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## 2 **Di-Silicate Dental Ceramic Surface Preparation by** 3 **1070 nm Fiber Laser: Thermal and Ultrastructural** 4 **Analysis**

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### 12 **Abstract:**

#### 13 **Background**

14 Lithium di-silicate dental ceramics bonding, realized by using different resins, is strictly dependent  
15 on micro-mechanical retention and chemical adhesion. The aim of this *in vitro* study was to  
16 investigate the capability of a 1070 nm fiber laser for their surface treatment.

#### 17 **Methods**

18 Samples were irradiated by a pulsed fiber laser at 1070 nm with different parameters (Peak Power  
19 from 5 kW to 5 kW, RR 20 kHz, speed from 10 to 50 mm/s, total Energy Density from 1.3 to 27  
20 kW/cm<sup>2</sup>) and the thermal elevation during the experiment was recorded by a Fiber Bragg Grating  
21 (FBG) temperature sensor. Subsequently, the surface modifications were analysed by optical  
22 microscope, Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDS).

#### 23 **Results**

24 With a Peak Power of 5 kW, RR of 20 kHz and speed of 50 mm/s, the microscopic observation of the  
25 irradiated surface showed increased roughness with small areas of melting and carbonization. EDS  
26 analysis revealed that, with these parameters, there are no evident differences between laser-  
27 processed samples and controls. Thermal elevation during laser irradiation ranged between 5 °C  
28 and 9 °C.

#### 29 **Conclusions**

30 1070 nm fiber laser can be considered as a good device to increase the adhesion of Lithium di-silicate  
31 ceramics.

32 **Keywords:** di-silicate ceramics; fiber lasers; Fiber Bragg Grating; Energy Dispersive X-ray  
33 Spectroscopy

### 34 **1. Introduction**

35 The demand of ceramic prosthetic restorations has increasingly become common in daily dentistry.  
36 Moreover, the continuous need for increased precision level, particularly in cosmetic dentistry.  
37 where new materials, such as feldspathic ceramics, play an important role in prosthetic  
38 rehabilitations, is considered crucially important. Unfortunately, failure resulting from porcelain  
39 fracture has been reported as ranging from 2.3% to 8%. Nevertheless, it seems to be a function of a  
40 multi-factorial reason [1-3], with the key cause attributed to the composite resin adhesion with  
41 porcelain. Therefore, it is necessary to condition the ceramic surface which is considered very  
42 interesting [4,5].

44 The inside surface of the ceramic prosthetics must be conditioned for optimized micro-mechanical  
45 retention by the resin penetration into the ceramic micro-roughness; this treatment enhances the  
46 mechanical retention of cement by enlarging the surface in contact with the tooth structure through  
47 the creation of micro-porosities. [6,7].  
48 For producing surface roughness and for promoting micro-mechanical retention, different treatment  
49 methods such as diamond roughening, air-particle abrasion with aluminium oxide and acids etching  
50 have been proposed in the literature [6,7]. All these techniques have been investigated under *in vitro*  
51 conditions [8-10].  
52 The use of laser technology for surface treatment has already been successfully applied in many  
53 industrial applications by the utilization of high power sources. Today, this technology represents a  
54 controllable and flexible technique for the modification of surface properties for different various  
55 materials [11,12], since laser parameters have the capability to influence and alter the surface  
56 microstructure [13].  
57 The *in vitro* study here reported has the aim to verify the possibility of performing the surface  
58 treatment of Lithium di-silicate ceramic specimens by the irradiation of a 1070 nm pulsed fiber laser.

## 59 **2. Materials and Methods**

60 The circular faces of twelve cylinders of Lithium di-silicate ceramics (e.max Press, Ivoclar, Italy) with  
61 10 mm diameter and 8 mm length were processed into three 3 x 3 mm square zones by using a 1070  
62 nm pulsed fiber laser (AREX 20) provided by Datalogic, Italy. This source has a maximum average  
63 output power of 20 W and a fixed pulse duration of 100 ns, thus providing a maximum peak power  
64 of 10 kW for a repetition rate of 20 kHz. Each square zone on the sample faces has been processed  
65 with different laser parameters. Particularly, the output power and speed have been varied from  
66 100% to 30% and 50 to 5 mm/s.

