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Effect of Fractional CO_2 Laser on the Surface Roughness and Bonding of Zirconium 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

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بسم الله الرحمن الرحيم

وَفَوْقَ كُلِّ ذِي عِلْمٍ عَلِيم

صدق الله العظيم
سورة يوسف
الآية٧٦

Dedication

To the love of my parents,

to the love and care of my husband, to my little angles; my three daughters and

to the soul of my beloved brother Amer (mercy upon him)

Luma

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First and foremost, I am grateful to my creator "**Allah**" for giving me the strength, enablement, knowledge and understanding required to complete this work.

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ABSTRACT

Background: The enhancement of shear bond strength of zirconia ceramic to resin cement using conventional methods and different lasers was covered by several studies.

The aim of current study: to enhance the shear bond strength of zirconia ceramic to resin cement and to investigate the effect of temperature elevation of zirconia ceramic using fractional CO_2 laser.

Material and methods: Forty four sintered zirconium oxide discs (10 mm in diameter, 2 mm in thickness) were prepared. Three zirconia discs were used as a control group and for the pilot study, 14 zirconia discs were used to determine the spot distance, time interval, and scan number. For the study group, 30 zirconia discs were divided in to three groups (N=10), five diffident pulse durations per spot were used in each groups (0.1, 0.5, 1, 5 and 10 ms)and each two specimens were treated with the same pulse duration. Group A was treated with fractional CO_2 laser with a power setting of 10 W, group B 20 W and group C 30 W. During laser irradiation, temperature elevation measurement was recorded for each specimen. A luting cement was bonded to the irradiated zirconia surfaces and cured for 30 seconds. Shear bond strength was evaluated using a universal testing machine at a crosshead speed of 1mm/min until failure occurred.

Results: The minimum temperature elevation measurement of the irradiated specimen which gave maximum shear bond strength (as 15.5± 0.3 Mpa) was about 1.6 Ċ higher than ambient room temperature with 20 W/0.1 ms pulse duration .Apparent micromechanical roughening and irregularities were seen in the treated samples and microcracks formation with increased irradiation time and power setting as observed in scanning electron microscope that resulted in higher surface roughness.

Conclusion: Fractional CO_2 laser is an effective alternative method for shear bond strength increment of zirconia ceramic to resin cement, taking into account the temperature elevation as a vital factor in the surface roughness of zirconia ceramic with fractional CO_2 laser irradiation.

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List of Abbreviations

Symbol	Term
Nd:YAG	**Neodymium doped Yttrium –Aluminum Garnet**
Er:YAG	**Erbium doped Yttrium –Aluminum Garnet**
CO_2	**Carbon dioxide**
µm	**Micrometer(=10^{-6} m)**
MPa	**Mega Pascal**
Er,Cr:YSGG	**Erbium –Chromium doped yttrium-scandium-gallium-garnet**
FDA	**Food and Drugs Administration**
FPD	**Fixed Partial Denture**
SPSS	**Statistical Package for the Social Science**
nm	**Nanometer (= 10^{-9} m)**
W	**Watt(Unit of Power)**
SBS	**Shear Bond Strength**
mJ	**milli joule (unit of energy)**
Y-TZP	**Yttrium-stabilized Tetragonal Zirconia Polycrystals**
AFM	**Atomic Force Microscope**
SEM	**Scanning Electron Microscope**

λ	**wavelength**
ms	**millisecond**
Hz	**Hertz (unit of frequency)**
(CAD/CAM)	**Computer Aided Design–Computer Aided Manufacturing**
HIP	**Hot Isostatic Pressed**
LED	**Light Emitting Diode**

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Chapter One

Introduction and Basic Concepts

Chapter One: Introduction and Basic Concepts

1.1 Introduction

Clinically, there is a growing demand for metal-free, fixed partial dentures (FPD), which has led to the production of ceramic materials with enhanced mechanical properties [1]. Zirconia ceramic has shown several important features that make it suitable for dental applications, including: ideal mechanical properties, good biocompatibility, high strength and esthetics [2-4]. New advances in dental ceramics have led to the use of yttrium-stabilized tetragonal zirconia polycrystals (Y-TZP) as a substitute for fixed partial dentures and complete coverage crowns especially when esthetic is required. Although mechanical properties are very important, clinical success depends in many cases on the strength and durability of the bond of resin cement to ceramic substrates and teeth, which has to integrate all parts of the system into one coherent structure for improving the retention, marginal adaptation, fracture resistance and bond strength of restorations [5, 6]. The problem in this respect is the low capacity of resin bonding to zirconia ceramic; which is related to glass-free and highly crystalline composition of zirconia [7]. A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires surface roughening and cleaning for adequate surface activation[8]. Surface treatment of ceramics increases the surface area and creates microporosities on the ceramic surface, which enhance the potential for mechanical retention of the luting composite resin [9]. Common treatment alternatives consist of grinding, abrasion with diamond rotary instruments [10], airborne particle abrasion with Al_2O_3 [11], acid etching [12], sandblasting, laser irradiation [13], and combination of any of these methods [14]. However, the composition and physical properties of high-strength zirconium oxide ceramics differ substantially from silica-based ceramics. The absence of glassy phase and silicon dioxide makes them resistant to etching by hydrofluoric acid and not amenable to silanization [8, 15]. Airborne particle abrasion has been proposed to facilitate the micromechanical retention between the Y-TZP ceramics and resin cements. This method increases the surface roughness and bonding area [2].However, the risk of microcrack generation with the use of air abrasion was also reported. Excessive air abrasion even induces chipping or a high loss of ceramic material, compromising the mechanical properties and long-term performance of the ceramic restorations [16].

Lasers have been used for different purposes in dentistry among which is conditioning tooth structure or restorative surfaces [17]. Previous studies employed different lasers such as Nd:YAG [18], Er:YAG[19], femtosecond laser[20]and CO_2 [21] for surface modification of zirconia ceramic, and reported varying degrees of success. The CO_2 laser is well suited for the treatment of ceramic materials because Zirconia ceramic has a high potential of absorbing CO_2 laser wavelength [22]. During the process of heat induction of ceramic surfaces with a focused CO_2 laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [23]. To date, a few studies investigated the efficacy of conventional CO_2 laser for enhancing adhesion of bonding cement to oxide ceramics ,whereas studies employed a fractional type of CO_2 laser for zirconia surface treatment are very limited.

In the present study the effect of fractional CO_2 laser irradiation on surface roughness and bond strength of resin cement to zirconia ceramic regarding the temperature elevation will be investigated.

1.2 Crowns and Bridges

Bridges and crowns are fixed prosthetic devices that are cemented onto existing teeth or implants. Crowns are used most commonly to entirely cover or "cap" a damaged tooth or cover an implant. Bridges are commonly used to cover a space for one or more missing teeth. They are cemented to natural teeth or implants surrounding the space where the tooth once stood.

A crown that has been fixed to a tooth looks and works very much like a natural tooth. Crowns are made from various types of materials, depending on which tooth needs a crown. Permanent crowns can be made of metal (such as stainless steel, gold or another alloy), porcelain-fused-to-metal, all resin, or all ceramic. There has been an increasing interest and demand in the use of all-ceramic materials due to their nonmetallic, biocompatible and improved esthetic features [24]. The core materials of all-ceramics include silica-based glass ceramics, such as Lithium-disilicate ($2SiO_2$-Li_2O), Leucite (SiO_2-Al_2O_3-K_2O) and Feldspathic (SiO_2- Al_2O_3-Na_2O-K_2O) and silica-free high strength ceramics, such as Zirconia (ZrO_2) and Alumina (Al_2O_3) [25]. Among numerous other ceramic options, zirconia drew considerable attention of the scientists and clinicians in the dental field due to its favorable mechanical properties [26, 27]. This biocompatible, chemically bioinert ceramic has potential for versatile applications in dentistry other than FDPs such as root posts, orthodontics brackets, implants, or implant abutments. Although, it can be use as single restorative material, it is mainly used as a core material, veneered by a stratified feldspathic ceramic [28].

1.3 Zirconium biomaterial

Zirconium (Zr) is a chemical element and it belongs to the transitional metals. Its atomic number is 40 and its atomic mass is 91.224 g/mol. The melting temperature of Zr is 2680°C and the boiling temperature is 4371°C. Zr was originally discovered by the chemist Martin Heinrich Klaproth in Germany in 1789 and was isolated by the Swedish chemist Jons Jacob Berzelius in 1824. The first reported biomedical application of Zr was in 1969 by Helmer and Driskell [29]. Zr is never found as a native metal in nature. It is part of igneous rocks mixed with other elements such as iron, titanium and silicon oxide. The main source of Zr is Zircon ($ZrSiO_4$) which is found primarily in Australia, South Africa, Brazil, India, Russia, and the United States. Zr also found in many other mineral species including baddeleyite [30].

1.3.1 Concepts in Zirconia Ceramics

Zirconia is a polycrystalline material, which can exhibit structural polymorphism (monoclinic, tetragonal and cubic form) depending on pressure and temperature conditions. Pure zirconia is monoclinic at room temperature and could be stable up to 1170°C. Above this temperature, it transforms into more dense, tetragonal phase, with volume decrease (5%) and initiation of cracks within its structure. The tetragonal form is stable between 1170 and 2370°C, while at higher temperatures ZrO_2 acquires a cubic structure as shown in Fig. (1-1). Reversely, during cooling, tetragonal to monoclinic (T-M) transformation occurs, followed by volume expansion (3-4%).

The addition of stabilizing oxide to pure zirconia enables inhibition of phase transformations and allows generation of a multiphase material at room temperature, termed stabilized zirconia. Fully stabilized zirconia (additives: CaO, MgO, Y_2O_3) has a cubic form and presents material of increased hardness and high thermal shock resistance, familiar in engineering ceramics [31].

The other type of multiphase zirconia with desirable mechanical properties is partially stabilized zirconia (PSZ).Its microstructure, at room temperature, consists generally of cubic zirconia as main phase, and monoclinic and tetragonal zirconia precipitates as minor phase.

The proofs of in vitro biocompatibility of ZrO_2 and advances in computer aided design–computer aided manufacturing (CAD/CAM) technology were the essential facts that promoted this material in modern dentistry in a variety of clinical applications; root canal posts, orthodontic brackets, implants, implant abutments and frameworks for all-ceramic restorations [32].

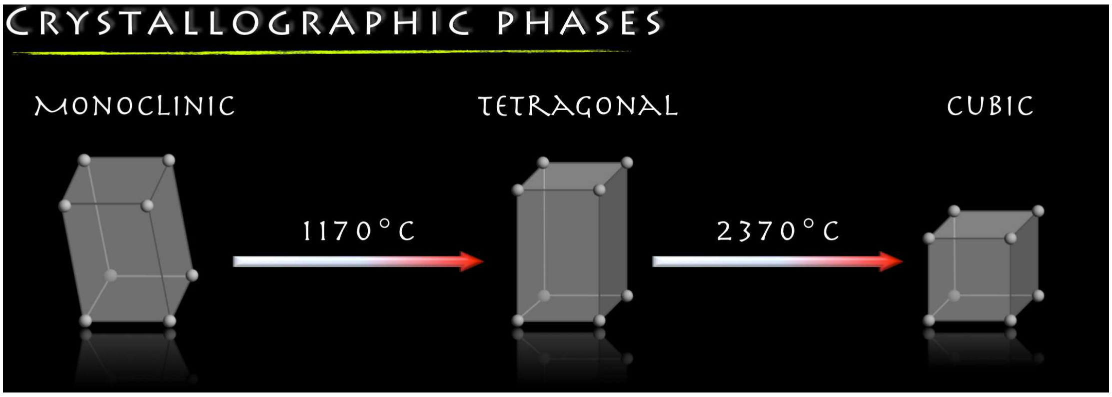


Figure (1-1). Crystallographic and relative temperature of the three zirconia phases [32].

1.3.2 Characteristics of Zirconia

1.3.2.1 Optical characteristics

Zirconia has an adequate level of opacity. Its opaque optical behavior can be attributed to the fact that the grain size is greater than the wavelength of light, and also that zirconia has a high refractive index, low absorption coefficient and high opacity in the visible and infrared spectrum. [4]. The opacity of zirconia is very useful in clinical situations where polychromatic substrates need to be masked so as not to interfere with the aesthetic result. Blackened teeth, pins and metal cores can be adequately masked by zirconia infrastructures.

Because of its opacity, zirconia cannot be used as a restorative material alone. With the current processing technologies and due to their homogeneity, high density (residual porosity <0.05%), even in limited thickness (0.5 mm) and they also allow a controlled translucency after lamination, thus, zirconia mostly used as an infrastructure, covered with translucent ceramics which exhibit characteristics that may look like natural teeth [33].

1.3.2.2 Biological characteristics

Studies have been confirmed a high biocompatibility of zirconia, especially when it is completely purified of its radioactive contents [34]. Generally, ceramics are inert materials, which have no adverse local or general tissue reactions. As the ceramic prostheses are made with highly polished surface, they can contact the gum tissue and assist in the maintenance of gingival architecture. Depending on the smoothness, the ceramics prevent the formation of plaque, making a favorable surface for the gingival tissues. Zirconia based ceramics are chemically inert materials, allowing good cell adhesion, and no adverse systemic reactions have been associated with it [35]. However, immune localized inflammatory reaction can be promoted by particles from the degradation of zirconia at low temperature or from the manufacturing process [36].

1.3.2.3 Mechanical characteristics

- **Flexural strength**

Fracture toughness is defined as the level of critical stress at which a particular defect starts to grow. It is determined numerically as the critical value that causes crack extension in the "mode I", or the opening mode by tension perpendicular to the crack. It is strongly affected by the size of flaws and defects on the surface of the material tested [37]. As microcracks and defects that inherently grow during the thermal and mechanical processes can influence the measurement of resistance [38], confirmed that surface defects and microcracks in ceramic Y-TZP zirconia are made internally on the surface machined by CAD-CAM techniques. Milling may introduce residual surface compressive stresses that can increase the resistance of zirconia ceramics. On the other hand, severe wear can make profound defects, which act as stress concentrating areas. Other methods, such as the partial sintering of ceramics manufacturing, and wear-free procedures, should be progressed to obtain crowns and bridges of the Y-TZP system with increased strength and reliability. Another important fact is that the accumulation of microcracks resulting from loading in an aqueous environment can cause surface defects that create a tension in areas of local concentration, facilitating the initiation of fracture under low level applied stresses[39].

- **Subcritical crack growth**

The subcritical crack growth (SCG), is one of the major causes of damage to ceramics and usually occurs as a function of time, which consists of a slow propagation of failures, SCG occurs under constant load due to the corrosive action in the region at the crack tip. In ceramics, the cyclic loading also accelerates the crack propagation and decreases its threshold due to degradation of toughening mechanisms [40]**.** Spread of the crack is observed faster in the presence of water, due to a high concentration of water molecules around the crack that increases the rate of crack growth because it facilitates the Zr-O-Zr union cleavage at the end of the crack [41].

1.4 Types of zirconia ceramics

There are three types of zirconia ceramic available for dental health care applications [42]:

1. Magnesium cation-doped partially stabilized zirconia (Mg-PSZ),
2. zirconia-toughened alumina (ZTA),
3. yttrium cation-doped tetragonal zirconia polycrystal (3Y-TZP)

1. Mg-PSZ (magnesia partially stabilized zirconia)

The microstructure of Mg-PSZ consists of an array of cubic zirconia partially stabilized by 8 to 10mol% of magnesium oxide. Fully sintered blocks have been manufactured with this material, and require rigid and strong machining systems [4], such a material has not been extensively used, neither has it encountered large popularity due to its remarkable porosity, large grain size (30–60 µm), low stability, tendency to framework wear, and overall poor mechanical properties, especially when compared to 3Y-TZP.). A dental ceramic system called Denzir–M (Dentronic AB, Skellefteå, Sweden) is an example of a fully sintered Mg-PSZ ceramic for dental crown and bridge work [43].

