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ABSTRACT

Purpose: This study was carried out to compare the effect of laser surface treatment on cubic and tetragonal zirconia ceramics regarding; phase transformation tetragonal-monoclinic (T-M), surface topography, mechanical properties and biaxial flexural strength before and after low thermal degradation (LTD) aging. **Materials and Methods**: sixty discs (N=60) of CAD/CAM zirconia were used in this study. These specimens were divided into two main groups according to type of zirconia. Group I: (n=30) Cubic Zirconia (DD cubeX^2), while Group II: (n=30) Tetragonal zirconia (Superfect-Zir). Then each group further subdivided into two subgroups according to surface treatment, subgroup (1): control without surface treatment, subgroup (2): laser surface treatment .Specimens form each subgroups were tested for the following; Phase transformation, surface topography, and mechanical properties before and after Low Thermal Degradation (LTD) aging test. **Results**: Statistical analysis revealed that there was a statistical significant difference between the two tested groups regarding the surface topography, biaxial flexural strength, and surface hardness. Also there was a statistical significant difference regarding the surface treatment and before/after LTD and laser among the tested zirconia ceramics. **Conclusions**: laser surface treatment improved the properties of tetragonal / cubic zirconia, and increases their resistance to low thermal degradation LTD aging.

KEYWORDS

Cubic Zirconia,
Thermal Degradation,
Laser Surface Topography,
Biaxial Flexural Strength.

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INTRODUCTION

Good aesthetic a reasonable factor for the use of ceramics in dentistry [(1)], however, the major problem is their low fracture-resistance. Yttria-stabilized tetragonal zirconia (Y-TZP) has become the best for crowns/bridges, implant abutments, and cores; due to its high strength and toughness mechanism [(2-4)].

Zirconia is present in three forms; cubic, tetragonal, and monoclinic forms. Zirconia has cubic structure at 2680˚C and on cooling from this temperature crystallographic transformations occur; tetragonal and monoclinic phase at 2370°C and 1170˚C respectively [(4)]. This crystal transformation induces internal stresses with a 3-5% expansion [(5,6)]. Stabilizing agents as yttria and ceria are added to the tetragonal phase to be stable at 37°C and to control volumetric expansion [(7)]. This mechanism results in increasing the mechanical properties of Y-TZP. Also, this transformation may occur at low temperatures and in presence of water which commonly called low thermal degradation (LTD) [(8)].

The LTD phenomenon occurs as follows; at temperatures (65-300^{o}C) in presence of water phase transformation (t–m) starts, where it proceeds gradually from the surface into the internal surface of the ceramic. Subsequently, micro- and macro cracks develop due to volume expansion occurs [(9)]. This result in increased surface roughness, and decreasing strength, toughness, and density [(10)].

The introduction of (CAD/CAM) has become an alternative technique to the traditional techniques [(11,12)] as it decreases time of processing and abrasion of the cutting instruments during milling. Many CAD/CAM systems use partially sintered instead of fully sintered ones [(13)]. The partially sintered material is then subjected to final sintering after milling.

Different methods have been used to modify either the internal or external surface of the dental restoration. One of these methods is the usage of laser, which has been used to modify the surface properties of dental ceramics; through influencing the surface microstructure, roughness, and mechanical properties causing modifications to these materials [(14)]. Therefore, the objective of this work was to compare the effect of laser on two CAD/CAM zirconia ceramics; cubic and tetragonal on their resistance to degradation [(15,16)]. This study was carried out to evaluate the effects of surface treatment or heat treatments on the properties of the zirconia ceramics as; flexural strength, surface hardness, and surface roughness of the dental ceramics [(17,18)].

MATERIAL AND METHODS

Specimens' grouping

A total of sixty discs of two brands of CAD/CAM zirconia ceramics were used in this in-vitro study. The specimens were divided into two main groups according to type of zirconia.

Group I (n=30): Cubic zirconia (DD cubeX^2-Dental Direkt materials- Germany).

Group II (n=30): Tetragonal zirconia (SuperfectZir-ST-14 Aidite high-technical Ceramics Company-Germany)

Each group was further subdivided into two subgroups (n=15) according to surface treatment.

Subgroup (1): Control (without surface treatment).

Subgroup (2): Laser surface treatment.

Phase transformation, surface topography, and mechanical properties before and after Low Thermal Degradation (LTD) aging test were performed to the control and laser treated subgroups of zirconia ceramics materials.

Specimens' preparation

Designing of the specimens; the required shape of zirconia blanks was designed using digital software Exocad system (developed by university of Chicago) in order to accurately design the cylinder shape from zirconia blank.

Milling of the zirconia blank; the blanks were inserted inside the milling machine (Roland-DWX-510-Japan), and milled according to the design imported data with an approximate 20-25% oversize to compensate for sintering shrinkage according to manufacture instructions.

Fabrication of the discs; Sixty zirconia discs were then cut from their respective cylinder using a low speed diamond saw (Isomet 4000 precision cut,Buehler, USA) with cutting speed 2500 rpm, under cooling system.

Sintering of zirconia discs; the sintering process of the sixty pre-sintered zirconia discs, started by placing them on a sintering tray containing the appropriate sized of zirconia beads in the sintering furnace (TABEO-1/5/ZIRKON-1000-Germany). The discs were then sintered in the firing oven to complete sintering following the firing schedule illustrated in the manufacturer's instructions. Digital caliper (Guilin Measuring of cutting Tool Co.,Ltd-China) was used to verify the final thickness of the discs after sintering, the final dimensions of the specimens were 10 mm diameter × 1.2 mm thickness according to ISO 6872:2008[19].

Finishing and polishing of the sintered zirconia discs; the discs were minimally finished using (Eve-Diasynt plus and Diacera zirkonoxid zirconia-Germany) according to manufacture instruction with minimal pressure and under water coolant. Then all specimens were ultrasonically cleaned using (Vita-Vitasonic II-china) for 5 minutes, and then dried to remove any debris contaminations.

