Ministry of Higher Education and Scientific Research University of Baghdad
Institute of Laser for Postgraduate Studies

The Effect of Fractional Er:Cr YSGG Laser on Bond Strength of Zirconia Ceramic to Resin Cement

A Thesis Submitted to the Institute of Laser for Postgraduate Studies, University of Baghdad in Partial Fulfillment of the Requirements for the Degree of Master of Science in Laser / dentistry

By

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بسم الله الرحمن الرحيم

﴿وَعَلَّمَ آَدَمَ الْأَسْمَاءَ كُلَّهَا ثُمَّ عَرَضَهُمْ عَلَى
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كُنْتُمْ صَادِقِينَ ، قَالُوا سُبْحَانَكَ لَا عِلْمَ لَنَا
إِلَّا مَا عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ﴾

سورة البقرة: (30–32)

Certification

I certify that this thesis was prepared under my supervision at the Institute of Laser for Postgraduate Studies, University of Baghdad, as a partial fulfillment of the requirement for the degree of ‘Master of Science of Laser/Dentistry

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Dedication

Mother and father..

I would’nt reach this far in my life’s journey,

if it is was’nt for your love and support.

To all of my relatives and friends

who wished me luck and happiness

Acknowledgements

My heart is overflowing with your love, dear 'allah'. And I am greatfull for all the power and graces you've been giving me. And greatfull for having the prophet muhammad as a leader to my heart and soul.

I am fulled with gratitude to my kind supervisor: Prof. Dr. Hussein A. Jawad for showing me the way, as I was lost in the greatness of the science world .

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ABSTRACT

Background: Various studies assessed different traditional techniques and laser applications, for the purpose of achieving an enhanced shear bond strength of zirconia ceramic material to resin cement. The aim of the present study was to examine the effect of pulsed erbium, chromium: Yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser parameters (pulse duration, fluence, irradiation time) on surface roughness for enhancing the shear bond strength of zirconia ceramic (Vita YZ HT ,Zahnfabrik, Germany) to an adhesive resin cement.

Material and method: Presintered zirconium oxide blocks were milled into 124 zirconia discs for the desired dimensions (9 mm diameter, 2 mm height). 82 specimens were used for the pilot study for the determination of the most effect (fluence, pulse repetition rate, pulse duration, laser irradiation time) on zirconia surface. 42 specimens are divided into six groups each of seven samples (control and laser groups). The laser groups are: Group (A): 20 s, 60 μs pulse duration, group (B) : 30 s, 60 μs pulse duration, group (C): 40 s, 60 μs pulse duration, group (D): 20 s, 700 μs pulse duration, and group (E): 30 s, 700 μs pulse duration. All group's samples were laser irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.4, 25, 28.57) J/cm^2. Luting adhesive cement was bonded to the laser irradiated zone of the zirconia disc's surfaces and light cured for 40 second. Shear bond strength was evaluated using a universal testing machine and the optained results were statistically analyzed. Also the average surface roughness was analyzed, by the atomic force microscope. The bond failur modes were also examined.

Results: Obvious laser pulse holes were observed on the laser treated samples, under the scanning electron microscope, and under the optical microscope, with an increase in their distinctness and depth at high laser fluence and long irradiation time, contirbuting to a clear enhance in the mean pulse depth, average surface roughness, and thereby in the shear bond strength. For the short

pulse duration (60 µs) groups, group B (28, 57 J/cm^2) specimen had statistically differences of high significancy for the average surface roughness and depth of pulses values. And had the highest shear bond strength value, of very high significant difference, compared to other groups of same and different pulse duration. With no microcracks formation.

Conclusion: There was an essential role for the Er,Cr:YSGG short pulse duration (60µs), that it's effect is better than the long pulse duration (700 µs) effect, in surface roughening and in shear bond strength enhancing. Er,Cr:YSGG laser could be considered as an effective alternating conditioning method for enhancing the bonding strength.

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List of abbreviations

Symbol	term
CO_2	Carbon dioxide
C°	Degree Celsius (unit of temperature)
Er:YAG	Erbium doped yttrium-aluminum garnet
Er,Cr:YSGG	Erbium_chromium doped yttrium-scandium-gallium-garnet
FDA	Food And Drugs Administration
Hz	Hertz (unit of frequency)
K	Kelvin (unit of temperature)
Mpa	Mega Pascal
mJ	Milli joule (unit of energy)
mm	Millimeter
Nd:YAG	Neodinium doped Yttrium-Aluminum-garnet
nm	Nanometer ($=10^{-9}$ m)
s	Second
W	Watt (unit of Power)
µm	Micrometer ($=10^{-6\ m}$)
µs	Microsecond ($=10^{-6\ m}$)
λ	Wavelength
SBS	Shear bond strength
rpm	Revolutions per minute

Chapter One

Introduction and basic concepts

1-1 Introduction

In search for the ultimate esthetic restorative material, new ceramic systems have been produced. Ceramics offers the potential for excellent esthetics, long-term stability, biocompatibility [1], and ideal mechanical properties [2]. One material currently having a great interest is zirconia. Zirconia is considered as the strongest and toughest ceramic material available for use in dental applications now adays [3]. Zirconia has the potential of being used for multi unit all-ceramic restorations, for high-stress alveolar ridge bearing areas, such as in posterior region of the mouth. Thus, zirconia frameworks with high bending strength and toughness , prepared by CAD/ CAM, and constructed with other dental ceramics, can be utilized for replacement of the traditional metal-ceramics (MC) prostheses [4]. Introducing yttrium-stabilized tetragonal zirconia polycrystals (Y-TZP) to the dental field had opened new limits for designing and application of CR, allowing for minimum thickness of framework (0.5 mm) with the remaining thickness of restoration used for building ceramic veneer [5]. Therefore, Y- TZP has been accepted as a durable and suitable core material for fixed partial dentures (FPDs) [6] . Clinically, the mechanical properties are very important, , but the success of polycrystalline ceramics depends on adequate adhesion to dental elements, allowing for higher fracture resistance, increasing retention, and microleakage elimination [7], that would prevent marginal discoloration, recurrent caries, and subsequentiy failure [8]. Conventional techniques for cementation with zinc phosphate and resin- modified glass ionomer cements could be applied to zirconia surfaces. However, adhesive cementation is more recommended for obtaining adequate retention and marginal sealing [9]. The problem with this respect is the low capacity of the resin bond to zirconia ceramics, which is related to the zirconia glass–free, highly crystalline composition [10]. Therefore, they are not conventionally conditioned by hydrofluoric acid, constituting major problems for the adhesive bonding [7]. Chemical and /or mechanical surface treatments provide a reliable adhesive bonding to resin cement and ceramic on metal-free prosthetic restorations . Air -borne particle abrasion [9], diamond bur grinding

, sandblasting, zirconia primers [11], tribochemical silica coating [12], selective infiltration technique [13], and Nano-alumina coating [14], have been applied for surface conditioning. Blasting treatments would decrease the dental ceramic prostheses strength due to the formation of subcritical micro cracks and phase transformation within zirconia surface. Which might negatively affect the mechanical properties [15]. Airborne particle abrasion of prosthodontic materials, has the potential for removing significant amount of material , which could subsequently affect their clinical adaptation [16]. Tribochemical silica-coating using (Co Jet) system at the dentist‟s office is a widely used conditioning technique for ceramic structure. Nevertheless, it has been criticized for possible subcritical crack propagation within zirconia thin restorations [17]. Lasers have been proposed as an alternative dental material surface conditioning method, using different wavelengths, such as: Er:YAG, Nd:YAG, CO_2, Er,Cr:YSGG [12], and fractional CO_2[18]. Studies investigating the impact of pulsed Er,Cr:YSGG (erbium, chromium: yttrium, scandium, gallium, garnet) laser power on adhesion efficiency between resin cement and ceramics are very limited. Accordingly, the objective of this research is to evaluate the influence of pulsed Er,Cr:YSGG laser irradiation power and pulse duration on surface roughness and bond strength of resin cement to zirconia ceramic.

1.1.1 Aim of the study:

1- Evaluation of Er,Cr:YSGG laser parameters (pulse duration, fluence, irradiation time) in adhesion efficiency of zirconia ceramic to an adhesive resin cement.

2- Investigation the role of pulse duration in enhancing the surface roughness and the laser pulse depth.

1.2 Anatomy of crown

The anatomical crown of a tooth: is the area above the cemento-enamel junction or neck of tooth. It is completely covered with the highly mineralized enamel, that is: the tissue exposed to oral cavity, which forms a protecting layer at the teeth's anatomical crown [19]. While , the term clinical crown refers to any part of tooth visible in mouth , as shown in figure (1-1).

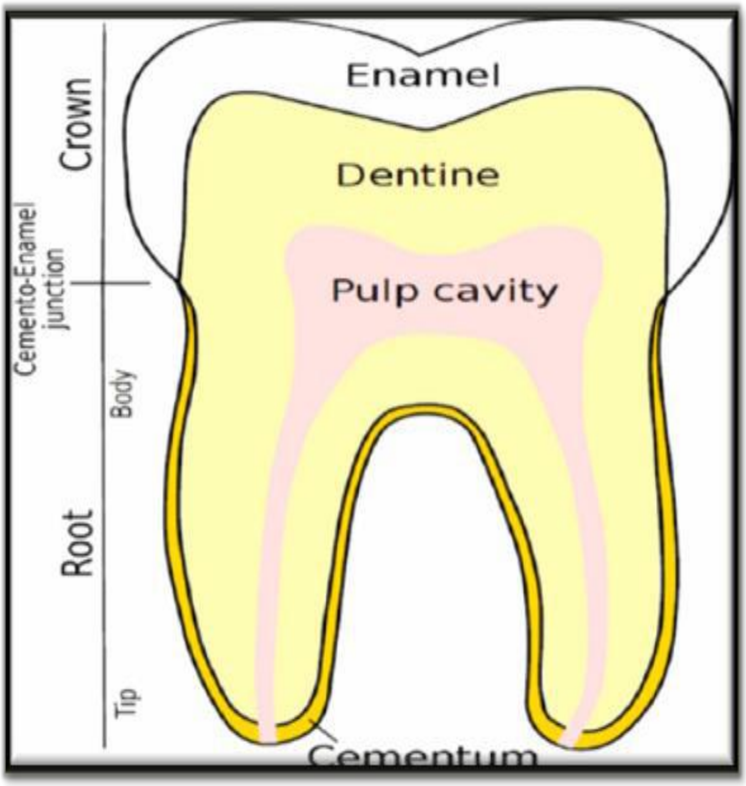

Figure (1-1): Diagrammatic representation of a human molar showing t he cellular tooth tissue [20]

Dental crowns are caps that are mimicing teeth by covering and restoring their size, shape and durability. Crowns are bonded to tooth surface by dental cement (resin or acid-base cement) [21]. Crowns are made from various types of materials, depending on the location of tooth needing a crown. Permanent crowns can be made of all-metal (such as gold or another alloy), porcelain-fused to metal, all resin, or all ceramic [9]. Metal- ceramic restorations have superior physical properties, and their marginal adaptation and aesthetics are clinically acceptable [21]. However, light reflection from the opaque porcelain at the restoration’s cervical third might cause a light grey appearance. Which had led to the search for more esthetic solutions.

Several types of full ceramic systems have been developed for meeting the highly esthetic demands for a more naturally appearing restorations. According to this classification, all-ceramic materials can be categorized into three groups, depending on the phase/phases of their chemical composition :

1) Glass-matrix ceramics: Containing a glass phase [feldspathic, leucite- reinforced glass ceramics, lithium disilicate glass ceramics [22].

2) Polycrystalline ceramics: No-glass containing, only a crystalline phase.

- Aluminum-oxide: The first material introduced for anterior-posterior full ceramic restorations [23].
- Yttria-stabilized zirconia polycrystals: Presenting superior mechanical proprieties, allowing for the construction of full anterior-posterior fixed ceramic partial denture [22].

3) Resin-matrix ceramics: Polymer matrix of predominantly inorganic refractory compounds [24]

All-ceramic materials can be used for the producing of all kinds of tooth restorations [25] such as: veneers, inlays, onlays, crowns and posts. lithium desilicated ceramic can be utilized for the production of 3-unit bridges (in the anterior and premolar region). Whereas, multi-unit bridges can only be made by stabilized zirconia [26]. Zirconia shows same mechanical properties to stainless steel [5], as it has been the strongest and toughest dental ceramics [27]

1.3 Zirconium biomaterial

Zirconium (Zr): Is a transitional metal with the atomic number (40). It was first discovered in 1789 by the chemist Martin Klaproth [28]. The material has a density of (6.49 g/cm^3), melting point (1852 °C), and boiling point (3580 °C). It has a hexagonal crystal structure and it is grayish in color. Zr is not found in a pure manar in nature, As it is found as zirconium dioxide (ZrO_2), representing the ceramic material, either combined with silicate oxide with a mineral named: Zircon (ZrO_2 - SiO_2) or as a free oxide (ZrO_2) with a mineral named: Baddeleyite [5]. These minerals cannot be used as primary materials in dentistry because of the impurities of these metal elements, that affect their color. And because of the natural radionuclides like

;Urania and thorium, which make it radioactive [29]. About 0.02% of the earth's crust comprises zirconium, with the largest deposits in Brazil and South Africa as Baddeleyite (monoclinic zirconia) and high persent in Australia and India, where it is found as zircon ($ZrSiO_4$) [30].

1.3.1 Zirconia ceramics concept

Zirconia is available as white, crystalline powder, after a cost-intensive purification and production process [5]. ZrO_2 is a polymorphic material that it is found in three forms: monoclinic,

tetragonal, and cubic. The monoclinic phase is stable at room temperature up to (1170 °C). The tetragonal at temperature of (1170-2370 °C). And the cubic phase at over (2370 °C), as shown in figure (1-2) [31]. During the cooling process of a pure zirconia molten mass, the transformation from the tetragonal to the monoclinic phase, which is also described as a martensitic transformation, is characterized by an increase in volume of approximately 3- 4 % [5]. This abrupt volume increase poses an excessively high tension and an unwanted cracks development in ceramic structure , making it impossible to produce sintered ceramics from pure zirconia. Zirconia tetragonal-to-monoclinic phase transformation can also be induced by stress, and surface treatments [32]. This martensitic transformation can be hindered, however, by the addition of stabilizing oxides, that stabilizes the tetragonal and/or cubic phases, such as: Magnesia (MgO) [33], yttrium (Y_2O_3), calcic (CaO)[34], alumina (Al_2O_3) [35], and ceria (CeO)
[36], allowing the generation of multiphase materials known as partially

stabilized zirconia (PSZ) of, generally, a cubic zirconia microstructure at room temperature, as major phase, and a monoclinic and tetragonal zirconia precipitates as minor phase [5]. The stabilizers are incorporated into the crystal lattice of zirconia. Zirconia used in dentistry was only made possible with use of the CAD/CAM technology [37]. Computer-aided design/ computer-assisted manufacture (CAD/CAM) system promoted the material in modern dentistry for multiple clinical all-ceramic applications; root canal posts, orthodontic brackets, implant abutments and frameworks [38].

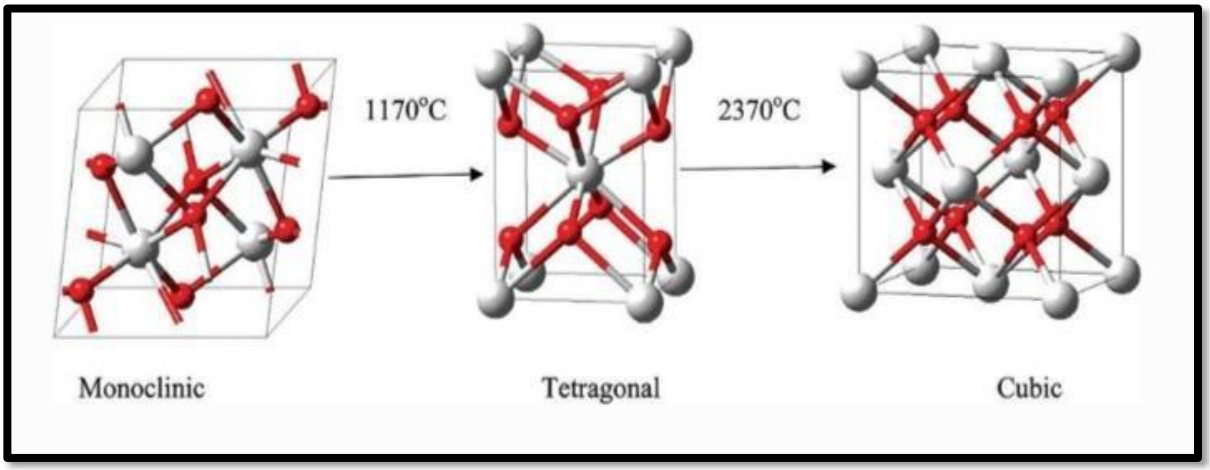

Figure (1-2): Crystallographic and relative temperature of the three zirconia phases [39].

