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Low Efficiency Gratings For 3rd Harmonic Diagnostics Applications

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ABSTRACT

The baseline design of the National Ignition Facility (NIF) calls for sampling gratings to provide third-harmonic energy diagnostics in the highly constrained area of the target chamber. These 40x40 cm transmission gratings are to diffract at (order +1) nominally 0.3% of the incident 351 nm light at a small angle onto a focusing mirror and into a calorimeter. The design calls for a plane grating of 500 lines/mm, and approximately 30 nm deep, etched into a fused silica focusing lens and subsequently overcoated with a sol-gel antireflective coating. Gratings of similar aperture and feature size have been produced for other applications by ion etching processes, but, in an effort to reduce substantially the cost of such optics, we are studying the feasibility of making these gratings by wet chemical etching techniques. Experimentation with high-quality fused silica substrates on 5 and 15 cm. scale has led to a wet etching process which can meet the design goals and which offers no significant scaleup barriers to full sized optics.

The grating is produced by holographic exposure and a series of processing steps using only a photoresist mask and a final hydrofluoric acid etch. Gratings on 15 cm diameter test substrates exhibit absolute diffraction efficiencies from 0.2-0.4% with a standard deviation of about 15% of the mean over the full aperture. The efficiency variation is due to variation in linewidth caused by spatial nonuniformities in exposure energy. Uniformity improvements can be realized by using a smaller, more uniform portion of the exposure beam and exposing for longer times. The laser damage threshold for these gratings has been measured at LLNL and found to be identical to that of the fused silica substrate. Scaleup to full-sized substrates will use techniques such as meniscus coating for photoresist, large-aperture holography and other processes already established at LLNL for optics of this size. A prototype sampling grating to be installed on the Beamlet laser will be produced in early 1996.

Keywords: gratings, damage

1. INTRODUCTION

The baseline design for the NIF calls for low-efficiency gratings in the target chamber vacuum vessel to direct a small fraction of the incident 3rd harmonic beams into calorimeters. Optical quality fused silica is the only substrate material of practical choice due to laser damage concerns, and the grating etched into the optic must not degrade its laser damage threshold. Of course, as is the case for all NIF components, cost of manufacture is an overriding concern. To this end, we have begun to investigate the feasibility of a manufacturing process for sampling gratings involving holographic exposure of photoresist grating masks followed by wet chemical etching of the SiO_2. Such a process requires no deposition or etching operations involving vacuum systems, and is in principle scaleable to the large apertures of the NIF. Gratings of similar aperture, period and groove depth have been made for aerospace applications by ion-etching[1,2] but this is a very costly process for the number of gratings needed. Ruled gratings of this aperture and feature size are not practical from a manufacturing viewpoint due to tool wear, ghost diffractions and the sheer number of parts needed. Although as a rule the etching of micron-sized features is the realm of dry etching[3,4], the very small aspect ratio of the grooves needed for low-efficiency diffraction $O(10^{-2})$ suggests that isotropic wet chemical etching would be suitable for this application.

The specifications for the 3rd harmonic sampling grating as stated in the NIF Conceptual Design Report (CDR) are that it be a plane grating to diffract in transmission 0.01% of the incident 3ω light at an angle of 10^o. The angle fixes the period of the grating to 2 μm. The efficiency specification of this grating has been subsequently revised to 0.3% based on what is achievable in practice, since a 0.01% efficiency in transmission requires depth control to atomistic dimensions. Modeling of performance of shallow gratings suggests that a groove depth of approximately 30 nm is required. The grating is to be overcoated with a sol-gel AR coating, which will reduce the efficiency of the bare grating structure.

