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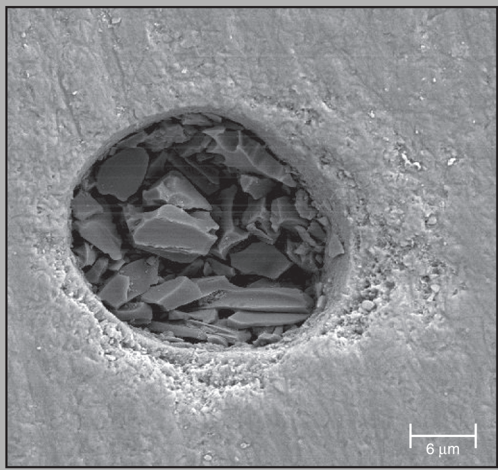
Abstract: The study of the interaction of intense laser light with matter, as well as transient response of atoms and molecules is very appropriated because of the laser energy concentration in a femtosecond optical pulses. The fundamental problem to be solved is to find tools and techniques which allow us to observe and manipulate on a femtosecond time scale the photonics events on and into the matter. Six third human extracted molars were exposed to a femtosecond Ti:Sapphire Q-switched and mode locked laser (Libra-S, Coherent, Palo Alto, CA, USA), emitting pulses with 70 fs width, radiation wavelength of 801 nm, at a constant pulse repetition rate of 1 KHz. The laser was operated at different power levels (70 to 400 mW) with constant exposition time of 10 seconds, at focused and defocused mode. Enamel and dentin surfaces were evaluated concerned ablation rate and morphological aspects under scanning electron microscopic. The results in this present experiment suggest that at the focused mode and under higher average power, enamel tissues present microcavities with higher depth and very precise edges, but, while dentin shows a larger melt-flushing, lower depth and melting and solidification aspect. In conclusion, it is possible to choose hard or soft ablation, under lower and higher average power, respectively, revealing different aspects of dental enamel and dentin, depending on the average power, fluence and distance from the focal point of the ultra-short pulse laser on the tooth surface.



Well-defined edges and original feature into microcavity is observed under an average power of 191 mW (300×), at enamel surface, using focused position

Selective ablation of dental enamel and dentin using femtosecond laser pulses

R.F.Z. Lizarelli, [1,*] *M.M. Costa,* [1] *E. Carvalho-Filho,* [1] *F.D. Nunes,* [2] *and V.S. Bagnato* [1]

[1] Physics Institute of São Carlos, USP, São Carlos, SP, Brasil
[2] Electronics and System Department, Federal University of Pernambuco, Recife, PE, Brasil

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1. Introduction

The use of ultrafast laser pulses is having an impact on materials and biological tissues processing in profound ways. Machining with femtosecond pulses affords considerable advantages over nanosecond pulses, such nanoselective removal, where ablated spot dimensions are below that achieved when longer pulses are focused to the minimum spot size dictated by optical physics.

The most important aspect is that the laser pulse is significantly shorter than the time it takes for electrons to transfer their energy to the lattice via electron-phonon coupling [1].

* Corresponding author: e-mail: lizarelli@if.sc.usp.br

The laser has firmly established itself as an indispensable tool for several applications in many areas. One branch of laser processing is laser micromachining, which generally includes machining with feature sizes less than 100 μm. Several laser systems for the removal and preparation of hard dental tissues are currently under intense investigation and some procedures are already in clinical use. The laser as a replacement for mechanical tools in many hard tissue applications is still strongly debated. One of the strongest limitations is the slow material removal rate, and, in several cases, unacceptable collateral damage, usually caused by overheating [2–9].

High power densities can promote plasma-induced ablation achieving a very clean or well-defined tissue removal. Ultra-short laser pulses (USLP) being certainly the situation, and many femtosecond pulses applications are presently under investigation [10–13]. During the last ten years many published papers have presented the general aspects of femtosecond laser ablation [11,14–17].

