Manufacturing High-efficiency, High Damage Threshold Diffraction Gratings with Lift-off Processing*

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Abstract

High-efficiency, high damage threshold diffraction gratings fabricated out of multilayers of dielectric materials are needed for the application of chirped-pulse amplification (CPA) in the Petawatt Laser Project. The underlying multilayers are deposited onto a flat substrate by standard e-beam evaporation. The grating structures themselves, however, can either be etched into a plane layer or deposited between a photoresist grating mask which is subsequently lifted off. The latter procedure, although more easily applied to large apertures, requires high-aspect ratio, vertical sidewall photoresist grating masks with, preferably, an overhanging structure to facilitate liftoff. By varying factors in each processing step, sample gratings exhibiting these characteristics were fabricated.

Using a high-contrast profile photoresist (AZ7710), we have been able to create grating masks with both vertical sidewalls and high-aspect ratios (>4.5). We have also had some encouraging preliminary results in making overhanging structures by including a pre-development chlorobenzene soak in the processing steps. Once these samples are deposited with an oxide and the grating masks lifted off to create the final grating, a more definitive processing method can be developed based on the results.

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1. Introduction

The application of chirped pulse amplification (CPA) (Figure 1) in high power laser systems has required the manufacture of large aperture, high-efficiency, high damage threshold diffraction gratings[1]. At the Lawrence Livermore National Laboratory, we have attempted to fabricate such gratings for a 1000-TW (petawatt) laser system. To achieve such high power with CPA, a laser pulse is often diffracted four times in a typical double-pass compressor. Thus, the diffraction gratings must exhibit both a high efficiency in the m = - 1 order and a high damage threshold to maximize the energy per area that can be tolerated.

Presently, CPA lasers use gold coated photoresist diffraction gratings[2]. However, higher efficiency and higher damage threshold gratings can be made of multilayers of dielectric materials since these do not absorb radiation as does gold[3]. The actual grating structures are often etched into the top layer of a multilayer stack of alternating high and low index of refraction. Another possible method involves creating a photoresist grating mask on top of the multilayer stack and depositing the actual grating material between the structures. The mask is then lifted off by dissolution, leaving ridges of dielectric material in between. An illustration of this process will appear in the next section. This paper presents in detail the processing steps involved in the latter procedure and presents early results of our fabrication experiments.

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[1] M. D. Perry and G. Mourou, "High-efficiency multilayer dielectric diffraction gratings."
[2] R. D. Boyd *et al.*, "High-efficiency metallic diffraction gratings for laser applications."
[3] M. D. Perry *et al.*, "High-efficiency multilayer dielectric diffraction gratings."

