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ABSTRACT

Multilayer dielectric (MLD) diffraction gratings[1] for Petawatt-class laser systems possess unique laser damage characteristics. Details of the shape of the grating lines[2,3] and the concentration of absorbing impurities on the surface of the grating structures both have strong effects on laser damage threshold. It is known that electric field enhancement in the solid material comprising the grating lines varies directly with the linewidth and inversely with the line height for equivalent diffraction efficiency[2,3]. Here, we present an overview of laser damage characteristics of MLD gratings, and describe a process for post-processing ion-beam etched grating lines using very dilute buffered hydrofluoric acid solutions. This process acts simultaneously to reduce grating linewidth and remove surface contaminants, thereby improving laser damage thresholds through two pathways.

1. INTRODUCTION

The technique of chirped pulse amplification[4] (CPA) has enabled the generation of Petawatt-class laser system throughout the world. Existing Petawatt-class lasers today are mostly based on Nd:glass architectures with laser pulses nominally of 500 J energy and 500 fs pulse duration. The design and construction of a new generation of high-energy Petawatt-class laser systems is currently proceeding at a number of institutions around the world[5-7]. These lasers require large aperture diffraction gratings to compress amplified, temporally-stretched pulses[4]. The potential for increased energy and power handling capacity of multilayer dielectric (MLD) gratings make them the optic of choice for these new lasers, supplanting the gold-overcoated photoresist gratings used in the world's first Petawatt laser at LLNL[8] and currently in use elsewhere[9,10]. MLD gratings were first successfully demonstrated at Lawrence Livermore National Laboratory (LLNL) in the mid 1990's[1]. They are considerably more complex to manufacture compared with gold-overcoated gratings.

Modeling of the electric field distribution in the vicinity of a high diffraction-efficiency grating illuminated by a coherent plane wave shows periodic maxima set up by interference between the incident and diffracted waves. Figure 1 shows such a distribution for a model grating. The high e-field penetrates into the solid material comprising the grating along the back side of the grating, as referenced from the side of the incoming wave. Field enhancements in these 'hot' zones are invariably greater than unity, normalized to the field strength of the incoming wave. Field enhancements this large are not typically seen in the solid material of a multilayer high-reflector, for example, so we expect the damage characteristics to be different between MLD mirrors and MLD gratings. Stuart et al.[11] have published data on the damage characteristics of MLD gratings that show damage localized to the back edges of the grating ridges, in correspondence with model results (see Fig. 1).