Maintaining spatial uniformity of the efficiency across the grating aperture is the most challenging processing requirement. We have made very uniform gold overcoated high-efficiency reflective gratings by fixing the groove depth corresponding to the calculated maximum efficiency[5,6]. This is done by applying the photoresist film at this thickness, exposing the grating structure and developing the grating using in-situ monitoring[6] of the evolving grating structure to assure that the exposed resist has cleared to the substrate. Variations in exposure intensity therefore affect the grating linewidth but not the height. At the groove depth corresponding to maximum efficiency in high-efficiency gratings, the derivative of the efficiency with respect to groove depth is of course zero, and the efficiency variation with linewidth is relatively insensitive as well. However, for low-efficiency gratings, the efficiency is a strong function of both the groove depth and linewidth, and, for etched gratings, the linewidth variation is directly translated into the substrate during the etch process.

Beam diagnostic packages employing diffraction gratings at other locations along the NIF beamlines are being considered. These are reflection gratings at 1.053 μm, for which an efficiency of 0.01% is feasible. These gratings differ in details of groove depth and spacing from the gratings discussed in this work, but a manufacturing process developed for 3ω sampling gratings in the NIF baseline design is expected to be applied straightforwardly to 1ω sampling gratings. The following describes the results of our R&D effort towards this manufacturing process.

2. EXPERIMENTAL

Optical quality fused silica substrates which were chemically/mechanically polished to minimize subsurface damage were obtained from Zygo Corp. These were washed with soap and water followed by NaOH rinse, then rinsed with deionized water and dried using dry, filtered and ionized nitrogen. The substrates were then baked at 220C for 2-3 hours to drive off surface and subsurface adsorbed water. The oven was then cooled to 110C and hexamethyl disilazane (HMDS) was introduced, to evaporate and vapor-prime the surface for enhanced adhesion of the photoresist. Following cooling, a 300 nm photoresist film was applied either by spinning for small samples, or meniscus coating[7] for 15 cm substrates, and the substrates softbaked at 80C for 30 min. The cooled substrates were painted on the rear surface with an absorbing strippable coating to attenuate back surface reflections, and then exposed holographically with a fringe-locked grating interferometer[5,6] shown schematically in Figure 1, which uses a Coherent Inova Kr-ion laser operating at 413 nm. The period of the grating was 1.97 μm. Approximately 85% of the total beam was used to expose 15 cm. diameter parts. At average intensities of 8 mW at the target plane (2W total laser power) exposures of only 30s were required. The latent gratings were developed using in-situ monitoring[6] to determine when the exposed resist cleared to the substrate. The gratings were then well rinsed with deionized water and placed immediately into an aqueous 1%HF, 15% NH_4F solution at room temperature for 90 s, resulting in an etch depth of the exposed silica of nominally 25 nm. The gratings were then again rinsed, and the resist mask removed by flood exposure with a UV source followed by development, rinsing and drying. The resulting gratings in fused silica were subjected to photometric and laser damage testing.

