The impact of laser scanning on zirconia coating and shear bond strength using veneer ceramic material

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Laser scanning is one of the methods that can be used for surface treatments of zirconia. Application of the laser to the surface of zirconia has diverse effects, depending on the type of laser. A carbon dioxide (CO₂) laser has high irradiation power and can alter the surface properties. This study investigated the surface coating of zirconia as a core material that subsequently coated with a veneering ceramic (v-c) material. This study compared laser scanning and conventional sintering processes. Various properties including surface topography, interface evaluation, phase transformation, elemental compositions, failure mode patterns, and contact angle were examined through X-ray diffractometry (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analyses. Results were confirmed that the bond strength between the v-c and the substrate recorded through laser scanning was higher than that determined through conventional sintering.

Keywords: Laser scanning, Sintering process, Surface treatment, Shear test, Contact angle

INTRODUCTION

The increasing demand for aesthetics and the advancements in technology have contributed to the rise in popularity of all-ceramic restorations. In the last two decades, the continuous progression in the ceramic materials science for dental applications has permitted the fabrication of high-strength materials. Zirconia-based ceramics have improved in terms of fracture resistance and long-term viability in comparison with other silica-based materials¹. These properties have promoted the utilization of zirconia in dental fields such as in fixed partial dentures (FPDs)^{2,3}.

Zirconia exhibits superior mechanical properties and is the preferred substructure material for single and multi-unit FPDs⁴. The material occurs in three forms, namely, monoclinic, cubic, and tetragonal. A monoclinic pure zirconia is present at room temperature and is stable up to 1,170°C. Above this temperature, it transforms into other phases such as cubic and tetragonal phases at 2,370°C. Pure zirconia can be alloyed with stabilizing oxides⁵.

For applications as a substructure, zirconia has been improved in terms of mechanical properties to withstand mastication forces¹. In addition, zirconia cores for crown frameworks still require application of veneering ceramics, typically with specialized porcelain, to achieve optimum aesthetics¹. Certain studies have reported the clinical failures of veneered zirconia FPDs due to veneer debonding, chipping, or fracture defects which were reported to be around 15% after 2–5

years^{1,5–7}. The adhesion between the zirconia substrate and veneer ceramics (v-c) is based on various factors such as chemical bonding, mechanical interlocking, wetting properties, interfacial stress, thermal expansion mismatch and glass transition temperature^{1,8}.

Insufficient consensus has been established with regard to efficient surface treatment method for achieving optimum bond strength between zirconia cores and v-c. The quality and bond strength between the core and v-c require enhancement to optimize successful bi-layered restorations⁹.

Diverse approaches for zirconia surface treatment have been studied; such approaches include laser techniques, airborne-particle abrasion, grinding, heat treatment, acid etching and plasma, which are used alone or in combination. The commonly used treatment is airborne with different sized aluminum oxide particles, with or without a layer of silica coating¹⁰.

Certain methods, such as airborne particle abrasion and liner application, exert a positive impact on surface treatments of zirconia, leading to an increase in the bond strength¹¹. However, airborne particle abrasion causes phase transition on the surface by altering its phase from tetragonal to monoclinic¹². In addition, this process may produce defects such as surface flaws which may cause detrimental impact on the core material by reducing the survival rate of FPDs¹³.

Further investigations are required to evaluate the bond strength between zirconia substructure and v-c material. The enhancement of the adhesion and the success rate of FPDs are pivotal to the development of adequate surface treatment of materials^{8,14}.

At present, researchers have focused on laser

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techniques with sophisticated methods to achieve optimum properties on the treated substrates¹⁰. Laser applications using CO₂, YAG, excimer, dye, argon-ion and diode lasers have also been studied^{15,16}. The use of laser energy on zirconia surface treatment has gained increasing research interest¹⁰. However, limited studies have investigated the application of laser scanning technology to zirconia. Furthermore, the impact of laser treatment between v-c and the zirconia substrate remains questionable.

Therefore, the aim of the study was to investigate the impact of CO₂ laser scanning on a densely sintered zirconia for surface coating and compare the process with a conventional sintering. The study hypothesized that the laser scanning can demonstrate superior bonding strength at the v-c interface than the counterpart sintering process.

