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Comparative study of Laser Induced Damage (LID) of laser materials

Lead Beneficiary:

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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

Both coated and uncoated optical samples were investigated by high fluence laser irradiation, specifically “1 on 1” and “S on 1” laser induced damage threshold (LIDT) measurements were carried out. Within these measurements online and offline diagnostics were applied for damage arbitration. As diode pumped laser relevant materials doped and undoped $Ca{:}F_2$ as well as fused silica and Nd:YAG substrates were chosen so far. The participants VULRC and FSU-IOQ deployed their advanced test benches for measurements at vacuum and cryogenic temperature conditions. These conditions are significant for modern cryogenic cooled diode pumped lasers. Many LIDT damage tests are furthermore required for benchmarking laser components and to develop reliable laser systems.

B. Deliverable Report

1 Introduction

Laser Induced Damage (LID) thresholds are critical parameters for the design of future high peak-power diode-pumped laser sources because the highest acceptable fluence at the laser medium and passive optics determines the overall efficiency of such a laser. At the same time this parameter bears the highest source for risk of failure. Modern laser concepts for high energy and short pulse operation based on low temperatures of optics that stay under vacuum conditions may suffer from it. At those extremes conditions, LID differ from what could be observed in air. To get a deeper insight into mechanisms of laser damage under such environment, comparative studies of different laser materials and surface treatments as well as coatings are required in order to provide a reliable database for laser designers and help to develop long lasting components.

2 Objectives

Objective of this task is to serve as legwork for high power laser development by investigation of LID on laser materials from partner institutions such as (un)coated Yb:YAG, $Yb{:}CaF_2$, Ti:sapphire and passive optical elements (mirrors and anti-reflective coated lenses) at various ambient conditions (gas pressure and temperature). According to gas and vacuum purity as well as surface treatment and coating preparation, limitations and constraints to the laser engineering will be investigated with strong impact on all large scale existing and upcoming installations (ELI, HiPER and others) that utilizes short pulse laser amplifiers and OPA's with and without cryogenic cooling.

3 Work performed / results / description

3.1 Laser induced damage test measurements at damage test facility of the IOQ Jena

<u>The damage test procedure</u>

All damage tests at the IOQ are based on ISO 112541-1 as 1-on-1 measurements. The test facility is described in detail in the report Laserlab EuroLite WP33 Deliverable 17. A specialty of this facility is the possibility to apply a pulse structure by overlapping two beams. The amplitudes of the two consecutive pulses with a delay of some nanoseconds can be freely adjusted. Because they share the same amplifier in collinear propagation both beam profiles are exactly the same. This allows to simulate stress to a laser medium equivalent to a propagation in multi-pass amplifiers.

In a first step samples were irradiated with the laser pulses with one shot per position on the sample by stepwise increasing pulse energy. Per energy level ten shots were applied. The second step is inspection of the test sites. From the number of damaged sites versus the applied pulse energy a damage probability versus fluence is estimated and a linear approximation towards zero valued probability will result in the laser induced damage threshold for the inspected sample. Damaged sites are all that show any permanent visible degradation. In case of surfaces with coatings these could be of several kinds as shown below. For all measurements described here the pulse width was fixed to 3 ns at a wavelength of 1030nm, i.e. the lasing wavelength of the investigated Yb-doped materials. The laser spot has a Gaussian shape shown in figure 1 with a diameter of roughly 200 micron. The laser spot is measured online for every pulse and together with the pulse energy measurement the applied energy density can be evaluated exactly and independently from any variations of the laser.

Samples were always placed in vacuum, if measured at room-temperature or cooled down to 130 K.

<u>Sample preparation</u>

In this project period the focus of investigation was on Yb-doped calcium fluoride, as a laser material which provides a very broad band emission, supporting amplification of pulses well below 200 fs, exhibiting a long fluorescence life time, and showing a high absorption cross section at 980 nm where high power laser diodes are available. Since in laser applications the coating is mostly limiting the maximum allowed fluence in this first campaign undoped calcium fluoride was used as a model substrate.

In order to be able to distinguish between differences of the influence to the damage threshold by surface preparation, bulk material or coating techniques a matrix of combinations of different preparation techniques were studied. For the surface polishing three different qualities were available an inspection polishing (IP) a laser grade polishing (LP) and a UV grade polishing as the highest standard for applications in lithography (UP). Materials from three different vendors were used. The anti-reflection coatings were prepared by another three vendors with ion beam sputtering technique (IBS) and ion assisted evaporation deposition technique IAE.

