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Optics and photonics — Lasers and laser-related equipment — Laser-induced molecular contamination testing

Qualification des composants optiques laser pour les applications spatiales

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Foreword

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This document was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 9, *Electro-optical systems*.

Introduction

Laser technique is becoming increasingly important for space applications. Complex laser systems are used both for Earth observation and for planetary exploration. For long-term operations, optical components have to satisfy stringent requirements concerning precision and reliability. Before being used in space, all optical components have to be tested extensively. For standardized determination of laser damage threshold, ISO 21254 (all parts) should be applied. For characterization of optics for space applications, corresponding tests should be performed under vacuum conditions. In addition to laser damage issues, laser-induced molecular contamination (LIMC) should be taken into account. LIMC denotes the interaction of laser radiation, especially in case of high fluences and short wavelengths with volatile molecules and the resulting formation of deposits on optical components. LIMC proved to be particularly critical, if the laser system is operated under vacuum conditions and could considerably reduce the functionality of the whole laser system. Molecular contamination is mainly caused by organic materials and silicones, e.g. glues, adhesives, insulating material or circuit boards due to stronger outgassing rates compared to inorganic materials. The outgassing can be reduced but not totally prevented by selection of suitable materials and preconditioning, e.g. bake-out at elevated temperature well above the planned operating temperature. The outgassing behaviour of materials is generally characterized by these parameters: collected volatile condensable material (CVCM), total mass loss (TML), recovered mass loss (RML), volatile condensable material (VCM) and water vapour regained (WVR). Definitions and corresponding measuring specifications for these quantities can be found in ECSS-Standard Q-ST-70-02C, ASTM-E595-07 and ASTM-E1559.

This document outlines the test procedure for investigations of laser-induced molecular contamination in order to compare the growth of laser-induced depositions on optical surfaces for different molecular contamination materials.

Optics and photonics — Lasers and laser-related equipment — Laser-induced molecular contamination testing

1 Scope

This document describes the setup, test procedure and analysis of measured data for investigation of laser-induced molecular contamination (LIMC) for space and vacuum applications.

LIMC is the formation of depositions on optical surfaces due to interaction of intense light radiation with outgassing molecules especially from organic materials. It is a phenomenon of molecular contamination and it is distinguished from particle contamination, which can occur during manufacturing, assembly, integration or test of the optical components.

Formation of laser-induced depositions can lead to deterioration of the performance of an optical system. Phase distortion, scattering and absorption can be increased by LIMC. LIMC is of particular relevance, if a laser system is operated in vacuum at short wavelength and short pulse duration. In such a case, even small partial pressure of contamination material in the range of 10^{-5} hPa could have strong negative impact on optical performance. It was also shown that the laser-induced damage threshold could be reduced by a factor of 10 and more if laser-induced depositions are involved.

Laser-induced molecular contamination and laser-induced damage are both phenomena, for which the interaction of laser radiation with optical surfaces plays a major role, in case of LIMC with additional molecular contamination. Therefore, this document is treated in relation to ISO 21254 (all parts) which specifies the test methods for the determination of laser-induced damage thresholds.

This method was derived to evaluate qualitatively, whether the material under investigation causes deposits on optical surfaces in a low-pressure environment in the presence of high-energy nanosecond pulsed laser irradiation at a wavelength of 355 nm. Due to the nature of photochemical surface reactions, this result cannot be directly transferred to scenarios where the properties of the irradiation are altered (especially wavelength, repetition rate, pulse duration, etc.). Due to the non-linear growth of the laser-induced contamination and its detection methods, this technique does not provide quantitative means to evaluate the deposit and, therefore, it should be seen as a means to compare materials relatively with respect to their laser-induced contamination behaviour.

