LASER ETCHING OF ENAMEL FOR BONDING ORTHODONTIC BRACKETS

by

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LASER ETCHING OF ENAMEL FOR BONDING ORTHODONTIC BRACKETS

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ABSTRACT

LASER ETCHING OF ENAMEL FOR BONDING ORTHODONTIC BRACKETS

The aim of this study is to determine the optimum laser parameters to provide the maximum bonding strength with reference to conventional bonding technique for orthodontic braces. 1940 nm Thulium Fiber Laser was used in pulse mode and in two different parameters for enamel etching procedure. In addition, there is a control group which had conventional acid etching technique. Laser etching procedure was carried out by two different approaches: Lasing without cooling and with cooling. For the first approach, according to the whitening of the enamel initiation, application time of laser irradiation was defined for each laser parameters. For the second approach, in order to prevent damage on alive tissue, water spray and air pressure cooling was applied during lasing to limit pulp temperature to maximum 5.5 °C which is the critical temperature. After the bonding of ceramic braces, they were debonded from the enamel surface using mechanical test machine. When the bonding strength of the braces were analyzed, it was observed that the mean bonding strength of both the etching without cooling group and etching with cooling group are significantly different from acid etched group ($p<0.05$, student t-test). SEM evaluations of etched surface with laser showed that surface morphologies were not similar with acid etching group.

Keywords: Laser, Etching, Bracket.

ÖZET

ORTODONDİK BRAKETLERİN YAPIŞTILMASI İÇİN MİNE YÜZEYİNİN LASERLE PÜRÜZLENDİRİLMESİ

Bu çalışmanın amacı geleneksel asitle pürüzlendirme tekniği referans alınarak, ortodontik braketler için maksimum yapışma gücünün sağlanabilmesi için uygun laser parametrelerinin belirlenmesidir. Mine pürüzlendirme işlemi için 1940- nm Thulyum Fiber Laser, 2 farklı dozda, darbeli moda uygulanmıştır. Bunun yanında geleneksel yöntem ile pürüzlendirdiğimiz grubumuzda vardır. Laserle pürüzlendirme iki farklı yaklaşımla gerçekleştirilmiştir: Soğutmasız laser kullanımı ve soğutmalı laser kullanımı.İlk yaklaşımda, her bir laser parametresi için minedeki beyazlama başlangıcına göre uygulama süresi belirlenmiştir. İkinci yaklaşımda, laser kullanımı sırasında, canlı dokuya zarar vermeyi önlemek amacıyla su spreyli soğutma ve basınçlı havayla soğutma yapılarak pulpa sıcaklığı kritik sıcaklık olan 5.5 °C de sınırlandırılmıştır. Seramik braketler yapıştırıldıktan sonra, mekanik test makinesi ile mine yüzeyinden koparılmıştır. Braketlerin kopma kuvvetleri incelendiği zaman, soğutmalı ve soğutmasız olarak pürüzlendirilen grupların kopma kuvvetleri asitle pürüzlendirilen grubunkinden anlamlı olarak farklı olduğu gözlemlenmiştir ($p<0.05$, student t-test). SEM değerlendirmeleri laserle pürüzlendirilen yüzeylerin yüzey morfolojilerinin asitle pür üzlendirilen grubunkiyle benzer olmadığını göstermiştir.

Anahtar Sözcükler: Lazer , Pürüzlendirme , Braket.

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LIST OF SYMBOLS

Δ	Difference of energy levels
h	Constant of Planck
ν	Frequency of radiated light
c	Speed of light
λ	Wavelength of radiated light

LIST OF ABBREVIATIONS

LASER	Light Amplification by the Stimulated Emission of Radiation
nm	nanometer
°C	Celcius Degree
CW	Continuous Wave
CO_2	Carbon dioxide
ER:YAG	Erbium-doped Yttrium Aluminium Garnet
Er,Cr:YSGG	Erbium, Chromium- doped Yttrium Scandium Gallium Garnet
Nd:YAG	Neodymium-doped Yttrium Aluminium Garnet
μm	micrometer
Bis-GMA	Bisphenol-a and methylmethacrylate
4-META MMA	4-methacryloxyethyl trimellitate anhydride
mm	millimeter
NIR	Near Infra-red
N	Newton
W	Watt
sec	second(s)
mm^2	millimetersquare
MPa	Megapascal
cm	centimeter
s	second(s)
J	Joules
SEM	Scanning Electron Microscope
US	United States