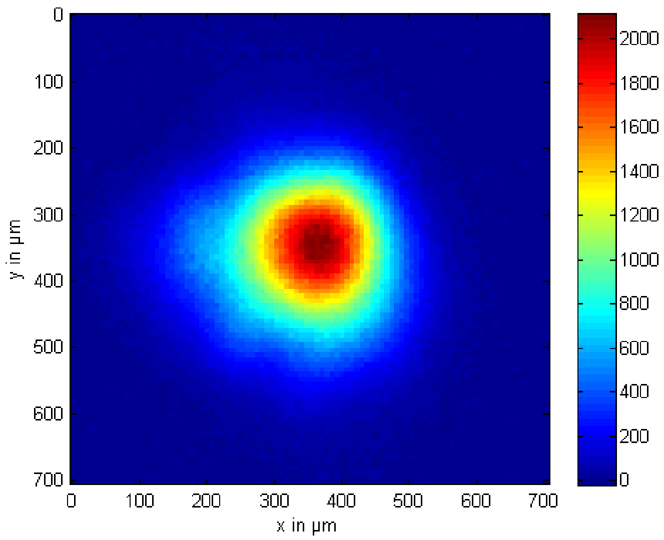


Figure 1: Beam Profile of the laser in the focal plane.

The vendors of the material, the polishing and the coatings were Hellma Materials GmbH (Germany), Layertec GmbH (Germany), Carl Zeiss AG (Germany), CVI-Melles Griot (UK), Neneo-Precisition-IBS-Coatings GmbH (Germany), and Laseroptik Garbsen GmbH (Germany). It is intended to not reveal the assignment of the different vendors to the indentation in the following since we have no permission from the companies to publish the results in direct comparison. Therefore vendors are numbered A to E but not in the order as listed above. From vendor E a coating sample on fused silica substrate was also prepared for comparison. And a measurement of uncoated Yb-doped YAG was added as a reference for the measurement under same conditions.

Results

The measurement results are listed in Table 1. In the last two columns the laser induced damage thresholds are given in J/cm^2 at a temperature of 300K and a temperature of 130K. Most of the measurements were done twice in order to average and to be able to estimate an error value. In those cases where no second sample or not enough surface area for all measurements was available the error was estimated from the probability curve. In the case of material from vendor B, UP polishing and without coating we have not seen any surface damage but at the given energy densities bulk damage occurred. This indicate that for the given pulse properties about 60 J/cm^2 seem to be the ultimate upper limit since it is known that doping will decrease the fracture toughness of the material, i.e. it will finally cause a lower damage threshold.

Table *1: 1-on-1 laser-induced damage thresholds in J/cm², vendors A to E in parenthesis.*

Material	Polishing	Coating	at 300K	at 130K
CaF_2 (B)	UP (C)	IBS (A)	7 ± 4	7 ± 3
CaF_2 (B)	IP (C)	IBS (A)	6.5 ± 2.5	10.5 ± 2.5
CaF_2 (B)	LP (A)	IBS (A)	15 ± 6	15 ± 6
CaF_2 (B)	UP (C)	IAE (C)	11.5 ± 2.5	11 ± 3
CaF_2 (E)	LP (E)	IAE (E)	11.5 ± 1	11.5 ± 1
CaF_2 (D)	LP (D)	IAE (E)	7 ± 1	7.5 ± 1
CaF_2 (D)	LP (D)	IAE (D)	2.8 ± 0.6	4 ± 0.7
CaF_2 (D)	LP (D)	No coating	42 ± 5	45 ± 6
CaF_2 (B)	UP (C)	No coating	> 52 *	> 58 *
Yb:YAG	standard	No coating	23 ± 4	29 ± 6
Fused Silica (E)	laser grade (E)	IAE (E)	35.5 ± 3	39 ± 3

** onset of volume damage, no surface damage detectable*

Comparing the results one can conclude that except for vendor D, where the coating process seem to be a limiting factor in the whole sample preparation, damage thresholds of coated samples are in the range of 6 to 12 J/cm^2,whereas for the uncoated samples a five times higher damage threshold was observed. Comparing the values with the reference of anti-reflection coated fused silica, room for improvement by better adaption of the coating to the substrate could be argued.

By simply inspecting the damage threshold values, there is a small indication for an advantage of ion beam sputtering as the coating technique. But this is within the error limits of the measurement. But looking furthermore to the damage morphology as exemplarily shown in figure 2 to 5, it could be concluded that damages of the harder, more dense IBS coatings (fig. 2 and 3) result in damage sites that can extend the irradiated area since fissures with large extensions may occur. This behavior is not represented by the damage threshold values. Nevertheless, it can have an impact in laser applications with large beams, where some damages are acceptable by blocking parts of the laser beam in this area. In these cases care must be taken in damage measurements to put the damage test sites far enough away from each other. In our case the spots were separated by about 1.5 millimeter. Such fissions are not observable for evaporated films (fig. 4 and 5). Nevertheless even there coatings with lower thresholds tend to be peel off (fig. 5).

