Laser irradiation effects on structural, morphological and mechanical properties of ZirCAD dental ceramic

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In the present study, the effects of laser irradiation on microstructure, morphology and hardness of yttria-stabilized tetragonal zirconia (ZrO_2) commercially known as ZirCAD are examined. Three specimen discs were cut from the ZirCAD block. After polishing two of them were irradiated by Nd: YAG pulsed laser, with 100 and 150 laser pulses respectively and the remaining one unexposed specimen was kept as a standard. The XRD analysis confirmed that no phase change occurred after laser irradiation however an increase in dislocation line density (DLD) was observed. The surface morphology was observed by optical and scanning electron microscopy (SEM). Laser irradiation initially caused the upper surface to melt therefore resulting in a smooth and glazed surface. Further increase in laser pulses produced porosity within the sample. The optical microscopy revealed that the crater size decreased and the heat-affected zone increased with the increased number of laser pulses. Vickers hardness was employed to observe the hardness of unirradiated and irradiated samples and a 32% increase in hardness was observed when the material was irradiated by 100 laser pulses. The glazed surface exhibited more hardness as compared to the unirradiated and porous surfaces. The improved surface morphology and high hardness achieved in this work with laser irradiation for ZirCAD ceramic are quite beneficial in dental applications.

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1. Introduction

The great advancement in the field of dentistry has been seen in the recent past. The clinical use of all-ceramic restorations has grown substantially, owing to the increase in demand for aesthetic restorations [1]. Zirconia is an efficient material and has a prominent position among ceramisc. High mechanical strength and flexural resistance, make it ideal material for crowns, bridges, and implants [2]. It is polymorphic and exists in three different crystalline forms i.e. monoclinic, tetragonal, and cubic, at different temperatures. Yttria stabilized tetragonal zirconia polycrystal, commercially known as ZirCAD, is considered a promising candidate among dental ceramics due to its superior mechanical and optical properties [3].

Despite the superior mechanical properties of zirconia as a restorative material, it faces the clinical challenges of chipping of the veneering smaterial and its inability to bond with the tooth structure by conventional methods. Good cementation ensures the clinical success of indirect restorations which requires roughening of the cementing surface. To ensure optimal bonding with the tooth, there is a range of different surface treatment methods recommended for different ceramics. Hydrofluoric acid etching along with silane coupling agent application is considered as the establised procedure to create a rough surface for good resin-to-glass ceramics bonding. However, for roughening the polycrystalline ceramics e.g. zirconia (not silica-based), the conventional gold-standard protocol is not effective. Therefore techniques like air-particle abrasion with Al_2O_3 or SiO_2 and laser surface treatments are suggested to enhance the adhesion to resin

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cements [4,5]. The surface management techniques with laser processing have been developed significantly over the last two decades. The laser treatment procedure is usually very safe and is much more quick and convenient than the conventional alternatives. These factors have led to its widespread application in treatments of dental materials, etching metals, and bleaching procedures, decreasing tooth sensitivity, etc [6].

Commonly used dental lasers cover a broad range of wavelengths from visible light (Argon-ion laser, 488– 514.5 nm; He–Ne laser, 632.8 nm; diode laser, 635– 980 nm) to the infrared region Nd: YAG, 1064 nm; Er: YAG, 2940 nm; CO_2, 9300, 9600, 10600 nm) [7,8]. According to the current literature, there is no optimum wavelength for all dental applications. Each wavelength has different treatment advantages [9]. Among different lasers, Nd: YAG laser (1064 nm) is being widely used in dentistry for caries removal, the treatment of dentine sensitivity, bleaching of teeth and creating surface roughness to increase the bond strength between resin and ceramic crown. Many studies have investigated the effect of Nd: YAG laser, as a surface treatment method [10,11]. The irradiation of Nd: YAG laser on feldspathic porcelain improved bond strength values as compared to hydrofluoric acid etching [12]. The Nd: YAG laser produced more surface roughness when in comparison to that produced by the Er: YAG laser [13].

The objective of this investigation was to evaluate the structural, morphological changes as well as hardness of ZirCAD after irradiation with Nd: YAG laser.