Materials and biological tissues at nano dimensions exhibit totally different properties compared to their bulk and atomic states. The understanding of resulted surfaces and subsurfaces of irradiated dental tissues could open up innovative and more conservative therapies to dental hard tissues pathologies. One of the arguments to use laser in the removal of dental hard tissue is the possible selectivity that can be achieved. It is possible to remove portions of the tissue that presents different compositions using short pulses of high power densities [15–17]. This possibility opens up a new type of selectivity ablation, which may allow an ultra-conservative dentistry procedure.

The ablation of dental hard tissue in the femtosecond regimen is still in a very early stage. While comparisons of morphological aspects using micro, nano- and picosecond have been widely studied, there is still a lack of data regarding the adequate morphological aspects using femtosecond laser ablation. The purpose of this study is to present interesting observed aspects during laser ablation of human teeth: selective and controlled ablation.

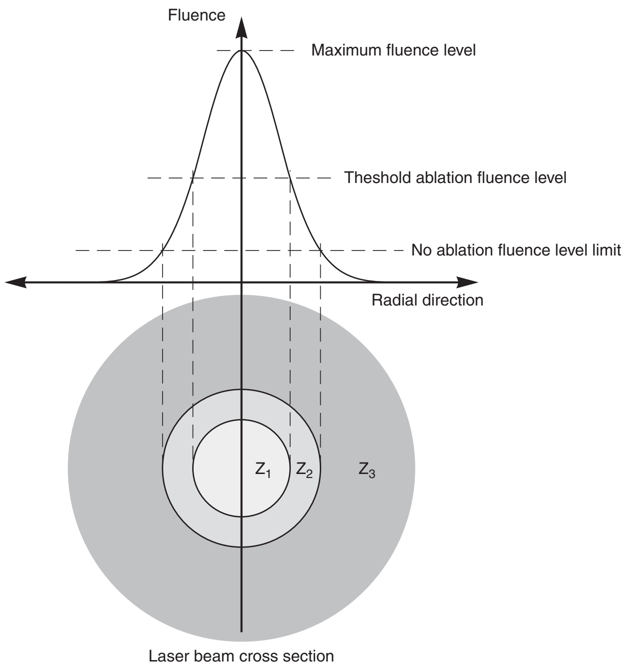

Figure 1 Laser beam cross section regions exhibiting different regions with borders defined by fluence levels corresponding to threshold ablation and no ablation fluence limit

2. Materials and methods

Six third human extracted molars were chosen for the present investigation. Roots were removed and buccal surfaces were cut (IsoMet 1000, Buehler Ltd., Lake Bluff, IL, USA) and polished with 600-grit water sand paper, resulting in flattened areas with dimensions of 5.0×5.0 mm^2 in enamel and dentin. This procedure was done to allow better laser coupling at 90°.

Three dental crowns were fixed on a xyz translation stage in order to provide spatial positioning relative to the laser beam. The prepared surfaces were placed at the focus of a 20.0 cm focused length lens, and under defocused mode at distances of – 20.0 mm, – 10.0 mm, + 10.0 m, and + 20.0 mm.

The laser used in the experiment was a femtosecond Ti:Sapphire Q-switched and mode locked laser (Libra-S, Coherent, Palo Alto, CA, USA), emitting pulses with 70 fs width, radiation wavelength of 801 nm, at a constant pulse repetition rate of 1 KHz. At the focus, the Gaussian laser beam presents a waist (ω_0) of 20 μm.

The laser was operated at different power levels (70 to 400 mW) with constant exposition time of 10 seconds. Under such conditions, tooth exposure to the laser beam produced microcavities which were examined to evaluate different aspects of the ablation. Only natural cooling was used.

3. Results and discussion

The obtained microcavities were observed under a scanning electron microscope (SEM). To evaluated these resulted microcavities, a classification of three possible distinct zones of the microcavities, previously suggested [18] through a SEM (scanning electron microscope) micro morphological analysis, was considered as follows: Z1 – region correspondent where the material was in fact removed and where the depth and diameter measures were made; Z2 – intermediate region between the removed region and the region around the original microcavity, being the margin or edge; and, Z3 – region around the original microcavity which was not laser irradiated. These three regions correspond to the region of the laser beam delimited by fluence levels as shown in Fig. 1.