67 After a preliminary pilot study using different parameters, it was decided to conduct all the tests at  
68 RR of 20kHz.

69 The lens used with the AREX 20 laser has a focal length of 160 mm. In this configuration, the laser  
70 beam has a spot-size of 80  $\mu$ m. Each square zone on the sample surface has been processed using a  
71 meshed filling pattern with a distance between lines of 0.03 mm.

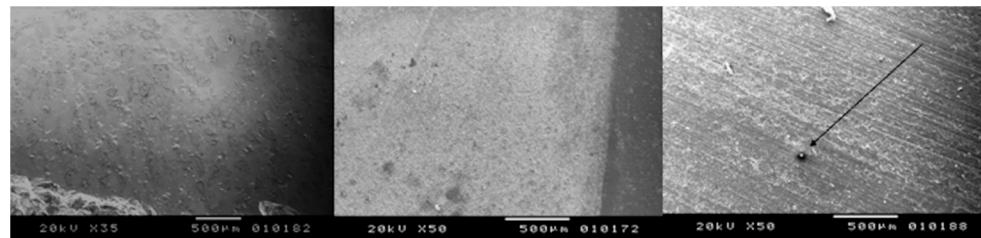
72 The laser beam focalization was checked by a metal cylinder of the same dimension of the samples.  
73 The Power per unit area deposited on the material ranged between 1.3 and 27 kW/cm<sup>2</sup>.  
74 The specimens were subsequently observed by an optical microscope (Olympus MTV-3, Japan), then  
75 metallized and analysed by a SEM (Ion sputter Jeol JFC 1100E, USA) and an EDS system (JSM-35CF,  
76 Jeol Ltd., Japan).

77 During the irradiation of the sample with the best laser parameters, the thermal elevation was  
78 recorded by a FBG-based temperature sensor connected to an interrogator. The fiber sensor was  
79 positioned into the groove in the middle of the sample. Dynamic Optical Sensing Interrogator sm130-  
80 500 (Micron Optics Inc, Atlanta, USA) was used to measure the FBG wavelength shift induced by the  
81 temperature increase). This device is also considered as a compact, industrial grade, dynamic optical  
82 sensor interrogation module, field proven for robust, reliable, and long term operation. The software  
83 included with the sensing interrogator system provides a single suite of tools for data acquisition,  
84 computation, and analysis of optical sensor networks. A 25 mm-long FBG with centre wavelength of  
85 1550 nm, reflectivity of 96% and acrylate coating, imprinted in a standard SMF (AOS GmbH,  
86 Germany), has been connected to the interrogator for performing the temperature change  
87 measurement. A temperature-induced wavelength shift of about 13 pm/°C has been considered for  
88 the FBG at 1550 nm.

## 89 **3. Results**

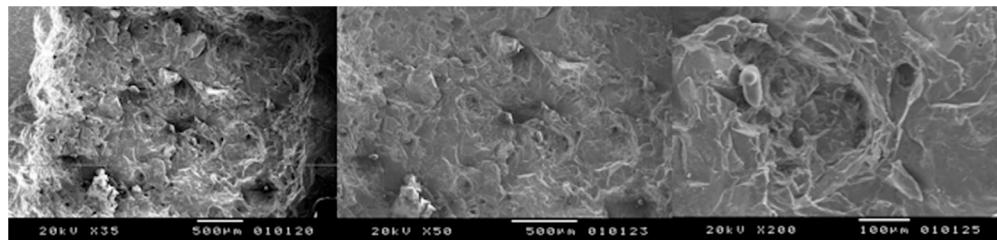
### 90 *3.1. SEM observation*

91 By comparing at higher magnification, the control group (non-irradiated samples) to the cylinders  
92 processed by the fiber laser, greater differences can be noticed (Fig. 1).

