

Investigation of Laser Damage Thresholds of an Output Coupler for TEA CO₂ Laser

Mohamad Nour Lababidi^{a*}, Khaled Mayya^a

^aHigher Institute for Applied Sciences and Technology (HIAST), Department of Applied Physics, 31983, Damascus, Syria.

Received: January 13, 2022; Revised: June 09, 2022; Accepted: June 12, 2022

Output couplers for TEA CO₂ lasers were made from Ge substrate coated with dielectric layers of ZnS, Ge and Y₂O₃ using physical evaporation technique (PVD). The laser-induced damage thresholds (LIDTs) of manufactured mirrors have been investigated using an experimental setup, based on TEA CO₂ laser. The effect of preparation conditions, such as grinding, polishing and cleaning, is shown. The maximum value of LIDT about 8 J/cm² was achieved using a fine grinding with loose abrasive grade 3 μm, then polishing with alumina powder grade 0.3 μm and finally hot cleaning with ultrasonic waves.

Keywords: Laser-induced damage thresholds, optical materials, coupler mirrors, CO₂ laser.

1. Introduction

TEA CO₂ laser is still considered very perspective and used for many applications such as marking, paint stripping, non-destructive ultrasonic testing and there has been a renewed interest in its high power and high repetition rate especially for dielectric materials in micro- and nano- engineering^{1,2}. One of the key technologies involved in the successful design and construction of TEA CO₂ laser is to manufacture a damage-resistant optics for use internally in laser resonator or externally to manipulate the output beam. There are many optical materials used successfully in this field such as Ge, ZnSe, NaCl, KCl^{1,3}. Nevertheless, the damage threshold of these materials depends on many parameters: bulk material properties^{4,5}, methods of optical surface preparation^{6,7}, coating methods^{8,9} and finally treatment (especially laser treatment) of optical surface before or after coating^{10,11}.

The optical surface preparing methods (mainly grinding and polishing) give three main layers: a smooth surface layer or hydrated layer (thickness up to 100 nm), a subsurface defect layer (thickness up to 1 μm) and then the bulk material. In the case of uncoated optical surfaces, all damages initiate from surface layer (scratches, digs, micro- or nano-inclusions) or from subsurface defect layer¹²⁻¹⁴.

In the case of coated optical surfaces, the situation is more complicated, because the defects arising from the coating process often play a major role in the beginning of the emergence of damage, which are intrinsically related to the structure of the optical surface^{15,16}.

Following our success in the previous reports work¹⁷, a new and inexpensive method for manufacturing an output coupler for a relatively high peak power and pulse repetitive TEA CO₂ laser has been demonstrated. This output coupler provides an ideal transmittance coefficient for the indicated laser and, on the other hand, withstands the nominal value of laser peak power.

The transmittance coefficient of the indicated output coupler was measured in the range 18-22% at the laser

emission wavelength ($\lambda=10.6 \mu\text{m}$). Therefore, when using in laser resonator, it has been noticed a significant improvement in the laser pulse energy value. This previous used mirror was uncoated germanium ($T_{oc}=46\%$) and the laser pulse energy did not exceed 300 mJ, while when using the new manufactured mirror in our laboratory, the energy of the laser pulse was improved and became about 430 mJ.

This paper reports the verification of new finding as well as the parameters affecting the laser-induced damage threshold (LIDT) on the fabrication parameters affecting the laser damage threshold with emphasizing to manufacture these coupler mirrors locally with high efficiency output and relatively low cost.

2. Samples design and preparation

In order to study and measure LIDT, a set of samples were prepared with the specifications shown in Table 1. They are all manufactured according to the design mentioned in the article¹⁷, a germanium substrate (6 mm thick, 30 mm diameter) coated with multi layers of ZnS and Ge dielectric materials and an Y₂O₃ protection layer. Samples were prepared by applying different conditions of grinding, polishing and final cleaning processes prior to deposition. It's worth mentioning that our optical workshop is intended for manufacturing traditional optics usually used in visible and IR system and for the first time is used to prepare damage-resistant laser optics.