1. INTRODUCTION

Straight teeth do not only provide individuals to bite chew and speak effectively, they are also important cosmetically. Orthodontic treatments have been used to correct the impairments to provide healthy and straight teeth [1]. The most common orthodontic treatment for the tooth irregularity is the dental braces. [1, 2]

For the dental treatment with braces, generally metal or plastic wires are inserted into orthodontic brackets, which can be made from stainless steel, ceramic material or plastic. Ceramic brackets are widely used because of their aesthetic appearance, higher strength, resistance to deformation, and better color stability. [1, 2, 3, 4]

The first step for the orthodontic treatment with brackets is to etch the enamel surfaces to provide mechanical retention of composite resin. To bond the ceramic brackets, more mechanical retention is necessary than other type of brackets. To provide more mechanical retention, extra acid etching and extra adhesive are required. This conditions cause more risks of enamel damage or decay. After the bracket bonding, wires interact with the brackets and the force applied by the wire to move the teeth to the desired direction [1].Dental braces are removed at the end of the treatment.

Laser application has been proposed as an alternative method to acid etching. [2]. In the literature, different types of laser (CO_2, Er: YAG, Nd: YAG, and Er,Cr: YSGG) have been used in orthodontics for enamel conditioning to bond brackets[3-7]. Some laser types provide local melting and ablation on dental hard tissue. In that manner, laser etched enamel may has the similar topology of acid etched. The depth of the surface roughness depends on the power and the wave length of the laser [8]. However, beside its many advantages, the thermal effect during the laser radiation can cause undesirable results for the dental tissues.

In the majority of the studies, safety threshold of intra pulpal temperature increase has been chosen as 5.5 °C for intra pulpal described by Zach and Cohen as a critical rise in order to prevent the thermal damage for the nerves in the pulp [9-12].

1.1 MOTIVATION AND OBJECTIVE

In clinical conditions, enamel surfaces have been etched chemically by acid solution to provide surface roughness for bonding brackets. Although acid etching is a common method, new alternative methods have being investigated since the acid application causes some disadvantages like demineralization and contamination of enamel surface in long term. Motivation of this study is to etch enamel with laser to decrease the risk of tooth decay caused by acid etching technique.

The aim of this study was to determine a laser-etching protocol with minimum thermal effect to pulp and with maximum bonding strength with reference to conventional acid-etching technique to bond the orthodontic brackets. 1940 nm Thulium Fiber Laser was used for the laser etching procedure. During this procedure, to prevent the pulpal injury, temperature increase was controlled and limited to maximum $5,5\,^{\circ}C$ which is the critical temperature value for the pulp. After bonding, ceramic brackets were debonded from the enamel surface to measure the bond strength. Debonding procedure was quantified with a universal testing machine. Breaking load was recorded during this procedure. Results were compared with acid-etching samples.

1.2 OUTLINE

In Chapter 2, orthodontics and orthodontics treatments, tooth structure, basic laser principles, laser tissue interaction and some laser etching studies in literature are explained.

In Chapter3, detailed information about materials and procedure used in this study are mentioned.

In Chapter 4, results about measurements of debonding forces from teeth samples which were etched in different laser parameters and SEM images of enamel surface specimens are explained.

In Chapter 5, results of the study are discussed and compared with other studies.

In Chapter 6, the conclusion of the study and future studies are explained.

2. BACKGROUND

2.1 ORTHODONTICS

Orthodontics consists of two Greek words “orthos” and “dontos”. Orthos means normal or straight and dontos means teeth. Orthodontics is a branch of dentistry concerned with prevention, interception and correction of malocclusion and other abnormalities of the dental region.

The teeth play an important role in speech and a role in food processing. Foods are masticated, or chewed, by opening and closing our jaws and moving them from side to side while continually using tongue to move the food between the teeth. In the process, the teeth tear and grind the food, breaking it down into smaller fragments.

Normal adults have 32 teeth, which are arranged in two dental arches called the maxillary arch and the mandibular arch. The first teeth formed are the milk (deciduous) teeth, replaced later by the permanent teeth. Human teeth have various shapes and have different functions. The incisors are the cutting teeth. The canines, which are next to incisors, help with tearing and fixing. In addition, the chewing surfaces of the premolars and molars grind and perform the major part of the work of chewing. Incisors and canines are also called the anterior (labial) teeth and the premolars and molars the posterior (buccal) teeth [13-16].