1.3.2 Characteristics of zirconia

1.3.2.1 Biological characteristics

In vitro and in vivo studies had confirmed the Y-TZP high biocompatibility when used as a very pure zirconia powder, that have been purged of their radioactive content [40]. No proved localized or systematic adverse reactions to the material. Depending on the smoothness of ceramic, it shows a plaque formation inhibition, which create a favorable surface for gingival tissue [41]. Although some data quantified and explored differences in the biocompatibility of zirconia, no instances of gingival inflammation or periodontitis could be shown [42]. Different studies have demonstrated fewer bacterial accumulation around Y-TZP than with titanium [43]. However, localized immune-inflammatory reactions could be promoted by particles from the zirconia low-temperature degradation, or from the manufacturing process [44].

1.3.2.2 Optical characteristics

Zirconia has a whitish opaque appearance, due to the extremely high number of interfaces for the numerous very small crystal structures through which light has to pass. And also duo to the high light refractive index [40]. Ceramic system used in dentistry must have an adequate translucency to achieve good dental aesthetic while providing adequate strength during mastication. These two properties cannot be obtained by a single material. Thus, an oxide ceramic material should be used as basic framework, while a glass or feldspathic ceramic must be used as an esthetic veneering material for a good masking of a darkened substrate, which allows for a controlled translucency after lamination procedure [2]. The index of refraction of conventional zirconia is anisotropic in different crystallographic directions, inducing reflection and refraction phenomena at the grain boundaries, Thus, reducing light transmittance. Therefore, a new approach to increase the translucency of zirconia was to develop an isotropic cubic zirconia material, [45]. This was achieved by an increased percentage of yttria for stabilizing zirconia composition, that resulted in 10 to 15% increase in the cubic crystalline phase [46]. And also by reducing the grain size [47], which should be less than the

visible wavelength ideally under (100 nm) [45]. Kim et al., concluded that a short sintering time yielded small grain size and increased the light transmittance values of zirconia ceramic. The transmittance also increases with the rise of sintering temperature (from 1350 to 1550 ºC) that the polycrystalline structure would become more compact, less porous [48]. Zirconia frameworks veneered with silicate ceramic, have high chipping rates of these veneered frames, especially on posterior molar region [49]. Therefore, to avoid chipping of the veneered zirconia layers, full-contour restorations (monolithic) [50], with a flexural strength that is higher than that of the core ceramics stratified with layering porcelain, were tested [51].

1.3.2.3 Mechanical characteristics

- **Flexural strength**

It is the final force required to cause a fracture. It is strongly affected by the size of flaws and by defects on the tested material`s surface [42]. Zirconia is a ceramic material that can be used in the posterior region because of it"s high flexural strength. One of the reasons for this high strength is it"s high crystalline content [52].

- **Aging**

It is the zirconia degradation at low temperature (LTD). It is a spontaneous and a progressive phenomenon that is exacerbated in presence of water, steam, or fluids. Consequences for zirconia"s aging process are many, including: surface deterioration, micro cracks, and decreased resistance. Aging occurs through a slow surface transformation to the more stable monoclinical phase. This phenomenon leads to occurrane of cascade of events in the neighboring particles, causing volume increase that stresses the particles , and resulting in subcritical crack growth (SCG), offering a way for water to penetrate inside the material [44]. the main factors affecting zirconia aging are the stabilizer type (oxides) and it"s content, the grain size and the residual stress. The best resistance to LTD can be achieved by adding the most appropriate stabilizer; Y_2O_3 between 3.5 and 8 mol % [53], sintering at (1450°C) with one h dwelltime [54], reducing the particle size, or even the formation of composites with Al_2O_3 [55]

❖ **Phase transformation toughening**

It is a self-heal crack property [56]. The t–m transformation followed by volume expansion could be used to enhance the fracture toughness of partially stabilized zirconia-based materials. This mechanism was explained as „oriented nucleation of microcracks‟ [57]. When a restoration containing metastable tetragonal-zirconia is subject to an external source of energy, as in case of tensile stress, temperature shock or overloading in patient with parafunction, cracks may occur. Zirconium oxide grains are transformed from their tetragonal to monoclinic form accompanied by a volumetric expansion of the grains thus restricting the crack. Since this expansion is constrained by the surrounding material, the net result is compressive stress on the crack‟s surface, thus hindering crack propagation, and preventing failure of zirconia restoration. This is the reason why this phenomenon is called "phase transformation toughening [58].

1.4 Types of zirconia ceramics

Various types of zirconia are available for dental applications, including

- ❖ Partially stabilized zirconia (PSZ),
- ❖ Tetragonal zirconia polycrystal (TZP),
- ❖ Zirconia toughened alumina (ZTA), and
- ❖ Fully cubic stabilized zirconia (CSZ) [50]

1. Magnesia partially stabilized zirconia (Mg-PSZ)

It is one of the most commonly used zirconia-based engineering ceramics [59]. Factors that have discouraged the interest of ceramic manufacturers in development Mg-PSZ for biomedical applications are: difficulties in obtaining precursors free of impurities, residual porosity in the material"s mass, and a rather coarse grain size (30-40 µm) [5], which may lead to surface wear and large crack propagation. The sintering temperature is much higher (1680–1800 °C) than other composites. It has been reported that reinforcement by phase transformation toughening is less pronounced in Mg-PSZ than in Y-TZP. The microstructure of Mg-PSZ consists of an array of cubic zirconia partially stabilized by 8 to 10% (by mol) of magnesium oxide. A dental ceramic system called Denzir-M (Dentronic AB, Skellefteå, Sweden) is an example for a fully sintered Mg-PSZ ceramic for dental crown and bridge that requires rigid and strong machining system [59].

2. Yttria stabilized tetragonal zirconia polycrystals (3Y-TZP)

One of the mostly used dental ceramic material, and the first version of conventional zirconia, was the high-strength tetragonal-crystalline phase zirconia, stabilized with 3 mol % yttria (Y_2O_3) and enhanced with 0.25 % alumina to minimize the LTD [60]. Tetragonal zirconia can be fully stabilized with 8 mol % yttria. However, concentrations above or below 3mol% yttria shows a decrease in strength of ceramic [61]. Stable tetragonal form helps control stresses incorporated during the tetragonal to monoclinic phase transformation, creating a material with a crack propagation arresting, for a higher toughness, making it suitable for dental applications[5]. (Y-TZP) had shown greater strength compared to other molecular types of zirconia [36]. The material exhibits brilliant mechanical properties; high flexural strength (700-1200 MPa) and high fracture toughness (7-10 MPa m1/2) [48], hardness, corrosion resistance, wear and tear under both acidic as well as basic ambient conditions, color stability, and greater effectiveness of diagnostic radiographs [62]. The grain size significantly influences the mechanical properties of zirconia 3Y-TZP. High temperature and longer sintering periods produce larger grain sizes and will subsequently diminish the mechanical properties due to large pore sizes [63]. Prosthetic restorations with 3Y-TZP are obtained by CAD/CAM milling of the pre-sintered blocks, followed by another subsequent sintering performed at a high temperature, or by machining sintered blocks [64].

3- Zirconia – toughened alumina

Zirconia-toughened alumina is composed, by weight, of 70% - 90% alumina, and 10% - 20% zirconia. Similar to the toughening of Y-TZP, ZTA is toughened by a stress-induced transformation mechanism. In ZTA microstructure, the stress-induced transformation toughening occurs from the uniform internal strain that causes the zirconia structure to crack and the zirconia particles to undergo phase transformation. During this process, the number of zirconia particles increases and this change induces compressive stress within the alumina structure. The result of this process is that the strength is doubled and the toughness is increased two to four times [65]. Although there is a relatively low concentration of zirconia in these composites, they shows a similar hardness value in comparison with other materials such as Al_2O_3 [61]. ZTA can be manufactured according to two different processes: soft machining or slip casting. The latter offers the advantage of a more limited shrinkage, but, when compared with 3Y-TZP it showes a higher porosity and poorer mechanical properties [66].

4- Fully cubic stabilized zirconia (CSZ)

This new generation of zirconia has excellent optical features compared to the other types of zirconia. Increasing the yttria content of zirconia to more than 8 mol % will help in stabilizing the cubic stage inside the plan [45]. The new translucent zirconia has a molecular structure which is different from the conventional zirconia. Changes in the formulation has not only lowered flexural strength from 1000 to 600 Mpa, but also have eliminated the unique transformation toughening [67]. So these ceramics are only indicated in less- bearing clinical situations [68]. In 2015, two new products were introduced: cubic ultra-translucent zirconia (UT-550 Mpa). and super-translucent zirconia (ST-750 MPa) [69].

1.5 Dental applications of zirconia

1- Zirconia-based implant and implant abutments

Experimental studies showed that the peri-implant soft tissues react successfully to zirconia implant surfaces [70], and that, ZrO2 implants are able to sustain chewing stresses [71]. In addition, no implants were lost during the observation period [72]. Zirconia as implant abutment material was first introduced in 1996 [73]. It shows, both in vivo and in vitro, excellent biocompatibility and good radiopacity. Moreover, it is not soluble in water. With a negligible corrosion susceptibility in oral environment [28]. There has been no report on zirconia implant abutments fractures in any clinical trial [74]. No significant differences in bone levels were found, when comparing zirconia and titanium abutments after 3-years of follow-up, [75].

2- Zirconia-based dental posts

The requirement for more esthetic posts, especially under all-ceramic restorations for restoring anterior teeth, has started the development of new post materials. metal posts may result in unfavorable esthetic, such as grey discoloration of the translucent all-ceramic crowns and surrounding gingival margin [76]. Additionally, the corrosive reactions of prefabricated metal posts may cause metallic taste, oral burning, pain, sensitization, and other reactions [77]. Number of researchers have introduced stabilized zirconia ceramic for the fabrication of post system, because of it's higher strength and fracture toughness, compared to other ceramics. [76] reported that the zirconia post showed a high success rate. Zirconia posts are available as smooth, tapered, and parallel, or tapering at apex. Zirconia posts can be used with both direct or indirect techniques. With excellent light transmission via both the root and the coronal restoration [78].

3- Zirconia-based esthetic orthodontic brackets

Polycrystalline ZrO_2 brackets, have been presented as an alternative to Al_2O_3 ceramic brackets [79]. ZrO_2 has been reported to be cheaper than crystalline Al_2O_3 ceramic brackets, of favorable sliding properties with both stainless steel and nickel-titanium arch wires along with reduced plaque adhesion and clinically acceptable bond strengths at the bracket/adhesive interface [80].

4- Zirconia-based crown and bridge

ZrO_2 is often used for anterior teeth with superior mechanical properties (fracture toughness, strength, and hardness) compared to that of the metal- based materials [81]. It"s load-bearing capacity proved to be higher than other conventional all- ceramics materials, such as

lithium dislocate, glass ceramics
and zirconia- reinforced glass infiltrated alumina
[82].

1.6 Prefabrication procedures

Zirconia is mechanically processed. As a result, blanks with specific shapes are pressed from ZrO_2 powder to be processed using special (CAD/ CAM) machining system [83]. Zirconia ceramics can be milled in the green state, in the partially sintered state, or in the fully sintered state [84].

1.6.1 Soft machining techniques

The most popular manufacturing process that is based on milling presintered zirconia blanks, which are fabricated by cold-isostatic pressing of the zirconia powder, stabilizing oxides, and binding agent's mixture. Soft machining generates negligible internal porosities (about 20–30 nm) of the highly homogenous and easy to mill zirconia, reducing production time, machinery wear and surface flaws. The downside is that this process abapt a 25% precise oversizing of the framework to be milled, implementing dimensional inaccuracies, particularly in the presence of complex framework geometry as in figure (1-3) [28]. Following CAD/CAM system milling, the enlarged prosthesis substructure undergo‟s a sintering process at (1350–1500°C), to reach the material‟s final structural densification (more than 99 %), so that the ultimate properties of the material are achieved, as shown in figure (1-4) [85]. The term “white body” means that zirconia has already undergone pre- sintering at high temperatures; the binders were burnt out in this period and the material hardened minimally. The sintering parameters vary from one manufacturer to another [83]. Most manufacturers of CAD/CAM systems have adapted the soft zirconia processing. The disadvantages of these versions are the cost-intensive sintering special ovens [86].

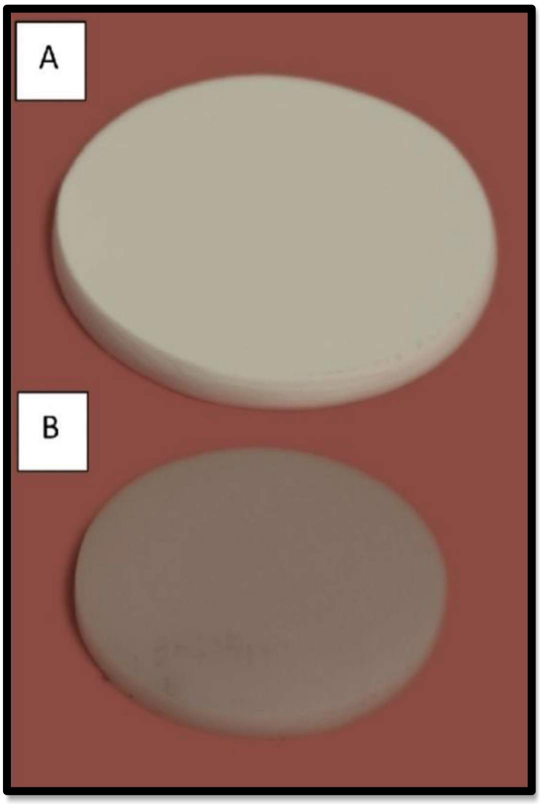

Figure (1-3): (A). Presintered milled zirconia disc. (B). Volumetric zirconia disc shrinkage following sintering procedure [87]

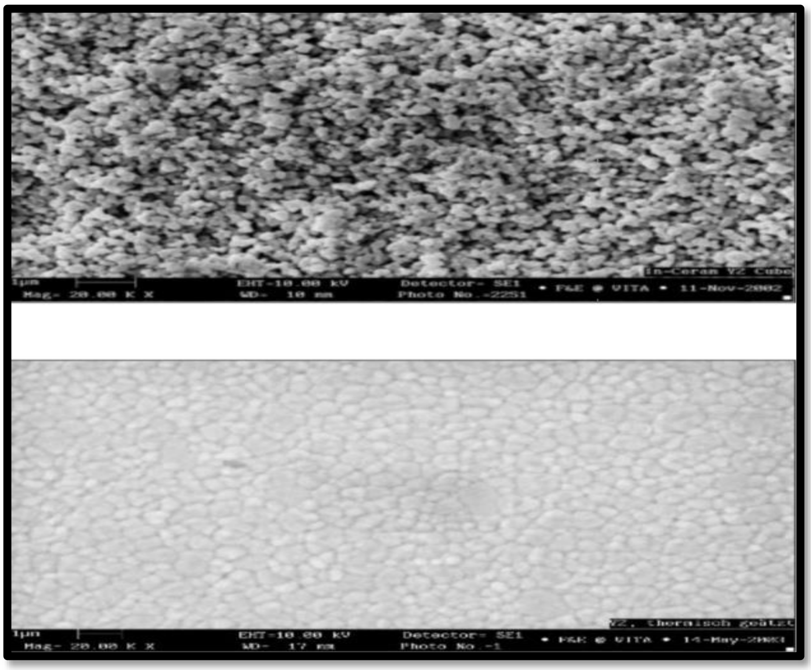

Figure (1-4): Scanning electron microscope of presintered zirconia (above) and after sintering (below). (source: vita in-ceram YZ manual).

1.6.2 Hard machining technique

Involves the fully sintered zirconia blocks which are generally produced by hot isostatic pressing (HIP) at 1400°-1500 °C [28]. Hipped zirconia (known as white blocks) material, is in it‟s final high strength, and is characterized by a constant grading and better homogeneity. It has the highest flexural strength compared to several presintered zirconia materials tested after sintering [88]. This approach eliminated the problem of post-milling shrinkage, since neither oversizing nor sintering are necessary. Nevertheless, hard machining needs longer milling time, and more complex manufacturing, presented as; higher costs from accelerated machinery production wear. Moreover, zirconia frameworks, right after hard machining, experiences certain amount of monoclinic phase transformation due to mechanical stress from the friction of the working burs and the overheated machined zirconia material. With the increased susceptibility to LTD.

The choice is mainly guided by shape, volume, and complexity of the prosthetic geometry as well as available time and cost of the milling procedures [28].

1.7 Luting of zirconia

Glass ionomer (GIC) and resin-based cement are the primary choices for bonding ceramic restorations to the underlying tooth structure, mostly because these cements are very easy to use [89]. Adhesive cementation of indirect restorations provides many benefits such as: enhancement of marginal adaptation, reducing micro leakage, improving retention and fracture resistance [21]. The success of indirect restorations is greatly dependent on the efficient adhesion at the restoration-cement-tooth interfaces [90]. Variations of the chemical formulae of self-adhesive resin cement played an important role in determining the micro tensile bond strength to the ceramic materials [91]. The most favorable and effective cement for all types of ceramic restorations is the Physio-chemical interaction of the resin-based composites containing 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP) monomer [89]. Zirconia resin bond strength changes by several factors, such as surface treatment and wettability of resin cement [92].

1.8 Surface roughness

In order to optimize the zirconia bonding efficacy, micromechanical and/or chemical surface treatments should be used. Different surface roughening techniques and adhesion promoting agents were applied [93]. Zirconia inertness and low surface energy [94], is the result of absent glassy phase in zirconia structure. This makes it resistant to hydrofluoric acid etching. For this reason, conventional surface preparation methods are ineffective for zirconia ceramics[95], and alternative techniques are necessary [96]. Roughening may include grinding with diamond rotary instruments [97], air abrasion using aluminum oxide particles, silica coating, selective infiltration etching, laser [98], Nano alumina coating [13], or combination of any of these procedures [99].

