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Investigation of High Power Nd : YAG Laser Processed Zirconium Silicate Surface

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ABSTRACT

In this, study the photothermal of high power Nd: YAG laser at 1064 nm wavelength for different durations on the surface properties of zirconium silicate (ZrSiO4) ceramics was investigated. Specimens of zirconium silicate (ZrSiO4) ceramic pieces were divided into four samples according to irradiation duration as follows: one control sample (no treatment), and three samples irradiated with Nd: YAG laser at irradiation durations 3, 4 and 5 minutes. The irradiation was applied with fixed output power (60 W) with continuous mode. The samples hardness was measured then SEM, EDX and FTIR characterization was done. The results show that high power Nd: YAG laser provide higher hardness surfaces compared to non-treated surface. SEM images demonstrate the formation of microstructures, smoother surface and solidification process occurring confirming the hardness results. FTIR spectra denotes the presence of quartz with small particle that improves mechanical strength of the zirconium silicate. Furthermore, EDX results reveal that laser irradiation does not change the chemical surface composition of ceramics. A linear correlation between laser irradiation duration and hardness, tensile strength and surface solidification was fond, without causing material defect.

Keywords : Laser Irradiation; Laser-matter interaction; Surface morphology; Surface Treatment; Zirconia ceramics.

I. INTRODUCTION

Lasers have diverse applications in different fields such as medical, industrial, military and other different fields. In the industrial field lasers can used in many processes such as welding, cutting, drilling or surface solidifications. Laser-matter effects can be in diversified manners such as photothermal (represented by vaporization), ablation of matter (based on absorption) or in photochemical (direct breakage of chemical matter bonds).

Zirconia ceramic is a functional material and it has a leading position among ceramic materials. Its special properties, such as high mechanical strength, flexural resistance, make this ceramic material ideal for esthetic crowns, bridges, and frameworks in the anterior and posterior region (El-Ghany and Sherief, 2016).

There have been numerous research studies the effect of laser matter irradiation; for example, on killing bacteria (laser milk pasteurization) (Marouf and Sara, 2018)(Amna and Marouf, 2018), silicon surface modification in solar cells (Marouf et al., 2014), food irradiation (bee honey irradiation) (Al Humira and Marouf, 2017) and production of highly value materials from agricultural waste (Gawbah et al., 2017 and Gawbah et al., 2018). Other numerous researchs studies the interaction of low-level lasers with biological materials such as blood; for examples; investigating the effect of He-Ne laser on human whole blood (Haimid, et al., 2019 a) (Haimid, et al., 2019 b) and it also used to induce emission in human teeth to distinguish between dental caries and sound teeth (Marouf and Khairallah, 2019).

Meanwhile, ceramic materials in structural applications burned by large kilns to enhance its mechanical and surface properties. Also, different ceramic compositions are available for dental use, with varying properties that respond to different clinical indications. Now a days, the Nd:YAG laser has been suggested for employing to dental hard tissues for various usages, such as the effects of laser on surface morphology of dental restorative materials (Sanusi, et al., 2012)(Garcia-Sanz, et al., 2018), surface modification of Ti dental implants by laser (Braga, et al., 2014), laser treatment of dental ceramic/cement layers (Pich, et al., 2015), effects of laser on filling materials (Türkmen, et al., 2006) and effect of laser in hardness of dental ceramic (Ahmed, et al., 2014).

The aim of this study was to investigate the surface morphology and hardness of zirconium silicate following high power Nd: YAG laser irradiation.

II. MATERIALS AND METHODS

2.1 Specimens

The materials used were commercial. Materials preparation went through the following stage: first grinding raw materials (Nile clay, silica sand, weather granite, kaolin, sodium silica, S.T.P.P, master mix) and then sprayed and kept in silos and pressed using (SAMI PH3590 PRESS) and pressed from 160 to 180 Bar. Then dried and it passed the glaze line which consists of (quartz, feldspar, Ball clay, I. Kaolin, Engobe frit-19, matt frit-13, opaque frit-188, Transparent frit-575, Zirconium silicate ($ZrSiO_4$), calcined alumina, transparent printing-106, transparent printing-1000, reactive printing powder-606). Four rectangular specimens A, B, C and D were made with length of 2 cm, width 1 cm and thickness 2 mm. Three of these specimens (B, C and D) were exposed Nd: YAG laser with 60 W output power at continuous mode, with different irradiation times (3, 4 and 5) min and one specimen was left without treatment (A) as reference.

