Evaluation of outgassing contamination effects on optical surfaces of the LIL

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ABSTRACT

The Ligne d’Intégration Laser (LIL) is a prototype installation at scale 1 of one of the 30 lasers of future Laser Méga Joule. It is intended to validate technological choices made for LMJ and to prepare its exploitation. The facility will contain nearly 10,000 optics and over 4000 m² of mirrors. Cleanliness will be an essential matter in the facility since contamination of optics can reduce their laser damage threshold. Hence, airborne molecular contamination (AMC) has been sampled near optics in strategic places of the LIL. These samplings have shown high levels of organic compounds, notably in the amplifying section, which is expected to be the most sensitive part in the LIL. Suspecting a local source of contamination, outgassing tests of typical materials constituting the amplifying section have been carried out. Among them, one sealing material has been identified as a source of organic contamination near the optics. Effects of this pollution have been investigated by a measurement of laser damage threshold after intentional contamination of optics. This work shows the complexity of the outgassing contamination issue, since several steps are necessary to evaluate the effects of this contamination on optical surfaces: air samplings, identification of sources, outgassing tests, intentional contamination of optics and finally measurement of laser damage threshold.

Keywords: contamination, outgassing, LIL, LMJ, optics, surface.

1. INTRODUCTION

The French Atomic Energy Commission (CEA) has started to lay the foundations of the building that will house Europe’s biggest laser, known as Laser Méga Joule (LMJ). LMJ will consist of 30 separate lasers, each containing eight beams, and will deliver a total energy of 1.8 MJ. The beams will be focused onto micron-sized targets made of hydrogen atoms to create the extreme conditions of temperature and pressure that exist inside a thermonuclear weapon when it explodes. Reactions of the same type take place in the core of stars such as our sun. The Ligne d’Intégration Laser (LIL) is a prototype installation at scale 1 of one of the 30 lasers of future Laser Méga Joule. It is intended to validate technological choices made for LMJ and to prepare its exploitation.

In order to direct and amplify the laser signal, numerous optics are necessary. The facility will contain nearly 10,000 optics and over 4000 m² of mirrors. Contamination is a critical issue for the performances of those optics. Two fields in particular have studied first airborne molecular contamination on optics: microlithography and aerospace. For them, Airborne Molecular Contaminants (AMCs) are most conveniently classified as:

- Acids: corrosive materials whose chemical reaction characteristic is that of an electron acceptor,
- Bases: corrosive materials whose chemical reaction characteristic is that of an electron donor,
- Condensables: chemical substances, typically having a boiling point above room temperature at atmospheric pressure, capable of condensation on a clean surface (excluding water),

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As the principal source of AMCs is outgassing from materials present in closed environment, several researches have been carried out to identify and quantify the compounds emitted by typical materials used in microlithography and aerospace: seals, gloves, resins, packages of wafers, plasticizers, foams, oils and coatings constituting the Hubble satellite. In particular, photodegradation of polymers under UV light has been largely studied. Condensables include organic compounds with low volatility. Among them, phthalates, organosilicones, and aromatics have been proved to cause serious damages to optics by deposition on surfaces. To show the effects of these contaminants on signal transmission, some authors have intentionally contaminated optics with typical contaminants.

Indeed, even if airborne molecular contamination in confined environments (such as cleanrooms, microlithography units, space modules) has aroused more and more interest for the last decades, it has been only recently discovered that high power lasers can suffer from this kind of contamination. Actually, contamination of optics constituting a high power laser can result in a formation of a haze on optics, a degradation of the coating, reduce the optics performances such as laser damage threshold or transmission, and reduce laser signal itself.

The aim of this study was to evaluate the outgassing contamination effects on optics originating from the LIL. This work divides into several parts: first, air samplings were carried out inside different parts of the LIL, that permitted to identify the sources. Then outgassing tests of typical materials were carried out to identify the principal contaminants. Optics were then intentionally contaminated by a forced outgassing of one material, and finally this study was concluded by a measurement of laser damage threshold.

2. ORGANIC CONTAMINATION IN STRATEGIC PLACES OF THE LIL

2.1 Sampling and analytical conditions

Organic measurements in the air were done using TENAX TA (60/80 mesh, Macherey & Nagel) cartridges, connected to a pump and a mass flow meter. Before using the Tenax TA, it was purified with methanol inside a soxhlet extractor and subsequently dried. One hundred milligrams of Tenax TA were packed into each tube and fixed with silanized glass wood. The adsorption tubes were conditioned for 5 h at 300°C by flushing them continuously with nitrogen (99.999 % purity) at 10 ml/min. Both ends of the adsorption tubes were closed with Swagelock fittings.

Each sampling lasted 30 min at a flow of 100 ml/min corresponding to a 3 L air sample. When a sample was obtained, the cartridge was closed and kept at a temperature of 5°C until analysis.