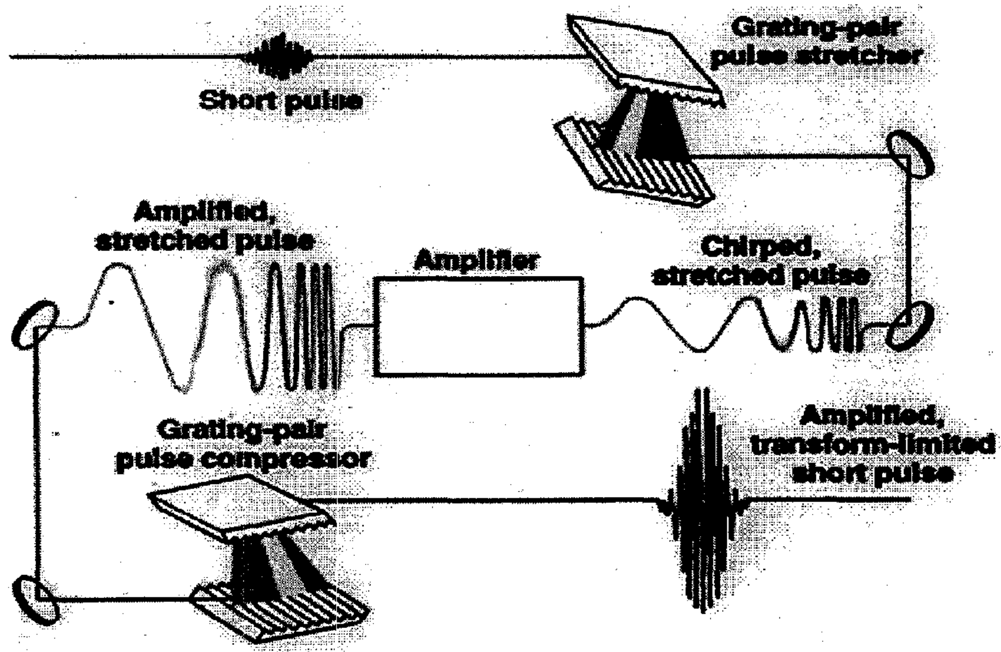

Figure 1. Chirped pulse amplification (CPA) concept

We have been able to construct grating masks that facilitate lift-off. Tall structures with high aspect ratios are preferred for the lift-off process. Structures with an overhang are also favored for they can easily be lifted off to maintain an ideal grating. We show that by using a high-contrast profile resist and including some preprocessing steps, adequate grating masks may be constructed, and the lift-off process is indeed feasible.

2. Procedure

The procedure we use to manufacture high-efficiency diffraction gratings with lift-off processing entails the following steps:

1. Substrate preparation
2. Exposure
3. Preprocessing
4. Development
5. Oxide deposition and lift-off

Variations in processing techniques can affect the shape and overall effectiveness of the grating mask, and thus, influence the efficiency of the final grating. By adjusting different factors in each processing step, we were able to develop an optimal procedure and create potential grating masks.

2.1 Substrate preparation

The actual diffraction gratings we will use for CPA are created on large, optically flat 80 cm diameter glass substrates layered with dielectric material. However, to analyze the grating mask profiles, microscope slides, 50 × 77 mm, are used. They are first scrubbed in a 3% NaOH detergent solution and later subject to ultrasonic cleaning in the same solution for 30 minutes. After rinsed in distilled water and dried with dry-nitrogen, the substrates are coated with a nominally 1 mm thick AZ7710-series photoresist layer. For small substrates (<12 cm width) the ARC and photoresist layers are applied by spin coating. For larger substrates (≥12 cm width) a meniscus coater[4] is used. When spin coating, photoresist thickness can be controlled either by the solute concentration in the resist solution or by the angular speed of rotation. When applying the resist with a meniscus coater, thickness is again dependent on concentration and on the speed at which the substrate traverses laterally. The photoresist- coated substrates are baked at 70° for 20 minutes, and finally, the back sides are painted with a peelable, black varnish to prevent back reflections during exposure.

2.2 Exposure

The surface relief pattern is holographically produced in the photoresist by exposing the samples to the intersection of two highly collimated laser beams of ultraviolet radiation (Figure 2). The interference pattern is produced by an equal-path, fringe-stabilized interferometer connected in an electronic feedback loop. A lateral-effect photodiode detects the movement of coarse fringes constructed by recombining portions of each laser beam. As a result, an error

[4] W. A. Bookless, ed., "Meniscus Coating."