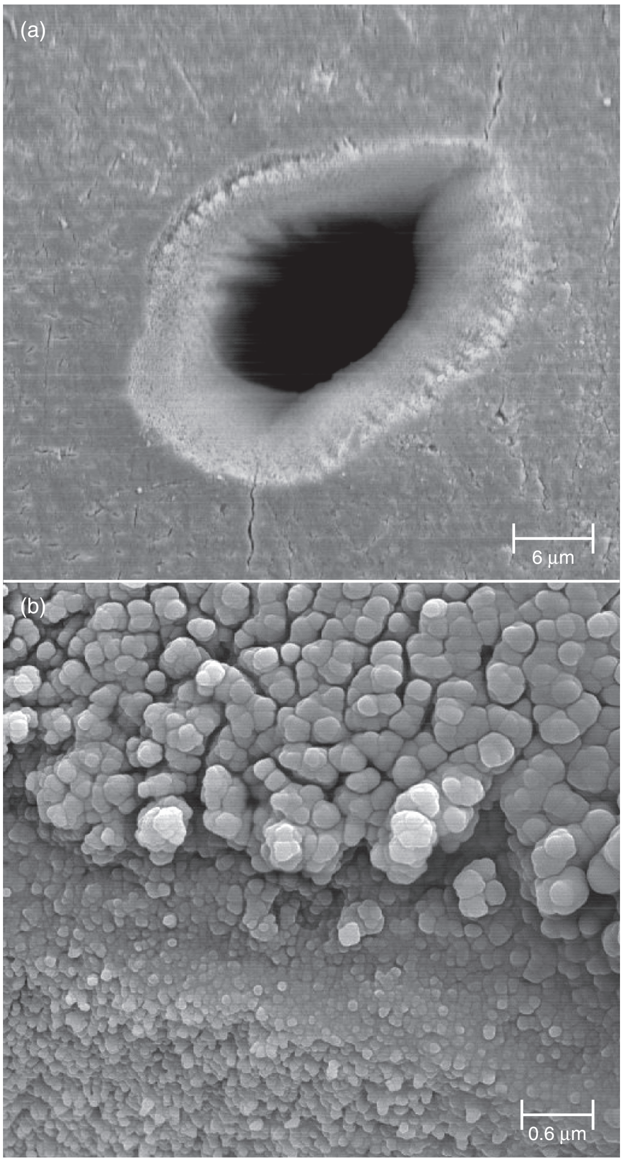

Figure 2 Resulted microcavity from irradiation under average power of 76 mW and fluence of 13 J/cm^2; (a) – 300× and (b) – 3000×, at enamel surface

	Fig. 2	Fig. 3
Average power, mW	76	191
Fluence, J/cm^2	13	32

Table 1 Irradiation parameters using the focused laser beam on the target tissue

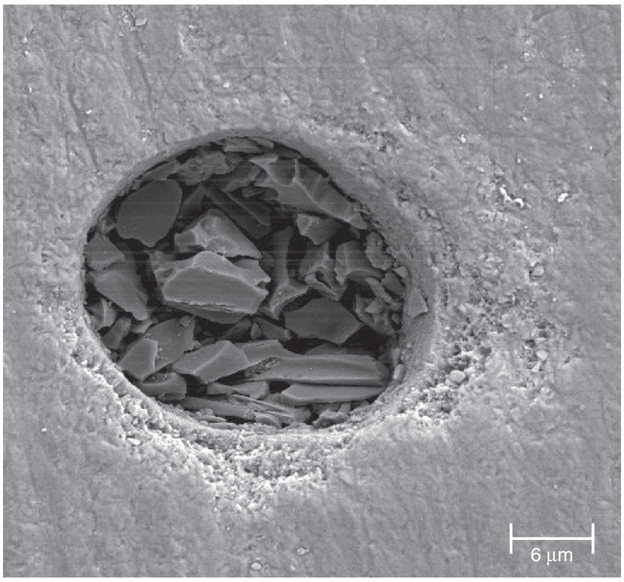

Figure 3 Well-defined edges and original feature into microcavity is observed under an average power of 191 mW (300×), at enamel surface, using focused position

Threshold ablation fluence level to enamel and dentin was measure by Krüger et al. [11] under a femtosecond laser ablation, being 0.3 J/cm^2 for dentin and 0.6 J/cm^2 for enamel. They have shown that enamel and dentin can be ablated successfully avoids thermal damage to neighboring tissue and microcracking.