Laser surface treatment

The laser subgroups were treated using laser device (Laser Diode Driver LDD 50-Tokyo-Japan), in the National Institute of Laser Enhanced Science (NILES) at Faculty of Science, Cairo University.

Diode laser was used with the following parameters: wavelength 780-1000 Nanometer, maximum power 5watt, continuous mode.

The specimens were maintained perpendicularly to the laser beam by attaching them in special holder at 3cm distance, the specimens were subjected to laser beam for 10 seconds with a speed of 0.3cm/sec[20] according to previously pilot study using different parameter. The laser power was measured using power meter (Solo2-Laser power-Energy meter-gentec-China) in order to adjust and fix the power value for all the specimens.

Low thermal degradation (LTD) Aging

The tested groups were subjected to low thermal degradation (LTD) aging test using an autoclave (TS Tau Steril -Fino Mornasco-Italy). The discs of each subgroup were packed in small labeled sterilization packs which were arranged in the autoclave trays. The autoclave was programmed at 134^{o}C, 2bars pressure for 5 hours (10 cycles). The autoclave cycle starts from zero pressure and increased to the desired pressure (2 bars) in 15 minutes so the autoclave cycle (45 minutes) was calculated only as 30 minutes.

Prior and after hydrothermal degradation, each specimen was characterized by the following:

I- Phase transformation

The effect of surface treatment on (t-m) phase transformation and crystalline structure of the tested zirconia ceramics was examined using X-ray diffractometer (XRD) (Max B; Shimadzu, Tokyo, Japan). Quantitative analysis of the tetragonal to monoclinic (t–m) XRD spectrum was collected over a range between 27° and 33° at a scan speed of 1°/min and a step size of 0.02.

The relative amount of the monoclinic phase fraction (*X*m) was calculated by equation given by Gravies and Nicholson [21].

$$X\text{m} = \frac{[I\text{m}\,(111) + I\text{m}(11\bar{1})]}{[I\text{m}\,(111) + I\text{m}(11\bar{1}) + I\text{t}\,(111)]} \times 100$$

II-Surface topography

1-Scanning electron microscope (SEM)

The specimens were scanned using SEM (model Quanta 250; FEG Field Emission Gun, at the Egyptian Mineral Resource Authority). The scanning was done to investigate the change in microstructure of the specimens after surface treatment. The specimens were viewed at magnification power 2000x. Analysis of data was performed using SPSS 17 (Statistical Package for Scientific Studies) for Windows.

2. Surface roughness using Atomic Force Microscope (AFM)

The surface roughness of the specimens was examined using Atomic Force Microscope (AFM) (Model: MLCT-MT-A). AFM scan an area of 10×10 μm with number of data points 256*256, and scanning rate 1Hz. The specimens were fixed on adhesive tape of magnetic specimen stubs, and the surface roughness was represented in x, y, z directions. The topographical data of the surface were recorded by computer software to give 3D images of the surface.

III-Mechanical properties

1-Biaxial flexural strength

Biaxial flexural test with a ball on ring was chosen in this study. Testing was done with universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). Data was recorded using computer software (Instron-Blue hill Lite Software). The biaxial flexure strength was calculated according to the following equation [22].

$$\sigma = P/h^2 \{(1+\nu)[0.485x \ In(a/h) + 0.52] + 0.48\}$$

2- Surface hardness

Surface hardness of the specimens was determined using digital display Vickers hardness Tester (Model: HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China). A load of 200g was applied to the surface of the specimens for 20 seconds. Micro-hardness was obtained from the following equation [23]:

$$HV=1.854 \ P/d^2$$

Statistical analysis

Numerical data was explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. Three-way mixed model ANOVA was used to study the effect of different tested variables and their interaction. Comparison of main and simple effects was done using paired t-test for aging and independent t-test for other variables. The significance level was set at $P \leq 0.05$ within all tests. Statistical analysis was performed with IBM-SPSS® Statistics Version 25 for Windows.

RESULTS

I. Phase transformation of zirconia ceramics using X-ray diffractometer (XRD)

Phase transformation of the two zirconia ceramics (cubic and tetragonal) used in this in-vitro study was examined using XRD after subjected to different surface treatment.

Regarding the cubic zirconia; The XRD pattern of the control specimens of cubic zirconia ceramics revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.24; and then at 2Θ=30.46. Other well defined peaks were found at 2Θ=50.35 and 2Θ=50.58 which are corresponding to relative intensity I/I_0=46.33 and I/I_0=43.92 respectively (figure1 a). The XRD pattern of the cubic zirconia ceramics specimens subjected to laser surface treatment revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.25 other well defined peak was found at 2Θ =50.8 which corresponding to relative intensity I/I_0= 63.02 (figure 1b).The XRD pattern of control cubic zirconia ceramics specimens after subjected to LTD revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=29.92.Other well defined peaks were found at 2Θ=50.08 and 2Θ=59.33 which are corresponding to relative intensity I/I_0=63.95 and I/I_0=39.57 respectively(figure 1c). Laser treated specimens after subjected to LTD revealed that maximum peak relative I/I_0 intensity of 100% was found at2Θ =29.9 other well defined peaks was found at 2Θ=50.26 which corresponding to relative intensity I/I_0=44.25 (figure 1d).

XRD patterns of the low temperature degradation specimen displayed only broad diffraction peak at approximately 2Θ= 59.3 and 2Θ=62.4. These peaks were attributable to the combined peaks of the cubic and tetragonal phases. As low thermal degradation (LTD) aging progressed, monoclinic peaks appeared in the XRD pattern at 2Θ=59.3, and 2Θ= 62.4 respectively. The relative amount of the monoclinic mass fraction Xm as calculated from the equation given by Gravies and Nicholson and resulted into 3% phase transformation.

