Minimizing Contamination to Multilayer-Dielectric-Diffraction Gratings Within a Large Vacuum System

B. Ashe, K. L. Marshall, D. Mastrosimone, and C. McAtee
University of Rochester
Laboratory for Laser Energetics

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Outline

• Overview of the OMEGA EP Laser System
• Overview of the grating compression chamber (GCC) and multilayer-dielectric-diffraction (MLD) gratings
• GCC and component cleaning procedures
• Qualification of vacuum-compatible materials
• Multilayer-dielectric-diffraction-(MLD) grating cleaning
• GCC cleanliness verification
• Summary
Summary

Systematic approaches to minimize contamination of MLD gratings within the large GCC vacuum system have been implemented.

- The presence of contaminants (particle or molecular) in the vacuum system places the MLD gratings at risk with respect to lowered damage threshold.
- Numerous protocols have been developed and implemented at LLE to minimize MLD grating contamination.
- Vacuum surfaces and components are cleaned through multiple qualified cleaning procedures.
- Qualification, testing methods, and studies of materials for use within the GCC have been implemented.
- A piranha clean process has been developed and will be used to clean the large-scale-diffraction gratings.
- *In-situ* coupon gratings samples are periodically measured to verify that the large-aperture gratings continuously meet the laser-damage-threshold specification.

Keeping the GCC and MLD gratings clean is critical as the OMEGA EP Laser System becomes operational.
The Laboratory for Laser Energetics (LLE) was established in 1970

LLE’s Mission Statement

• To conduct implosion experiments and basic physics experiments in support of the National Inertial Confinement Fusion (ICF) Program

• To operate the National Laser Users’ Facility (NLUF)

• To develop new laser and materials technology

• To conduct research and development in advanced technology related to high-energy-density phenomena

• To provide graduate and undergraduate education in electro-optics, high-power lasers, high-energy-density physics, plasma physics, and nuclear fusion technology
The extended performance (EP) addition to OMEGA has five primary missions

1. Extend HED research capabilities with high-energy and high-brightness backlighting
2. Perform integrated advanced-ignition experiments
3. Develop advanced backlighter techniques for HED physics
4. Provide a staging facility for the NIF to improve its effectiveness
5. Conduct ultrahigh-intensity laser–matter interactions research
The OMEGA EP (extended performance) is located next to the existing OMEGA facility.

**OMEGA**
- Two Class-1000 cleanrooms
- 60 28-cm round beam, 351-nm UV system
- >30-kJ UV on target in 1 ns

**OMEGA EP**
- Single Class-1000 clean room
- Four 40-cm square beam, 1054-nm IR system
- Two 2.6-kJ short-pulse beams into OMEGA target chamber, or OMEGA EP target chamber
- Up to four 6.5-kJ long-pulse beams into OMEGA EP target chamber
OMEGA EP uses tiled multilayer-dielectric-diffraction gratings and chirped-pulse amplification (CPA) to compress pulses from nanoseconds to picoseconds.

Chirped-pulse amplification to create picosecond pulse

- Longer (red) wavelengths at the chirped (stretched) incoming pulse front experience larger diffraction, but arrive at G4 at the same time as the shorter (blue) wavelengths.

- Maximum system output is limited by the laser-damage resistance of TGA4 (~2.7 J/cm² at 1054-nm, 10-ps pulse measured beam normal)
A critical component for OMEGA EP is the grating compression chamber (GCC)

- 15-ft × 15-ft × 70-ft internal dimensions (15,750 ft³)
- Isolated payload support pylons (quantity 28)
- 2.5-ft × 6.6-ft man doors (quantity 2)
- 7.5-ft × 15-ft payload entry door
- 3.3-ft × 6.6-ft top payload access port
- Weight: 400,000 lbs
- Internal wall material: 304L SS
Inserting the GCC into the laser bay proved to be challenging.

- Seven 10-ft lengths of the 15-ft × 15-ft chamber sections were delivered from California to New York.
The GCC contains critical optics for the success of the OMEGA EP project

- 14 custom fabricated optical tables
- >100 individual optics
- 32 high-fluence beamline optics
- 8 multilayer-dielectric-tiled-grating assemblies (TGA’s) (each assembly hold 3 gratings)

The optics and chamber are a challenge to clean and keep clean to LLE specifications.
Tiled multilayer-dielectric- (MLD) diffraction gratings are required to produce 2.6-kJ output IR energy per beam at 10 ps.

- Each tiled-grating assembly (TGA) is comprised of three multilayer-dielectric- (MLD) gratings consisting of alternating layers of HfO₂ and SiO₂.

- Grating specifications
  - Dimensions: 43 cm × 47 cm × 10 cm
  - Grating period: 1740 lines/mm
  - Diffraction efficiency: >95%
  - Wavefront (p–v): <λ/4 at 1054 nm
  - Damage threshold: >2.7 J/cm² at 10-ps pulse measured beam normal
Metal components are cleaned and tested before being deployed into the GCC.

Incoming part

- Component electropolished, passivated, or plated
  - Yes: IPA\(^2\) wipedown for bay entry
  - No: Spot/local passivation?
    - Yes: Citrisurf\(^1\) component
    - No: Size dependent
      - Yes: LaCave ultrasonic cleaning
      - No: High-pressure wash

- Precision wipedown
  - Size dependent
    - Precision pressure wash
      - Size dependent
        - Precision ultrasonic wash (OAA)
      - NVR
        - PASSED: Release for installation
        - FAILED

\(^1\) Citrisurf product sheet, Stellar Solutions, Inc., McHenry, IL 600500 (http://www.stellarsolutions.net).
\(^2\) IPA = Isopropyl Alcohol
The internal vacuum surfaces and components were cleaned to meet the IEST-STD-CC1246D cleanliness and NVR standards.