TENAX cartridges were analysed using a gas chromatograph coupled with a mass spectrometer (Perkin Elmer Turbomass). The adsorption tubes were placed inside an automated thermal desorption device (Perkin Elmer ATD 400) and thermally desorbed by flushing the heated tubes (320°C) for 10 min. The desorbed organic compounds were cryogenically trapped at –30°C. By heating up the trap to 300°C, the contaminants were transferred to the gas chromatograph and the sample was injected into the chromatographic column. Analytes were separated with a 60 m * 0.32 mm fused silica column with a 0.5 μm PM-5MS stationary phase. For the Gas Chromatograph (GC), the initial oven temperature was set at 50 °C, and then ramped to 250 °C at a rate of 5 °C/min, followed by a holding at 250 °C for 2 min. Helium was used as carrier gas at a flow rate of 1 mL/min.

2.2 Sampling Sites

Airborne organic contamination has been sampled near optics of the LIL. Sampling sites were strategically chosen to be the most representative of the main parts of the LIL.

A scheme in figure 1 summarizes the different parts of the LIL and the sampling sites.
2.3 Results
The results obtained for the samplings done in typical environments of the LIL are reported in table 1.

<table>
<thead>
<tr>
<th>Sampling Sites</th>
<th>Average Concentration in ppbv</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre-Amplifying Module (MPA)</td>
<td>34.7</td>
</tr>
<tr>
<td>Injection</td>
<td>21.2</td>
</tr>
<tr>
<td>Demi-tour (about-turn)</td>
<td>12.5</td>
</tr>
<tr>
<td>Transport Amplifier</td>
<td>55.9</td>
</tr>
<tr>
<td>Cavity Amplifier</td>
<td>41.8</td>
</tr>
<tr>
<td>End of Cavity</td>
<td>57.4</td>
</tr>
<tr>
<td>Transport Mirror MT1</td>
<td>16.8</td>
</tr>
<tr>
<td>Frequency Conversion System (SCF)</td>
<td>30.3</td>
</tr>
</tbody>
</table>

Table 1: Average concentrations in organics (Total Volatile Organic Compounds = TVOC) at different places of the LIL.

We immediately notice that there is a large scale of contamination levels in the LIL. The less contaminated places are the transport zone, the 3ω zone and the laser source. However, the amplifying section shows high levels of organic compounds (from 40 up to 200 ppbV), notably for Injection and Demi-Tour. To make a comparison, recommended values for micro-electronic fabs are 20-40 ppbV of organic condensables (condensables only represent a part of TVOC measured) and recommended values for DUV photolithography are less than 1 ppbV.

The TVOC measurements give global information on airborne organic contamination levels, but they are not necessarily representative of the risks to which the optics are exposed. Indeed, it has been proved \(^ {3, 5, 14, 16, 19}\) that some chemical species are more dangerous than others: siloxanes, aromatics and phthalates in particular can deposit easily on optics because of their low volatility. That is the reason why it is interesting to identify the main chemical families present. An example of chromatogram obtained for organic measurements is given in figure 2. This chromatogram shows that several species were identified: aromatics (benzen, toluene), siloxanes and alcohols for example.

Once the principal contaminants had been identified in several sites of the LIL, the next step was to find the sources of this contamination.
3. OUTGASSING TESTS OF TYPICAL MATERIALS

Outgassing from materials is one of the principal sources of contamination in closed environments\textsuperscript{1-6}. In this study, we focused our work on the essential and more sensitive part of the LIL: the amplifying section. Therefore, outgassing tests of typical materials constituting the amplifying section were carried out.

3.1 Experimental conditions

The analytical method used for outgassing measurements was inspired by several industry standards:

- ECSS-Q-70-02A : “Thermal vacuum outgassing test for the screening of space materials”

Outgassing conditions were as follows: about 1 cm\textsuperscript{2} of material was heated in an inox tube at 50°C for 30 minutes. The organic compounds emitted were analysed by TD-GC-MS for identification and GC-FID for quantification.

Nine materials present in the amplifying section were tested:

- fluoroelastomer seal (Viton®)
- PTFE seal
- chlorosulfonated PE (Hypalon®)
- sealing paste (Voranol®)
- aluminium self adhesive tape
- 40 shores cast silicone seal
- 40 shores extruded silicone seal
- 60 shores cast silicone seal
- 60 shores extruded silicone seal

3.2 Results

The results are presented in figure 3.
Outgassing rates of typical materials of the LIL (in 30 min at 50°C). The three materials showing the highest outgassing rates are: 40 shores cast silicone seal, aluminium self adhesive tape, and 60 shores extruded silicone seal. Among them, 60 shores extruded silicone seal was suspected to be a potential source of contamination of the amplifying section, notably by the nature of the organic compounds emitted, which were also identified in the amplifiers. Indeed, figure 4 shows that identical compounds were observed between the sampling made in the end of cavity and the outgassing test carried out for 60 shores extruded seal.