Furthermore, it is out of the scope of this method to select representative quantities of contamination materials — representative with respect to the material partial pressure present in the vicinity of the optical surface in a real laser system. This is carefully derived with other methods and is a mandatory parameter to be fixed before applying this method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11145, *Optics and photonics — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 21254 (all parts), *Lasers and laser-related equipment — Test methods for laser-induced damage threshold*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11145 and ISO 21254 (all parts) and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
laser-induced deposition
material growth on optical surfaces as a result of photochemical or photothermal mechanisms triggered by the interaction of laser radiation with volatile molecules from outgassing process of especially organic materials

3.2
laser-induced fluorescence
light emission from any substance that has been excited to singlet states by absorption of electromagnetic radiation

4 Symbols and abbreviated terms

Symbol	Unit	Term
α	rad	Angle of incidence
d	m	Beam diameter
N_p	—	Number of pulses per site
H_{peak}	J/m^2	Peak energy density
τ	s	Pulse duration
f_p	Hz	Pulse repetition rate
E	J	Pulse energy
p	Pa	Pressure
T_c	K	Contamination temperature
λ	m	Wavelength

5 Test method

5.1 Test setup

5.1.1 Vacuum chamber

A typical setup for tests of anti-reflective optics (angle of incidence: 0°) is shown in Figure 1. The main part is an ultra-high vacuum chamber. It should be ensured that no organic materials are present in this chamber. Especially an oilfree pumping system and metallic sealings instead of plastic o-rings should be used. In particular, the use of silicone-based materials (e.g. pump oils) should be absolutely avoided. In general, silicones have a very low vapour pressure and it is very laborious to get rid of it once the test chamber is contaminated with it. Moreover, silicones are known to produce laser-induced depositions even at ambient or oxygen atmosphere. In contrast to this, for hydrocarbons, the formation of laser-induced depositions is strongly reduced or totally inhibited by oxygen at least in case of UV laser radiation. The vacuum chamber is composed of copper-sealed stainless steel components, e.g. according to ConFlat (CF) standard. The pumping system consists of a turbomolecular pump and an oilfree forepump (e.g. scroll pump). For pressure sensing Pirani, Penning or capacitance manometer should be used. Additionally, a quartz crystal microbalance (QCM) sensor and/or a mass spectrometer can be integrated for determination of outgassing rate and composition of contamination material. By using a mass spectrometer, it is possible to determine the partial pressure of the contamination material in the main chamber, provided that the total pressure is below 10^{-5} hPa. If the total pressure is higher, a differentially pumped mass spectrometer should be used. The mount for the optical sample under test is positioned at the centre of the vacuum chamber. This mount is movable in horizontal direction by a vacuum feedthrough. This enables execution of sequential tests on different positions on the sample surface without breaking the vacuum. The contamination sample to be tested is located in a small source chamber which is separated by a needle valve from the main chamber. The contamination chamber can be heated from outside by electrical heating bands for acceleration of the outgassing. The mass flow into the main chamber should be regulated by a needle valve to enable a temporally constant partial pressure of contamination material in the main chamber. A modified test setup for investigation of 45° high-reflection (HR) optics is shown in Figure 2.

5.1.2 Laser source and optical beam line

As already mentioned in the introduction, LIMC is especially a problem in case of UV irradiation. Therefore, in the following, a laser source and an optical beam line for pulsed 355 nm light are described. But with other laser sources and corresponding beam line optics, the LIMC tests can also be performed for other wavelengths, either pulsed or cw. For investigation of LIMC in the UV, a pulsed Nd:YAG laser with subsequent frequency doubling and tripling unit can be used. To create the required beam diameter, a Galileo's telescope could be installed. By an optical attenuator consisting of a half wave plate and a polarizer, the pulse energy and power, respectively, can be changed without major impact on the beam profile. By a 50 % beam splitter, two identical beams can be created. One of the two beams is the actual measuring beam for the optical sample under test; the other beam is used as a reference. It passes the vacuum chamber but not the optical sample. Focusing lenses ensure that the beam diameter on the windows is larger than on the sample. Consequently, the laser power density on the windows is lower than on the sample, and contamination is mainly formed on the sample and not on the windows. For further reduction of contamination growth, the windows should be heated. Standard vacuum compatible window flanges can be heated up to 200 °C.