MATERIALS AND METHODS

Material composition

The standard compositions of Zenostar®T zirconia substrate and IPS e.max Ceram materials are summarized in Table 1.

Preparation of zirconia specimen

Eighty Zenostar®T Y-TZP (Wieland Dental, Technik, Ivoclar Vivadent, Pforzheim, Germany) cubic-shape specimens were prepared by milling with CoriTec-245i CAD/CAM machine (imes-icore®in CNC & Dental Solutions, Eiterfeld, Germany).

All the specimens were densely sintered in a furnace (Austromat®, Dekema, Dental Keramiköfen, Freilassing, Germany) according to manufacturer's instruction. The dimensional change that occurred was approximately (20 vol%) in each specimen, and the size of each specimen was finally set as 10 mm in length; width; and height, respectively. The surface of the cubic specimens was polished using the Zenostar®T polishing set (Wieland Dental, Technik, Ivoclar Vivadent). All specimens were ultrasonically cleaned in an ethanol solution using a digital ultrasonic cleaner (Jeken®, PS-20A, Shenzhen, Guangdong, China) for 10 min and dried using an electric heat blast oven (Model DGG-9240B, Nanjing, China) for 5 min prior to subjecting specimens to surface coating.

Naming of experimental groups

All the specimens were divided into two main experimental groups named laser scanning and conventional sintering as following:

Group L (laser scanning group $n=40$): laser scanning was conducted to coat zirconia surface after adjusting CO₂ laser machine's software using carbon dioxide laser machine (Julong laser machine JL-K6040, Laser engraving, Shandong, China), the summary of the laser parameters is shown in Table 2.

Group C (Conventional sintering group $n=40$): No special treatment was carried out to modify the zirconia core surfaces. The surfaces were polished with Zenostar®T polishing set after densely sintering through the furnace (Programat P310, Ivoclar

Table 1 Chemical compositions of Zenostar®T and IPS e.max Ceram v-c materials

Materials	Chemical composition (%)	Manufacturer name
Zenostar®T	Zirconium oxide (ZrO ₂ , HfO ₂ , Y ₂ O ₃) ≥99.0, Yttrium oxide (Y ₂ O ₃) 4.5≤6.0, Hafnium oxide (HfO ₂)≤5.0, Aluminum oxide and other oxides≤1.0	Wieland Dental, Technik, Ivoclar Vivadent, Pforzheim, Germany
IPS e.max Ceram veneer-ceramic, material	SiO ₂ 60–65, Al ₂ O ₃ 8–12, Na ₂ O 6–9, K ₂ O 6–8, ZnO 2–3, CaO; P ₂ O ₅ ; F 2–6, other oxides 2–8.5, and pigments 0.1–1.5	FL-9494, Ivoclar Vivadent, Schaan, Liechtenstein

Table 2 Typical laser scanning parameters and laser properties for v-c on the substrate

Laser parameters	Value	Laser properties	Value
Scanning speed	30 mm s ⁻¹	Laser type	CO ₂ laser
Output power	25 Watt	Wavelength	10.6 μm
Distance between laser tube and the substrate	20 mm	Frequency	50 Hz
Space between scanned lines	0.25 mm	Laser tube diameter	8 mm
Scanning duration	70 s	Spot size	0.2 mm
		Intensity	0–1.6×10 ⁶ W/cm ²
		Pulse length	10–50 μs
		Pulse energy	150 W
		Feed speed	0–300 mm s ⁻¹

Vivadent, Schaan, Liechtenstein, Germany).

Dividing specimens according to the corresponding groups

In each group ($n=40$), based on randomization method, 10 samples were selected to evaluate the coated v-c layer on the top surface ($n=5$) and at the interface ($n=5$) of the core material. From the rest of 30 samples, 20 samples were subjected to shear bond strength (SBS) test ($n=20$). The specimens which were assigned for shear test were further subdivided; 10 specimens were stored in distilled water (DW) ($n=10$) for 2 weeks, the rest of the samples were subjected to the shear test without storage condition ($n=10$). Ten specimens were also subjected to contact angle measurement ($n=10$). The experimental route is summarized in Fig. 1.