Regarding the tetragonal zirconia; The XRD pattern of the control specimens of tetragonal zirconia ceramics revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.6. Other well defined peaks were found at 2Θ=50.58 and 2Θ=50.73 which corresponding to relative intensity I/I_0=41.40 and I/I_0=26.17 respectively (figure 1e). The XRD pattern of the tetragonal zirconia ceramics specimens which subjected to laser surface treatment revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.08.Other well defined peaks was found at 2Θ=29.66 and 2Θ=50.05 which corresponding to relative intensity I/I_0=63.40 and I/I_0=55.08 respectively (figure 1f). The XRD pattern of the control tetragonal zirconia ceramics specimens after subjected to LTD revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.56.Other well defined peaks were found at 2Θ=50.55 and 2Θ=50.70 which corresponding to relative intensity I/I_0=42.09 and I/I_0=23.57 respectively (figure 1g). The XRD pattern of the laser treated tetragonal zirconia ceramics specimens after subjected to LTD revealed that maximum peak relative I/I_0 intensity of 100% was found at 2Θ=30.04 (figure 1h).

XRD patterns of the low thermal degradation specimen at134^{o}C in water steam resulted in a notable t–m phase transformation. XRD patterns displayed only one single broad diffraction peak at approximately 2Θ=29.03, 2Θ=42.03, and2Θ= 45.04. These peaks were attributable to the combined peaks of the cubic and tetragonal phases. As low thermal degradation (LTD) aging progressed, monoclinic peaks appeared in the XRD pattern at and resulted into 7% phase transformation.

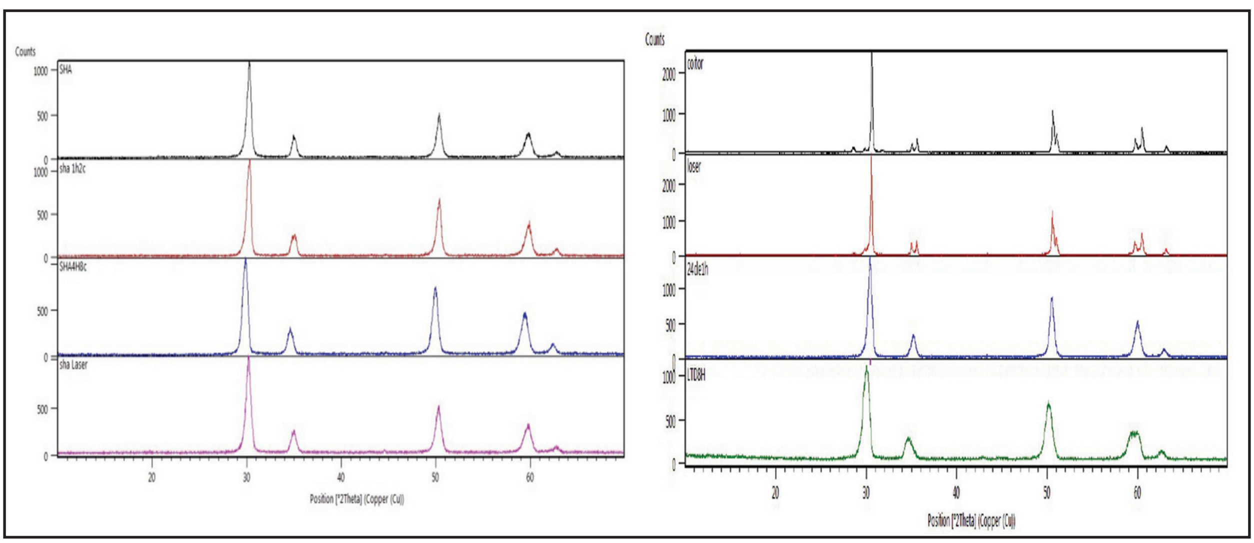

Figure (1) XRD pattern of cubic zirconia ceramics a) control specimen, b) laser surface treatment specimen, c) control after LTD aging specimen d) laser after LTD. While XRD pattern of tetragonal zirconia ceramics specimens after surface treatment: e) control specimen, f) laser surface treatment specimen, g) LTD aging specimen, h) laser after LTD

II-Surface Topography

1-Scanning Electron Microscope (SEM)

The change in the surface morphology of the zirconia ceramics used in this in- vitro study was examined using SEM.

Regarding cubic zirconia; the morphologic surface for the control specimen of cubic zirconia show a clear apperance of the crystalline structure. The specimen possessed an average grain size of 0.5 μm, as determined by the linear intercept method [(24)] (figure 2a).After LTD, the morphologic surface of the control specimens of the cubic zirconia ceramics show the presence of microcracks, irregular holes on rough surface (figure2b).After laser surface treatment of the cubic zirconia ceramics,the morphologic surface show smooth surface with multiple burned areas embedded into a wide smooth melted region compared to the clear crystal structure of the control specimens (figure 2c). LTD after laser surface treatment, the morphologic surface of the laser treated cubic zirconia ceramics after subjected to LTD, show the presence of microcracks, irregular multiple holes and melting areas with some burned areas on rough surface (figure 2d).

Regarding tetragonal zirconia; the morphologic surface for the control specimen of tetragonal zirconia show the clear apperance of the crystalline structure with obviously visible grain boundaries. The specimen possessed an average grain size of 0.3 μm, as determined by the linear intercept method (figure 2e).After LTD, the morphologic surface of the control specimens of the tetragonal zirconia ceramics show the presence of microcracks, multiple irregular holes on relatively rough surface (figure 2f).After laser surface treatment to the tetragonal zirconia ceramics, the morphologic surface show zirconia melted layer with a smooth and glassy morphology with shiny appearance (figure 2g). After LTD, the morphologic surface of the tetragonal zirconia ceramics show the presence of shallow and smooth depressions on rough surface, microcracks, irregular holes and melting areas (figure 2h).