Through additional beam splitters, the beams can be further divided, such that several samples can be tested simultaneously.

5.2 Setup for data acquisition

5.2.1 Beam profile monitoring

Prior to the test, a wedge is brought into the beam line between the focusing lens and the vacuum chamber (see Figure 3). The beam profile is monitored by a CCD camera, which is positioned in such a way that the optical path length, l_1, between the wedge and the camera is the same as between the wedge and the optical sample, l_2, so that the measured beam profile represents the beam profile at the position of the optical sample. For determination of the background, a measurement with blocked beam should be performed. By fitting an elliptical Gaussian profile to the background corrected data points, the minor and major axes of the fitted profile at $1/e^2$ of intensity should be evaluated. The effective beam diameter d_{eff} is the geometric mean of minor axis a and major axis b as shown in Formula (1):

$$d_{eff} = (a \times b)^{1/2} \quad (1)$$

5.2.2 Measurement of pulse energy and determination of transmission

Through an optical wedge, a part of the undivided beam is coupled out and used for online monitoring of pulse energy. The entrance energy on the optical sample can be obtained from these data through a calibration using an additional detector placed in the beam line between focusing lens and vacuum chamber (see Figure 4). From this, a calibration factor, $F_1 = E_4/E_1$, can be derived. For calibration of the exit pulse energy, a measurement with detector 3 is performed initially (see Figure 5). Therefore, a detector 5 is brought into the direct exit beam and the calibration factor $F_2 = E_5/E_3$ is obtained. During the LIMC test, online measurements with detectors 1 and 3 enable the determination of transmission values: $T = E_5/E_4 = (F_2/F_1) \times (E_3/E_1)$. Corresponding calibration has to be performed for the reference beam. The transmission losses by the windows should be derived from the reference beam data and should be used for correction of the transmission values for the measuring beam to receive the transmission values for the optical sample.

5.2.3 Laser-induced fluorescence monitoring

If LIMC tests are performed with UV laser light, laser induced fluorescence should be used for *in-situ* monitoring of deposit growth. Once organic material is deposited on the surface, it is stimulated by the UV laser to emit fluorescence light. Most suitable for fluorescence light detection are CCD cameras with integrated electron multiplier (EM-CCD). These cameras have a very high signal-to-noise ratio and a large dynamic range. During LIMC test, fluorescence pictures are recorded in regular time intervals (Δt = 1 min to 10 min). For suppression of scattered UV light, a corresponding filter should be implemented in recording optics. Figure 6 shows a typical fluorescence picture. For a quantitative analysis, the fluorescence intensity values of all pixels in an area, B_1, containing the fluorescence spot are summed, giving I_1. For background determination, a corresponding intensity sum I_2 is calculated for a region, B_2, which has the same area as, B_1, but is located outside the fluorescence spot. Background corrected intensity sum $I = I_1 - I_2$ should be plotted in dependence on irradiation time as a measure for deposit growth.

5.3 Test preparation

5.3.1 Bakeout

To avoid cross contamination by residual contamination material from preceding tests, the vacuum chamber has to be baked out prior to each new test sequence. Therefore, the chamber is wrapped with heating bands as completely as possible to ensure homogenous temperature distribution. The use of additional flange heaters facilitates an effective heating of massive flanges and avoids heat sinks. Maximum bakeout temperature depends on the components of the vacuum chamber, in particular, windows, pressure sensors and other measuring devices should be kept in mind. Heat sensitive cables and sensor housings should be removed before bakeout if possible. Furthermore, possible restrictions to temperature ratings, especially for the windows should be noted. If this is taken into account, metal sealed vacuum chambers are heatable up to 200 °C. Duration of bakeout should be at least 24 h or more. The pumping system should be running during bakeout. The removal of outgassing molecules can be improved by purging the vacuum chamber during bakeout with dry nitrogen. The pressure in the vacuum chamber should be lower than 10^{-8} hPa after bakeout. In order to check the effectivity of the bakeout, a blank test (see 5.3.3) should be performed thereafter.