2. Experimental details

Three specimen discs were cut from IPS e.max ZirCAD block pre-sintered (Ivoclar Vivadent) [Zirconium oxide (ZrO_2) 88.0 – 95.5 % - Yttrium oxide (Y_2O_3) > 4.5 % – ≤ 7.0 % - Aluminium oxide (Al_2O_3) ≤ 1.0% -Other oxides ≤ 1.5 %)] by using 5 axes cutting machine ARUM (Korea). The samples were cut oversized by 25% and sintered to the full density at 1500°C in the air for 2 hours in a software-controlled furnace (VITA). During the sintering, the sample shrank 25%, and the required size of 10 mm (in diameter) and 2mm (in thickness) was obtained. The post-sintered samples were then polished by a silicon polisher and afterward cleaned ultrasonically.

In the second step two samples were irradiated by Nd: YAG pulsed laser (1064 nm, 125 mJ, 6ns, 10Hz) for 100 and 150 laser pulses. The laser beam was targeted on the material at 0° to the normal surface. The focal length of the lens used was 20 cm and the distance between the sample and the lens was kept at 19 cm to avoid the drilling of the samples. The whole experiment was performed in air at standard pressure.

The structural properties were studied using “PanalyticaX'PertPro with scan range 5° - 70°. Optical Microscopy was accomplished with OLYMPUS U-TVO.5xC-3 Japan with LMPI anFLN 5x/0.13 lens. SEM analysis was performed by Jeol JSM 6480 LV (Japan) to study the surface morphology. The Microhardness of the samples was measured by Vickers hardness tester Zwick/Roell ZHV 5030 (Germany) under a test load of 3kg with a dwell time of 5 seconds.

3. Results and discussions

3.1. X-ray diffraction analysis

The XRD pattern of unirradiated and irradiated ZirCAD samples with 100 and 150 laser pulses is shown in Fig.1, showing the tetragonal phase when compared with the standard crystallographic data card PDF#50-1089 of ZrO_2 [14–16]. The absence of peaks of yttrium and other constituents confirmed that they were properly doped [17].

For unexposed and exposed samples, seven peaks were obtained for each sample with reflection planes (011), (002), (012), (112), (013), (121) and (202) with minor deviation in 2θ. It is clear from the results that the X-ray diffraction pattern of laser-irradiated and un-irradiated specimens was identical with one another indicating that laser irradiation had no effect on the internal microstructure of ZirCAD and it was just a surface treatment. The XRD parameters for (011) planes were taken for the microstructural analysis and are given in Table 1.

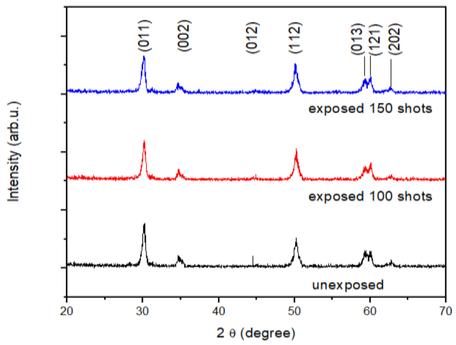

Fig. 1. XRD pattern of un-irradiated and irradiated ZirCAD.

The crystallite size was calculated by using Scherer's formula [18–21] given in equation (1).

$$D = k\lambda/\beta cos\theta \quad (1)$$

where D is the average size of the crystallite, λ is X-ray wavelength (1.5406 Å), θ is the angle in radian where the peak is observed and β is the peak broadening (FWHM). To calculate the quantity of defects after laser exposure of the samples, the dislocation line density (DLD) was also calculated by using equation (2) [18].

$$\delta = 1/D^2 \quad (2)$$

where δ is the dislocation line density and D is the crystallite size.

The residual strain corresponding to the plane (011) of ZirCAD was evaluated by using equation 3 [22],

$$\text{Strain}(\sigma) = d\text{-}d_o/d_o \quad (3)$$

where d_o is the original or unstrained plane spacing, and σ is induced strain

Table 1. XRD parameters of ZirCAD using (011) plane.

Material	d- spacing	FWHM	Crystallite Size	DLD $x10^{15}$	Strain
	Å	Deg.	nm	lines/m^2	No units
Un- Exposed	2.972	0.53397	16.09	3.86	Nil
Exposed 100 pulses	2.971	0.55208	15.56	4.13	$- 3.4\times10^{-4}$
Exposed 150 pulses	2.978	0.55963	15.35	4.24	$+ 2.0\times10^{-3}$

Table 1 shows that the FWHM value which indicates the structural disorder increased consistently with the increased number of laser pulses from 0.53397° for the unirradiated sample and reached a maximum value of 0.55963° for the sample irradiated by 150 laser pulses. The highest disorder in the crystalline structures was due to work hardening. The added energy transported to existing dislocation and endorsed a large number of new dislocations as evident from table 1. For the structural analysis of the laser-irradiated material, the crystallite size of the samples was also considered. The minimum crystallite size of 15.35 nm was observed for the sample exposed with150 laser pulses. The decrease in crystallite size restricted the slipping of planes and improved the mechanical properties of the material.