1.8.1 Mechanical methods

Surface roughness is an important factor for adhesion [98]. Micromechanical attachment is one of the key mechanisms for a reliable adhesion to dental hard tissues and restorative materials [100]. Aggressive mechanical surface abrasion methods that lead to surface flaws and reduced strength of the material are neccessory to roughen the zirconia surface [101]. Surface treatment methods ranging from air abrasion with aluminum oxide (Al_2O_3), silica coating with silica-modified Al_2O_3 particles [102], and laser applications, are frequently used on Y-TZP restorations. Surface treatments tend to accelerate surface degradation. Areas with residual scratches and defects may also promote an additional t-m phase transformation [103]. Al_2O_3 air abrasion is one of the common procedures used for increasing surface roughness [104], that superior bonding to zirconia was obtained with this method. Studies showed that sandblasting did not have a significant effect on improving the bonding of resin cement to zirconia surface [105]. It may cause flaws and phase transformation that accelerate the micro-crack formation and could altere zirconia mechanical properties [15]. Tribochemical silica-coating (silicatization) is a widely used conditioning method for ceramic cementation [15]. It has been used to create a silica layer on ceramic surfaces through the high-speed surface impact of the silica-modified alumina particles that can penetrate up to (15 µm) into ceramic substrates [89], without any application of additional heat or light [106]. It was reported that infiltrating fused glass micro-pearls, to the ZrO_2 surface, increases the bond strength of resin cement to ZrO_2. The fused glass film enhanced surface roughness and increased micro-retention, throught the hydrofluoric acid etching. The silica- rich film also allows for silanization of ZrO_2 before bonding [107].

1.8.2 Chemical methods

Etching ceramic surface with hydrofluoric acid prior to silane coupling agent, reported a proved success [21]. In case of zirconia, the use of coupling agents like silane can be adopted only after a tribochemical processing or after infiltrating the zirconia surface with a thin layer of glassy ceramic. However, the latter approach could create excessive ceramic thickness. Also the adhesion efficiency between glassy matrix and polycrystalline network still remained unclear [108]. Selective infiltration etching process, changes the dense, non-retentive, low energy, relatively smooth zirconia surface, to a highly active, well bonding surface [109]. Hot etching technique could form an ideal surface roughness for zirconia. It proved to be producing a higher shear bond strength of zirconia to resin cement compared to air born-particle abrasion, while avoiding the T-M surface phase transforming [110]. However, [111] showed that, this method created unfavorable deep grooves which decreased the bonding strength. The 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP), which is an Acidic monomer, is known to increase the bonding strength of zirconia ceramics, especially when used with Al_2O_3 airborne-particle abrasion or tribochemical coating. MDP silane- coupling agents, provide siloxane bonds, which are required for chemical bonding, resulting in increased zirconia-resin cement bond strength [101]. Gas plasma creates chemically active areas between resin and zirconia [112]. It has the advantage of producing a reactive surface without causing any physical trauma on zirconia surface [113].

1.8.3 Surface roughness by laser

The most important interaction between laser light and the substrate is the absorption of laser energy by substrate [114]. Lasers have been introduced to modify surfaces of the materials in relatively safe and easy means [99]. Treating the surface of zirconia with lasers has shown to be an effective way to achieve a higher bond strength [115]. Er:YAG, Nd:YAG, CO_2, and Er,Cr:YSGG lasers have been proposed as an alternative surface treatment to condition the surfaces of dental materials [104]. The wavelength of Er:YAG (2940nm) and Er,Cr:YSGG (2780nm) lasers are considered similar, so their results could be compared [116]. Some studies reported that, in comparison with control group, the application of Er:YAG, Diode, and CO_2 lasers, increased the bond strength between resin cement to zirconia ceramic [104].

The Er:YAG laser irradiation of zirconia surface, was found to increase the surface roughness, with no microcracks observation, with increasing the shear bond strength of ceramic to dentin [115]. Whereas [117], studied the Er:YAG laser irradiation effect with water coolant on zirconia and found that laser treatment decreased the bonding strength of resin to zirconia framework. Nd: YAG laser was also used to roughen zirconia and feldspathic ceramic. Nd: YAG laser treatment caused surface changes due to the micro -explosions resulting in the formation of voids and melting of the most superficial ceramic layer followed by solidification to form a smooth blister-like surface [118]. Nd YAG laser irradiation of zirconia causes color change to black with many cracks with reduced oxygen content [119].

CO_2 laser wavelength (10600 nm) is well absorbed by ceramic materials. Temperature increase and surface ablation due to laser absorption by ceramic, create some surface porosity, inducing an increase in the micromechanical retention of resin cement to zirconia ceramic [104]. Sudden temperature changes could create internal tensions that might affect the bond strength [99].
fractional CO_2 laser application can improve the bond strength of resin- zirconia ceramic [18].

1.9 Laser

A laser is a device that emits light through a process of optical amplification based on the stimulated emission of electromagnetic radiation. The term "laser" originated as an acronym for "light amplification by stimulated emission of radiation" [120]. The first theoretical foundation of laser was given by Einstein in (1917) using Plank"s law of radiation that was based on probability coefficients (Einstein coefficients) for absorption and spontaneous and stimulated emission of electromagnetic radiation [120]. The first laser was built in (1960) by Theodore H. Maimane, based on a theoretical work by Charles Hard and Arthur Leonard [120]. Basically, every laser system essentially has an active/gain medium, placed between a pair of optically parallel and highly reflecting mirrors with one of them being partially transmitting, and an energy source to pump the active medium. The gain media could be solid, liquid, or a gas medium, having the property of amplifying the light wave amplitude, whenever passing through it by the stimulated emission, Pumping mechanism may be electrical or optical. The gain medium being placed between pair of mirrors in such a way that light oscillation between mirrors passes each time through the gain medium. After attaining considerable amplification, the laser light finally emits through the transmitting mirror, as shown in figure (1-5) [121].

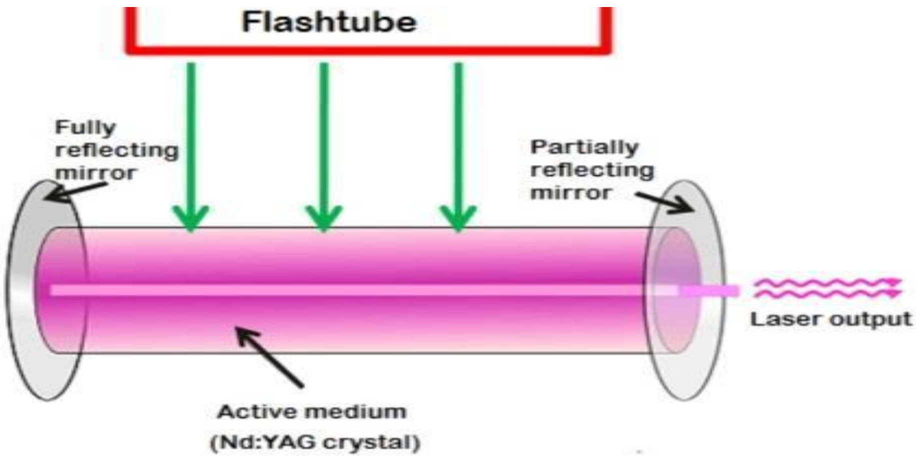

Figure (1-5): Laser active medium [122].

1.9.1 Properties of laser

The laser output beam has a group of properties:

- Coherency: That the laser light waves are physically identical in their amplitude and frequency. Coherence causes collimation of a laser beam over an extremely large distance. allowing the beam to be extremely finely focused. Any given laser beam can be focused to a final diameter that equals the wavelength of the specific laser[123].
- Monochromatic: Lasers expresses one color, or specifically

have a single wavelength from UV to infrared. Lasers, of varying types, emits an individual wavelength [123].

- ❖ Collimation (directionality): Laser beam travels parallel to each other. The emitted beam has a constant size and shape.
- ❖ Brightness: This property arises from the parallelism or collimation of the laser light as it moves through space, maintaining it's concentration. The high brightness factor translates to high concentrations of energy when the laser is focused on a small spot. It means it is concentrated enough to be dangerous [123].

1.9.2 Lasers in dental practice

Can be classified by various methods: According to the used active medium, according to tissue applicability, according to the range of wavelength, and the risk associated with the application.

❖ Diode Laser

The active medium is a solid-state semiconductor, made of aluminum, gallium, arsenide, and occasionally indium [124]. Which produces Invisible, near-infrared laser wavelengths, ranging from (375-2000 nm). They can function with continuous wave or gated pulse modes. These lasers are used for laser-assisted tooth whitening and adjunctive periodontal therapy as they exhibit bactericidal capabilities and specific soft tissue procedures. This laser is only a soft tissue [125]. They are poorly absorbed by the hydroxyapatite and water present in the enamel and bone [126].

❖ CO_2 laser

Is a molecular laser that uses gas molecules (combination of carbon dioxide, nitrogen, and helium) as the active medium [127]. This laser emits light at different wavelengths (9,300, 9,600, 10,600 nm) in the far-infrared region of the electromagnetic spectrum. The traditional CO_2 (10600 nm) is for soft tissue uses [128], with a very shallow depth of penetration. CO_2 laser is an excellent tool for incising tissue for multiple purposes with good hemostasis. A (9300 nm), hard tissue capable CO_2 laser wavelength, has become available [124].

❖ Nd:YAG laser

Is a solid-state laser that uses dopants (Neodymium (Nd 3+) dispersed in a crystalline matrix (a complex crystal of Yttrium-Aluminum-Garnet (YAG), to generate a free-running pulsed laser light of an output wavelength of 1064 nm in the near-infrared region of the spectrum [127]. It is being highly absorbed in melanin, less absorbed in hemoglobin, and slightly absorbed in water, making it an effective surgical laser for cutting and coagulating dental soft tissues, for a good hemostasis [129], and a nonsurgical sulcular debriding tool, used in periodontal disease control [130].

❖ Erbium lasers

Er: Cr YSGG (2,780 nm) laser wavelength, has an active medium of yttrium-scandium-gallium- garnet, doped with erbium and chromium ions. And Er:YAG (2,940) laser wavelength, has the active medium of yttrium- aluminum-garnet doped with erbium ions [128]. Both wavelengths have high absorption in water, collagen and hydroxyapatite [131]. In general practice, Er,Cr:YSGG is used for soft tissue lesions, tooth structure, and bone [132]. Er,Cr:YSGG laser is popularly used for cavity preparation. It is capable of producing surface roughness comparable to that produced by acid etching of enamel and dentin surface [133]. Er:YAG laser device was cleared for marketing by the U.S. FDA in (1997) for certain hard and soft tissue procedures, such as caries removal and cavity preparation, as well as incision and excision of intraoral soft tissues. Other Er:YAG laser instruments were then cleared for sulcular debridement in (1999), and in (2004) for osseous surgery [134]. For both lasers, many procedures can be done without local anesthesia [135]. The smear layer is virtually eliminated, with significant disinfection effect on the enamel and dentin to be restored [136]. The novel concept of fractional photo hemolysis (FP) was first introduced in (2003), BY Huzaira et al, in it‟s basic applications [137]. In (2004) and (2005) the first full reports and applications, by Man stein et al. followed [138]. It is a non-invasive treatment that uses a device to generate thousands of microscopic treatment zones (MTZ) by scanning the target area. [139]. The MTZs are usually smaller than (400 µm) in diameter and can penetrate up to varying depths (1,300 µm), depending on the wavelength, pulse energy, and the chosen device [138]. Fractional technologies can be divided into two main categories, based on the wavelength‟s affinity for water: Those devices with wavelengths that are highly absorbed by water are termed ablative, which include both (Er:YAG; 2,940 nm) and (Er,Cr:YSGG; 2,790 nm) and (CO_2; 10,600 nm) lasers. And non-ablative wavelengths: (Er:glass and gallium arsenide (GaAs) lasers at 1540 nm) [140]. CO_2 fractional lasers, have been introduced since (2007), and fractional versions of Er:YAG lasers have also followed [141].

1.9.3 Laser ceramic material interaction

In case of ceramic materials, the laser-material interaction is considered a more difficult than those of metallic material, since higher energy is required for electrons excitation from the valence band. Therefore, short and ultra-short pulsed laser with high peak power are indicated [142]. The outcome of laser material processing is highly governed by laser power, interaction time, pulse width, wavelength, energy density, the material"s coefficients of reflection and absorption, boiling and melting point, and surface shape [143]. Different physical phenomena occur on material"s surface, once the laser beam strikes, including: reflection, absorption, scattering, and transmission.

❖ **Absorption**: Is the interaction of electromagnetic radiation with electrons in the material. It is the most required one in laser material processing [127]. Absorption of laser energy by the material, depends on:

1- Optical and thermal properties of substrate material.

2- Laser parameters, including: wavelength, mode of operation and power [144].

3- The orientation of ceramic surface with respect to the beam direction, that reaches it"s highest value with an incident angel above 80 degree [145]. Absorption of laser energy causes heat generation on the workspace, subsequently, triggering the desired effect. Heat dissipation into core material results in heat affected zone, presenting as cracks [146].

❖ **Scattering (diffusion:)** The change in direction of a light wave on a single or multiple occasions, during interaction with a small particle or object, within inhomogeneous and/or turbid material. That the laser light is transformed from a narrow collimated beam to a broad, diffused one. The quantity of scattering depends on the wavelength, and relative particle sizes[147].

- ❖ **Reflection:** The bouncing of the laser beam off the surface, with no interaction or penetration at all. It is usually an undesirable effect since the energy could be unintentionally redirected to a target, such as the eyes, which is a major safety concern for laser operators.
- ❖ **Transmission**: The propagation of laser energy through an object, without affecting it. It is highly dependent on laser wavelength. Only the non-absorbed, non-reflected, forward scattered photons, will be transmitted through material [147].

1.9.4 Thermal effects

In laser material processing, the material can be removed by three different ways: A. Melting, B. Vaporization (both ways are categorized as thermal processing), and C. Chemical degradation. As for the thermal way, the intensity of absorbed laser beam must be high enough to generate the required thermal energy for melting and vaporization of material. While, a direct bond breakdown take place after absorption of incident laser beam, in the chemical way [148].

- **Melting**

At high laser power densities, the surface temperature increases, with increasing irradiation time. When the temperature of ceramic surface reaches the melting point, the material removal is facilitated [149].

- **Vaporization and plasma formation**

As the temperature of the ceramic surface reaches the boiling point, further increase in laser power density or pulse duration, removes the material by evaporation instead of melting. After the start of vaporization, the liquid- vapor interface moves further inside the material, with supply of laser energy. The degree of ionization is an important parameter, which gives an indication whether plasma will be formed during the machining process. Accordingly, necessitate efforts to be taken, to overcome the harmful effects of plasma [149]. When the laser energy density exceeds a threshold limit, the material immediately vaporizes, then ionized forming plasma, with temperatures as high as 50,000 K and pressure up to 500 Mpa [150]. This thermal stress could result in cracking. So optimized parameters should be set. Crack formation can be avoided by precluding the formation of a plasma induced-intense laser [151].

- **Ablation**

When the material is exposed to a sufficiently large incident laser energy, the temperature of the material`s surface exceeds the boiling point, causing rapid vaporization and subsequent material removal by a process referred to as thermal ablation [152]. Ablation take place when laser energy exceeds a characteristic threshold of the processed material. Energy above ablation threshold facilitate the material removal by bond breaking. Whereas thermal effects take place with energy below ablation threshold. Absorption properties of the ceramic and incident laser parameters determine the location at which the absorbed energy reaches the ablation threshold, thus determining the depth of ablation [149].

1.9.5 Laser hazard classification

According to the American national standard institute (ANSI) & to the occupational safety and health administration (OSHA) standards: CLASS DESCRIPTION

- **CLASS I**: Low powered lasers that are safe under all conditions. Eg: Nd: YAG Laser used in a dental laboratory.
- **CLASS IIa:** Low powered lasers, in the visible portion of spectrum (0.4-0.7µm), that are hazardous only when viewed directly for longer than 1,000 s. eg: Visible red aiming beam of a surgical laser.
- **CLASS IIb**: Low powered visible lasers which have a dangerous viewing time of one-fourth of a second.
- **CLASS III:** Medium powered lasers (0.5W maximum). Direct viewing is hazardous to the eye. eg: Low power diode laser used for biostimulation.
- **CLASS IV:** High powered lasers (> 0.5W), visible and invisible. produces ocular, skin and fire hazards, from direct or indirect viewing. may produce hazardous diffuse reflections. eg: All lasers used for cutting and drilling in oral surgery, whitening and cavity preparation [123].

1.9.5.1 Types of laser hazards

1- **Environmental hazards**: Inhaled airborne contaminants, emitted in the form of smoke or plume, during laser-material thermal interaction or during the accidental escape of toxic chemicals and gases from the laser itself, may be hazards to the respiratory system, for both dentist and patient. Most surgical lasers in dentistry are capable of generating the plume. Ablation of infected tissue poses an even greater hazard due to the possible presence of infectious agents like HIV within the plume. Different chemicals are found after soft tissue laser irradiation, like formaldehyde, climates, acrolein, cyclohexane, acetone, xylene, etc. The Avoidance of these hazards is by wearing surgical masks, using high volume evacuators, and using surgical smoke evacuation equipments[123].