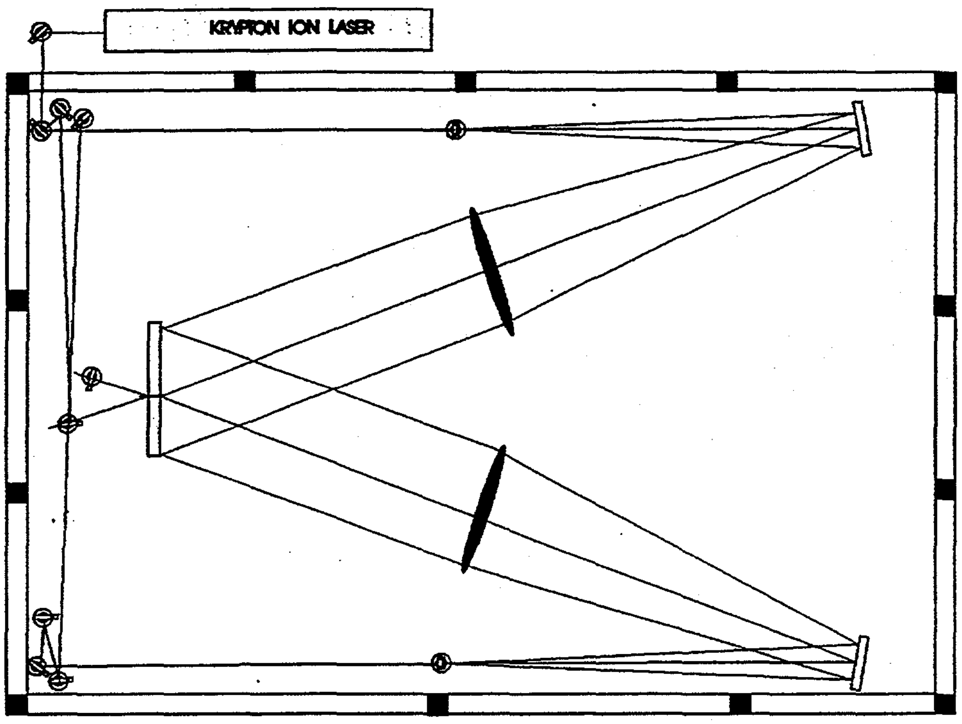

Figure 1. Schematic of holographic exposure facility.

3. RESULTS AND DISCUSSION

By use of the above procedure, gratings can be produced using only photoresist as a mask for the HF etching step. The process is extremely simple and no specialized equipment outside of the grating interferometer is required. The process time is largely taken up during the substrate prebake and the photoresist curing steps.

The transmitted (+1) diffraction efficiency at 355 nm of a 15 cm diameter grating wet-etched in to fused silica is shown in Figure 2. The average efficiency is 0.39% with a standard deviation of 13.5% of the mean over the entire aperture. The spatial variation of the diffraction efficiency is an inverse imprint of the intensity of the laser used to expose the pattern in the photoresist. This laser propagates the TEM_{00}

gaussian, and TEM_{10} and TEM_{01} toroidal modes simultaneously(5), to give an approximately top-hat beam profile that is, however, nonuniform by several percent. The intensity is maximal in the toroidal modes during typical operational conditions. A relative increase in intensity results in thinner grating lines in the photoresist, and this is transferred into the optic during the etch step. Thus, for the grating illustrated in Fig. 2 which was exposed using approximately 85% of the full beam, the diffraction efficiency is lowest in the toroidal region where the exposure intensity was highest, because in this area the increased fluence reduced the grating linewidth relative to the center of the optic where development monitoring was performed. It is important to note that intensity variations caused by linewidth variation during exposure/development would be evident in ion-etched as well as wet-etched gratings. The uniformity can be much improved by expanding the interfering beams and using only the central portion. This will extend exposure times, but the stability of the fringe-locked interferometer is sufficient for indefinite exposure times.

