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Comparative study of “fatigue” LID on laser optics

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GSI HELMOLTZZENTUM FÜR SCHWERIONENFORSCHUNG - GSI

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Deliverable Nature	
R = Report, P = Prototype, D = Demonstrator, O = Other	R
Dissemination Level	
PU = Public PP = Restricted to other programme participants (incl. the Commission Services) RE = Restricted to a group specified by the consortium (incl. the Commission Services) CO = Confidential, only for members of the consortium (incl. the Commission Services)	PP

A. Abstract / Executive Summary

High reflectivity (HR) mirror coatings were investigated by the means of optical fatigue effect. So called "S on 1" laser induced damage threshold (LIDT) measurements were carried out at VULRC. Two types of deposition technologies were investigated. Ambient air, vacuum and cryogenic cooling conditions were used to test TiO_2-SiO_2 mirror samples with infrared femtosecond pulses. Furthermore, a set of ultraviolet (UV) mirrors was also investigated with nanosecond pulses. In this case HfO_2-SiO_2 HR mirrors were tested under flow of different external gases. Effect of annealing was also investigated. For fs pulses it was found out that the damage threshold behavior differs between deposition technologies; however, no apparent changes were found for investigated environmental conditions. The ns results reveal no apparent changes with respect to different gas flow; however effect of annealing was significant. The nature of damage morphology indicates presence of the coating defects originating form deposition process.

B. Deliverable Report

1 Introduction

Lifetime of laser optics can be evaluated in terms of LIDT parameter as a function of repetitive laser pulses. Decay type of curves indicates so called fatigue effect thus meaning a significant risk of failure for high power laser systems. Modern laser concepts include many optical elements operating under low temperature and vacuum conditions that may suffer from it. Some bibliographic inputs indicate effects of vacuum that lead to an organic contamination of the surface irradiated. This phenomenon reduces the lifetime of optical component. Experimental conditions such as laser characteristics, environment composition and structure of the coating strongly influence the fatigue effects and laser induced contamination mechanisms. To get a deeper insight into mechanisms of laser damage under such environment, comparative studies of different environmental conditions are required on laser mirror coatings in order to provide a reliable database for laser designers and help to develop long lasting components. This report presents the observations made and discusses the possible physical mechanisms involved.

2 Objectives

This study aims at describing of the laser-induced fatigue effect in E-Beam evaporated and Ion Beam Sputtered mirror coatings both with femtosecond and nanosecond pulses. Various ambient conditions (gas, air pressure and temperature) as well as irradiation conditions (distinct pulse duration and wavelengths) have been have been used for testing. Observed trends in fatigue effect of highly reflecting optical elements should be useful for upcoming installations (ELI, HiPER and others) utilizing short pulse laser amplifiers and OPA's with and without cryogenic cooling.

3 Work performed / results / description

3.1 Study of femtosecond laser-induced damage test measurements on high reflectivity mirrors at distinct environmental and temperature conditions

Sample preparation

For temperature dependence LIDT testing in vacuum as well as for vacuum-air comparison tests we choose TiO_2-SiO_2 high reflectivity multilayer mirror coatings (99.8%) designed for 720-880 nm bandwidth and deposited on fused silica substrates. Before the deposition substrates were polished by using conventional loose abrasive polishing method. Cleaning has been carried out in an ultrasonic bath using specialized chemicals, water, then deionized water, and finally the samples were dried in an infrared oven. Substrates were deposited at the State Scientific Research Institute of Natural Sciences and Technology Center at the Institute of Physics by using two distinct deposition methods and namely ion beam sputtering (IBS) and electron beam evaporation (E-Beam). Silicon dioxide (SiO_2, n = ~1.44) was used as a low refractive index material while titanium oxide (TiO_2, n = ~2.40) was used for high refractive index layers. Electron beam evaporation (Fig. 1, left) was covered with 22 alternating layers. Each layer was of the same optical thickness and namely quarter-wave of optical thickness – QWOT at 800 nm wavelength. Physical thickness of TiO_2 sublayer was 82.11 nm and 136.8 nm for SiO_2 sublayers respectively. The final top layer is a double QWOT layer of SiO_2 overcoat. Another substrate (Fig. 1, right) was covered with 30 layers by using IBS sputtering. Physical thicknesses of TiO_2 and SiO_2 coatings were 85.211 nm and 134.608 nm respectively. All samples were additionally cleaned with acetone before measurements.

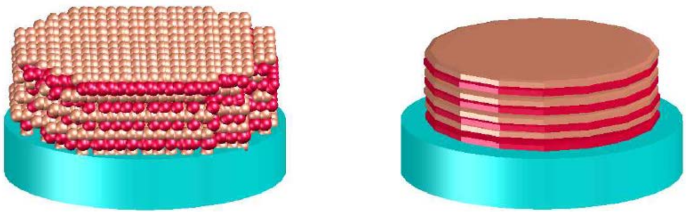

Fig. 1. Illustration of porous E-Beam (left) and dense IBS mirror coatings (right).