Airbrush spraying technique

To achieve a uniform thin layer of the v-c material on the core material, each sample was subjected to spraying technique prior to conduct laser scanning and conventional sintering. The surface of the zirconia specimens 10×10 mm was coated with IPS e.max Ceram Type I, Class II Dentin C2/TI3 material v-c material (FL-9494, Ivoclar Vivadent).

The v-c powder was weighed using digitalize analytical balance (Sartorius Entris Analytical Balance, Göttingen, Germany) and mixed with the corresponding liquid following the manufacturer's direction. A mini magnetic stirrer (SH-II-2, Huanghua Faithful Instrument, Cangzhou, China) was used to achieve uniform mixture. The spraying process at a fixed perpendicular distance 15 cm from the substrate surface

Fig. 1 Flow chart illustrates the summary of the experimental route.

Table 3 Coating parameters for airbrush spraying technique

Coating parameters	Values
Mixing amount of veneer-ceramic and liquid for each sample	1 g powder, 1.5 mL liquid
Mixing speed and duration via mini magnetic stirrer	300 *rpm/min for 10 min
Distance between zirconia top surface and airbrush nozzle tip	15 cm
Nozzle diameter	0.2 mm
Working pressure	37 **psi (2.5 bar)
Spray time	10 s
Dryness time in ambient room temperature	5 min

*rpm: rounds per minute, **psi: pounds per square inch

was performed using a mini airbrush spray gun (Dual action airbrush, Sheng, Model HS-35, Zhejiang, China). The samples were placed on a flat surface to achieve a uniform coated layer of v-c on the substructure. The protocol of coating parameters and airbrush spraying are summarized in Table 3.

Laser scanning and conventional sintering processes

To perform laser scanning, the specimens ($n=40$) were transferred to the flat X-Y laser table after deposition of a thin v-c layer through spraying technique. Each sample was subjected to laser scanning to permit formation of a v-c layer on the core material. The laser scanning parameters were set using CorelLaser 2013.02 and CorelDraw Suite X4 SP2 software program to scan the airbrush sprayed 10×10 mm surface area. After completing the process, the specimens were left to cool down slowly.

The similar process of airbrush spraying was followed as mentioned earlier for specimens which were assigned to be treated through sintering. After the samples were airbrushed, they were transferred to the furnace (Programat P310, Ivoclar Vivadent) and sintered as per the manufacturer's instructions. The specimens were left to cool down to normal room temperature.

Analysis of surface roughness

The surface roughness of the specimens for each group were analyzed with a profilometer (Surtronic 25, Taylor Hobson, Leicester, UK). R_a which represents arithmetical mean roughness was determined to indicate of the surface roughness of the substructure. For each new specimen, recalibration was conducted using a standard sample ($6 \mu\text{m}$) that was provided by the manufacturer. The cut off length ($0.03 \text{ inch}=0.8 \text{ mm}$) was determined according to recommended ISO 4288-1996. For each group, randomly selected samples ($n=10$) were subjected to surface roughness analysis on a flat surface. For each sample, four readings of roughness were recorded and the mean value was calculated.

X-ray diffractometry (XRD) examination

Phase ingredients were identified using SmartLab X-ray diffractometer (Rigaku SmartLab, Rigaku, Tokyo,

Japan) with CuK α radiation at 200 mA and 40 kV. Diffraction data were collected within the 2θ range of 20° – 90° at a step size of 0.02° and step time of 8 min. The peaks were read using (MDI Jade v6.0, Livermore, CA, USA) software program to identify the phases. The peaks' patterns were drawn using the OriginPro 2016 (OriginLab v.93E, Northampton, MA, USA) software program. The patterns were plotted by a set of line positions 2θ (deg) against intensity (arbitrary units).

Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) examination

The coated core material of each group ($n=10$) was investigated after random selection. Samples in each group were further subdivided to evaluate surface topography ($n=5$) and interface ($n=5$) through SEM (Ultra plus ZEISS, Oberkochen, Germany) equipped with EDS.

The specimens to be examined at interface were sliced to the desired shape by using a controlled-speed diamond saw machine (Sherline 5410, Sherline products, Vista, CA, USA) on a slow speed mode with water coolant. The interface of the specimens was polished with SiC grit sizes #600 and #800 on a flat surface under running water.