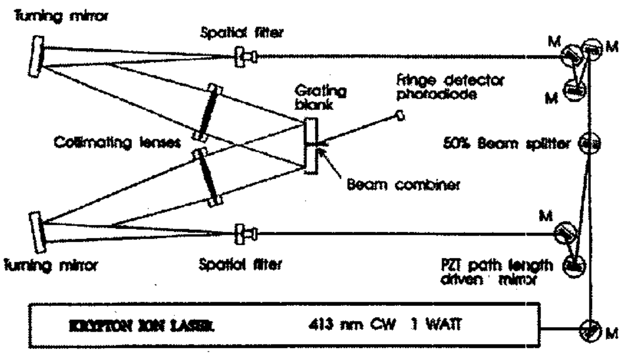

Figure 2. Holographic exposure setup

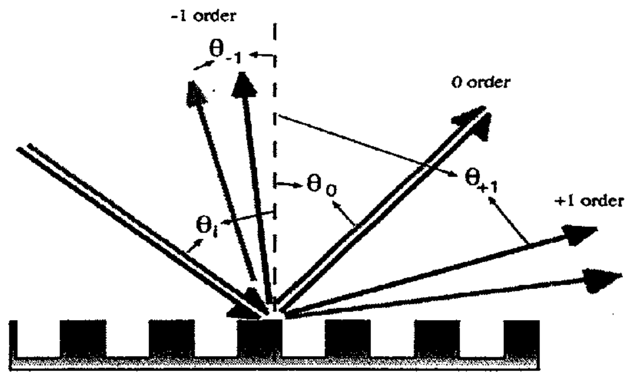

Figure 3. Diffraction equation concept

signal is generated, changing the path length of one arm of the beam. This compensates for any slight vibrations that may occur, thus keeping the fringes locked in place and stabilizing the interference pattern.

The grating equation which relates the incident angle to the angle of diffraction is as follows:

$$\sin \theta_m = \sin \theta_i + \frac{m\lambda}{d} \qquad (1)$$

where θ_i is the incident angle, θ_m is the diffracted angle for order m, λ is the wavelength of incident light, and d is the groove spacing of the grating (Figure 3). If the diffraction grating is to be used at the $m = -1$ order, at the Littrow angle, the grating equation becomes

$$\sin \theta_i = \frac{\lambda}{2d}. \qquad (2)$$

Thus, for a specific incident angle during usage, a certain groove spacing is required. This groove spacing can be achieved by controlling the angles of the exposure beams according to this equation (Figure 4):

$$\mathrm{d} = \frac{\lambda_e}{2\sin\theta_e \cos\phi}. \qquad (3)$$

Using equation (2), the groove spacing required for an incident angle of 51.2° of 1053 nm radiation is 675 nm. To achieve this, the exposure angle must be set to 17.8°, assuming the bisector of the angle

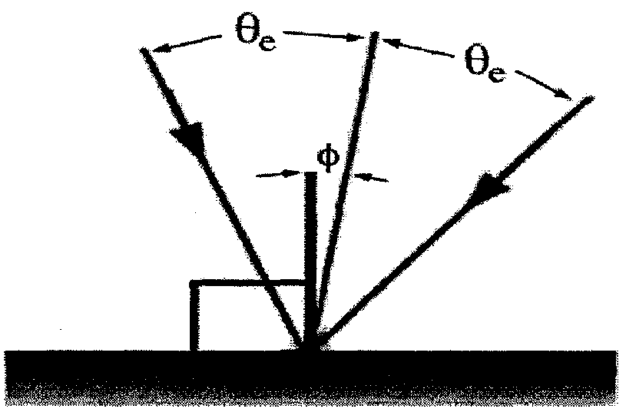

Figure 4. Holographic exposure geometry

between beam vectors is normal to the target grating surface.

Numerous factors influence the exposure time necessary for optimal grating performance. In general, exposure time, often measured as fluence, is inversely related to development time. If a longer development time is desired, the duration of exposure must be shorter. Another factor to be considered is the preferred duty cycle of the grating. For a larger base width, a shorter exposure time may be used. With a high-contrast photoresist, however, there is an exposure fluence threshold under which very little exposed resist will actually dissolve away during development. Fluences we used for lift-off ranged from 130 to 160 mJ/cm^2.

2.3 Preprocessing

To facilitate lift-off, various forms of preprocessing can be utilized[5]. After exposure, our substrates are subject to a chlorobenzene pre-development soak. This fractioning solvent is absorbed in the top surface of the exposed photoresist and serves to inhibit dissolution during development. Consequently, structures with an overhang are produced. The thickness of the overhang layer is affected by the soak time and the impurities in the chlorobenzene. The substrates are again rinsed with distilled water.