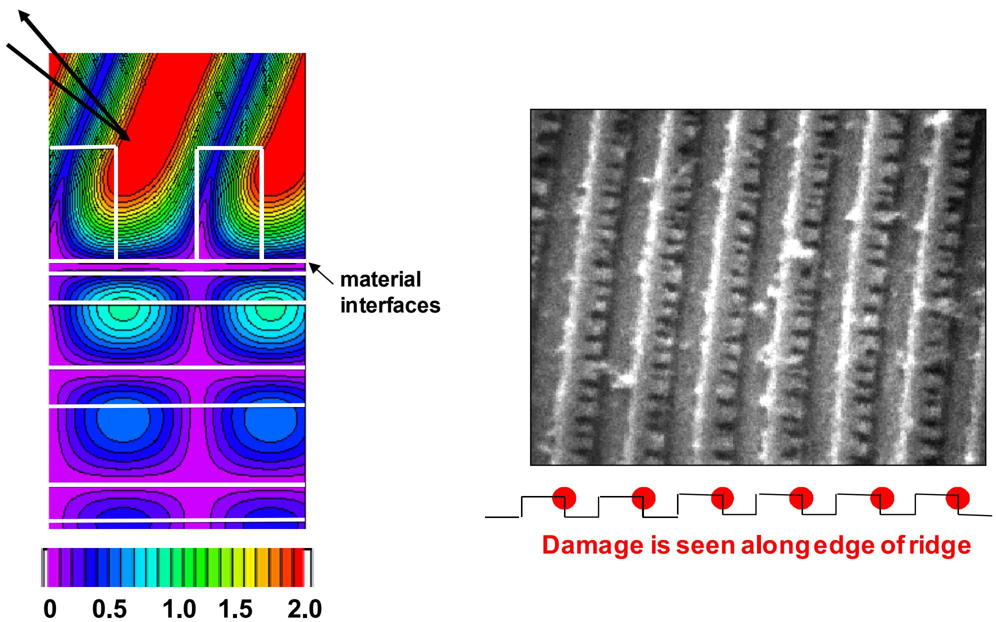


Figure 1, A. (Left); Distribution of electric field in the vicinity of a grating with laser radiation incident from the left. Material interfaces shown in white outline. B (Right); Scanning electron micrograph showing damage along edge of grating ridges. (from Ref 11).

2. DESIGN OF MLD GRATINGS

The optical design of a high-efficiency MLD grating is subject to a number of constraints related to its manufacturability[12]. We choose to design a dichroic multilayer coating that is highly reflective at the use angle and wavelength, and minimally reflective at the holographic exposure angle and wavelength. This is to minimize standing-wave effects common to pattern generation in photoresist on reflective structures that impact linewidth control. The second criterion in particular places demands on the accuracy of the coating deposition. We could choose to deposit a simpler quarter-wave design and use a sacrificial absorptive coating between the multilayer stack and the photoresist film, but this increases complexity and risk for other aspects of the grating manufacturing process, particularly at large apertures. The design must also be insensitive to coating deposition and grating linewidth variations that can be expected to occur over the meter-size apertures. The grating can be etched into one or several of the deposited dielectric layers. We choose to have a single thick SiO_2 layer comprising the grating due to the intrinsic high laser damage threshold of SiO_2. The high-and low index layers comprising the stack are made of HfO_2 and SiO_2. The final design is the result of numerous iterations based on performance and manufacturing error-tolerance considerations.

Once the MLD stack design is finalized, the grating profiles are optimized for minimal electric field intensities in the solid grating material, while at the same time maximizing the efficiency. Figure 2 shows a plot of the calculated diffraction efficiency at 1053 nm, 76.5^0 incidence angle and TE polarization, of a 1780 line/mm MLD grating as a function of grating height on the vertical axis and grating duty cycle (linewidth/period) on the horizontal axis, for a HfO_2/SiO_2 MLD grating with the grating in the top SiO_2 layer. A surface of high efficiency >99% extends from the lower right to the upper left of this plot, as shown. It is generally true that the electric field strength in the solid material along a line of constant diffraction efficiency decreases as the grating height increases and the duty cycle decreases. Gratings with calculated electric field enhancements of ~1.1 are possible with manufacturable designs having duty cycles of -25% and height:width aspect ratios of ~5: 1. Neauport et al.[3] have published data that show evidence that for fixed incident angle and materials the damage of an MLD grating is directly related to the electric field intensity, which depends on the groove profile as described above.