The samples were gold-sputtered using a sputter coater machine (JS-1600, Beijing HTCY, Beijing, China). The specimens were then examined in different magnifications through SEM. The spot and line EDS spectra were then recorded and analyzed.

Procedure of veneering

Randomly selected specimens for each group ($n=10$) were ultrasonically cleaned in an ethanol solution for 10 min and dried using an electric heat blast oven. A silicon mold was fabricated from a dimensionally stable polyvinyl siloxane impression material, which was used as a standard guide for preparing the v-c cylindrical shape. The fabrication process of v-c cylinder is illustrated in Fig. 2a. The IPS e.max Ceram powder and the corresponding liquid were mixed in the recommended ratios following the manufacturer's instructions. The mixture of v-c slurry was condensed into the silicon mold through a pre-made cylindrical shape hole (5 mm in

Fig. 2 Schematic of veneer ceramic (v-c) preparation on zirconia core substrate. The final dimension of the v-c cylinder is 5 mm in diameter and 3 mm in thickness (a, b). Seated specimen in electromechanical universal testing machine (c). Separated v-c cylinder from the core material following shear test (d).

Fig. 3 Illustrates sessile drop test and dot plot graph for tested specimens; pre-surface treatment of zirconia specimen showed an average contact angle value (ACAV) around 75.25° (a), the core material specimens after coating with v-c by sintering and laser scanning showed ACAV around 37.95° and 26.25° , respectively (b, c). The dot plot graph shows the outcome correlation between contact angle θ and shear bond strength SBS of specimens subjected to θ measurement; $r=-0.450$, $p<0.05$ (d).

diameter and 3 mm in thickness) to build up a cylindrical shape of v-c at the center of the coated surface area on the core material. A lubricating agent (Marbocote®220, Mühlmeier, Bärnau, Germany) was used around the prefabricated hole of the silicon mold to overcome the adhesion between the v-c slurry and the custom-made silicon mold.

After gentle vibration, excess liquid was removed using tissue paper. The firing protocol for v-c material was followed in the furnace as per the manufacturer's instruction (Programat P310). A second layer of the v-c slurry was applied to compensate the shrinkage of the v-c material after the first firing cycle.

Contact angle measurement

The contact angle θ values of the zirconia specimens ($n=10$ in each group) were recorded (Figs. 3a–c). The sessile drop method (Drop Shape Analyzer DSA25, KRÜSS, Hamburg, Germany) was used to measure the θ . The hydrophilicity of specimens was evaluated by measuring θ values between the solid and the liquid. A time frame of 30 s for each measurement was recorded and evaluated. Two measurements (right and left contact angles) were carried out for each droplet. Contact angles were determined at normal laboratory atmospheric environment, and photographs were taken after droplets impacted on the zirconia surface in a most stable and measurable state. The volume of each water drop was 1 μL . The procedure was performed by the same trained operator.

SBS test procedure

In each group ($n=20$) randomly selected samples were subjected to the above mentioned test. The specimens were then equally subdivided: the specimens ($n=10$) that were not stored in DW and the specimens ($n=10$) that were immersed in DW at 37°C for 2 weeks. The samples were secured on an electromechanical universal testing machine (E44.304, MTS Systems, Shenzhen, China) with a special holder to conduct the test. A semicircular metal jig was positioned close to the interface between the v-c and the substrate at a cross head speed of 1 mm/

min until failure of each specimen was occurred (Figs. 2b–d). The measured force was recorded in newton, and the bond strength for each sample was calculated by dividing the peak load values force in newton by the surface area (mm^2) to record the strength in MPa. The recorded load was calculated based on the following formula $b=f/s$ where b , f and s represent bonding, force and surface area, respectively.

Fracture mode examination

The fracture modes of the specimens were examined visually. The outcomes were reconfirmed through SEM equipped with EDS to support the fracture mode patterns. The failure modes were defined as adhesive failure (separation of v-c and the substrate), cohesive failure (separation through the v-c material) and a mixed mode of failure (combination of the cohesive and the mixed modes). The fracture mode patterns for each tested group were recorded in percentage.

