

LANE 2012

## D-scan measurement of the ablation threshold and incubation parameter of optical materials in the ultrafast regime

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### Abstract

Machining with ultrafast laser pulses demands the selection of correct conditions to obtain precise, and yet, efficient material extraction, in which the control of volume and physical state of the matter being etched is fundamental. Usually, the production of volumetric structures needs overlapping of many pulses, and the incubation effects and its dependence on process parameters are of prime importance. Hence, in this work this parameter and the damage thresholds for many different pulses overlapping were measured for some optical glasses with an alternative method which is faster than the traditional one, and is closer to the real machining condition.

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*Keywords:* ultrashort pulses; femtosecond machining; damage threshold

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### 1. Introduction

Sculpting complex structures with femtosecond laser pulses is becoming commercially feasible mainly due to the development of new systems with improved average power. In such commercial framework, not only batch production needs a high throughput but, also the first prototypes need to be quickly produced and optimized. This is only possible when a process chart has already been developed for that specific material and design. It is fundamental to know the exact response of the material to the process parameters being used, not only to obtain the better dimensional accuracy, but also to improve cosmetic appearance and decrease collateral effects.

Hence, the damage threshold in the high and low fluence regime ranges as function of laser parameters must be known, and also related to the effects caused to the part being processed. Due to incubation effects however, these relations change as laser shots are accumulated on a unique spot. This is the case

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of *milling* surfaces where pulses superposition along with pulse density determinate the area and depth of the ablated volume. Even this incubation effect may vary for different process parameters, like temporal pulsewidth and energy density.

With the aim of studying the influence of some laser parameters on the incubation factor, and at the same time obtaining fast acquisition of related data, this work has developed a method suitable to measure the ablation threshold  $F_{th}$  as function of the number  $N$  of overlapped pulses closely reproducing the case of real machining.

Ultrashort pulses ablation is a nonlinear process [1], [2], [3] that also depends on the presence of impurities, defects, excitons, etc. [4], [5], [6], which either create intermediate levels in the bandgap or modify the local electronic density and lower the ablation threshold fluence  $F_{th}$  values. These defects are frequently produced when processing solids with superimposed shots which lower the  $F_{th}$  for subsequent pulses. These are cumulative phenomena, known as incubation effects [7], [8], [9], and modifications in  $F_{th}$  induced by them, change the ablated volume as function of  $N$ , and therefore must be taken into account when machining a material.

## 2. Experimental

This work used the Diagonal Scan (D-Scan) technique [10], [11], instead of the traditional “zero damage” method [12], to measure the ablation threshold as function of the pulses overlapping  $N$ . In this case, the sample is placed with its surface orthogonally to a  $TEM_{00}$  Gaussian beam, and is moved in two directions simultaneously, parallel and perpendicular to the beam axis. In a position close to the beam waist, an etched profile like shown in Fig. 1 will appear. The ablated track exhibits a minimum width at the focus position and two maximum lobes with width  $2\rho_{max}$ , symmetrically located before and after it.

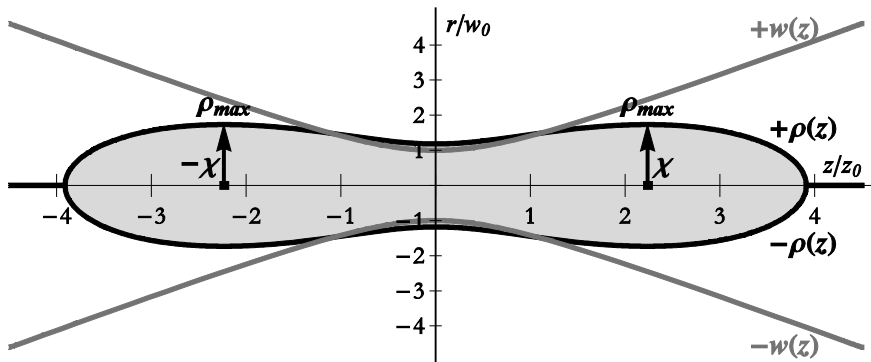


Fig. 1. Profile etched in the sample surface which has two maximum transversal lobes at position  $\chi$  with dimension  $2\rho_{max}$ . The lines  $+w(z)$  and  $-w(z)$  represents the laser beam radius at  $1/e^2$  of peak intensity

If there are no significant heat effects, and the experiment is performed above certain intensity, it can be shown [10] that for  $TEM_{00}$  Gaussian beam, the ablation threshold is directly related to the  $\rho_{max}$  dimension and the laser pulse energy  $E_0$ , through the very simple expression:

$$F_{th} = \frac{E_0}{e\pi\rho_{max}^2} \cong 0.117 \frac{E_0}{\rho_{max}^2}, \quad (1)$$

