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INVESTIGATION OF THE EFFECTS OF HIGH-POWER ND: YAG LASER ON ZIRCONIUM SILICATE SURFACE PROPERTIES

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Abstract : ***In this, study the influence of high power Nd: YAG laser irradiation on the hardness and surface properties of zirconium silicate ($ZrSiO_4$) ceramics was investigated. Specimens of zirconium silicate ($ZrSiO_4$) ceramic pieces were separated into four samples according to irradiation duration as follows: one control sample (no treatment), and three samples irradiated with Nd: YAG laser at irradiation times 3, 4 and 5 minutes. The irradiation was applied with fixed output power 60 W. The hardness and tensile strength were measured, SEM and EDX characterization was done and the optical properties were characterized by UV-vis spectroscopy. The present results show that high power (60 W) Nd: YAG laser provide higher hardness compared to non-treated surfaces. Extending irradiation time can induce higher hardness of the ceramics. SEM images demonstrate the formation of microstructures, smoother surface and solidification process occurring confirming the hardness results. EDX results reveal that laser irradiation does not change the chemical surface composition of ceramics. Moreover, Increase in transmittance of the irradiated zirconium silicate in the visible and near infrared range was also found using UV-vis spectroscopy.***

Keywords- Hardness; Laser Irradiation; Laser-matter interaction; Surface morphology; Surface Solidification; Zirconium silicate.

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 [1].

There have been numerous research studies the effect of laser matter irradiation; for example, on killing bacteria (laser milk pasteurization) [2, 3], silicon surface modification in solar cells [4], food irradiation (bee honey irradiation) [5] and production of highly value materials from agricultural waste [6, 7]. 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 [8, 9] and it also used to induce emission in human teeth to distinguish between dental caries and sound teeth [10].

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 [11, 12], surface modification of Ti dental implants by laser [13], laser treatment of dental ceramic/cement layers [14], effects of laser on filling materials [15] and effect of laser in hardness of dental ceramic [16].

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

I. MATERIALS AND METHODS

A. 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.

B. 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.

C. 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:

$$Ts = 3.45 \times HB$$

D. SEM and EDX 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.

E. UV-vis spectroscopy

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 carried out using a Jasco-670 UV-Visible spectrometer.

II. 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:

F. 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 that the hardness of the irradiated samples was obviously increased. A considerable increasing in hardness with increasing irradiation time from 3 to 5 minutes was found; it changed at a constant irradiation time at one minute. It increased about one third (37.5%) at irradiation time three minutes, it increased the half (50%) at four minutes and about two thirds (62.5%) at five minutes.

TABLE I

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

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

$$Ts = 3.45 \times HB$$

Table 2

Tensile strength results for 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

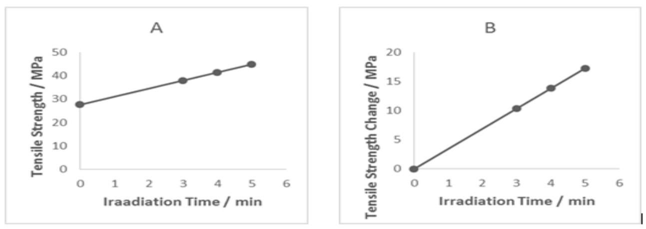


Fig. 2 Effect of irradiation time on zirconium silicate samples' A) tensile strength B) tensile strength change

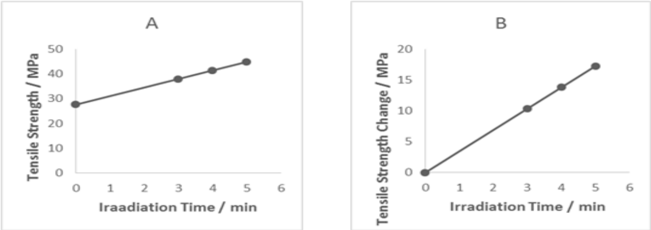


Fig. 2 Effect of irradiation time on zirconium silicate samples' A) tensile strength B) tensile strength change

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.

Fig. 1 A) depict the linear correlation between hardness and irradiation time, while Fig. 1 B) depict the linear correlation between hardness change and irradiation time.

Fig. 2 depict A) depict the linear correlation between tensile strength and irradiation time, while Fig. 2 B) depict the linear correlation between tensile strength change and irradiation time.

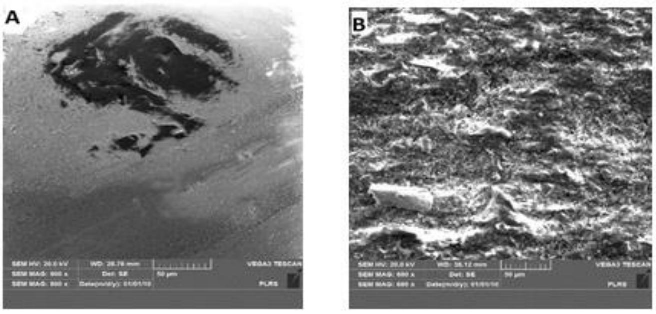
Graphs in Figures 1 and 2 presented that extending irradiation time can induce higher hardness of the ceramics.

B. SEM Results

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 Fig. 3. 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 (Fig. 3B) to a smooth one (Fig. 3D), 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.