Previous experience for uncoated samples of calcium fluoride was that UV grade polishing is able to improve the surface damage threshold a lot. While the two measurements on uncoated samples listed here also follow this assumption the coatings from vendor A show, that this is of less importance when the coating sets the limit in damage threshold. Similarly to the comparison of different coating techniques a higher quality polishing with smaller surface roughness always result in smaller damage sites (comp. fig. 2 and 3) even if the applied fluence was higher (comp. fig. 4 and 5). And again this is not represented by the threshold values.

It is clearly to be seen in figure 2 and 4 that for the UV grade polished and coated surface, where the damage sites are small enough that the primary damage sources are often aligned in straight lines. The reason for this behavior could not yet really be explained. Since the underlying substrate is a single crystal, one possibility for such structures are small angle grain boundaries. At these boundaries the chemical and optical properties can differ from the rest of the sample. But other reasons like small invisible scratches or subsurface defects could be looking similar as well as residual abrasives or cleaning agents. Since the latter are very unlikely crystal intrinsic defects are assumed to be the source. More measurements are needed to prove this assumption.

In all cases except for the vendor D coating a higher damage threshold is always achieved if the polishing and coating was made by the same vendor. This is a hint that surface contamination or surface degradation at storage time will be a source of limited damage thresholds. It is planned to investigate this possible effect further.

Finally we compare the results for different temperatures. In Table 1 the listed damage thresholds for room temperature and 130 K are very close to each other so that conclusion is allowed that temperature does not play any role here. This is again in contradiction with previous measurements on uncoated samples what showed slightly higher damage threshold in case of cold sample together with always smaller damage diameters.

Figure 2: Damage site with UP polishing (C) and IBS coating (A) (@ 67 J/cm^2 / 300K) magnification:5.

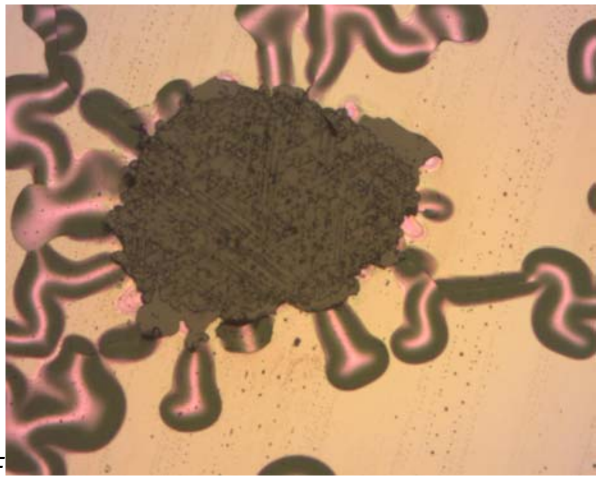


Figure 3: Damage site with IP polishing (C) and IBS coating (A) (@ 51 J/cm^2 / 300K) magnification:5.

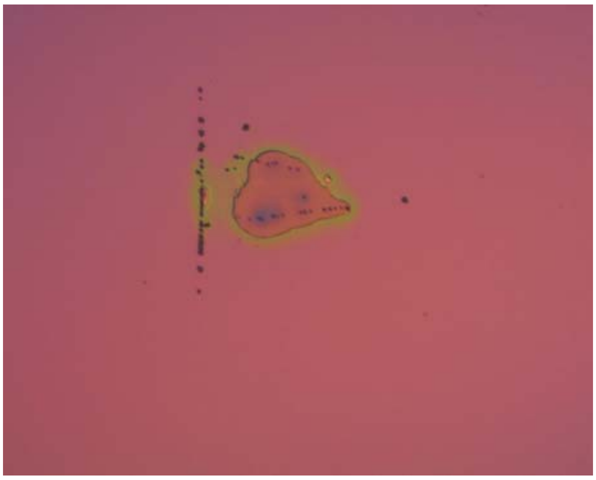


Figure 4: Damage site with UP polishing (C) and IAE coating (C) (@ 60 J/cm^2 / 300K) magnification:5.

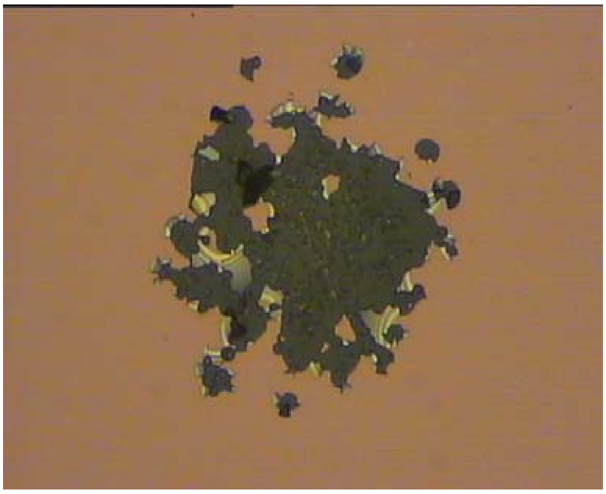


Figure 5: Damage site with LP polishing (D) and IAE coating (E) (@ 26 J/cm^2 / 300K) magnification:5.