A typical cohesive fracture mode for the laser group and a mixed type for the conventional sintering were imported into the OriginPro 2016 software program and analyzed in 3D format to confirm and distinguish between fracture modes.

Statistical analysis

The final results were statistically analyzed using an IBM Statistical Package for Social Sciences software (SPSS v.20.0, IBM®SPSS®, Chicago, IL, USA). Independent t -test was performed to compare the means of groups for surface roughness, shear test, and contact angle values.

ANOVA repeated measure was used to determine the outcomes of tests between subjects-effects, the mean of treatment methods and the mean of aging condition (before and after storage of specimens in DW) were recorded. Pearson test was used to analyze correlations (r) between R_a and aging condition, contact angle and shear test. The fracture modes were recorded in percentages, chi-square test was used to compare means of fractured patterns. The statistical significance was set at $\alpha=0.05$.

RESULTS

Table 4 shows the outcomes of surface roughness R_a , shear test, contact angle θ values, and fracture mode patterns. The mean and standard deviation (M $\pm$ SD) of R_a for the laser group 'GL' was $1.087\pm0.143\ \mu\text{m}$, which was higher than that in the conventional sintering process 'GC' $0.715\pm0.117\ \mu\text{m}$. R_a values showed statistically significant difference between the two groups ($p<0.05$).

GL showed low θ $24.39\pm1.09^\circ$, whereas the sintering technique showed θ of $36.44\pm1.38^\circ$. Independent sample t -test showed a statistically significant difference of θ between the two groups ($p<0.05$). Pearson test demonstrated a negative significant correlation between contact angle and SBS test (Fig. 3d). Specimens which showed low contact angle provided higher SBS values.

The shear test values before and after storage of specimens in distilled water (DW) showed a statistically significant difference between the two groups ($p<0.05$). The laser scanned specimens recorded higher values of shear test before storage in DW $33.2\pm4.184\ \text{MPa}$; however, the lowest value of $26.242\pm3.97\ \text{MPa}$ was found in the GC group after storage of specimens in DW.

The fracture mode patterns of the specimens were recorded in percentages. The highest percentage of adhesive fracture mode was recorded in GC (70%), whereas the lowest value was detected in GL (5%). Interestingly, the highest failure mode in GL was a mixed pattern (55%). A cohesive type of failure mode

(40%) was recorded in GL, whereas the percentage of that mode was 0% in the GC group. The chi-square test showed a statistically significant difference in failure mode patterns between the GL and GC groups ($p<0.05$).

ANOVA repeated measure of variance demonstrated that the effect of treatment methods (GL and GC) on aging condition was not statistically significant ($p>0.05$, Table 5). Pearson test did not find correlation between surface roughness and aging condition, the outcome was not statistically significant ($p>0.05$).

XRD analyses did not show phase transformation, and only the tetragonal (t) phase existed on the zirconia surface after coating the substructure and after performing the shear test through both methods (Figs. 4a–c). The major peak of t phase was detected at 30.170° which corresponds to the crystallographic plane pdf #48-0224. The other t phases were also found at 34.742° , 35.278° , and 50.212° , which correspond to different crystallographic planes pdf #25-0757, pdf #14-0534, and pdf #82-1243, respectively.

SEM examination of the top surface and interface of the substrate after being coated through both techniques presented different outcomes. In GL, the SEM showed irregularities with certain micro holes within the v-c material following laser scanning as shown in Fig. 5a. The interface between the v-c layer and zirconia showed proper adhesion in such a way that it could not be distinguished the line fusion between the materials as shown in Fig. 5b. The coated v-c layer showed homogenous thickness $72.2\text{--}99.4\ \mu\text{m}$ following the laser

Table 4 Summarized outcomes of surface roughness, contact angle, shear test values, and fracture mode patterns of studied groups

Groups	R_a after coating (μm) (Mean $\pm$ SD)	Contact angle (deg.) (Mean $\pm$ SD)	Shear test values (MPa) (Mean $\pm$ SD)		Fracture mode pattern (%)		
			Specimens before storage in DW	Specimens after storage in DW	Adhesive	Cohesive	Mixed
GL	1.087 ± 0.143^a	24.39 ± 1.09^a	33.2 ± 4.184^a	31.7 ± 3.56^a	5	40	55
GC	0.715 ± 0.117^b	36.44 ± 1.38^b	28.48 ± 4.435^b	26.242 ± 3.97^b	70	0	30

Values marked with different letters indicate for statistically significant difference between studied groups ($p<0.05$).