The damage test procedure

The test bench developed at Laser Research Center of Vilnius University has been used for femtosecond laser damage experiments to explore vacuum and temperature effects. The laser system consists of Ti:Sapphire amplifier with integrated oscillator and pump laser delivering linearly polarized pulses of ~100 fs duration (FWHM) with beam diameter in target plane (1/e): 140 ± 1.1 µm (average from 64 pulses, F = 50 cm lens) at 0 deg the angle of incidence and 100 Hz rep. rate. The fluence of fundamental harmonic (800 nm) was adjusted by using motorized attenuator consisting of half-wave plate and polarizer. A mechanical shutter is employed in order to pick up separate shots from a pulse train of 50 Hz repetition frequency. The online damage detection system was used for tracking of changes in optical scattering upon the damage event. The off-line inspection of irradiated sites was performed by Nomarski microscopy after irradiation exposure. For criterion of damage we consider catastrophic damage. Also we register any visible modifications that can be seen by Nomarski microscope to estimate the threshold of vacuum induced contamination. The sample was placed into custom designed holder (Fig. 2) that was cooled via cooper braids by closed cycle “Cryodyne Model 350” refrigerator in combination “Cryo-con Model C22“ temperature controller. PFEIFFER (TC 400) vacuum pump was used for regulation of pressure inside the vacuum chamber. Both samples were tested at 7 distinct ambient conditions as summarized in Fig. 3.

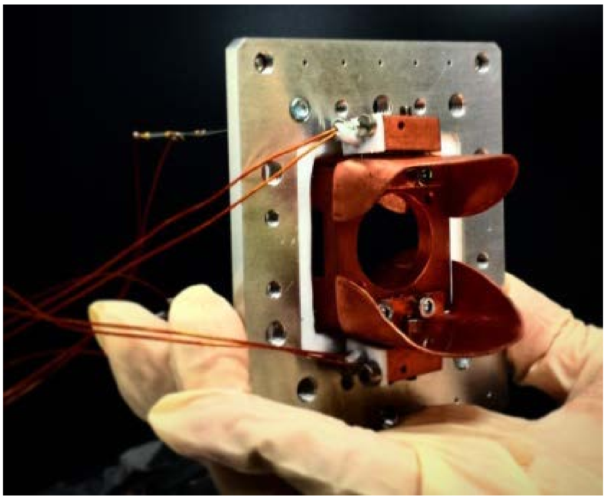

Fig. 2. Customized sample holder for cryo-cooling of one inch optics.

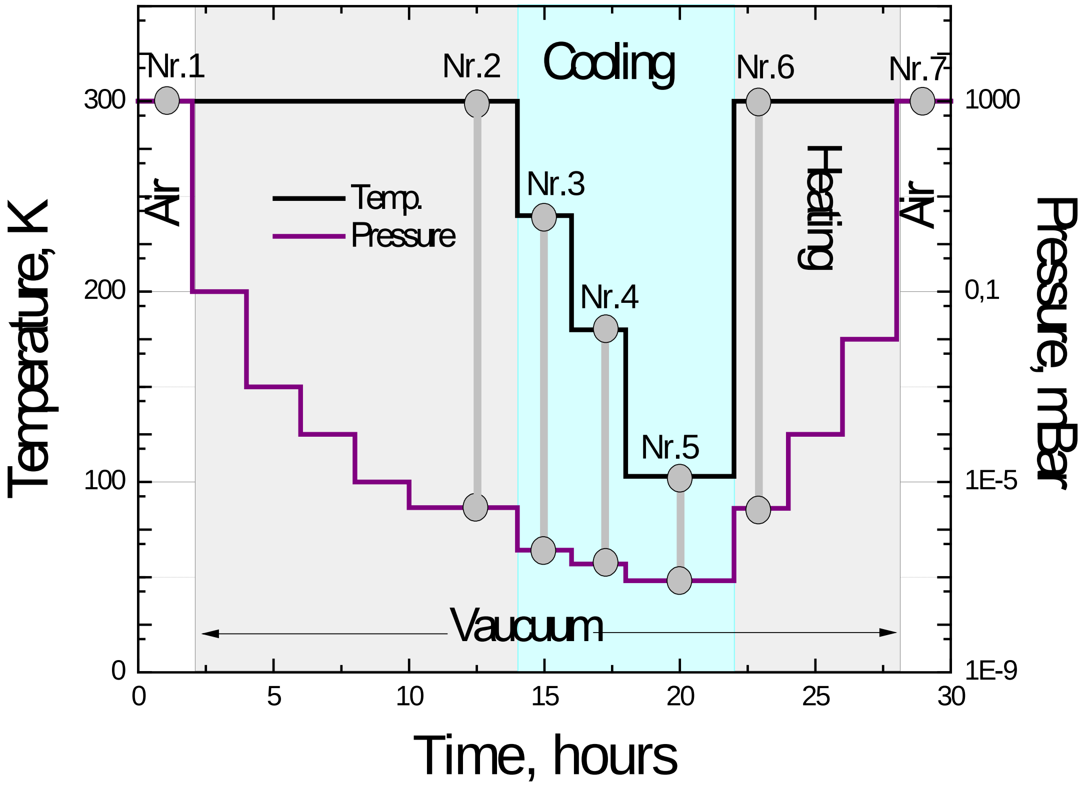

Fig. 3. Summary of 7 distinct conditions that were used for LIDT testing.