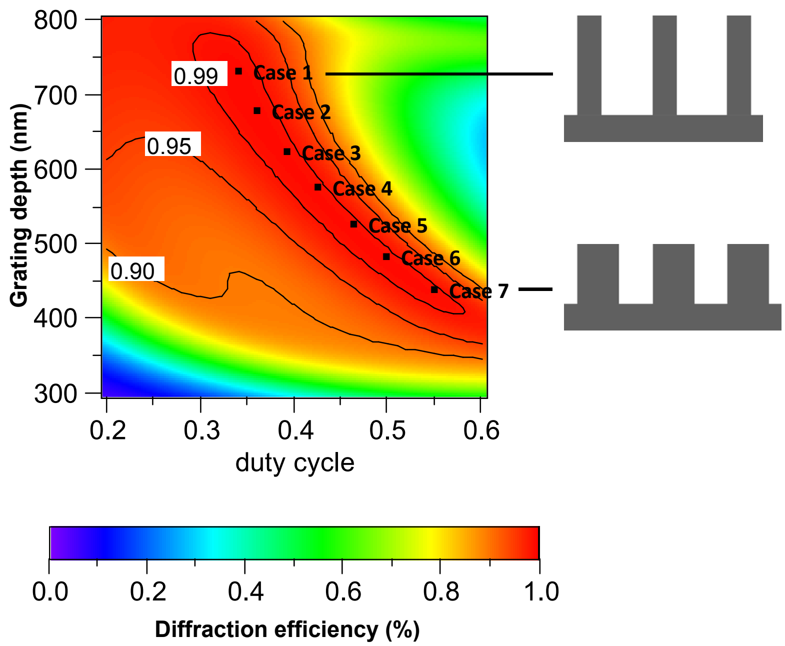
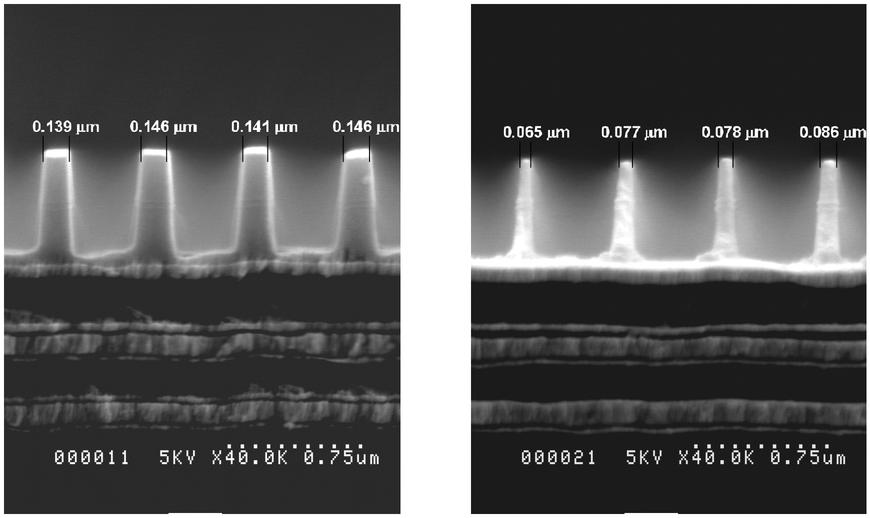


Figure 2. A plot of calculated diffraction efficiency as function of grating height and duty cycle for 1780 line/mm MLD grating for 1053 nm, TE polarization, 76.5° incidence angle. For a given efficiency, maximum electric field in the solid grating material decreases in the direction as the grating height increases and the duty cycle decreases (from Ref. 2).