The first grinding process G1 was carried out using loose abrasive in three stages, starting at 25 μm grain size, then 12 μm and finally 6 μm, for a period of 20 min for each grain size degree. Fine grinding of the second type G2 was achieved also using loose abrasive in four stages, starting at 25 μm grain size, then 12 μm, then 6 μm and finally 3 μm for a period of 20 min for each grain size degree. (with these parameters: rotation speed $v=15 \text{ rpm}$, arm frequency $f=0.3\text{s}^{-1}$, pressure $p=56 \text{ g/cm}^2$ in grinding and polishing machine -4axis).

The polishing of the first type P1 was carried out using of diamond powder of 1 μm grain size and then 0.3 μm,

*e-mail: mhamadnour.lababidi@hiast.edu.sy

Table 1. Preparation conditions of samples.

Notes	Cleaning	Polishing	Grinding	
Grinding 6 –polishing Al-cold cleaning	C1	P2	G1	Sample N1
Grinding 6 –polishing dia-cold cleaning	C1	P1	G1	Sample N2
Grinding 3 –polishing Al-cold cleaning	C1	P2	G2	Sample N3
Grinding 3 –polishing dia-cold cleaning	C1	P1	G2	Sample N4
Grinding 6 –polishing Al-hot cleaning	C2	P2	G1	Sample N5
Grinding 6 –polishing dia-hot cleaning	C2	P1	G1	Sample N6
Grinding 3 –polishing Al-hot cleaning	C2	P2	G2	Sample N7
Grinding 3 –polishing dia-hot cleaning	C2	P1	G2	Sample N8

while the polishing of the second type P2 was carried out in two stages, the first using alumina powder of grain size of 1 μm and then 0.3 μm . In both cases, a polisher (polishing pad) of the pitch type was used with distilled water at 25°C. (with these parameters: $v=66$ rpm, $f=0.17\text{s}^{-1}$, $p=50$ g/cm² in grinding and polishing machine -4axis).

Cleaning process was traditional ultrasonic waves at a frequency of 35 MHz in distilled water as a first stage and then in alcohol as a second stage for an hour in each stage. The first type of cleaning C1 was carried out at 20°C and the second type of cleaning C2 was carried out at 45°C.

All experiments were done at a laboratory temperature.

3. LIDT measurement

In order to measure the LIDT threshold, we have prepared the experimental arrangement shown in Figure 1. The TEA CO₂ laser used in the measurements has an internal design as in¹⁸ but its technical specifications as indicated in Table 2. The distribution of energy in the laser spot (Figure 2 but with a diaphragm of 15 mm diameter) has approximately a flat-top (top-hat) type, which is usually used in measuring LIDT according to ISO-21154-2011 (of course in addition to Gaussian type)¹⁹⁻²². The laser pulse shape is characteristic for TEA CO₂ lasers and consists of two parts as shown in Figure 3: the first high spike (100 ns, 30% pulse energy) and the second low tail (2.5 μs , 70% pulse energy). Therefore, pulse power is equal to 1.15 MW. The aim of using such type of laser and with these specifications is to measure LIDT of the mirrors under the same operating conditions they will be used later.

FL focusing lens of meniscus type, focal point $f=5''$ (127mm), diameter 33mm, made of ZnSe material, was used so the diameter of laser spot was 0.85 mm; that is, the highest power density that could be obtained using this experimental arrangement was 13.67 J/cm² by using the laser beam directly without beam splitter, BS or attenuator²³. When attenuator and the laser beam splitter are used, the obtained power density in the lens focal point can be in the range 0.2–9.8 J/cm².

The damage criterion was monitored by measuring the distortion of the scattered beam from the laser effective area. This was obtained by measuring the scattering of Helium-Neon laser beams (online measurement) using a Si-PD silicon photodiode and then examining the samples under the microscope after removing from the two-dimensional XY target holder (offline measurement).

Table 2. Specifications of the laser used in the measurement.

TEA CO ₂	Laser type
350 mJ	Pulse energy
100 ns	Pulse width
25 Hz	Maximum pulse frequency
15×15 mm ²	Laser spot dimensions
7.4	Beam quality parameter M²
± 2.5%	Pulse-to-Pulse Stability

The pulse energy was measured using an EM energy meter (model PM100D with sensor probe model ES245C²⁴). The samples were fixed on a movable optical holder in the XY plane in the focal plane of the lens, which was placed in a clean chamber that was fed with clean dry air at atmospheric pressure and temperature of 20°C. The angle of incidence of the laser beam on the samples is 10° and a damper was used to eliminate the effects of the reflected beam.