Results

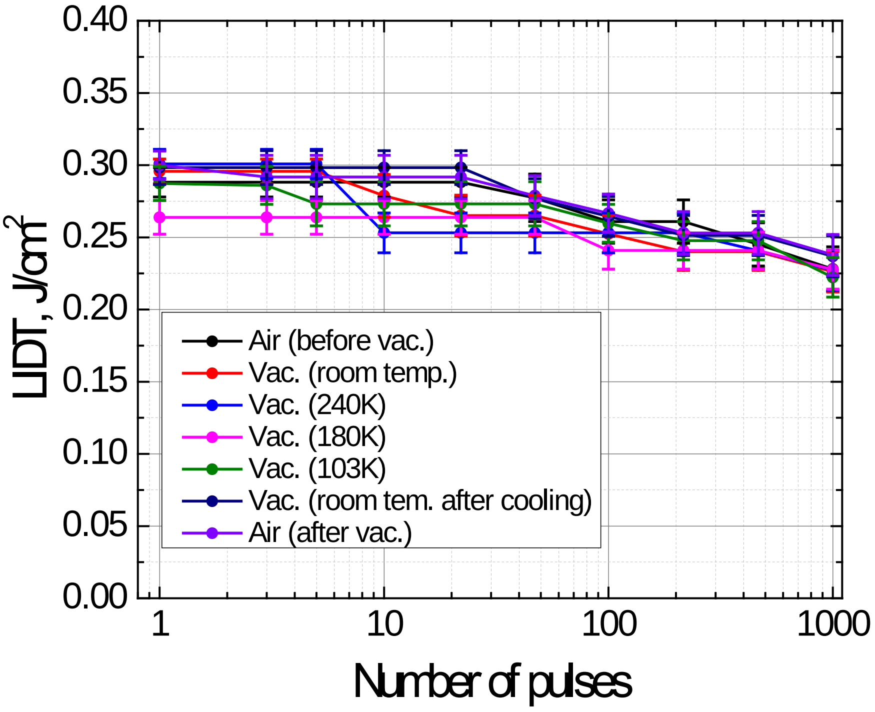

Fig. 4. LIDT test results of E-Beam coatings
(800 nm wavelength, 100 fs pulse duration, 100 Hz rep rate).

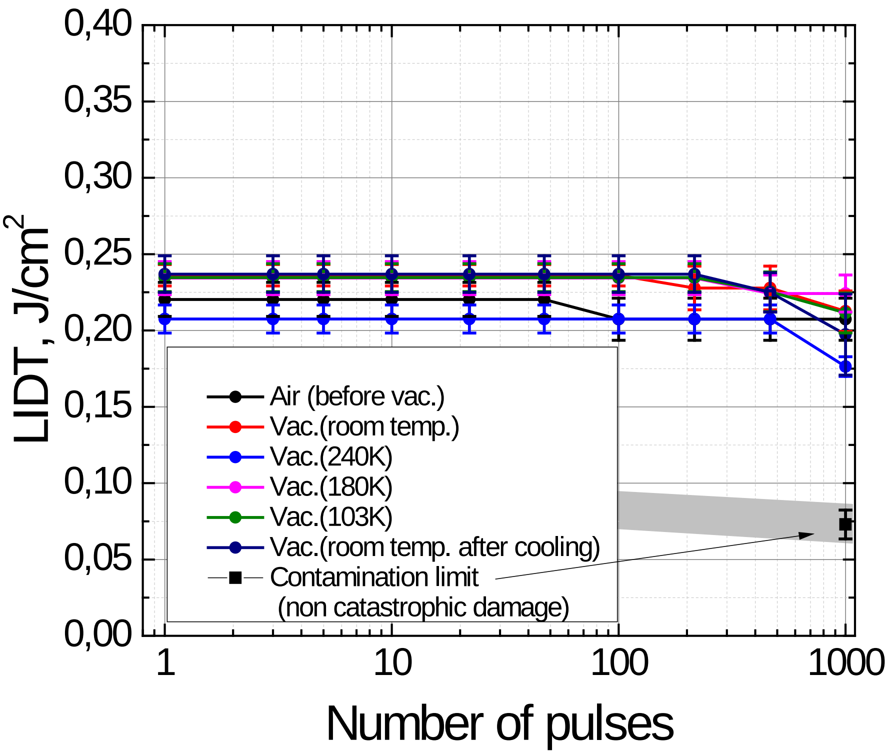

Fig. 5. LIDT results of IBS coatings
(800 nm wavelength, 100 fs pulse duration, 100 Hz rep rate).

Table 2. Laser induced damage in E-Beam mirror coatings.