Double pulse LIDT measurements

The double pulse option provided by the Yb:YAG laser system was used to model the pulse structure of a typical multipass amplifier. In a first attempt two passes were assumed and the energies of the two pulses were adjusted to be equal. A detailed description of the laser equipment that is required for generating this temporal structure is described in the report for deliverable 33.17. An example for such a measurement is given in figure 6. There the measurement with the double pulse is compared with a measurement using a single pulse what exhibits the same total pulse energy. In the double pulse measurement fluence correspond to the sum of both pulses.

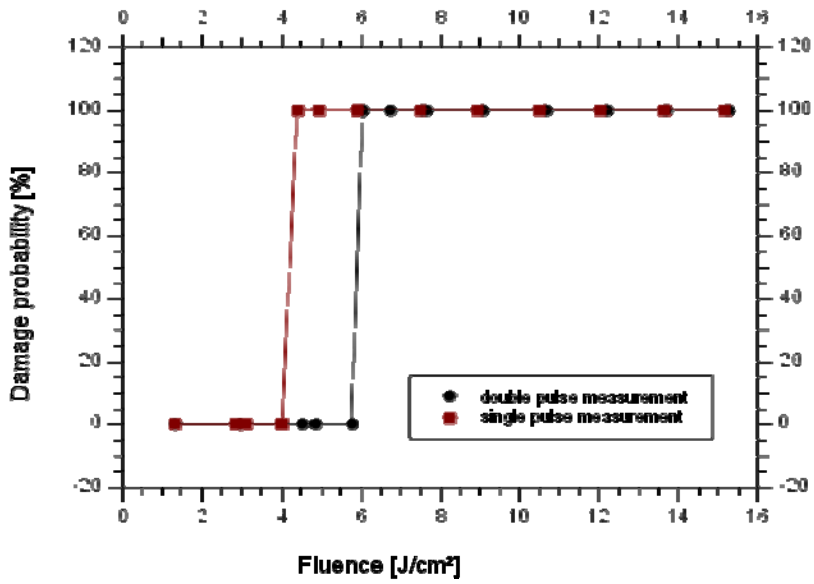


Figure 6: Comparison of measured damage probability for a single pulse and a double pulse. In the double pulse measurement the fluence correspond to the total fluence of both pulses. This measurement was carried out on an AR-coated CaF_2 sample.

One can conclude that the laser damage threshold reduces by the square root of two, what is exactly the same factor one would expect for a two times longer pulse. This result is in accordance with the assumption that the pulse delay does not allow the material to settle down after the first pulse impact. This has to be considered in any multipass and regenerative amplifiers. On the other hand it shows that with pulse trains high extraction fluences in laser amplifiers can be tolerated.

3.2 Laser induced damage test measurements at damage test facility of the VU LRC Vilnius

The damage test procedure

All damage tests that are performed at VU-LRC were performed according to ISO S-on-1 procedure which is a multi-shot technique that also involves 1-on-1 case described above. In this case every site on optic surface is irradiated by fixed (S) amount of repetitive pulses. In our case maximum 10,000 pulses were used per site. Similarly as a before energy was increased stepwise. Per energy level ten sites were irradiated. From the number of damaged sites versus the applied pulse energy a damage probability versus fluence is estimated in each class of laser shots. Damage threshold is then estimated by fitting damage probability statistics versus applied pulse energy relation by appropriate extrapolation model using maximum likelihood algorithm [1].

Damage threshold is reported as a function of incident laser pulses. This technique is well suited for estimation of ageing effects in laser optics. The same procedure can be applied at different pulse repetition rates and environmental conditions. As in previous case test facility is described in detail within the report Laserlab EuroLite WP33 Deliverable 17. Before its irradiation all optics had been cleaned using lens tissue and acetone. The dwell time in vacuum was set to at least 12h before starting the irradiation. The test laser beam shape was close to Gaussian and the beam diameter used to test the optics was approximately 451 µm diameter ($1/e^2$). Other parameters were - Laser pulse width (FWHM) - 11.1ns, Rep. Frequency 50 Hz, Injection seeded, Pressure ~ 1 x10^{-6}mbar, Pumping time - 17h.

Sample preparation

Two Anti-reflection coating (AR) and two High-reflecting mirror coating (HR) samples have been manufactured in the same two coating runs using a customized research deposition chamber. On high quality super-polished fused silica substrates with a surface quality of σRMS<0.1nm dielectric multilayer systems of alternating layers of SiO2 and Ta2O5 were deposited finished by a silica top layer. These materials are commonly used for the production of high power laser optics.

