Digest Journal of Nanomaterials and Biostructures Vol. 16, No. 4, October-December 2021, p. 1519-1525

The influence of Nd: YAG pulsed laser treatment on the structure, morphology and hardness of e.max ceram dental material

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The use of ceramics in dentistry is increasing day by day owing to their more aesthetic look, better mechanical properties, and easy-to-fit approach. The material used in the study is IPS e.max Ceram by Ivoclar Vivadent which has been widely adopted as the ceramic of choice by many dentists as it is ideally suitable for the veneering of more opaque substructures. The study aims to elucidate the result of Nd: YAG pulsed laser irradiation at different numbers of pulses on structure, surface morphology, and hardness of e.max Ceram dental material. XRD shows the amorphous nature of the material before the laser irradiation, whereas the crystallinity in the material is slightly induced after the exposure of e.max Ceram with 150 laser shots. The surface morphology and hardness degradation are attributed to the swelling potential of the material and induced increase of the residual micro-stresses during laser irradiation. This technique could provide more ease for bonding and de-bonding of orthodontic brackets and might be used as a chairside tool.

(Received July 3, 2021; Accepted November 24, 2021)

Keywords: e.max Ceram, Microstructure, Surface morphology, Vickers hardness

1. Introduction

Dental glass-ceramics are extremely eye-catching for dental clinicians and patients due to their combination of excellent physical and chemical properties, such as fabulous esthetics, translucency, low heat conductivity, satisfactory strength and durability, biocompatibility, wear-resistance, and non-allergic nature. Moreover, this family of materials has also attracted great interest from researchers during the last two decades [1]. The idea of non-invasive dental restoration is getting appreciation in current dental practice, and thus all-ceramic veneers are being increasingly used [2].

Tooth veneers can help to achieve the look one desires. They become more significant when to correct tooth shape or to cover up dental stains. These thin custom-made veneer coverings are made to fit the tooth and bonded to the front surfaces of the teeth. The shapes of these ceramic coverings are idealized to improve one's smile [3,4]. These are also a better option for patients who have little room between their teeth.

IPS e.max Ceram by Ivoclar Vivadent is a veneering ceramic designed for use in combination with all-ceramic structures. The standard composition contains SiO_2, Al_2O_3, LiO_2, Na_2O, K_2O, ZnO, CaO, P_2O_5, F, with 60-65 wt % SiO_2 as base veneering material and 8-12 wt % Al_2O_3 as strengthening veneering material. As an improved blend of materials having a low coefficient of thermal expansion (CTE) along with low firing temperature, IPS e.max Ceram can be used as a veneer for all IPS e.max frameworks of different materials e.g. IPS e.max Press and IPS e.max ZirCAD [5]. Silvia Brandt et al have also recommended IPS e.max products by Ivoclar Vivadent after a long clinical study of 66 months or more [6].

Laser has many applications in dentistry e.g. cavity preparation, disinfection of root canal, acceleration of tooth movement, bone transformation, enamel roughness for attachments, de-bonding/removal of ceramic brackets, etc.[7]. Due to the increase in demand for orthodontic

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treatment in adults, orthodontists at times have to bond attachments to ceramic crowns in patients already gone through prosthodontic procedures. Bonding with ceramics is a challenge for the clinician, even though different types of surface preparations techniques are available and each has its own pros and cons. Surface roughness is required for creating micromechanical retention for the adhesive bonding of orthodontic brackets. Traditional methods include sandblasting and acid etching with hydrofluoric (HF) acid etc. With the development of laser systems and their role in dentistry, research has focused on the efficacy of laser etching of ceramics [8].

Use of CO_2, Er: YAG, and Nd: YAG for the surface treatment of zirconia, has been reported [9]. According to Soltaninejad et al, the surface treatment with Nd: YAG laser resulted in surface roughness along with increased shear bond strength [10]. However according to Liu et al Nd: YAG laser did not improve the surface roughness of zirconia [11]. Moser et al used CO_2 laser and concluded that laser treatment of zirconia had significantly increased the shear bond strength (SBS) values [9]. Use of Nd: YAG laser pulses also have been reported to enhance hydroxyapatite properties by modifying the enamel surface [12]. Both fractional CO_2 laser and Q switched Nd: YAG laser treatments showed higher bond strength than unexposed zirconia by another study [9].