- Surfaces were cleaned using either a high-pressure (3000 psi) spray of 5% surfactant solution in heated (120°F) DI water clean or a precision ultrasonic (40 kHz) wash process.
- Nonvolatile residue (NVR) analysis was performed to determine if the vessel passed the IEST-STD-CC1246D A/10 specification: \( A/10: <0.10 \text{ mg/ft}^2 \)
- Bright/UV light inspections were performed to understand particle contamination along with extractive sampling.
Individual materials were tested for vacuum compatibility using an HP 5988A CG-MS system

- Ion source: electron impact (EI)
- Mass filter: single quadrupole
- Mass range: 10 to 1000 amu
- Mass accuracy: ±0.13 amu
- Dynamic range: $2 \times 10^6$
- Detection limit: 500 picograms
- Scan speed: 2000 amu/s

<table>
<thead>
<tr>
<th>Sample Inlet</th>
<th>Sample Form</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP 5890 GC (liquid injection)</td>
<td>“Neat” material or solution</td>
<td>Surface contamination analysis (solvent extract)</td>
</tr>
<tr>
<td>HP 5890 GC (“Headspace”)</td>
<td>Solid or liquid</td>
<td>Volatile component analysis (outgassing)</td>
</tr>
<tr>
<td>Direct-insertion probe</td>
<td>Solid, viscous liquid</td>
<td>Vacuum outgassing</td>
</tr>
</tbody>
</table>
Materials were tested for vacuum compatibility using the direct-insertion-probe method before they were accepted for deployment in the GCC

- The top trace shows the total ion chromatogram (TIC), which is the total abundance of all ions evolved as a function of time. Each point on the TIC is a complete mass spectrum.
- This sample shows a large amount of volatile organic material evolved approximately 25 min into the run.
- The lower trace shows the mass spectrum obtained at the peak maximum of the TIC, which indicates the largest concentration of volatile components present.
- In order for the sample to be acceptable for vacuum use, it must not show any ion abundance signal in excess of 1000 amu (as indicated by the “red” zone in the figure).
Components and assemblies were tested for vacuum compatibility using a Hiden Analytical Mass Spectrometer.

- A 28-in. oval vacuum chamber equipped with a Hiden Analytical HPR30 manifold system integrated with an HAL 12501-9 RC triple-filter quadrupole mass spectrometer.

- The system utilizes a dual Faraday/single-channel, electron multiplier detector configured for low mass (up to 300 amu) and high mass (up to 1500 amu).

- An internal heater within the chamber to heat components during outgassing analysis and to bake out components before deployment.
Components were tested for vacuum compatibility before they were accepted for deployment in the GCC.

- Unlike the previous method, concentration of ionic species in terms of the partial pressure of residual gases are reported rather than the TIC.
- Individual mass spectra are reported as a mass-to-charge ratio (m/e) of the resulting fragments.
- Components were qualitatively tested for vacuum material outgassing.
- Samples were heated and analyzed for 24 h or longer.
- Samples exhibiting peaks $>1 \times 10^{-10}$ torr after 24 h fail the test.
Components that failed the outgassing or the NVR analysis were either recleaned or refabricated.

Hydrocarbon signature during outgassing test. Sample was recleaned to remove residual surfactant from the clean process.

Component was recleaned twice then analyzed again. Hydrocarbon signature was gone.
Grating-surface cleanliness is of paramount importance to achieve high-laser-damage thresholds. A previously developed piranha \((\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2)\) cleaning process\(^1\) will be used to periodically clean the large-scale diffraction gratings.

**DOE Cleaning Results: 10 ps N-on-1**

Total time = 30 min (Bath 1: 20 min/Bath 2: 10 min)

![Graph showing damage threshold vs. bath temperature]

Damage threshold (J/cm\(^2\))

\[50 \quad 60 \quad 70 \quad 80 \quad 90 \quad 100 \quad 110\]

Bath temperature (C)

LLE specification: \(>2.7 \text{ J/cm}^2\)

\[2.0 \quad 2.2 \quad 2.4 \quad 2.6 \quad 2.8 \quad 3.0 \quad 3.2 \quad 3.4 \quad 3.6\]

**Piranha 4:1**

**Piranha 2:1**

**Piranha 6:1**

Cleaning diffraction gratings is critical to achieving and maintaining high-laser-damage thresholds (LDT’s).

Periodic cleaning of the OMEGA EP gratings will maintain high-laser-damage thresholds.

- Required LDT for OMEGA EP is 2.7 J/cm² at 10 ps
- Actual LDT is monitored by *in-situ* coupon samples

Automated Acid Piranha Process (H₂SO₄:H₂O₂)

3.3 J/cm² at 10 ps has been achieved on small samples.
Grating-damage-threshold degradation from exposure to the GCC environment is a serious concern

- Actual laser-damage threshold is monitored by *in-situ* MLD grating coupon.

- The *in-situ* coupon samples (previously piranha cleaned and tested) are recovered periodically from the GCC and retested.

- If there is a significant decrease in damage threshold, the large-aperture gratings are taken out and piranha cleaned and redeployed into the GCC.

Results of the first test were positive with no significant damage-threshold degradation.
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The OMEGA EP Laser System is complete and initial experiments are underway
Acknowledgment

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