2- **Electrical hazard**: Because class IV surgical lasers often use high electrical current, there are several electrical hazards involved , like; electrical shock hazard, electrical fire hazard, and explosion hazard. Insulation circuit, shielding, grounding, and housing of high voltage electrical components provide proper protection from electrical injury. The clinicians should never attempt to repair or remove safety panels from the laser [123].

3-**Skin hazard**: The potential for skin damage through class IIIB and IV laser exposure, is related to the ablation threshold of skin structure and incident laser energy. Subablative power levels will pose little threat (reversible tissue warming). Visible and near-infrared wavelengths (400-1400 nm) have the potential to pass through the epidermis into superficial and deeper structures respectively. Mid to far-infrared wavelengths (1400- 10,600 nm) will interact with surface structures. The governing factor of the structural damage is the absorption potential of the laser wavelength, relative to the tissue elements (chromophores) such as pigment (shorter wavelengths) and water (longer wavelengths), together with the power density, duration of laser exposure, and spot size [153].

4- **Fire hazard**: The high temperature with use of class IV and certain class IIIB lasers can either cause ignition of material and gases or promote flash- point ignition. Therefore, the use of aerosols, alcohol-soaked gauze, and alcohol-based anesthetics are to be avoided [154].

1.9.6 Laser controlling area warning signs

The aim of warning signs, is to deliver a rapid and visual hazard- alerting message to the others, that there is a laser system hazard in the area. So many protocols should be followed, Including [155]:

- ❖ The laser system should be positioned in a side-room, so there is more than one door before the system.
- ❖ Warning signs should be big, colorful, and in an obvious position.
- ❖ Knocking before entering.
- ❖ Light signs should be illuminated during usage.
- ❖ Laser eye protection wears, should be available.
- ❖ The area should be restricted for authorized persons only.
- ❖ The operator should have enough training about the system parameters and uses.

1.10 Literature Review of Zirconia Surface Roughening Using Different Laser Types

In 2014, Ghasemi A, et al [156] evaluated the effect of Er,Cr:YSGG laser treatment on microshear bond strength of zirconia to resin cement before and after sintering. And compared that effect with (50 µm) alumina powder air abrasion surface treatment and concluded that: Laser treatment of presintered Y-TZP cannot be recommended for the bonding improvment. Although sandblasting of sintered Y-TZP yielded better results than the rest of the groups, Er,Cr:YSGG (3 W) laser power postsintering can also be effective in enhancing the bonding strength of resin cement to zirconia..

V. A. Zanjan et al. 2014 [157] evaluated the effect of sandblasting, CO_2, Er,Cr:YSGG lasers, in zirconia ceramic surfaces roughening for enhancing the bond strenght of resin cement to zirconia. CO_2 laser at (4 W), and Er,Cr:YSGG laser at only (3 W) output power can be considered as surface treatment options for roughening zirconia surface for gaining a better bond strength to resin cement.

Dede D. Ö. Et al 2016 [158] investigated the effect of CO_2 and Er:YAG laser irradiations on bonding strength of sintered zirconia ceramic to resin cement. And concluded that: CO_2 and Er:YAG laser irradiation techniques could increase the shear bond strength values of the tested zirconia ceramics.

Thereby, it could be recommended for clinicians as an alternative pretreatment technique.

L. C. Zeidan et al. 2017 [159] evaluated the effect of different output powers of Er,Cr:YSGG laser in comparison with tribochemical silica coating, on the bond strength between zirconia ceramic to two resin cements. And concluded that: The lowest tested power was suitable, which showed bond strength values similar to tribochemical silica deposition. And the light curing is important to adhesion.

G. E. Kunt et al. 2018 [160] analyzed the surface roughness of (Y-TZP) ceramic after different laser treatments (CO_2, ER:YAG) and concluded that: Surface roughness of zirconium oxide ceramic was increased with CO_2 laser.

L. R. Sofi et al. 2018 [161] investigated the effect of Er:YAG laser, sandblast and several types of universal bonds on shear bond strength of zirconia to composite resin. their results showed: Er:YAG laser is a more appropriate method for increasing bonding strength as compared with sandblasting,

J. Saade et al. 2020 [162] evaluated the effect of different surface treatment on resin-zirconia bonding. The use of Er,Cr:YSGG laser for zirconia surface treatment with predetermined parameters (2 minutes at a power of 5.5
W, 20 Hz with 100 mJ energy) yielded a useful non-destructive surface roughening method. The Er,Cr:YSGG laser increases zirconia surface roughness and thus, enhances surface wettability for a better adhesion to resin cement.

R. Kara. 2020 [163] evaluated the effect of Er,Cr:YSGG laser surface treatment method on the shear bond strength of resin cement to zirconia ceramic. Resulting in highest values for the laser abrasion group. Although laser group was not statistically significant from the silica coated group, the sandblasting group and the acid etching group were statistically different from other groups. Conclusion: Er,Cr:YSGG laser and silica coating treatments can be an alternative method for enhancing the bonding of resin cement to dental zirconia ceramic.

Elkallaf et al. 2020 [164] investigated the effect of alternative surface treatments to enhance the zirconia-resin bonding. Results: the highest shear bond strength was recorded for (Hand grinding) while the lowest mean value was recorded for control group. Conclusions: Treatment of zirconia with CO_2
and Er:YAG lasers increased shear bond strength of zirconia to resin cement, with the CO_2 laser values of the group being higher than Er:YAG laser.

Tarek et al. 2020 [165] evaluated the effect of different surface conditioning methods on the shear bond strength of zirconia ceramic to resin cement and their results were: The mean shear bond strength values for the control, silica coating, sandblasting, and laser groups were 10.37± 0.92, 15.99
± 2.01, 12.88 ± 0.98, and 18.87 ± 1.17 MPa respectively. Conclusions: The use of Nd:YAG laser irradiation increases the bond strength of bonding resin cement to zirconia ceramics. Zirconia ceramic sandblasting treatment alone is not effective for achieving good bond strength.

Table (1-1): The effect of different types of lasers on the shear bond strength (SBS) of resin cement to zirconia ceramic

Reported Study	Laser Type	Mean SBS ±SD(Mpa) of Laser Group	Mean SBS ±SD(Mpa) of Sandblasting Group	Mean SBS ±SD(MPa) of Control Group
Unal et al, 2015 [98]	YbPL (1064nm)	11.03 ±1.08	3.55 ±0.78	3.54 ±1.28
Aras et al, 2016 [176]	Er,Cr:YSGG (2780nm)	6.6 ±5.9	9.1 ±3.1	3.6 ±1.9
Saygin et al, 2017 [166]	Nd:YAG (1064nm)	44.7 ±10.5		48 ±10.1
	Er:YAG (2940nm)	40.3 ±11.8		
Ahmed et al, 2018 [167]	CO_2 (1060nm)/ Sandblast/Silan	1.56	Sandblast /Silan	
			0.92	
Elkallaf et al, 2020 [164]	CO2 (1060nm)	11.5 ±1.7	14.8 ±5.4	4.8 ±1.43
	Er:YAG (2940nm)	7.4 ±1.43		
Abdelrehim et al, 2020 [165]	Nd:YAG (1064nm)	18.8 ±1.17	12.8 ±0.98	10.37 ±0.9
Dawood et al, 2020 [168]	Er,Cr:YSGG (2780nm)	4.68 ±0.32	7.5 ±0.38	1.66 ±0.29

Chapter Two

Materials and methods

This chapter includes a detailed description of all the materials and equipments used in the present study, with the methods used to perform this study.

2.1 Materials

The following table (2-1) contains all of the materials that were used for the current study

Table (2-1): Materials and devices used in the current study

Materials	Company	Origin
Zirconium blanks vita YZ HT 98.4 / h 10 mm	Vita Zahnfabrik, lot no. 75330	Germany
Relyx U200 self-adhesive resin cement	3M ESPE, lot no. 6095231	Germany
Silicone apparatus for resin cement molding	Technician Customized	Iraq
Cold cure acrylic	FORMED, lot no. 51/992	Poland
Teflon molde holding zirconia specimens	Technician Customized	Iraq
Deionized distilled water	Pioneer	Iraq
Alkohol	Al-Kafeel	Iraq
Covered Plastic container	Imidro	Iran

2.2 Equipments

The following table (2-2) contains every used equipments for the current study:

Table (2-2): Equipments used in the current study

Equipment	Company	Origin
Er,Cr:YSGG laser system	Waterless I Plus, Biolase Technology	USA
Sintering zirconia furnace	Zirkonzahn, oven 600/V2, , serial no: 003V1180050AC-2018	South Tyrol
LED light curing system	Ivoclar Vivadent, 220-240V, 50-60 Hz	Liechtenstein
universal testing machine	Instron-1195, 2-53/2/116, serial no. H3166	England
Stop watch	sony	Japan
Ultrasonic cleaner	CD-4820	China
Digital camera	Sony	Japan
CAD-CAM milling machine	Zirkonzahn, type: M1, serial no:001M1180230BK-2018	South Tyrol
Atomic force microscope	Anstgrom Advanced Inc., AA3000	USA
Stereo microscope	Euronext, ME, 2665	Holland
Digital water bath	Labtech, Daihan Labtech	Korea
Scanning electron microscope	Tescan, Vega	Czechia
Pumped sputter coater	Quorum Tec, Q150R Rotary-Pumped Sputter Coater	UK

2.3 Laser system

Er,Cr:YSGG laser system, shown in figure (2-1) (Waterless I Plus, Biolase Technology, USA), has the following specification:

- Laser wavelength: 2780 nm.
- Total output power: up to 9W at 15 Hz
- Pulse duration.
  1. Hard tissue 'H' mode: for 60 μs
  2. Soft tissue 'S' mode: for 700 μs
- Energy per circular spot: 10-40 mJ
- Spot diameter: 200-300 μm
- Frequency: 5-100 Hz
- Pulse energy: 0-600 mJ
- Mode: multimode
- Aiming beam: 635 nm (red) laser, 1mw max (safety classification I)
- Fluence per spot: 20-120 J/cm^2
- Depth of one crater: up to 0.5 mm

Figure (2-1) Er,Cr:YSGG laser device

2.4 Method

Zirconia disc samples were obtained by the following procedure:

2.4.1 Construction of zirconia specimens

Presented zirconium oxide blocks were milled into (124) zirconia discs, figure (2-2) (Vita YZ HT Zahnfabrik/Germany), of the physical and chemical properties that are listed in tables (2-3) (2-4), to obtained the desired dimensions: (11.5mm diameter, 2.5mm height). Then sintered in a special furnace of zirconium oxide, figure (2-3) (Zirkonzahn, type: oven 600/V2-2018) at 1450 °C for 8 hours including cooling, following the manufacturer’s instructions, to the final disc

measurements, approximately: (9 mm diameter, 2 mm height). During sintering process, 3-dimensional volumetric shrinkage of the milled discs, of approximately 25%, took place. Explaining why the discs milling were approximately 25% larger in volume.

Figure (2-2): VITA YZ HT zirconium oxide block (zahnfabrik/Germany)

Figure (2-3): sintering furnace

Table (2-3): VITA in-cream YZ/VITA YZ HT physical properties

property	Unit	Value
Coefficient of thermal expansion – CTE (20 – 500°C)	10-6 K-1	10.5
Chemical solubility (ISO 6872)	µg/cm2	<20
Density after sinter firing	g/cm3	6.05
Flexural strength (ISO 6872)	MPa	>900

Table (2-4): VITA in-cream YZ/VITAYZ HT chemical properties

Components [Wt %]	VITA YZT	VITA YZ HT	VITA YZ ST	VITA YZ XT
ZrO_2	90-95	90-95	88-93	86-91
$Y_2 O_3$	4-6	4-6	6-8	8-10
HfO_3	1-3	1-3	1-3	1-3
$Al_2 O_3$	0-1	0-1	0-1	0-1
Pigments	0-1	0-1	0-1	0-1

Following sintering, each zirconia disc was measured (9mm diameter, 2mm height), shown in figure (2-4), by using the vernier caliper, figure (2-5).

Figure (2-4): CAD-CAM milled zirconia samples

Figure (2-5): Digital vernier

The bonding surfaces of zirconia discs were then polished consecutively with 600, 800 (at 5.000 rpm), 1000 and 1200 (at 10.000 rpm) grit silicon carbide abrasive papers with water coolant to standardize all sample surfaces. All specimens were whipped with a cotton and alcohol then ultrasonically cleaned in distilled water and 70% alcohol for 3 min., figure (2-6) to remove any contaminants and dried naturally in the atmosphere [169]. Then all the specimens were examined by an optical microscope, shown in figure (2-7) at a magnification power of 10X, for cracks , pits or/and fissures. Two samples, one with fissures and the other with pits, were substituted by other perfect ones.

Figure (2-6): Ultrasonic cleaner

Figure (2-7): Optical microscope

Specimens were divided into groups, as illustrated in figure (2-8):

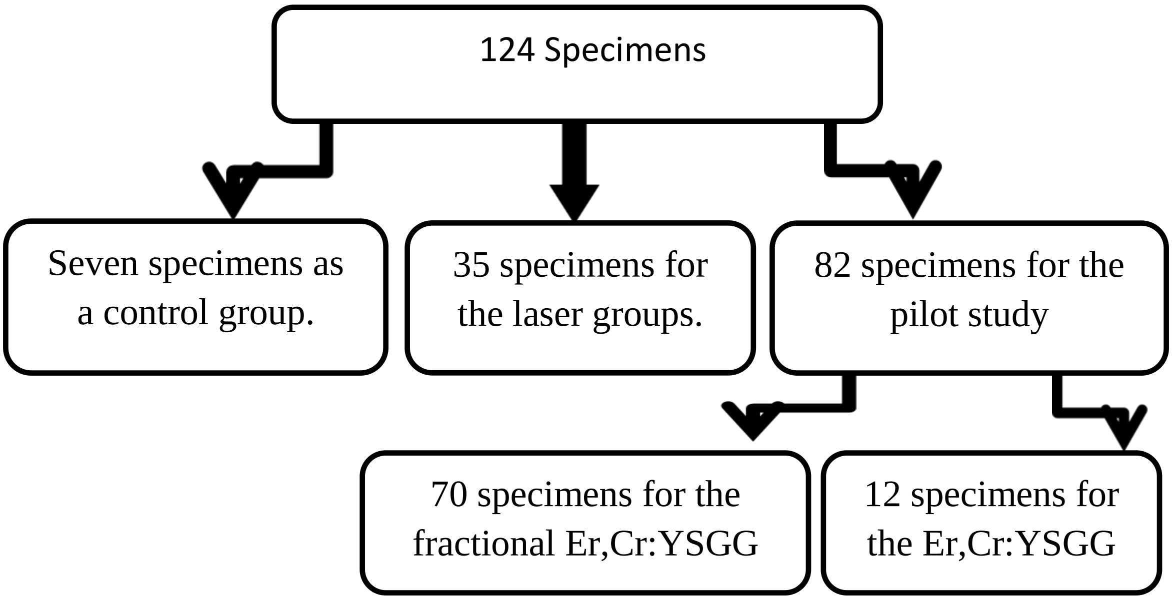

Figure (2-8): Zirconia specimens grouping

2.4.2 Construction of teflon mold

A teflon mold was constructed, by a technician, for holding the zirconia discs. The mold was designed as a cylinder with (5 cm) in height and (3 cm) in diameter shown in figure (2-9).

Figure (2-9): Teflon mold

2.4.3 Construction of the rubber mold

A mold of alumina was used for the construction of a circular silicon rubber mold with an external diameter of (30 mm and 2.5 mm) in height. In the middle of the rubber mold, there was a circular opening with a (5 mm) diameter for molding of the resin cement. The opening was surrounded by another circular border that encountered the zirconia disc with (0.5 mm) depth in order to fix the rubber mold over the specimen. The outer border of the rubber mold was surrounded by stainless steel ring for handling, as shown in figure (2-10).

Figure (2-10): Silicone mold with central opening for resin cement molding

2.5 The Experimental work

In order to optimize the laser effect during the experimental procedure for the laser groups, a pilot study was needed. And it is detailed as follow:

2.5.1 The pilot study

The Er,Cr:YSGG laser has different parameters that need to be modified during specimens laser irradiation, for the purpose of finding the optimum laser parameters to produce the desired effect on the zirconia sample surfaces with maximum shear bond strength. These parameters included the power setting, pulse duration, pulse repetion rate, as shown in figure (2-11).

Figure (2-11): Control panel of the Er,Cr:YSGG laser device

Laser energy was delivered as five separated irradiation zones, at a circular area of (6 mm) diameter in the middle of each (12) specimens. One spot of irradiation being centered in the circular area. Two upper spots, and two at the lower, each at the periphery of the 6 mm area, forming a square shape of laser pulses. Gold handpeice with 600 μm quartz core tip was held manually, perpendicular and (1 mm) distant from the sample surface (focused mode), which was fixed in a teflon mold constructed to engage the specimen. After laser irradiation procedure, all zirconia disc specimens were whipped with a cotton and alcohol, then ultrasonically cleaned with distilled water and 70% alcohol bath for (3min) before being examined under the optical microscope for determination of the most effective parameters that are to be used. The water and air flow levels were determined based on a previous study [170]: 65/55 %.