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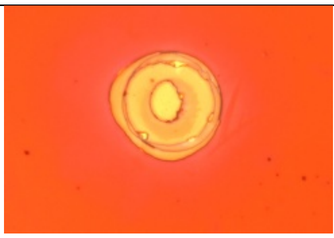
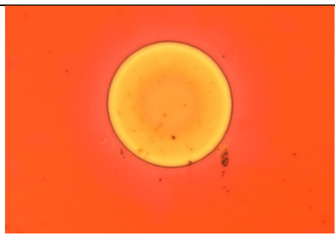
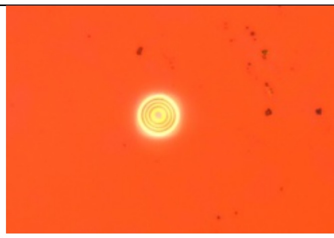
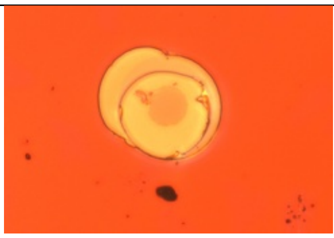
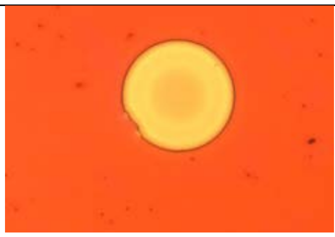
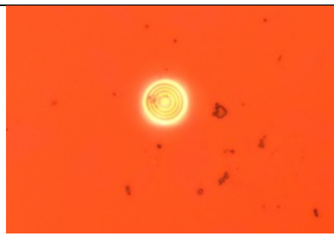
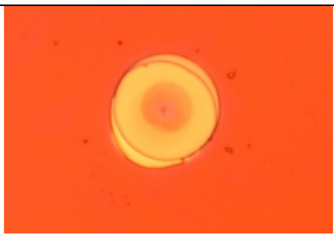
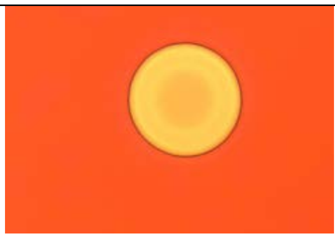
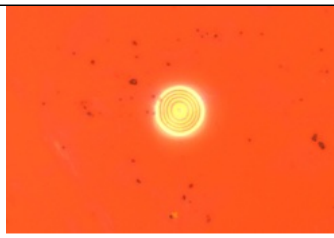
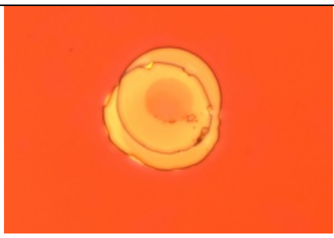
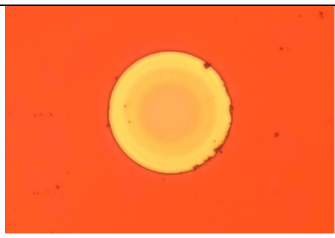
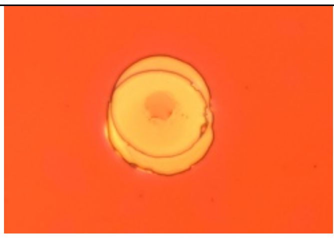
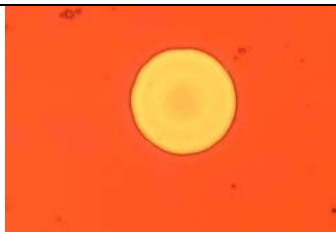
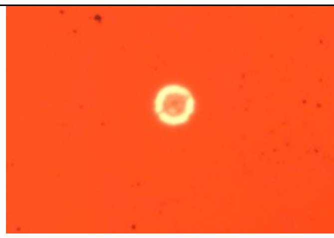
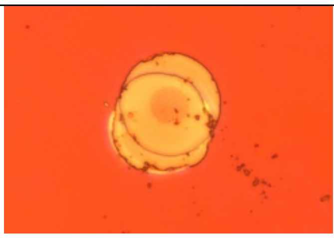
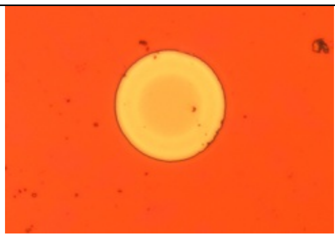
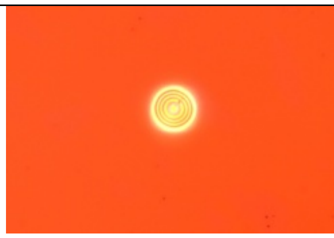
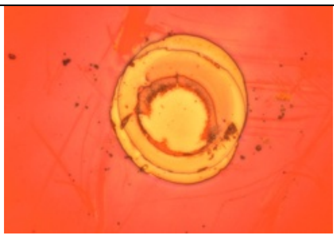
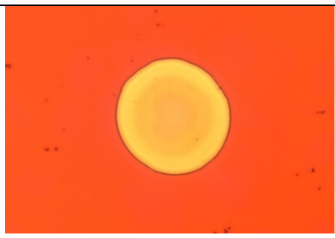
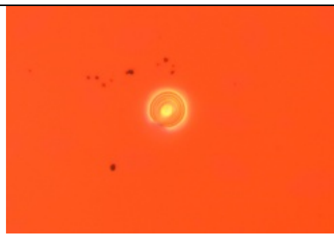
Oras				
	300			
	240			
Vacuum	180			
	103			
				
Air	300			

Table 2. Typical laser induced damage in IBS mirror coatings.