2.2 Laser Irradiation

An Nd: YAG laser system (Dornier Medilas fiber to 5100) operating at a wavelength of 1064 nm with continuous mode was used to irradiate specimen. Specimens were placed one by one and the Nd: YAG laser beam was projected perpendicular to the surface of the specimens. The distance from the laser window to the specimen surface was approximately 7 mm. The laser power was 60 W and laser irradiation treatments were carried out without any water spray (dry laser). All tests and characterizations were done on the exposed area in the samples.

2.3 Hardness and Tensile strength Tests

The hardness of the irradiated and non-irradiated specimens were tested using the Vickers Hardness method (ZHU250, ZWICK/ROELL, GERMANY, 2015). The principle of the Vickers Hardness method is similar to the Brinell method. The Vickers indenter is a 136 degrees square-based diamond pyramid. The impression, produced by the Vickers indenter is clearer than the impression of Brinell indenter;

therefore, this method is more accurate. The load, varying from 1kgf to 120 kgf, is usually applied for 30 seconds. The Vickers number (HV) is calculated by the following formula:

$$HV = 1.854 \times F / D^2$$

Where: F ≡ applied load/kg; D ≡ length of the impression diagonal/ mm

The length of the impression diagonal is measured using a microscope, which is usually an integral part of the Vickers Tester.

Tensile strength was calculated using the following equation:

$$Ts = 3.45 \times HB$$

2.4 Analysis

An analysis of the morphology of the surface layer was made for the irradiated and non-irradiated specimens using electron microscopy (SEM), including a local analysis of the chemical composition (EDX). The surface morphology of the impact sites was examined using a scanning electron microscope (Libusina trida 863/21, Brno, Czech Republic). All impact sites were photographed by a 3.3-megapixel digital camera (Coolpix 995, Nikon, Tokyo, Japan). The analysis of characteristic X-rays (EDX analysis) emitted from the sample gives more quantitative elemental information. Such X-ray analysis can be confined to analytical volumes as small as one cubic micron.

2.5 FTIR and UV-vis spectroscopy

The chemical groups presented in the irradiated area in the specimens and non-irradiated were identified by the Fourier transform infrared spectrometer. Impact sites were mixed with dry potassium bromide powder KBr with a ratio of 1:100, by applying sufficient pressure, the mixture was prepared to scan. FTIR spectra of the specimens were collected in the wavenumber range of (400 - 4000) cm^{-1} using (FTIR) spectrometer (Satellite FTIR 5000).

To study the effect of laser irradiation on the absorbance of the irradiated specimens and the non-irradiated specimens an aqueous suspension of the zirconium silicate was evaluated using a Jasco-670 UV-Visible spectrometer.

III. RESULTS AND DISCUSSIONS

The photothermal effect of the irradiation of zirconium silicate specimens with Nd: YAG laser at 1064 nm wavelength and 60 W output power with continuous mode for different durations generates heat witch caused in the following results:

3.1. Hardness and Tensile Strength Results

The results of irradiation with Nd: YAG laser (power 60 W) at different duration time (0, 3, 4 and 5) minutes on the zirconium silicate specimens' hardness were listed in Table 1. It shows considerable increasing in hardness with increasing irradiation time from 3 to 5 minutes; it changed at a constant irradiation time at one minute. It increased about one third (37.5%) at three minutes; it increased the half (50%) at four minutes and about two thirds (62.5%) at five minutes.

The results of irradiation on the zirconium silicate specimens' tensile strength were listed in Table 2. It was calculated by applying the following equation:

$$Ts = 3.45 \times HB$$

The experimental data presented in Tables 1 and 2 show that, in all experiments, a linear correlation between hardness or tensile strength and laser irradiation time was detected.