To account for incubation effects, the superposition of  $N$  different shots is considered as the ratio between the summation of the intensities produced at  $(\chi, \rho_{\max})$  by every pulse that hits the sample during its movement, and the intensity generated by the pulse centered at  $(\chi, 0)$ . Under this assumption, it can be shown [13] that:

$$N = \vartheta_3\left(0, e^{-\left(\frac{v_y}{f\rho_{\max}}\right)}\right), \quad (2)$$

where  $\vartheta_3$  is the Jacobi elliptic theta function of the third kind,  $f$  is the laser repetition rate and  $v_y$  is the sample transversal translation speed. For high repetition rates and low translation speeds, Eq. 2 can be approximated to:

$$N \sim 1.8f\rho_{\max}/v_y \quad (3)$$

The number  $N$  only accounts for shots in the immediate vicinity of  $\chi$ , since shots relatively far from this position do not contribute for the ablation process. Due to the relative movement, the superposition  $N$  is different as the one used in the traditional method, but reproduces much more closely the real situation occurring during real machining.

### 3. Results and Discussion

The experiments reported here were performed with a CPA Ti:Sapphire laser system (Femtopower Compact Pro CE-Phase HP/HR from Femtolasers), continuously generating 25 fs (FWHM) pulses centered at 775 nm with 40 nm of bandwidth (FWHM), at a maximum repetition rate of 4 kHz. These pulses were used to measure  $F_{th}$  and the incubation parameter  $k$  for sapphire and the optical glasses BK7 and Suprasil. All irradiations were done in air, and after etching the samples were cleaned with isopropyl alcohol in an ultrasonic cleaner to remove redeposited ablation debris. After cleaning, the samples were observed and photographed on an optical microscope, and the ablation dimensions were measured in the micrographs.

Fig. 2 (a) shows D-scan profiles in a BK7 sample surface for three different energies (109, 184 and 61  $\mu\text{J}$ , from top to bottom). Pictures show top and lateral views of the etched tracks; in this case it is possible to observe the depth related to the focus position. It is clear that the deepest etch it is not produced when the focus is on the surface, but in a position slightly below it. Fig. 2 (b) shows three tracks produced in Sapphire surface for the same energy  $E_0$  and three different longitudinal superposition. The values of  $N$  and corresponding  $F_{th}(N)$  are shown along with the position where  $\rho_{\max}$  were measured.

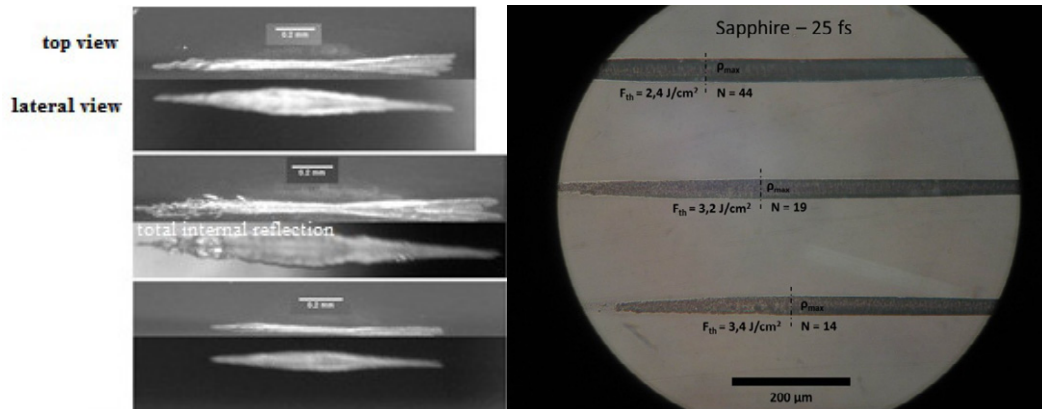


Fig. 2. (a) Top and lateral view of D-scan profiles in BK7 surface for three different energies: 109, 184 and 61  $\mu\text{J}$ , from top to bottom. (b) Tracks produced in Sapphire surface for the same energy  $E_0$  and three different longitudinal superposition, showing the position where  $\rho_{\text{max}}$  were measured. Obs.: in (a), only the bottom part of the lateral view must be considered, the top view is due to the total internal reflection

Fig. 3 shows part of the D-Scan track where  $\rho_{\text{max}}$  was measured on Sapphire surface. At the border of the etched trace the formation of ripples is observed. These Laser Induced Periodic Structures (LIPSS) [14], typical of ablation by Coulombian explosion, evidence that thermal effects were absent at this position.