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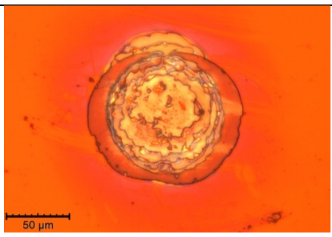
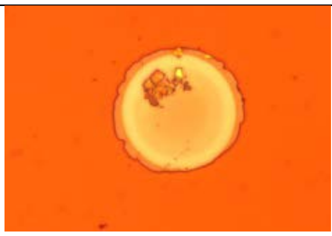
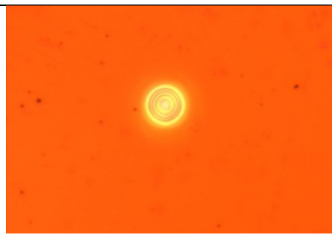
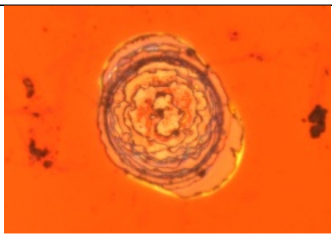
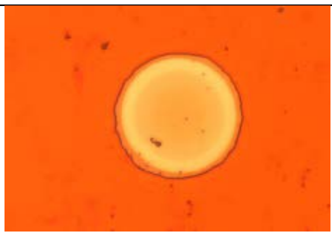
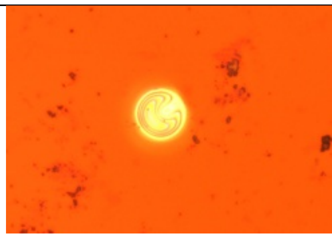
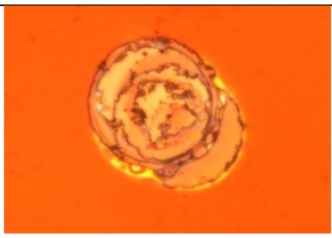
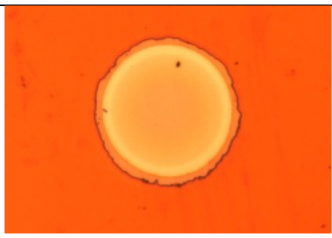
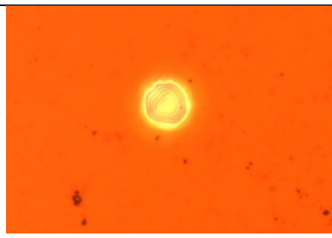
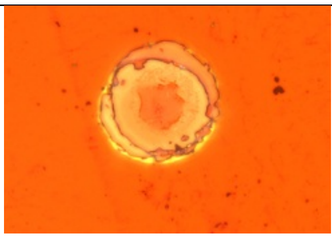
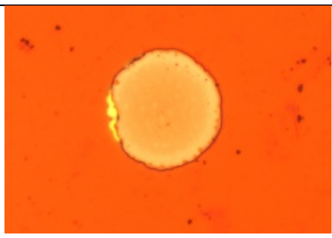
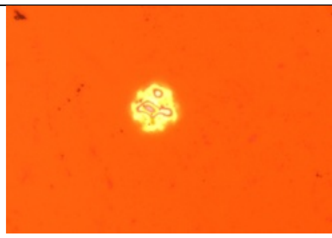
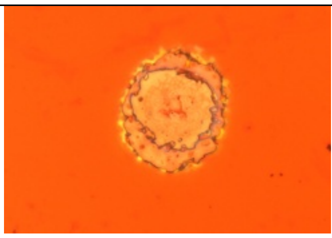
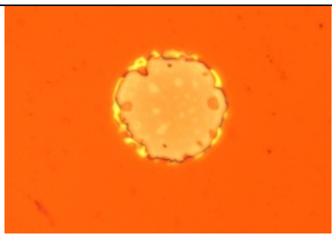
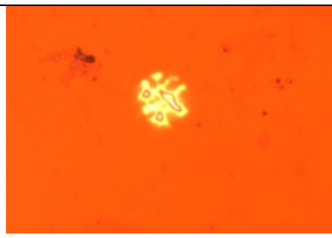
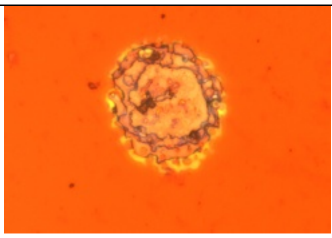
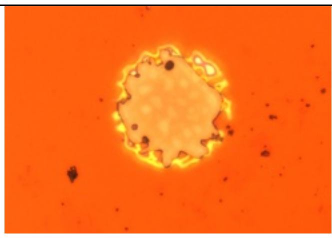
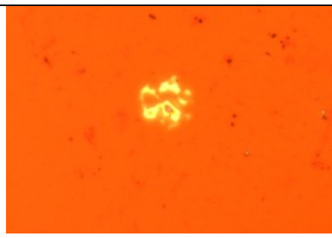
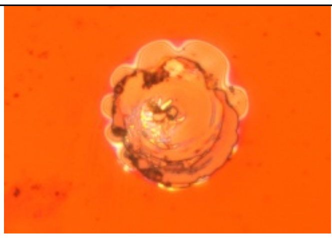
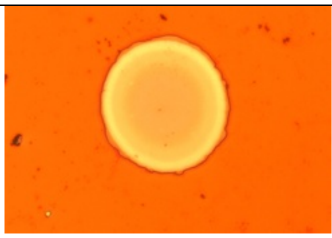
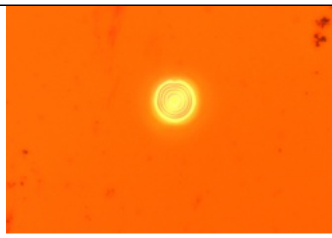
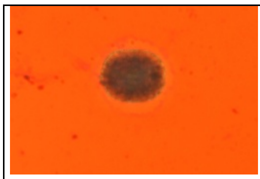
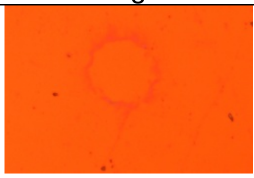
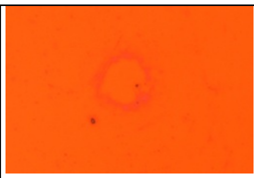
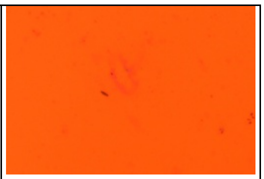
Air	300			
Vacuum				
	240			
	180			
	103			
	300			
Air				

Table 3. Typical morphology observed for IBS coatings bellow catastrophic laser damage threshold. Ring pattern (changes in color with Nomarski microscopy) was visible in all cases after vacuum treatment. Below the case of 180K cooling in vacuum is illustrated.