3. HF-ETCHBACK FOR GRATING LINEWIDTH TAYLORING

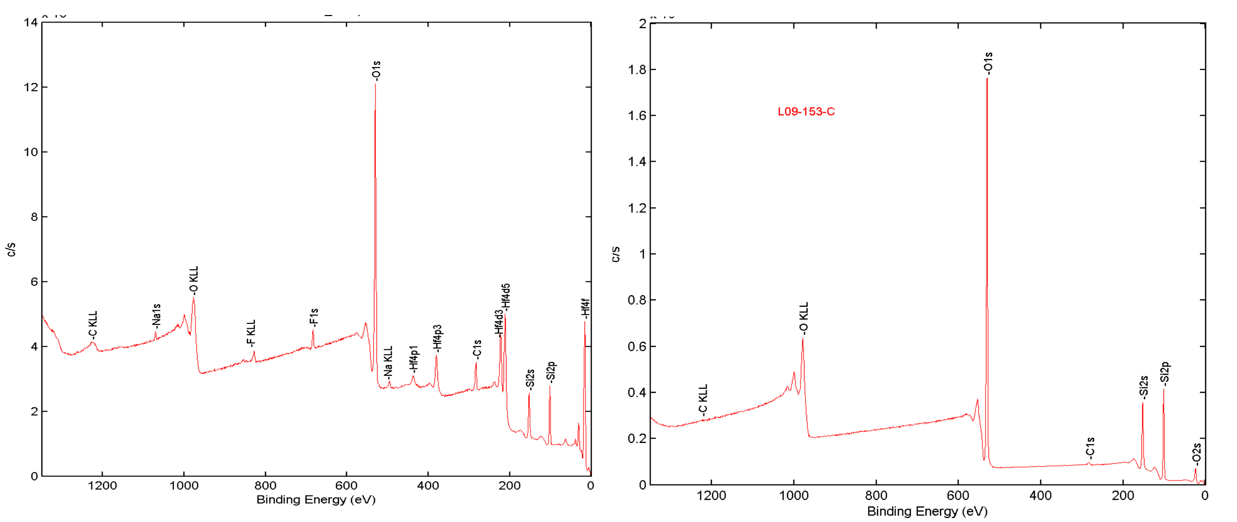
We have developed a process utilizing a very dilute buffered hydrofluoric (HF) acid solution to wet-etch grating profiles fabricated into the top SiO_2 layer of a multi-layer dielectric coated stack. This process acts simultaneously to reduce grating linewidth in a controlled and predictable fashion and remove surface contaminants, thereby improving laser damage thresholds through two pathways. The grating lines are initially formed via reactive ion-beam etching using a holographically-generated vertical-sidewall photoresist mask. This anisotropic etching process produces nearly vertical-sidewall gratings with the linewidth nominally fixed by the mask linewidth. Wet-etching, on the other hand, is an isotropic removal process wherein the surface recedes at a constant rate everywhere along the surface normal. For wet-etching of grating lines, this results in a profile evolution that affects the linewidth more strongly than the depth. Line profiles eventually evolve into cusplike shapes with sharp tips[13]. For wet-etching of high aspect-ratio submicron grating lines, precise control is required since very small removal totals have very large effects on duty cycle. Also of significance is that etch rates of deposited SiO_2 layers can be very much larger than for bulk fused silica due to the small residual porosity of these layers. Figure 3 shows SEM micrographs comparisons of grating profiles after standard processing and of grating profiles after subsequent HF etchback for 6 minutes.



A B

Figure 3. A. (Left); SEM micrograph of grating profiles after ion-beam etching and cleaning. B. (Right); SEM micrograph of grating profiles after subsequent HF etchback for 6 minutes.

Since wet etching removes the entire surface layer, it can be expected that contamination left on the surface by prior processing would be removed as well. Figures 4 and 5 show XPS and EXP/EDAX surface analysis data of a witness grating before and after HF etchback treatment. The data indicate a significant reduction in surface contaminant levels after the etchback treatment. In particular, bond-energy analysis shows the removal of C-F compounds that are a residue from our reactive ion-beam etching process.



A B

Figure 4. XPS surface analysis of MLD witness grating L09-153F after standard cleaning following ion beam etching with no O_2 (A), and grating L09-153C with same treatment followed by the HF etchback (A). Both show C, O, Si. (A) shows signals of F, Na, Hf as well.