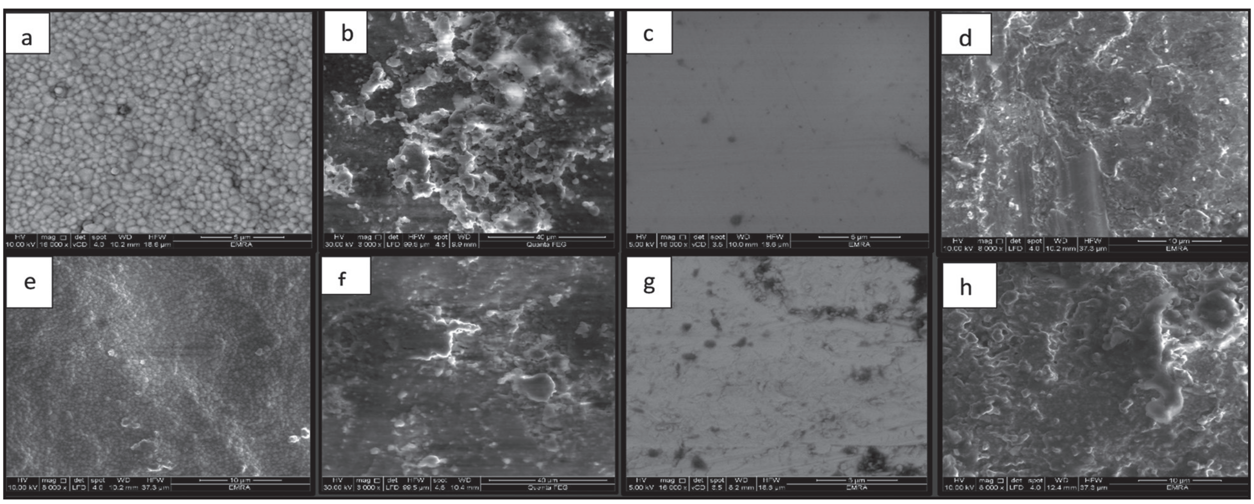

Figure (2) SE micrograph for cubic zirconia ;a)control specimen, b)control after LTD,c)Laser specimens,d) laser after LTD;While SE micrograph for tetragonal zirconia ;e)control specimen f)control after LTD;g)Laser specimens;h)laser specimen after LTD

2- Surface roughness

Tables (1,2) and figure (3) show mean and standard deviation (SD) values of surface roughness Ra (μm) for the tested zirconia (cubic and tetragonal) materials and surface treatment before and after (LTD) aging. **Regarding cubic zirconia;** mean values of surface roughness of control specimens after (LTD) aging showed statistically higher values than before (LTD) aging (25.44±0.38, 24.90±0.47μm respectively), however, the difference was not significant (P=0.104). Meanwhile, mean values of surface roughness of laser specimens after (LTD) aging showed statistically significant higher values than before (LTD) aging 25.37±0.05 μm, 25.14±0.03) respectively, (P< 0.001).**Regarding tetragonal zirconia**; mean surface roughness values of the control specimens after (LTD) aging showed a lower value (25.45±0.36 μm) than before (LTD) aging (25.66±0.24 μm), but the difference was not significant (P=0.28). Meanwhile, laser treated specimens after (LTD) aging showed a significantly higher mean surface roughness value (25.48±0.23 μm) than before (LTD) aging (25.16±0.06 μm) (P=0.027).Furthermore, statistical results revealed that tetragonal zirconia showed higher mean surface roughness values for both control and laser treated specimens than cubic zirconia (P≤ 0.05).Moreover, laser treated specimens of cubic zirconia and tetragonal zirconia before and after (LTD) aging had a significantly higher mean surface roughness values than the control specimens (P≤ 0.05).

Table (1) *Mean ± standard deviation (SD) of surface roughness for tested zirconia materials and surface treatment before and after (LTD) aging.*

Material	Aging	Surface treatment (mean±SD)		P-value
		Control	Laser	
Cubic zirconia	Before	24.90±0.47	25.14±0.03	0.294ns
	After	25.44±0.38	25.37±0.05	0.711ns
	P-value	0.104ns	<0.001*	
Tetragonal zirconia	Before	25.66±0.24	25.16±0.06	0.002*
	After	25.45±0.36	25.48±0.23	0.857ns
	P-value	0.280ns	0.027*	

Table (2) *Mean ± standard deviation (SD) of surface roughness Ra (µm) for different materials and surface treatments before and after (LTD) aging.*

Aging	Surface treatment	Material (mean±SD)		P-value
		Cubic zirconia	Tetragonal zirconia	
Before	Control	24.90±0.47	25.66±0.24	0.013*
	Laser	25.14±0.03	25.16±0.06	0.980ns
After	Control	25.44±0.38	25.45±0.36	0.560ns
	Laser	25.37±0.05	25.48±0.23	0.340ns

**; significant (p ≤ 0.05)*

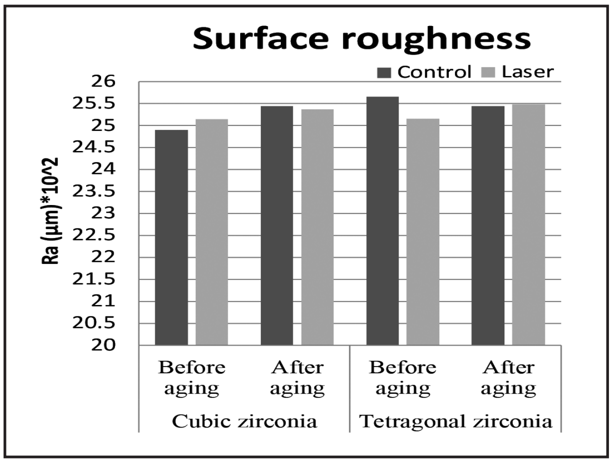

Figure (3) Bar chart showing average surface roughness Ra (μm) for tested materials and surface treatment before and after (LTD) aging.