The tooth is divided into three parts: crown, neck, and root. The crown extends above the gum and is covered by enamel. The center of the tooth is a pulp cavity, which is filled with blood vessels, nerves, and connective tissue called pulp. The pulp cavity (dental cavity) within the root is called the root canal. The nerves and blood vessels of the tooth reach the pulp through a hole at each root called the apical foramen. The pulp cavity is surrounded by a living, cellular, and calcified tissue called dentin. The dentin of the tooth crown is covered by an extremely hard, nonliving, a cellular

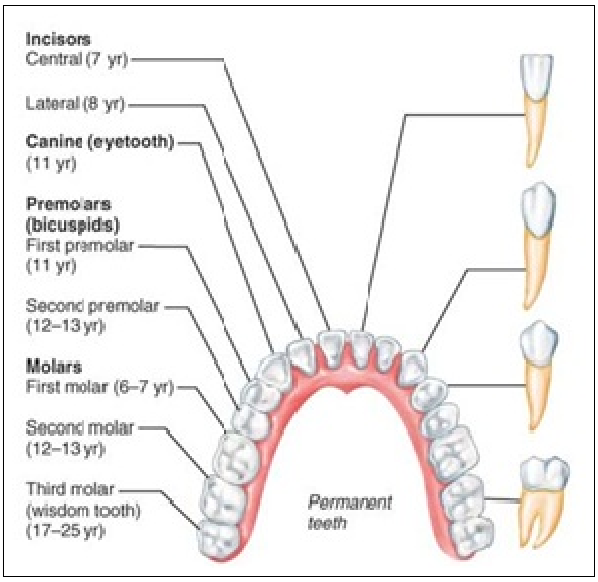

Figure 2.1 Human deciduous and permanent teeth of the lower jaw: The shapes of individual teeth and approximate age at which tooth is shown [17].

substance called enamel, which protects the tooth against abrasion and acids produced by bacteria in the mouth. The surface of the dentin in the root is covered with a cellular, bonelike substance, called cementum, which helps anchor the tooth in the jaw. Dentin is sensitive to pain. Enamel is the hardest substance in the human body and consists of 97% inorganic salts (chiefly hydroxyapatite). Dentin includes 70% inorganic substances, cementum 65%. The part of the tooth where cementum and enamel meet is called the neck of the tooth [16]. In an orthodontic treatment, four main types of

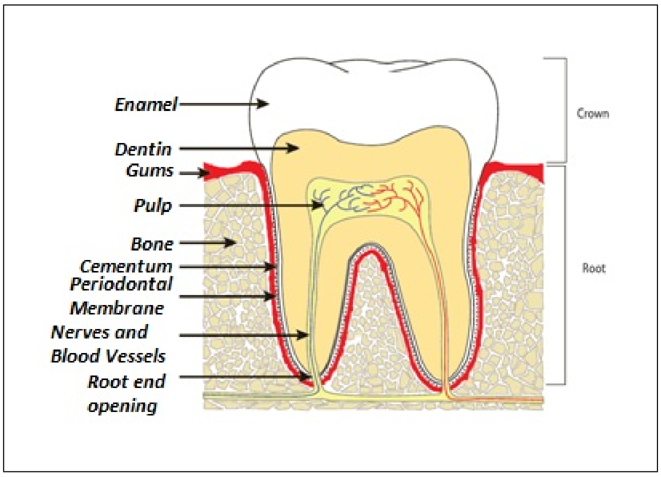

Figure 2.2 Cross section human molar with name of its structures [17].

appliance, which are removable, fixed, functional and extra oral devices, can be used.