2. Zirconia-toughened alumina (ZTA)

ZTA utilizes the stress-induced transformation capacity of zirconia in an alumina matrix. ZTA can be manufactured according to two different processes: soft machining or slip casting. The latter presents the advantage of a more limited shrinkage but, at the same time, higher porosity and poorer mechanical properties than yttrium partially-stabilized tetragonal zirconia polycrystal (3Y-TZP), Moreover, stabilization by cerium oxide provides better thermal stability and resistance to low temperature degradation than Y-TZP [44, 45].

3. Yttria stabilized tetragonal zirconia polycrystal (3Y-TZP)

This type consists of an array of partially stabilized zirconia with a 2-4 mol% yttria oxide. Since 1977, it was reported that ZrO_2 fine grain (usually <0.5 μm) with small concentrations of Y_2O_3 stabilizers could contain up to 98% of the metastable tetragonal phase after sintering. The main feature of this microstructure is to be formed by tetragonal grains of uniform diameter in the order of nanometers, sometimes combined with a small fraction of the cubic phase. The YSZ (yttrium oxide) is suitable for optical applications due to its high refractive index, low absorption coefficient and high opacity in the visible and infrared spectrum. Its first applied in the medical field of orthopedics, with significant success due to its good mechanical properties and biocompatibility [29]. In dental applications, it is fabricated with microstructures containing small grains (0.2 to 0.5 μm in diameter) depending on the sintering temperature, which avoids the phenomenon of structural deterioration or destabilization in the presence of saliva, slowing the growth of subcritical cracks [46]. 3Y-TZP was available in dentistry for the fabrication of dental crowns and fixed partial dentures. The restorations were processed either by soft machining of presintered blanks followed by sintering at high temperature, or by hard machining of fully sintered blocks [47]. Its mechanical properties are highly depended on the grain size [42]. The sintering conditions had a strong impact on both stability and mechanical properties of the final product as they dictate the grain size [48].

Sintering with higher temperatures and longer times leaded to larger grain sizes [49]. Later; 3Y-TZP for soft machining of dental restorations was developed, It utilizes final sintering temperatures varying between 1350 and 1550 ◦C depending on the manufacturer. This sintering temperature with such a wide range was therefore likely to have an influence on the grain size and later the phase stability of 3Y-TZP for dental applications.

1.5 Dental Application of Zirconia

1. Zirconia-Based Crown and Bridge

The fabrication of zirconia frameworks of either presintered or highly isostatic pressed zirconia for crown and bridge has been employed widely [50]. Zirconia frameworks offer new perspectives in metal free fixed partial dentures and single tooth reconstructions because of zirconium's high flexural strength of more than 900 MPa and showed good first clinical results. Y-TZP is the most recent framework material for the fabrication of all-ceramic FPDs either in anterior or posterior sites. The load bearing capacity of Y-TZP FPDs was found to be significantly higher than other conventional all-ceramic systems, such as lithium-disilicate glass ceramics and zirconia-reinforced glass-infiltrated alumina [51].

2. Zirconia-Based Dental Posts

A metal post and core system restricts light transmission and thus gives an undesirable dark shadow in the root and cervical areas, especially through thin periodontal tissues and significantly decreases the value of the coronal part of the restoration [52]. With the introduction of custom made all-ceramic posts and cores or zirconium dioxide (ZrO_2) prefabricated posts [53, 54], an unique esthetic approach has been developed in combination with all-ceramic crowns. Dentin-like shade all-ceramic posts and cores contribute to a deeper diffusion of light and therefore provide an appropriate depth of translucency [55]. In situations where all-ceramic restorations are used for restoring anterior teeth, metal posts may result in unfavorable esthetic results [54, 56]. Additionally, corrosive reactions with prefabricated posts may cause complications involving the surrounding tissues and oral environment, including a metallic taste, oral burning sensitization, oral pain, and other reactions [57]. These concerns have led to the development of white or translucent posts made of zirconia and other ceramic materials.

3. Zirconia-Based Esthetic Orthodontic Brackets

Zirconia has been applied for the fabrication of esthetic orthodontic brackets [58]. Polycrystalline zirconia brackets, which reportedly have the greatest toughness amongst all ceramics, have been offered as an alternative to alumina ceramic brackets [59]. Y-TZP orthodontic brackets provide enhanced strength, superior resistance to deformation and wear, reduced plaque adhesion, and improved esthetics.

4. Zirconia-Based Implant Abutments

As a result of utilizing the zirconia ceramics for the fabrication of tooth-supported restorations, this encouraged the clinicians to extend its application for implant-supported restorations. Utilizing zirconia as implant-supported restorations is due to the higher toughness and modulus of elasticity. In stabilized and transformation toughened forms, zirconia provides some advantages over alumina in order to solve the problem of alumina brittleness and the consequent potential failure of implants [60]. These Y-TZP implant abutments are distinguished by their tooth-matched color, enhanced biocompatibility, metal-like radiopacity for better radiographic evaluation, and, ultimately, reduced bacterial adhesion, plaque accumulation, and inflammation risk [61].

1.6 Configuration of Zirconia

The zirconia in dentistry is milled in pre-sintered stage as observed in Fig. (1-2). This configuration is a soft, chalk-like stage that is called "green" stage. During the sintering process, the material shrinks and reduces with the volume shrinkage of about 20-25%. It's very important to know the exact volume shrinkage information for the individual zirconia blank blocks in order to optimize the fitting of the restoration. Sintering is an important step in processing zirconia; the zirconia is raised to a high temperature (about 1500ºC) having the furnace raising temperature in a steady rate. The process results in pores being reduced between particles, increasing strength but results in shrinkage of the structure [62]

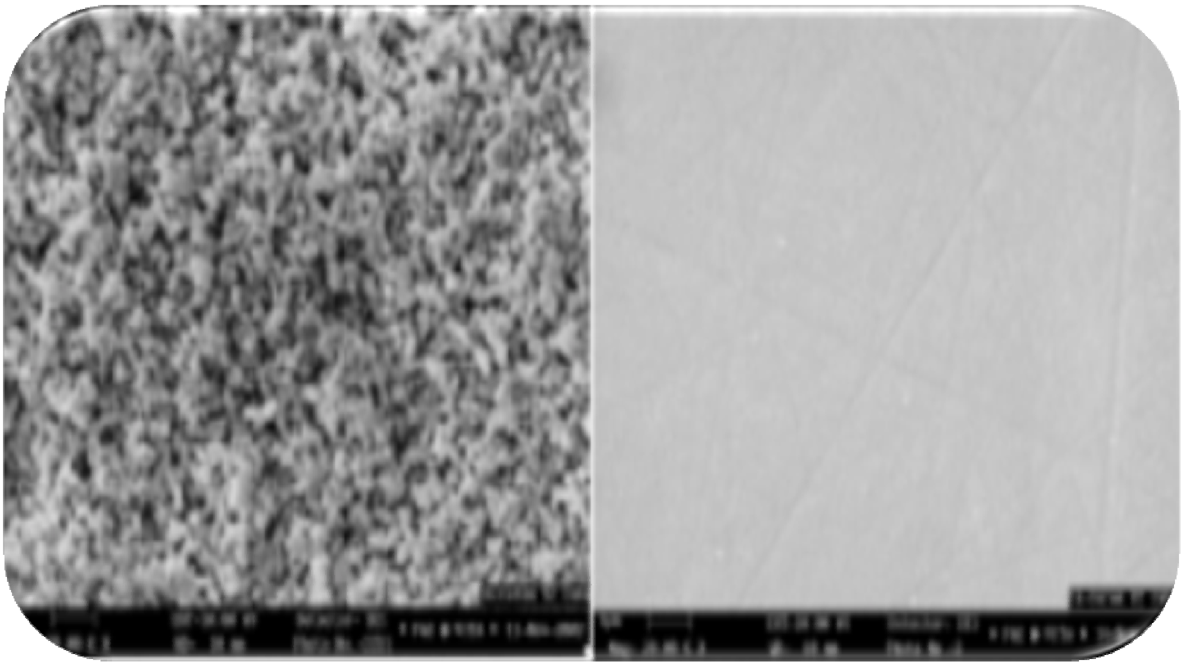


Figure (1-2) Microscopic image of pre-sintered zirconia (left) and after sintering (right). (Source: VITA In-ceram YZ manual).

The zirconia is called hipped (hot iso-static pressed) when the material is industrially sintered. Hipped zirconia has a constant grading and thus a more homogeneous quality. As expected, milling time and wear of the milling tools is higher in comparison to the pre-sintered variants.

1.7 Prefabrication Procedures

The most commonly used techniques for prefabrication of zirconia frameworks are: “soft machining” of pre-sintered blocks or “hard machining” of fully sintered blocks [63].

1.7.1 The soft machining process

This procedure is based on milling of pre-sintered zirconia blocks that are fully sintered at a final stage. It is the most common manufacturing system for 3Y-TZP, which was developed in 2001. It consists of compacting zirconia powder in the presence of a binder that makes it suitable for pressing, through a cold, isostatic pressing process; this leads to homogeneous distribution of the components inside the block [64]. The binder is eliminated during a pre-sintering heat treatment. This step has to be controlled carefully by manufacturers, particularly the heating rate and the pre-sintering temperature. If the heating rate is too fast, the elimination of the binder and associated burn out products can lead to cracking of the blanks. Slow heating rates are therefore preferred [4]. Processing at a proper pre-sintering temperature of zirconia is an important factor because this parameter influences hardness, machinability and roughness of the blocks. These proprieties act as reverse factors; this means if hardness of block is adequate, manipulation of blocks is performed safely, but, high hardness is unsuitable for machinability. Moreover, an increase in pre-sintering temperatures generates rougher surfaces. Soft milling involves machining enlarged frameworks of pre-sintered blanks of zirconia, also called the ‘‘green’’ state. Usually, CAD software programs design the enlarged framework to compensate shrinkage. After this step, the zirconia is then sintered completely in a furnace to achieve its final shape, strength, and physical properties, which is accompanied by shrinkage of the milled framework [65].

1.7.2 Hard-milling hot isostatic pressed (fully sintered)

In this procedure the 3Y-TZP blocks are sintered and condensed at high temperatures (1400–1500°C) and under high pressure in inert gas medium. These blocks are very hard, dense and homogeneous [66]. The blocks can then be machined using a specially designed milling system. Due to the high hardness and low machinability of fully sintered Y-TZP, the milling system has to be particularly robust, as the hard-milling may introduce micro cracks in the framework during the milling process, which affects the physical strength and longevity of the prosthesis.

However, HIPed zirconia provids superior marginal fit because no shrinkage is involved in the manufacturing process, also precise margins is difficult to be achieved with non-HIPed zirconia, giving rise to recurrent caries, periodontal conditions and unaesthetic margins[65].

The major disadvantage of the hard machining technique is more time-consuming and requires very tough and wear-resistant cutting devices. Provision of these pieces of equipment makes the production procedure more costly [67].

1.8 Bonding of Zirconia

The zirconium oxide, with its silica free composition, makes it difficult to bond to traditional resin composite cements. Resin cements have been selected for their advantageous mechanical and adhesive properties compared with conventional cements. The clinical application of resin-bonded fixed restorations requires a strong and stable resin bond to the ceramic [68]. The choice of resin cements also plays a significant role in adhesion to zirconia. Phosphate ester monomer-containing adhesion promoters with polar functional groups such as 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) could make covalent bonds with the hydroxyl groups on the zirconia surface [69]. Kern and Wegner were the first to report the long-term bond strength of phosphate monomer-containing resin-based composite cements to ZrO_2. Wolfart et al. evaluated the bond strength and the durability of two composite resins to zirconia ceramic. They concluded that the use of the (10-MDP) or MDP-containing composite resin Panavia F (Kuraray Medical Inc., Kurashiki, Japan)can be recommended for bonding of ZrO_2 for clinical use[70]. Unfortunately, low degree of conversion of such acidic monomers, less hydrolytic stability, and low diffusion level in to the dentin are limitations of self-adhesive cements [71].

1.9 Surface Roughness of Zirconia

To achieve reliable adhesion to ceramics, it is necessary to carry out a surface pre-treatment. Roughened ceramic surfaces may allow resin cements to flow into the microretentions and create a stronger micromechanical interlock [72]. The long-term success of zirconia-based restorations depends on the preparation technique of the internal surfaces of ceramics prior to cementation, cement properties and bond strength between the cement and the ceramic [1]. A strong bond depends on micromechanical interlocking and chemical bonding to the ceramic surface that requires surface roughening and cleaning for a suitable surface activation. Surface treatment of ceramics increases the surface area and creates microporosities on the ceramic surface, which enhance the potential for mechanical retention of the luting composite resin. Common surface treatment methods (STM) consist of (1) Acid etching (typically hydrofluoric acid(HF)) (2) Grinding. (3) Abrasion with diamond (or other) rotary instruments,(4) Air abrasion with alumina (or other) particles(5) Application of different laser types and(6) A combination of any of these techniques that actually roughen surfaces [14].

1.9.1. Mechanical Methods

Since zirconia is resistant to chemical treatment, very aggressive mechanical abrasion methods must be used to provide sufficient surface roughness. Surface abrasion or roughening (grinding, airborne particle abrasion using Al_2O_3 or other abrasive particles ranging in size from 50 to 250 µm, rotary abrasion using diamond burs), establishes adhesion only through micro-mechanical retention [73]. These treatment methods have both positive and negative effects on the mechanical properties of Y-TZP materials in that they are generally easy to apply in a dental environment. However, research has shown that surface grinding techniques, have no significant effect on increasing the bond strength of zirconia to resin cements [17]. Another problem with these techniques is that they can create sharp crack tips and structural defects, making zirconia more susceptible to radial cracking during function [74]. Several studies have showed that sandblasting surface treatment with aluminum oxide particles is the most effective when compared to fine polishing, grinding with an abrasive wheel, or grinding using a diamond bur [75]. In the selective infiltration etching (SIE) method the surface of zirconia is coated with a glass-containing conditioning agent. Later on, the material is heated above glass transition temperature, until the optimal grain boundary diffusion is achieved. After cooling to room temperature, the glass is dissolved in an acidic bath to eliminate all traces of conditioning agent. [12]. SIE transforms dense, nono-retentive, relatively smooth and low energy surface of zirconia to highly active and well bonding surface by increasing the surface area available for bonding with increased microtensile bond strength[76].

Hot chemical etching solution is also recommended for conditioning the zirconia substrate. It selectively etches the zirconia and creates micro-retentions on the surface by modifying the grain boundaries through removal of the less arranged atoms. It is possible that this technique could enhance the mechanical retention of ZrO_2 greater than SIE [77].

Also the application of fused glass micropearls is another surface treatment method where slurry of micro-pearls is painted on a ZrO_2 surface and fired in a furnace. The fused glass film increased surface roughness, and significantly increased the bond strength of resin cements to ZrO_2 [78].

1.9.2 Chemical Methods

One of the most common STM of ceramic restorations is based on micromechanical bond obtained with HF acid etching. HF removes the glassy matrix of glass ceramics creating a high surface energy substrate with microporosities for the penetration and polymerization of resin composites, that is, enabling a micromechanical interlocking [79].

Silanes coupling agents are also commonly used in dentistry. Silanes are also believed to promote surface wetting, which enhances potential micromechanical retention with low viscosity resin cements [80]. Studies showed that application of silane alone for the enhancement of luting of ZrO_2 resulted in low bond strength.Alternatively, the use of a zirconate coupling agent has been explored for pure zirconium and ZrO_2 [17]. This coupling agent enhanced bonding to resin cements but bond strength is significantly decreased after thermocycling.

Tribochemical silica coating(TBC) techniques also have employed which uses alumina particles modified with silica for air abrasion and embedding silica particles in the ceramic surface. Silica particles create a base for micromechanical bonding and interlocking in ceramic. The next step is application of a silane which enables chemical adhesion between ceramic and resin cement [81].

Other process is Gas-phase fluorination process which is chemically modifying zirconia by creating thin oxyfluoride conversion layer on its surface which would further react with organo-silanes, enabling silicon bonding to the surface. The recent studies show that the fluorination treatment on roughened or polished zirconia displayed higher bond strength as compared to commercially available treatments [82].

1.10 Laser basics

Laser is a light with specific properties. Light is an electromagnetic energy that exists as tiny particles called photons, move in space as waves. The term Laser is an acronym for Light Amplification by Stimulated Emission of Radiation [83].