5.3.2 Pretreatment of optical samples

Prior to the tests, the optical samples should be cleaned. For removal of dust and adhesive particles, the optical sample should be blown with dry nitrogen. Then, the optic should be cleaned by drop and drag procedure: through a pipette, a drop of ultrapure acetone is applied to a lens cleaning paper. The wet part of the paper is put on the optic and slowly drawn over the surface until the optic and paper are almost dry.

In addition, the optical sample could be cleaned by ozone. Ozone should be produced by UV light, e.g. by a mercury lamp. The optical samples are brought in vicinity of the mercury lamp and exposed up to 12 h to ozone.

After cleaning, the optical sample is inspected by differential interference contrast (DIC) microscopy (magnification at least 100 times). If any residual pollution or streaks are detectable, the cleaning procedure should be repeated.

5.3.3 Blank test

Prior to LIMC tests, a so-called “blank test” has to be performed to ensure that the vacuum chamber is free of residual organic material from former LIMC tests. The blank test is run without any contaminant but in other respects (fluence, wavelength, repetition rate, beam profile and duration) under identical conditions like the scheduled LIMC test. For the blank test and the LIMC test, the same optical sample or samples from the same batch should be used. Only if no contaminations are detectable, the blank test can be considered as successful. Otherwise, the bake out has to be repeated.

5.4 LIMC test

5.4.1 Test parameters

The test should be characterized by the following parameters:

a) laser wavelength, λ;

b) pulse duration, τ;

c) pulse repetition rate, f_p;

d) beam diameter in the target plane, d;

e) pulse energy, E;

f) angle of incidence, α (e.g. 0° for AR and 45° for HR coated samples);

g) temperature of contamination source, T_c;

h) partial pressure of contaminant gas, p;

i) duration of irradiation, Δt;

j) dimensions of optical sample (thickness, diameter);

k) coating of optical sample (high-reflective, anti-reflective, etc.).

5.4.2 Individual steps for LIMC test

The LIMC test should be performed by following this procedure:

— cleaning of optical sample (see 5.3.2);

— mass determination of contamination sample;

— mounting of optical sample into vacuum chamber;

— insertion of contamination material to contamination source;

— pumping down of vacuum chamber ($p < 10^{-6}$ hPa);

— determination of beam profile and beam caustic (see 5.2.1);

— calibration of energy sensors (see 5.2.2);

— setting of contamination pressure;

— adjustment of laser energy density;

— start of irradiation.

During LIMC test, the following data should be recorded online:

— transmission and reflection respectively;

— laser-induced fluorescence(see 5.3.2);

— pressure;

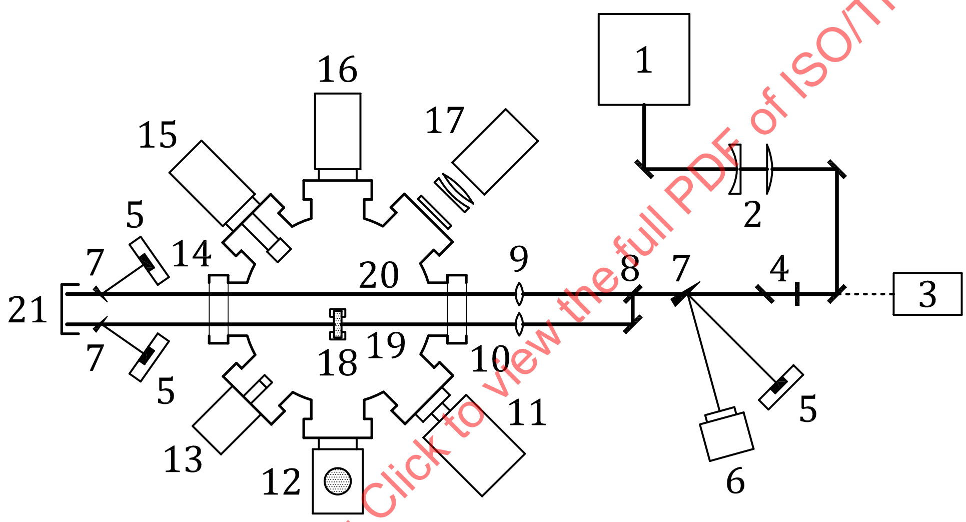
— temperature;

— outgassing rate (QCM).