12 zirconia specimens were randomly divided into three groups:

1- Group of laser pulse repetition rate: Four zirconia disc specimens were irradiated, each with the following parameters: (20s, 55% air flow, 65% water flow, 60 μs), and with four different laser pulse repetition rate (20, 30, 40, 50) Hz, and four corresponding fluences, as shown in figure (2-12). The (50) Hz, at 21.4 J/cm^2 specimen had the deepest pulse depth value (3 μm) with no evident laser optical damage.

Fixed laser parametes

Pulse duration (µs)	60
Irradiation time (s)	20
Air flow %	55
Water flow %	65

No. of samples	pulse repetition ratee(Hz)	Fluence (J/cm^2)
1	20	53.5
2	30	35.7
3	40	26.7
4	50	21.4

Figure (2-12): Pulse repetition rate group specimens for their applied parameters

2- Group of laser irradiation time: Three zirconia disc specimens were irradiated, each with the following parameters: (21.4 J/cm^2 , 50 Hz, 55% airflow, 65% water flow, 60 µs), and with three different laser irradiation times per second (10 s, 15 s, 20 s) as shown in figure (2-13). The (20 s) laser irradiation was when the laser pulse effect became obvious.

Fixed laser parameters

Pulse duration (µs)	60
fluence (J/cm^2)	21.4
pulse repetition rate (Hz)	50
Air flow %	55
Water flow %	65

No. of samples	Irradiation time (s)
1	10
2	15
3	20

Figure (2-13): Irradiation time group specimens for their applied parameters

3- Group of laser power: Five zirconia disc specimens were irradiated, each with the following parameters: (50Hz, 20s, 55% airflow, 65% water flow,60 µs), and with five different laser fluences (3.5, 5.35, 7.1, 21.4, 35.7) J/cm^2, shown in figure (2-14).

Fixed laser parameters

Pulse duration (µs)	60
Irradiation time (s)	20
pulse repetition rate (Hz)	50
Air flow %	55
Water flow %	65

No. of samples	Fluence (J/cm^2)
1	3.5
2	5.35
3	7.1
4	21.4
5	35.7

Figure (2-14): Laser fluence group specimens for their applied parameters

The (7.1) laser fluence was when the laser pulse starts it's effect on the zerconia surface as shown in fig. (2-15) A- B.

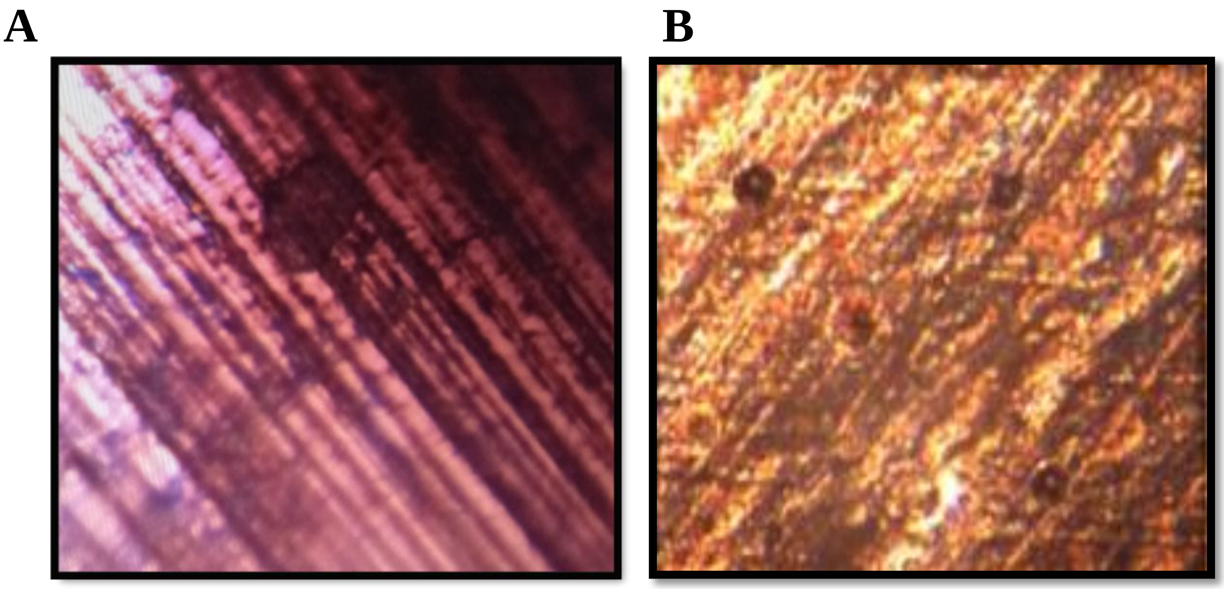

Figure (2-15) A-B: Microscopical view (100 mag.) of the Er,Cr:YSGG laser treated specimens with: (50Hz, 20s, 55% air flow, 65% water flow,60 µs), for two different laser fluence (**A**. 7.1 J/cm^2). (**B**. 21.4 J/cm^2)**.**

The pilot study showed that the most suitable laser parameters were (50 Hz. 65/55% water/air flow) to be fixed in each group. And the (7.1J/cm^2, 20s) laser parameters were when the laser irradiation effect started to show microscopically on the zirconia surface and become effective.

2.5.2.The fractional Er,Cr:YSGG pilot study

70 zirconia specimens were laser irradiated with the provided fractional head of the Waterlase I Plus Er,Cr:YSGG device. Each zirconia specimen was fixed in the teflon mold with the fractional head of (9 mm width, 2mm hight) being adapted in contact with the polished zirconia surface, perpendicular to it's horizontal plane. This was facilitated by the rectangular shape of the laser output opening of the handpeice, that allowed itself to be easily supported.

The 70 zirconia specimens were randomly divided into two main groups: 60 µs pulse duration group of 35 specimen, and 700 µs pulse duration group of 35 specimen. Each group was further sub divided into five groups and as illustrated in figure (2-16).

All samples in this fractional laser pilot study groups, was irradiated with the fixed laser parameter (50/10 % water/air level)

35 specimens for 60 µs pulse duration groups

↓

Two groups

each group of seven samples

(60 µs) groups	Irradiation time (s)	repetition rate (Hz)
1)Group Frac/D	30	50
2)Group Frac/E	40	50
Each group with seven different laser fluences (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2		

(60 µs) groups	Irradiation time (s)	repetition rate (Hz)
1)Group Frac/A	20	25
2)Group Frac/B	30	25
3)Group Frac/C	40	25
Each group with seven different laser fluences (14.2, 21.4, 28.5, 35.7, 42.8, 50, 57.14) J/cm^2		

35 specimens for 700 μs pulse duration groups

↓

Two groups

each group of seven samples

(700 μs) groups	Irradiation time (s)	repetition rate (Hz)
1)Group Frac/d	30	50
2)Group Frac/e	40	50
Each group with seven different laser fluences (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2		

(700 μs) groups	Irradiation time (s)	repetition rate (Hz)
1)Group Frac/a	20	25
2)Group Frac/b	30	25
3)Group Frac/c	40	25
Each group with seven different laser fluences (14.2, 21.4, 28.5, 35.7, 42.8, 50, 57.14) J/cm^2		

Figure (2-16): Fractional laser pilot study groups, for their applied parameters

After laser irradiation procedure, all treated zirconia disc specimens were examined under the optical microscope with the same above procedure used for the previous pilot study. The obtained images showed no laser effect to be considered in this study.

2.6 The control group

In this group no surface treatment was performed. Each zirconia disc was embedded horizontally in a mixed cold cure acrylic mold to about (1.5) mm and the remaining (0.5) mm of zirconia disc height was left exposed, ensuring that the ceramic surface remained intact for the bonding procedure, as in figure (2-17). Then the silicon rubber mold 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 disc. After that, an adequate amount of the adhesive cement (Relyx U200 self-adhesive resin cement, 3M ESPE Germany) shown in figure (2-18) was outo mixed and delivered into to the opening of the silicon mold. The excess cement was then removed with the explorer tip from the periphery of the mold and the luting agent was then photo polymerized using light curing system that illustrated in figure (2-19) for 40 seconds, following the manufacturer’s instruction

Figure (2-17): Acrylic blocks with zircons discs

Figure (2-18): Relyx U200 self-adhesive

Figure (2-19): Light curing system

The silicon mold was removed as in figure (2-20). And finally, one hour after cementation, the specimens were stored in distilled water in a plastic container as in figure (2-21), and placed in a digital water bath as shown in figure (2-22) at 37 °C for 24 hours before SBS testing for completion of resin cement polymerization reaction [171].

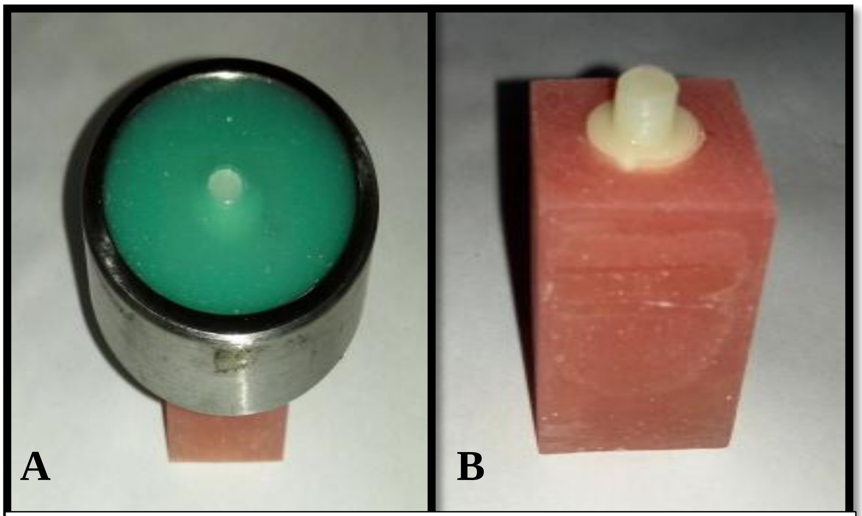

Figure (2-20): Cementation procedure. A. Silicon mold fitted over the acrylic cube. B. Molded cement.

Figure (2-21): Water bath storage

Figure (2-22): Digital water bath

2.6.1 Shear bond strength test

The specimens were attached to a universal testing machine, as in figure (2-23) (Instron-1195, England). And subjected to a shear force using a stainless steel chiseled-shaped rod (0.5 mm) with across head that illustrated in figure (2-24) at a crosshead speed of 1mm/min until failure occurred.

Figure (2-23): Universal testing machine

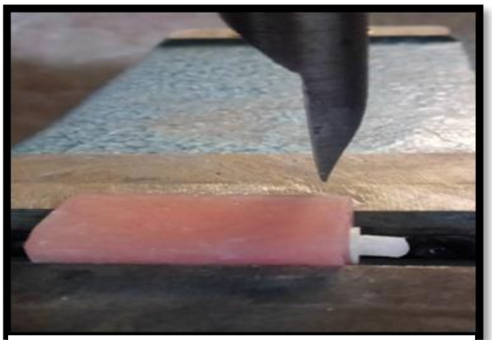

Figure (2-24): chisel end rod

The tested specimens were placed in the lower part of the testing machine, while the acrylic block was held in a horizontal position in such a way that the long axis of the chisel-shaped rod is placed parallel to the horizontal surface of the zirconia disc. The chisel end of the rod was positioned at the zirconia-cement interface. The specimen was secured tightly in place, to ensure that the zirconia disc was always at 90 degrees to the vertical plane. Then the specimens were stressed to failure. The shear bond test values were calculated from this measurement and expressed in Map. Shear strength (Map) = maximum force (N) / bonding area (mm), as in figure (2-25).

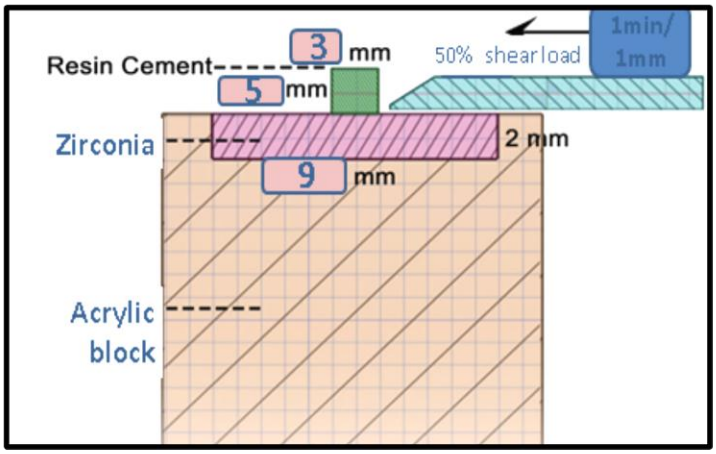

Figure (2-25): Shear bond testing procedure.

2.7 The Laser groups

Each zirconia disc bonding surface was irradiated with the Er,Cr:YSGG laser with the same performed procedure in the pilot study, after determination of the most appropriate parameters which are to be fixed: (50 Hz), and those to be included as minimum parameters; (20s, 1W), in this group.

The specimens were randomly divided into five sub-groups, each with seven samples:

1- **Group (A):** The specimens of this group were irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2, each for 20 s, 60 µs pulse duration.

2- **Group (B):** The specimens of this group were irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2, each for 30 s, 60 µs pulse duration.

3- **Group (C):** The specimens of this group were irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2, each for 40 s, 60 µs pulse duration.

4- **Group (D):** The specimens of this group were irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2, each for 20 s, 700 µs pulse duration.

5- **Group (E):** The specimens of this group were irradiated with seven different fluences: (7.1, 10.7, 14.28, 17.85, 21.42, 25, 28.57) J/cm^2, each for 30 s, 700 µs pulse duration.

As simplified in figure (2-26).

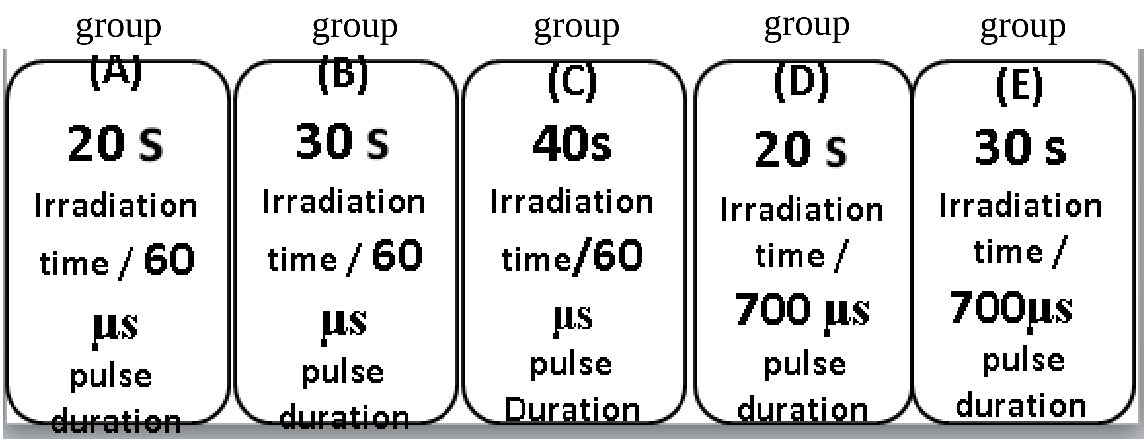

Figure (2-26): Laser groups for both 60 µs, 700 µs pulse durations, for different irradiation times.

An acrylic mold was constructed for each zirconia disc and the entire procedure, for the resin cementation, that was performed for the control group was beening repeated with each specimen of this group. The SBS test was measured for all the groups. The fractured specimens were examined under a stereo microscope at (40X mag) to determine the failure type.

The failure modes were classified as:

A. **Adhesive failure:** When the fracture occurred at the cement-ceramic interface.
B. **Cohesive failure:** When the fracture occurred in ceramic or in the cement. without damaging the interface.
C. **Mixed failure**: Was for the involvement of both the material (ceramic or cement) and their interface[172].

2.8 Scanning electron microscope (SEM):

One untreated sample and five other samples of the highest SBS value from each laser group were examined for their surface morphology by coating their bonding surfaces with gold-palladium (Q150R Rotary-Pumped Sputter Coater, Quorum Tec., UK) shown in figure (2-27) and then observed under SEM (TESCAN, VEGA/Czechia) figure (2-28).

Figure (2-27): Pumped Sputter Coater

Figure (2-28): Scanning Electron Microscope (SEM)

2.9 Surface roughness analysis:

One untreated sample and five other samples, that showed the highest SBS values, from each laser group underwent surface roughness analysis by the atomic force microscope (AFM) (AA3000, AnstgromAdvanced.Inc., USA) shown in figure (2-29). The device diamond tip of a (3 Hz) scanning rate, was passed through the sample surface in contact mode for determination of the average surface roughness (Ra).

Figure (2-29): Atomic force microscope (AFM).

2.10 Statistical analysis

Statistical analysis was performed with SPSS software version 23/France. Statistical methods were used in order to analyze and assess the results, including:

A- Descriptive Statistics:

1- Statistical tables, including:

- ✓ Mean value.
- ✓ Standard deviation ‘SD’

2- Graphical presentation by histogram and diagram.

