage 500 V. Deep-etched high-density sub-micron gratings can show interesting diffraction properties for novel optical elements. Also, because of the excellent optical quality of fused silica, the fabricated sub-micron gratings etched in the fused silica can be widely used for various optical information processing systems.

Acknowledgements

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