Ion beam sputtering deposition (IBS) has been selected as coating process for the manufacturing of the optics. This PVD process is well known for delivering dense coatings without wavelength shifts occurring in air-vacuum transitions, which makes IBS coatings ideally suited for low pressure applications like high-power optical components in harsh space environment.

The antireflective coatings were designed to have a remaining reflection of less than 0.1%. Only one side of each sample has been coated to avoid back side damages occurring during the LIDT-measurements. Characterization of the AR samples in calorimetric measurements revealed an absorption level of 7ppm at a test wavelength of 1064nm.

The mirrors had a reflection of at least >99.9% and showed total losses of approximately 650ppm measured with cavity ring down (CRD) technique at the wavelength 1064nm. The absorption of the HR samples was also 7ppm at the test wavelength.

Furthermore one uncoated Nd:YAG crystal was prepared as an example of active medium. In this case substrate was doped with Nd ions with concentration ranging between 0,8 % and 1 %. The sample was polished by loose abrasive polishing technique with diamond powder of different fractions.

Results

Herewith we report on measurements in vacuum environment of the laser-induced damage thresholds (LIDT) of fused silica coated with AR and HR and optimized for 1064 nm wavelength. All the samples were placed simultaneously in multi sample holder and tested in the same sequence without opening of the vacuum chamber. An overview of the results for the LIDT-measurements on both types of samples evaluated according to the ISO standard is depicted in Figure 7 and Table 2.

Table 2. LIDT results of AR and HR coatings.

Sample	1-on-1 LIDT, J/cm²	(S=10000)-on-1 LIDT, J/cm²
AR1	7.84 < 9.55 < 10.69	3.28 < 4.80 < 6.13
AR2	6.07 < 9.06 < 10.65	4.68 < 5.87 < 6.67
HR1	24.05 < 28.04 < 30.04	10.08 < 12.08 < 13.07
HR2	34.03 < 40.02 < 43.99	13.57 < 17.57 < 19.06

As can be seen from differences between 10000-on-1 and 1-on-1 LIDT results all samples exhibit an ageing behavior under repetitive irradiation, thus meaning that multi-shot LIDT is always lower than that of single shot. For a pulse class of 10.000 the difference between the lowest and the highest measured HR sample threshold is about ~5 J/cm². This signifies an discrepancy for LIDT-measurements on comparable optics from the same coating run. It has to be stated that the results are within the specified error budget, however 1-on-1 results exceeds these limits. This indicates the presence of vacuum effects that are probably changing in time. Difference between two HR measurements vacuum exposure is roughly 4 hours. AR coatings indicated almost identical results. The difference between AR and HR coatings probably could be explained by the fact that damage initiation most likely starts at the coating-substrate interface because of polishing defects. This is confirmed by differences in damage morphology: in the AR case some localized spots are visible. Accordingly, there is no big difference in LIDT, since the surface coating-vacuum is not limiting the LIDT. In contrary for HR coatings, the electric field does not penetrate till the substrate and the maximal intensity is located at vacuum-coating interface thus any changes in surface properties are more likely affecting the LIDT of mirrors. Additional investigations including post damage surface contamination analysis are necessary to explain these effects.

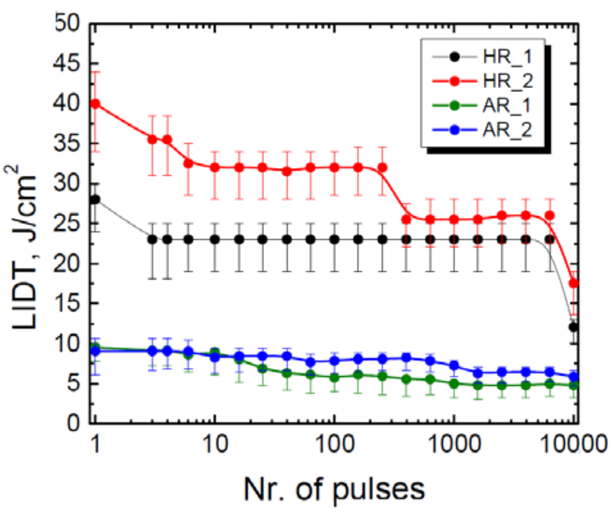


Figure 7: S-on-1 laser-induced damage thresholds of HR and AR coatings in J/cm² as a function of number of incident pulses.