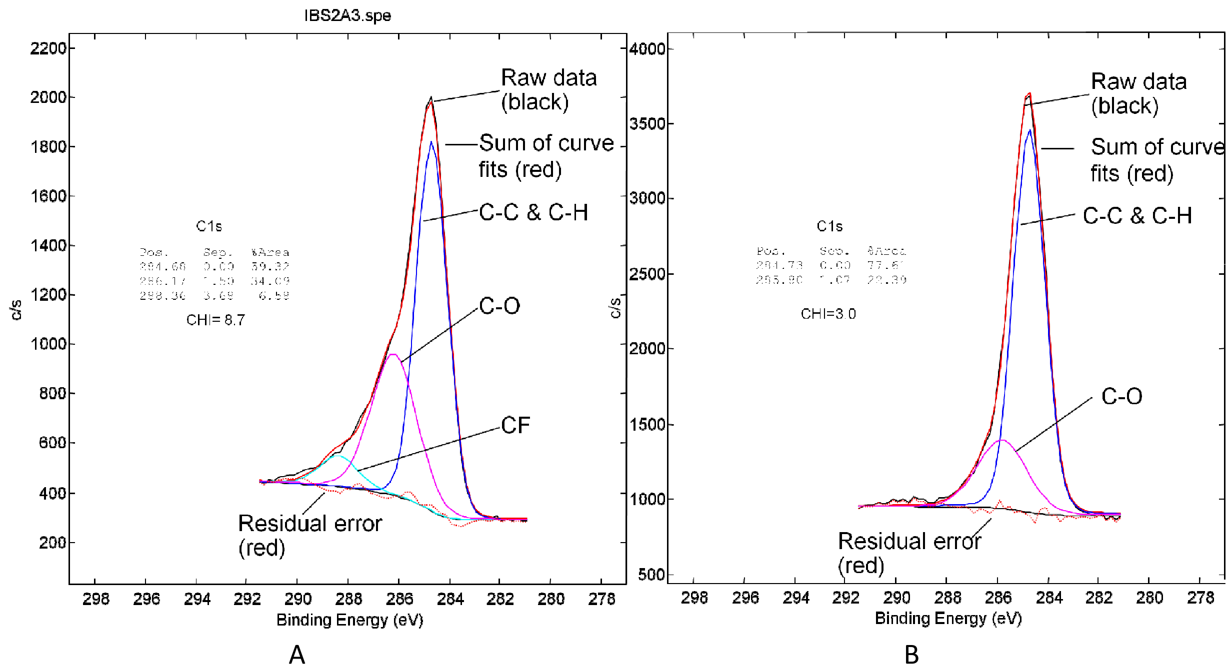


Figure 5. Analysis of EXP/EDAX carbon binding energies of ion-milled surface before (A) and after (B) cleaning process. CF-bonds are present before cleaning.

The HF etchback process also allows the ability to easily modify ion-milled profiles to recover or tailor performance if linewidths are larger than optimal, making it a processing tool that can increase yield as well as improve performance. Figure 6 shows diffraction efficiency comparisons of a 850x450 mm MLD grating after standard processing and after subsequent HF etchback. Of course linewidths less than optimum cannot be improved by this process.