Fig. 2 shows the aspects of 2 microcavities obtained from the teeth under different average power levels. The conditions used on the ablation procedures are resumed on Table 1 and laser beam focused on the teeth surface, representing all resulted microcavities.

Fig. 2a shows a resulted microcavity when average power was 76 mW and fluence of 13 J/cm^2. Under this parameter, it is possible to observe a conventional ablation pattern with photo thermal effect. A very similar pattern can be observed after longer irradiation pulses [19]. There is a clear separation between the interaction regions in three distinct zones: Z1, the real ablated area; Z2, a modified area; and, Z3 with no apparent modification.

In this case, only part of the energy was enough to cause the ablation process. Being the beam waist (ω_0) of 20 μm, the microcavity showed on Fig. 2a presents this diameter adding Z1 and Z2, where 2/3 of the stored energy was capable of ablating (Z1) a lower diameter (14 μm). However, 1/3 of the entire energy, which corresponds to the Gaussian beam boundary, does not represent an interaction capable of promoting this ablation process, causing thus a "soft ablation" and modifying just the tissue surface (Fig. 2b).

A line between Z1 and Z2 can be observed with more details (3000×) in Fig. 2b. The transition is quite dramatic due to the enamel heterogeneous structure.

The fact that the ablation process may have been smaller than the laser beam shows that there is an energy density threshold above the one where the absorption

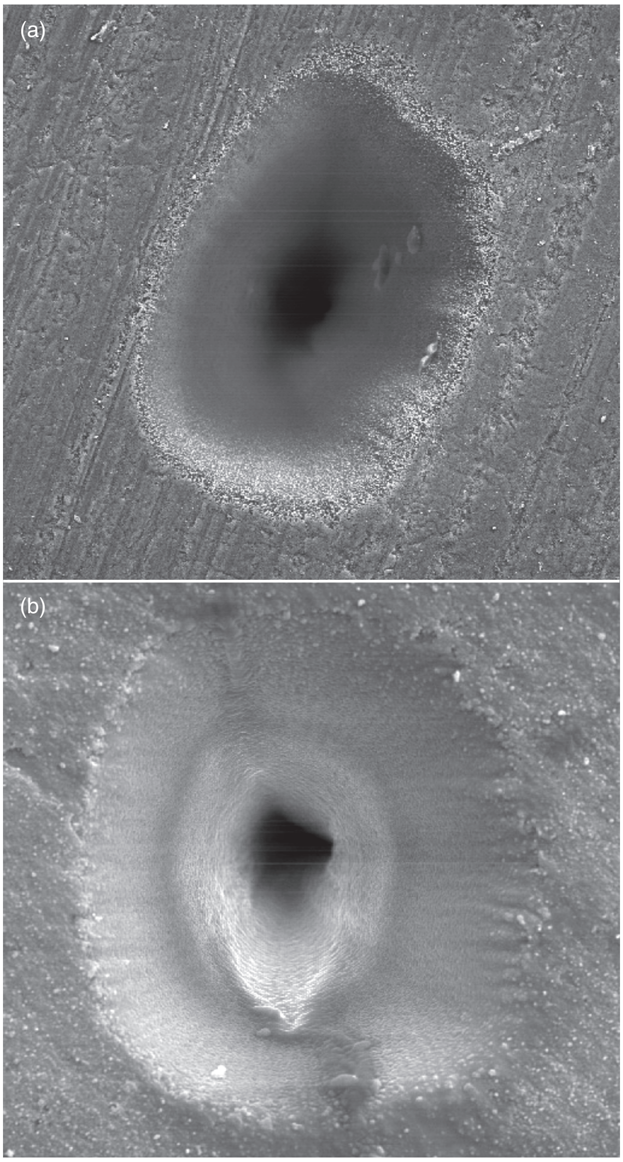

Figure 4 Non-defined edges and original feature into microcavity is observed under an average power of 191 mW (300×), at dentin surface, under focused (a) and defocused mode of + 10.0 mm (b)