Table 1 Hardness results for untreated zirconium silicate sample (A) and zirconia treated with Nd: YAG laser specimens (B, C and D)

Sample	Hardness/ HV=1.8544 F/D²	Hardness/HB	Hardness changes/HB	Change %
A	8.1	8	0	0
B	10.5	11	3	37.5
C	11.7	12	4	50
D	12.6	13	5	62.5

Table 2 Tensile strength results fo
r untreated zirconium silicate sample (A) and zirconia treated with Nd: YAG laser specimens (B, C and D)

Sample	Hardness/ HB	Tensile strength/ MPa	Tensile strength changes/ MPa	Change %
A	8	27.6	0	0
B	11	37.95	10.35	27.27
C	12	41.4	13.8	33.33
D	13	44.85	17.25	38.46

3.1. SEM Results

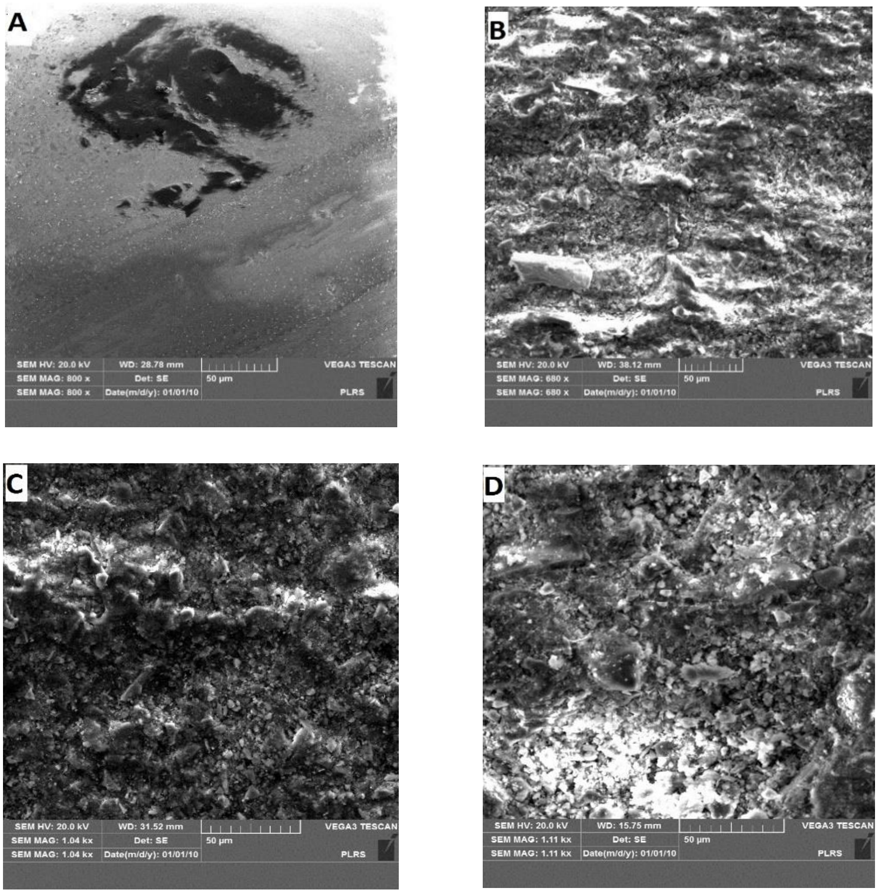


Figure 1: SEM images of zirconium silicate surface after Nd: YAG laser irradiation (60 W): (A) 0 min irradiation duration, (B) 3 min irradiation duration, (C) 4 min irradiation duration and (D) 5 min irradiation duration

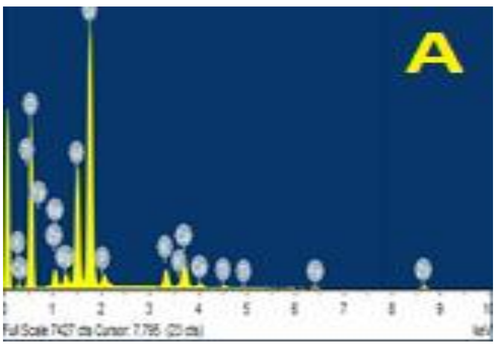
The SEM images (50 μm scale) of the zirconium silicate surface obtained with irradiation duration equal to 3, 4 and 5 min are shown in Figure 1. It shows that different irradiation times of irradiation induced different surface morphology.

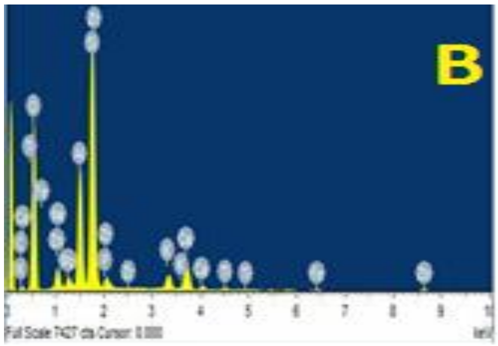
The SEM images of Nd: YAG laser-treated specimens B, C and D revealed a nonhomogeneous rough surface with many irregularities and holes compared to untreated specimen A. This surface includes the formation of microstructures in the produced dimples and grooves. The presence of thermal damaging effects, such as melting, burning, and cracks, was not detected, no microcracks were observed proving that the irradiation process does not cause material defect.