Generally, fixed appliances are used for multiple tooth movements and alignment of ectopic teeth treatment because the results are more predictable. In order to operate with fixed appliances, a fixed point of attachment is produced to control the position of the teeth. The fixed point is attached brackets to the teeth. An arch wire is placed into the bracket slots to move the teeth [1]. The orthodontic braces are known as dental

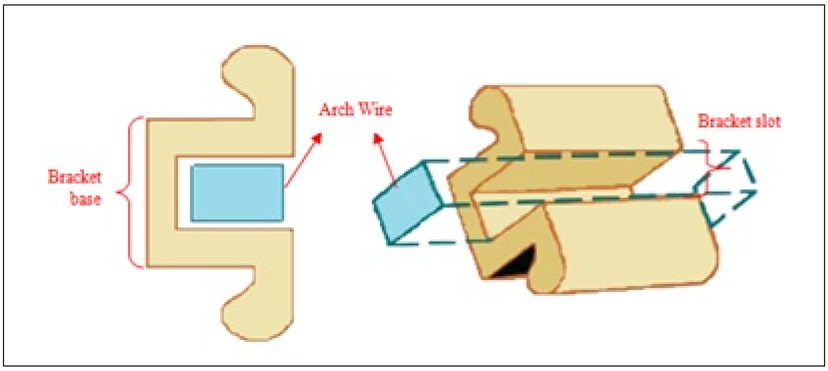

Figure 2.3 Bracket and arch wire :In a rectangular slot, rectangular wire is placed to fully control the teeth in three dimensions[28].

braces or simply braces. In an orthodontic treatment, braces are used in aligning and straightening teeth with treating several dental problems for example crowded teeth, buck teeth, protruded teeth, spaced teeth, overbites, under bites etc. The typical period of such a treatment is about two or three years.

For the dental treatment with braces, generally metal or plastic wires are inserted into orthodontic brackets, which can be made from stainless steel, ceramic material or plastic.

Ceramic braces are most preferable types by orthodontic patients who want to look good during treatment. Ceramic brackets offer many advantages over traditional aesthetic appliances (initially made from acrylic and later composite) because they provide higher strength, more resistance to wear and deformation, better color stability and, most important to patients, superior aesthetics.

All currently available ceramic brackets are composed of aluminum oxide; however, because of their distinctly different methods of fabrication, there are two types of ceramic brackets, namely, polycrystalline and monocrystalline [18-24].

The most apparent difference between the two is their optical clarity; monocrystalline ceramic brackets are noticeably clearer. Because of their rough surface, polycrystalline ceramics have a higher coefficient of friction than monocrystalline ceramics and stainless steel. The previous studies showed that debonding of polycrystalline brackets required higher force than mono crystalline ones [19,23]. In this study polycrystalline brackets were used because of their availability.

Figure 2.4 Bonded brackets with arc wire on teeth [28].

Although ceramic brackets are most preferable, they have some disadvantages. Ceramic brackets cannot bond chemically with acrylic and diacrylate bonding adhesives due to their inert aluminium oxide composition. For this reason, a sealant is used to provide chemical mediator between the bracket base and adhesive resin and enamel and adhesive resin. This chemical retention causes a risk on enamel damage in the form of crack during the debonding.

Bonding ceremic brackets currently occur with mechanical retention, using light or chemically cured adhesive without adding special bonding agents. There are several mechanical base designs. Some of them are microcrystalline, mechanical ball, dovetail, dimpled chemo/mechanical, silane coated buttons and polymeric. Several researchers have determined the debonding force of ceramic brackets with different adhesion mechanisms. According to their results, mechanically retained brackets have sufficient bond strength and cause less enamel damage at debonding compared to the chemically retained variety [24, 25]. Bond strength can show variation according to adhesive types for bonding and etching times of enamel [26, 27]. According to Omana et al., the mean bond strength of ceramic brackets is higher than other types of brackets.

First process is etching enamel surfaces to attach brackets with a composite resin [29]. Etching of tooth surface provides a key and lock relation between the enamel surface and the braket resin. In order to promote micromechanical bonding, several methods are used for producing surface roughness but acid etching is the most common method. Acid etching of enamel was introduced by Buonocore in 1955. Generally, to produce equally distrubuted etch pattern, a 37% phosphoric acid solution reported by Silverstone in1964 is used [30]. Although acid etching is a common method, new alternative methods have being researched since the acid application causes some disadvantages like deminerilazation and contamination of enamel surface [31]. The achiving of bonding orthodontic brackets depends on etching of the enamel surface and the bond between the tooth and bracket. For metalic braces, the tissue side of the bracket requires some mechanical retention. Bonding ceramic brackets is carried out coating its base with a sealent employed to bond inorganic fillers to resin matrix of resin-based composite. A plastic bracket is primed with a solvent.