B-Inferential Statistics:

One-way ANOVA (analysis of variance) and least significant difference (LSD) tests were performed for finding any significant differences among the group’s means.

Statistical significance according to probability value (P) was determined as:

- ❖ Non – significant at $P > 0.05$
- ❖ Significant at $P \leq 0.05$
- ❖ Highly Significant at $P \leq 0.01$

Chapter Three

Results and Discussion

This chapter is concerned with the results of the research work and discussion of these results that were obtained after Er,Cr:YSGG laser irradiation of zirconia ceramic surface specimens with different laser powers, irradiation time, and pulse durations. The evaluation of the laser effect on the shear bond strength of zirconia ceramic to resin cement, laser pulse depths, the failure modes, and the surface morphological and textural properties, yielded an analysis for these results and thereafter the conclusions. A future suggested work will be mentioned at the end of this chapter

3.1 Shear bond strength test:

Table (3-1) shows the mean and standard error values for SBS of the six groups (control and laser groups). The one-way analysis of variance (ANOVA) test indicated that there was a highly significant difference in SBS values among laser groups, for the same applied fluence, mainly those treated with 28.57 J/cm^2 (P = 0.001), that the LSD test of this variance for group B (30 s, 60 µs) was significantly the highest SBS mean values, as (a) followed by group A (20 s, 60 µs) and group D (20 s, 700 µs) shown as (b) and group E (30 s, 700 µs) as in (c), and lastly, group C (40 s, 60 µs) which showed the least SBS values as expressed in (d). The 25 J/cm^2 (p = 0.05) follows, that the higher SBS value of the LSD test was for group E, as in (a), followed by groups B, C, D each as in (b). With the least SBS value founded in group A. And the 21.4 J/cm^2 (P = 0.01) then follows. Whereas no significant differences in the P-value were founded for the 7.1-17.85 J/cm^2. Comparing between control and the laser groups, regarding the same fluence setting, the highly significant differences were founded for the 21.4, 25, and 28,57 J/cm^2 each for (P = 0.0001), Followed by 14.28, 17.85 J/cm^2 each for (P =

0.001) and less significant differences were found with 7.1, 10.7 J/cm^2 each for (P = 0.01). Each laser group showed a P-value = (0.0001), which is a highly significant difference when compared to the control group.

Also, the LSD test of the same laser group for varying laser fluence showed that the higher significant difference (P = 0.001) was for 21.4 and 25 J/cm^2 of group C expressed in (A), followed by group A and B each for (P = 0.01) 28.57 J/cm^2 expressed in (A). And least significant differences were detected with lower fluences. Whereas no significant differences of the SBS mean values were detected in groups D and E for specimens treated with different fluences. All laser groups expressed the same P-value(0.0001), when compared with the control group.

For the short pulse duration (60µs) laser groups, there was a clear increment in SBS with fluence increasing. Laser irradiation time increase 20s- 30s yielded SBS increase for all the applied fluence. While laser irradiation time from the 30s- 40s had no increased effect on the SBS values.

For the long pulse duration (700 µs) groups, the 700 µs pulse duration created no SBS enhancement for every fluence increasing. Laser irradiation time increase 20s- 30s yielded no enhancement for the SBS in return, except for fluence (21.4, 25) J/cm^2 .

Generally, when comparing the SBS mean values of the 60 µs, 700 µs pulse duration groups, all of the700 µs pulse duration group specimens showed that their obtained SBS values were higher than those obtained with the 60 µs pulse duration groups, except for fluence 28.57 J/cm^2 for both of the700 µs groups; D, E

Table (3-1): Shear bond strength measurements for different fluence of Er,Cr:YSGG laser

Fluence J/cm^2/ Shear bond	20 sec/60 µs		30 Sec / 60 µs		40 Sec /60 µs		20 Sec /700 µs		30 Sec /700 µs		P value C. VS G.	P value L.G.
	SBS Mean	Std. Error	SBS Mean	Std. Error	SBS Mean	Std. Error	SBS Mean	Std. Error	SBS Mean	Std. Error		
7.1	C 5.24	0.20	C 6.26	0.12	C 5.94	0.41	6.84	0.33	5.79	0.91	0.01	NS
10.7	C 5.30	0.13	B 6.76	0.30	C 5.44	0.10	6.80	0.68	6.49	0.43	0.01	NS
14.28	C 5.79	0.25	C 6.44	0.30	B 6.14	0.15	6.74	0.00	6.53	0.17	0.001	NS
17.85	C 5.86	0.42	C 6.33	0.46	B 6.24	0.03	6.51	0.33	6.59	0.11	0.001	NS
21.42	B 6.16 d	0.03	B 6.86 b	0.37	A 6.56 c	0.00	6.91 b	0.42	7.44 a	0.54	0.0001	0.05
25	B 6.79 c	0.05	B 6.96 b	0.00	A 6.99 b	0.20	7.05 b	0.20	7.64 a	0.02	0.0001	0.01
28.57	A 7.23 b	0.14	A 8.63 a	0.13	C 5.74 d	0.00	6.94 b	0.02	6.48 c	0.19	0.0001	0.001
CONTROL	4.49	0.16	4.49	0.16	4.49	0.16	4.49	0.16	4.49	0.16	------	-------
*P value C VS L.G.	0.0001		0.0001		0.0001		0.0001		0.0001			
P value L.G.	0.01		0.01		0.001		NS		NS			

C VS L.G: p value between control and tested laser group/ L.G: P value between the tested group. LSD test was used to calculate the significant differences between tested mean, the letters (A, B, C, and D for column and a, b, c and d rows) represented levels of significance. highly significant start from the letter (A or a) and decreasing with the last one. Similar letters mean there are no significant differences between tested mean.

- Non-significant at $P > 0.05$.
- significant at $P \leq 0.05$.
- Highly significant at $P \leq 0.01$.
- Very high significant $p \leq 0.001$

The SBS mean values of the 60 µs, 700 µs pulse duration groups are drawn as a function of fluence, and shown in figure (3-1) and figure (3-2) respectively. The general behavior of the five groups is the SBS increase with fluence increasing. For the short pulse duration (60 µs), it was observed that the maximum SBS was obtained with 30 s, 60 µs, 28.57 J/cm^2 laser parameters and the minimum SBS was obtained with 20 s, 60 µs, 7.1 J/cm^2 laser parameters. For group A and B, the fluence increasing was accompanied by little SBS enhancements, except for 30 s, 28.57 J/cm^2 specimen, which witnessed a marked elevation in the SBS value. In group C, the 60 µs for 40 s laser irradiation time revealed that the threshold for the SBS increase was at the 21.42 J/cm^2, and the maximum SBS value was reached at 28.57 J/cm^2, beyond which, no further gain in the SBS enhancement was observed.

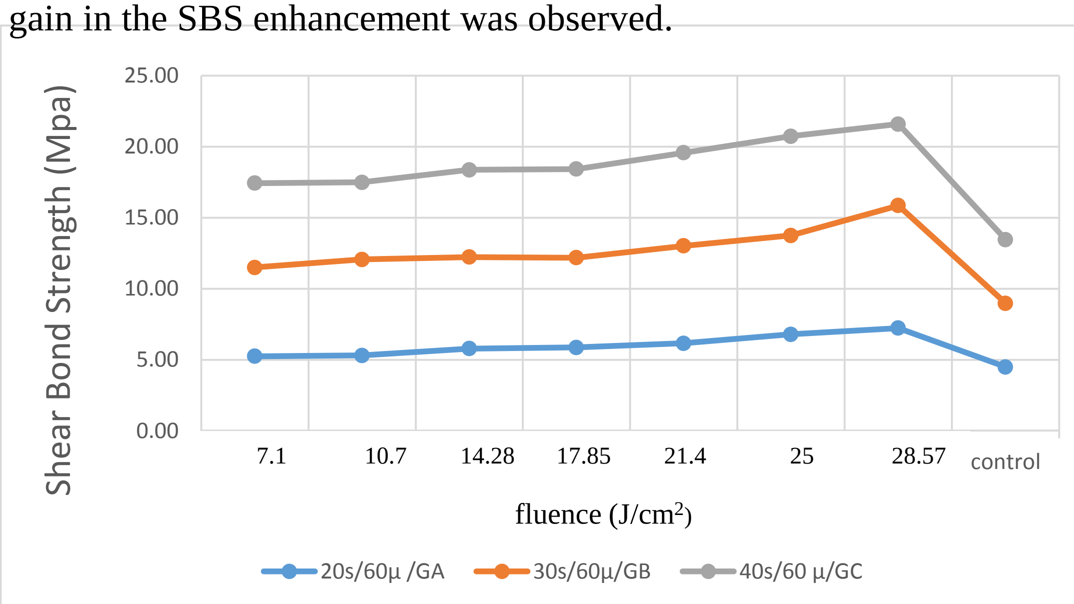

Figure (3-1): The shear bond strength as a function of the Er,Cr:YSGG laser fluence for the groups related to the pulse duration (60 µs) (Group A/20s, 60µs. Group B/30s, 60µs. Group C/40s, 60µs).

For the long pulse duration (700 µs), the maximum SBS was obtained with 30 s, 700 µs, 25 J/cm^2 laser parameters and the minimum SBS was obtained with 20 s, 700 µs, 7.1 J/cm^2 laser parameters. Laser irradiation for 20s with 700 µs had no effect on enhancing the SBS. The threshold for the SBS increase, of the 30s laser irradiation time was at fluence 21.4 J/cm^2, reaching it's maximum value at 25 J/cm^2. Then experienced a fall in its enhanced SBS value. Nevertheless, the maximum reached SBS value for the 60 µs short pulse duration was higher than the value that was reached with the 700 µs long pulse duration

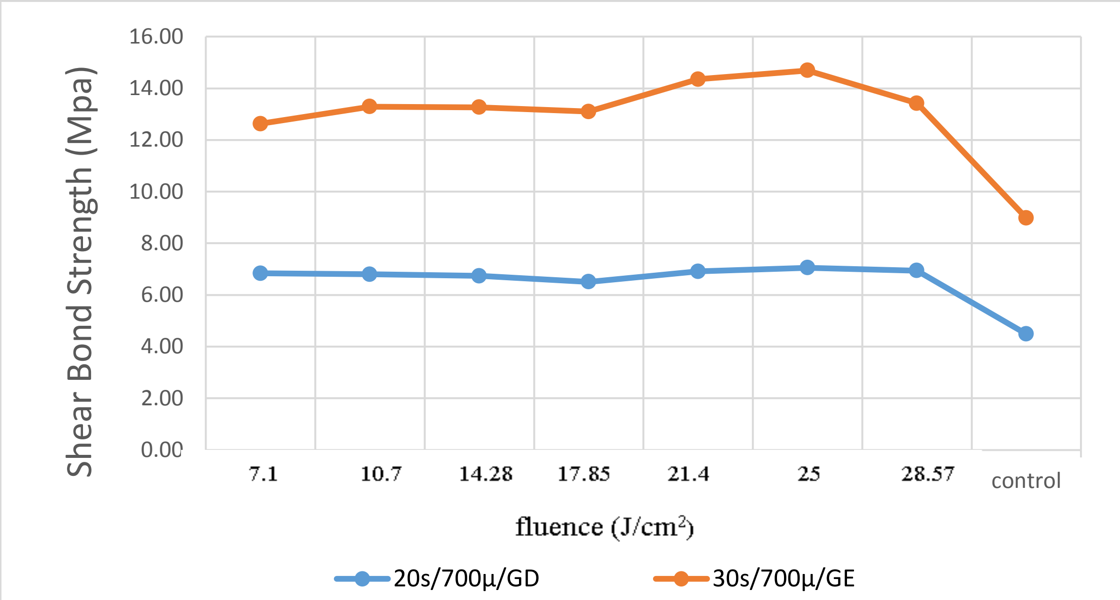

Figure (3-2): The shear bond strength as a function of the Er,Cr:YSGG laser fluence for the groups related to the pulse duration (700 µs) (Group D/20s, 700µs. Group E/30s, 700µs).

3.2 Laser pulse depth examination

Table (3-2) shows the mean and standard error values for LPD of the five laser groups. One-way ANOVA test indicated that the highest significant difference in the LPD mean values, for the different laser groups, was for those specimens treated with 28.57 J/cm^2 of groups B (30 s, 60 µs), and E (30 s, 700 µs): (P=0.0001), which was indicated by the LSD test as;(a). Followed by 17.85 J/cm^2 for groups C (40 s, 60 µs) and E (30 s, 700 µs), 21.4 J/cm^2 for group C, and 25 J/cm^2 for groups C, E, where the (P = 0.001) and the LSD test expressed as; (a) for each indicated group specimen. And the least significant difference was shown with 7.1 J/cm^2 group A

as; (c) for P value=0.05.

Also, for the same group, of different laser fluences, the highest significant difference (P= 0.001) was for group B and E (28.57 J/cm^2) as; (A), and for group C (25 J/cm^2). Whereas, no significant difference in the LPD mean values was for the specimens treated with different laser fluences of group D. Also the table clearly shows that, except for group D, all laser groups experienced an increase in the LPD with increasing the laser fluence. The 700 µs pulse duration groups had higher LPD values than the 60 µs groups for 14.28 J/cm^2 and above. Laser irradiation time increase, for the 700 µs laser groups, necessitated a significant increase in the LPD values. The 60 µs laser groups also showed a significant increase in the LPD values, but with less mean values than those for the 700 µs pulse duration laser groups.

Table (3-2): Laser pulse depths measurements for different fluences of Er,Cr:YSGG laser

fluence J/cm^2/ Depth of laser	20 sec/60 µs		30 Sec / 60 µs		40 Sec /60 µs		20 Sec /700 µs		30 Sec /700 µs		* P value
	Pulse depth Mean	Std. Error	Pulse depth Mean	Std. Error	Pulse depth Mean	Std. Error	Pulse depth Mean	Std. Error	Pulse depth Mean	Std. Error	
7.1	D 1.70 c	0.40	E 2.10 b	0.30	F 4.50 a	0.35	2.67 b	0.69	C 2.00 b	0.20	0.05
10.7	C 2.30 d	0.21	D 4.20 a	0.15	E 3.90 b	0.10	3.40 c	0.00	C 2.30 d	0.10	0.01
14.28	C 2.00 d	0.15	D 4.80 b	0.56	D 4.50 b	0.12	3.70 c	0.40	B 6.20 a	0.21	0.01
17.85	C 2.50 d	0.12	D 4.70 b	0.53	C 6.2 a	0.72	3.50 c	0.29	B 6.30 a	0.56	0.001
21.42	B 3.00 d	0.00	C 6.00 c	0.23	B 7.50 a	0.20	3.50 d	0.25	B 6.50 b	0.12	0.001
25	A 4.10 c	0.51	B 6.90 b	0.06	A 8.00 a	0.58	3.30 d	0.15	A 7.90 a	0.17	0.001
28.57	B 3.30 c	0.65	A 8.00 a	0.21	B 7.70 b	0.35	3.70 c	0.12	A 8.20 a	0.12	0.0001
P value	0.01		0.001		0.001		NS		0.001		

P value between the tested group. LSD test was used to calculate the significant differences between tested mean, the letters (A, B, C, and D for column and a, b, c and d rows) represented the levels of significant, highly significant start from the letter (A or a) and decreasing with the last one. Similar letters mean there are no significant differences between tested mean.

The LPD mean values of the 60 µs, 700 µs pulse duration groups are drawn as a function of fluence, shown in fig. (3-2) and figure (3-3) respectively. The general behavior of the five groups is LPD increase with fluence increasing. For the 60 µs pulse duration groups, the maximum laser pulse depth was obtained with group B 30 s, 60 µs, 28.57 J/cm^2 laser parameters and the minimum laser pulse depth was obtained with group A 20 s, 60 µs, 7.1 J/cm^2 laser parameters. The maximum LPD values for group A and C were for: 25 J/cm^2. Group A 14.28 J/cm^2, group B 17.85 J/cm^2 and group C 10.7 J/cm^2 had a slight decrease in their LPD value along the plot of the gradual increasing LPD values.

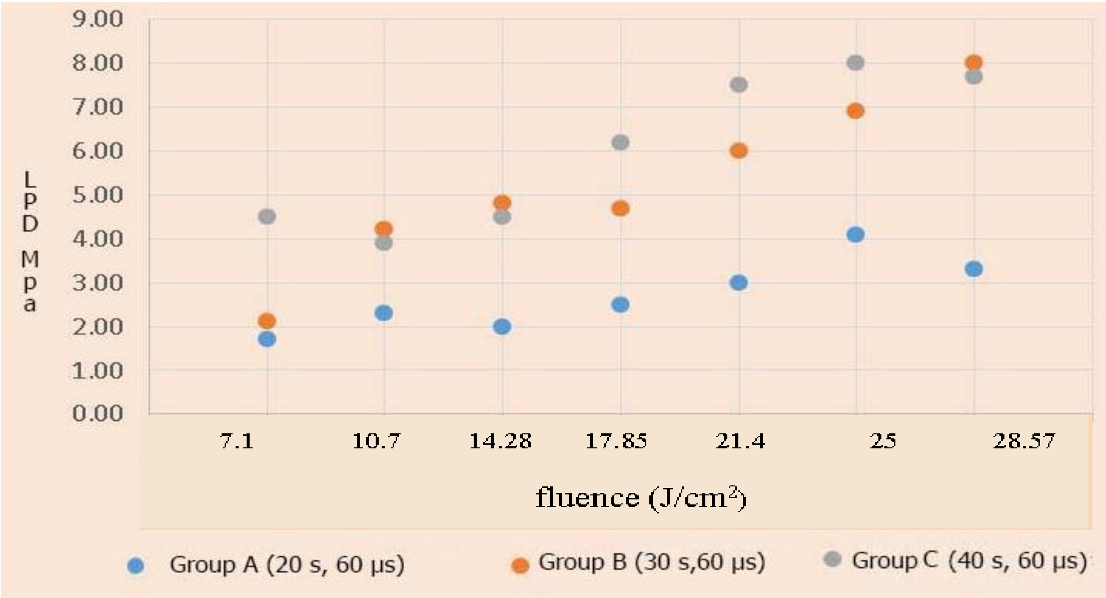

Figure (3-3): The laser pulse depth as a function of the fluence for the groups related to the pulse duration (60 µs).