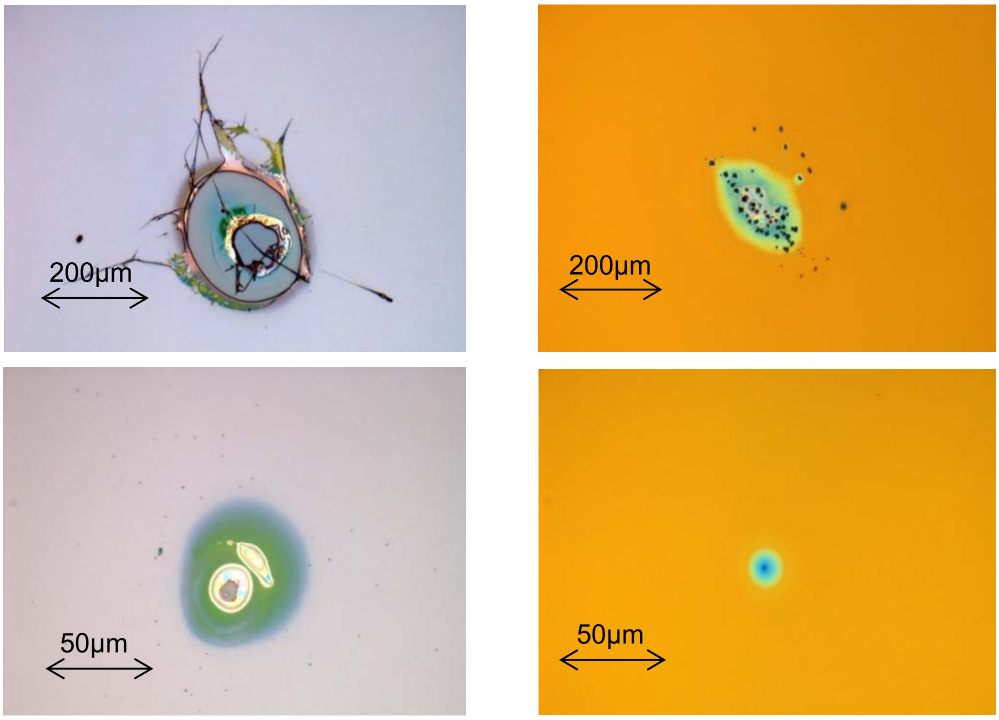


Fig. 7. Damage morphologies in vacuum observed for HR coatings (on the left) and AR coatings (on the right)

Further, uncoated doped Nd:YAG crystal has been investigated by the means of S-on-1 LIDT test under two distinct environmental conditions: air and high vacuum (10^{-6} mbar). As in previous case sample has been exposed with 1064 nm, 11 ns, 50 Hz laser irradiation featuring Gaussian shaped beam intensity profile with diameter of (98.0 ± 2.8 um) at $1/e^2$. *S*-on-1 LIDT measurement procedure has been performed according ISO 21254 standard (slight exception has been made regarding small beam diameter due to limitations in available testing surface). Each tested site was exposed with burst of maximum 1000 pulses unless it was recognized the case when damage apeared earlier. Absolute fluctuations of the laser peak fluence have been characterized to be ~8% (1 std.). Laser – Induced damage threshold is calculated by non-linear fitting based on model function and maximum-likelihood search algorithm for the best estimates of parameters [1]. Determined LIDT values as a function of number of incident laser pulses are presented in Fig. 8 and listed in Table 3. Typical damage morphologies are presented in Table 4.