1.10.1 Components of laser system

1. The optical cavity (resonator): It forms the laser cavity and has two mirrors, one at each end. They cause the bouncing of the photons to stimulate the release of more photons; therefore, one of the mirrors has high reflectance, while the other has less reflectance. This stimulation process may cause increase in temperature; therefore, heat must be controlled by using cooling systems [84].

2. Active medium: It may be gas (CO_2), solid (Nd:YAG), or liquid, placed in the cavity. It is the source of the photons, and it amplifies the photon chain reaction that results from stimulated emission when its atoms are excited (84).

3. Pumping medium: It is the energy source that applies energy to pump the atoms in laser excited state. It may be flash lamb strobe device, electrical circuit, or others.

The illustration in Figure 1-3 shows the main components of lasers.

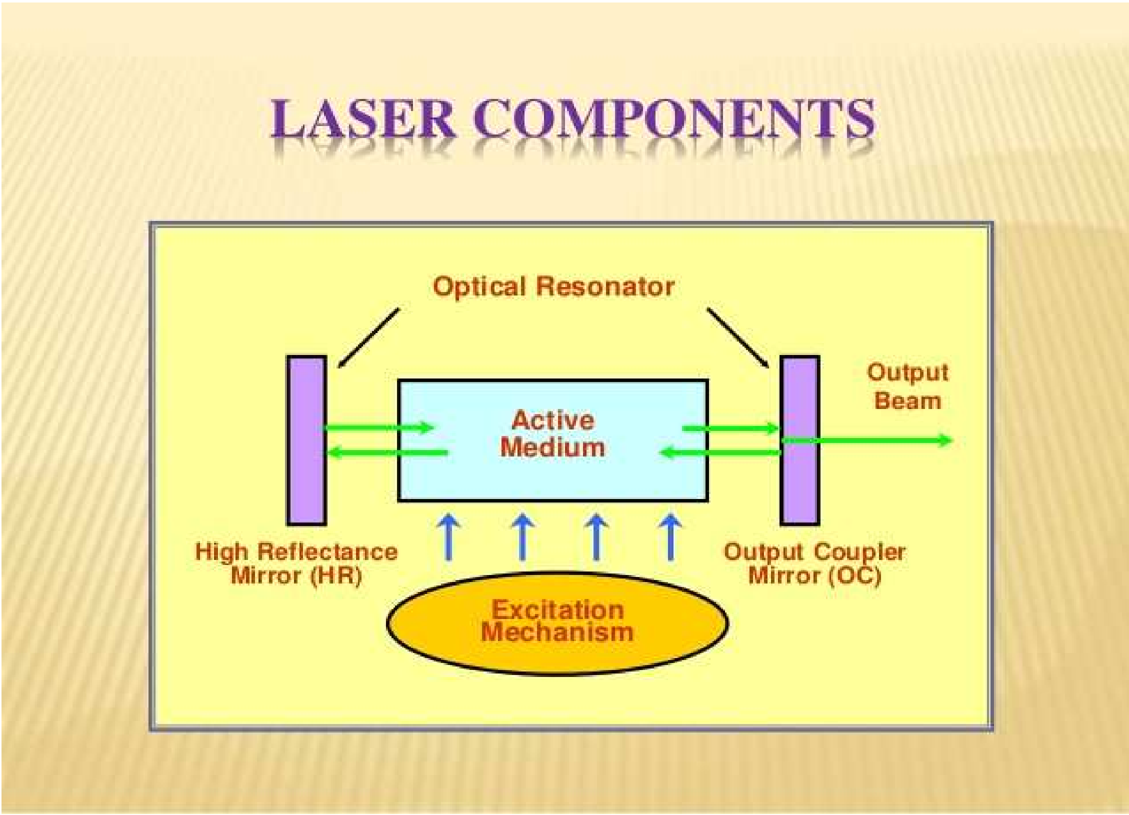


Figure (1-3) Laser components [85]

1.10.2 Important terms in laser:

Laser Wavelength λ (m): It is the length of the light wave, or the shortest distance at which the wave pattern usually repeats itself. In laser studies, we use micrometer and nanometers to describe the wavelength.

Laser Frequency υ (Hz): Is the number of times that the wave oscillates per second.

Pulse Duration (pulse width): It is the time of the single pulse in pulsed laser**.**

Spot Size: It is the diameter of the beam of laser radiation. It influences the concentration of photons in the target.

Pulse Repetition Rate: It is the number of pulses per second.

Power Density (Intensity) (Irradiance) W/cm^2: It is the concentration of photons in a unit area, or the power of laser beam divided by its cross section.

Energy Density (Fluence) J/cm^2: It is the total energy delivered by laser on a unit area during an expose time.

Peak Power (P peak): It is the maximum amount of power that the laser single pulse delivers to matter.

P peak = energy of single pulse / pulse duration**.**

Average Power (P ave): It is the amount of energy released over the period of the cycle.

P ave = energy of single pulse * Pulse Repetition Rate (PRR)[86].

1.10.3. Characteristics of Laser

- **Coherence**

Each wave of laser light is identical in physical size and shape. This means that the amplitude and frequency of all the waves of photons are identical, producing a specific form of focused electromagnetic energy [87]. Temporal or longitudinal coherence is referring to the closeness in phase of various portions of the laser frequency bandwidth. While the closeness in phase of different spatial portions of the beam after the beam has propagated a certain distance is referred to as spatial or transverse coherence [88].

❖ **Collimation**

All the waves are traveling in a specific direction and hence they are all parallel to each other and travel for long distance. Lasers produce the most collimated light with the least divergence angle [87, 88].

❖ **Monochromaticity**

Laser light is generated only as one color. This color can be either visible or invisible to the human eye.

Monochromaticity refers to how pure in color (frequency or wavelength) the laser beam is, as all the photons have the same wavelength [88].

❖ **Focusability**

It is the ability of focusing the laser beam to be precisely to a very small spot size . Transverse electromagnetic mode (00) can produce a laser light with the smallest beam diameter, nearest to the dimension of the wavelength of the laser [87, 88].

❖ **Brightnees**

It translates to the high concentrations of energy when the laser is focused on a small spot. This property arises from the high degree of collimation of laser light as it moves through space maintaining its concentration, thus the characteristic brightness [89].

1.10.4 Laser in dentistry

The dental lasers in common use today are Erbium, Nd:YAG, Diode, and CO_2 as illustrated in Fig.(1-4). Each type of laser has specific biological effects and procedures associated with them.

- **Erbium Lasers**

The Er,Cr:YSGG laser has an active medium of yttrium-scandium-gallium-garnet doped with erbium and chromium ions and a wavelength of 2,780 nm. The Er:YAG laser has an active medium of yttrium-aluminium-garnet doped with erbium ions and a wavelength of 2,940 nm. The erbium lasers are hard and soft tissue capable and have the most FDA clearances for a host of dental procedures [90, 91]. Their primary chromophore is water, but hydroxyapatite absorption occurs to a lesser degree. Tooth preparation is quite efficient with erbium devices and many procedures can be done without local anesthesia. Smear layer is virtually eliminated and the laser has a significant disinfecting effect on the dentin and enamel to be restored. Bone cutting with erbium lasers results in minimal thermal and mechanical trauma to adjacent tissues. Erbium lasers are excellent soft tissue devices as well. Rapid healing with minimal postoperative pain when soft tissue procedures are done with erbium lasers, including periodontal procedures, gingival contouring, biopsies, frenectomies, pre-prosthetic procedures and the like. Erbium lasers can also be used to safely scale root surfaces during periodontal procedures which has the added benefit of root surface decontamination [92].

- **CO_2 Laser**

CO_2 Laser has been available in medicine since the early 1970's and have been used in dentistry for more than 25 years. The CO_2 gas is mixed with nitrogen and helium gases to form the active medium which is pumped with an electrical current. Articulated arms or hollow waveguides are used to transmit CO_2 laser beams, and it emits infrared light at different wavelengths (9,300, 9,600, 10,300 and 10,600 nm). It is highly absorbed by water and exhibit excellent homeostasis. The traditional CO_2 (10600nm) are currently for soft tissue uses [91]. They are continuous wave lasers that can be operated in gated wave modes.

CO_2 laser is excellent tools for incising tissue for multiple purposes. Incisional and excisional biopsies, frenectomy, gingivectomy, pre prosthetic procedures, and the like are all achieved with excellent homeostasis. Sutures are rarely needed and the controlled thermal effects and sealing of nerve endings often makes for a very comfortable post-operative experience for the patient. This wavelength is also very effective for ablation and vaporization of leukoplakia and dysplasia.

A hard tissue capable CO_2 laser has become available recently with 9300 nm wavelength. This particular wavelength has a high absorption affinity for hydroxyapatite that allows for efficient vaporization of tooth structure [93].

❖ **Nd:YAG Laser**

It is the classical and most widely used solid- state laser [94]. The Neodymium:YAG lasers were the first types of true pulsed lasers to be marketed exclusively for dental use in 1990, emitting its strongest fundamental wave length at 1,064 nm. This wavelength is absorbed by pigment in the tissue, primarily hemoglobin and melanin [95]. Photothermal interaction predominates and the laser energy here can penetrate deeply into tissues. Nd:YAG also has excellent biostimulative properties. It has the unique capacity to stimulate fibrin formation.It is primarily used for periodontal treatments and its proclivity for pigmented tissue allows for effective debridement and disinfection of periodontal pockets. Bacterial decontamination in tissues treated with Nd:YAG laser energy also contributes to resolution of periodontal infection. Nd:YAG lasers can also be used for multiple soft tissue procedures such as gingivectomy, frenectomy, impression troughing, and biopsy [96].

Diode Lasers

A specialized semiconductor that produces monochromatic light when stimulated electrically is common to all diode lasers. Diode lasers are invisible near infrared wavelengths and current lasers range from 805–1064 nm. One exception is the Diagnodent caries diagnostic laser which uses a visible red wavelength of 655 nm. They can function with continuous wave or gated pulse modes. Diode lasers are becoming quite popular due to their compact size and relatively affordable pricing. Diode lasers are soft tissue only. The chromophores are pigments such as hemoglobin and melanin, similar to the Nd:YAG. They are quite effective for intraoral soft tissue procedures such as gingivectomy, biopsy, impression troughing, and frenectomy. Diode lasers also exhibit bactericidal capabilities and can be used for adjunctive periodontal procedures. They also are used for laser assisted tooth whitening [96].

- **Fractional CO_2 laser**

A CO_2 laser of wavelength 10600 nm with an advanced technique referred to as a factional CO_2 laser. Fractionated lasers deliver energy in parallel vertical columns of multiple microscopic thermal spots called microscopic treatment zones (MTZs), while the distance between spots remains intact and untreated [97,98].

The concept of fractional photothermolysis(FP) was introduced in 2003 [99]. In the fall of 2004, FP became commercially available for clinical use [100].

Studies have shown that fractional delivery may be superior to the traditional uniform delivery of heat due to the fact that higher irradiation within the columns results in more effect.

This can be achieved without increasing the power of the optical source, also due to increased surface-to-volume ratio of the (MTZ) with larger safety margins [99].

In dentistry, when surface treatment is of concern, the use of fractional CO_2 laser may have several advantages. The exact irradiation area can be predetermined by the apparatus and the laser irradiates multiple zones in the target area with predefined space between them.

As a result a more homogenous etching pattern can be obtained; also the need for manual movement of the laser handpiece is restricted during conditioning. In addition, less thermal damage to the underlying area compared to that occurs with conventional CO_2 laser [97].

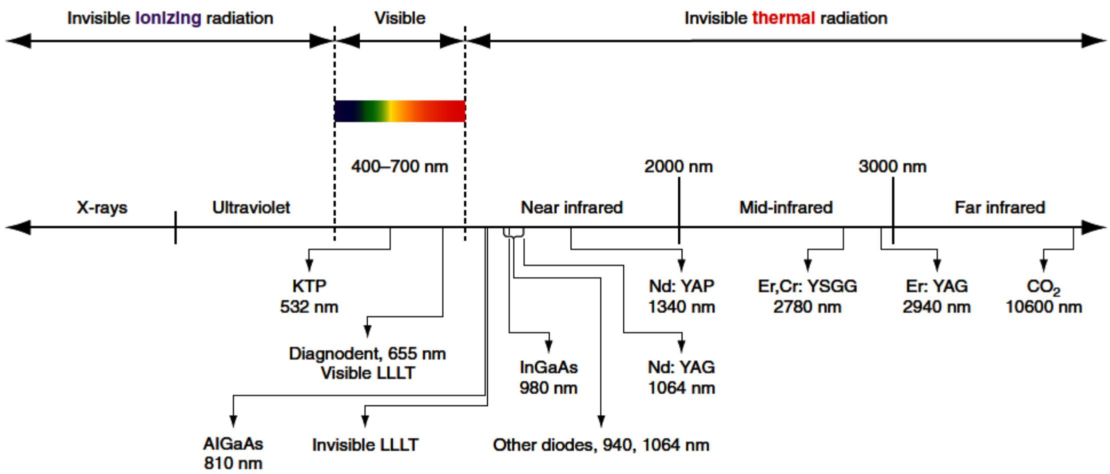


Figure (1-4) Currently available dental laser wavelengths on the electromagnetic spectrum [87].

1.10.5 Laser ceramic interaction

Structural ceramics those are commonly used in medical application including hips, teeth, and joints are zirconia (ZrO_2), alumina (Al_2O_3), hydroxyapatite and bioglass [101]. The interaction of laser beams with these materials is a complex phenomenon that depends on many factors. The energy density of the laser beam, the time of irradiation or pulse length, the wavelength, and the energy distribution within the beam are related to laser characteristics. The reflection and absorption coefficients, surface shape, homogeneity, melting point, and boiling point are factors related to the material [102]. Four physical phenomena can be distinguished when the laser beam is incident on the ceramic surface. These are reflection, absorption, scattering, and transmission as shown in Figure (1-5).

Reflection – The laser beam bounces off the surface with no penetration or interaction at all. Reflection is usually an undesired effect, because the energy could be redirected to an unintentional target, such as the eyes, which is a major safety concern for laser operation [103].

Scattering - the change in direction of a light wave on single or multiple occasions when it interacts with a small particle or object within inhomogeneous and/or turbid materials. The laser light passing through the object undergoes multiple scattering processes and is transformed from a narrow collimated beam to a broad diffused beam. The angle or quantity of scattering depends on the wavelength and relative sizes of the particles [103].

Transmission – when the laser light is transmitted through the object unchanged, as if it is transparent to the laser beam, i.e. It is the propagation of laser energy through the object, without effect on it.

This effect is also highly dependent on the wavelength of laser light. Only nonreflected and nonabsorbed or forward scattered photons will be transmitted through the tissue [103].

Absorption - Which is the usual desirable effect and the crucial of all the phenomena, is described as the interaction of electromagnetic radiation with the electrons of the material. It depends on both the wavelength and the spectral absorptivity characteristics of ceramics (e.g., reflection coefficient). The absorptivity is also influenced by the orientation of the ceramic surface with respect to the beam direction and reaches a maximum value for angles of incidence above 80° [104]. Thermal conductivity of structural ceramics is generally smaller than that of most metals; so radiation absorption sets in faster. Absorbed energy is converted into heat and its subsequent conduction into the material establishes the temperature distribution within the material.

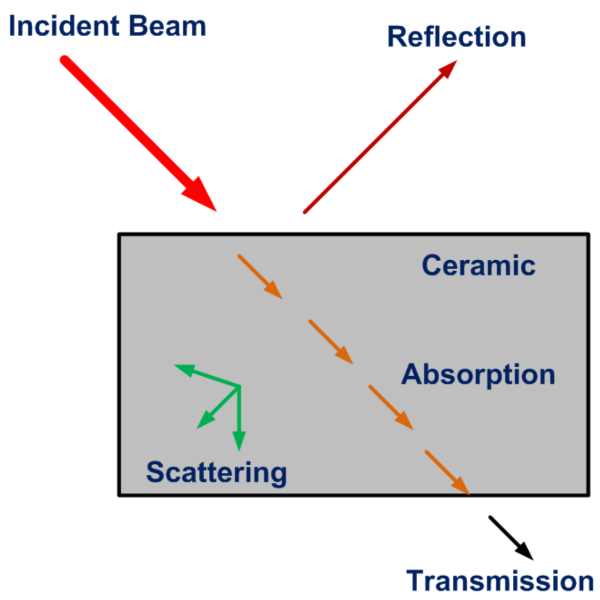


Figure (1-5) Interactions of incident laser beam with ceramic [105].

