Iraqi J. Laser, Part A, Vol.17, pp. 23-31 (2018)

IRAQI JOURNAL OF LASER

Influence of Fractional CO_2 Laser Irradiation on Temperature Elevation and Bonding Strength of Resin Cement to the Zirconia Ceramic

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(Received 3 October 2017; accepted 18 December 2017)

Abstract: Objectives: To investigate the effect of temperature elevation on the bonding strength of resin cement to the zirconia ceramic using fractional CO_2 laser. **Background:** Fractional CO_2 laser is an effective surface treatment of zirconia ceramic, as it increases the bonding strength of zirconia to resin cement. **Methods:** Thirty sintered zirconia discs (10 mm diameter, 2 mm thickness) were prepared and divided to three groups (N=10) and five diffident pulse durations were used in each group (0.1, 0.5, 1, 5 and 10 ms). Group A was treated with 10 W power setting, group B with 20 W and group C with 30 W. During laser irradiation, temperature elevation measurement was recorded for each specimen. Luting cement was bonded to the treated zirconia surfaces and cured for 30 seconds. Shear bond strength was evaluated by a testing machine (universal) with bond failure mode determination. **Results:** The lowest temperature elevation measurement of the irradiated specimen which gave maximum shear bond strength was about 1.6±0.3 Ċ higher than ambient room temperature (27±0.2 °C). Apparent micromechanical irregularities were seen in the treated samples and cracks formation with increased pulse duration and power setting were also observed. **Conclusions:** The temperature elevation is a vital factor in the surface roughness of zirconia ceramic with fractional CO_2 laser irradiation and the lowest temperature elevation at best shear bond strength of zirconia ceramic to the resin cement is satisfied with the shorter pulse duration of 0.1 millisecond.

Keywords: Fractional CO_2 Laser, Shear Bond Strength, Temperature Elevation

Introduction

Zirconia ceramic is widely used nowadays as an alternative to metal restorations in dental clinics (Kasraei et al., 2015). Since pure zirconia is an unstable compound of zirconia dioxide; therefore, yttria stabilized tetragonal zirconia polycrystalline (Y-TZP) is mostly used in dentistry (Denry et al.,2008; Blatz et al., 2004). Y-TZP exhibits exceptional chemical and mechanical properties, including high flexural strength, fracture toughness, hardness, wear and corrosion resistance in both acidic and basic ambient conditions, translucency, color acceptance, high effectiveness radiographically and biocompatibility (Raigrodski et al., 2004; Odman et al., 2001; Pjetursson et al., 2007). Although both of these properties are very important, clinical long-term success depends to a great extent on the strength and durability of resin cement bond to ceramic substrates and teeth that have to integrate all system parts into a coherent structure for enhancing the retention, marginal adaptation, fracture resistance and bonding strength of ceramic dental restorations (Burke et al., 2002; Rosenstiel et al., 1998). A strong cement-ceramic bond depends on micromechanical interlocking and chemical bonding to the ceramic restoration surface, which needs surface roughening procedure and cleaning for a suitable surface activation (Borges Et al., 2003). Different methods of surface roughening of dental ceramics surface have been introduced to increase the surface area and create microporosities on the ceramic surface, which enhance the mechanical retention of the luting composite resin (Ayad et al., 2008). The most commonly used methods include grinding, abrasion with rotary instruments (diamond), abrasion with airborne particle (Al_2O_3), acid etching (Ersu et al., 2009), sandblasting (Della Bona et al., 2007; Nagayassu MP. et al.. 2006), and laser irradiation and combination of two or more of these roughening methods (Unal et al., 2015). However, the composition and physical properties of high-strength zirconium oxide ceramics differ considerably from silica-based ceramics. The lack of glassy phase makes these materials resistant to hydrofluoric acid etching (Borges et al., 2003). Airborne particle abrasion has also been used to facilitate the mechanical retention between the Y-TZP ceramics and luting cements. This method increases the surface roughness and bonding area (Xie et al., 2011). However, excessive air abrasion can induce a high loss (chipping) of ceramic material, which adversely affects the mechanical properties and long-term effectiveness of the zirconia ceramic restorations (Zhang et al., 2004). In the recent years, lasers have been employed for different purposes in dentistry which include conditioning the tooth structure or restorative surfaces (Fischer J. et al. 2008). Previous studies used different lasers such as Nd:YAG (Edelhoff et al. 2007; Aboushelib et al., 2006) Er:YAG (Blatz et al. 2004) , femtosecond laser (Kara Ozlem et al., 2015) and CO_2(conventional and fractional) (Ural et al., 2012; Ahrari et al., 2015) for surface roughness of zirconia ceramic, and varying degrees of success have been reported.