When the sample was exposed to 100 laser pulses compressive strain was observed. However, by increasing the number of pulses to 150, the tensile strain was noticed as given in table 1. The compressive strain is attributed to an increase in density of the material while tensile strains might be due to the thermal expansion of the material owing to more energy transferred by the increasing number of laser pulses.

3.2. Optical microscopy

Fig. 2 (a,b,c) shows the optical micrograph of unirradiated and irradiated with100 and 150 laser pulses respectively. The presence of small scratches on the micrograph was due to the polishing of ZirCAD. Three zones were observed in each micrograph after laser irradiation. First is the crater, the second is the heat-affected zone (HAZ) and the third is the unaffected area. The maximum heat is produced at the center of the laser spot due to the Gaussian profile of the laser beam due to which crater is formed. Beyond the laser-irradiated spot, the irregular crater diameter and heat-affected zone are due to non-uniform heat conduction at the ZirCAD surface during laser irradiation. The diameter of the crater and the HAZ versus the laser pulses are shown in Fig. 3.

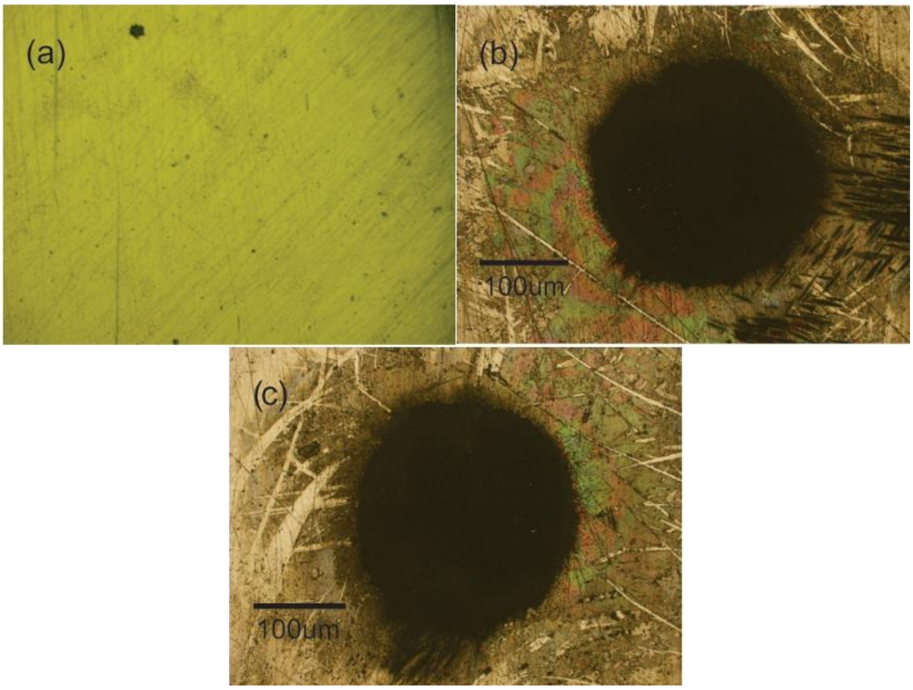

Fig. 2. Optical micrograph of ZirCAD samples. (a) unirradiated (b) irradiated with 100 pulses and (c) irradiated with 150 pulses.

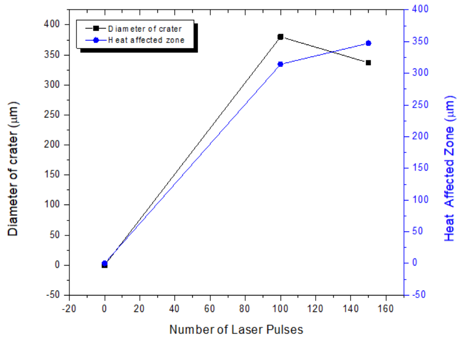

Fig. 3. Heat affected zone and crater diameter as a function of laser pulses.