LIDT was measured using the protocol adopted in the international standard ISO-21254¹⁹⁻²², especially the first and second tests: the first is the one-shot test, or what is called 1-on-1 test, and the second is the multi-shot test or what it is called S-on-1. In both tests, 30 positions on the sample were exposed to laser pulses, and based on the experimental data; the damage probability curve of the sample was extracted according to the model shown in Figure 4. The value of the LIDT was determined in Figure 4 based on its definition as the highest power density at which the optical element has a zero damage probability. The accuracy of the LIDT measurement depends on many factors, the most important of which are: the accuracy of measuring the laser pulse energy, the stability of the laser energy from pulse to pulse, the accuracy of measuring the diameter of the focused laser spot, the accuracy of the damage criterion and the values between online or offline measurement. In our case, the overall accuracy was in the range ±15%, while the offline measurement was less than the online measurement by 30%, as we will see in Figure 5 while discussing the results.

4. Results and Discussion

Before applying deposition on the surface of the samples according to the design detailed in the article¹⁷, the surface roughness of the eight different samples in Table 1 was measured using Tencor Alpha Step 200 system and they had similar roughness value about $R_a=0.9$ nm. Their degree of scratches and digs were also estimated; where the S/D threshold ratio was 5/10. It is worth mentioning that the

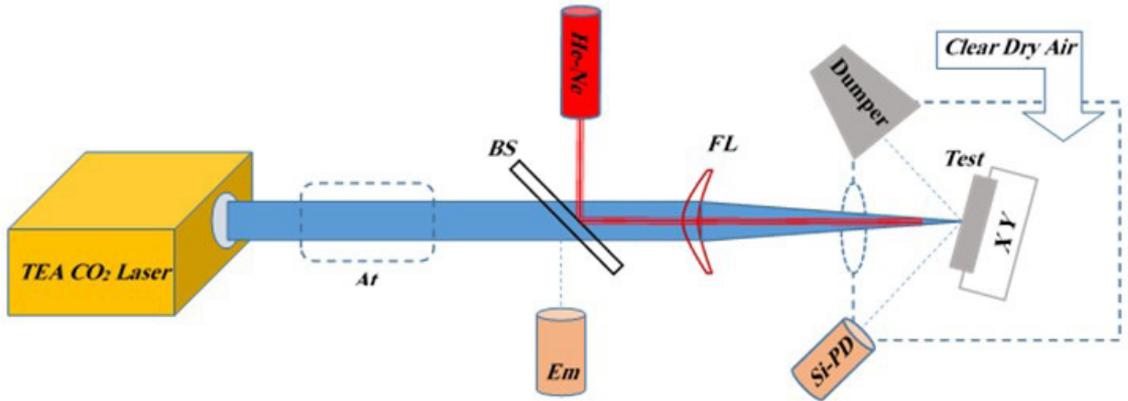


Figure 1. LIDT measurement Setup.



Figure 2. Laser Spot Trace on a Fax Paper.

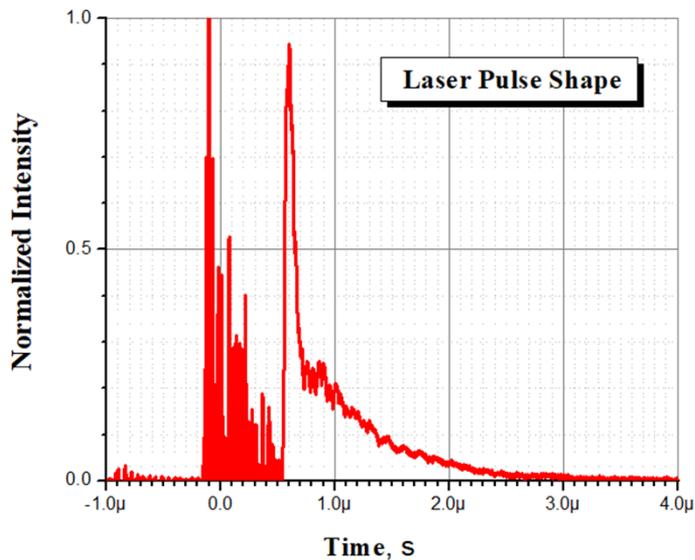


Figure 3. Laser Pulse Temporal Shape .

polishing period of 40 h was sufficient to achieve a clean surface from scratches and digs as well as accepted degree of roughness, while increasing the polishing time did not give better results.