The importance of laser effect lies in converting radiant energy to heat energy, as the absorption of laser energy by the surface of the material is the most important interaction between the zirconia ceramic and laser (Usumez et al., 2013) .The CO_2 laser is well suited for the surface treatment of ceramic materials because zirconium oxide ceramic has a high potential of absorbing CO_2 laser wavelength (Akyil et al., 2010). Due to the heat induction process of ceramic surfaces using a focused CO_2 laser, shell shaped tears—typical effects of surface warming—are formed. These tears are believed to increase mechanical retention between composite resin and zirconia (Ural et al., 2010). Although good results were obtained, the effect of temperature elevation of zirconia ceramic during laser irradiation is an important factor concerning its bonding strength to resin cement. Nowadays, a few studies have investigated the efficacy of fractional CO_2 laser for improving the adhesion of resin cement to oxide ceramics (Akhavan Zanjani et al., 2014; Kasraei et al., 2014; Paranhos et al., 2011) , whereas there is very poor investigation concerning the effect of temperature elevation on zirconia ceramic surface and subsequently on the shear bond strength (SBS) of resin cement to zirconia ceramic using fractional CO_2 laser. Fractional CO_2 laser is a CO_2 laser with a wavelength of 10.6 μm. Fractional lasers deliver energy in parallel vertical columns of multiple spots with a predefined space between them that remains intact and untreated during laser irradiation (Huzaira et al., 2003).

The concept of fractional photothermolysis was introduced in 2003 (Ahrari et al., 2013). In dentistry, fractional CO_2 laser as a technique may have several advantages regarding surface treatment. Predetermination of the exact irradiated surface area by the laser controlling panel produces a more homogenous etching pattern. Also, the manual movement of the laser hand piece is restricted during surface conditioning. Furthermore, less thermal damage is compared to that developed using conventional CO_2 laser (Ahrari et al., 2015).

In the present study, the effect of temperature elevation is investigated during the assessment of the shear bond strength (SBS) of resin cement to zirconia ceramic by using different parameters of fractional CO_2 laser as a surface treatment.

Materials and Methods

In this experimental study, 30 Y-TZP discs each measuring 12.5 mm in diameter and 2.5 mm in thicknesses were milled from presintered zirconium oxide blocks (Ceramill Zi, Amann Girrbach AG, Koblach, Austria). First of all, each block was divided into blanks with the dimensions (13 mm, 13 mm, and 12 mm) by a cutting saw; each blank was then glued into a fitting pin. The fitting pin was then placed into the designated place in the milling machine, in a way allowing a rotational movement around its axis at a high speed. At the same time, the cutting saw was moving around the blank in circular movement and upward movement providing a precise cutting to obtain the desired dimensions. The obtained zirconia discs were then sintered in furnace (Ceramill; Amann Girrbach AG, Koblach,Austria) at 1450 °C for

10 hours including cooling, following the manufacturer's instructions. After sintering, each zirconia disc was measured approximately (10 mm in diameter, 2 mm in thickness). Before the surface treatments, all specimens were ultrasonically cleaned in distilled water and acetone (C_3H_6O) for 15 min. to remove any contaminants and dried naturally in the atmosphere. Samples with cracks or fissures were substituted by other perfect samples.