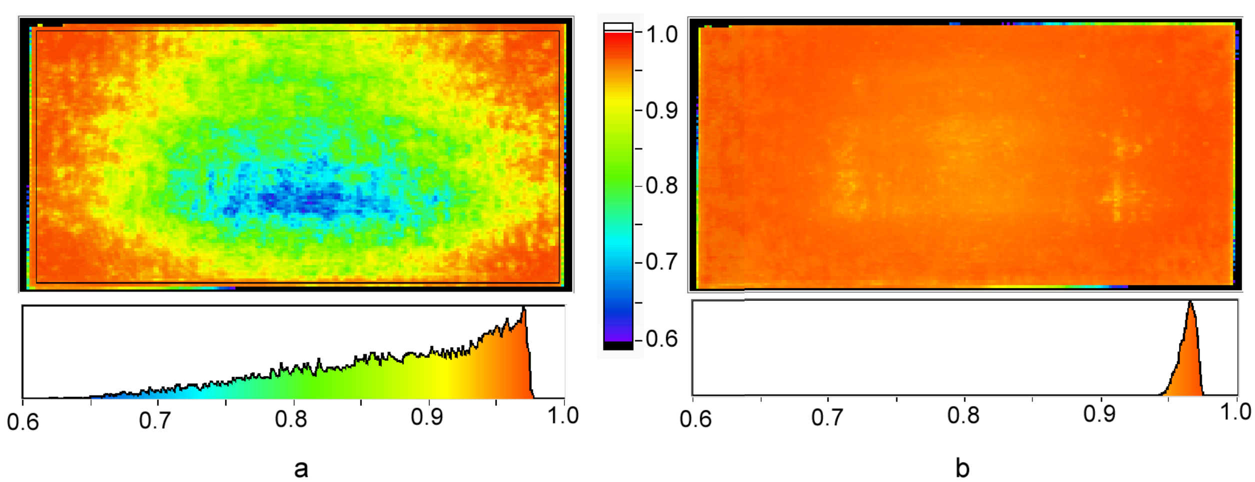


Figure 6. Diffraction efficiency map of 850x450 mm MLD ARC grating 38035 (1782 l/mm) at 1053 nm, 76.5° before (a) and after (b) profile etchback with dilute buffered HF solution.

Fourteen sample gratings were tested at LLNL's small-scale damage test facility before and after HF-etchback treatment. Figure 7 presents laser damage resistance of several small witness gratings measured at 1053 nm, nominal 10 ps pulse duration, 76.5^{o} incidence angle in air at 10 Hz. Damage thresholds are reported as normal-incidence fluence. Damage was defined as the onset of a visible surface change under brightfield illumination at 10X magnification. An average value for this onset based on 10 sites subjected to a slowly ramped fluence is reported. Seven of the 14 witness showed an increase in damage threshold above 10%, with three improving above 40%. The other half exhibited an insignificant change in performance.

Sample	Part #	Onset damage threshold (J/cm^2)		
		Standard process	after HF etchback	% change
1	L09-83C	4.3	4.5	5.4
2	L09-153C	3.1	4.4	42.1
3	L09-153D	3.2	4.4	36.6
4	L09-153E	2.4	3.6	48.2
5	L09-153F	3.2	3.8	18.8
6	L09-236A	3.9	3.9	0.8
7	L09-236B	5.0	4.9	-3.4
8	L08-549A	4.1	5.7	40.0
9	L09-101A	4.4	4.5	0.9
10	L09-059C	4.4	5.5	26.2
11	L09-103B	3.9	4.0	3.5
12	L09-234C	3.3	4.4	31.7
13	L09-236C	3.7	3.6	-1.5
14	L09-237B	4.5	4.9	9.1
	Average	**3.8**	**4.4**	**18.5**

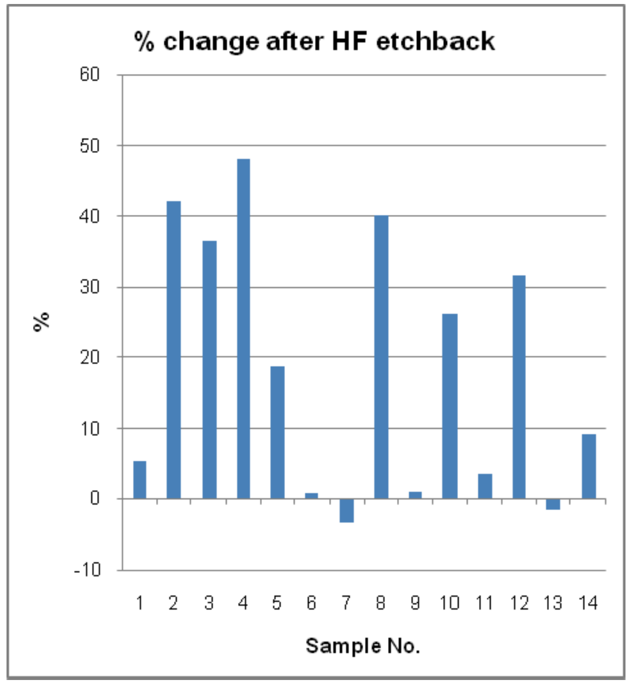


Figure 7. Onset of laser damage of 13 MLD witness gratings measured at LLNL (10 ps, 76.5^{o} incidence, 1053 nm, normal incidence fluence), before and after HF etchback process.