LIDT for the substrate (uncoated germanium) was measured before coating, and we did not observe any damage. This is mostly attributed to the fact that the substrate damage threshold ($0.6\text{--}11\text{ MW/mm}^2$ at 100ns^4) is much greater than

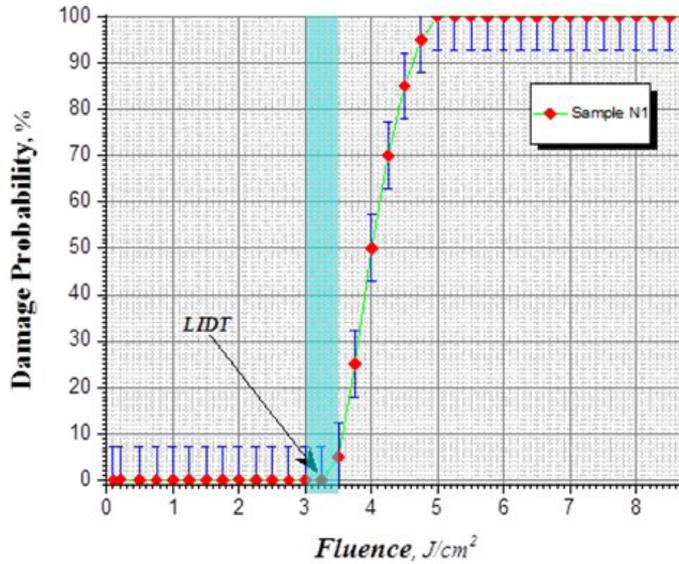


Figure 4. An example of the sample damage probability curve N°1.

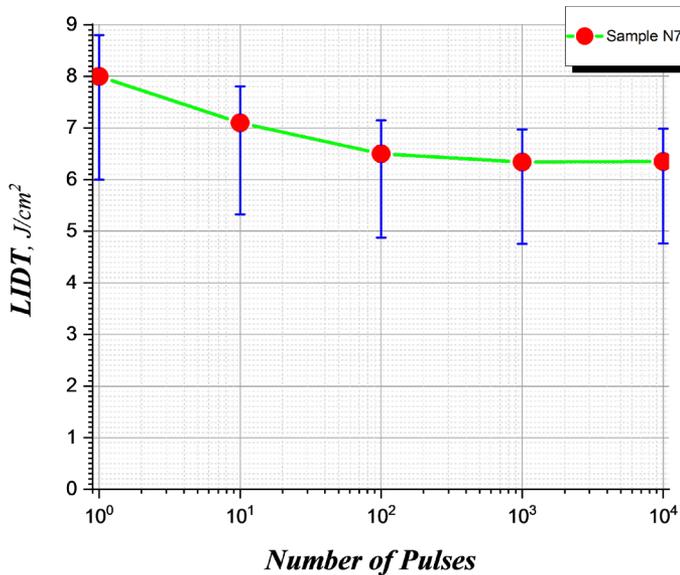


Figure 5. Results of S-on-1 test for sample N°7 in Table 1.

the power density that can be achieved by the laser in Figure 1; that is 13.67 J/cm^2 .

The results of the single-pulse test measurements in Figure 6 show that the damage threshold is in the range of $2.75\text{--}8 \text{ J/cm}^2$ and that the best sample is the sample N°7. This is due to the fine grinding process that makes the subsurface defect layer at its lowest limits. Additionally, polishing with alumina powder gives better results than diamond powder, although the fineness of both grains is the same $0.3 \mu\text{m}$. We also noticed that the type of damage in the samples polished with diamond powder is different from that using alumina samples as shown in the Figure 7. Cleaning process is effective to dispose of polishing powder residues, but not sufficient. What is required for a more indicative comparison is the use

more developed methods of post-polishing processes, such as MRF (Magneto-Rheological Finishing) accomplished with etching, then laser conditioning¹⁰. Unfortunately, these techniques are not available in our laboratory.