However, no such study is conducted so far on e.max Ceram which is a veneering material for many ceramic crowns. So the objective of the study was to evaluate the structure, surface morphology, and hardness of IPS e.max Ceram by Ivoclar Vivadent before and after irradiation with Nd: YAG laser.

2. Material and method

2.1. Preparation of specimen

First, a stainless steel mold was used to fabricate three specimens from e.max Ceram, measuring 10 mm in diameter and 2 mm in thickness. Then, Ceram powder was mixed with the respective solvent to form a slurry, and then the mixed slurry was loaded into the mold as a layer. The specimens were then placed on a firing tray where they were dried and sintered in a vacuum furnace (Artis UGin dantene Furnace) according to the manufacturer's instructions.

2.2. Experimentation

The Sintered samples were then polished with a silicon polisher and ultrasonically cleaned in the second phase. Later, the two specimens were irradiated with 100 and 150 laser pulses from a Neodymium Yttrium Aluminium Garnet (Nd: YAG) pulsed laser (1064 nm, 125 mJ, 6ns, 10Hz), and one sample was kept unexposed as a control. The laser beam was aimed at the material at an angle of 0 degrees to the normal surface. To avoid drilling while focusing in close quarters, the target material (specimen) was kept 19 cm away from a convex lens with a focal length of 20 cm. The entire experiment was carried out at standard atmospheric pressure.

The structural properties were studied by using "PanalyticaX'PertPro emitting $CuK_{(\alpha),(\beta)}$1.5406Å X-Rays at 45 kV with scan range 5° - 70° in 5min 28 sec. Optical Microscopy was accomplished with OLYMPUS U-TVO.5xC-3 Japan with LMPI anFLN 5x/0.13 lens. Jeol JSM 6480 LV (Japan) was used for SEM analysis to investigate the surface morphology. Vickers hardness tester Zwick/Roell ZHV 5030 (Germany) was used to determine the microhardness of the samples under a high test load of 3kgf and a dwell time of 5 seconds. .

3. Results and discussions

3.1. XRD analysis

The diffraction outcome from the interaction of X-rays with a specimen showing a poorly- or non-crystalline nature of the material. The crystalline phases of material present strident diffraction peaks while glassy and poorly-ordered phases due to lack of periodicity, produce broad X-ray scattering profiles [13]. Generally, for amorphous phases XRD patterns do not exhibit sharp peaks, however owing to variations in short-range structural order and chemistry, many amorphous materials display unique scattering contours.

XRD patterns of materials with several amorphous phases are difficult to investigate. So to the identification of a single phase within the mixture is difficult to achieve with diffraction data alone[14]. It is clear from Fig. 1 that there is no peak for the unexposed and the sample exposed by 100 laser pulses, so the material may be regarded as predominately vitreous. However, when the incident energy was increased by increasing the number of laser pulses to 150, two minor peaks appeared in the amorphous matrix, indicating the emergence of a primary crystalline phase that could be interpreted as microstructural change as a function of heat treatment variables like time and temperature. [15,16]. The first sharp peak was observed at 2θ = 31.24° and the second peak at 2θ = 55.82° corresponding to reflection planes (400) and (-333) when compared with the crystalline phase of Al_2O_3 using PDF card #.11-0517 [17].