It can also be observed that the diameter of the crater was 380 µm for 100 laser pulses and then it was decreased to 337µm as the number of laser pulses increased to 150. The formation of the large crater can be attributed to the fact that the laser energy was initially absorbed and transmitted by the upper surface while the decrease in crater diameter might be due to increase in ablation depth per pulse and thus making the material porous as shown in SEM micrograph 4(e,f). The heat-affected zone (HAZ) on the other hand was 314.42 µm when 100 laser pulses were used which increased to 347µm when 150 laser pulses were used. The increase in HAZ is attributed to the more and more heat produced due to more energy deposition with an increasing number of laser pulses.

3.3. SEM analysis

The scanning electron microscope was also used to investigate the surface morphology of unexposed and laser exposed ZirCAD surfaces in detail. Fig. 4(a,b) taken at ×1k and ×5k respectively showed SEM micrographs of unexposed polished ZirCAD surface having some cracks and holes. Fig. 4(c,d) showed the SEM micrographs of the ZirCAD surface irradiated with 100 laser pulses. It is clear from the micrograph that laser has melted the upper surface of ZirCAD and the melted material flew and filled the scratches, reduced the depth as compared to the unexposed sample which was also observed in other studies [16,23]. Fig. 4(e,f) showed the ZirCAD surface irradiated with 150 laser pulses. The micrograph Fig. 4(e) at ×1k showed that the laser interaction produced the porous (foamy) structure at the ZirCAD surface. The pores were formed when more laser pulses were applied which evaporated the ZirCAD surface to some extent [24]. However the formation of pores was more visible in the micrograph Fig. 4(f).

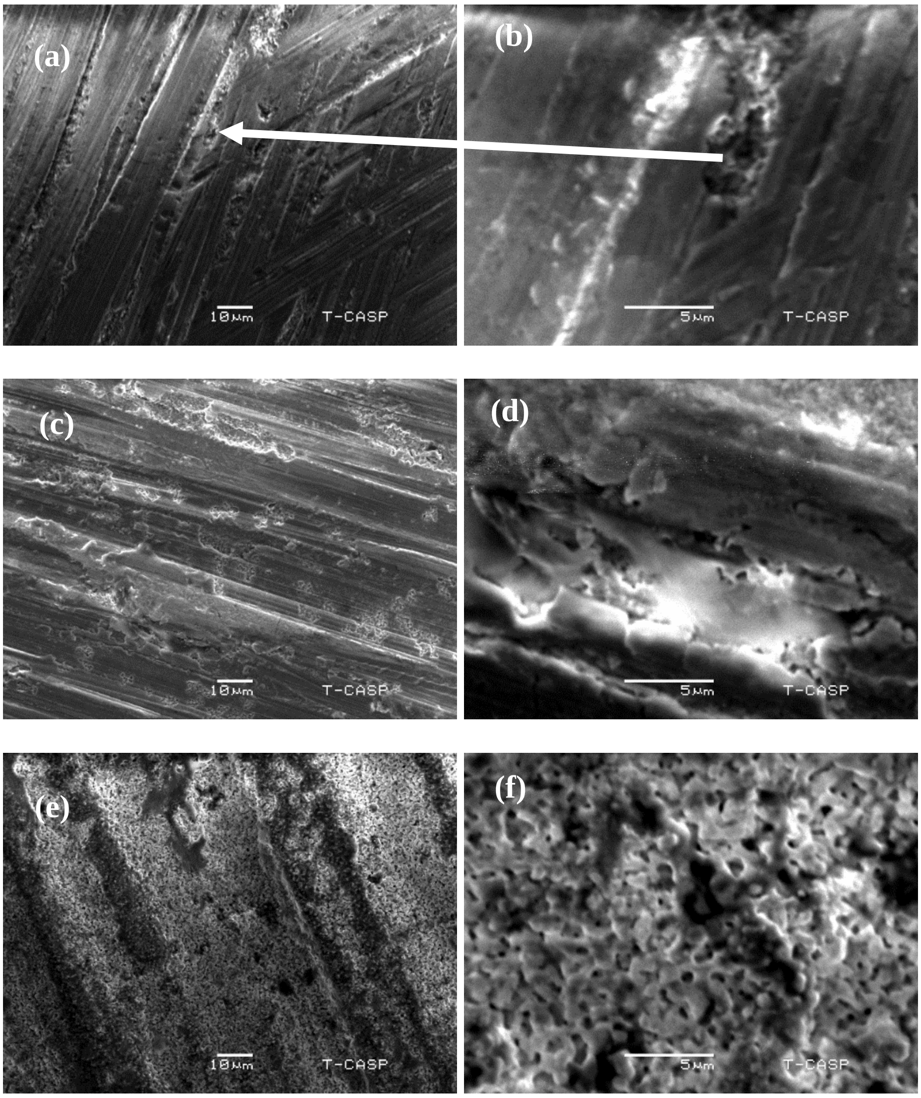

Fig. 4. SEM micrographs of ZirCAD samples (a, b) unirradiated ×1K, ×5k (c, d) irradiated with 100 laser pulses ×1k, ×5k (e, f) irradiated with 150 Laser pulses ×1k, ×5k.