As for the 700 µs pulse duration groups, the maximum laser pulse depth was obtained with group E 30 s, 700 µs, 28.57 J/cm^2 laser parameters and the minimum laser pulse depth was obtained with group D 20 s, 700 µs, 7.1 J/cm^2 laser parameters. Both groups had their maximum LPD values at 28.57 J/cm^2 . Group D showed a sudden and a high increase in LPD mean, for 14.28 J/cm^2 fluence, compared with other fluences of the same group. fluence increasing for group D samples was unable to increase the LPD values.

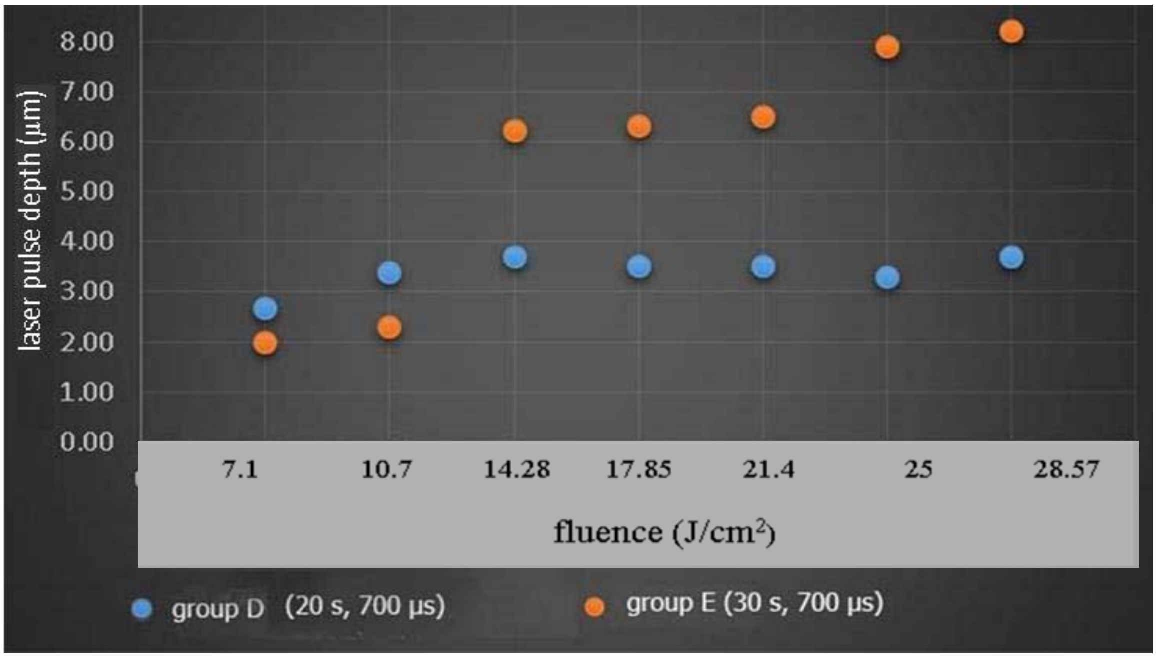

Figure (3-4): The laser pulse depth as a function of the fluence for the groups related to the pulse duration (700 µs).

3.3 Failure mode

Frequency of failure mode after shear bond strength test of each group is shown in table (3-3). The results indicated that the failure mode of the groups varied with different laser parameters. In group A and C, type (1) failure mode was frequently observed in those specimens treated with 7.1- 21.4 J/cm^2 for both groups. On the contrary, failure mode of type 3 was mostly detected in group D and E excep for 14.28- 17.85 J/cm^2 and 7.1- 10.7 J/cm^2 respectively. And type (2) had the least frequency in group B, 28.57 J/cm^2 and E, 25 J/cm^2.

Table (3-3): Failure mode distribution following the shear bond strength test for the five laser groups (A, B, C, D, E).

Groups	Adhesive Failure (1)		Cohesive Failure (2)		Mixed Failure (3)	
	No.	%	No.	%	No.	%
A	5	50			2	20
B	3	30	1	10	3	30
C	5	50			2	20
D	2	20			5	50
E	2	20	1	10	4	40

3.4 Scanning electron microscope analysis

The SEM images of the zirconia disc surfaces is shown in figure (3-4) A-F. The effect of laser energy pulses on the surfaces is observed as holes with no altering in the surface texture surrounding each laser pulse hole. No obvious cracks or charring were seen. Therefore, no laser optical damages. Picture

(A) represent the surface morphology of the untreated specimen (control) with very fine scratches resulted from the polishing procedure. The Er,Cr:YSGG laser treated samples: Picture (B) is for the (20s/60µ/ 28.57 J/cm^2) laser treatment. The effect of laser pulses shows as small pale holes, compared to other sample pictures, as the LPD was beaning shallow (3.30µ), which yielded SBS (7.23 Mpa) for the used laser parameters. Picture (C) for the (30s/60µ/ 28.57 J/cm^2) laser treatment of the highest SBS (8.63 Mpa) and the deepest LPD (8µ) appears with wide and very defined laser pulse holes. Picture (D) the (40s/60µ/ 25 J/cm^2) laser treated specimen, shares the same LPD value(8µ) and almost the same laser holes appearance with the previous specimen picture, with a slight decrease in its SBS (6.99Mpa) for the increased laser irradiation time been used.

Picture (E) for the (20s/700µ/25 J/cm^2) showing the appearance of a pale shallow pulse holes (3.30 µ) as a result of the laser heat dissipation that had occurred with the 700 µs pulse duration. While, picture (F) of (30s/700µ/25 J/cm^2) for the same pule duration shows the effect of the increased laser irradiation time/sec. that is distinguished as dark, well defined laser holes, correlated to the increased LPD (7.90µ) which produced SBS of (7.64Mpa).

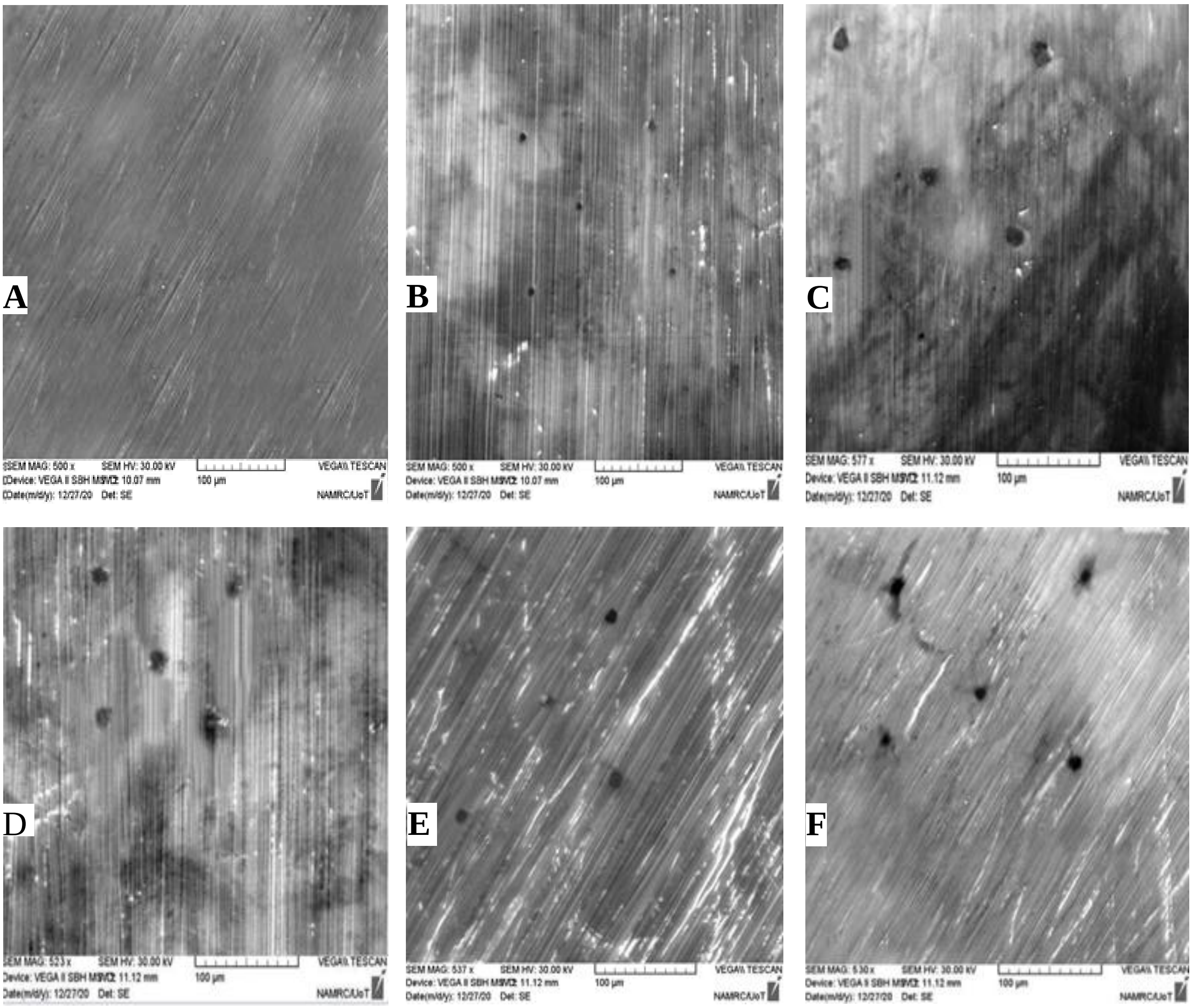

Figure (3-5): SEM Pictures of zirconia specimens (500X). A. Control (untreated) Specimen. The Er,Cr:YSGG laser treated samples B. (20s/60µ/28.57 J/cm^2). C. (30s/60µ/28.57 J/cm^2). D. (40 s/60µ/25J/cm^2). E.(20s/700µ/25J/cm^2). F.(30s/700µ/25J/cm^2).

3.5 Surface roughness analysis

The three-dimensional (3D) roughness measurements of different laser treated specimens are showed in figure (3-5). The roughness average (Ra) was increased with the laser higher fluence settings, with increased number of pulses per sec., and with the longer laser pulse duration groups, as seen with specimen (30s, 700µs, 25J/cm^2) of the highest Ra value than with the other surface treated specimen.

Picture (A) presents specimen with the lowest Ra value (3.05nm) duo to absent laser surface treatment. Picture (B) of the 20s/60µs/28.57J/cm^2 Er,Cr:YSGG laser treated specimen, had the lowest Ra=7.73nm for the lowest LPD mean (3.30µ) among the AFM examined specimens. Picture (C) for the 30s/60µs/28.57J/cm^2 laser treatment, had an increase in the Ra (8.88nm) value for the highest LPD value (8µ) among all 60 µs laser treated specimens for the higher laser irradiation time that zirconia surface had been exposed, as compared to the previous specimen. Picture (D) of 40s/60µs/25J/cm^2 laser treatment showed further Ra increase (10.2nm) with LPD (8µ) for the much increased laser irradiation time. Picture (E) of 20s/700µs/25J/cm^2 laser treatment, showing the effect of the 700 µs laser pule duration in creating an enhanced roughness (Ra=12.4nm) with more prominent surface irregularities as shown in the following figure, for the lowest LPD value of (3.30 µ) of all the AFM analyzed surface specimens. Picture (F) of the (30s/700µs/25J/cm^2) laser treatment, had the highest Ra (14.5nm) value of all the AFM analyzed surfaces, with the larger, unevenly distributed surface irregularities presented in the 3-D dimensional picture. And the highest LPD (7.90 µ) for the increased laser irradiation time that were used per unit sec.

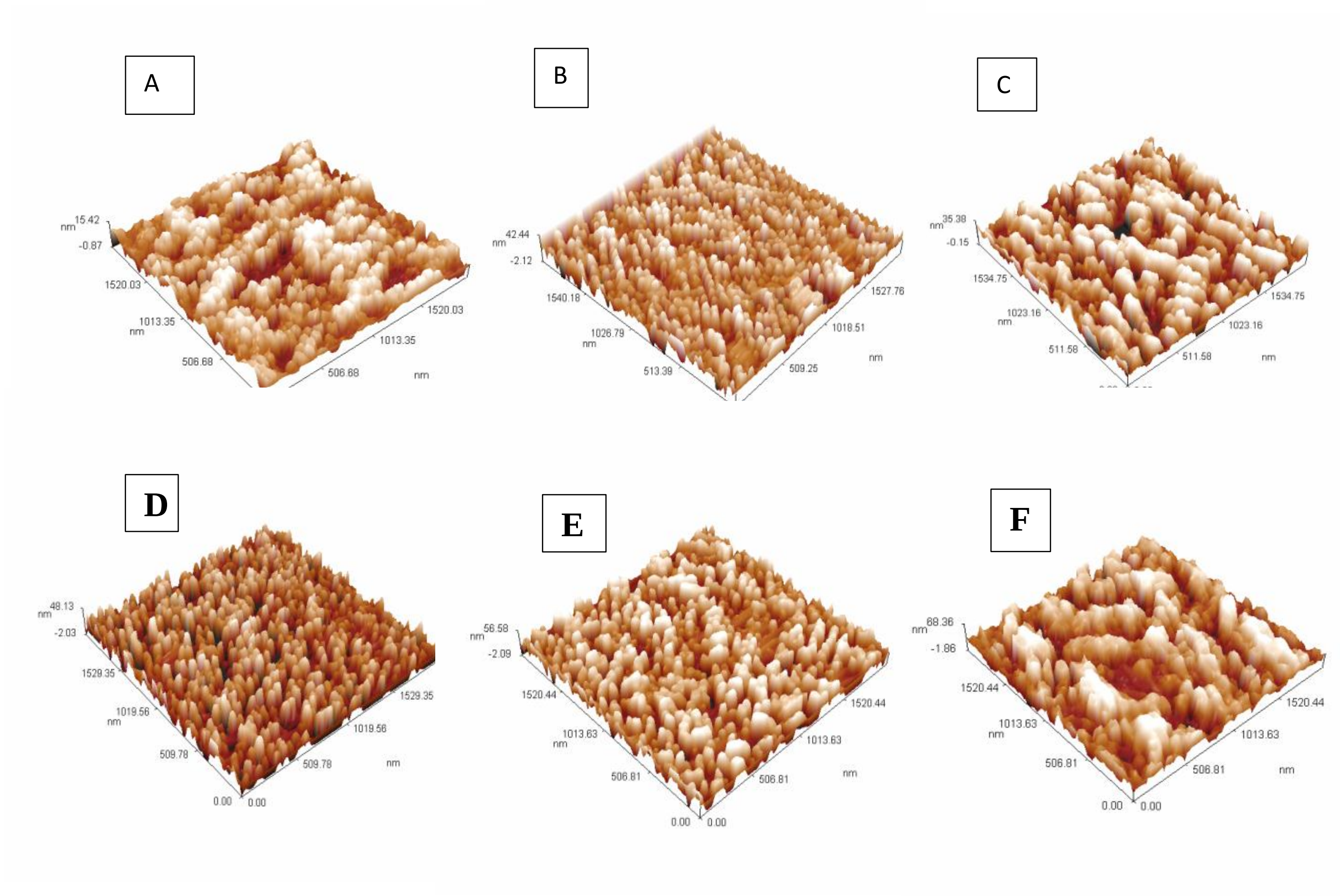

Figure (3-6): (3-dimentional AFM pictures). A. Untreated specimen (Ra=3.05nm). Er,Cr:YSGG laser treated specimens: B.(20s/60µs/28.57J/cm^2)Ra=7.73nm, C.(30s/60µs/28.57J/cm^2)Ra=8.88nm,D.(40s/60µs/25J/cm^2)Ra=10.2nm, E.(20s/700µs/25J/cm^2)Ra=12.4nm, F.(30s/700µs/25J/cm^2)Ra=14.5nm

3.6 Discussion

Laser was proposed by many authors as a new surface treatment method to improve surface roughness and bond strength of zirconia ceramics to resin cements [165]. Laser application remove particles by micro explosion, a process called ablation. Er,Cr:YSGG laser induced roughness has the advantage of increasing potential area for zirconia bonding[173].