1.10.5.1Thermal Effects

The excitation energy provided by the laser is rapidly converted into heat and this is followed by various heat transfer processes such as conduction into the materials, convection and radiation from the surface [106].

The temperature distribution within the material as a result of these heat transfer processes depends on the thermo- physical properties of the material (density, absorptivity, emissivity, thermal conductivity, specific heat, thermal diffusivity), dimensions of sample(thickness) and laser processing parameters (absorbed energy, beam cross-sectional area).

The magnitude of temperature rise due to heating governs the different physical effects in the material such as melting, vaporization, plasma formation and ablation.

- **Melting**

At high laser power densities, the surface temperature of the ceramic may reach the melting point and material removal takes place by melting as shown in Figure (1-6). The surface temperature increases with increasing irradiation time [107].

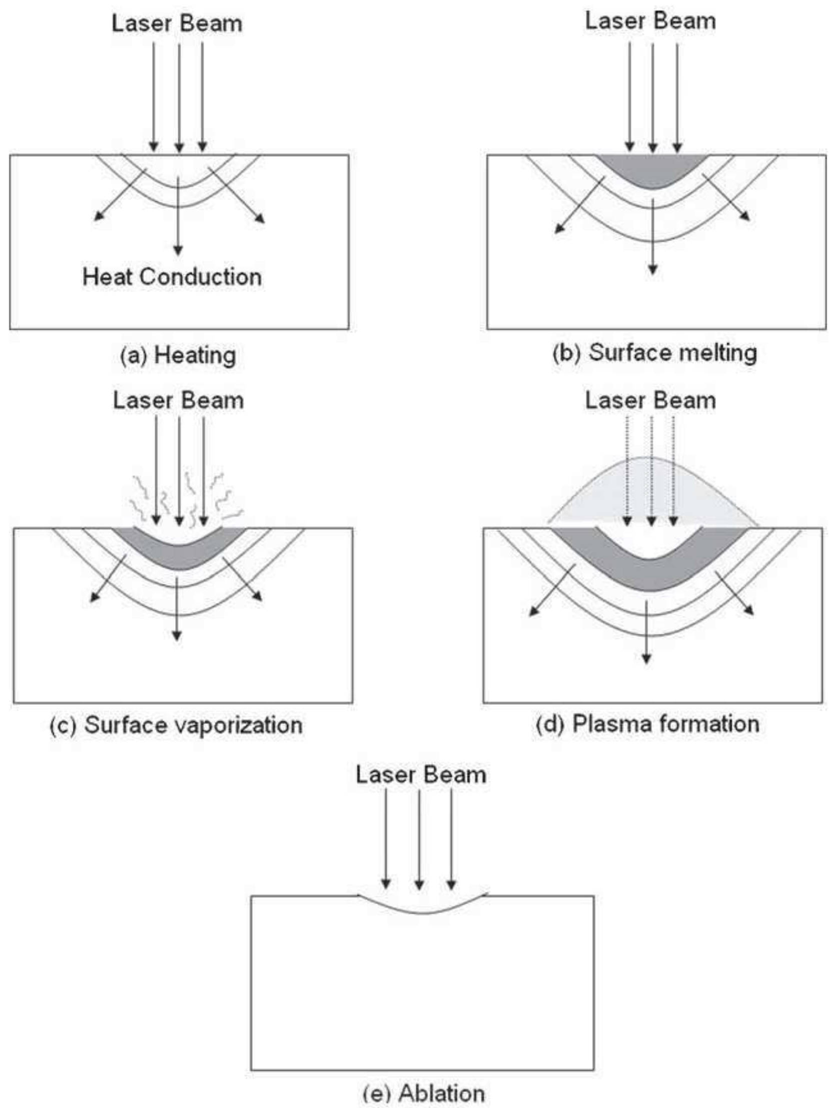


Figure (1-6). Various physical phenomena during laser-ceramic interaction [106].

❖ **Vaporization and plasma formation**

As the surface temperature of ceramic reaches the boiling point, further increase in laser power density or pulse time removes the material by evaporation instead of melting. After vaporization starts at the material surface, the liquid-vapor interface moves further inside the material with supply of laser energy and material is removed by evaporation from the surface above the liquid-vapor interface [106].

When the laser energy density surpasses a certain threshold limit, the material immediately vaporizes, gets ionized and forms plasma having temperatures as high as 50,000 K and pressures up to 500 MPa [108]. This thermal stress can result in cracking, so that optimized parameters should be set to keep the heat input to the bulk material lows. Mostly, cracks formation is avoided by precluding the formation of intense laser induced plasma [109].

- **Ablation**

When the material is exposed to sufficiently large incident laser energy, the temperature of the surface exceeds the boiling point of the material causing rapid vaporization and subsequent material removal by the process referred to as thermal ablation [110].

Ablation takes place when laser energy exceeds the characteristic threshold laser energy which represents the minimum energy required to remove material by ablation.

Above ablation threshold energy, material removal is facilitated by photo-chemical (bond breaking) processes, whereas photo-thermal (vibrational heating) effects take place below ablation threshold energy [106].

1.10.6. Surface Roughness by Laser

Different alternative methods to treat ZrO_2 surfaces have been proposed and evaluated in order to produce a reliable adhesion. Recently, due to advances in laser techniques, some studies have suggested the application of lasers. The underling principal of laser application is the convention of light energy into heat energy.

The most important interaction between laser light and the substrate is the absorption of laser energy by the substrate [111]. Previous studies employed Er:YAG to bring about changes on zirconia ceramics and improve their bond to tooth structures.

Laser application removed particles by microexplosions and by vaporization, a process called ablation. As a result macroscopic and microscopic irregularities formed on the zirconia surface after Er:YAG laser application increase the surface area for adhesion and may play an important role in bonding [112].

Some studies have shown Nd:YAG laser improves the bond strength to zirconia ceramics [18, 19]. These studies concluded that laser effects on surface roughness are obtained due to local temperature changes (heating and cooling) which roughens the zirconia surface by melting and creating random crystallization [113].

Recently researchers have shown that Zirconia ceramic can completely absorb the energy of the CO_2 laser beam. After absorption of laser energy, a process called heat induction produces shell-like rupture (conchoidal tears)—typical effects of surface warming—appear on the ceramic surface. These tears are believed to provide mechanical retention between resin composite and ceramic surface after resin tags penetrate into these cracks and set [114].

Femtosecond (FS) laser, with its ultrashort light pulses, is an innovative laser technology that can be used for multiple applications [115]. The FS laser pulses create minimal thermal and mechanical damage to the surrounding area during laser imaging, drilling, and ablation.

This quality makes them a good candidate for use in dental practices including surface treatment [116]. It have been found that the FS produced extensive surface fissuring, and this fissuring was more homogenous and had more regular surface characteristics which makes the FS to be an effective method for bonding resin cement to zirconia ceramic surfaces [20].

The disadvantages of lasers include stepped local temperature changes during the heating and cooling phases, which could create internal tensions damaging to teeth and dental materials [14].

1.10.7 Laser Safety Standards and Hazard Classification

Classification of a laser can be determined using the American National Standard Institute (ANSI) Z136.1-2014. This classification is based on their potential to cause biological damage, extending from Class 1 lasers, which may pose no implicit risk, to Class 4 lasers for which all safety measures are applicable.

1. Class 1 Laser System:

Any laser or laser system containing a laser that cannot emit laser radiation at levels that are known to cause eye or skin injury during normal operation. It is except from any control measures and considered safe. No requirement for special safety measures.

2. Class 1M Laser System:

Class 1M laser system is considered to be incapable of producing hazardous exposure conditions during normal operation unless the is viewed with an optical instrument such as an eye-loupe (diverging beam), and except from any control measures other than to prevent potentially hazardous optically aided viewing; and is except from other forms of surveillance.

3. Class 2 Laser System:

The emission of this class lasers is in the visible portion of the spectrum (0.4 to 0.7 μm), that are known to cause skin or eye injury within the time period of human eye aversion response(0.25seconds) and eye protection is normally afforded by the aversion response.

4. Class 2M Laser System:

The emission of this class lasers is in the visible portion of the spectrum (0.4 to 0.7 µm), and Eye protection is normally afforded by the aversion response for unaided viewing. However, Class 2M is potentially hazardous if viewed with collecting optical aids.

5. Class 3 Laser System (medium-power):

Lasers in this class are may be hazardous under direct and specular reflection viewing conditions, but is normally not a diffuse reflection or fire hazard.

There are two subclasses:

• A Class 3R laser system is potentially hazardous under some direct and specular reflection viewing condition if the eye is appropriately focused and stable, but the probability of an actual injury is small. This laser will not pose either a fire hazard or diffuse-reflection hazard.

• A Class 3B laser system (Medium powered lasers)visible or invisible regions that present a potential eye hazard for intrabeam(direct) or specular (mirror like)conditions. Class 3B do not present a diffuse(scatter)hazard or significant skin hazard except for higher powered 3B lasers operating at certain wavelengths.

6. Class 4 Laser System (ALL DENTAL AND MEDICAL SURGICAL LASERS)

High-powered lasers(visible or invisible) considered to present potential acute hazard to the eye and skin for both direct (intrabeam)and scatter(diffused) conditions. Also have potential hazard considerations for fire (ignition) and byproduct emission from target or process materials [117].

1.10.7.1 Hazards of Laser Radiation and Its Biological Effects

Because of the intensity of the output beam and the ability of lasers to produce very high concentrations of optical power at considerable distances, these lasers can cause serious injuries to the eyes and can also burn the skin.

These biological effects depend on the radiant exposure, wavelength, exposure time, environmental conditions, and individual susceptibility [118].

1. **Laser Hazards to the Eye**

The human eye is the most vulnerable tissue to all types of laser radiation. The tissue in the retina is susceptible to damage because the lens concentrates and focuses the laser beam on the retina.

In terms of the scope for repair, retinal injuries are more serious and may initially pass unnoticed, due to the lack of pain receptors [119]. Retinal burns are possible in the visible (400 - 700 nm) and nearinfrared (700 - 1400 nm) regions, that can cause partial or complete blindness. Although the cornea and lens structures are less easily damaged than the retina, excessive energy absorption can cause cell damage and impairment of vision [118].

Laser emissions in the ultraviolet (< 400 nm) and infrared to farinfrared (> 1400 nm) regions are primarily absorbed by and cause damage to the cornea that resulting in ablation, scarring and distortion of vision [120]. In the near-ultraviolet range (315 - 400 nm), some of the radiation reaches the lens of the eye [121].

So the different structures of the eye can be damaged from laser light based on the wavelength of the laser radiation.

2. Laser Hazards to the Skin

The effect of laser radiation on the skin depends on the wavelength and the pigmentation of the skin. In the visible spectrum the skin can reflect much of them, while in the infrared and ultraviolet regions, the skin becomes highly absorbing.

For infrared exposure, the results can be thermal burns or excessively dry skin depending on the intensity of the radiation. In the 230-380 nm range of ultraviolet light, erythema (sunburn), skin cancer, or accelerated skin aging are possible [121].

Also high power laser (class3B&4) can inflect considerable skin burns. However, the laser injury to the skin may be less serious than those to the eye [122].

3. Hazards of laser on Respiratory System

The inhalation of laser plume by the operator is hazard and it is called Laser Generated Airborne Contaminants (LGAC). This plume may contain vital stains of Human Papilloma Virus or any other organisms. This hazard can be controlled by using a high volume evacuation system during irradiation to clear the plume; also the operator should wear a surgical mask [123]

4. Hazards of Fire and Explosions

The laser systems include some flammable materials that may pose significant hazards. These flammable solids, liquids or gases are easily ignited if exposed to laser beam [124].

5. Hazards of Electric shock

The Class 4 laser systems have high powers, so there is a chance of electric shock hazards. If the lasing is directed to oral soft tissue, the tooth should be protected [124].

1.10.7.2 Laser controlling area warning signs

The purpose of the warning signs is to convey a rapid visual hazard-alerting message to the others that there is a laser system hazard in the area, and many protocols should by followed. These protocols include [125]:

- The laser system should be positioned in side-room, so there are more than one door before the system;
- Warning signs should be big, colorful, and in obvious position;
- Knocking before entering;
- Light signs should be illuminated during using;
- Laser Eye Protection Wears should be available;
- The area should be restricted for authorized persons only; and
- The operator should have enough training about the system parameters and uses.

1.11 Literature Review of Zirconia Surface Roughening Using Different Lasers Types

The enhancement of surface roughness and shear bond strength of resin cement to zirconia ceramic using different lasers was evaluated by several studies as shown in Table(1-1).

Table (1-1) The effects of different types of lasers on the shear bond strength (SBS) of resin cement to the zirconia ceramics.

Laser type	Wavelength (µm)	Mode of operation	Typical power(W)	Mean(SBS) ±SD(MPa)of laser group	Mean(SBS) ±SD(MPa)of control group
CO_2	10.6	continuous [126]	8	6.68±1.69	1.78±0.56
		Pulsed[127]	2	21.0 ± 2.7	
		Pulsed[19]	3	20.9±3.7	13.4±3.1
		Pulsed[128]	3	12.12 ± 3.02	5.95± 1.14
		Pulsed[129]	3	18.12 ± 0.82	9.17 ± 0.57
		Fractional [130]	20	28±4.9	9.4±1.2
Nd:YAG	1.064	Pulsed[131]	3	6.33±1.69	3.87±1.17
CO_2 and Er:YAG	10.6	Pulsed[132]	3	12.12 ± 3.02	5.97±1.14
	2.94	Pulsed[132]	2	8.65±1.77	
CO_2 and Er,Cr:YSGG	10.6	Continuous [133]	4	29.08±6.59	
	2.78	Pulsed[133]	3	27.52±4.85	
CO_2 and Nd:YAG	10.6	Pulsed[134]	3	14.00± 1.96	10.35±3.12
	1.064	Pulsed[134]	2	18.95±3.46	
CO_2 and Nd:YAG	10.6	Pulsed[135]	3	6.24±1.98	4.65±1.32
	1.064	Pulsed[135]	3	14.09±1.05	

1.12. Aim of the study

- Evaluation of the enhancement of the shear bond strength of zirconia ceramic to resin cement using fractional CO_2 laser as a surface treatment.
- Assessment of the temperature elevation of the zirconia specimen during irradiation with laser to determine the appropriate laser setting parameter that safely affect the zirconia surface.

Chapter Two

Materials and Methods

Chapter Two: Materials and Methods

2.1 Introduction

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

2-2 Materials

1. Zirconium blanks(Ceramill zolid 71XS/Amann Girrbach/Austria,lot no.1603000-68)
2. Bisco dual-cured adhesive resin cement, paste/paste mixing system (BisCem , lot no.1600007616, USA)
3. Customize made teflon holder to hold the ziconia specimen and fix laser hand piece in place.
4. Customize made silicone apparatus to mold the adhesive resin cement.
5. Cold Cure acrylic (Re ACROMED, Iran)
6. Deionized distilled water (IONTECH, Taiwan)
7. Plastic containers with their covers (IMIDRO, Iran).
8. Chisel end rod of stainless steel specially made for the shear testing.
9. Acetone alcohol (DAWD, Jordan)

2.3 Equipment

1. Fractional CO_2 laser system (CO_2 Fractional Laser Brochure,JHC1180, China)

2. Thermometer (AMPROBE TMD®-56, Everett, WA, USA).

The specifications are:

a. Highly accurate with 0.1% basic accuracy.

b. Dual input T1, T2

c. K-type thermocouple with range of (-200°C to 1372°C) and head diameter of 0.8 mm.

d. Measures temperature every one second.

e. All the collected data are arranged and processed with system software.

3. Sintering zirconia furnace (Ceramill; Amann Girrbach AG, Koblach, Austria)

4. Venire caliper (TOPEX Sp. Z o.o. S.K., Warsaw, Poland), the specification are:

a. Resolution: 0.01 mm.

b. Measurement accuracy: ± 0.02 mm.