Experimental Work

Each zirconia disc bonding surface was irradiated with 4 scans of fractional CO_2 (gas laser) of λ= 10.6 μm (Brochure, JHC1180, China), chopped (gated) laser with maximum output power of 30 W. The energy of laser was delivered in the order mode and a circular area of 8 mm diameter was irradiated at the middle of the ceramic specimen. The spot size of the laser beam was 0.05 mm^2. The delivery system consisting of 7 articulated arms connected to a hand piece was held manually perpendicular and fixed to a Teflon mold constructed to hold the specimen at a distance of 5 cm. The mold was provided with a circular opening to enable a thermometer (AMPROBE TMD56, Everett, WA, USA) to touch the specimen for measuring the temperature during the laser irradiation. The experiment was held at an ambient room temperature of (27±0.2 ºC). The thermometer was connected to a software program specialized to collect and analyze the thermometer data every 1 second. After laser irradiation, each zirconia disc was horizontally embedded in mixed cold cure acrylic to about 1.5 mm and the remaining 0.5 mm of the zirconia disc height being exposed ensuring that the ceramic surface remain intact for the bonding procedure .A circular silicon mold with an external diameter of 20 mm and 2 mm in height provided with a central circular opening of 5 mm in diameter was positioned over the acrylic mold and properly fitted in a way that the circular opening of the silicon mold was positioned on the center of the zirconia disc. Adequate amount of the adhesive cement (Bisco-BisCem-self-adhesive resin cement, USA) was automixed using disposable mixing tips supplied with the cement kit and dispensed into the opening of the silicon mold. The excess cement was removed with an explorer tip from the periphery of the mold and the luting agent was then photopolymerized using light curing system (Quayle Dental, Sussex, UK) for 30 seconds following the manufacturer's instructions. The silicon mold was removed and finally, one hour after cementation, specimens were stored in distilled water at 37 °C for 24 hours before SBS testing.

The Study Groups

The specimens were randomly assigned into 3 groups (N=10) and 5 different pulse durations (0.1, 0.5, 1, 5 and 10 ms) were used in each group:

GROUP (A): The specimens were treated with a power setting of 10 W.

GROUP (B): The specimens were treated with a power setting of 20 W.

GROUP (C): The specimens were treated with a power setting of 30 W, while the spot energy density of groups was as in Table 1.

Table.1. The Spot Energy Density of the Groups

Pulse duration /ms	**Group A Energy density J/cm²**	**Group B Energy density J/cm²**	**Group C Energy density J/cm²**
0.1	2	4	6
0.5	10	20	30
1	20	40	60
5	100	200	300
10	200	400	600

The SBS was determined by subjecting the samples to a shearing force at zirconia-cement interface in a universal testing machine (LARYEE, WDW-50,50KN, China) with a chisel-ended blade at a rate of 1 mm/min. The SBS values, evaluated in MPa, were calculated using the following formula:

ž Shear strength [MPa] =Failure Load (N)/Surface area (mm^2)

All the fractured samples were evaluated under a stereo microscope (ME, 2665, Euromex, Holland)) at 40X magnification. The mode of failure was classified as follows:

(1) Adhesive: de-bonding only at the cement-ceramic interface.

(2) Cohesive: rupture in the cement or zirconia ceramic.

(3) Mixed: shows both adhesive and cohesive failure modes

Surface Analysis

Three samples from the study groups were coated with gold-palladium (Q150R Rotary-Pumped Sputter Coater, Quorum Tec., UK) and observed under scanning electron microscope (SEM) (Inspect S50, FEI, USA) with 200X magnifications and 15 kV voltage**.**

Statistical Analysis

This analysis was done with SPSS software version 23/France. Data was analyzed by one way ANOVA-test (analysis of variance) to calculate the P-value between the groups' means of temperature elevation measurement in the study. Least significant difference- LSD test was done also to compare between the groups' means.

Results

Shear bond strength and Temperature Measurement

Table (2) presents the descriptive statistics of the SBS values and temperature elevation measurements of the three groups. Also the results of statistical analysis for comparison of temperature measurements among the study groups are shown in the Table.

The specimens treated with fractional CO_2 laser of 20 W/0.1 ms pulse duration exhibited the highest SBS values, and the minimum temperature elevation measurements were observed in groups A and B with 0.1 ms pulse duration whereas the maximum measurement was observed in group C with 10 ms pulse duration. Also it was shown that the temperature increased with increasing the pulse duration with the same power setting .One way ANOVA test and LSD test indicate that there was a significant difference in the temperature elevation measurement of the three groups.

Table (2) Shear Bond Strength and Temperature Elevation Measurements of the Groups.