After the test is completed, the optical samples should be investigated in detail *ex-situ*. Laser-induced depositions on the optical samples should be inspected with DIC and fluorescence microscopy. Fluorescence microscopy is a sensitive tool for detection of organic materials. Even thin films with thickness in the range of some nanometers can be detected. For analysis of surface structure, white light interference and atomic force microscopy could be used. Chemical composition of laser-induced depositions could be analysed by Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) or other surface analysing methods.

5.5 Evaluation of test results

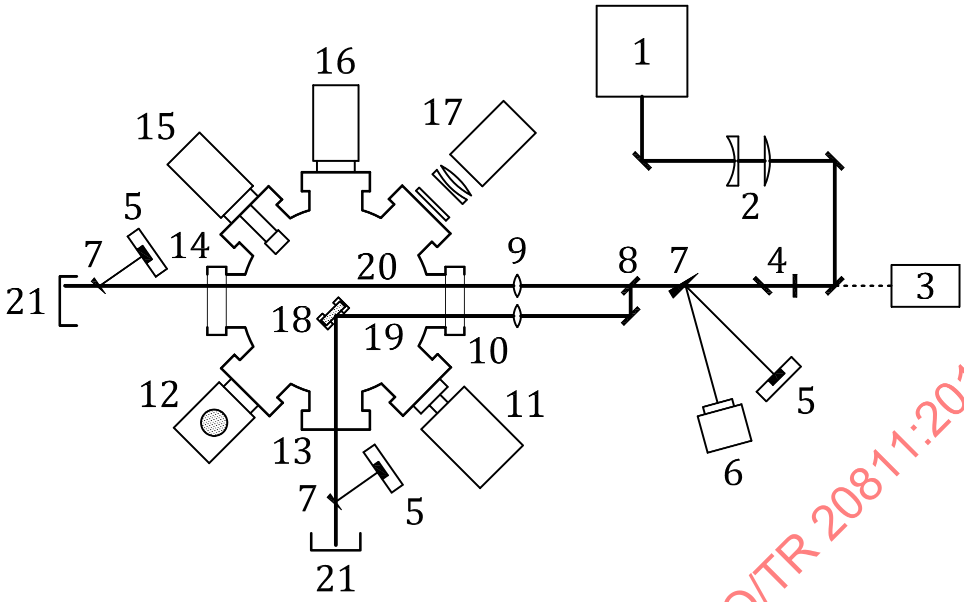
Decrease of reflection for high-reflective coated optics or decrease of transmission for anti-reflective coated optics and increase of fluorescence intensity during LIMC test provides clear indication for the formation of laser-induced depositions. Through a comparison of reflection or transmission decrease and fluorescence intensity for different optics tested in the same run or under identical conditions, the performance of the tested optics with respect to laser-induced molecular contamination can be assessed. Abrupt degradation of transmission or reflection is clear evidence for damage of the coatings potentially enhanced by formation of laser-induced depositions.