If we look at the compounds emitted by this seal, we notice that it is able to emit dangerous organic compounds:
- dangerous because of their physical and chemical properties: they are sticky compounds, with low volatility and deposit easily on surfaces (for example: cyclopentasiloxane, decamethyl, or benzyl alcohol).
4. INTENTIONAL CONTAMINATION OF OPTICS AND LASER TEST

4.1 Experimental section
Typical optics of the amplifying section were intentionally contaminated by outgassing the 60 shores extruded seal. The aim of this experiment was to simulate optics contamination phenomena in order to determine contamination kinetics and surfaces sensitivity (interactions substrates / chemical species). Moreover, the impact of this contamination was also examined since a laser damage test completed this experiment.

The scheme of our system is presented in figure 5. The material was outgassed at 50°C and the gas charged with contaminants was directed towards the mirror (maintained at 20°C in order to help condensation of organics on the surface).

![Scheme of the system permitting intentional contamination of optics by outgassing a material.](image)

Three dielectric mirrors were used. Mirror MEG 23 H served as a reference, since it was exposed 1 hour to the system, but without the contaminating material, in order to characterize the contamination emanating from the system itself. Mirror MEG 23 I and mirror MEG 23 J were exposed respectively 3 hours and 15 hours to the outgassing material airflow.

![Scheme of the system permitting intentional contamination of optics by outgassing a material.](image)

4.2 Surface contamination
Thermodesorption of the mirrors, carried out in a specific chamber (temperature ramp from 30°C up to 350°C for 100 min, under ultraclean N₂ flux), followed by a tenax sampling, permitted to identify and quantify the contamination deposited on their surfaces. Among the 27 principal organic compounds identified, 21 originated from the 60 shores extruded seal, proving that the mirror was well contaminated by the components outgassed from the material.

We observe a global increase of the contamination after 3 hours (about 15 % more), but the contamination seems to be levelling off after 15 hours (figure 6).

But if we look more closely at the evolution of the different chemical species (figure 7), we notice that they do not have the same evolution in time. Aromatics and siloxanes seem to stay quite stable after 15 hours of exposition. It seems that these contaminants remain on the surface. However, alcanes and alkenes seem to diminish in favor of oxidized compounds such as aldehydes, alcohols and acids. This could be explained by different deposition kinetics according to the chemical nature of the contaminants and the surface reactivity.
4.3 Laser Damage Threshold measurement

The effects of this contamination were investigated by a measurement of Laser Induced Damage Threshold (LIDT). Laser tests were carried out at 1.064 µm, 45°s polarization, with an automatic damage test facility, which has been previously described. Multiple pulses of increasing energy are sent on a mirror site until a scattered light diagnostic detects a certain threshold of diffused He-Ne light. This test is called R/1, ramp on a site. With an automatic test bench, more than one hundred sites are targeted on a 50 mm diameter sample, which gives a statistical weight to the peak fluence, in the laser beam, at which the damage occurred. R/1 damage threshold is always higher than 1/1, one shot on a site, damage threshold because the mirror is optically conditioned as well as damage tested due to the accumulation of increased fluence shots on a site. Under the term “conditioning” lie multiple physical effects, not always understood, in which “laser cleaning” could be added. Any difference of laser damage threshold that can be measured in a R/1 laser damage test would be measured with a more drastic effect in any other laser induced damage test. Results are visible on figure 8. LIDT is about 15 % lower after 3h of exposure to the forced material outgassing, but no visible effect is observed after 15 hours of exposure. However, we notice that these results are correlated with the mass of organic contamination deposited on the surface of the mirror. The degradation of LIDT induced by contamination of the mirror surface was already observed with the forced outgassing of polypropylene boxes at 80 °C. The degradation was more pronounced but the contamination mass was also 2.5 times what is presented in this work. Once again we show that very small amount of contamination can induce the degradation of LIDT of very high resistance laser mirrors. That such degradation can be induced by outgassing volatile contaminants at near “room temperature” will lead to a more stringent choice of materials inside the Megajoule Laser.
5. CONCLUSIONS

Effects on optics of contamination originating from a typical material present in the LIL were investigated by a laser induced damage threshold test. To achieve this study, it was necessary to identify a potential source of contamination present in the LIL among the different materials constituting the system. Therefore, airborne organic contamination was sampled near optics inside the different sections of the LIL. These samplings revealed high levels of contamination in some places, notably dangerous species such as siloxanes, aromatics or phthalates, which can deposit easily on optics because of their low volatility. A comparison between contaminants identified by these samplings and contaminants emitted by materials outgassing was carried out and allowed to identify a silicone seal as a potential source of contamination on optics. The laser induced damage threshold measurements confirmed that this material has an impact on optics performances. Further outgassing experiments are scheduled and will allow us to better understand the deposition mechanisms. Complementary tests, and particularly ellipsometric measurements, will help evaluating the real impact of surface contamination thickness and decrease of optics performances.

REFERENCES