Pulse duration/ ms	**Group A Mean±SD**		**Group B Mean±SD**		**Group C Mean±SD**	
	Temp./Ċ	**SBS/ Mpa**	**Temp./Ċ**	**SBS/ Mpa**	**Temp./Ċ**	**SBS/ Mpa**
0.1	*0.3±0.1 a	7.85±0.2	*1.6±0.3 a	15.5±0.3	*3.1±0.2 b	12.7±0.1
0.5	**2.6±0.2 a	7.6±0.1	**3.3±0.2 a	12.65±0.1	**8.2±0.4 b	11.7±0.2
1	**3.1±0.2 a	7.4±0.1	**5.4±0.2 a	11.5±0.2	**13.1±0.3 b	10.75±0.3
5	***10.2±0.2 a	7.15±0.1	***51.1±0.1 b	10.52±0.3	***63.2±0.2 c	9.65±0.1
10	****42.2±0.2a	6.75±0.2	***62.6±0.2 b	9.55±0.2	***72.2±0.1 c	8.6±0.2
P-value of temp. elevation	0.01		0.01		0.01	

One-way ANOVA test and LSD test of groups mean ±SD of temperature elevation measurements. LSD for each pulse duration includes all powers (raws) expressed as (a, b, c) LSD for each power includes all pulse duration (columns) expressed as (*, **, ***). The similarity of symbols between two groups means that there is no significant difference between them.

- Non-significant at P> 0.05,
- Significant at P≤ 0.05,
- Highly significant at P≤0.01.

Failure Mode

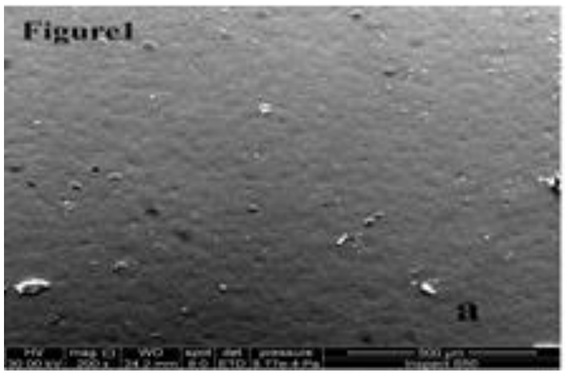
Frequency of failure mode after shear bond strength test of each group was showed in Table (3). The results indicated that the failure mode of the groups with different parameters varied. In group A type 1 failure mode was frequently observed. On the contrary, failure mode of type 2 or type 3 was detected in group B. However, type 1 failure mode was found in those treated with (20W for10ms). In group C failure mode types 1 was observed except for (0.1 ms and 0.5 ms) which showed failure mode of type 2.

Table (3). Failure Modes Distribution.

Group	**Pulse Duration /ms**	**Adhesive(1)**		**Cohesive(2)**		**Mixed(3)**	
		No.	%	No.	%	No.	%
A	0.1	1	10	1	10		
	0.5	1	10	1	10		
	1	2	20				
	5	2	20				
	10	2	20				
B	0.1					2	20
	0.5			2	20	1	10
	1			1	10	1	10
	5			1	10		
	10	2	20				
C	0.1			2	20		
	0.5			2	20		
	1	2	20				
	5	2	20				
	10	2	20				

Surface Analysis

The SEM pictures of zirconia disc surface is illustrated in **Fig.1(a-c)** .The texture of the surface shows micromechanical irregularities with irregular micro and macrcracks in those specimens treated with (20W for 10ms) as shown in **Fig.1(b).** These cracks are connected to each other like a network and large flaws could be detected easily on the ceramic surface as shown in specimens treated with (30W for 10ms) as in **Fig.1(c).**