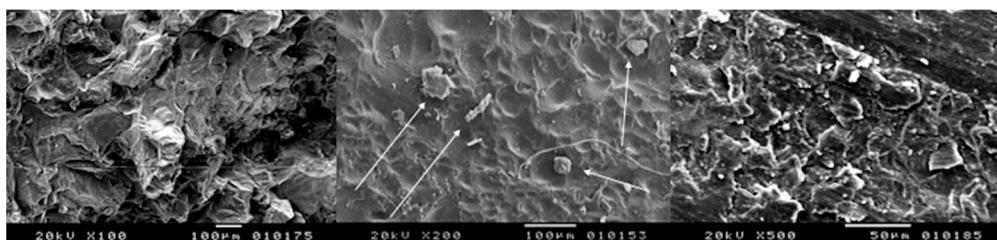
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**Figure 1.** (Left): Non-irradiated sample. (center): peak power of 7.5 kW and 50 mm/sec speed. (right): peak power of 7.5 kW and 10 mm/sec speed with a carbonization spot. (left: X35; center and right: X50)

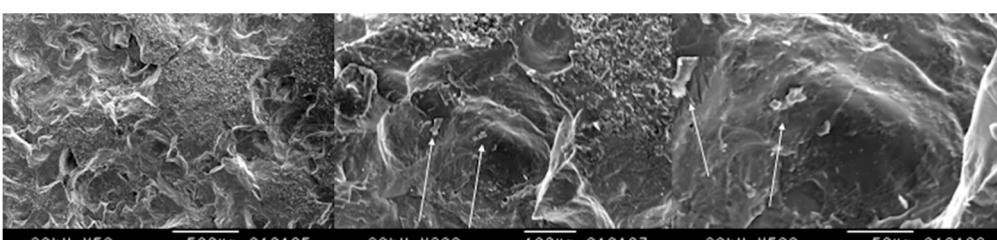
97 In fact, all the treated surfaces show a rough surface with many holes and irregularities. It is evident  
98 that the samples irradiated at different lasing parameters experienced some areas of melting and  
99 burning when the highest energy level was used, due to the cumulative effect of the laser energy. The  
100 presence of some cracks with variable intensities are also found, due to the thermal effects of laser  
101 irradiation (Figs. 2-3-4-5).

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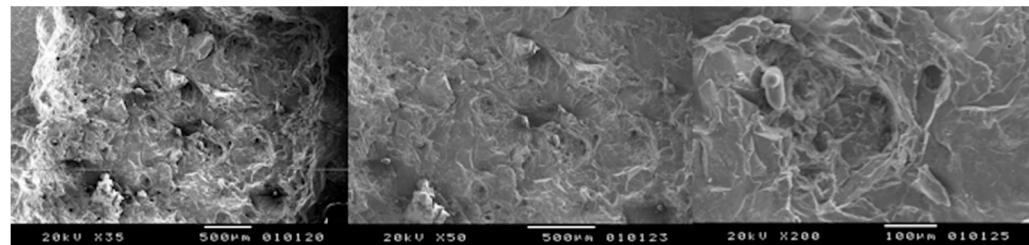
**Figure 2.** Peak power of 10 kW, speed of 10 mm/s: many zones with melting and carbonization are shown. (left: X35, center: X200, right: X500)

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**Figure 3.** Peak power of 10 kW, speed of 50 mm/s: some points with melting are shown. (left: X100, center: X200, right: X500)

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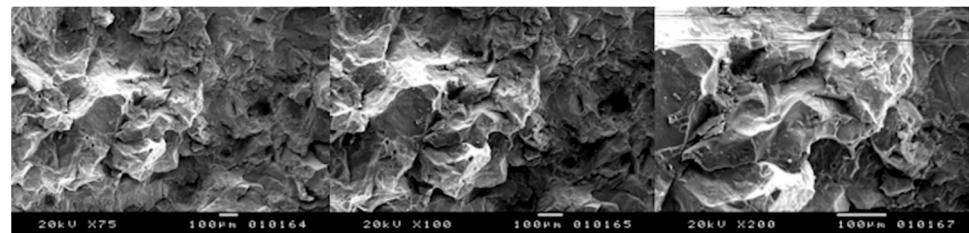
**Figure 4.** Peak power of 7.5 kW, speed of 50 mm/s: presence of melting and carbonization in some areas of the sample. (left: X35, center: X50, right: X200)



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112 **Figure 5.** Peak power of 5 kW, speed of 10 mm/s: evidence of some zones with melting (left: X50,  
113 centre: X100, right: X500).