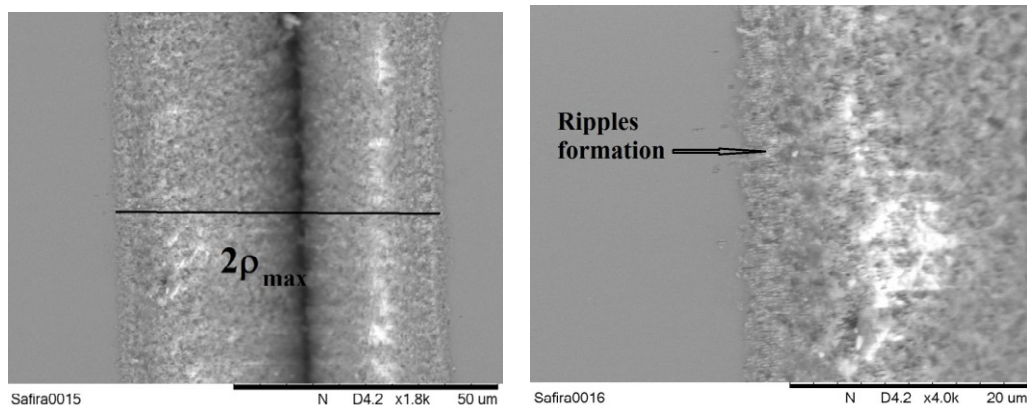


Fig. 3. (a) Region of the etching where  $\rho_{\text{max}}$  were measured in Sapphire. (b) Ripples formed at the edge of the track in Sapphire surface

Fig. 4 shows data for  $F_{\text{th}}$  as function of  $N$  measured in BK7 surface by the traditional and by the D-Scan methods, and in both cases, the laser beam was focused by a 38 mm of focal length lens. The number of superposed shots ranged from 1 to 1020; in the still sample case, the energies used were 2.9, 5, 7, 8.5, 12, 14 and 18.5  $\mu\text{J}$ , for D-Scan three energies were used, 31, 71 and 134  $\mu\text{J}$ . Different superposition conditions were obtained by combining the repetition rate with the sample transversal displacement speeds; these frequencies were 50, 100, 500, 1000, 2000 and 4000 Hz;  $v_y$  were 6, 12, 25, 50

and 100 mm/min. The longitudinal  $v_z$  displacements speeds, were chosen according to  $v_y$  to produce an elongated etched profile.

The results shown in Fig. 4 for the ablation thresholds for many pulses measured by the two methods shows a good agreement, validating the D-Scan method for  $F_{th}(N)$  measurements. An incubation effects model that considers a saturation of the defects accumulation in dielectrics, is given by [15]:

$$F_{th}(N) = F_{th\infty} + (F_{th1} - F_{th\infty}) \cdot \exp[-k(N-1)] \tag{4}$$

where  $k$  is the sample incubation parameter,  $F_{th}(N)$  and  $F_{th,1}$  are the ablation thresholds for  $N$  and single pulses, respectively, and  $F_{th,\infty}$  is the ablation threshold for infinite pulses, when saturation occurs.

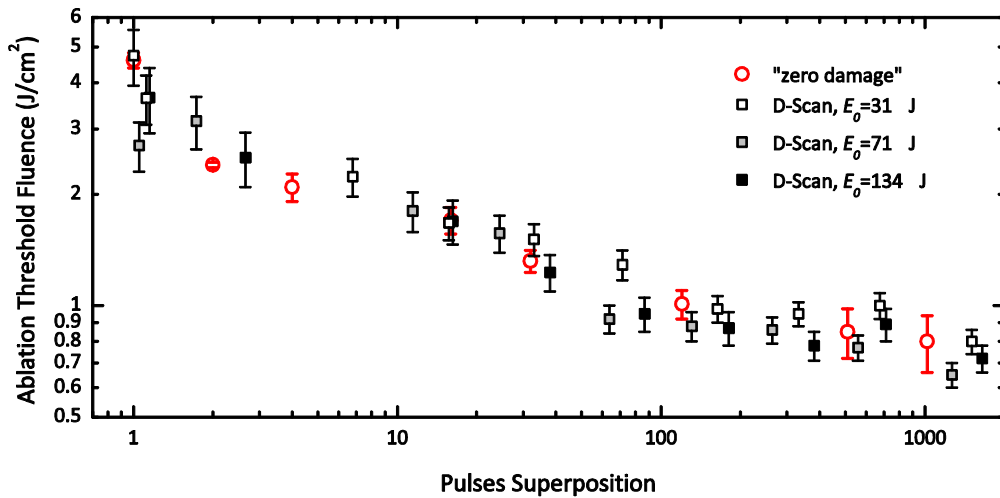


Fig. 4.  $F_{th}$  as function of  $N$  for BK7, obtained by the “zero damage” and the D-Scan methods