5. LED Light curing system (QUAYLE DENTAL ,Spectrum 101,sussex,UK)

6. Instron Universal testing machine (LARYEE, WDW-50,50 KN,China)

7. CO_2 laser protective eyeglasses.

8. Stop watch (Software Application in cellphone)

9. Tourna machine (Hobbymat, Germany)

10. Atomic force microscope (AA3000,AnstgromAdvanced.Inc.,USA).

11. Stereo microscope (ME, 2665, Euromex, Holand)in which all the samples pictures are saved with system software.

12. Digital water bath (LABTECH, DAIHAN LABTECH, Korea, water thermal conductivity is 0.6 W/cm C).

13. Scanning Electron Microscope (SEM) (Inspect S50, FEI, USA).

14. Pumped Sputter Coater (Q150R Rotary-Pumped Sputter Coater, Quorum Tec., UK).

15. Ultrasonic cleaner (CD-7810A, New Trent, China)

16. Digital camera (China).

2.4 Laser System

Fractional CO_2 laser system that shown in Figure (2-1) (CO_2 Fractional Laser Brochure,JHC1180, China) has the following specification:

a. Laser wavelength: 10600 nm

b. Output power: ≤30 W

c. Pulse Duration Time per spot: 0.1-10 ms adjustable

d. Spots distance: 0.1 – 2.6 mm adjustable

e. Interval time (time between pulses): 0.1 ms-500 ms adjustable

f. Mode of scan: order, disorder, parallel (switching) g. Pulse energy: maximum 300 mJ

h. Area of Focal Spot: 0.05 mm^2

i. Output graphic: square, rectangle, triangle, circle, oval, hexagon, linear (scalable)

j. Graphic area: ≤ 20 mm × 20 mm

k. Optical system: 7 articulated arms

Figure (2-1) Fractional CO_2 laser device

2.5 Method

2.5.1 Construction of Zirconia Specimens

Forty four zirconia discs were milled from Pre-sintered Y-TZP zirconium oxide blocks, Figure (2-2)(Ceramill zolid/Amann Girrbach/Austria)with the physical and chemical properties are listed in Tables(2-1) ,(2-2) . First of all, each block was divided into blanks with the dimensions(13mm,13mm,12mm) by a cutting saw as shown in Figure (2-3), each blank was then glued into a fitting pin as Figure (2-4). The fitting pin was then placed into the designated place in the milling machine, in a way allowing a rotational movement around its axis at a high speed. At the same time, a circular shape carbide bur (Gerdau, Brazil) was used for cutting the sides of the sample.

The bur was moving around the blank in circular movement and upward movement providing a precise cutting to obtain the disc shape specimen with the desired dimensions (12.5mm in diameter and 2.5 mm in height) as illustrated in Figure (2-5). The obtained discs were then sintered in a special furnace of zirconium shown in Figure (2-6) according to manufacture instructions at 1450 C/ for 10 hours including cooling, according to manufacturer’s instructions. During this process a 3-dimensional volumetric shrinkage of the milled discs of approximately 25% took place that is why the discs were milled approximately 25% larger. Following sintering each zirconia disc was (10 mm in diameter , 2 mm in height)as in Figure(2-7).,that was measured by using the vernier caliper that shown in Figure(2-8). Before the surface treatments, all specimens were ultrasonically cleaned in distilled water and acetone for 15 minutes as in Figure (2-9), to remove contaminants and dried naturally in the atmosphere [131]. Then all the specimens were microscopically examined at a magnification power of 100X and those having bubbles or fissures were replaced by perfect specimens.

Table (2-1): Zoild physical properties (Ceramill Systems, Amann Girrbach, Germany,2012).

Bending strength(4-point)	>1000 Mpa
Elastic module	>200 Gpa
Grain size	≤0.6 μm
Density	≥6.06 g/cm^3
Open porosity	0%
Thermal expansion coefficient (25-500 °C)	$10.4 \pm 0.5 \times 10^{-6}$ 1/K
Chemical solubility	<5 $\mu g/cm^2$
radioactivity	<0.2 Bq/g

Table (2-2): Zoild chemical properties (Ceramill Systems, Amann Girrbach, Germany,2012).

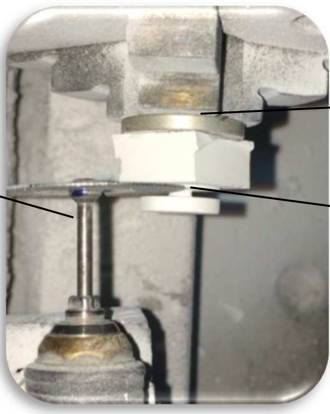
Oxide	Mass percentage
$ZrO_2+HfO_2+Y_2O_3$	>99.0
Y_2O_3	4.5-5.4
HfO_2	<5
Al_2O_3	<0.5
Other oxides	<0.5

Figure (2-2)Y-TZP zirconium oxide blocks,

(Ceramill zolid/Amann Girrbach/Austria)

Figure (2-3) block was divided by a cutting saw

Figure (2-4) fitting pin



Figure(2-5) cutting with a carbide bur

Figure (2-6) Sintering furnace

Figure (2-7): Final shape and size of the zirconia sample

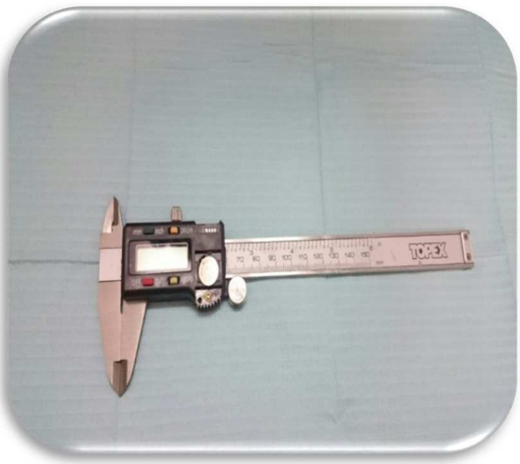


Figure (2-8) Digital vernire

Figure (2-9) Ultrasonic cleaner

Specimens were divided into the following:

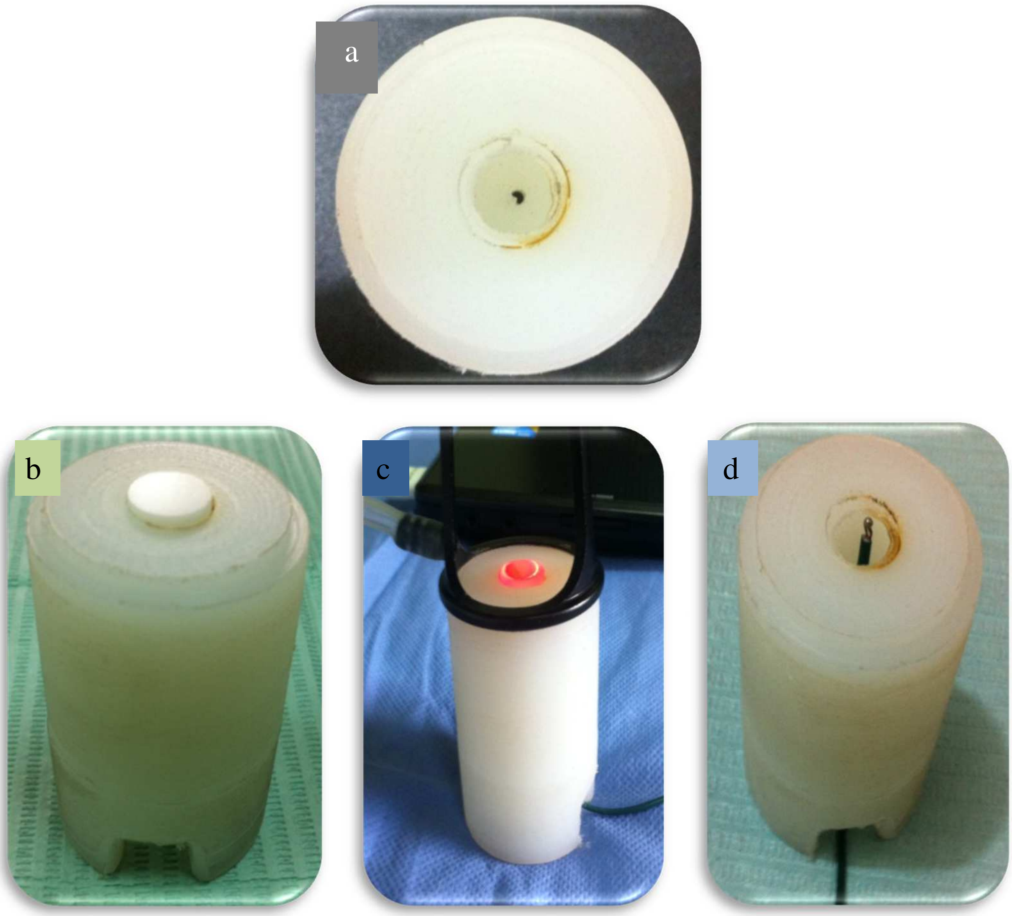
1. 4 specimens as a control group
2. 11 specimens for pilot study.
3. 30 specimens for the study groups

2.5.2 Construction of Teflon mold

A Teflon mold which was constructed by a technician that was used for holding the zirconia disc and at the same time for the fixation of the fractional CO_2 laser apparatus at appropriate distance from the specimen.

The mold was designed as a cylinder with 5 cm in height and 3.5 cm in diameter. In the middle there was a circular opening with 10 mm external diameter and 9mm of the inner diameter to hold the disc in place.

The opening was extended along the entire length of the mold to enable the thermometer to touch the specimen for measuring the temperature during the laser radiation. The external diameter of the mold was reduced about 2 mm in depth and width to fix the laser hand piece during irradiation as shown in Figure (2-10).



Figure(2-10) Teflon mold –a-Top view with a central opening for the thermometer, b- zirconia sample in the middle whole, c- Fixation of laser handpeice during laser irradiation d-Thermometer fixed in position to be in contact with the zirconia sample.

2.5.3. 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 20 mm and 2.5 mm in height. In the middle of the rubber mold there was a circular opening with 5 mm diameter for molding of the resin cement. The opening was surrounded by other circular border that encountered the zirconia disc with 0.5 mm depth in order to fit 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 (2-11)

Figure (2-11) Silicone mold with a central opening for molding the resin cement

2.5.4 Temperature Measuring

The specimen was fixed on the Teflon mold and the experiment was held at an ambient room temperature (27±0.2 ºC) with the K-type thermocouple fixed in contact with other side of the specimen through an opening that was prepared to measure the temperature elevation during lasing. The other side of the thermocouple was connected to thermometer which was connected with to a programmed computer that have software specialized to collect and analyze the thermometer data every 1 sec as illustrated in Figure (2-12)

Figure (2-12) Temperature Measuring during laser irradiation

2.5.5. The experimental work

2.5.5.1. The control group

In this group no surface treatment was performed and each zirconia disc was embedded horizontally in mixed cold cure acrylic to about 1.5 mm and the remaining 0.5 mm of the zirconia disc height being exposed ensuring that the ceramic surface remained intact for the bonding procedure as in Fig.(2-13).

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 adequate amount of the adhesive cement, (Bisco-BisCem-self-adhesive resin cement, USA) shown in Figure (2-14) was automixed (using disposable mixing tips supplied with the cement kit) and dispensed into the opening of the silicon mold.

The excess cement was removed with the explorer tip from the periphery of the mold and the luting agent was then photopolymerized using light curing system that illustrated in Figure(2-15) for 30 seconds following the manufacturer's instruction.

The silicon mold was removed as in Figure(2-16) and finally ,one hour after cementation, specimens were stored in distilled water in a plastic container and placed in the water bath shown in Figure(2-17)at 37°C for 24 hours before SBS testing for completion of resin cement polymerization reaction[136].

Figure (2-13) Acrylic block of zirconia disc

Figure (2-14) Bisco adhesive resin cement

Figure (2-15) Cementation procedure over the zirconia disc

Figure (2-16) LED (Light curing system)

Figure (2-17) Digital Waterbath

2.5.5.2 Shear bond strength test (SBS)

The specimens were attached to a universal testing machine, as in Figure (2-18) and subjected to a shear force using a stainless steel chiseled-shaped rod with across head that illustrated in Figure(2-19), at a crosshead speed of 1mm/min until failure occurred.

The tested specimens were placed in the lower part (jaw) of the testing machine. 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 so that to ensure that the zirconia disc was always at 90 degree to the vertical plane, the specimens were stressed to failure.

The Shear bond test values were calculated from this measurement and expressed in MPa.

Shear strength [MPa] = maximum force [N] / bonding area [mm^2].

The SBS the control group was 5.96 MPa.

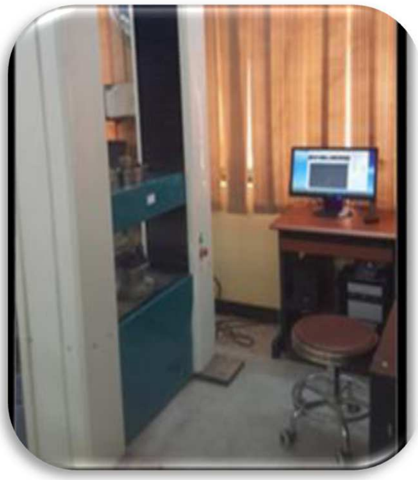


Figure (2-18) Universal testing machine

Figure (2-19) Chisel end rod

2.6 The Pilot study

The bonding surface of each specimen in this group was treated with fractional CO_2 laser. This type of laser has different parameters that can be modified during irradiation in order to find the optimum laser parameters to produce the desired effect on the zirconia surface with the maximum shear bond strength. These parameters included the power setting, distance between spots, the time intervals, pulse duration per spot and the number of scans used as shown in Figure (2-20). That is why the specimens in this group were divided in to subgroups.

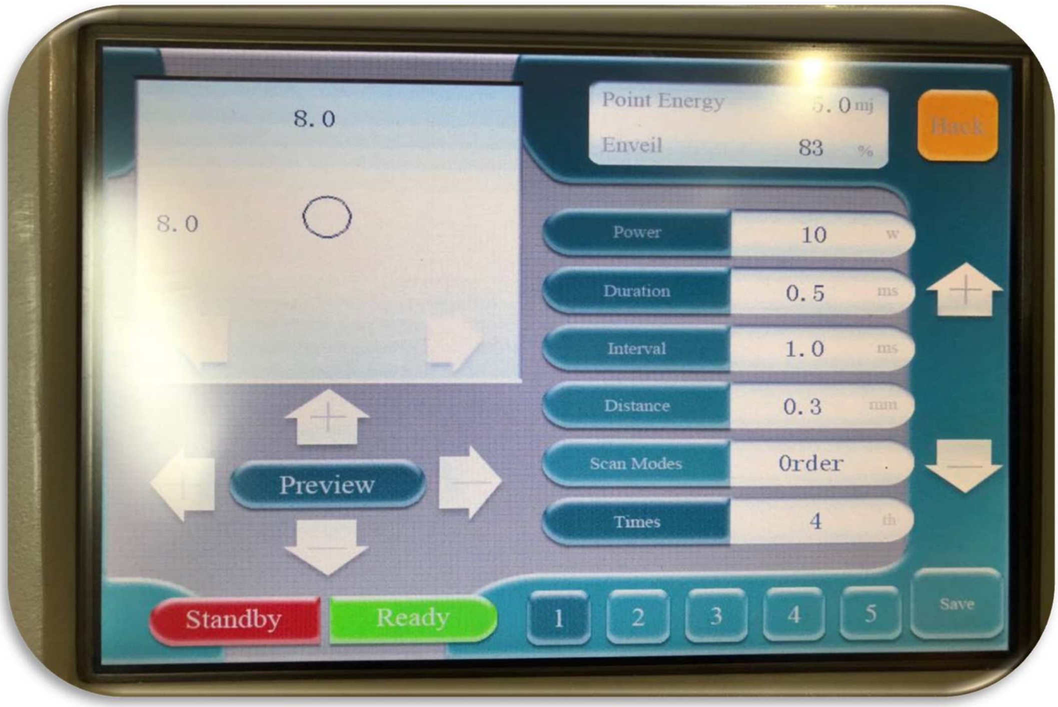


Figure (2-20) Control panel of the laser device

The laser energy was delivered in the order mode and a circular area of 8 mm diameter was irradiated at the middle of the ceramic specimen. The handpiece of the laser was held manually perpendicular and fixed to the Teflon mold at a distance of 5 cm .The groups are randomly assigned as following:

Group A (N=3): in which the specimens were treated with 3 different distances between spots (0.1, 0.3, and 0.6) mm, while the other parameters were fixed as (power: 30 W, pulse duration per spot: 10 ms, scan no.: 4 and intervals: 1 ms). The SBS was shown in Table (2-3).