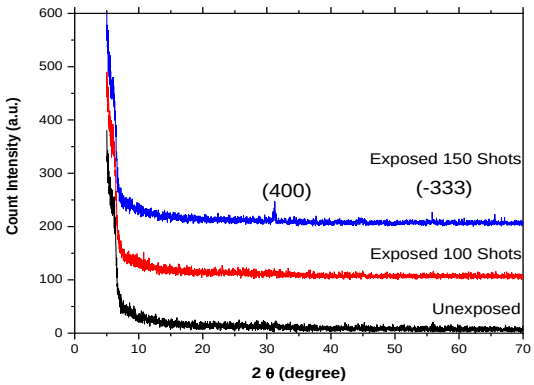

Fig. 1. XRD pattern of un-irradiated and irradiated e.max Ceram.

3.2. SEM analysis

Scanning electron microscopy (SEM) has established its significance as a power full tool in explaining the surfaces before and after irradiation. Topographic changes such as voids, cracks, or holes can easily be detected under the scanning electron microscope. SEM micrographs for unexposed and laser exposed e.max Ceram surfaces are presented in Fig. 2.

Fig. 2 (a, b) shows the surface morphology of an unexposed annealed surface of e.max Ceram using x1k magnification and x5k magnification respectively. It shows that the surface texture is uniform. This represents the structure of the veneer surface prepared for the tooth. Fig. 2(c, d) shows the surface morphology of the sample exposed with 100 laser shots with x1k magnification and x5k magnification respectively. This micrograph displays an image-like molten surface. This condition indicates the uneven surface of the material and the non-uniformity of the surface texture is evident. This non-uniformity of the surface is due to the inconsistent thickness of the material throughout the surface due to the flow of material [18]. Fig 2(e, f) shows the surface morphology of e.max Ceram exposed with 150 laser shots. From the micrograph holes, voids and micro-cracks can be seen, this might be due to thermal stresses induced by the rapid generation of large temperature gradients [19]. This may increase the bond strength of adhesive material sufficient enough to withstand orthodontic and handy forces, similarly use of Nd: YAG may also be used in the process of deboning of the bracket without causing damage to ceramic [6].