3- Topographic assessment of the tested zirconia ceramics

Figures (4 a,b,c,d) show topographic 3D images of the control specimens and then after LTD. Laser surface treated specimens, and then after LTD of the cubic zirconia ceramics.

Topographic 3D images for control specimens of the cubic zirconia ceramics show relative rough surface with number of irregularities which demarcate the grain and the crystalline structure of the specimens as shown in figure (4a).However, after subjected to LTD the specimens show relatively high sharp peaks started to appear on the surface as shown in figure (4b).Topographic 3D images for laser surface treatment specimens of the cubic zirconia ceramics, show well demarcated smooth surface features compared to the control specimen as a function of laser surface treatment as shown in figure (4c). After low thermal degradation (LTD) aging, the topographic 3D images of the laser treated specimen show numerous highly peaks with sharp pointed edge spread over relative smooth abraded surface irregularities in figure (4d).

Topographic 3D images for the tetragonal zirconia ceramics, the control specimens show irregular surface and multiple elevations, deep grooves and valleys figure (4e). However; after LTD increase in the surface elevation and irregularity nodular like appearance were seen as in figure (4f).While after laser surface treatment fewer irregularities were noticed with obvious and clear smooth surface compared to the control specimen as show in figure (4g). After low thermal degradation (LTD) of the laser treated specimens their topographic 3D images show rough surface with rounded smoothened edges scattered all over the surface (figure 4h)

III-Mechanical properties

1-Biaxial flexural strength

Table (3,4) and figure (5) show mean and standard deviation (SD) values of biaxial flexural strength (Mpa) for the tested zirconia (cubic and tetragonal) materials and surface treatment before and after (LTD) aging. **Regarding cubic zirconia**; mean biaxial flexural strength values of both control specimens and laser treated specimens after (LTD) aging showed statistically lower values (610.85± 2.56 Mpa, 655.57± 29.67 Mpa respectively) than before aging (621.04± 3.99 Mpa, 819.90± 33.15 Mpa respectively) (P≤ 0.05).

Regarding tetragonal zirconia; mean biaxial flexural strength values of the control specimens after aging showed a lower value (1210.25± 9.50 Mpa) than before (LTD) aging (1270.06±57.99 Mpa), but the difference was not significant (P=0.070). Meanwhile, laser treated specimens after(LTD)aging showed a significantly higher mean biaxial flexural strength value (1283.93± 46.73 Mpa) than before (LTD) aging (1408.70± 64.85 Mpa) (P=0.001).Furthermore, generally, statistical results revealed that tetragonal zirconia showed

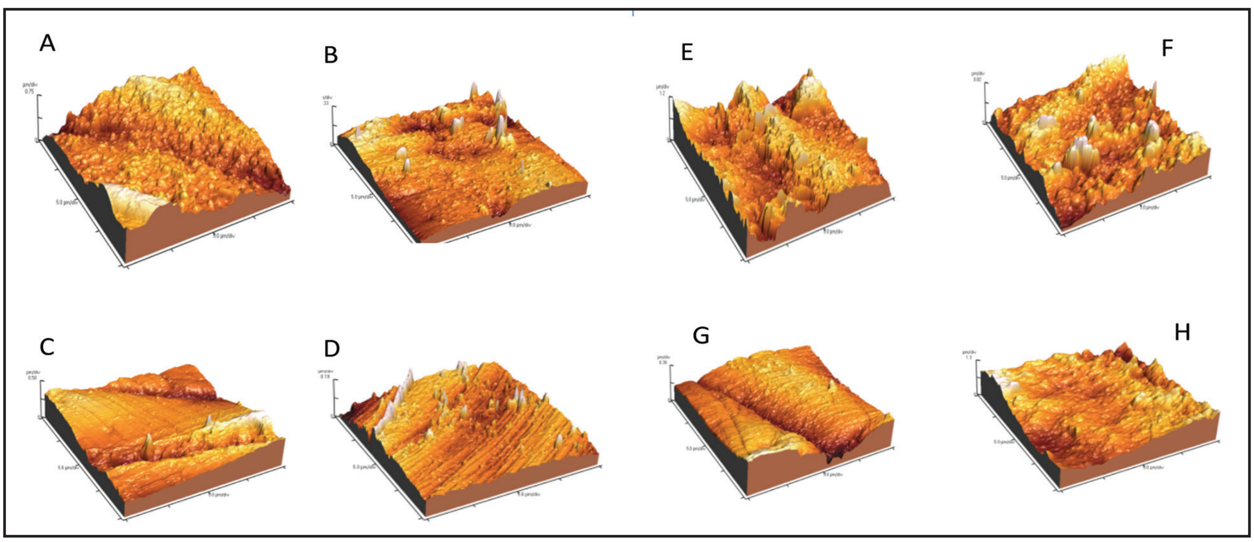

Figure (4) Topograghic 3D image for cubic zirconia; a) control specimen b) control after LTD c) laser specimen ,d) laser specimen after While LTD Topograghic 3D image for tetragonal zirconia; e) control specimen,f) control after LTD ,g) laser specimen, h) laser specimen after LTD statistically higher mean biaxial flexural strength values for both control and laser treated specimens than cubic zirconia (P≤ 0.05).Moreover, laser treated specimens of cubic zirconia and tetragonal zirconia before and after (LTD) aging had a significantly higher mean biaxial flexural strength values than the control specimens (P≤ 0.05).

Table (3): *Mean ± standard deviation (SD) of biaxial flexural strength (Mpa) for tested zirconia materials and surface treatment before and after (LTD) aging*.

Material	Aging	Surface treatment (mean±SD)		P-value
		Control	Laser	
Cubic zirconia	Before	621.04± 3.99	819.90 ± 33.15	**<0.001***
	After	610.85± 2.56	655.57 ± 29.67	**0.010***
	P-value	**0.013***	**<0.001***	
Tetragonal zirconia	Before	1270.06 ±57.99	1408.70 ± 64.85	**0.007***
	After	1210.25± 9.50	1283.93 ± 46.73	**0.009***
	P-value	**0.070ns**	**0.001***	

**; significant (p ≤ 0.05)*

Table (4): *Mean ± standard deviation (SD) of biaxial flexural strength (Mpa) for tested Zirconia materials and surface treatment before and after (LTD) aging*.