2.4 Development

After preprocessing, the samples are brought to the developing station (Figure 5). They are placed in the same exposure holder in the original orientation so that alignment can be maintained for *in situ* development monitoring[6]. A laser beam of

[5] W. M. Moureau, "Semiconductor lithography: principles, practices, and materials."
[6] J. A. Britten *et al.*, "*In situ* end-point detection during development of submicrometer grating structures in photoresist."

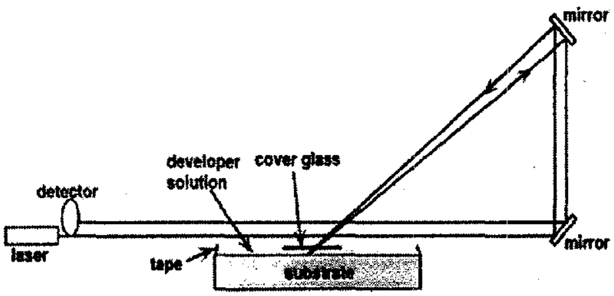

Figure 5. *In situ* development station schematic

unexposing wavelength illuminates the sample at the $m = -1$ order, Littrow angle such that the diffracted beam can be measured with a photodetector. The sample is placed in the reservoir under a wave-suppressing window. At time zero, the sample is immersed in 100% AZ MIF 300 developer. Although development times may vary for each sample, the end-point is determined by the shape of the curve of the monitored diffraction intensity. For tall structures, as the grating emerges, the intensity will cycle through a series of minima and maxima until it finally levels off. At this point, development through the entire photoresist layer has occurred, and the sample can be removed from the developer solution. Further development will merely cause the sidewalls of the gratings structures to dissolve, resulting in a decreased duty cycle. The grating mask sample is then rinsed with distilled water and dried with dry-nitrogen.

2.5 Oxide deposition and lift-off

Once the grating mask has been made, the actual grating itself can be formed. Both small and large substrates can be placed on a magnetron sputtering chamber and undergo oxide deposition. Hafnium oxide is first evaporated at an angle to produce half of a cap at the tips of the grating mask (Figure 6a). Angled evaporation from the other side occurs to produce the other half of the cap (Figure 6b). Oxide evaporation normal to the substrate then deposits the actual grating between the photoresist structures (Figure 6c). The photoresist grating mask is subsequently lifted off by dissolution in 3% NaOH (Figure 6d).

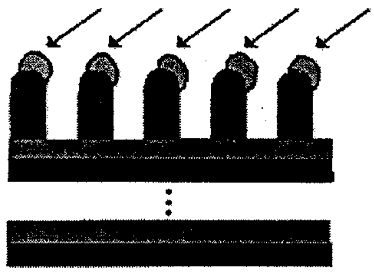

Figure 6a. Angled evaporation of oxide to form half of overghanging cap

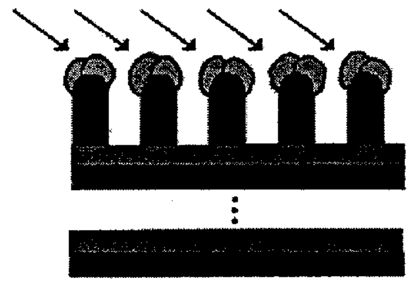

Figure 6b. Angled evaporation of oxide from other side to form other half of cap

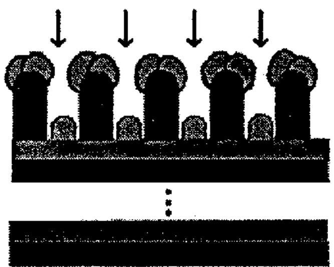

Figure 6c. Normal evaporation of oxide between photoresist grating mask

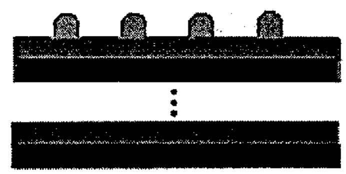

Figure 6d. Photoresist grating mask is lifted off by dissolution

3. Results and Analysis

We have been able to fabricate several different grating masks for the lift-off process (Figures 7-9). Some samples were deposited with an oxide and subsequently stripped of its photoresist grating mask. The samples were then broken and examined with scanning electron microscopy (SEM).