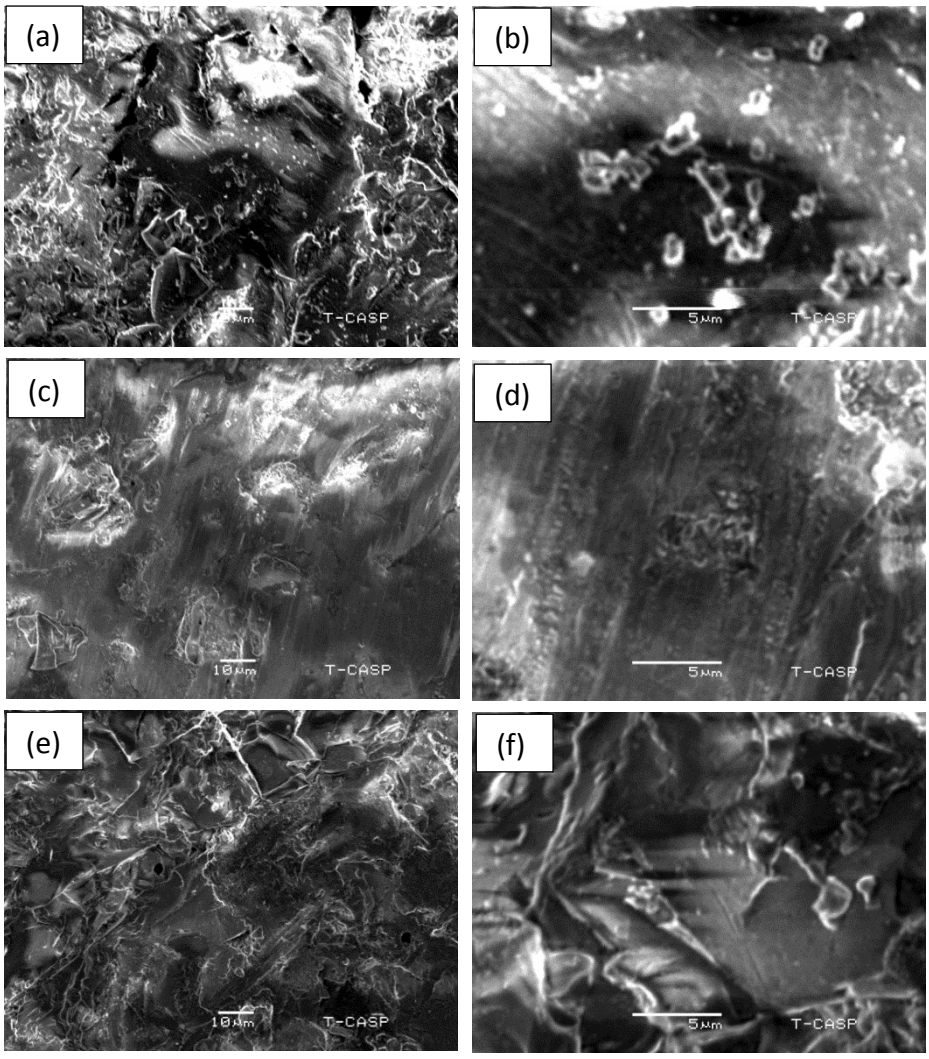

Fig. 2. SEM micrographs of e.max Ceram samples (a) unirradiated (x1k), (b) unirradiated (x5k), (c) irradiated with 100 laser pulses (x1k), (d) irradiated with 100laser pulses (x5k), (e) irradiated with 150 laser pulses (x1k) and (f) irradiated with 150 laser pulses (x5k).

3.3. Optical Microscopy

The optical microscopy was used to examine the unexposed and laser exposed e.max Ceram surface for the different number of laser pulses. The e.max Ceram surface irradiated with 100 and 150 laser pulses are as shown in Figures 3(a) and 3(b) respectively. The presence of small scratches on the unexposed area of the micrograph shown in Fig 3(a) was due to the polishing of e.max Ceram surface. Each micrograph revealed three distinct zones: the crater, the heat-affected zone (HAZ), and the unaffected area. Due to the Gaussian profile of the laser beam, the most heat is created to the centre of the laser spot, resulting in the formation of a crater. Outside the laser-irradiated spot, the irregular crater and heat-affected zone can be seen [20]. In Figure 4, the diameter of the crater and the HAZ are presented as a function of the number of laser pulses. It is observed that the diameter of the crater increased from ~ 253 µm to ~ 322 µm which is plotted in Fig 4. It is observed that the diameter of the crater increased from ~ 253 µm to ~ 322 µm as the number of laser pulses was increased from 100 to 150. The increase in crater diameter was attributed to more energy absorption of laser light and the transportation of heat inside the material through a larger distance as depicted in [19]. During laser-matter interaction the laser energy was initially absorbed and transmitted by the upper surface. The heat-affected zone (HAZ) was ~ 56 µm when the material was irradiated by 100 laser pulses and it decreased to ~ 48 µm when 150 laser pulses were shined on the material. The scattering of the laser beam due to the uneven e.max

Ceram surface at that spot is responsible for the decrease in HAZ. . The burnt material might be seen on the surface in micrographs. For 100 laser pulses, less removed material is seen but as the number of laser pulses was increased the sputter/ablated material was also increased.

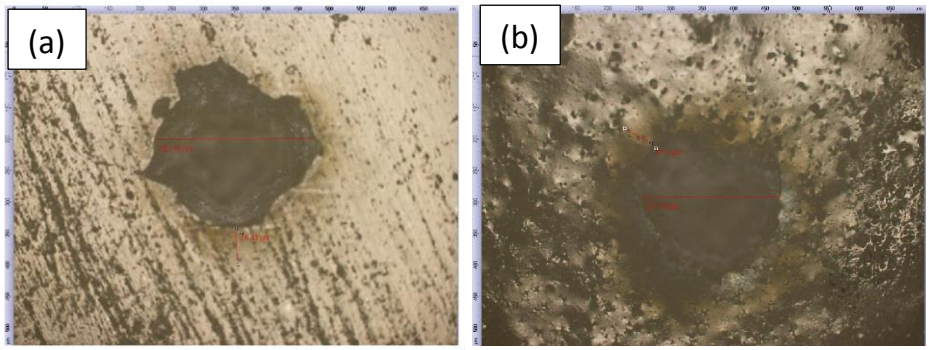

Fig. 3. Optical micrograph of e.max Ceram samples at ×5 (a) irradiated with 100 pulses and (b) irradiated with 150 pulses.