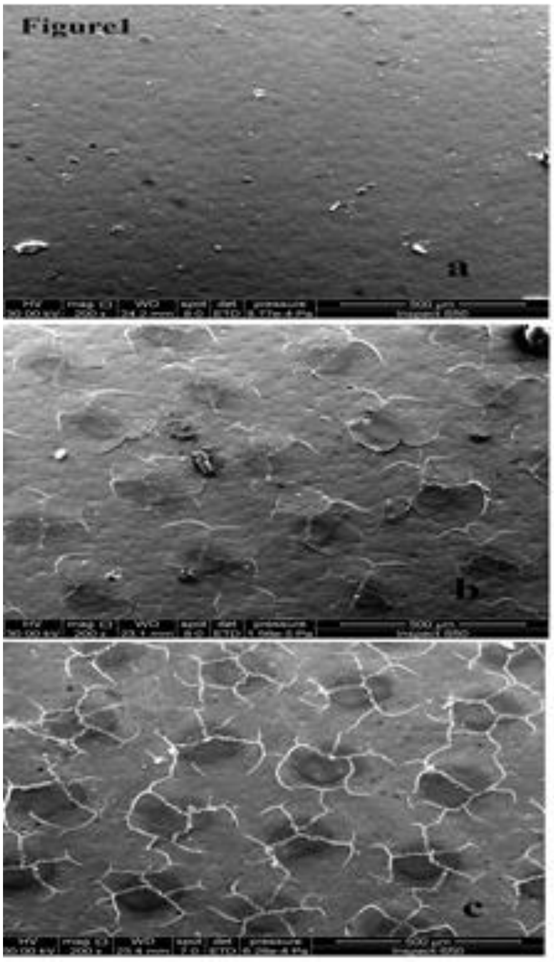


Fig. (1): SEM Pictures of Zirconia specimens (200X). a .Control (untreated) Specimen. b. (20 W/10 ms).c. (30 W/10 ms)

Discussion

Different lasers have been proposed for surface roughening of zirconia ceramic, among them, CO_2 lasers were shown to be a suitable surface treatment of ceramic materials (Usumez et al., 2013). In the present study, the effects of temperature elevation on the SBS of resin cements to zirconium oxide surface using fractional CO_2 laser with different power setting and pulse durations are evaluated and compared.

Shear bond strength test was used to evaluate the bonding property of zirconia ceramics to (BISCO-USA) dual cured resin cement. BisCem-Bisco adhesive cement has a very simple application technique and can be done in a single step clinically. Resin cement is a critical part of today's clinical practice as it provides enhanced adhesion to ceramic restorations because of its high compressive and tensile strengths, low solubility with a favorable aesthetic qualities (Hill et al., 2011).

During laser irradiation, short pulse duration should be used to confine the optical energy to the zirconia surface in order to avoid micro and macro cracks formation. SBS of the specimens treated with the fractional CO_2 laser (20 W/0.1 ms) as (15.5±0.3 Mpa) with 5 seconds working time was significantly greater than those of the other groups for the same or for different pulse duration, which is greater about 20% than that of the conventional CO_2 laser (which showed a maximum SBS of 12.12± 3.02 Mpa) or other lasers such as Er:YAG(which showed a maximum SBS of 8.65± 1.77 Mpa) used with the same type of zirconia ceramic system (Kasraei et al., 2014). This result was given due to the effect of fractional CO_2 laser in roughening the bonding surface through the process of thermal conduction of concentrated laser energy within short pulse duration with a more homogeneous etching pattern resulting in micromechanical irregularities that increased mechanical retention thereby enhanced the bonding strength of zirconia ceramic to resin cement.

The maximum temperature elevation measurement obtained in those specimens treated with 30 W/10 ms pulse duration was about 72 Ċ higher than the ambient room temperature (27± 0.2 Ċ) and the minimum temperature elevation measurement of the laser irradiated specimen giving the maximum shear bond strength was 28.6± 0.3 Ċ, with the laser parameter of 20 W/0.1 ms pulse duration which is about 1.6 Ċ higher than ambient room temperature. An increase in the power setting or pulse duration resulted in more thermal effects on the zirconia surface as observed in temperature elevation after irradiation with different laser parameters. The higher temperature elevation didn't reveal higher SBS due to the fact that this thermal effect resulted in more heat dissipation and subsequently cracks formation which could adversely affect the material structure and its bonding strength, confirming that heat confinement is a critical factor in the surface roughness of zirconia ceramic with laser irradiation.