Key

1 laser source
2 telescope
3 He-Ne Laser
4 attenuator
5 energy detector
6 CCD (beam profiling)
7 wedge
8 beam splitter
9 focusing lenses
10 entrance window
11 pressure sensor
12 contamination source
13 quartz crystal microbalance
14 exit window
15 mass spectrometer
16 pumping system
17 EM-CCD (fluorescence monitoring)
18 optical sample
19 measuring beam
20 reference beam
21 beam dump

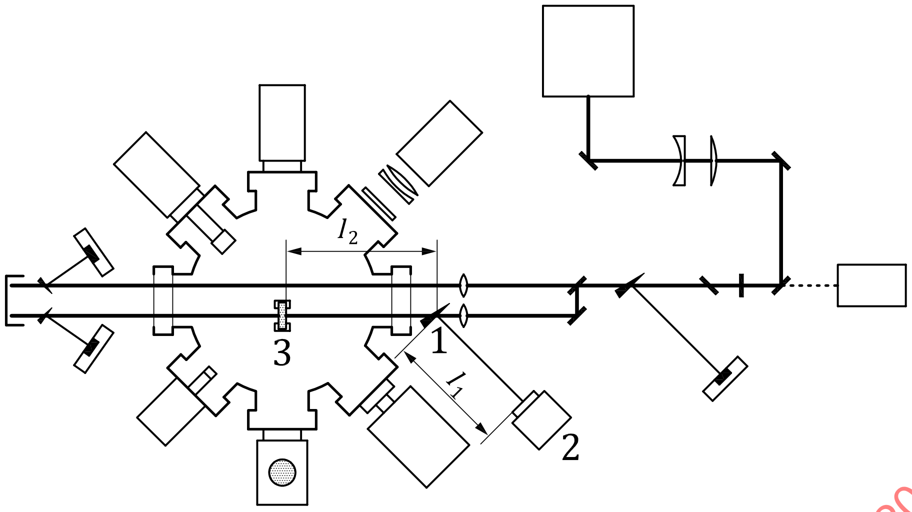
Figure 1 — Experimental setup for LIMC tests of AR optics



Key

1	laser source	12	contamination source
2	telescope	13	exit window
3	He-Ne Laser	14	exit window
4	attenuator	15	mass spectrometer
5	energy detector	16	pumping system
6	CCD (beam profiling)	17	EM-CCD (fluorescence monitoring)
7	wedge	18	optical sample
8	beam splitter	19	measuring beam
9	focusing lenses	20	reference beam
10	entrance window	21	beam dump
11	pressure sensor		

Figure 2 — Experimental setup for LIMC tests of 45° HR optics



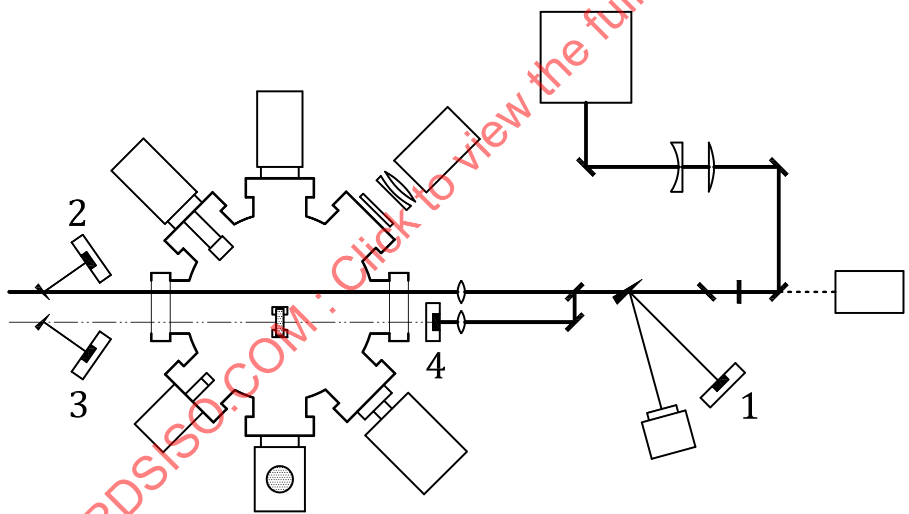
Key

1 wedge

2 CCD-camera

3 optical sample

Figure 3 — Beam profile measurement



Key

1 energy detector 1

2 energy detector 2

3 energy detector 3

4 energy detector 4

Figure 4 — Calibration of entrance pulse energy