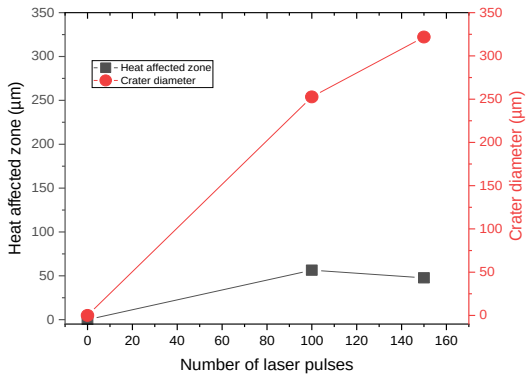

Fig. 4. Heat affected zone and crater diameter as a function of laser pulses.

3.4. Hardness

The variation of hardness as a function of the number of laser pulses is shown in figure 5. The figure shows that the hardness of all the laser-irradiated samples is less than the unirradiated e.max Ceram sample. For each sample, the hardness measurement was carried out ten times, and then the mean value of the hardness number was taken. The value of micro-hardness for the unexposed sample was 5345 MPa which decreased to 5269 MPa when the sample was exposed to 100 laser pulses. The hardness continued to decrease to 5095 MPa as the number of laser pulses was increased to 150. The decrease in hardness as a function of the number of laser pulses is attributed to some factors. First is the fast heating during nanosecond laser irradiation generated a high level of volumetric changes causing the stresses upon the surface resulting in the reduction in hardness. Second, the laser-induced thermal process led to the melting of the material surface, this displacement of the material led to a decrease in the hardness. The decrease in hardness by increasing the number of laser pulses from 100 to 150 laser pulses is attributed to the fact that the more number of laser pulses delivered extra energy to the surface of the specimen which caused thermal sputtering and removed the material from the surface as depicted in Fig 3(b). The ablation of material by using Nd: YAG laser with 150 laser shots might also be the cause of a decrease in hardness and the increase in roughness of the surface [21]. The increase in crystallinity might also be responsible for the decrease in hardness values as indicated in [15]. This might be due to crystallization that occurred on the surface of the material which hindered the sintering process and reduced the sample shrinkage due to which the sample became softened as given in [16]. The maximal decrement of 4.7 % was observed in hardness in e.max Ceram in this work.

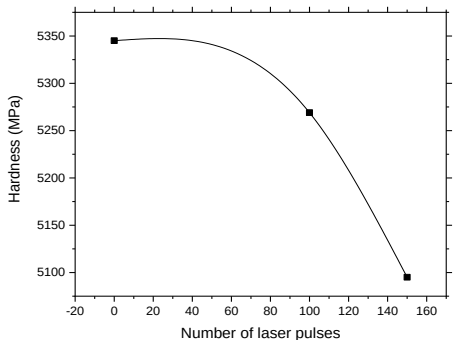

Fig. 5. Hardness of e.max Ceram as a function of the number of laser pulses.

4. Conclusion

The following conclusion can be drawn after Nd: YAG laser irradiation of IPS e.max Ceram.

1- XRD characterization of the exam Ceram before laser irradiation confirmed its glassy nature. Nd: YAG laser irradiation exhibited no adequate modification in its structure when the sample was exposed with 100 laser shots but laser exposure of the material with 150 laser pulses induced the crystallinity in the material but not up to the larger extent.

2-Laser surface treatment changed the surface morphology of Ceram into roughness with a 4.7 % decrease in hardness.

3 -Nd: YAG -laser can be used as a chairside tool for etching of e.max Ceram and de-bonding of brackets from veneers.

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