				
0,257 J/cm^2	0,242 J/cm^2	0,194 J/cm^2	0,164 J/cm^2	0,135 J/cm^2

Summarized LIDT test results and damage morphologies are provided in Figs 4 and 5. and Tables 1 – 3 respectively. At the first glance to the data it is obvious that E-beam coatings indicated slightly higher LIDT values if compared to IBS. However, E-beam mirror curves are not saturated after 1000 shots and show further trend of fatigue for larger amount of laser pulses. IBS coatings show somewhat lower but comparable values. Furthermore IBS coating indicate some changes in morphology at low temperature and shortly after that. The damage crater changes from nicely round shape to fragmented isle like morphology (Table 2, 103 K) thus indicating some changes in damage mechanism, however further studies are needed to explain such behavior. What is more: some signs of contamination are visible below the threshold of catastrophic damage in case of IBS coatings (Table 3). Such contamination appears in the form of colored rings and was not observed for E-beam coatings.

3.2 Study of nanosecond laser-induced damage threshold behavior by using distinct ambient gas at room temperature

Sample preparation and annealing

The coating consisting of 30 alternating HfO_2-SiO_2 layers was designed to meet HR@355nm, AOI = 0 deg specification. It was deposited by the same IBS coating chamber as described in previous chapter. 5 samples were deposited in total. Four samples were treated by e*x-situ* annealing for 12 hours at 300 C temperatures in order to reduce stress as well as absorption losses. Absorption in HfO_2-SiO_2 films is mostly related to incomplete oxidation of Hf particles and decreases with increasing temperature. One sample was left „As deposited“ in order to estimate the effect of annealing.

The damage test procedure

Another test bench developed at Laser Research Center of Vilnius University has been used for experiments with nanosecond pulses in order to explore effect of ambient gas. The system is based on a single longitudinal mode injection seeded Nd:YAG laser (Innolas, SpitLight Hybrid) delivering linearly polarized pulses of 5.7 ns duration (FWHM) at frequency tripled wavelength 355 nm. The fluence of pulses is adjusted with a motorized attenuator consisting of half-wave plate and polarizer. A mechanical shutter is employed to pick up separate shots from a pulse train of 50 Hz repetition frequency. The online damage detection system was used to track changes of light scattering due to laser-induced surface changes. The off-line inspection of irradiated sites was performed by

Nomarski microscopy after irradiation exposure. For criterion of damage we consider any visible modifications that can be seen by Nomarski microscope. LIDTs are evaluated by applying advanced fitting procedure described in reference [1]. It takes into account fluctuations of laser fluence as a fit parameter. Conventionally polished fused silica samples were chosen for deposition of mirror coatings. The beam size was focused down to 117.2 ± 5.6 μm ($1/e^2$) by using plano-convex lens. Statistical measurements were carried out on different samples by using standard S-on-1 ISO 21254-2 test procedure. The error bars for the probability measurements are calculated by using the procedures described previously. They correspond to a confidence level of 95%.

To figure out the effect of environmental conditions and annealing optical coatings were LIDT tested by using external gas flow on every irradiated surface site. Flow of different gases and namely nitrogen, oxygen, helium was compared. For the sake of comparison one two samples were measured by using normal atmospheric conditions - the air without flow. For the ambient experiments we used clean compressed gas with a steady flow reducer with resulting in 2 L/min flow from the tube located by ~5 mm distance to the targeted area that was exposed by a laser. The LIDT measurement methodology has been kept the same as described by ISO standard. Experiments were carried out on 5 samples.

Results

The effect of annealing

First of all effect of annealing was investigated. Spectra of coating samples were registered before and after annealing process (Fig. 6.). The increased transmittance after annealing indicates reduced absorption and thus more complete oxidation of coatings.