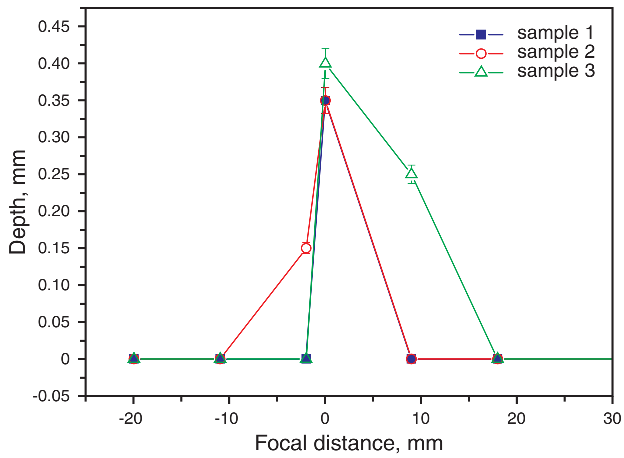

Figure 5 (online color at www.lphys.org) Depth curves of microcavities under different focal distance at enamel surfaces

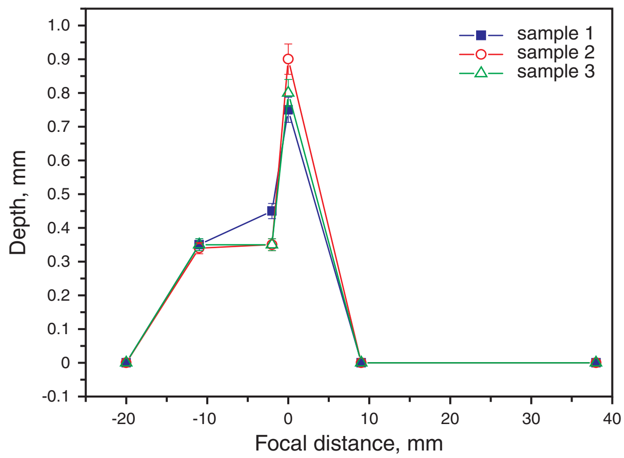

Figure 6 (online color at www.lphys.org) Depth curves of microcavities under different focal distance at dentin surfaces

lies while below this threshold this phenomenon is not observed.

This way the stored energy at that limit region between Z1and Z2 under the laser beam periphery under an average power of 76 mW was preferentially absorbed by the enamel organic portion exposing the prismatic "heads" structure.

On the other hand, Fig. 3 shows a resulted microcavity under an average power of 191 mW and fluence of 32 J/cm^2. The aspects show well-defined edges or rim and inside the cavity (bottom) the presence of original crystal structure of the material.

The cavity results from ablation showed a sharp edge or rim definition preserving the original characteristics of the enamel and a highly prismatic structure. Results obtained by other researchers [11,12,16,19,20] are in accordance to this ablation description.

A competition between absorption and dissipation takes place at this stored level energy parameter. Once the pulse width is of 70 fs and the vibration relaxation time on the crystalline net (photon relaxation time) presented by a dielectric solid, such as the enamel, is of 100 fs [6] there is no time for the energy dissipation phenomenon to Z3 to happen as heat. The entire illuminated area lies above the ablation density.

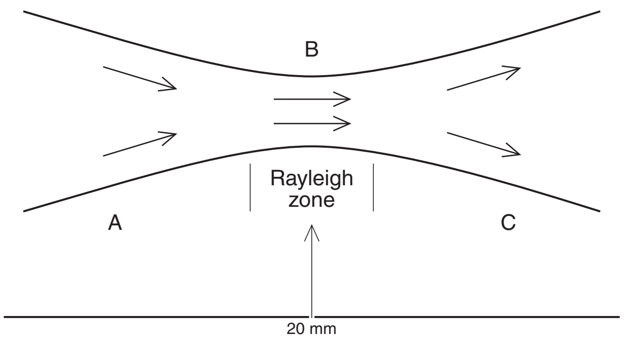

Figure 7 Laser beam geometry: before (A) and after (C) of focal point (B)