GL; Laser scanning group, GC; Conventional sintering group, R_a ; surface roughness, μm ; micron, SD; standard deviation, deg.; degree, MPa; Mega Pascal, DW; distilled water, %; percentage.

Table 5 ANOVA procedure, repeated measures of variance, and tests of hypothesis between subjects-effects

Source	ANOVA sum of square	df	Mean square	F	Sig.
Treatment	258.9792100	1	258.9792100	9.21	0.0071*
Aging	34.39161000	1	34.39161000	7.44	0.0138*
Treatment $\times$ Aging	1.3616000	1	1.3616000	0.29	0.5969

Treatment; the treatment methods so called laser scanning and conventional sintering. Aging; the outcome values of shear test before and after storage of specimens in distilled water for 2 weeks. Treatment $\times$ aging: The effect of each treatment method "laser and sintering" on aging condition.

*The superscript asterix indicates for statistically significant results $p<0.05$.

Fig. 4 XRD shows crystallographic peaks without phase transformation.
The sole zirconia surface without coating by veneer ceramic v-c material (a), the core material specimens coated with v-c followed laser scanning and sintering, respectively (b, c).

scanning process (Fig. 5c).

In GC, (Fig. 5d) shows micro holes similar to laser scanning, however, the line fusion between the v-c and the core material was easily observed. Along the mentioned line, micro gaps at the interface level can be distinguished (Fig. 5e). The coated v-c layer was thicker than the laser-treated specimens, and the thickness ranged from 114.2–128.8 μm following sintering (Fig. 5f).

The results of line EDS analyses after the substrate was coated through both methods and prior to conducting SBS test are demonstrated in (Figs. 6a, b). Si, Al and zirconia were the most prominent components found during the EDS analyses. The first two elements were the main components of the v-c, and the latter was the main component of the substrate material. Similar outcome of line EDS analyses was recorded after performing shear test followed laser and sintering (Figs. 6c, d).

Figures 7a–h show the SEM images (a–c, e–g) and 3D (d, h) evaluations of the interface and fracture surface pattern of the specimens that treated through both techniques after shear testing. Figures 7a, e show the interface of the treated specimens following laser scanning and sintering, respectively. At the interface, certain features were detected such as micro cracks and tiny micro gaps between the v-c and the core material in the specimens treated by laser scanning (Fig. 7a). The larger non-uniform pores within the v-c layer and larger gaps between the core and the v-c were observed at the interface in specimens treated through sintering (Fig. 7e).

The fracture mode patterns were observed after performing shear test for specimens treated through mentioned techniques. Figures 7b, c (SEM) show the

Fig. 5 SEM images showing the top surfaces and interface after coating the specimens with veneer ceramic (v-c) material through laser scanning and sintering. The white arrows indicate micro retention means on the top surface followed the mentioned techniques, respectively (a, d); the black arrows indicate line fusion at the interface between the v-c and the core material; the white arrows indicate certain micro gaps within the v-c material followed the mentioned techniques, respectively (b, e); the thickness of the coated v-c layer measured at the interface level followed the mentioned techniques, respectively (c, f).

Fig. 6 EDS line analyses at the interface for determining elemental compositions. EDS line analysis before performing shear test followed laser scanning (a), and conventional sintering (b). EDS line analysis after performing shear test followed the mentioned techniques, respectively (c, d).

Fig. 7 SEM images illustrating the interface and fracture patterns on the top surfaces after performing shear test.