Aging	Surface treatment	Material (mean±SD)		P-value
		Cubic zirconia	Tetragonal zirconia	
Before	Control	621.0 4± 3.99	1270.06 ±57.99	**<0.001***
	Laser	819.9 0± 33.15	1408.70 ± 64.85	**<0.001***
After	Control	610.85± 2.56	1210.25± 9.50	**<0.001***
	Laser	655.57± 29.67	1283.93± 46.73	**<0.001***

**; significant (p ≤ 0.05)*

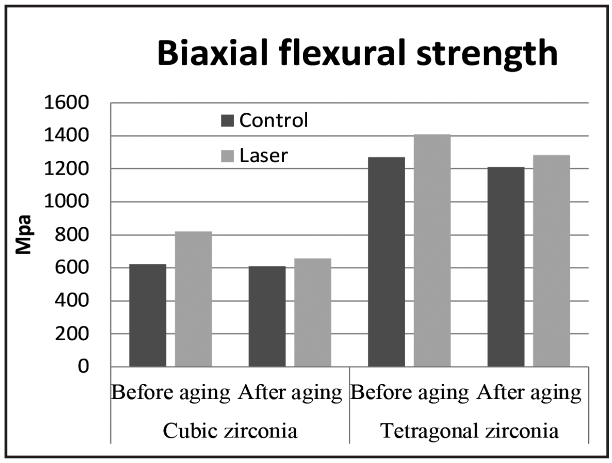

Figure (5) Bar chart show mean biaxial flexural strength (Mpa) for tested materials and surface treatment before and after (LTD) aging.

2- Surface hardness

Tables (5,6) and figure (6) show mean and standard deviation (SD) values of surface hardness for the tested zirconia (cubic and tetragonal) materials and surface treatment before and after (LTD) aging. **Regarding cubic zirconia**; mean surface hardness values of both control specimens and laser treated specimens after (LTD) aging showed statistically lower values (869.27±17.65Kg/mm^2, 1050.66±103.2865Kg/mm^2 respectively) (P=0.005) than before (LTD) aging (1059.73±42.0465Kg/mm^2, 1154.79±102.16Kg/mm^2), but the difference was not significant (P= 0.09). **Regarding tetragonal zirconia**; mean surface hardness values of the control specimens after (LTD) aging showed a lower value (1096.20±52.85 Kg/mm^2) than before (LTD) aging (1222.85±67.01 Kg/mm^2), but the difference was not significant (P=0.059). Meanwhile, laser treated specimens after (LTD) aging showed a lower surface hardness value (1322.33± 71.58 Kg/mm^2) than before (LTD) aging (1379.40± 100.76 Kg/mm^2), with insignificant difference (P=0.264). Furthermore, statistical results revealed that tetragonal zirconia showed statistically significant higher mean surface hardness values for both control and laser treated specimens than cubic zirconia (P≤0.05). Moreover, laser treated specimens of cubic zirconia and tetragonal zirconia before and after (LTD) aging showed higher mean of surface hardness values than the control specimens, but the difference was not significant (P>0.05).

Table (5): *Mean ± standard deviation (SD) of surface hardness (Kg/mm²) for tested zirconia materials and surface treatment before and after (LTD) aging.*

Material	Aging	Surface treatment (mean±SD)		P-value
		Control	Laser	
Cubic zirconia	Before	1059.73 ±42.04	1154.79 ± 102.16	0.091ns
	After	869.27 ± 17.65	1050.66 ± 103.28	0.005*
	P-value	<0.001*	0.096ns	
Tetragonal zirconia	Before	1222.85 ±67.01	1379.40 ± 100.76	0.001*
	After	1096.20 ±52.85	1322.33 ± 71.58	0.053ns
	P-value	0.059ns	0.264ns	

**; significant (p ≤ 0.05) .*

Table (6): *Mean ± standard deviation (SD) of surface hardness (Kg/mm ²) for tested Zirconia materials and surface treatment before and after (LTD) aging.*

Aging	Surface treatment	Material(mean±SD)		P-value
		Cubic zirconia	Tetragonal zirconia	
Before	Control	1059.73 ±42.04	1096.20 ±52.85	0.262ns
	Laser	1154.79 ± 102.16	1379.40 ± 100.76	0.008*
After	Control	869.27 ± 17.65	1222.85 ±67.01	<0.001*
	Laser	1050.66 ± 103.28	1322.33 ± 71.58	0.001*

**; significant (p ≤ 0.05) .*

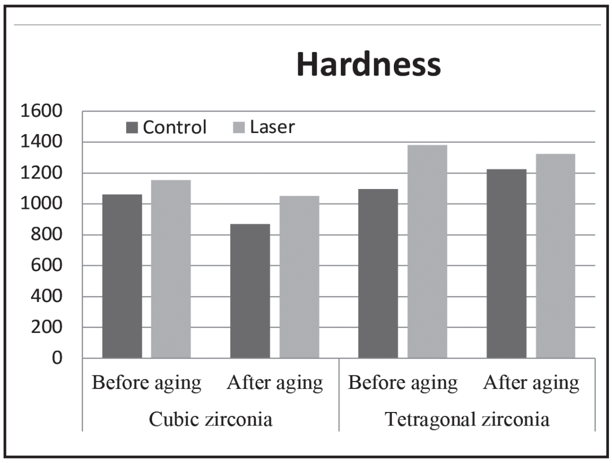

Figure (6) Bar chart showing mean of surface hardness (Kg/mm 2) for tested materials and surface treatment before and after (LTD) aging.