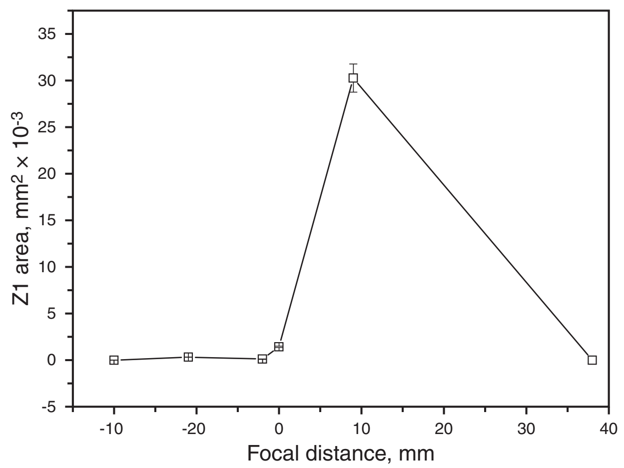

Figure 8 Ablated area (Z1) average values of microcavities under different focal distance at enamel surfaces

Then, the result is a precise ablation with well-defined borders. These features can be observed on the microcavity shown on Fig. 3. No raised rims are observed, which are very common features of laser ablation with long pulses, when thermal effect is present most of the time. The superficial tissue change on the microstructure is not even noticed.

Under the average power of 191 mW a preservation of the dental enamel original features is observed meaning the presence of the highly mineralized prismatic rods removing only the material containing the more organic portion, which is responsible to keep these prisms together. This morphology has a very similar aspect when compared with chemically treated teeth with 37% phosphoric acid by 15 seconds, reported by Silverstone et al. [21], as Type II.

The advantage of USLP ablation in comparison with chemical attack lies in the former calcium ions removal, which are very important to maintain the cariogenic resistance of this tissue into oral cavity [22]. On the other hand USLP ablation should remove phosphoric elements, due

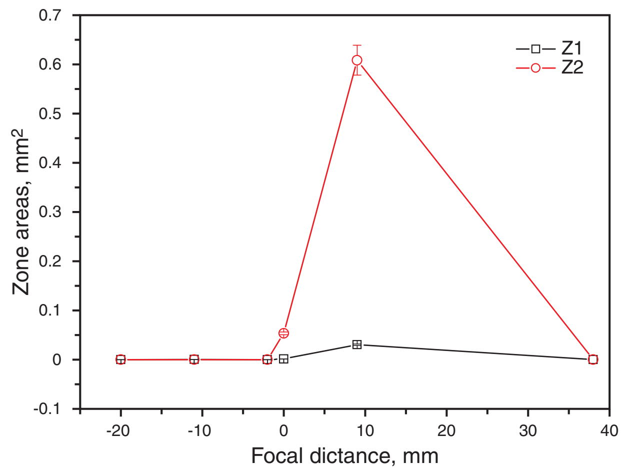

Figure 9 (online color at www.lphys.org) Ablated area (Z1) and non-defined edges (Z2) average values of microcavities under different focal distance at dentin surfaces

to light interaction with water since phosphor atoms are strongly bonded to water molecules.

To enamel tissue, the interesting practical result is that the data obtained in our experiments suggest that controlling fluence delivered to the tissue using a femtosecond laser, should be possible to perform such an irradiation condition that leads to the preservation of the existent prismatic structure, as well building up extremely favorable surface conditions to restoring material adherence.

By the other side, considering dentin tissue, Fig. 4 shows two microcavities resulted from the same average power of 191 mW, at focused (a) and defocused mode of + 10.0 mm (b). Non-defined edges, as melt-flushing aspect, normally present under nanosecond or even under microsecond pulsed laser ablation, are present here. It seems very clear the even under the same parameters, the chemical composition and microstructure of the target tissue promotes different results, in agreement with Krüger et al. [11].

When laser beam was defocused 10.0 mm from the focal distance of 20.0 mm (laser was 30.0 mm distant to the target surface), Z2 is larger than under focused mode. The aspect of melt-flushing at the edges, cited by Russo et al. [14], as commonly to nanosecond laser ablation, is present here.