4. CONCLUSION

We have demonstrated a process utilizing a very dilute buffered hydrofluoric (HF) acid solution to wet-etch in a controlled fashion high aspect-ratio grating SiO_2 grating lines fabricated in a multi-layer dielectric coated stack. This process acts to reduce the grating linewidth and remove surface contaminants, thereby improving laser damage resistance via two mechanisms. The process also allows the ability to easily modify ion-milled profiles to recover or tailor performance if linewidths are larger than optimal resulting in a processing tool that can increase yield as well as improve performance.

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REFERENCES

1. M. D. Perry, R. D. Boyd, J. A. Britten, B. W. Shore, C. Shannon and L. Li, "High efficiency multilayer dielectric diffraction gratings," Opt. Lett. **20**, 940-942 (1995)

2. J.A. Britten, W.A. Molander, A.M. Komashko and C.P.J. Barty, 'Multilayer Dielectric Gratings for Petawatt-Class Laser Systems'. Proc. SPIE, V5273, 1-7, (2004)

3. J. Neauport, E. Lavastre, G. Raze, G. Dupuy, N. Bonod, M. Balas, G. de Villele, J. Flamand, S. Kaladgew, F. Desserouer, 'Effect of electric Field on Laser Induced Damage Threshold of Multilayer DieIectric Gratings', Optics Express, 15(19), 12508-12522 (2007)

4. D. Strickland and G. Mourou, 'Compression of Amplified Chirped Optical Pulses', Opt. Commun., 56, 219-221, (1985)

5. N. Blanchot et. al., Proc. Intl. Fusion Sciences Applications Conf., Monterey, CA (2003)

6. D. Meyerhofer et. al., Proc. Intl. Fusion Sciences Applications Conf., Monterey, CA (2003)

7. R. Kodama et al, Plasma Phys. Control. Fusion 44, B109 (2002)

8. M.D. Perry, D. Pennington, B.C. Stuart, G. Tietbohl, J.A. Britten, C. Brown, S. Herman, B. Golick, M. Kartz, J. Miller, H. T. Powell, M. Vergino, V. Yanovsky, "Petawatt laser pulses" Optics Letters, 24, 160-162, (1999)

9. Kitagawa, Y., Fujita, H. Kodama, R., Yoshida, H., Matsuo, S, Jitsuno, T., Kawasaki, T., Kitamura, H., Kanabe, T., Sakabe, S., Shigemori, K., Miyanaga, N., Izawa, Y., "Prepulse-free PetaWatt laser for a fast ignitor" IEEE J. Quantum Electronics, 40, 281_293, (2004)

10. M.H.R Hutchinson, D. Neely, B.E. Wyborn, 'Vulcan Upgrade: a PetaWatt laser facility for experiments at 10^{21} W cm^{-2}', Proc. SPIE, V4424, 63-69, (2001)

11. B.C. Stuart, M.D. Feit, S.M. Herman, A.M. Rubenchik, B.W. Shore, M.D. Perry, 'Ultrashort-Pulse Optical Damage' Laser-Induced Damage in Optical Materials, 27* Boulder Damage Symposium, H.E. Bennett et al., Proc. SPIE V2714, 616-627, (1995)

12. B.W. Shore, M.D. Perry, J.A. Britten, R.D. Boyd, M.D. Feit, H. T. Nguyen, R. Chow, G. Loomis and L. Li, 'Design of high-efficiency dielectric reflection gratings' Journal of the Optical Society of America A, 14(5), 1124-1136, (1997)

13. J.A. Britten and L.J. Summers, 'Multiscale, multifunction diffractive structures wet-etched into fused silica for high-laser damage threshold applications' Applied Optics, 37(30), 7049-7054, (1998)