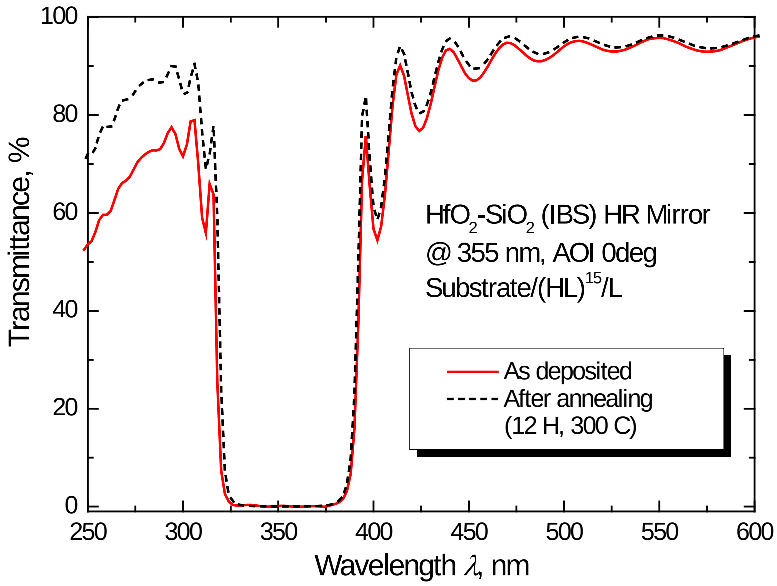


Fig. 6. Transmission spectra of „as deposited“ and annealed samples

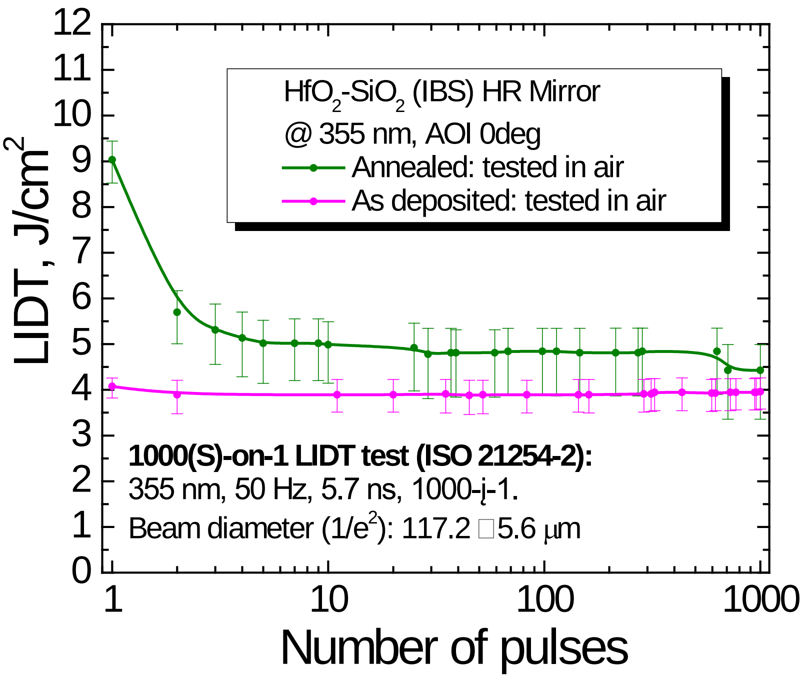


Fig. 7. Direct comparison of LIDT for annealed and as deposited samples.

From the results above we can draw a simple conclusion: annealing helps. First of all, “as deposited” sample has lower transmittance indicating higher absorption if compared to annealed sample and thus not surprisingly, annealed samples indicated > 2x higher 1-on-1 damage threshold. In case of 1000-on-1 test, this increase was only 5-15%..

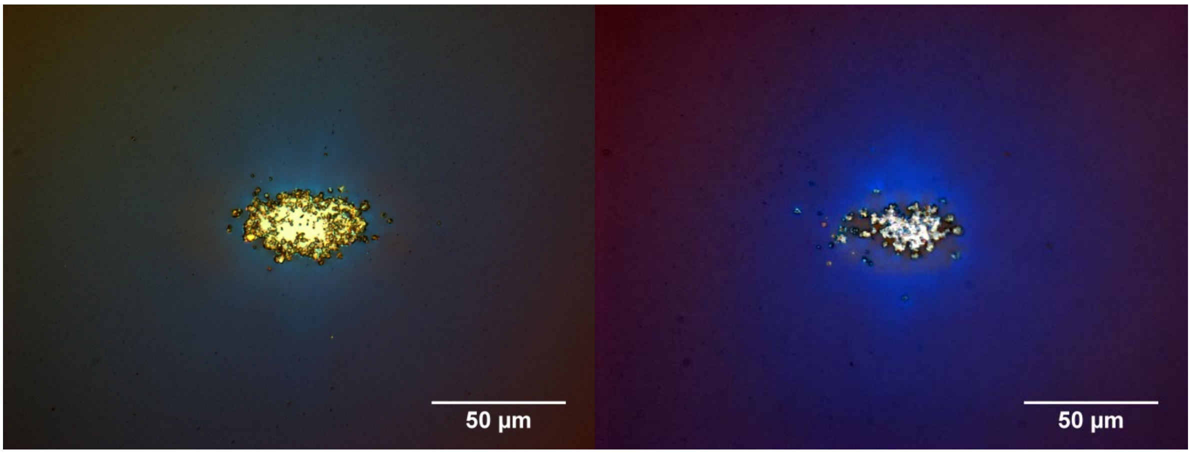


Fig. 8. Comparission of 1-on-1 damage morphologies for As deposited (left) and Annealed (rigt) samples slightly above LIDT.

By comparing the damage morphologies one can conclude that no principal differences exist for 1-on-1 morphology obtained slightly above single shot LIDT. Signature of defect driven damage can be recognized in this case: most likely coming from nodular defects created within deposition process.