In 1955 Micheal Buonocore reported the first meaningful proof of intraoral adhesion. He etched the enamel with phosphoric acid to get micromechanically roughened surface. Once bonding resin was applied the etched surface, the monomers of resin flowed into the etch pits and genereated mechanical bonding (Fig4). [32] Generally, phosphoris acid at concetration 37% is preferred etching agent. Once the enamel is etched, the acid solution should be rinsed away completely with water. After drying the enamel, it shows a white and dull appearance. Although etching of enamel with acid solution increases the bonding force of enamel, contamination of etched surface can decreases this force.If contamination occurs, it shoulde be removed and etchind procedure should be repeated. If proper procedures are followed, the bond strength of the resin to tooth surface is succeeded. For ceramic brackets, bonding failure often takes place at the wing of the brackets. [32] Resin polimerization can be achieved by conventional chemical-cure system or by light activation. In our study, we used

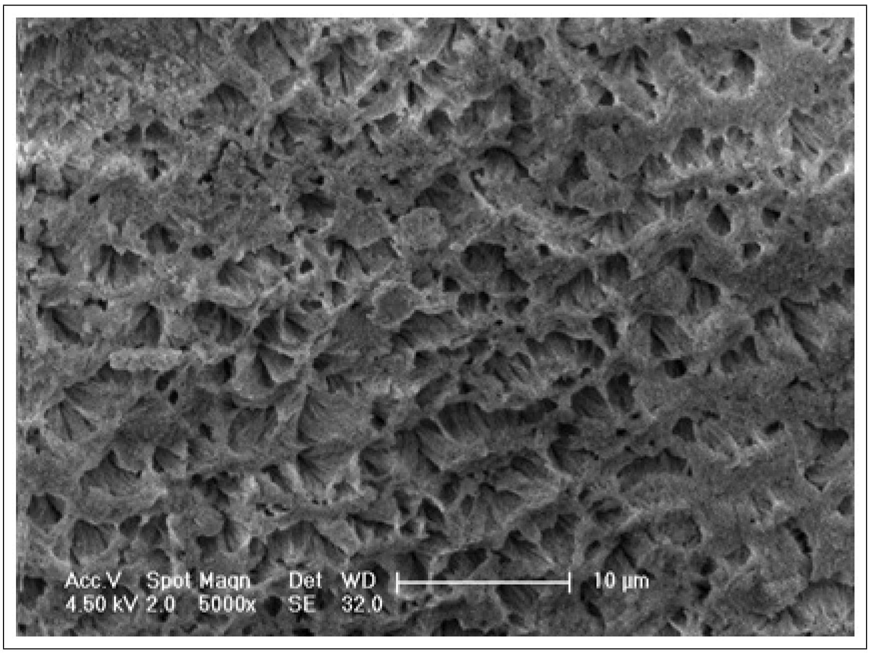

Figure 2.5 Acit etched enamel surface shows etced pits.

conventional chemical-cure system.

After the bracket bonding, wires interact with the brackets and the force applied by the wire to move teeth into the desired direction [29]. Dental braces are removed after completion of the treatment. Bracket removal is sometimes hard when the bond of resin to the bracket and etched enamel is strong.

A universally accepted test method does not exist to test orthodontic bond strength. But many of the studies have been compared their results with Reynolds et al. article in which they had recommended a 6.0 to 8.0 MPa minimum range as a normal value for orthodontic shear bond strength[30].

2.1.1 Risks in Orthodontic Treatment with Braces

Enamel demineralization and decalcification usually on smooth surfaces, is a common complication in orthodontics. According to study of Gorelick et al. white spot formation in children treated with fixed appliances, found that half of their patients had at least one white spot onto their teeth after treatment [34]. O'Reilly and Featherstone and Oggard et al. found that demineralization can occur rapidly, within the first month of fixed appliance treatment[35, 36]. During and after the treatment oral hygiene is vital. In order to prevent the risk of decalcification sugar intake should be controlled with a diet and to prevent white spot formation fluoride mouthwashes should be used [37, 38].Floride application can be helpful in remineralising of etched enamel and reduce the decalcification [39-42]. Laser application has been proposed as an alternative method to acid etching. [31].