114 The laser parameters which seem to be the most effective for surface conditioning of the  
115 materials without causing any damages are peak power of 5 kW, repetition rate of 20 kHz  
116 and speed of 50 mm/sec. In fact, the samples irradiated with these parameters revealed a  
117 rough surface with holes, irregularities, cavities and recesses, while the presence of thermal  
118 damaging effects, such as melting, burning and cracks, were not evident (Fig.6).



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120 **Figure 6.** Peak power of 5 kW, speed of 50 mm/s: no evidence of carbonization and melting zones.  
121 (left: X75, center: X100, right: X200)

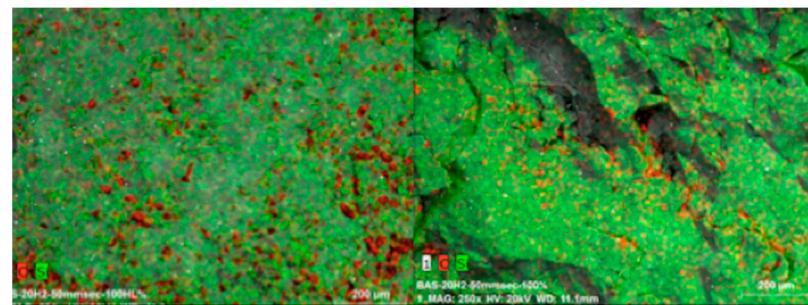
### 122 3.2. EDS analysis

123 The EDS analysis consists of the percentage recording of chemical elements in the point where the  
124 probe is placed. Analyzed samples showed, in general, slight differences in the chemical composition  
125 between control groups and irradiated samples, even smaller variations by changed lasing  
126 parameters were detected thus confirming the information given by the SEM observation.

127 The differences of elemental composition between the non-irradiated areas in the different samples  
128 may be explained by the structure of the ceramic which is not homogeneous, thus resulting in  
129 structural variations of the tested zones. (Fig.1, Left)

130 The samples treated with laser operating at peak power of 10 kW, repetition rate of 20 kHz and speed  
131 of 50 mm/s experienced some zones (red spots) of lower percentage of C when compared to the  
132 control group. On the other hand, O and Al elements were slightly higher in the affected zones (Fig.  
133 7).

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Concentration (%) of some elements before and after laser irradiation (10 kW, 50 mm/sec)

	C	O	Si	K	Al	Na
<b>non-irradiated</b>	16.70	41.40	25.70	6.10	4.30	3.40
<b>irradiated</b>	7.40	44.40	23.70	8.00	6.70	5.50

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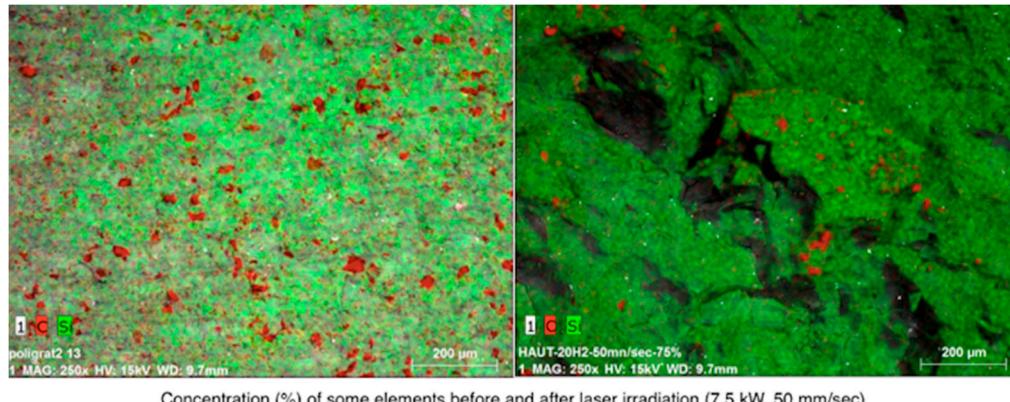
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**Figure 7.** (Left) Control group and (right) samples irradiated with peak power of 10 kW and speed of 50 mm/s: in red the C concentration.