The quality of zirconia-cement bond was also assisted by the bond failure mode analyses as it provided important illustrations of the bonding effectiveness (Toledano et al., 2007). The bond failure of group A was mostly adhesive due to the inadequate micro retentive interlocking obtained with low power laser irradiation as observed in microscopical examination. An adhesive failure mode was also found in group C indicating that the micromechanical interlocking of this group was not strong enough due to macro cracks formation that resulted in low bond quality except for specimens treated with 0.1ms and 0.5ms pulse durations of group C which showed cohesive failure mode. In group B, Cohesive and mixed failure modes in resin cement were the most predominant. This means that the bond strength between cement and ceramics was mostly higher than the cohesion bond of the cement material.

The specimens irradiated with 20 W/10 ms and 30 W/10 ms showed significant superficial changes in SEM observations. The high temperature due to laser irradiation effectively facilitated localized surface heating and created a damaged surface layer with micro and macro cracks due to an increase in the heat dissipation following laser irradiation with long pulse duration which may affect the strength of the structure of zirconia ceramic adversely. This means that both of the pulse duration and the power setting had a compromising effect on the temperature elevation and subsequently on SBS of zirconia ceramic to the resin cement. Researchers concluded that power setting and irradiation time must be carefully considered using high-energy lasers to avoid cracks formation on zirconia surfaces (Unal et al., 2015; Liu L. et al., 2015). However conclusions obtained from one commercial zirconia ceramic system may not be suitable or applicable to other commercial systems. Future studies are suggested to evaluate the effect of fractional CO_2 laser irradiation on the surface properties and bonding strength of other zirconia ceramic types to the resin cement.

Conclusion

Within the limitation of the current study, it can be concluded that:

The lowest temperature elevation measurement of the fractional CO_2 laser irradiated specimen which gave the maximum shear bond strength is obtained with the laser parameter of 20 W/0.1 ms pulse duration.

The temperature elevation is a vital factor in the surface roughness of zirconia ceramic with laser irradiation to enhance its bonding strength to the resin cement.

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تأثير تشعيع ليزر CO_2 المجزئ على ارتفاع درجة الحرارة وقوة التصاق الاسمنت الراتنج إلى الزركونيا السيراميك

لمى الحسني حسين علي جواد

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الخلاصة : الأهداف: للتحقيق في تأثير ارتفاع درجة الحرارة أثناء تقييم قوة التصاق الاسمنت الراتنج إلى السيراميك زركونيا باستخدام ليزر CO_2 المجزئ. **الخلفية:** الليزر CO_2 المجزئ هو معالجة سطحية فعالة للسيراميك زركونيا، لأنه يزيد من قوة الترابط من زركونيا السيراميك للاسمنت الراتنج. **الطريقة**: تم تحضير ثلاثين قرص زركونيا السيراميك (قطر 10 ملم، سمك 2 ملم) وقسمت إلى ثلاث مجموعات(10 = N) ، واستخدمت خمس فترات نبضة متفرقة في كل مجموعة (0.1 و 0.5 و 1 و 5 و 10 مللي ثانية) . تم التعامل مع المجموعة A مع إعداد طاقة 10 واط، المجموعة B مع 20 واط ومجموعة C مع 30واط.و خلال أشعة الليزر، تم تسجيل قياس ارتفاع درجة الحرارة لكل عينة. وتم صب الاسمنت الراتنج إلى أسطح الزركونيا المعالجة وعولج لمدة 30 ثانية. تم تقييم قوة اللقص بواسطة آلة اختبار (عالمية) مع تحديد وضع فشل الالتصاق **النتائج:** كان الحد الأدنى من قياس ارتفاع درجة الحرارة للعينة المشععة التي أعطت أقصى قوة التصاق حوالي 1.6 Ċ أعلى من درجة حرارة الغرفة المحيطة (27 ±0.2 ºC) لوحظ وجود اختلالات ميكانيكية واضحة في العينات المعالجة وتشكيل الشقوق مع زيادة مدة النبض وزياده الطاقه. **الاستنتاجات:** ارتفاع درجة الحرارة هو عامل حيوي في خشونة السطح لزركونيا السيراميك مع تشعيع ليزر CO_2 المجزئ وأدنى ارتفاع درجة الحرارة في أفضل التصاق من زركونيا السيراميك إلى الاسمنت الراتنج كان متوافقا مع مدة نبضه 0.1 ميلي ثانية .