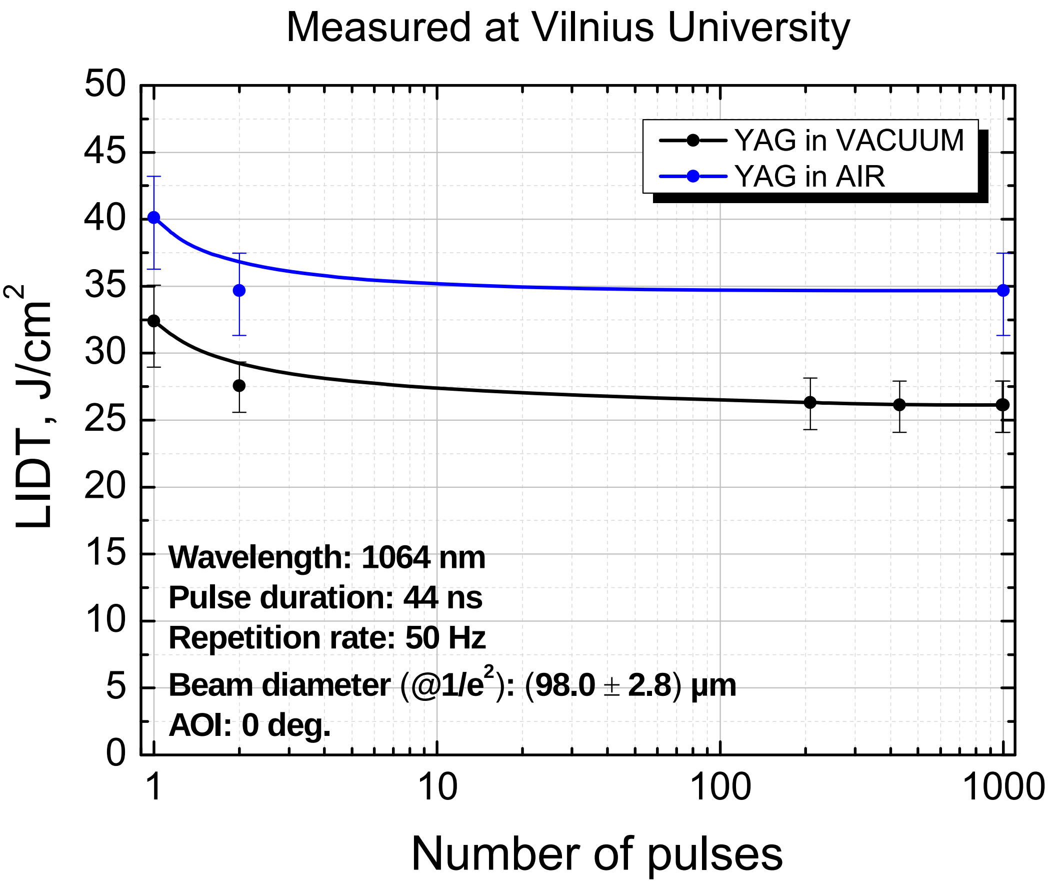
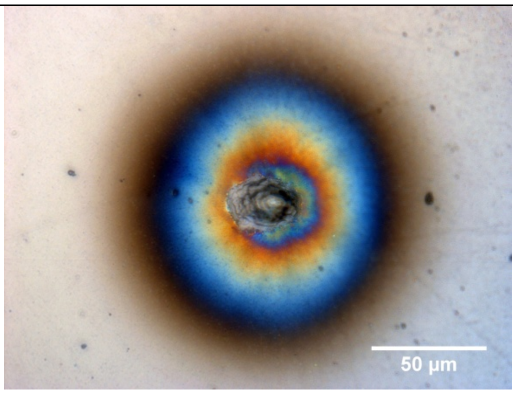
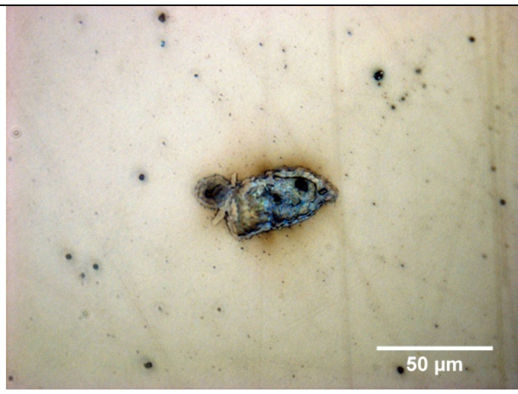
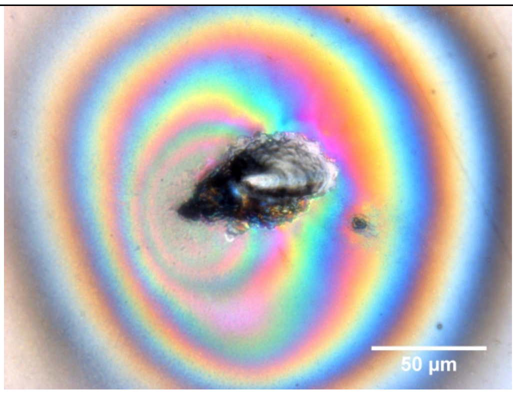
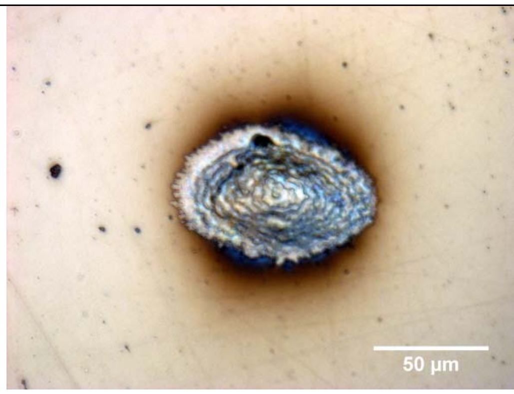
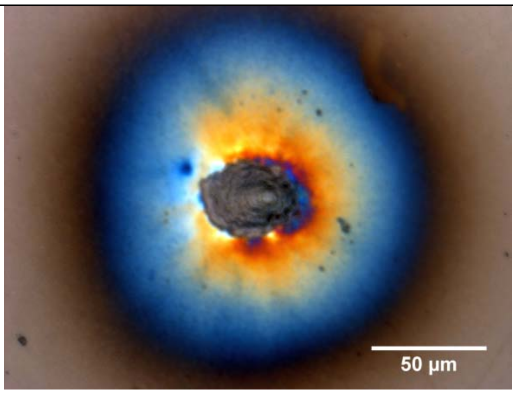
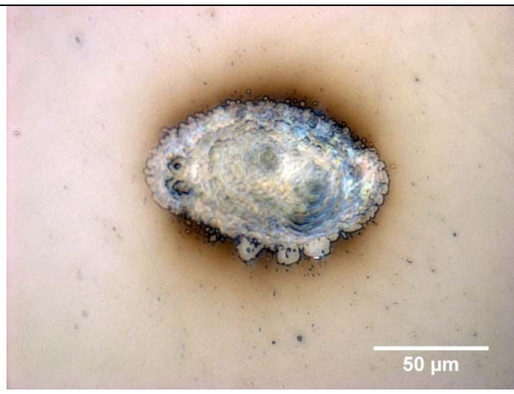