Fig. 5 shows the damage threshold  $F_{th}$  as function of  $N$ , and Eq. (4) fitted to the data, obtained by the D-Scan technic for Suprasil and Sapphire. Here the transversal speed was twice the longitudinal one ( $v_y = 2 \cdot v_z$ ), the focal length of the focusing lens was 50 mm and pulse width was 25 fs. Pulse energy of 94  $\mu$ J was used in the case of Suprasil and 126  $\mu$ J in the case of Sapphire. The parameters obtained from the fittings are shown in table 1.

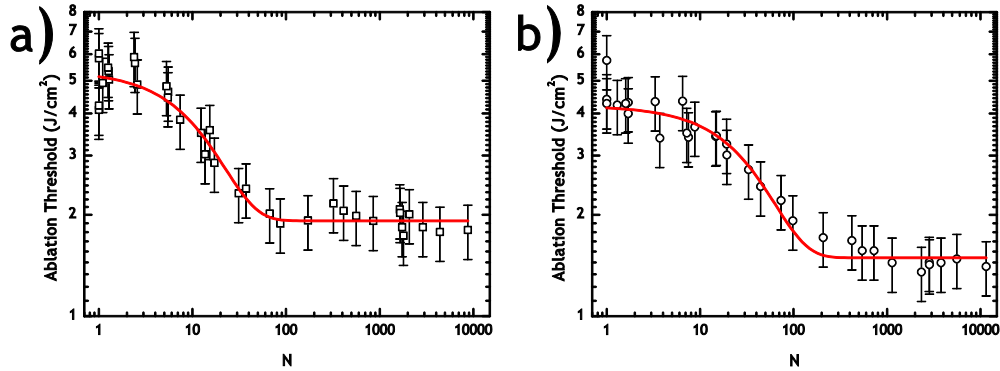


Fig. 5. Ablation thresholds as a function of the pulses superposition  $N$  for a) Suprasil and b) Sapphire. Red lines represents Eq. 4 fitted to the data

During the accumulation of some pulses, less than 10 in both cases, a decrease in the damage threshold is not evident, but after that, a strong incubation effect takes place and  $F_{th}$  decrease steadily with the number of overlapped shots. The figure is the same for both materials up to the appearance of saturation, which happens after approximately 80 pulses for Suprasil and for more than 200 pulses for Sapphire, reflecting lower  $k$  parameter for the last one.

Table 1. Fit parameters for the data shown in Fig. 5

Sample	$F_{th,1}$ ( $J/cm^2$ )	$F_{th,\infty}$ ( $J/cm^2$ )	$k$
Suprasil	5.1(1)	1.92(5)	0.064(8)
Sapphire	4.2(1)	1.49(4)	0.022(3)

It is evident from these numbers, that machining involving overlapping of pulses, mainly in the range where  $N < \sim 100$ , must take into account the variation of  $F_{th}(N)$ . This not only assures a higher accuracy, but also helps to keep the processes far from the high fluence condition. Based on these concepts, we proposed a method to “mill” dielectrics[16] in which engraving is performed by layers of different depths, where in each one the machining parameters are adjusted according to the number  $N$  previously stricken on the surface. Fig. 6 below shows an example of machining microchannels in BK7 where this method was used.

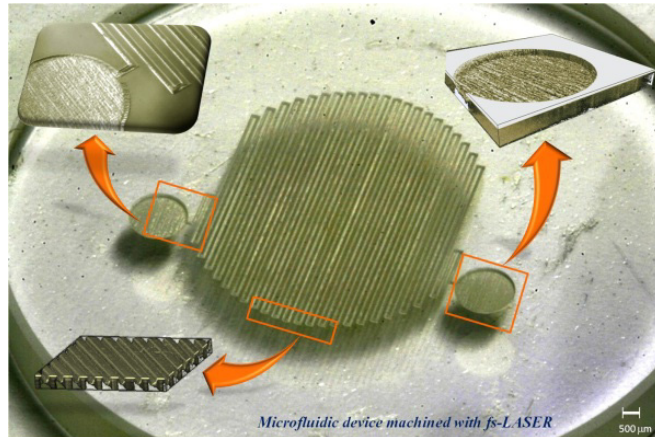


Fig. 6.  $100\mu\text{m} \times 100\mu\text{m}$  microchannels serpentine machined on BK7 optical glass surface. Details top right and bottom left were obtained by OCT

In conclusion, we demonstrated that D-Scan is a valid and useful method for the measurement of the ablation thresholds and incubation parameters for the superposition of ultrashort pulses for dielectrics. The use of these values to adjust process parameters during machining progression helps to avoid unwanted heat effects and to obtain increased dimension accuracy.