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The samples irradiated with peak power of 7.5 kW, repetition rate of 20 kHz and speed of 50 mm/s showed that only the Carbon concentration was higher in the control group (13.6%), while all the other elements, such as O, Si, K, Al and Na, presented higher concentration values on the treated surfaces (Fig. 8).



Concentration (%) of some elements before and after laser irradiation (7.5 kW, 50 mm/sec)

	C	O	Si	K	Al	Na
non-irradiated	13.60	41.20	27.10	7.40	4.50	3.40
irradiated	3.80	44.40	27.10	8.90	5.90	4.70

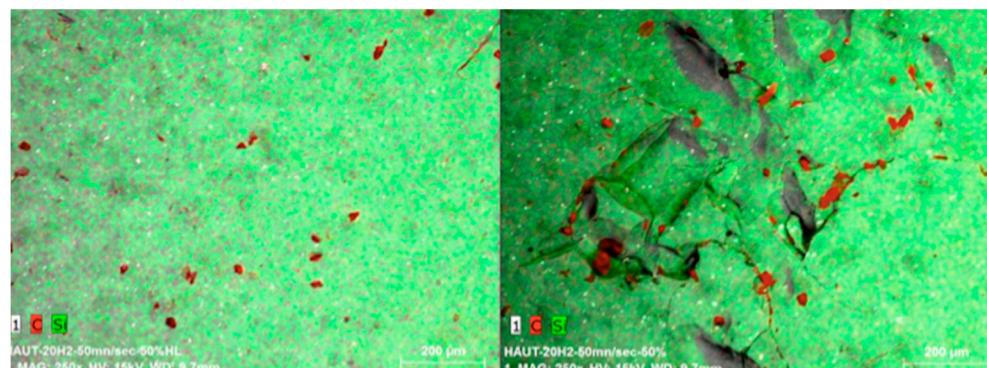
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**Figure 8.** (Left) Control group and (right) samples irradiated with peak power of 7.5 kW and speed of 50 mm/s: in red the C concentration.

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Carbon is one of chemical ceramic composition of lithium di-silicate. The presence of carbon on ceramic surface is due to the high energy of laser irradiation that leads to the burning and melting of ceramic surface.

SEM observations of the samples irradiated with the parameters such as peak power of 5 kW, repetition rate of 20 kHz and speed of 50 mm/s demonstrated the best results. The analysis, in this case, was conducted in four different zones. Results showed slight differences for all the elements concentration in each analyzed zone. These data, confirmed also by SEM observation, demonstrated a poor modification of the ceramic chemical structure caused by laser operating with the optimum parameters (Fig. 9).



Concentration (%) of some elements before and after laser irradiation (5 kW, 50 mm/sec)

	C	O	Si	K	Al	Na
non-irradiated	5.70	43.10	27.50	9.50	6.10	4.40
irradiated	4.30	43.50	28.00	9.50	6.40	4.50

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**Figure 9.** (Left) Control group and (right) samples irradiated with peak power of 5 kW and speed of 50 mm/s: in red the C concentration.

## 161 3.3. Thermal analysis

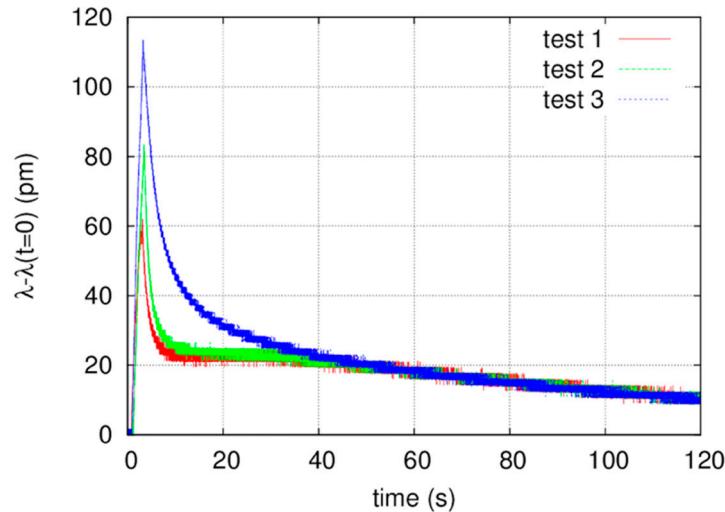
162 The temperature increase during the laser irradiation has been measured only when the source  
 163 operates with the best parameters as per the observation of SEM and EDS analysis. The aim of this  
 164 measurement was to provide the maximum value of the temperature rise, induced by laser, that the  
 165 di-silicate ceramic material can withstand, without being damaged. Higher energy laser treatments  
 166 provide more significant temperature change, which is associated with the detrimental surface  
 167 modifications as shown by SEM and EDS analysis.