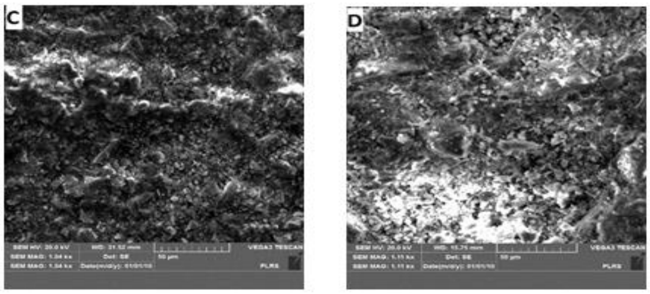


Fig. 3: SEM images of zirconium silicate surface after Nd: YAG laser irradiation (60 W): (A) 0 min irradiation duration, (B) 3 min irradiation duration, (C) 4 min irradiation duration and (D) 5 min irradiation duration

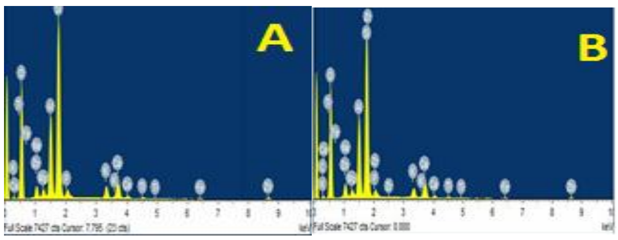
C. EDX Results

The EDX spectra shown in Fig. 4 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 do 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 as shown in Table 3.

Table 3

Edx results of zirconium silicate sample (a) before, sample b) and samples c) after nd: yag laser irradiation (60 w): 0, 4 and 5 min irradiation duration respectively

Element	Sample A		Sample C		Sample D	
	Weight%	**Atomic%**	**Weight%**	**Atomic%**	**Weight%**	**Atomic%**
C K	0	0	2.95	4.87	6.20	10.04
O K	54.14	68.64	54.31	67.30	51.70	62.82
Na K	1.70	1.50	1.61	1.39	1.88	1.59
Mg K	1.09	0.91	0.89	0.73	0.81	0.65
Al K	9.51	7.15	8.94	6.57	8.78	6.32
Si K	23.59	17.03	21.25	15.00	21.19	14.67
P K	1.24	0.81	0.64	0.41	1.21	0.76
K K	2.22	1.15	1.76	0.89	1.75	0.87
Ca K	3.71	1.88	3.45	1.71	3.23	1.57
Ti K	0.26	0.11	0.23	0.09	0	0
Fe K	0.57	0.21	0.29	0.10	0.27	0.09
Zn K	1.97	0.61	1.73	0.52	1.63	0.49
Zr L	0	0	1.96	0.43	0	0
Pt M	0	0	0	0	1.34	0.13
Totals	100.00		100.00		100.00	



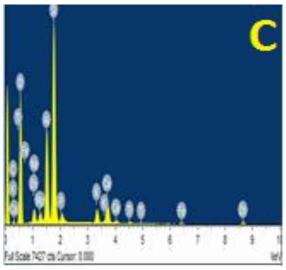


Fig. 4: EDX spectra of zirconium silicate (A) before, B) and C) after Nd: YAG laser irradiation (60 W): 0, 4 and 5 min irradiation duration respectively

D. UV-vis spectroscopy Results

The UV-vis spectroscopy spectra of the four samples are shown in Fig. 5. Decreased absorbance was observed in the laser-irradiated samples in the visible and near infrared range (350-800 nm). When comparing non-irradiated sample with each of the laser irradiated samples, non-irradiated sample always showed higher absorbance. The process of laser irradiation presented effect in transmittance of the zirconium silicate.

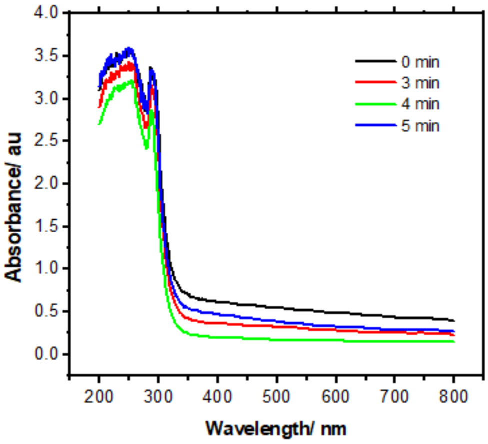


Fig. 5: UV spectra of zirconium silicate specimens before and after laser irradiation

III. CONCLUSIONS

In summary, the experimental results obtained from high power Nd: YAG laser irradiation of zirconium silicate show that the hardness of zirconium silicate could be increased with irradiation. A linear correlation between hardness or tensile strength and laser irradiation time was detected. SEM images demonstrate the formation of microstructures, smoother surface and solidification process occurring confirming the hardness results. EDX results reveal that laser irradiation do not change the chemical surface composition of ceramics. Moreover, UV-vis spectra show more transmittance of irradiated zirconium silicate specimens compared to the non-irradiated specimens. Nd: YAG laser irradiation with (60 W) can increase the hardness of zirconium silicate ceramics.

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