As for the results of the multi-pulse measurement S-on-1, Figure 5 shows a characteristic curve for the best sample N°7 where the LIDT value decreases from 8 J/cm^2 for one pulse to a stable value of about 6.3 J/cm^2 for the number of pulses greater than 100. The curve is repeated for the rest of the samples but the difference between the LIDT value for a single pulse and multiple pulses is much larger and that mainly relates to the nanoprecursor absorption centers embedded in the surface or subsurface structure.

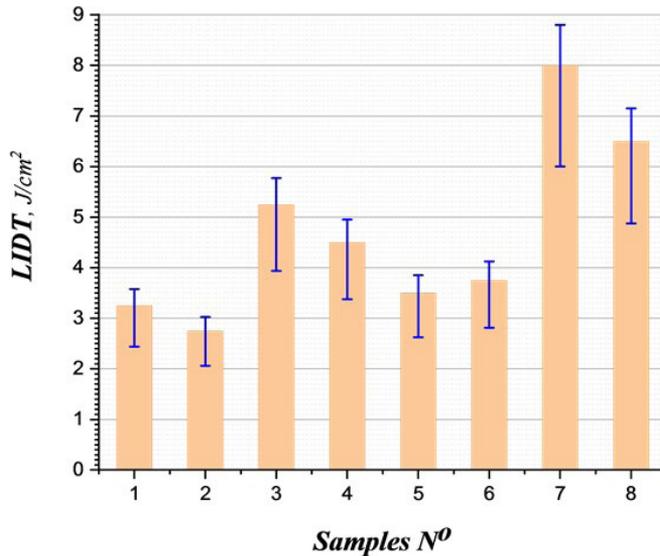


Figure 6. Results of 1-on-1 test for the eight samples in Table 1.

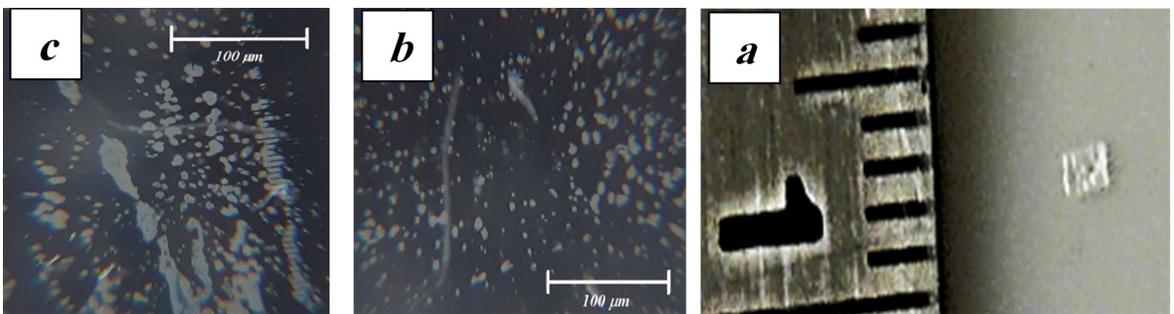


Figure 7. Morphology of damage (a) Polyamide sample; (b) Sample N°7; (c) Sample N°8.

5. Conclusion

In this work, we have measured the LIDT of a relatively inexpensive laser output mirror made from dielectric coating on a germanium substrate. The effect of mirror manufacturing conditions (grinding, polishing, and cleaning) on the LIDT value was studied and the ideal method (using traditional tools of own optical workshop in our laboratory) was determined to obtain the highest LIDT threshold of 8 J/cm² by applying fine grinding and polishing processes using 0.3 µm alumina powder and hot cleaning. In general, we have used similar bulk substrate materials but in form of thin nanolayers materials to enhance its characterizations and adapt these new prepared nanolayers materials for various applications in the optic field science.

The main goal is to obtain an output mirror within the required specifications, and to improve and develop it within the available capabilities and through the effect of surface treatments using traditional methods, which, as we have shown in this article, have a significant impact on the beginning of the formation of defects that affected the threshold of laser sabotage of the studied output mirror with the possibility of studying also the mechanisms of Removing abrasive and polishing materials.

6. Acknowledgement

The authors would like to thank the HIAST (Higher Institute for Applied Sciences and Technology) for encouragement and support.