The current study aimed to investigate and compare the effect of different fluence settings of Er,Cr:YSGG laser of 60, 700 µs pulse duration with different laser irradiation times on the SBS of resin cement to zerconia surface. The shear bond strength test was used to investigate the bonding efficacy of zerconia ceramic to (Relay X U200, US) dual cure adhesive resin cement. This test was chosen because it is relatively simple, easy to perform and it simulates the human oral shear stresses which are an important contributor to deterioration and deboning of restorative materials [9]. Different types of cements (conventional, glass ionomer, adhesive resin cements) have been proposed for zirconia cementation. Among these, adhesive resin cements are the preferred choice because of their enhanced marginal fit and retention [174], Favorable aesthetic properties, high compressive and tensile strength [175]. In this study Relay X U200 Self-adhesive composite cements was used as it allows for a good bond between the resin and zirconia without the application of additional primer and promises a simple cementing procedure according to manufacturer instructions.

3.6.1 Shear Bond Strength And Bond Failure

Heat confinement is a crucial factor in zerconia ceramic surface roughness with laser irradiation [18]. The results of this study shows that using Er,Cr:YSGG laser on the bonding surface of zirconia surface created a significantly enhanced shear bond strength compared with the control group, probably because of the surface roughness and LPD of the zirconia bonding surface that enhanced the zirconia- resin cements interlocking. the optained results showed that the highest SBS mean, in the specimens treated with the Er,Cr:YSGG laser for 700 µs pulse duration groups, was: 7.64 Mpa, and LPD: (8.2µm), for specimen treated with (30 s, 700 µs, 25J/cm^2). While for the short pulse duration (60 µs), the highest SBS mean was: 8.63 Mpa, and LPD: (8µm), for specimen treated with (30 s, 60 µs, 28.57J/cm^2), which was significantly greater than those of the other groups for the same or for different pulse duration. This findings could be explained by the effect of Er,Cr:YSGG focused laser irradiation in ablating the zirconia surface particles with concentrated, short laser pulse durations (60 µs) with no lateral laser-heat dissipation, thereby with no damaging effect to zerconia material. The LPD strongly influenced by the fluence increase. The applied high fluence (28.57J/cm^2) and the long laser exposure time (30s) facilitated for an efficient laser-zirconia ceramic interaction and later on, for an ablation of deeper layers of the material’s structure forming well defined holes of much increased depth as compared to the results of samples irradiated with other laser parameters. The resulted increase in the LPD, means the increase in the surface area. Which allowed for increased zirconia surface exposure to resin cement that facilitated the flow of resin and it’s steeliness to be photo polymerized into a strong resin-zerconia bond of an enhanced mechanical interlocking. Specimen treated with (20 s, 60 µs, 7.1 J/cm^2) exhibited the lowest SBS (5.24 Mpa), and LPD: (1.7µm), among the laser groups. This result could be duo to the low fluence (28.57 J/cm^2) being used for the short laser irradiation time (20 s) that was unable to ablate a sufficient amount of zirconia particles needed for creating adequate LPD for increasing the surface area and gaining a proper mechanical retention.

The laser effect of the short pulse duration on a hard material such as zirconia ceramic differs from the long pulse duration effect. The delivery of short pulse durations(60µs) requires an adequat fluence efficiency for acheiving suffucient ablation depth presented as surface roughness and laser holes(LPD), and also requiers for an adequate laser exposure time, which is a particular number of laser pulses per second, as the short laser pulse has a limitted interaction zone and time, because of the very short laser energy delivary, that the laser heat would not be allowed to diffuse and interacte with areas beyond application site. There was no SBS enhancement with fluence increasing, for the 20s laser irradiation, as the number of laser pulses available within the given irradiation time was not enough for creating LPD of that much of differences within it's values to show it's behavior as an enhanced results. fluences 7.1- 21.4 J/cm^2 for 30s laser irradiation time, proved not to be the needed fluences, for this type of zirconia, to achieve attainment of the SBS threshold of enhancment. Whereas fluences25- 28.57 J/cm^2 had a marked enhance in it's SBS values duo to the increased numder of laser pulses beening used for the 30s laser irradiation, these high fluences that supplied the laser ablation process with a more material's particles removal through a faster and effective layer fragmentation, creating an enhanced depth (LPD) needed for a stronger resin-zirconia attachment, that is an enhanced SBS

As for the long pulse duration (700µs), The provided longer and repeated laser exposure time allows for, the laser heat to be diffused lateral to the laser interacted zone, and the temperature elevation in the laser affected zone that the low fluences capacities would not be effective, and the higher fluences would have a loss in their heat confinement and damaging effect for the material. That is way, the 20s exposure time had no enhancing effect on the SBS for all the applied fluences, nor an increase in the obtained LPD values even as proceeding towards higher fluences. the increased laser irradiation time from 20s to 30s had no enhancing effect on the SBS, except for fluences 21.4-25J/cm^2 that witnessed a marked SBS increase which was related to the huge differences in there LPD values, for the applied 30s irradiation time. These two laser fluences had sufficient amount of energy to be deposited in the material as a laser heat, creating an enhanced surface roughness pattern needed for SBS enhancement despite the laser energy dissipation. the huge elevation in LPD value for fluence 14.28 J/cm^2, and the continued increase in LPD for 25- 28.57 fluences, could be considered as a better LPD results than the 60 µs pulse duration groups. Nevertheless, the LPD values for the 700 µs group was accompanied by zirconia structure morphological changes beyond the LPD periphery that contributed to the presented high depths, but had also weakened the resin-cement interlocking as it shows from the SBS results. So, the resulted zirconia surface effect would be different from the shorter pulse duration.

This came in a good agreement with (Gomes et al 2015) [176] who stated that, the increased bonding strength values had been reported after Er:YAG laser irradiation. And with those results of (kara R et al 2020) [163] who showed that the Er,Cr:YSGG laser treatment method provides durable bond strength when the correct energy and time are applied and can be an alternative pretreatment to increase the bond strength of the resin cement. However, the results disagreed with (Aboushelib et al 2014) [177], Whom concluded that, the use of high energy level of pulsed Er,Cr:YSGG failed to increase the bond strength to ceramics. this can be explained by the fact that the authors in their study used different laser parameters. The bond quality, in the current study, was also assisted by bond failure mode analysis which supply influential information of bonding efficacy. Cohesive and mixed (cohesive failure type combined with an adhesive mode) fracture patterns are clinically preferable to entirely adhesive failure type, since the last one is generally accompanied by low bond strength values [178]

The bond failure of group A and C mainly of adhesive tape showing that the micromechanical interlocking of this groups was not adequately strong to generate higher bond strength. As for group B, adhesive and mixed modes were the most predominant except for specimen treated with 28.57 J/cm^2 laser fluence which showed cohesive failure mode. Whereas group D and E showed mostly mixed failure mode. This means that groups B, D and E had the higher cement-ceramic bond strength than the other two groups of adhesive mode. These results are in different with (Aras et al 2016) [179], who reported that The increased surface area could increase adhesion but bond strength values were not higher after laser irradiation.

3.6.2 Number of laser pulses per second

The amount of laser pulses striking the irradiation site per second, strongly affect the absorption and the ablation process as it determines the amount of ablated particles and therefore the created depth that elevate the SBS to the optimized enhance. For the 60 µs pulse duration group, the laser heat confinement encourages for no laser power lose. So increasing the time of laser irradiation from 20s to 30s had in turn increased the number of laser pulses by two times without the need for fluence increase that gained the ablation process much laser energy per each second of irradiation for an efficient LPD ablation. So all the laser pulse holes had increased in depth exposing larger surface area for resin cement interlocking. While increasing the time of laser irradiation from 30s to 40s had shown an increased LPD values only for 17.85-25 J/cm^2 fluences which was not accompanied by SBS increase, as the high number of laser pulses striking the zirconia surface had caused for the laser-heat dissipation with low fluence and the incident laser energy being wasted, and for the higher fluences to cause lateral laser damage. The 30 s laser irradiation time for the low fluences 7.1- 10.7 J/cm^2 of the short pulse duration (60 µs) group, had the higher LPD values than the long pulse duration (700 µs) group. While for high fluences 14.28 J/cm^2 and above, the long pulse duration group had the highest LPD values compared to short pulse duration;(60µs, 30s) laser irradiation time group. This could be explained by the fact that the low fluences of the 60 µs laser pulses had a confined laser-heat dissipation within the irradiated zone, that all the laser energy per pulse was beening absorbed by the zerconia surface creating the well-defined depth with no loss of laser energy to area peripheral to the LPD. Which was not the case for the 700 µs pulse duration. And the higher fluences of the 700 µs , the zirconia surface turned over-heated with the repeated, high energy pulses, an effect that when it is combined with the heat dissipation by the 700 µs pulse duration, it would create an increase in the LPD values over the 60 µs pulse duration LPD mean values.

3.6.3 Surface analysis

The laser energy absorption by the material's surface is the most important interaction between the laser and the material. Therefore, it is crucial to choose suitable laser parameters in order to have a zerconia ceramic surface changing of a suitable surface treatment [180]. The surface roughness is an important parameter and could increase bonding strength values[181]. samples treated with Er,Cr:YSGG laser showed increased surface roughness as compared with untreated zerconia samples. Surface roughness increased with fluence increasing and with laser irradiation time. Samples of the long pulse duration (700 µs) showed the higher Ra values(14.5nm) compared with the short pulse duration samples(10.2nm), but, for the long pulse duration samples, SBS values was not enhanced. This could properly be duo to the effect of Er,Cr:YSGG laser long pulses in roughening the zerconia bonding surface with lateral laser-heat dissipation that may have created surface micro irrigularities in the laser irradiated zone, that contributed to surface roughness increase. Same result was found by (Alhassani et al 2018) [18]. Who reported that the surface roughness increased with increasing power and pulse duration but it does not enhance the bonding strength.

These results disagreed with Miranda et al. 2015[182], whom examined the surface roughness on Y-TZP surface after Er:YAG laser irradiation at 1.5 W/20 Hz and concluded that laser irradiation caused a decrease in surface roughness. The results were in a good agreement with (Kunt and Duran, 2018)[160], whom concluded that only 4W CO2 laser irradiation for Y-TZP ceramics is recommended as an alternative surface treatment to sandblasting.

3.6.4 The scanning electron microscope analysis

The continued laser energy deposition with repeated incident pulses on superficial layers of the material, produces ablation and surface irrigularities that are unique for the applied laser and for it's adapted parameters. This laser-surface morphology had to be scanned with the SEM technology. The general principle for laser-zirconia treatment, is to create a surface pattern of increased area of exposure for the resin cement to be strongly interlocked. This was the illustrated results for the five examined laser treated specimens, that the visualized micro holes had no cracks, fissures nor charring. The created micro irrigularities differ for the applied laser parameters, and aid to provide solid mechanical bond with the lute resin cement. The SEM measures the exacte degree of produced surface roughness, and pulses depths, that needed to be reached, for optimizing the SBS values. Thereby, giving an estimation for the laser ablation efficiency for producing surface changing to be considered as a desirable surface treatment.

3.7 Conclusions

According to previous results and discussion, the followings are to be concluded**:**

1- Er,Cr:YSGG laser irradiation of the zirconia ceramic surface at (30s, 60µs, 28.57 J/cm^2) were the most efficient in the enhancement of the bonding strength of zirconia to resin cement.

2- The enhanced SBS had depended on the surface roughness and pulse depths, created by different laser parameters surface treatment.

3- Fluences above 28.57 J/cm^2 and laser exposure times beyond 30s, might cause material damage.

4- The effect of the short pulse duration (60µs) on enhancing the SBS, is better than the long pulse duration (700µs).

3.8 Suggestions

1. Considering the Er,Cr:YSGG laser parameters effect on the SBS of super highly translusent zirconia to resin cement.
2. Evaluating the effect of metal primers combined with the Er,Cr:YSGG laser on the SBS of zirconia restorations.
3. Evaluating zirconia crystalline phase change, through the X-ray diffraction method.
4. Microscopical evidence for the lute cement, measuring the surface wettability
5. Zirconia surface temperature measurement, evaluating laser-thermal effect.

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PUBLISHED ARTICLES

1- (**THE INFLUENCE OF SHORT PULSE Er,Cr:YSGG LASER ON THE SHEAR BOND STRENGTH OF CAD/CAM ZIRCONIA MATERIAL TO RESIN CEMENT).**

Submitted to

The Indian Journal of Forensic Medicine and Toxicology

2- (**PULSED Er, Cr:YSGG LASER FOR SURFACE MODIFICATION OF DENTAL ZIRCONIA CERAMIC**).

Submitted to

Iraqi Journal of Laser

3- (**SURFACE MODIFICATION OF CAD-CAM MACHINED ZIRCONIA USING THE 60 µS, 700 µS Er,Cr:YSGG LASER PULSE DURATION**).

Submitted to

Journal of Research In Medical And Dental Science

الخلاصة:

الخلفية: قيمت دراسات عِدَّه، تقنيات تقليدية وتطبيقات ليزريه مختلفة، لغرض تعزيز قوة رابطة القص لمادة الزركونيا سيراميك مع الإسمنت الراتنج. كان الهدف من هذه الدراسة هو فحص تأثير معلمات Er, Cr: YSGG النبضي على تعزيز قوة رابطة القص للزركونيا سيراميك (Vita YZ HT، Zahnfabrik ، ألمانيا) إلى الإسمنت الراتنج اللاصق.

المواد وطريقة العمل: تم خراطة كتل أُكسيد الزركونيوم الغير مصلّبه، إلى ٥٤ قرصًا من الزركونيا حسب الأبعاد المطلوبة (قطر ٩ ملم ، ارتفاع ٢ ملم). تم استخدام ١٢عينة للدراسة التجريبية لتحديد العامل الليزريّ الاكثر تأثيراً (معدل تدفق الطاقه ، معدل تكرار النبض ، مدة النبضة ، زمن التشعيع) على سطح الزركونيا. ٣٥ عينة مقسّمة إلى ستة مجموعات كل منها من سبعه عينات (المجموعة الغير معامَله ومجموعات الليزر). مجموعات الليزر هي: المجموعة (أ): ٢٠ ثانية ، ٦٠ مايكرو ثانية مدة النبضة ، المجموعة (ب): ٣٠ ثانية ، ٦٠ مايكرو ثانية مدة النبضة ، المجموعة (ج): ٤٠ ثانية ، ٦٠ مايكرو ثانية مدة النبضة ، المجموعة (د): ٢٠ ثانيه ، ومدة النبضة ٧٠٠ مايكرو ثانية ، والمجموعة (ي): ٣٠ ثانية ، ٧٠٠ مايكرو ثانية مدة النبضه .تم تشعيع جميع عينات المجموعات بسبعة معدلات تدفق طاقه مختلفة: (٧.١، ١٠.٧، ١٤.٢٨، ١٧.٨٥، ٢١.٤، ٢٥، ٢٨.٥٧) جول/سم٢. تم لصق الإسمنت لأسطح اقراص الزركونيا المشععه بالليزر. وتم معالجتها بالضوء لمدة ٤٠ ثانية. تم تقييم قوة رابطة القص بإستخدام آلة إختبار عالمية وتم تحليل النتائج المختارة بدقة. و تم فحص متوسط خشونة السطح بواسطة مجهر القوة الذرية. كما و تم فحص أوضاع فشل السندات.

النتائج: لوحظ وجود ثقوب واضحة لنبض الليزر في العينات المعالجة ، تحت المجهر الإلكتروني الماسح وتحت المجهر الضوئي ، مع زيادة في وضوحها وعمقها مع زيادة معدل تدفق الطاقه ، وزمن التشعيع ، مما ساهم بشكل واضح في تعزيز قوة رابطة القص ، ومتوسط خشونة السطح ، وقيم عمق النبضات. بالنسبة لمجموعات النبضات القصيره (٦٠ مايكرو ثانية) ، كانت أعلى قيمة لقوه القص في المجموعة B (٢٨.٥٧ جول/سم٢) وكانت ذو فروقات إحصائية عالية الأهمية، وايضا لمعدل الخشونة السطحية ولعمق النبضات مع انعدام تشكيل شقوق مجهرية

الخلاصة: إن قوة الليزر ومدة النبض كلاهما معلَمتان حيويتان لزيادة قوة رابطة القص لسيراميك الزركونيا إلى الإسمنت الراتنج. كان هناك دور أساسي لمدة النبضه القصيره (٦٠ مايكرو ثانية) Er, Cr: YSGG ، حيث أن تأثيرها أفضل من تأثير مدة النبضة الطويلة (٧٠٠ مايكرو ثانية) ، في تخشين سطح الزركونيا وفي تعزيز قوة رابطة القص. يمكن إعتبار ليزر Er,Cr:YSGG طريقة تخشين سطحي بديلة و فعالة لتعزيز قوة الترابط لسيراميك الزركونيا (Vital) مع الإسمنت الراتنج

جمهورية العراق

وزارة التعليم العالي والبحث العلمي

جامعة بغداد

معهد الليزر للدراسات العليا

تأثير الليزر Er:Cr YSGG المجزء على قوة ارتباط سيراميك الزركونيا مع اسمنت الراتنج

رسالة مقدمة الى

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متطلبات نيل شهادة ماجستير علوم في الليزر / طب الاسنان

مقدمة من قبل

فاطمه سمير محمد حسين

بكالوريوس طب الاسنان – 2009 م

بإشراف

الاسُتاذ الدكتور حسين علي جواد

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