Figure 7 shows SEM's of a grating that was directionally evaporated with scandia. Deep cap-shaped overhangs were created on the scandia samples, and normal evaporation resulted in approximately 250 nm high ridges between the grating masks. After lift-off of the grating mask, scandia ridges remained with excess material along the sides (Figure 7b). These extensions are remnants of the oxide that existed on the sidewalls near the base of the photoresist mask structures. The angle deposited oxide penetrated too deeply into the grating mask, thus producing the final non-ideal grating. These results do show, however, that the lift-off process is possible.

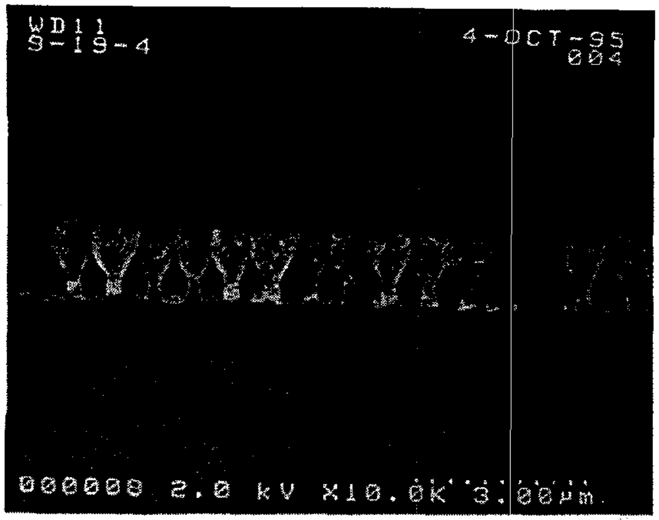

Figure 7a

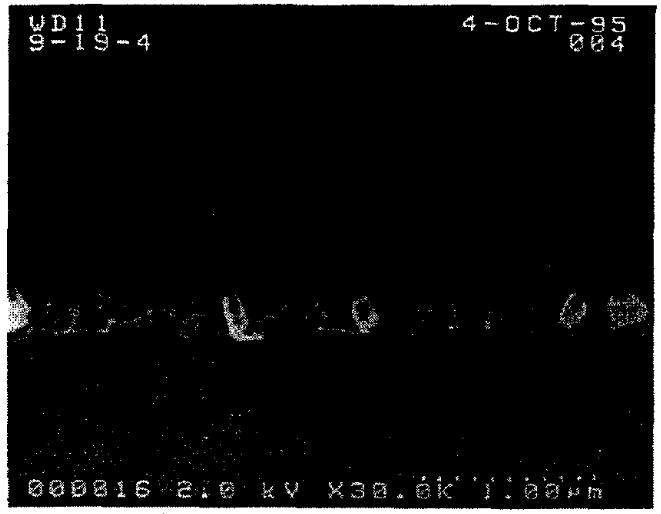

Figure 7b

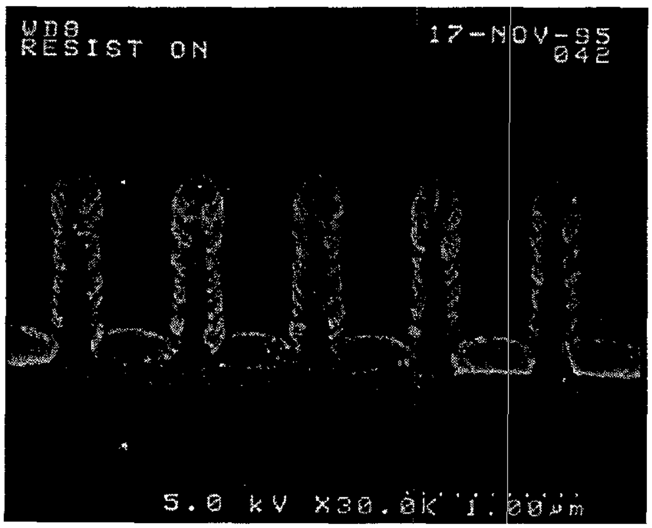

Figure 8a

Figure 8b

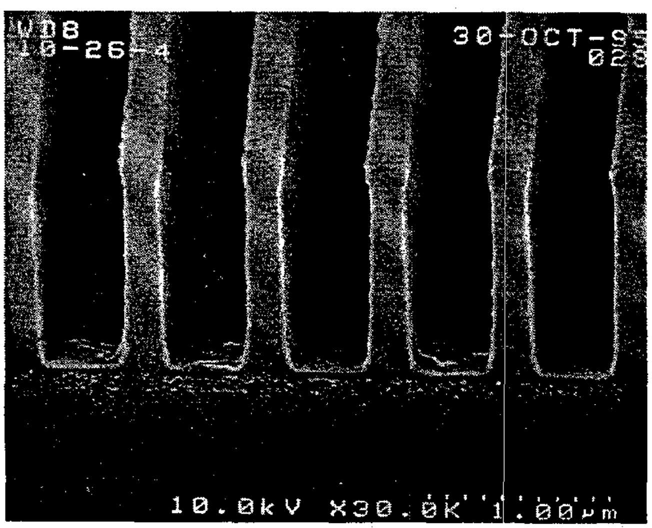

Figure 9a

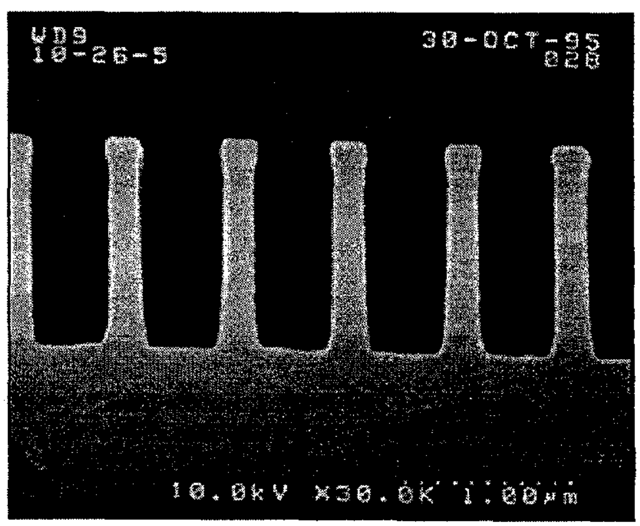

Figure 9b

Figure 8a shows a grating mask magnetron-sputtered with hafnium oxide. A high-contrast photoresist was used to create tall structures with vertical sidewalls. After normal deposition of the oxide, 200 nm high ridges remained between the grating mask structures. After the photoresist mask was stripped, the sample revealed almost no hint of a grating (Figure 8b). The accumulation of oxide on the sidewalls may have again caused problems during lift-off. On this microscope slide sample, the hafnium oxide did not adhere to the underlying surface adequately. This adhesion problem probably would not occur on an actual dielectric surface of high index of refraction.

In hopes of avoiding the sidewall accumulation problem, we attempted to create tall grating masks preprocessed to develop an overhanging structure. Gratings pre-soaked in chlorobenzene exemplify this characteristic, as shown in Figures 9a and b. These structures are 1.2 μm tall with a base width of 0.25 μm. We hope that tall structures such as these, with the appropriate angle of oxide evaporation, will prevent sidewall growth. The high aspect ratio of these structures (>4.5) may also facilitate lift-off by keeping the base of the photoresist clear of oxide. Thus, when the photoresist mask is dissolved, the actual grating material will remain intact.

4. Conclusions

Although the preliminary grating masks we have created did not generate ideal gratings, our limited success does prove that the creation of diffraction gratings by lift-off processing is indeed possible. Each process step contributes significantly to the resulting characteristics of the final grating mask. These characteristics include overall shape, height, basewidth, duty cycle, and adhesion to the substrate surface. We must continue our research to optimize the processing method. Only then will we have the ability to produce grating masks that will provide high-efficiency, high damage threshold diffraction gratings.

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