DISCUSSION

Zirconia-based restorations are commenly used prosthetic. However; their susceptibility to degradation remains abstract. It was postulated that CAD/CAM machining and subsequent surface treatments have great effect on the hydrothermal degradation behavior of Y-TZP[25].

In the present study, two brands of CAD/CAM zirconia ceramics material, tetragonal versus the newly introduced cubic zirconia (Cube X²). The cubeX2 system is based on a 5 % yttria oxide, leads to a stabilization of approximately 53% cubic and 47% tetragonal crystal structure and improved their properities with this new zirconia brand, so less transformation is observed .

Sixty discs (10 mm diameter x1.2 mm thickness) were cut from cylinders which were milled from zirconia blanks; these dimensions were chosen In order to standardize the dimension with biaxial flexural test according to ISO:6872[26]. Sintering of the specimens was done according to the manufacturers recommendation at high temperature furnace at 1450°C according to ISO 13356:2015 [27] , to generate dense structures, as dense material prevent penetration of the water to bulk of the material.. However, increasing the sintering temperature above 1550ºC, lead to formation of large grain, pores and surface cracks and water penetertation, hence,be more susceptable to LTD[28]. Finishing and polishing of the specimens was done in order to simulate the real state for the clinical situation, followed by ultrasonic cleaning and dryness of the specimens to remove any manipulative contamination that may affect the the results.

Laser has been used for different purposes in the dentistry among which roughning the bonding surface of ceramics through the process of thermomechanical ablation, which increases micromechanical retention, thereby enhancing the bond strength at thecement/ zirconia interface. However, laser is not yet commonly used as a way to modify the external surface of zirconia ceramics. Laser gives sufficient energy hence cause significant change in the surface morphology [28]. Several types of laser were used in dentistry; types as CO_2 ,Diode,Nd: YAG , and others.In the present study, diode laser was chosen as it is a broadband solid active medium laser, manufactured from semiconductor crystals using with wide range wavelengths from 700 nm to 980 nm compared to other types. In order to determine the most suitable parameters for the diode laser used in this study, a pilot study was done. It was found that 5 watt for 10 sec at 3 cm target distance gave the desired effect. Continuous mode was chosen over pulsed mode to allow broad coverage of the specimens with the laser beam which could not be achieved if pulsed mode was used.

Low thermal degradation (LTD) aging test of the zirconia specimens was induced by protocol for 5 hours as it simulate 15 year clinical conditions. This approach was in accordance with a previous study [29] who considered the treatment of zirconia specimens in the autoclave for 1 hour at 134ºc to be equivalent to 3-4 years of in-vivo aging. Moreover, other study reported that 5 hours aging at 134ºc corresponds to 15-20 years at 37ºC [30].

XRD was used in this study in order to identify and quantify the phase transformation (t-m) or any change in the crystalline sttrucure before and after LTD[31].

Several methods were used for surface topography analysis, such as scanning electron microscopy (SEM), atomic force microscope (AFM), and Profilometer. In this in- vitro study, change in the surface morphology of the tested zirconia ceramics was examined using scanning electron microscope. The atomic force microscope was used to measure the surface roughness and provides topographic 3D images of the tested zirconia ceramics. It is a powerful tool that is valuable for measuring incredibly small samples with a great degree of accuracy. Unlike other technologies, it does not require either vacuum or sample treatment that might cause damage [32].

Biaxial flexural strength was tested using piston on three balls and micro-indentation hardness testing was direct techniques and effectively to measure the mechanical properties of hydrothermally aged zirconia .

In the present study, XRD pattern after low thermal degradation (LTD) at134ºC in water steam resulted in a t–m phase transformation of 3% and 7% for the control group of the cubic and tetragonal zirconia respectively (figure 1). This could be attributed to the difference in their composition; as cubic zirconia contain high percent of the cubic phase (53%) which is a stable phase that does not easily transformed as the tetragonal phase. This result was also in accordance with other study [33] which stated that the coexistance of cubic with the tetragonal grains in cubic zirconia ceramics increased the resistance to LTD. Moreover, This might be due to the difference in the yttria content between the two tested zirconia ceramic materials, as the cubic zirconia contains 5% yttria, while the tetragonal one contains only 3% which plays a role in decreasing their stability when subjected to LTD[34]. Other important point to focus on is that;

Transformation of zirconia preceded from grain to grain and the growth continue, the presence of small amount of oxides in the zirconia matrix can reduce this transformation by reducing the contact area between grains. the presence of the alumina in the zirconia matrix leading to increase the stiffness of the matrix and change in the elastic strain energy associated with the phase transformation which is consequently hinder [35] .

In the current study regarding the cubic zirconia, figures (2,4) show the morphological surface and topographic image for the control group specimens of the cubic zirconia .These images show a clear appearance of the crystalline structure. While after laser surface treatment of the their morphologic surface changed and show smooth surface with multiple burned areas embedded into a wide smooth melted region compared to the clear crystal structure of the control specimens as show in figures(2,4). The change in the surface morphology could be attributed to the modification of the surface profile as a function of the laser power.

In accordance with this research's results [36,37] others studied the effect of sandblast and lasers on bond shear strength of resin cement to zirconia, and reported that CO_2 laser reduces bond strength. Also reported that CO_2 laser treatment creates a smooth surface which means decrease in the surface roughness. But this conclusion depends on practical laser parameters. Since the main effect of laser energy is the transformation of light energy into heat, and the absorption of laser energy by material's surface is the most important interaction between material's surface and laser [38]. They concluded that this came from the low density of CO2 laser (159,22J/cm2),and a lower CO2 laser power (3W),

While after LTD figure (2,4) show the morphological surface and topographic image for the the laser treated group and the result show the presence of microcracks, irregular multiple holes on rough surface which indicate increase in the surface roughness as a function of LTD, because at low temperature transformation from tetragonal to monoclinic procedure begin to become rapid progress when temperature range between 125 and 150°C [39]. This mean that each grain is push out of the surface and the roughness of the surface increase due to low thermal degradation(LTD) aging .Moreover; in the laser treated group after LTD melting areas with some burned areas were notcied. This change of the surface may lead to degradation of mechanical property of conventional Y-TZP.