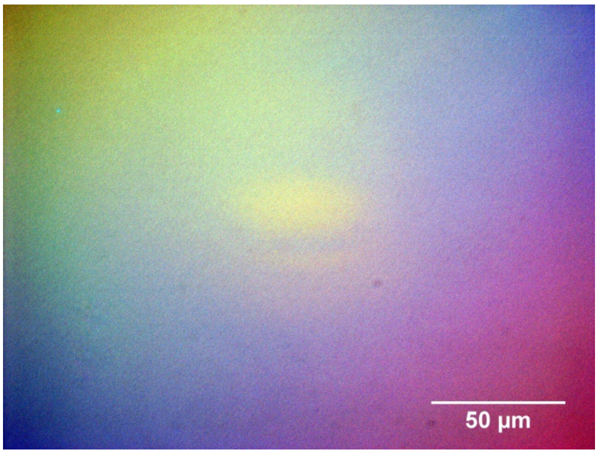
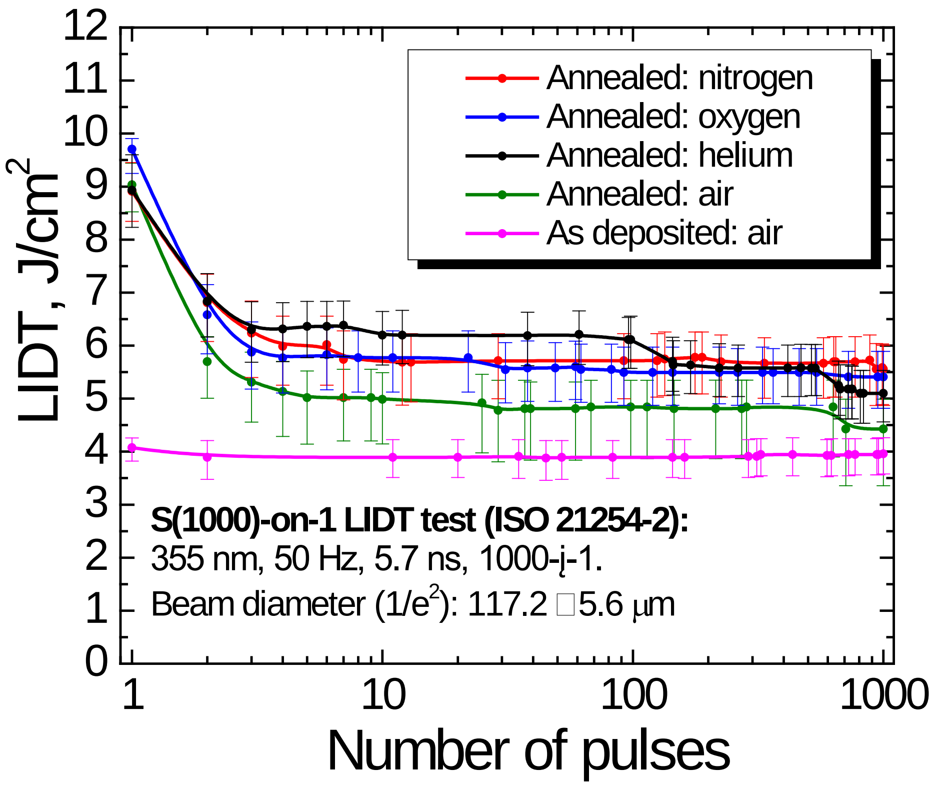


Fig. 8. Video-contrast enhanced
„As deosited“ site, after 1000 pulses (4,84 J/cm^2)

In case of S-on-1 morphology non catastrophic changes in color are visible thus indicating either laser induced contamination or annealing caused by light absorption.

Effect of ambient gas on annealed samples



The results of gas experiments are summarized in Fig. 9. The curves obtained at normal room air conditions indicate slightly lower LIDTs than those exhibited under gas flow. Nevertheless, the differences are rather minimal and non-exceeding the range of the measurement errors. No principal differences were observed for investigated gases tus meaning that damage mechanisms are not directly related with environment but rather to internal reasons such as burred defects that could recognized in Fig 8.

4 Conclusions

- All investigated mirror coatings (TiO_2-SiO_2 and HfO_2-SiO_2) experiences fatigue effect bot for ns (355 nm) and fs (800 nm) repetitive laser pulses.
- No apparent changes in multi-shot fs LIDT behavior were found among E-beam TiO_2-SiO_2 mirror coatings when changing ambient conditions (Air, vacuum, low temperature).
- Contamination effects as well as changes in damage morphology were observed for IBS TiO_2-SiO_2 high (@ 800 nm, 100 fs) when changing ambient conditions (Air, vacuum, low temperature). However observed morphology changes do not indicate any changes in measured catastrophic damage threshold.
- Differences in LIDT behavior between IBS vs E-beam coatings are more significant than ambient factor thus indicating, that damage is caused mainly by material intrinsic properties rather than ambient condition at least for the first 1000 laser shots.
- Experimentally found that the electron beam evaporation coated with TiO_2-SiO_2 mirror is resistant to vacuum and cryogenic temperature effects, as laser damage threshold varies within the range of error, and substantial damage morphology differences were observed.
- Annealing has strong positive effect on IBS mirror coatings and resulted in increased LIDT values and reduced absorption.

5 References/Publications

[1] G. Bataviciute, P. Grigas, L. Smalakys, and A. Melninkaitis, "Revision of laser-induced damage threshold evaluation from damage probability data.," *Rev. Sci. Instrum.*, vol. 84, no. 4, p. 045108, 2013.