The interface subjected to laser scanning and sintering processes, respectively (a, e); the surface fracture pattern shows cohesive type of failure through SEM analysis (b, c). 3D image shows different color scales; representing the v-c material (green) and the core material in mixed colors (yellow and red) followed laser scanning (d). The interface of a specimen after subjecting to conventional sintering (e). The surface fracture pattern shows mixed type of failure, as determined through SEM analysis (f, g); the 3D image demonstrates colored scale (h) followed conventional sintering.

typical cohesive type of failure for the specimens treated by laser scanning, the fracture line was irregular through the v-c layer. A typical mixed type of failure for the specimens which treated by conventional sintering presented in Figs. 7f, g. The 3D view of the fractured

pattern illustrated in Figs. 7d, h; the software program provided different colors, such as green representing the v-c material, and the mixed yellow-red color representing the core material.

Figures 8a–d show the spot EDS analyses for

Fig. 8 Spot EDS analyses showing the collected spectra of the fractured surfaces after performing shear test.

For cohesive failure pattern, the predominant peak represents Si after laser scanning (a, b). For mixed failure pattern, the prominent peak shows zirconia in the delaminated area (c), and Si was detected in the coated area by v-c material followed conventional sintering (d).

mentioned types of failures. The collected spectra showed Si as a prominent peak which was detected during analysis of the cohesive type of failure for laser group (Figs. 8a, b). The spot EDS analyses for sintering group showed mixed type of failure, Si was observed as a prominent peak on the core material after shear testing, whereas zirconia was detected as a predominant peak in the delaminated area (Figs. 8c, d).

DISCUSSION

The principal approach for surface coating of zirconia is to increase the surface roughness at the microscopic level to promote high surface energy and achieve satisfactory wetting property^{17,18}.

The enhancement of bonding between zirconia and v-c is a prerequisite for improving the success rate and longevity of FPD restorations^{1,8,19}. The recorded failure of zirconia in FPDs often occurs due to insufficient bond strength between the v-c and the core material which leads to delamination of the v-c layer¹⁹.

Key factors, such as adhesion and mechanical integrity, play a major role in the successful performance of v-c in FPD. The initial bond strength achieved from the *in vitro* investigations can provide useful information for the behavior and predictability of zirconia all-ceramic crowns to be used in the oral environment²⁰.

Various factors affect the integrity of the v-c interface; such factors include wetting property, surface roughness, thermal mismatch, presence of defects and the chemical interactions of the bonded materials²¹.

Laser has been used for certain decades in the dental

field and surface treatment²². For hard tooth tissues, the tissues turn into steam instantly, and the volume expansion leads to the micro explosion after absorption of the laser energy, which removes the ambient tissues and provide a rough surface²³. Nevertheless, in zirconia specimens water does not exist similar to that in the oral environment. The impact of laser scanning on zirconia surface may lead to rehardening of the surface material due to extensive absorption of laser energy²⁴. Such changes in the zirconia surface topography will ultimately result in different levels of surface roughness.

The effect of laser scanning on the coating zirconia substrate by v-c has been rarely reported. Laser systems used in previous studies were mostly designed for clinical approach, in which the maximum output energy of these systems is relatively lower than that in the present study.

High levels of output energy were reported to produce noticeable influences on the zirconia surface. Low output energy leads to less detrimental impacts and has been suggested as a method worthy of further investigation²⁵. In addition, various kinds of laser beams may not have the similar effect on zirconia bonding properties due to the variations in laser energy adsorption, *e.g.*, CO₂²⁶. Previous studies reported that an Nd:YAG laser system pulsed mode caused lower bond strength values, whereas the Er:YAG laser system pulsed mode and CO₂ laser continuous mode showed higher bond strengths in zirconia bonding²⁷. The present results are in agreement with previous studies, hence, using modified CO₂ laser parameters (continuous mode) with higher output energy provided sufficient bond strength between the

v-c and the core material²⁸⁾.

In this study, the surface roughness was promoted by adsorption of laser energy and resulted in the remodelling of the zirconia surfaces. Although conventional sintering and CO₂ laser scanning provided satisfactory surface roughness of zirconia specimens, the roughness value was higher in the laser group. This finding may be attributed to the effect of energy transmission and adsorption property. Previous studies recorded controversies about the relation of surface roughness to increase the bond strength. The current research found no correlation between *Ra* and aging condition. The current result is consistent with the previous literature²⁹⁾, however, it is inconsistent with an earlier investigation³⁰⁾.