Fig. 8. Measured *S*-on-1 damage threshold as a function of pulse number under two environmental conditions: air and high vacuum (10^{-6} mbar).

Table 3. Calculated damage threshold values for uncoated Nd:YAG crystal under two environmental conditions: air and high vacuum (10-6 mbar)

Environment \ irradiation	1 - on - 1	1000 - on - 1
Air	36.28 ≤ 40.11 ≤ 43.20	31.32 ≤ 34.68 ≤ 37.47
Vacuum	28.97 ≤ 32.39 ≤ 35.07	24.09 ≤ 26.14 ≤ 27.91

Table 4. Typical damage morphologies in air (left) and vacuum (right).

Air:	Vacuum:
	
Site 198, pulses 2, fluence 40.68 J/cm^2	Site 333, pulses 2, fluence 30.30 J/cm^2
	
Site 200, pulses 2, fluence 40.86 J/cm^2	Site 411, pulses 2, fluence 35.15 J/cm^2
	
Site 495, pulses 1, fluence 52.71 J/cm^2	Site 495, pulses 2, fluence 50.98 J/cm^2

As expected both 1-on-1 and S-on-1 damage thresholds were lower after exposure in vacuum if compared to case of air. Significant differences were observed in damage morphologies as well. Surroundings of damaged sites in vacuum were more clean than those produced in air. This is more likely due to the differences in mean free path for ejected particles and subsequent redeposition. In case of air redeposited layer of ablated particles is much more thick in air and resulted in colorfull newton rings due to gradient in thickness. On the other hand larger mean free path for ejected particles in vacuum increases the risk of contamination of all sample surface in closed space such as vacuum chamber as particles can be reflected back from chamber boundaries. This could be a possible scenario why the

LIDTs are lower in vacuum. There are also other assumptions: in example outgasing of chamber components that was nonbacked prior the present experimet,

4 Conclusions

In this measurement campaign undoped calcium fluoride crystals with and without coatings, with different applied coating techniques and treated by different polishing techniques were studied in damage threshold measurements at different temperatures. These samples are models for Yb-doped crystals that are used in short pulse laser amplifiers. Thresholds of coated samples were typically in the range of 6-12 J/cm^2, five times lower than for the pure uncoated crystal. Even if different temperatures, surface preparation techniques, and coating techniques result in different damage morphologies, they have less effect on the onset fluence value for laser damage. More investigations are needed to reveal this mismatch and to find hints for further improvements.

Nevertheless, it can be expected that in femtosecond laser amplifiers energy densities of up to 5 J/cm^2 are applicable, what allow reasonable high extraction efficiencies of up to 10%.

From measurements in S-on-1 regime it is evident that aging effects play a role in vacuum both for interference coatings and uncoated crystals: multi-shot LIDT is always lower than that of single shot LIDT. Furthermore, additional exposure in vacuum environment might result in changes of LIDT, thus additional investigations are necessary to evaluate vacuum exposure effects. This effect is more pronounced in the LIDT of HR coatings. The results of vacuum air measurement comparison confirmed the fact that LIDT is reduced due to exposure in vacuum.

5 References/Publications

[1] Gintarė Batavičiutė, Povilas Grigas, Linas Smalakys and Andrius Melninkaitis, Revision of laser-induced damage threshold evaluation from damage probability data, Review of Scientific Instruments, 84, 045108 (2013), DOI:http://dx.doi.org/10.1063/1.4801955

Research of VULRC will be published soon in:

Stefan Schrameyer, Heinrich Mädebach, Lars Jensen, Detlev Ristau, Clemens Heese, Jorge Piris, Alessandra Ciapponi, Bruno Sarti, Paul Allenspacher, Melanie Lammers, Wolfgang Riede, Andrius Melninkaitis, Gintare Bataviciute, Linas Smalakys and Valdas Sirutkaitis, "Round Robin experiment on LIDT measurements at 1064 nm in vacuum for space qualification of optics" Proc. SPIE, Est. pub. November 2013