Table (2-3) SBS of group A

distances between spots(mm)	Shear bond strength(MPa)
0.1	6.11
0.3	**12.42**
0.6	5.9

Group B (N=3): in which the specimens were treated with 3 different time intervals (1, 5, and 10) ms, while the other parameters were fixed as (power: 30W, pulse duration per spot: 10ms, distance: 0.3mm and scan no.:4, distance). The SBS of each specimen was as shown in Table (2-4).

Table(2-4) SBS of group B

Time intervals (ms)	Shear bond strength(MPa)
1	**12.42**
5	12.31
10	12.33

Group C (N=5): in which the specimens were treated with 5 different number of scans (1, 2, 3, 4, 5) while the other parameters were fixed as (power: 30W,distance: 0.3mm and intervals: 1ms and pulse duration per spot: 10ms). The SBS in each specimen was recorded as shown in Table (2-5).

Table (2-5) SBS of group C.

number of scans	Shear bond strength(MPa)
1	7.82
2	8.91
3	10.63
4	**12.42**
5	11.71

After completing the laser irradiation of each group, all the specimens were examined with light microscope with 100X magnification power as shown in Fig (2-21)and Fig (2-22).

An acrylic mold was constructed for each zirconia disc and the total procedure was performed for each sample the same as that was done for the control group as shown in Fig (2-23).

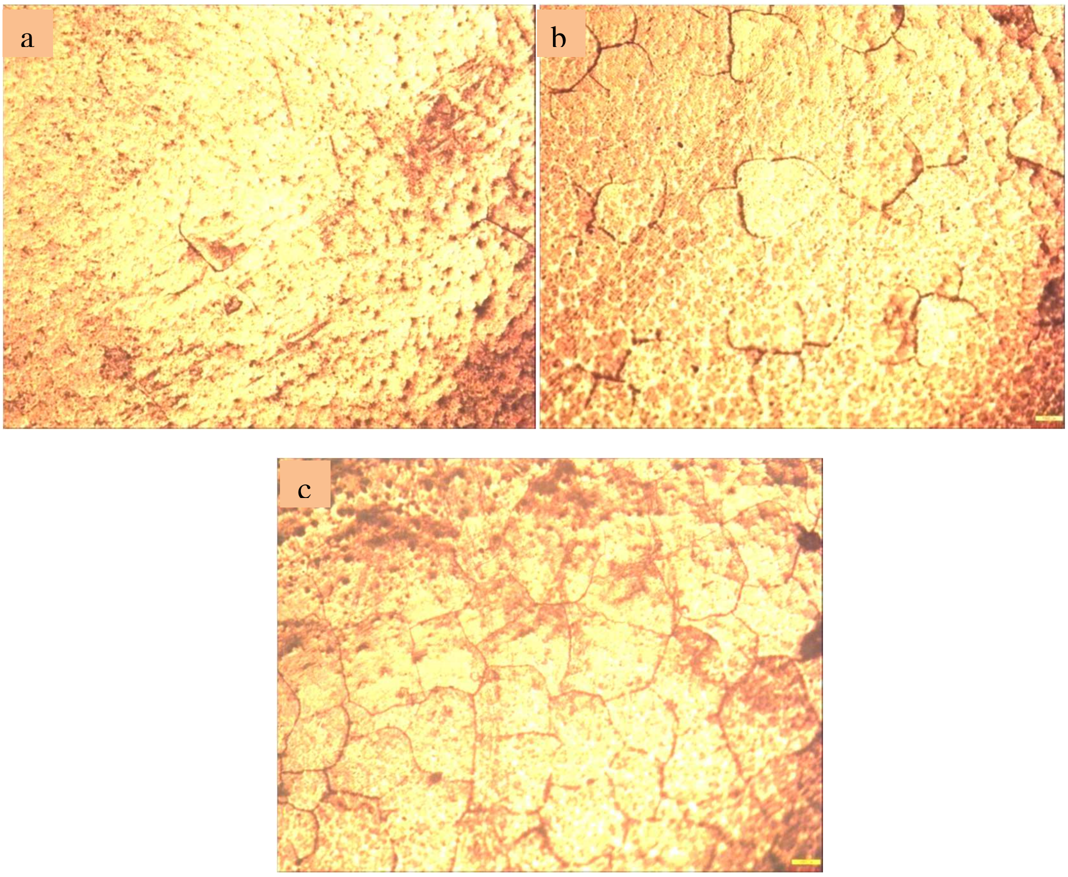


Figure (2-21). Microscopical view (100X) of specimens treated with fractional CO_2 laser 30 W /10 ms with different spots distances.
a. 0.6 mm spots distance. b. 0.3 mm. c. 0.1 mm.

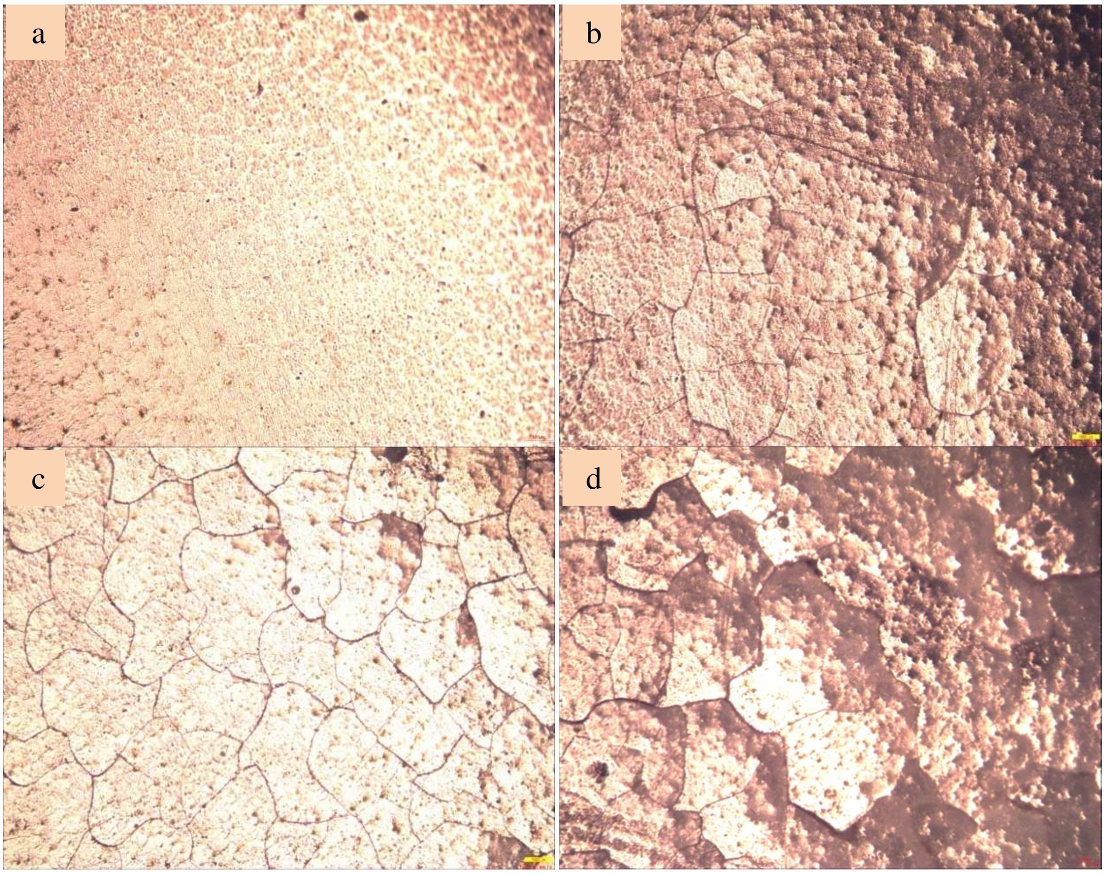


Figure (2-22). Microscopical view (100X) of specimens treated with fractional CO_2 laser 30 W /10 ms with different scan number. a. untreated specimen. b. 3 scans. c. 4 scans. d. 5 scans

The SBS test was measured for each group in order to determine the most appropriate parameters to be fixed in the study group.

The pilot study shows that the most suitable laser parameters were (distance 0.3mm, time interval 1ms and scan no.4) which showed higher SBS in all the groups as 12.42 Mpa.

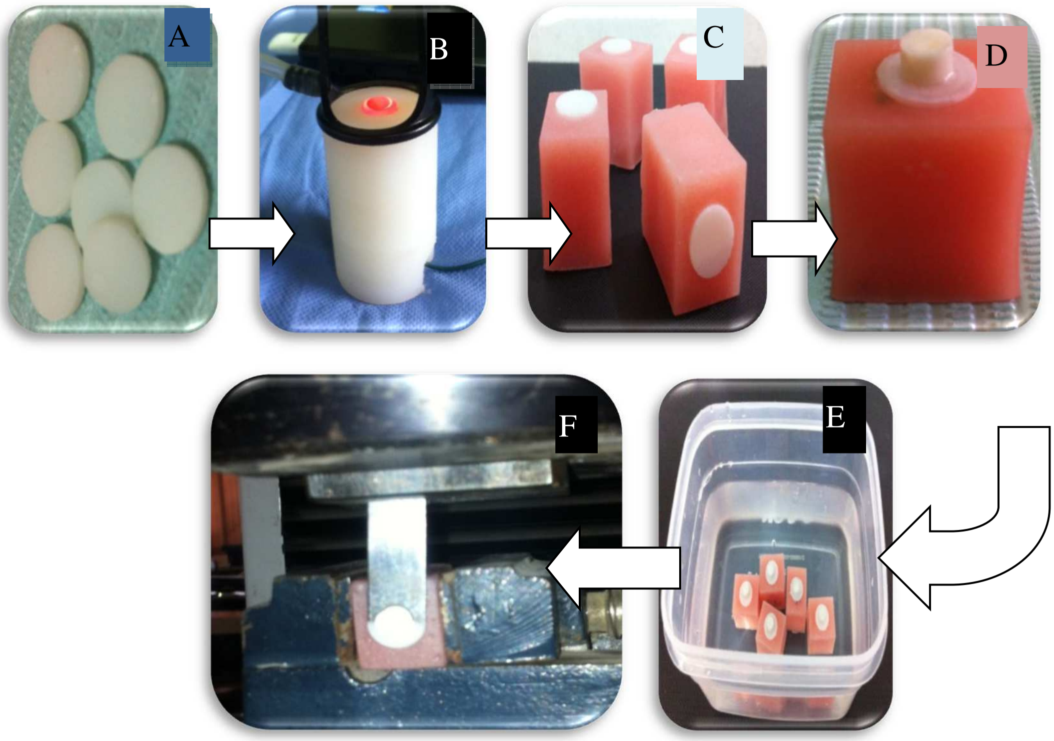


Figure (2-23) Experimental steps. A. specimens' preparation. B.laser irradiation.C. acrylic mold construction. D.cement molding. E.water bath storage. F. SBS test.

2.7 The Study Group

The bonding surface of each specimen in this group was treated with fractional CO_2 laser with the same procedure that was performed in the pilot study after determining the most appropriate laser parameters to be fixed in this group as(distance: 0.3mm,scan no.: 4 and intervals: 1ms).

The specimens were randomly assigned in to 3 subgroups each of 10 with 3 different powers setting (10, 20, and 30 W) as following:

GROUP (A): The specimens in this group were treated with 5 different pulse durations (0.1, 0.5, 1, 5, 10 ms), each 2 specimens with the same pulse duration per spot and a power setting of 10W for all the specimens.

GROUP (B): The specimens in this group were treated with 5 different pulse durations per spot (0.1, 0.5, 1, 5, 10) ms, each 2 specimens were treated with the same pulse duration per spot and a power setting of 20 W for all the specimens.

GROUP (C): The specimens in this group were treated with 5 different pulse durations per spot (0.1, 0.5, 1, 5, 10 ms), each 2 specimens with the same pulse duration per spot, and a power setting of 30 W for all the specimens.

An acrylic mold was constructed for each zirconia disc in each group and the total procedure was performed for each sample the same as that was done for the control group. The SBS test was measured for all the groups.

The fractured specimens were examined after debonding under a stereomicroscope at 40X magnification to determine the type of bond failure. The failure modes were classified as**: [137]**

1. Adhesive failure: was considered when it occurred at the ceramic/resin cement interface.

2. Cohesive failure: when it occurred in the resin cement or ceramic, with no damage to the interface.

3. Mixed failure: was defined as involving both the interface and the material

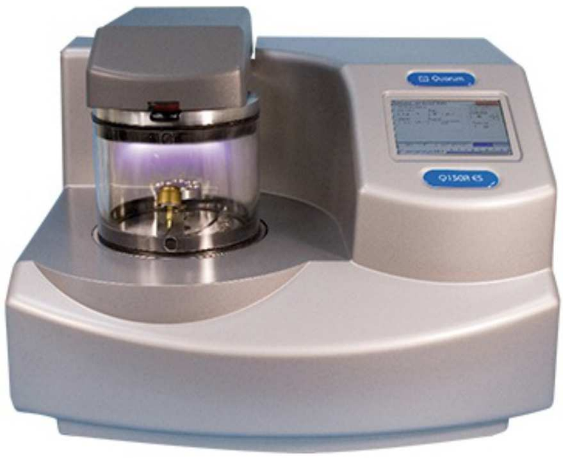
2.7.1 Evaluation of surface roughness

Surface roughness was assessed by Atomic Force Microscope (AFM), as shown in Fig (2-24). The diamond tip of the device with a scanning rate of 3 Hz was passed through the surface of the samples in a contact mode. The average surface roughness (Ra) was determined for one untreated specimen and other specimens those were determined according to the microscopical examination and the results that were provided by SBS test from the experimental group.

Figure (2-24) Atomic Force Microscope

2.7.2 Scanning Electron Microscope (SEM)

To evaluate the surface morphology, the same samples used for AFM were coated with gold-palladium by Rotary-Pumped Sputter Coater, illustrated in Fig.(2-25) and observed under SEM shown in Fig.(2-26) with 500X and 15 kV voltage.



Figure(2-25) Pumped Sputter Coater

Figure (2-26) The Scanning Electron Microscope system.

2.8. Statistical Analysis

Statistical analysis was performed with SPSS software version 23/France. Statistical methods were used in order to analyze and assess the result which includes:

A- Descriptive statistics:

1- Statistical tables including:

- Mean value.
- Standard deviation “SD”

2- Graphical presentation by diagram.

B-Inferential statistics:

- ❖ One-way ANOVA (analysis of variance) and lest significant difference (LSD) tests were carried out to see if there were any significant differences among the means of groups.

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

- Not-significant at $P > 0.05$.
- Significant at $P \leq 0.05$.
- Highly significant at $P \leq 0.01$.

Chapter Three

Results, Discussion and Conclusions

Chapter Three: Results, Discussion and Conclusions

3.1 Introduction

Chapter three is concerned with the results of this research work and discussion of these results. These results were obtained after irradiating of zirconia ceramic specimens with different power settings and pulse durations of fractional CO_2 laser.

After analyzing these results, conclusions were drawn based on evaluation of the laser effects on shear bond strength of zirconia ceramic to resin cement, the bond failure mode, temperature elevation measurements, and the surface texture and roughness properties. Also the future suggested work will be mentioned at the end of this chapter.