3.4. Hardness

The variation of hardness versus the number of laser pulses is presented in Fig. 5. The figure confirms that the hardness of all laser-irradiated samples is greater than the unirradiated sample. The hardness increases from 10.16 GPa to 13.48 GPa when the sample was exposed with 100 laser pulses which is 32.7 % greater than the unirradiated ZirCAD sample. However the hardness decreased to 11.94 GPa as the number of laser pulses were increased to 150.

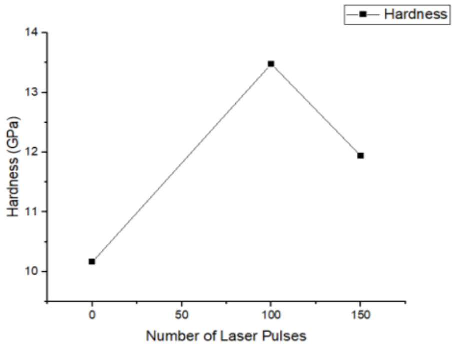

Fig. 5. Hardness of ZirCAD ceramics as a function of laser pulses.

The increase in hardness with the 100 laser pulses can be attributed to the different factors. First is the fast heating and cooling cycles during nanosecond laser irradiation leading to an increase in hardness [25]. Second, the laser-induced thermal process melted the material which filled the scratches and on re-solidification an increase in hardness is observed which was also noticed in other studies [26,27]. Thirdly the dislocation line density which shows crystal disorder in the material also increased at 100 laser pulses (as revealed by XRD data). This disorder in the crystal is also responsible for the increase in hardness values as mentioned by S. Naeem et al [18]. On the other hand, the decrease in hardness by increasing the number of laser pulses to 150 can be attributed to the fact that the more number of laser pulses resulted in more energy deposition onto the material causing more heating along with laser-induced thermal sputtering. Thus making irradiated sample more porous as was evident from SEM micrograph 4(e,f). The production of microscopic pores or cracks at the irradiated sample can decrease the hardness of the material [24]. Although the dislocation line density (DLD) of the samples irradiated with150 laser pulses was higher than the sample irradiated with 100 laser pulses but the formation of micropores overwhelms the increase of DLD. Therefore decreasing the hardness which is also reported previously by using Nd: YAP (10W) laser irradiation [28].

4. Conclusions

The superior mechanical properties and long-term stability make ZirCAD a promising dental material. This study investigated the effect of Nd: YAG laser irradiation on the structural, morphological, and mechanical properties of ZirCAD.

The XRD spectra confirmed that no phase conversion occurred in the sample after laser irradiation since the same planes are observed in all samples while the change in DLD took place after laser irradiation of the ZirCAD surface. The optical microscopy showed that the heat-affected zone (HAZ) increased while the crater size decreased with increased laser pulses. The increase in HAZ was credited to the increased number of laser pulses because the laser energy was initially absorbed and conducted by the upper surface. The reduction in crater diameter was due to an increase in ablation depth per pulse resulting in a porous material. The SEM results showed that the irradiated ZirCAD surface significantly changed and became smooth after irradiation with 100 laser pulses. The hardness data showed the maximum value when 100 laser pulses were applied which was attributed to factors like fast heating and cooling cycles, filling of flaws, and increased DLD. The glazed and smooth surface of laser irradiated ZirCAD will be less plaque retentive and less abrasive to the opposite natural dentition. The improved hardness of laser irradiated ZirCAD is another obvious advantage. With increased laser pulses 150 the material became porous and its hardness was reduced from its maximum value but it was still greater than the unirradiated sample.

The variation in the hardness of the material was attributed to the induced thermal strain for the various number of laser pulses. The rough and porous surface of ZirCAD might be more effective for adhesion with the tooth and also for better micromechanical fitting to veneering material like porcelain. The porosity of zirconia is also helpful for the clinical success of implants at the tissue material interface which determines the implant integration with the bone [29]. Therefore, the Nd: YAG laser treatment can be considered as a better ZirCAD surface treatment process without affecting its internal structure.

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