### Acknowledgements

This work was supported by FAPESP (grants 2008/00284-0 and 200907912-0) and CNPq (grants 20094732394-2 and 20093101119).

### References

- [1] Brinson, H. F.; Brinson, C. L.: Polymer Engineering Science and Viscoelasticity - An Introduction. Springer, New York, 2008.
- [2] N. Bloembergen, "Laser-Induced Electric Breakdown in Solids," IEEE J. Quantum Elec. **QE10**, 375-386 (1974).
- [3] D. Du, X. Liu, G. Korn, J. Squier, and G. Mourou, "Laser-induced breakdown by impact ionization in  $\text{SiO}_2$  with pulse widths from 7 ns to 150 fs," Appl. Phys. Lett. **64**, 3071-3073 (1994).
- [4] L. C. Courrol, R. E. Samad, L. Gomes, I. M. Ranieri, S. L. Baldochi, A. Z. de Freitas, and N. D. Vieira, "Color center production by femtosecond pulse laser irradiation in LiF crystals," Opt. Expr. **12**, 288-293 (2004).
- [5] F. Costache, S. Eckert, and J. Reif, "Near-damage threshold femtosecond laser irradiation of dielectric surfaces: desorbed ion kinetics and defect dynamics," Appl. Phys. A **92**, 897-902 (2008).
- [6] M. Mero, B. Clapp, J. C. Jasapara, W. Rudolph, D. Ristau, K. Starke, J. Kruger, S. Martin, and W. Kautek, "On the damage behavior of dielectric films when illuminated with multiple femtosecond laser pulses," Opt. Eng. **44**, 051107 (2005).
- [7] S. Martin, A. Hertwig, M. Lenzner, J. Kruger, and W. Kautek, "Spot-size dependence of the ablation threshold in dielectrics for femtosecond laser pulses," Appl. Phys. A **77**, 883-884 (2003).
- [8] D. Ashkenasi, M. Lorenz, R. Stoian, and A. Rosenfeld, "Surface damage threshold and structuring of dielectrics using femtosecond laser pulses: the role of incubation," Appl. Surf. Sci. **150**, 101-106 (1999).

- [9] Y. Jee, M. F. Becker, and R. M. Walser, "Laser-induced damage on single-crystal metal surfaces," *J. Opt. Soc. Am. B* **5**, 648-659 (1988).
- [10] R. E. Samad and N. D. Vieira, "Geometrical method for determining the surface damage threshold for femtosecond laser pulses," *Las. Phys.* **16**, 336-339 (2006).
- [11] R. E. Samad, S. L. Baldochi, and N. D. Vieira Jr, "Diagonal scan measurement of Cr:LiSAF 20 ps ablation threshold," *Appl. Opt.* **47**, 920-924 (2008).
- [12] J. M. Liu, "Simple technique for measurements of pulsed Gaussian-beam spot sizes," *Opt. Lett.* **7**, 196-198 (1982).
- [13] L. M. Machado, R. E. Samad, W. de Rossi, and N. D. Vieira Junior, "D-Scan measurement of ablation threshold incubation effects for ultrashort laser pulses," *Opt. Expr.* **20**, 4114-4123 (2012).
- [14] Reif J, Costache F, Varlamova O, Jia G, Ratzke M (2009) Self-organized regular surface patterning by pulsed laser ablation. *Phys. Status Solidi C* **6**: 681-686.
- [15] Ashkenasi D, Lorenz M, Stoian R, Rosenfeld A (1999) Surface damage threshold and structuring of dielectrics using femtosecond laser pulses: the role of incubation. *Appl. Surf. Sci.* **150**: 101-106.
- [16] Machado L M, Samad R E, de Freitas A Z, Vieira Jr N D, de Rossi W (2011) 37 Microchannels Direct Machining using the Femtosecond Smooth Ablation Method. *38 Phys. Procedia* **12**: 67-75.