2.2 LASERS IN DENTISTRY

2.2.1 Lasers

Lasers are devices that generate intense beams of light which are monochromatic, coherent, and highly collimated. The color (wavelength) of laser light is pure (monochromatic) when compared to other light sources, all photons that constitute the laser beam are coherence. Laser light has very low divergence. It can travel over great distances or can be focused to a very small spot. Because of all these properties of laser light, it is used several different areas. The term "laser" is an acronym for light amplification by stimulated emission of radiation. The term "light" is generally accepted to be electromagnetic radiation ranging from 1 nm to 1000 mm in wavelength. The visible spectrum ranges from approximately 400 to 700 nm. The wavelength range from 700 nm to 10 mm is called the near infrared (NIR), and above 10 mm is the far infrared (FIR). Conversely, 200 to 400 nm is called ultraviolet (UV); below 200 nm is the deep ultra- violet (DUV).

Laser light is based on electron stimulation between its energy states. Under the proper conditions an electron can move from its ground state (lowest-energy orbit) to an excited (higher) state, or it can go from a higher state to a lower state, but it cannot stay between these states. These energy states are called "quantum" states.

To excite an electron to higher quantum state, the atom must absorb energy from the outside. Similarly, when an electron goes down from a higher state to a lower state, the atom must release energy as electromagnetic radiation. Released energy is equal to energy differences between lower and higher states. The energy of this radiation is determined by the equation

$$\Delta E = h.\nu \tag{2.1}$$

where h is constant of Planck and ν is the frequency of the radiated light. The frequency and wavelength are related by the equation

$$\nu = \frac{c}{\lambda} \tag{2.2}$$

where c is the velocity of light in a vacuum and λ is the wavelength of the light. These equations show that the longer the wavelength of the light means the lower the energy of the photon; therefore, energy value of ultraviolet light is much more than infrared light.

Generally, when an electron is in an excited energy state, it must goes down to a lower state, releasing a photon of radiation. This is called spontaneous emission and the photon is emitted in a random direction and a random phase[43].

The amount of atoms in same level at a given time is called the population. For a normal population of atoms, there are always more atoms in the lower energy levels than in the upper ones. When the population of the upper level is much more than that of the lower level, it is called population inversion. To make a laser, a population inversion should be created.

A group of excited atoms in the same upper excited state are considered the incoming photon interacts with the first atom, causing stimulated emission of a coherent photon. These two photons interact with other next two excited atoms and this creates photons repeating self. At the end of the process, all coherent photons are in same phase and same direction. The upper energy state should have a long life time. The atoms should stay at the upper stable state for more time than at the lower level. The energy, which excites atoms, is provided externally by some energy source which is usually referred to as the pump source.

The four-level energy diagram shown below (Figure2.6) represents some real lasers. The electron is pumped (excited) into an upper level E4. It then passes through to E3, then to E2, and finally to the ground state E1. If a great population of such atoms, at equilibrium, is pumped continuously, a population inversion will be occur between E3 and E2 energy state, and a entered photon the population will be amplified coherently. To gain a laser light, a positive feedback mechanism is necessary to keep

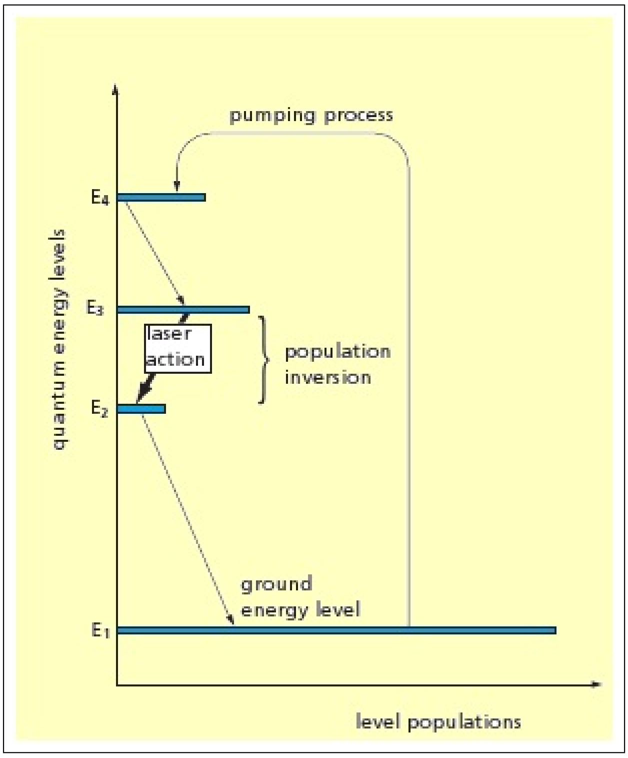

Figure 2.6 Population inversion [44].

coherent output. This feedback mechanism is called resonator, which includes system of mirrors to reflect photons sending back into the excited state population to amplify the output continuously [44].