168 Thermal elevation of the sample during the irradiation with the laser operating at a peak power of 5  
 169 kW, repetition rate of 20 kHz and speed of 50 mm/s, has been recorded with a FBG connected to an  
 170 interrogator. The FBG wavelength shift obtained in a time interval of 120 s, during the laser  
 171 processing, is reported in Fig. 10. The temperature measurement has been repeated three times, by  
 172 processing three square regions on the sample surface. The fiber sensor was placed in the centre of  
 173 the sample, approximately at the same distance from all the areas irradiated by the laser. Notice that  
 174 the wavelength shift measured by the interrogator is between 65 pm and 115 pm, respectively, in the  
 175 first and the third test. Consequently, the temperature rise due to the laser processing is between 5°C  
 176 and 9°C. The slight growth of the temperature value measured in the second and the third test can  
 177 be due to the gradual heating of the sample, originating from the previous laser processing.  
 178 Moreover, slight differences in the distance of the three zones irradiated by the laser with respect to  
 179 the sensitive part of the fiber sensor must be taken into consideration.

180 The measure of temperature rise during laser irradiation may throw some light on the explanation  
 181 behind the crack formations, after laser irradiations which could be explained through the high  
 182 thermal effects of laser processing, along with the consequence of an extreme physical stress in the  
 183 re-hardening ceramic surface.

184 It must also be underlined that the importance of the very short pulse duration given by the fiber  
 185 laser used in this study (100 ns) which may explain the greater difference between the fluences of  
 186 these tests, compared to those given in the cited works where irradiation had been performed in CW  
 187 or in  $\mu$ s.

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191 **Figure 10.** FBG sensor wavelength shift induced by temperature variations during and after the  
 192 laser irradiation with the best parameters (peak power of 5 kW, repetition rate of 20 kHz, speed of  
 193 50 mm/s).

194 **4. Discussion**

195 As contrast to the different surface treatment methodology mentioned in the introduction, this study  
 196 focused on the laser treatment of the ceramics materials. In current study, laser irradiation  
 197 demonstrated the capability to roughen ceramic surfaces which increases the contact area with the

198 tooth structure, by creating micro-porosities, and therefore enhancing the potential for mechanical  
199 retention of the cement.

200 Although different techniques used for ceramic surface conditioning have demonstrated several  
201 major limitations, the utilisation of laser is not free of problems, too. Particularly, some tests  
202 conducted on lithium-di-silicate [14] and CAD-CAM ceramics [15] with CW CO<sub>2</sub> laser at 10.6 μm  
203 confirmed the presence of micro-cracks and melting textures, due to the thermal effect of the laser  
204 irradiation at output powers higher than 10 W CW (3184.7 W/cm<sup>2</sup>). Moreover, the observation of the  
205 ceramics structure irradiated by a 10 W (14185 W /cm<sup>2</sup>) pulsed Nd:YAP laser at 1340 nm exhibited  
206 the presence of holes, micro-cracks and melted grains [14,15]. This is probably caused by the effect of  
207 high quantity of radiation energy given in a well-defined portion of the ceramic surface over a short  
208 period, thus leading to a very high energy density accumulation. Micro-cracks formation on ceramics  
209 after CO<sub>2</sub> and Nd:YAP laser irradiations may be related to the high thermal effects of laser processing  
210 which leads to an extreme physical stress in the re-hardening ceramic surface [16,17]. Also Er:YAG  
211 laser was used for surface treatment of feldspathic porcelain, however its effect resulted in  
212 significantly weaker surface than that of the HF treated surface. The probable assumption is that the  
213 laser energy from an Er:YAG laser is not well absorbed in porcelain and, therefore, not sufficient to  
214 create a micro-mechanical retention pattern for more favourable bonding [18]. In agreement with this  
215 study, some authors affirmed that, even at a very high energy (500 mJ), Er:YAG laser is not able to  
216 cause on the porcelain surface a roughness sufficient to promote reliable adhesion to the resin  
217 composite [19]. Recently, the so-called “ultra-short pulses” lighted up a greater interest in the field of  
218 mean roughness value [20]. However, due to the higher expense associated with this laser source, to  
219 date, it is still utilized only in few laboratories.