One important data is the microcavity depth and it was measured in our experiment following the method described afterwards. The first step was to focus the plane to be irradiated. The second one was to focus the bottom of the microcavity. The microcavity depth is measured by reading the microscope lens dislocation from the first to the second focus position.

The diameter and depth measures were analyzed through an optical microscope (40× magnification).

Figs. 5, 6, 8, and 9 show the resultant depth and area of the microcavity's top for three samples irradiated at several positions relative to the laser beam waist, located in the figure at 0 mm (focal position).

While Figs. 5 and 6 show depth curves of microcavities under different positions from focal point to enamel and dentin surfaces, respectively, Fig. 8 and 9 show areas of the entrance of microcavities (top). These curves give us very clear information about ablation progress process which is very close and dependent to the beam geometry (Fig. 7).

At focal position, we have the highest concentration of photons and they are traveling as parallel between them, resulting in a Rayleigh zone (Fig. 7). Before this focal point, photons are following a convergent direction, and, after this focal point, they are changed and following a divergent direction.

It means, that fluence is increasing before and up to focal point and it is decreasing from focal point to the C zone (Fig. 7).

Besides, considering threshold ablation to enamel and dentin [24], Figs. 5 and 6 agree with Krüger et al. [11] because dentin and enamel show the highest depths of 0.9 mm and 0.4 mm, respectively, it means that dentin has a chemical composition and very porous structure and it is reasons to become easy ablation under femtosecond pulsed laser. Indeed, this facilitation allows a rapidly plasma formation over target- zone, resulting in a more dense plasma into the same time under the same fluence, in comparison with enamel, resulting in a more intense plasma shielding.

This more dense plasma shielding to dentin than enamel promotes a higher reflection of incident laser beam. It explains why depth to enamel is higher than to dentin. Enamel femtosecond ablation results in less dense plasma shielding in comparison to dentin, then depth is higher to enamel. However, the area of the entrance to microcavity is smaller to enamel in comparison to dentin (Figs. 8 and 9), and once deposited energy is more used to ablation of enamel in depth, and more used to modification, through reflectivity and heat diffusion, of expanded area at the top of microcavity to dentin.

Ablation of human healthy human enamel and dentin could be explained by geometry beam and chemical and microstructure composition. When laser is deposited at the pre-focus position up to focal position, the fluence is converging and increasing, it allows a preferential penetration in enamel and dentin, it can observed at Figs. 5 and 6, even Figs. 8 and 9 shows that area is very restricted close to beam waist (ω_0) up to focal position, resulting in a very precise drilling. However, after the focal position, it is observed depth decreases and there is a expansion in area to both tissues, but dentin a more evident Z2 area with non-defined edges, confirm again its smaller threshold ablation.

All our findings show a possibility to select and control the enamel ablation using femtosecond pulsed laser, similar to the Er:YAG laser irradiation effects, which preferentially removes composite resin when compared with the enamel [23].

However, considering the ultra-short pulse system, the selectivity can be more precise because the enamel rods, which are the microstructure of this biological tissue, can be exposed. Not only as Krüger et al. [11] have mentioned, concerning depth of penetration, but even controlling region around microcavity, without cracks, micromachining a new aspect very convenient to receive a adhesive material diffusion, for example, when it is necessary a selective removal of less mineral portions.

Considering that an all-solid-state tunable Ti:Sapphire continuous wave laser was developed objecting new sources for spectroscopic measurements of various biophotonic information [10] and for micromachining of defects and refractive index change [24]. This study is very close to others [11,24,25] and contributes with a new method for restorative and aesthetic dentistry.

4. Conclusion

Formation of multiphotonic avalanche process which allows, through ultra-short pulsed laser, ablate a commonly nonabsorbing biologic tissue or target-material with the highest precision, is highly intensity-dependent [24].

In conclusion, it is possible to choose hard or soft ablation, under lower and higher average fluence, respectively, focused and defocused, revealing different aspects of dental enamel and dentin, depending on the average power and distance from the focal point of the ultra-short pulse laser on the tooth surface.

We demonstrated the possibility of selective control of refractive index change and application in the preferential removal of portions of dental hard tissues. This topic can open a new option in Ultra-Conservative Operative Dentistry procedures and into biomaterial science fields.

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