This result was support with the statistical analysis for surface roughness (Ra) as show in table (1,2) and figure (3).

Regarding cubic zirconia; mean values of surface roughness of control specimens after (LTD) aging showed statistically higher values than before (LTD) aging (25.44±0.38,24.90±0.47μm respectively), however, the difference was not significant (P=0.104). Meanwhile, mean values of surface roughness of laser specimens after (LTD) aging showed statistically significant higher values than before (LTD) aging 25.37±0.05 μm, 25.14±0.03) respectively, (P< 0.001).**Regarding tetragonal zirconia**; mean surface roughness values of the control specimens after (LTD) aging showed a lower value (25.45±0.36 μm) than before (LTD) aging (25.66±0.24 μm), but the difference was not significant (P=0.28). Meanwhile, laser treated specimens after (LTD) aging showed a significantly higher mean surface roughness value (25.48±0.23 μm) than before (LTD) aging (25.16±0.06 μm) (P=0.027).Furthermore, statistical results revealed that tetragonal zirconia showed higher mean surface roughness values for both control and laser treated specimens than cubic zirconia (P≤ 0.05).Moreover, laser treated specimens of cubic zirconia and tetragonal zirconia before and after (LTD) aging had a significantly higher mean surface roughness values than the control specimens (P≤ 0.05).

Our results were similar to a study [40] who concluded that the surface roughness of highly translucent zirconia increased after being subjected to hydrothermal aging at low temperature when aging procedure performed in an autoclave at 134 at 2 bars for 3hours.

This was in aggrement with other study who measure the surface topography by AFM and recorded that the thermally etched zirconia displayed smooth grains with no texture before LTD[41,42]

Few reports about hydrothermal degradation of dental Y-TZP on mechanical properties. They observed that there was no change in flexural strength for "Denzir" ceramic blocks after aging in 4% acetic acid at 80^{o}C /168 h [43]. Similar results were found for "InCeram YZ" ceramic bars. In this instance, a slight improvement in flexural strength was even reported after 7 days of aging in boiling water [44]

Regarding the biaxial flexural strength; Tables (3,4) and figure (5) show mean and standard deviation (SD) values of biaxial flexural strength (Mpa) for the tested zirconia (cubic and tetragonal) materials and surface treatment before and after (LTD) aging. Regarding cubic zirconia; mean biaxial flexural strength values of both control specimens and laser treated specimens after (LTD) aging showed statistically lower values (610.85± 2.56 Mpa, 655.57± 29.67 Mpa respectively) than before aging (621.04± 3.99 Mpa, 819.90± 33.15 Mpa respectively) (P≤ 0.05).Regarding tetragonal zirconia; mean biaxial flexural strength values of the control specimens after aging showed a lower not significant value (1210.25± 9.50 Mpa) than before (LTD) aging (1270.06±57.99 Mpa). Meanwhile, laser treated specimens after(LTD)aging showed a significantly higher mean biaxial flexural strength value (1283.93± 46.73 Mpa) than before (LTD) aging (1408.70± 64.85 Mpa) (P=0.001).

This result was in aggreement with another study [45] which found that there was significant reduction in biaxial flexural strength between aged in nonaged specimens, but there was no statistically significant difference between the two brands of zirconia regarding change in the flexural strength.

Furthermore, generally, statistical results revealed that tetragonal zirconia showed statistically higher mean biaxial flexural strength values for both control and laser treated specimens than cubic zirconia (P≤ 0.05). Also the results of the current study show that tetragonal zirconia showed a significantly higher biaxial flexural strength value (1293.24±86.93 Mpa) than cubic zirconia (676.84±88.84 Mpa) (P<0.001).

This was in accordance with other study who previously revealed that cubic zirconia had lowest biaxial flexural strength than tetragonal zirconia and was affected by hydrothermal degradation [46]. At the same time; composition could be reason for this difference. Also this behavior difference could be related to compositional difference, microstructure and flaw distribution of the tested zirconia ceramics[47].

Also in accordance with other studies [48] who stated that cubic zirconia had the lowest biaxial flexural strength in comparison with tetragonal after hydrothermal aging.Moreover, laser treated specimens of cubic zirconia and tetragonal zirconia before and after (LTD) aging had a significantly higher mean biaxial flexural strength values than the control specimens (P≤ 0.05).

Regarding the surface hardness;Tables (5,6) and figure (6) show mean and standard deviation (SD) values of surface hardness for the tested zirconia (cubic and tetragonal) materials and surface treatment before and after (LTD) aging. Regarding cubic zirconia; mean surface hardness values of both control specimens and laser treated specimens after (LTD) aging showed statistically lower values (869.27± 17.65Kg/mm^2, 1050.66± 103.2865Kg/mm^2 respectively) (P=0.005)than before (LTD) aging (1059.73±42.0465Kg/mm^2, 1154.79± 102.16Kg/mm^2), but the difference was not significant (P= 0.09). Regarding tetragonal zirconia; mean surface hardness values of the control specimens after (LTD) aging showed a lower value

(1096.20±52.85Kg/mm^2) than before (LTD) aging (1222.85±67.01 Kg/mm^2), but the difference was not significant (P=0.059). Meanwhile, laser treated specimens after (LTD) aging showed a lower surface hardness value (1322.33±71.58Kg/mm^2) than before (LTD) aging (1379.40±100.76Kg/mm^2), with insignificant difference (P=0.264).

CONCLUSIONS

Under the limitation of the present it could concluded that:

1. Laser surface treatment improve the mechanical properities of the CAD/CAM zirconia ceramics, and increase their resistance to LTD.
2. LTD reducing the properities of the zirconia ceramics materials.
3. Tetragonal zirconia posses superior properis than the cubic one.

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