3.2 Shear bond strength test

Table (3-1) shows the mean and standard deviation values for SBS of the three groups. One way ANOVA test indicated that there was a significant difference in SBS mean values of different groups especially those treated with 0.1 ms pulse duration ($P = 0.001$),followed by 0.5 and 1ms pulse duration($P = 0.01$)and less significant differences were shown with 5 and 10ms pulse duration.

Also LSD test indicated that group B had significantly higher SBS mean values with the same pulse duration as expressed in (a) followed by group C as expressed in (b) and group A as expressed in (c),also the LSD test for the same group with different pulse duration indicated that higher significant difference($P = 0.01$)was shown with 0.1ms pulse duration for group B and C as expressed in symbol(*)and lest significant differences were shown with 10 ms pulse duration as expressed in symbol(***),whereas no significant difference was shown for SBS mean value for specimens treated in group A for different pulse durations.

Table (3-1) Shear bond strength measurements for different powers of fractional CO_2 laser

Pulse duration (ms)	SBS(Mpa)			
	Group A (10 W) Mean±SD	Group B (20 W) Mean±SD	Group C (30 W) Mean±SD	P-value
0.1	7.85±0.2 c	*15.5±0.3 a	*12.7±0.1 b	0.001
0.5	7.6±0.1 c	**12.65±0.1 a	**11.7±0.2 b	0.01
1	7.4±0.1 c	**11.5±0.2 a	**10.75±0.3 b	0.01
5	7.15±0.1 c	**10.52±0.3 a	**9.65±0.1 b	0.05
10	6.75±0.2 c	***9.55±0.2 a	***8.6±0.2 b	0.05
P-value	NS	0.001	0.001	-----

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

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

The SBS mean values of the three groups for each pulse duration per spot are drawn as a function of power as shown in Figure (3-1).

The maximum shear bond strength was obtained with 20 W/0.1 ms laser parameters and the minimum shear bond strength was obtained with 10 W/10 ms laser parameters.

Also it can be clearly observed that increasing the pulse duration during the laser irradiation resulted in decreasing the SBS within the same group.

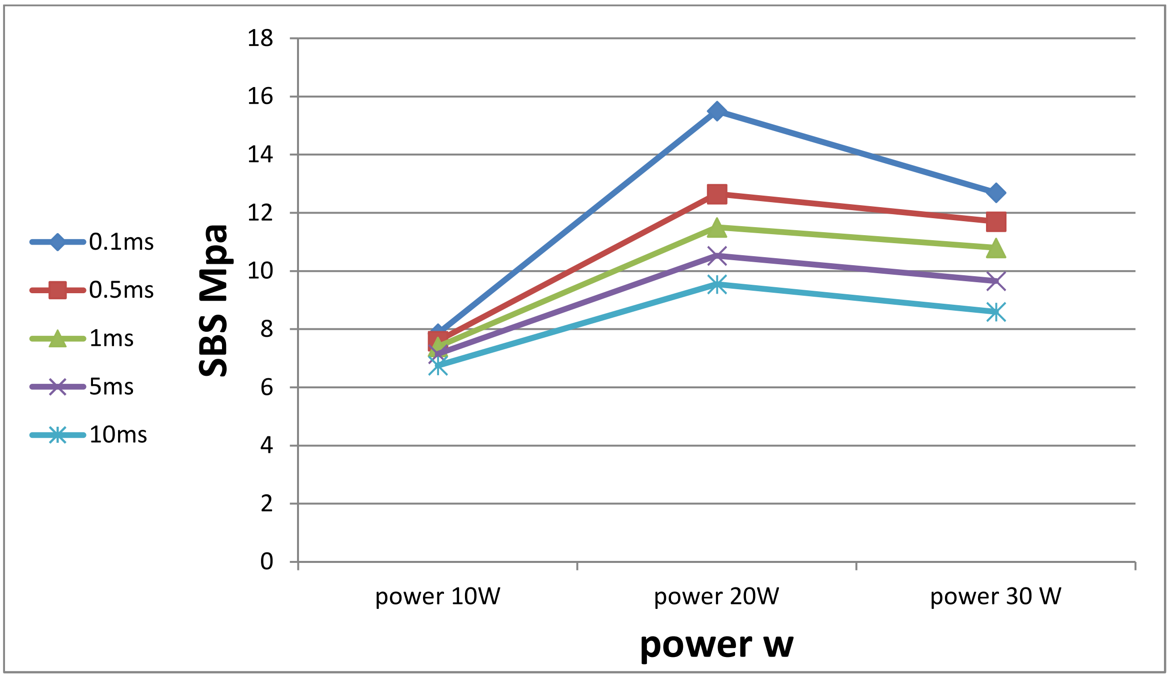


Figure (3-1) Diagram of Shear bond strength mean values of the three groups.

3.3 Temperature Measurements

Table (3-2) shows the mean and standard deviation values for the temperature elevation of the three groups. One way ANOVA test and LSD test indicated that there was a significant difference in the temperature elevation measurements for different pulse duration of the three groups with a P-value≤0.01.

Table (3-2) Temperature elevation measurements at different powers of fractional CO_2 laser

Pulse duration/ ms	Temperature/Ċ		
	Group A Mean±SD	Group B Mean±SD	Group C Mean±SD
0.1	*27.3±0.1 a	*28.6±0.3 a	*30.1±0.2 b
0.5	**29.6±0.2 a	**30.3±0.2 a	**35.2±0.4 b
1	**30.1±0.2 a	**32.4±0.2 a	**41.1±0.3 b
5	***37.2±0.2 a	***78.1±0.1 b	***90.2±0.2 c
10	****70.2±0.2 a	***89.6±0.2 b	***99.2±0.1 c
P-value	0.01	0.01	0.01

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

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

The temperature elevation measurements of the three groups for each pulse duration are expressed as a function of power as shown in Figure (3-2). The lowest temperature elevation measurements were observed in groups A (10 W) and B(20 W) with 0.1 ms pulse duration whereas the highest measurement was observed in group C(30 W) with 10 ms pulse duration. The temperature elevation measurement was increased with increasing the power setting of the groups.

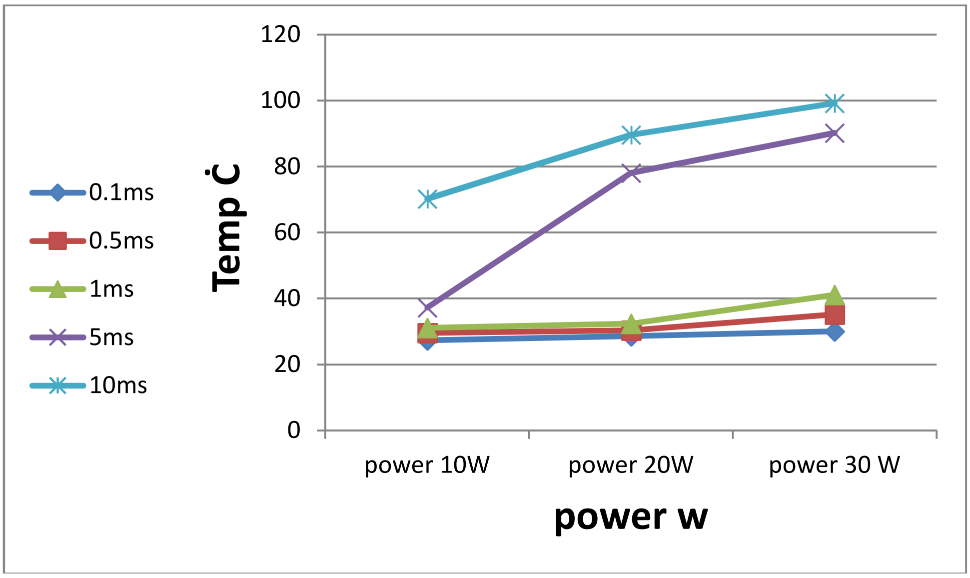


Figure (3-2) Diagram of the temperature elevation measurements' means of the three groups.

Also it can be noticed from these figures that the temperature elevation measurements were at the lowest values with (0.1, 0.5 and 1 ms) pulse durations for different groups and these measurements recording their highest values with (5 and 10 ms) pulse durations for different groups.

3.4 Failure Mode

Frequency of failure mode after shear bond strength test of each group was shown in Table (3-3). The results indicated that the failure mode of the groups with different parameters was varied.

In group A type 1 failure mode was frequently observed. On the contrary, failure mode of type 2 or type 3 was detected in group B, However type 1 failure mode was found on those treated with (20 W for10 ms). In group C failure mode types 1 was observed except for (0.1 ms and 0.5 ms) which show failure mode of type 2.

Table (3-3) Failure modes distribution.

Group	Pulse Duration per spot ms	Adhesive(1)		Cohesive(2)		Mixed(3)	
		No.	Percentage	No.	Percentage	No.	Percentage
A	0.1	1	10	1	10		
	0.5	1	10	1	10		
	1	2	20				
	5	2	20				
	10	2	20				
B	0.1					2	20
	0.5			2	20	1	10
	1			1	10	1	10
	5			1	10		
	10	2	20				
C	0.1			2	20		
	0.5			2	20		
	1	2	20				
	5	2	20				
	10	2	20				

3.5 The working time

The mean and standard deviation values of the working time for each pulse duration per spot of the groups was shown in Table (3-4).

One way ANOVA test indicated that there was no significant difference in the working time for the same pulse duration of different groups, whereas a high significant difference (p=0.001)was found for different pulse duration per spot of the same group and the lest working time was shown with 0.1ms pulse duration as observed with LSD test that represented with(*).

Table (3-4) Working time of the study groups.

Pulse duration (ms)	Working time/sec		
	Group A Mean±SD	Group B Mean±SD	Group C Mean±SD
0.1	*5±0.1	5±0.1	5±0.1
0.5	**6±0.03	6±0.03	6±0.03
1	***8±0.05	8±0.05	8±0.05
5	****15±0.1	15±0.1	15±0.1
10	*****26±0.2	26±0.2	26±0.2
P-value	0.001	0.001	0.001

The working time is plotted as a function of pulse duration of the study groups are shown in Figure (3-3).

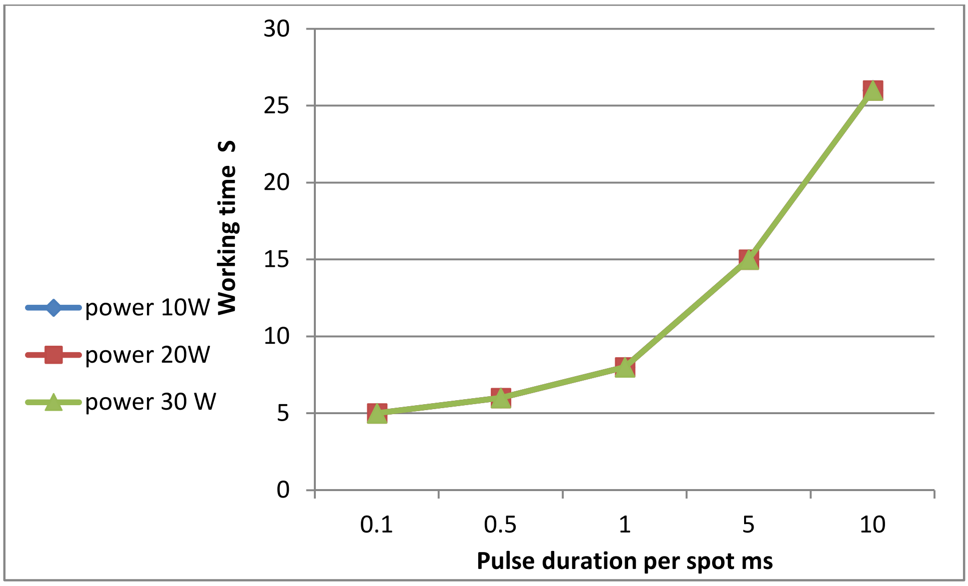


Figure (3-3) Diagram of the working times of study groups

It can be observed that the working time increased exponentially with increasing the pulse duration reaching to the maximum working time with 10 ms pulse duration.

3.6 Surface roughness analysis

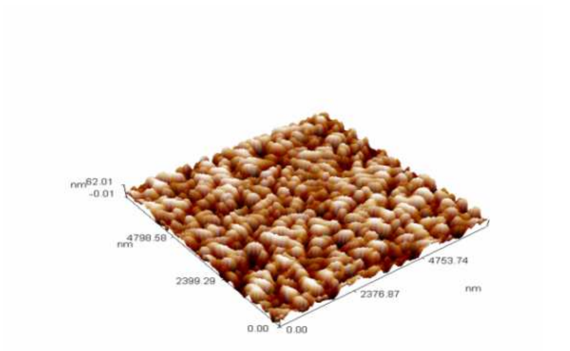
Table (3-5) shows surface roughness measurements of the different surface treatment groups.

Also Figures (3-4) a–d show the three-dimensional (3D) roughness measurements of the different surface treatment groups.

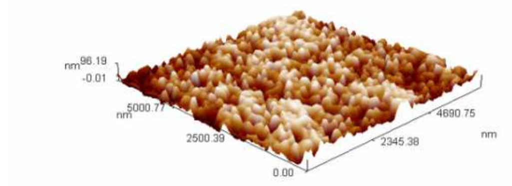
Ra was increased by laser irradiation with higher output power or by increasing the pulse duration per spot, as the higher Ra value was observed in specimens treated with (30 W for10 ms) than the other surface treatment groups.

Table(3-5) Surface roughness measurements

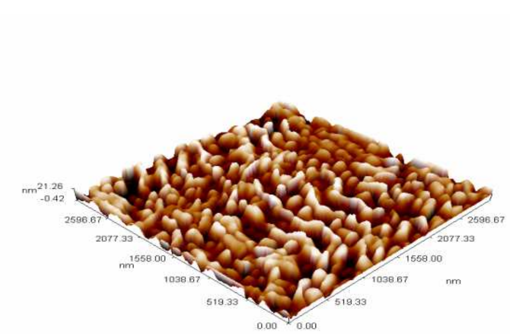
Fractional CO_2 laser parameters	**Ra(nm)**
Control(no treatment)	**5.42**
20 W/0.1 ms	**15.5**
20 W/10 ms	**21.4**
30 W/10 ms	**38.2**



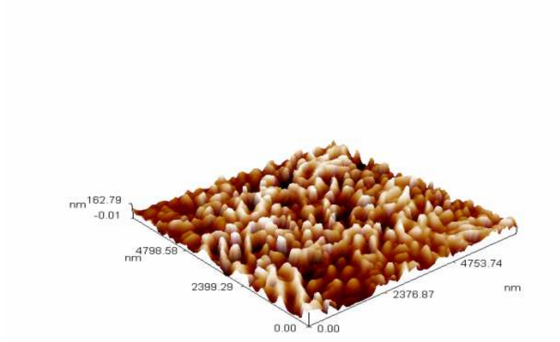
a



.b



c



d

Figure (3-4). (3-Dimentional AFM pictures). a. untreated specimen(Ra=5.42 nm);. b. Specimen treated with fractional CO_2 (20W/0.1ms) Ra=15.5 nm. c. (20 W/10 ms) Ra=21.4 nm. d. (30W/10 ms) Ra=38.2 nm**.**

The lowest Ra value was observed with the control specimen followed by that treated with fractional CO_2 of 20 W/0.1 ms laser parameter and the highest Ra value was shown with 30 W/10 ms laser parameter.

3.7 Scanning electron microscope analysis

SEM view of zirconia disc surface is shown in Fig. (3-5) a-e. A surface texture consisting of micromechanical irregularities with shallow dark pits and erosions were shown in a specimen treated with fractional CO_2 laser (20 W/ 0.1 ms) as shown in Fig. (3-5b). However irregular micro and macrocracks are seen all over the ceramic surface in those specimens treated with (20 W/0.5 ms) as shown in Figure (3-5c) and these cracks are connected to each other like a network as shown in specimens treated with (20 W/10 ms) and (30 W/10 ms) as in Fig. (3-5d)and (3-5e). Large flaws could be detected easily on the ceramic surface in specimen with 30 W.