It can be observed that they obtained morphology vary from a roughness surface (Figure 1B) to a smooth one (Figure 1D), depending of the irradiation duration, which is meaning solidification process occurring on the specimens' surfaces in all accumulated irradiation duration. This result is agreeable with the linear correlation between hardness and tensile strength with laser irradiation time.

3.2. EDX Results

The surfaces of irradiated zirconium silicate specimens were examined with EDX to evaluate changes in chemical composition, the spectra shown in Figure 2. It is giving the elemental analysis data of the samples before and after laser irradiation treatment (with 4 and 5 minutes). The presence of the same elements is coincides with the results from laser irradiated and non-irradiated specimens, which mean that laser irradiation does not change the chemical surface composition of ceramics. EDX spectra show that the zirconium and silicon have the highest peaks compared to the other elements.





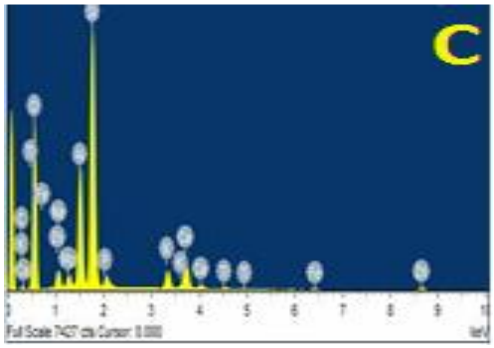


Figure 2: EDX spectra of un-irradiated zirconium silicate (A) and Nd: YAG laser (60 W) irradiated zirconium silicate specimens (B) and (C), 4 and 5 min irradiation duration, respectively.

3.3. FTIR Results

The FTIR spectra of zirconium silicate specimens a) before laser irradiation and b, c and d) and after laser irradiation are shown in Figure 3 and it was tabled in Table 3. The spectra showed hydroxyl groups (O-H) in deferent positions; the bands around 3696 cm-1 and 3620 cm-1 assigned outer and inner hydroxyl stretching vibration (Müller, et al., 2014; Kumararaja, et al., 2017), a broad band centered on 3442 cm-1 was due to the interlayer and intralayer H-bonded O-H stretching (Kumararaja, et al., 2017). Peak in the region of 3431 cm-1 also denotes the presence of hydroxyl groups (O-H) (Balan, et al., 2019).

Spectra also shwoed other functional groups such as amide, clay minerals, and silica. The amide A band (associated with N–H stretching) positions at 3423 cm-1 (Riaz, et al., 2018). The peak identified at 1025 cm-1 that correspond to the C–O stretching (Balan, et al., 2019). The 1033 cm-1 band is attributed to the Si-O stretching vibration (Vijayaragavan, et al., 2013). Clay minerals (Diopside) are characterized by the presence of 920 cm-1 (De Benedetto, et al., 2002). The band at **787** cm-1 is attributed to the stretching vibrations of Te-O bonds trigonal pyramids (Upender, and Prasad, 2017). The symmetrical bending vibration of the amorphous silica Si–O (quartz) group found at 695 cm-1 (Saikia, et al., 2008), this quartz with small particle size, improves mechanical strength of the ceramic bodies. The band at about 536-538 cm-1 is attributed to the coupling between the O-Si-O bending vibration and the K-O stretching vibration (Theodosoglou, et al., 2010). Al–O–Si and Si–O–Si bending vibrations produced the bands at 538 and 470 cm-1, respectively (Yin, et al., 2019).