220 Fiber lasers act as sources whereas, the active medium is an optical fiber with core doped with active  
221 ions, such as Nd (Neodymium), Yb (Ytterbium), Er (Erbium), Tm (Thulium) [21]. Fiber lasers differ  
222 from traditional solid-state lasers mainly by the form of the gain medium: in fact, bulk crystal lasers  
223 are typically based on conventional rod or slab geometries while in the case of fiber lasers, active ions  
224 are added into the core of an optical fiber, often with a length of many metres [22]. These lasers  
225 operating in continuous wave (CW) or pulsed mode and emit in a wide range of wavelengths, which  
226 is a function of the dopants and host materials. CW output powers of several kW [23] and pulse  
227 energies up to around 30 mJ [24,25] can be currently obtained with Yb-doped fiber lasers.

228 The most common applications of fiber lasers regard the industrial field, where they are used mainly  
229 for material processing (i.e., for cutting and marking). The main utilizations of fiber lasers in medicine  
230 are related to the lithotripsy [26], the surgical treatment of vascular lesions [27], the non-surgical skin  
231 aesthetic procedures [28, 29] and the eye surgery [30].

232 Recently also its use in the dental field started to be considered, particularly in the soft oral tissues  
233 surgery where it demonstrated to get some advantages consisting on the scanty overheating of the  
234 target, and consequently scanty tissue damages, probably also due to the shorter pulse duration  
235 (ns), compared to the emission normally used in dentistry (μs) [31].

236 This is also the reason of the great differences in the Power Densities utilised in this study (1.3 /27  
237 kW/cm<sup>2</sup>), compared to those used in the similar cited works [14,15] performed with different  
238 wavelengths.

239 The data here reported, according to Gamal et Al [32] confirmed that ceramic laser-irradiated surfaces  
240 show higher roughness values, when compared to non-irradiated surfaces, liable to enhance  
241 mechanical retention due to the extreme physical stress originating in the re-hardening ceramic  
242 surface by the characteristic photo-ionization.

## 243 5. Conclusions

244 This *in vitro* study demonstrated that the utilization of 1070 nm pulsed fiber lasers for the Lithium di-  
245 silicate ceramics surface conditioning is effective and damage-free. In fact, the results obtained using  
246 the proper laser parameters (peak power of 5 kW, repetition rate of 20 kHz and speed of 50 mm/s)  
247 show that it is possible to create an important ceramic rough surface, ready to incorporate in its  
248 cavities through the bonding agent. Moreover, thermal elevation recorded during irradiation was

249 found to be very low, thus explaining the few damages evidenced and, overall, the poor  
250 modifications in the ceramic structure, as shown by the EDS analysis.

251 The use of a pulsed fiber laser at 1070 nm represents a new approach in dentistry, especially in the  
252 field of prosthetics, opening new perspectives, which shall be confirmed by further *ex vivo* studies.  
253 Further analysis will have to be done for studying the mechanical properties of irradiated ceramic  
254 surface (micro-hardness, roughness) and the adhesion characteristics after ceramic sealing  
255 (wettability, shear bond strength and micro-leakage), to confirm the capacity of improving the  
256 adhesion of laser processed di-silicate ceramics to the dental tissues.

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260 the experiments; J.P. R. and N.B. analyzed the data; A.G. contributed materials; C.F. wrote the paper."

261 **Conflicts of Interest:** The authors declare no conflict of interest.

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