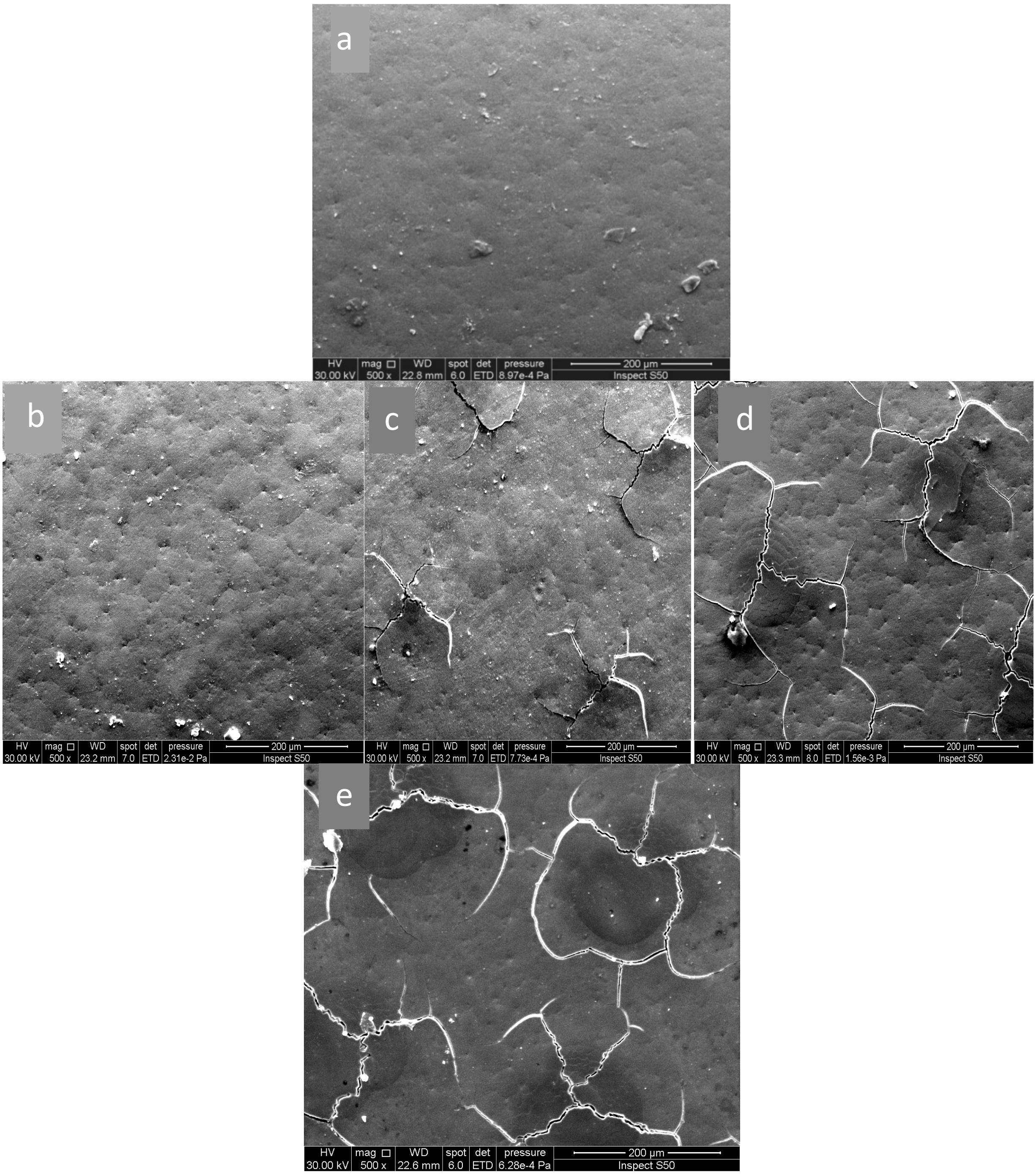


Figure (3-5). SEM pictures of zirconia specimens (500X). a. untreated specimen. b. Specimen treated with fractional CO_2 (20 W/0.1 ms). c.(20 W/0.5 ms). d. (20 W/10ms).e.(30 W/10 ms)

3.8 Discussion

Surface treatment of zirconium oxide ceramic by laser irradiation has been widely used nowadays as alternative methods. The critical factors that affecting the bond between the resin cement and treated zirconia ceramic surface are the type of laser and the laser parameters used.

The principal effect of laser energy is the conversion of light energy into heat (the thermo-mechanical effect), and the most important interaction between the laser and substrate is the absorption of the laser energy by the substrate [138] .Therefore, selection of appropriate laser parameters to change the surface properties of the zirconia surface is of the utmost importance. Ural et al. concluded that the output power of the laser and the energy level are critical factors affecting the bond strength of resin cement to zirconia ceramic [127].

There are different kinds of lasers and zirconia ceramics, so choosing the ones which will be good match for each other is crucial. Besides that, changing the irradiation setting can be beneficial as well.

CO_2 lasers have been shown to be well suited for the surface treatment of ceramic materials because these materials have a high potential of absorbing CO_2 laser wavelength [19, 22, 127]. Due to the process of heat induction of ceramic surfaces with a focused CO_2 laser, shell shaped tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [14].

Recently, few studies investigated the efficacy of conventional CO_2 laser for enhancing adhesion of bonding cement to oxide ceramics [132-136] whereas studies employed a fractional type of CO_2 laser for zirconia surface treatment are very limited.

The use of fractional CO_2 laser provides several advantages including predetermining the exact irradiation area by the laser controlling panel with a more homogenous etching pattern can be achieved. Also the need for manual movement of the laser handpiece is restricted during conditioning. Furthermore, thermal damage to the underlying area will be reduced compared to that occurs with conventional CO_2 laser [130].

In the current study, the effects of different power setting of fractional CO_2 laser with different pulse durations on the SBS of resin cements to zirconium oxide surface were evaluated and compared, while the spots distance, time interval and scan number used were constant (from a pilot study).

Shear stresses are believed to be major stresses leading to bonding failure of restorative materials, and the shear bond strength tests are very simple and quick tests and are considered the most appropriate tests for evaluating the bonding effectiveness of resin agents to ceramics [131]. In this study, shear bond strength test was used to investigate the bonding property of zirconia ceramics to (BISCO-USA) dual cured resin cement.

Resin cements are a major part of today's clinical practice as they provide enhanced adhesion to all-ceramic restorations because of their high compressive and tensile strengths, low solubility with a favorable aesthetic qualities. Their major disadvantages include difficult removal of excess cement, technique sensitivity, time-consuming process, and expensiveness [139, 140].

Bisco-BisCem adhesive resin cement provides good bond strengths to tooth structure without any pretreatment or bonding agents. Also its application is very simple and can be accomplished in a single clinical step.

3.8.1. Shear bond strength and failure mode

The SBS of the specimens treated with the fractional CO_2 laser (20 W/0.1 ms) was significantly greater than those of other groups for the same or for different pulse duration as (15.5±0.3 Mpa). This result could be attributed to the efficacy of fractional CO_2 laser in roughening the bonding surface through the process of thermal conduction of concentrated laser energy within short pulse duration which result in micromechanical irregularities that increases mechanical retention without any damaging effect to the zirconia surface, thereby enhancing the bond strength of zirconia ceramic to resin cement. Whereas specimens treated with (10 W/10 ms) exhibited the lowest SBS among the study groups (6.75±0.2 Mpa). This finding could be attributed to the low power used within along pulse duration (the laser energy is dissipated during long pulse duration) that was unable to produce any significant effect on the zirconia surface as observed in the microscopical examination.

These results are in difference from other results obtained from Ahrari F. et al. [130] who used fractional CO_2 laser at settings of (10 W/1.75 ms) and (20 W/0.58 ms) as a surface treatment method for high-purity zirconium oxide ceramic (Dental Direkt GmbH, Spenge, Germany). This could be attributed to the differences in the laser parameters those were fixed in the pilot study or due to the differences in the types of zirconia system or the resin cement material that had been used.

The quality of the bond in this study was also assisted by the bond failure mode analyses which provide important information of bonding effectiveness. Cohesive fracture and mixed (cohesive failure combines with adhesive failure) patterns are clinically preferable to total adhesive type of failure, since the last one is usually associated with low bond strength values [141].

The bond failure of group A and C was mainly of the adhesive type indicating that the micromechanical interlocking of this group was not strong enough to produce higher bond strength except for those specimens treated with 0.1 ms and 0.5 ms pulse durations of group C which showed cohesive failure mode. In group B, cohesive and mixed failure modes in resin cement were the most predominant.

This means that the bond strength between cement and ceramics was higher than the cohesion strength of the cement material. These results are in different with other results which were obtained from Ahrari et al. [130] who reported a lack of relationship between bond strength and failure mode after treating zirconia surface with fractional CO_2 laser with different parameters, as the failure mode was mostly adhesive with no significant difference among the groups.

3.8.2 Temperature measurement and surface analysis

Since the absorption of laser energy by material's surface is the most important interaction between material's surface and laser, it is crucial to choose suitable laser parameters, in order to have a change in the zirconia ceramic's surface and suitable surface treatment [128].

An increasing in the power setting or increasing pulse duration results in more thermal effects on the zirconia surface as observed in temperature elevation after irradiation with different laser parameters. This thermal effect resulted in more heat dissipation and subsequently cracks formation, confirming that heat confinement is a critical factor in the surface roughness of zirconia ceramic with laser irradiation.

Samples treated with fractional CO_2 laser showed increased surface roughness compared to the untreated zirconia sample. The surface roughness increased with increasing power setting and pulse duration but it does not enhance the bond strength. This may be due to the lack of micromechanical retention with larger surface cracks formation. This result came in a good agreement with that result which obtained from Ersu B. et al. [14] who reported that bond strengths were not dependent on surface roughness created by surface treatment methods.

An increase in output power or pulse duration was supposed to cause higher bond strength of the ceramics, which did not take place in this study. The specimens irradiated with 20 W/0.1 ms fractional CO_2 laser showed significant superficial changes in SEM observations. The temperature elevation due to laser irradiation effectively facilitates localized surface heating and creates micromechanical irregularities on the surface and subsequently increasing the bond strength.

The use of low power as in group A was unable to produce a considerable effect on the zirconia surface as observed in microscopical examination, whereas an increasing of the power setting as in group C showed reduction in SBS. In other words, SBS values increases as the power increases till a certain threshold beyond which macrocracks formation will occur resulted in decreasing SBS. Also increasing the pulse duration and subsequently increasing working time showed a decreasing in SBS.

This can be due to the formation of a damaged layer and macrocracks due to heat dissipation following laser application which may affect the strength of the ceramic structure adversely. This means both of the pulse duration and the power setting have a compromising effect on the SBS of zirconia ceramic to the resin cement.

Different reasons for crack formation on ceramic surface after CO_2 laser irradiation have been proposed. Stubinger et al. [142] reported that the main part of the absorbed laser energy was used during melting and only a small portion was spent for heating the mass of material. This feature causes rapid cooling of the molten layer as soon as the laser irradiation is removed and as a result, cracks are formed.

Some studies had shown that microcracks formation of on the zirconia ceramic surface increases the bond strength of resin cement to zirconia ceramic as these microcracks increased the resin penetration within the ceramic surface structure and subsequently higher SBS. [126,128,132].

Researchers concluded that output power and irradiation time must be carefully considered using high-energy Nd:YAG, Er:YAG and CO_2 lasers to avoid microcracks on zirconia surfaces [132,143].

The present study suggested fractional CO_2 laser as a suitable method for surface treatment of zirconium oxide ceramics, which can provide bond strength values remarkably greater about 20% than that of the conventional CO_2 laser (which showed a maximum SBS of 12.12± 3.02 Mpa) or other laser such as Er:YAG(which showed a maximum SBS of 8.65± 1.77 Mpa) had been used with the same type of zirconia ceramic system [128, 132], and without any significant damage to the substrate.

3.9 Conclusions

1. Irradiation of zirconia ceramic surface with fractional CO_2 laser at 20 W/0.1ms pulse duration was the most effective parameters for enhancing bond strength of the zirconia ceramic (Ceramill zolid/Amann Girrbach/Austria) to resin cement(Bisco-BisCem-self-adhesive resin cement, USA), and could be recommended as a suitable alternative to conventional methods of zirconia surface treatment.
2. Extending irradiation time and increasing output power cannot induce higher bond strength of dental zirconia ceramics and might cause material defect.
3. Surface roughness created by surface treatment with different laser parameters is not the only factor that increases the SBS of zirconia ceramic to resin cement.
4. Temperature elevation is a vital factor and can produce a significant damage to the zirconia ceramic surface with increasing pulse duration or power setting.

3.10 Suggestions for Future Studies

1. Conclusions drawn from one zirconia ceramic system may not be applicable to other commercial systems. Future studies are necessary to evaluate the effect of fractional CO_2 laser irradiation on the surface properties and bond strength of other zirconia ceramic types.
2. Further long-term studies are suggested to determine SBS values of laser treated zirconia after long term water storage or thermocycling to match the clinical situation.
3. Future study to evaluate the combined effect of fractional CO_2 laser and MDP containing primer on SBS of zirconia copings is also suggested.

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الخلاصه

مقدمه: هناك العديد من الدراسات المتعلقة بتعزيزالتصاق زركونيا السيراميك إلى الاسمنت الراتنج باستخدام الطرق التقليدية و الليزرات المختلفة. الهدف من الدراسة الحالية هو تعزيز قوه التصاق زركونيا السيراميك إلى الاسمنت الراتنج والتحقيق في تأثير ارتفاع درجة الحرارة زركونيا السيراميك باستخدام ليزرثاني اوكسيد الكاربون المجزئ كطريقة معالجة سطحية .

المواد وطريقه العمل: تم تحضير ٤٤ قرص من أوكسيد الزركونيوم (10mm في القطر، 2mm في السمك).تم استخدام ثلاث عينات لتحديد قيم السيطره وبالنسبة للدراسة التجريبية، استخدمت ١٤ قرصا زركونيا لتحديد المسافة والفترة الزمنية وعدد مرات المسح. وفيما يتعلق بمجموعة الدراسة، تم تقسيم ٣٠ قرص زركونيا إلى ثلاث مجموعات (n=10)، واستخدمت خمس فترات نبضات مختلفة في كل مجموعة (5,1,0.5,0.1 و10 ms)كل نموذجين بنفس مدة النبضة. تم معالجة المجموعة أ مع ليزر ثاني اوكسيد الكاربون المجزئ بقوة10 W ، المجموعة ب 20 W ومجموعة ج 30 W و خلال أشعة الليزر، تم تسجيل قياس ارتفاع درجة الحرارة لكل عينة وبعدها تم ربط الاسمنت بأسطح الزركونيا المشععة وتم معالجتها لمدة 30 sec. ثم تم تقييم قوة الالتصاق باستخدام آلة اختبار عالمية.

النتائج: إن الحد الأدنى من قياس ارتفاع درجة الحرارة للعينة المشععة الذي يعطي أقصى قدر من قوة الارتباط (15.5± 0.3 Mpa)هو حوالي 1.6 Ċ أعلى من درجة حرارة الغرفة المحيطة مع 20 W/0.1 ms .وقد لوحظ التقشير الميكانيكي في العينات المعالجة وتشكيل شقوق مع زيادة وقت التشعيع او زيادة توليد الطاقة اثناء الفحص التصويري للعينه مما يؤدي إلى خشونة أعلى للسطح.

الاستنتاج: ليزر ثاني أوكسيد الكربون المجزئ هو طريقة بديلة فعالة لتعزيز قوة التصاق زركونيا السيراميك إلى الاسمنت الراتنج ، مع الأخذ بعين الاعتبار ارتفاع درجة الحرارة كعامل حيوي في خشونة سطح زركونيا السيراميك اثناء التشعيع بليزر ثاني أكسيد الكربون المجزئ.

وزارة التعليم العالي والبحث العلمي

جامعة بغداد

معهد الليزر للدراسات العليا

تأثير ليزر ثنائي اوكسيد الكربون المجزّئ على خشونه السطح وإرتباط زركونيوم السيراميك مع الأسمنت الراتنج

رسالة مقدمة الى

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بكالوريوس طب وجراحه الفم والاسنان - ٢٠٠٣

بإشراف

الأستاذ المساعد الدكتورحسين علي جواد

١٤٣٩ هـ **٢٠١٧م**