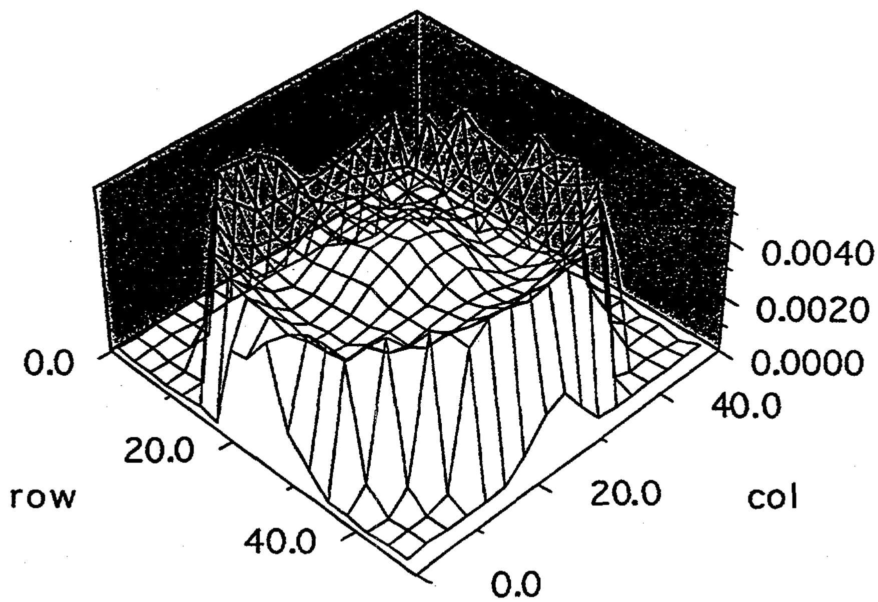

Figure 2. Efficiency of 1st diffracted (transmitted) order at 355 nm for 15 cm diameter grating wet-etched in fused silica. Average=0.398%; st. deviation=13.5% of average; min=0.190%; max=0.580%. (grating 4-21-3).

The depth of the acid-etched grooves is uniform to within about 2% across the aperture since, as noted in the literature[8] as well as in our lab, the etching of silica by HF is kinetically controlled, and independent of geometry and agitation that can result in lateral variations under conditions subject to mass transfer control. Thus, the wet etching process is easily scaleable to NIF sized parts.

A SEM of a sampling grating in superpolished fused silica is shown in Figure 3. This grating was made using a two-step transfer etch process with an intermediate chrome mask. Each wet transfer etch process reduces the grating linewidth. Therefore, the linewidth of this grating is less than that of the gratings made by the single-step photoresist mask process, but the feature details are illustrative. Some evidence of subsurface damage marks are evident in the etched grating grooves, but these features are very small.

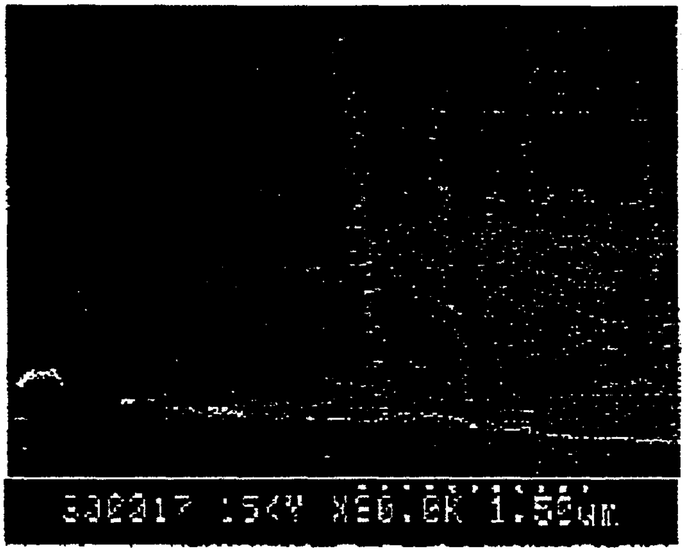

Figure 3. SEM of a grating wet-etched into a superpolished fused silica substrate.

Tests on one grating on a superpolished substrate show that the laser damage characteristics of the fused silica optic is not influenced by the shallow grating structure. Table 1 shows unconditioned and conditioned laser damage thresholds of a 15 cm diameter, 2.5 cm thick fused silica window coated with a 3w sol-gel AR coating on both the front (bare) and rear (grating) surfaces. The measurements were performed at the LLNL laser damage facility, using 355 nm light with 3 ns pulses at 10Hz rep rate. Within experimental error, the front and rear surface damage thresholds are identical, and neither surface showed improved performance with ramped-fluence (r:1) conditioning.

Table 1

Laser damage threshold of sol-gel AR coated grating (rear surface) and AR coated front surface (J/cm^2 @ 355nm, 3ns, 10^o incidence)

Surface	s:1 (unconditioned	r:1 (conditioned)
front	19.3+/-3	20.6+/-3
rear (grating)	22.6+/-3	19.8+/-3

These numbers fall within the statistical range expected for AR coated optical quality fused silica of 18-24 J/cm^2 under these conditions(9).