7. References

1. Steen WM, Mazumder J. Laser material processing. 4th ed. USA: Springer; 2010. 567 p.
2. Jitsuno T, Uno K. CO₂ lasers. In: Sugioka K, editor. Handbook of laser micro- and nano-engineering. USA: Springer; 2021. 689 p.
3. Patel BS. Optical suitability of window materials for CO₂ lasers. Appl Opt. 1977;16(5):1232-5.
4. Wood RM. Laser induced damage of optical materials. Reino Unido: IOP Publishing; 2003. 241 p.
5. Ristau D. Laser induced damage in optical materials. Boca Raton: CRC Press; 2015. 544 p.
6. Papernov S, Schmid AW. Laser-induced surface damage of optical materials: absorption sources, initiation, growth, and mitigation. Proc SPIE. 2008;7132:71321J.
7. Bloembergen N. Role of cracks, pores, and absorbing inclusions on laser induced damage threshold at surfaces of transparent dielectrics. Appl Opt. 1973;12(4):661-4.

8. Kozłowski MR. Damage-resistant laser coatings. In: Flory FR, editor. *Thin films for optical systems*. New York: Marcel Dekker; 1995. p. 521-49.
9. Stoltz CJ. Laser resistant coatings. In: Kaiser N, editor. *Optical interference coatings*. New York: Springer; 2003. p. 309-35.
10. Menapace JA, Davis PJ, Steele WA, Wong LL, Suratwala TI, Miller PE. MRF applications: measurement of process-dependent subsurface damage in optical materials using the MRF wedge technique. *Proc SPIE*;2005;5991:599103.
11. Lytvynenko IV, Maruschak PO. Analysis of the state of the modified nanotitanium surface with the use of the mathematical model of a cyclic random process. *Optoelectron Instrum Data Process*. 2015;51(3):254-63.
12. Paul Hed P, David FE. Optical glass fabrication technology. 2: relationship between surface roughness and subsurface damage. *Appl Opt*. 1987;26(21):4766-80.
13. Juškevičius K, Buzelis R, Kičas S, Tolenis T, Drazdys R, Batavičiūtė G, et al. Investigation of subsurface damage impact on resistance of laserradiation of fused silica substrates. *Proc SPIE*;2013;8885:888529.
14. Han W, Wang F, Zhou L, Li F, Feng B, Jia H, et al. Effect of laser beam size on laser-induced damage performance. *Proc SPIE*. 2011;8190:819012.
15. Kozłowski MR, Chow R. The role of defects in laser multilayer coatings. *Proc SPIE*. 1993;2114:640-9.
16. Dijon J, Poiroux T, Desrumaux C. Nano absorbing centers: a key point in the laser damage of thin films. *Proc SPIE*. 1997;2966:315-25.
17. Lababidi MN, Mayya K, Hassan M. Implementation of output coupler mirrors for high damage threshold CO₂ laser: new technical approach. *J Mater Environ Sci*. 2021;12(11):1383-91.
18. Qu YC, Liu FM, Hu XY, Ren DM, Zhao JS. Miniature high-repetition-rate TEA CO₂ laser with surface wire corona preionization. *Infrared Phys Technol*. 2000;41:139-41.
19. ISO: International Organization for Standardization. ISO 21254-1:2011. *Lasers and laser-related equipment—Test methods for laser-induced damage threshold—Part 1: Definitions and general principles*. Geneva: ISO; 2011.
20. ISO: International Organization for Standardization. ISO 21254-2:2011. *Lasers and laser-related equipment—Test methods for laser-induced damage threshold—Part 2: Threshold determination*. Geneva: ISO; 2011.
21. ISO: International Organization for Standardization. ISO 21254-3:2011. *Lasers and laser-related equipment—Test methods for laser-induced damage threshold—Part 3: Assurance of laser power (energy) handling capabilities*. Geneva: ISO; 2011.
22. ISO: International Organization for Standardization. ISO 21254-4:2011. *Lasers and laser-related equipment—Test methods for laser-induced damage threshold—Part 4: Inspection, detection and measurement (Technical Report)*. Geneva: ISO; 2011.
23. MacPherson RW. Variable attenuator for TEA CO₂ lasers. *Rev Sci Instrum*. 1973;45:316-7.
24. Thorlabs Co. THORLABS catalogue. Vol. 20. Newton: Thorlabs Co.; 2008. Light analysis catalogue; p. 1264-1351.