In this study, the laser beam subjected to the core material provided certain micro holes within the v-c after coating. These holes might act as micro mechanical retentive means to strengthen the integration between the v-c and the substructure for the specimens treated through laser scanning. This finding might be due to the high output laser power during scanning process.

XRD analysis showed that sintering and laser scanning did not cause crystallographic changes in the substrate. The stability of zirconia after laser scanning and sintering might be due to the steps followed in preparing the specimens (as mentioned in the MATERIALS AND METHODS section). This property of zirconia provided satisfactory SBS values without showing phase transformation from t-m. However, other studies have reported phase transformation from t-m during grinding and sandblasting^{31,32)}. The conflicting results may be contributed to the different methodologies followed by the present and previous studies. Therefore, the stability of the crystalline structure of zirconia is a promising indicator for preserving the structural integrity of zirconia as a core material that can be treated through laser technique.

After shear testing, certain micro cracks were detected at the interface in both groups. This finding may be due to the fact that the core material was subjected to repeated firing cycles to fabricate the cylindrical v-c layer on the substructure. After performing the shear test, the core surface material might have been subjected to an extreme internal pressure, which resulted in the formation of micro cracks at interface.

In this study, the laser specimens showed mixed and cohesive types of failure, while sintering specimens predominantly showed an adhesive failure pattern. The cohesive fracture is the most desirable bond failure, which indicates sufficient bond strength between v-c and substructure^{8,33)}. The existence of this type of failure might be attributed to changes in physical properties, such as fusion between the v-c and the substrate at the interface.

An adhesive fracture, which was the most frequent failure among fractured surfaces in conventional sintering, might due to the insufficient bond strength of v-c to the substrate. The finding might be due to the mismatch in the coefficient of thermal expansion (CTE) between the materials. Previous studies reported

that the thermal mismatch of less than $1 \times 10^{-6} \text{ K}^{-1}$ was estimated to be compatible and did not lead to crack formation during sintering³⁴⁾. In the current study, the CTE values of the v-c and the core material according to the manufacturer's data are $9.5 \pm 0.25 \times 10^{-6} \text{ K}^{-1}$ and $10.5 \pm 0.5 \times 10^{-6} \text{ K}^{-1}$, respectively.

EDS analyses revealed different elemental composition (mainly Si and Al) after coating and shear test. Silica was predominant during the analysis of the fractured surfaces and interfacial areas. As an important factor, differences in composition and microstructure of the v-c material and the substructure might result in achieving sufficient bond strength. The micromechanical interaction and chemical bonding of the specimens might increase the bond strength through an interlocking mechanism.

In the current study, the wetting property by means of measuring contact angle values between solid surface and liquid was determined through sessile drop technique^{35,36)}. High SBS and wetting property, that is high surface energy, was observed among specimens that showed low contact angle and treated by laser scanning. The finding is a promising indicator to enhance surface wettability of the core material following laser technique. The outcomes reflect the improvement in hydrophilic property.

The study was observed that the water contact angle decreased significantly after coating the substrate by v-c material. Laser group could demonstrate more hydrophilic property than sintering counterpart. The current result is in agreement with previous literature³⁷⁾. However, it is contradicted with previous investigation which was demonstrated a minimal impact of CO₂ laser irradiation on the contact angle values³⁸⁾. Hence, wetting property of the core material could be optimized to achieve satisfied bond strength following laser scanning.

The effects of laser scanning on the mechanical properties of zirconia, especially on fatigue tolerance properties, aging resistance, appropriate settings, and possible changes in chemical composition should be further studied. Based on the current outcomes, this study accepted the hypothesis that coating zirconia using v-c material through laser scanning provided better bond strength than its conventional sintering counterpart.

CONCLUSIONS

Within the limitation of this study, the following conclusions could be drawn:

- Laser scanning provided better bond strength than conventional sintering.
- No correlation was found between surface roughness and aging condition.
- The contact angle of treated zirconia surface was inversely related to the SBS. The lower the contact angle (high wettability), the higher the SBS would be.
- Laser scanning provided optimum adhesion of the v-c to the substructure. Both methods preserved

the integrity of the zirconia substructure since the treated specimens in both methods did not show phase transformation.

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CONFLICTS OF INTEREST

The authors report no conflicts of interest.

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