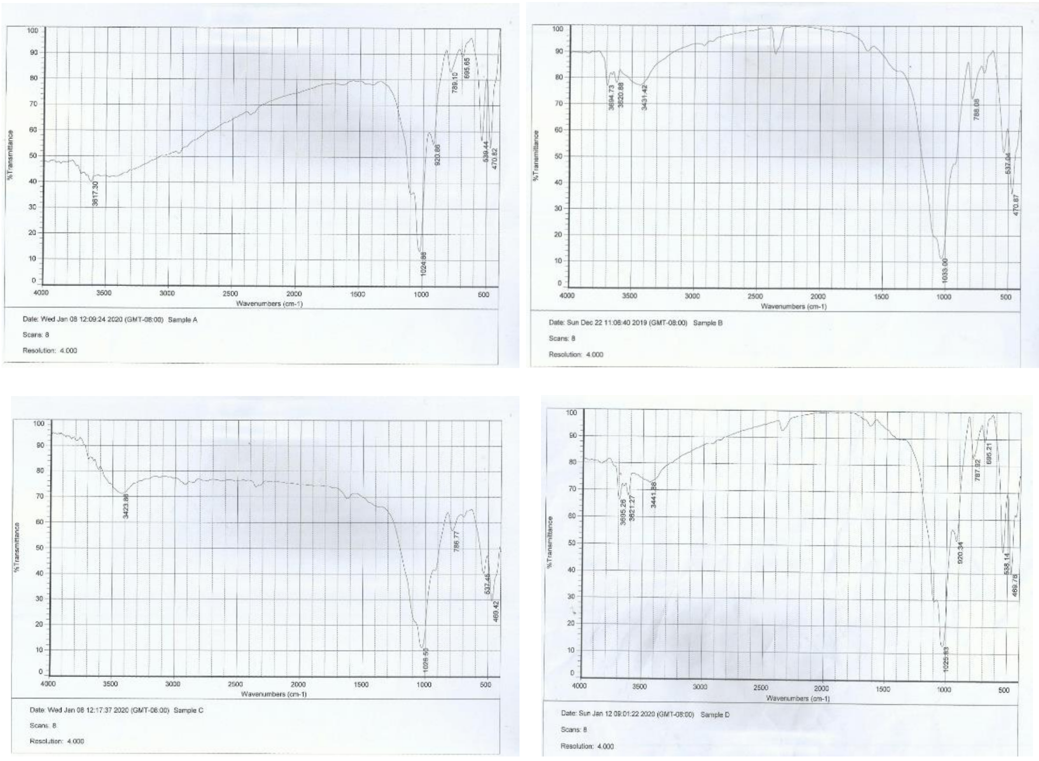


Figure 3 : FTIR spectra of zirconium silicate specimens a) before laser irradiation b,c and d) and after laser irradiation

Table 3. The main structural shifts observed in IR

Assignment	Characteristic Absorption (cm^{-1})				Reference
	Sample A	Sample B	Sample C	Sample D	
Inner surface-OH vibrations.	3695.26	-	3694.73	-	Müller, *et al.*, 2014
O-H stretching inner hydroxyl group	3621.27	3617.30	3620.88	-	Kumararaja, *et al.*, 2017
O-H stretching	3441.88	-	-	-	Kumararaja, *et al.*, 2017
hydroxyl groups (O-H)	-	-	3431.42	-	Balan, *et al.*, 2019
N–H stretching	-	-	-	3423.86	Riaz, *et al.*, 2018
C–O stretching	1025.83	1024.88	-	1026.50	Balan, *et al.*, 2019

Si-O stretching vibration	-	-	1033.00	-	Vijayaragavan, *et al.*, 2013
Diopside	920.34	920.86	-	-	De Benedetto, *et al.*, 2002
Te-O bonds	787.92	-	788.08	786.77	Upender, and Prasad, 2017
Amorphous silica Si–O (quartz)	695.21	-	-	-	Saikia, *et al.,* 2008
coupling between O-Si-O bending and K-O stretching vibrations	538.14	539.44	537.04	537.45	Theodosoglou, *et al.,* 2010
Si–O–Si bending vibrations	469.78	470.82	470.87	469.42	Yin, *et al.,* 2019

IV.CONCLUSION

The photothermal effect of the irradiation of zirconium silicate specimens with Nd: YAG laser at 1064 nm wavelength and 60 W output power with continuous mode for different durations was investigated. The hardness test show that Nd: YAG laser irradiation with (60 W) can increase the hardness of zirconium silicate ceramics. SEM images demonstrate the formation of microstructures, smoother surface and solidification process occurring confirming the hardness results. FTIR spectra denotes the presence of quartz with small particle size that improves mechanical strength of the zirconium silicate. Moreover, EDX results reveal that laser irradiation does not change the chemical surface composition of ceramics. In summary, the experimental results revealed a linear correlation between laser irradiation duration and hardness, tensile strength and surface solidification, without causing material defect.

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