Attack by HF on silica preferentially enhances microcracks and other subsurface flaws caused by mechanical polishing[8], leading to increased surface roughness, scattering losses and probably lowered laser damage thresholds in the manufacture of small surface relief, micron-sized grating structures. Figure 4 shows an atomic force microscope image of a sampling grating wet-etched into a substrate that was finished using an ordinary lap polish. The vertical scale is grossly exaggerated in this image, but the degree of surface roughness in the etched grooves can be contrasted with the very much smaller-scale roughness of the etched grating shown in Figure 3, which was chemically/mechanically polished. The grating of Figure 4 exhibited a laser damage threshold of 9 J/cm^2 for 355 nm, 3 ns pulses, and also exhibited some line liftoff during processing. These results underscore the need to begin with a properly polished substrate.

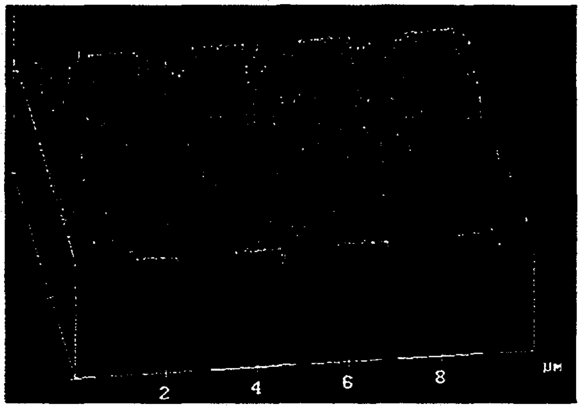

Figure 4. AFM image of a 2 μm period, 18 nm deep grating structure HF etched into lap-polished fused silica

Table 2 shows the effect of an AR coating on the transmitted diffracted efficiency for two test gratings. The coating was a standard porous sol-gel SiO_2 film[10] (n=1.2) applied by dip coating. The nominal film thickness of 75 nm is about twice as thick as the grating feature depths, but given the low aspect ratio of the gratings, little distortion of the grating shape is expected. The simple Rayleigh theory of grating performance cannot treat the case of a partially conformal overcoat, so the grating performance with AR coating was not modeled. Based on measured results, a reduction in the diffraction efficiency of approximately 40% can be expected due to the AR coating.

Table 2

Effect of 3ω sol-gel AR coating on transmitted diffraction efficiency

	Average % transmitted diffraction efficiency	
Grating ID no.	bare surfaces	AR coated
12-20-3	0.29	0.17
2-25-5	0.53	0.31

The key to the manufacture of low-amplitude sampling gratings in fused silica by wet chemical etching is to begin with a substrate that has been chemically-mechanically polished to minimize subsurface damage, and to thoroughly clean, bake and prime the substrate as outlined previously. This assures good adhesion of the resist mask during the processing steps, and to remove any organic contaminants which can act as a barrier to attack by the etchant. Early work focused on performing the lithography on a chrome layer sputtered onto the fused silica, transferring the resist grating into the chrome by a wet etch process, and using the chrome as a mask for HF etching of the fused silica. The intermediate chrome mask was necessary for grating transfer to substrates that were not chemically/mechanically polished initially, or that were not adequately baked prior to application of the resist, but by subscribing to the procedure outlined previously, the metal mask was proven to be unnecessary.

4. CONCLUSIONS

We have manufactured low-efficiency sampling gratings for transmitting high fluence 351 nm laser light by HF etching of fused silica substrates up to 15 cm in diameter, using a simple process involving only photoresist masks on superpolished substrates. The uniformity of the gratings can be improved by using a smaller, more uniform portion of the exposing beam and longer exposure times. The laser damage threshold of these gratings are the same as that of an AR coated bare surface. The role of subsurface damage in the manufacture of gratings by wet chemical etching is discussed.

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