Laser systems can be classified due to their wavelength, active material used, power or mode of operation. The most important parameter of a laser is the wavelength that determines laser penetration depth inside the tissue.

When laser light interacts with a biological tissue three different cases occur: Reflection, absorption and scattering (Figure 2.7). The direction of the reflected wave

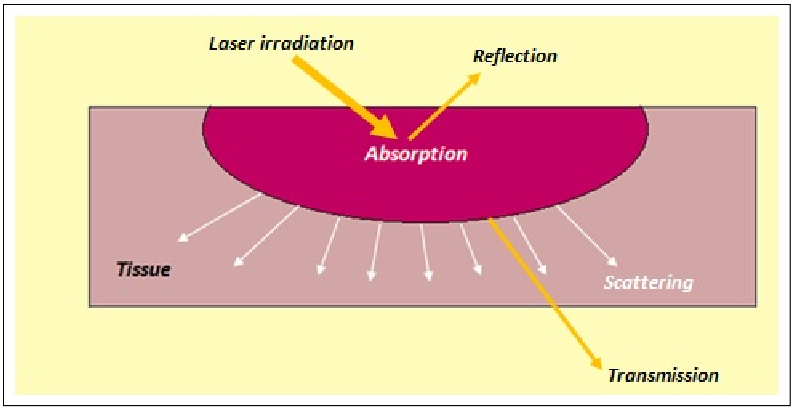

Figure 2.7 Geometry of reflection, absorption and scattering.

is often opposite the incident wave. In reflection, wavelength or photon energy of the light wave is not altered. When light is reflected from tissue surface, it does not cause thermal effect. Tissue can absorb the energy of the light and reemit this energy as another light which has smaller amount of energy. The energy of the light absorbed by tissue is converted into heat energy [45]. The optical properties of each tissue are depends on the concentration and allocation of their component substances Absorber substances in biological tissues are water molecules or macromolecules (such as hemoglobin). Absorption coefficient is the term determines how far into a material light of a particular wavelength can penetrate before it is absorbed. The graph in Figure11 shows that absorption of light by water is reversely proportional to the wavelength of light.

2.2.2 Lasers in Dentistry

Lasers are classified according to the content of their active (lasing) medium, wavelength and power or mode of operation. A number of laser systems have been

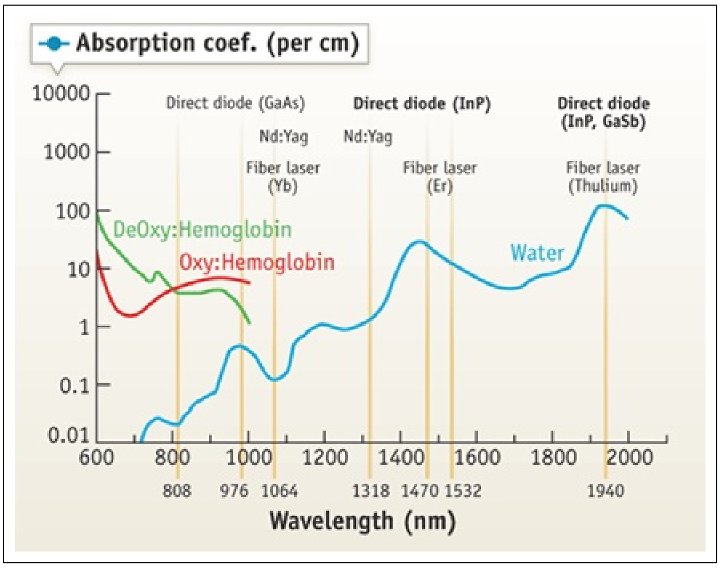

Figure 2.8 A Graph showing absorption coefficient of water vs. different laser wavelength.

used in dentistry. Many different applications have been developed, depending on the different parameters of the emitted laser light.

Lasers in dentistry were developed by Maiman who created the first operational laser (a ruby laser) in I960. This was followed within 3 years by the development of the argon, carbon dioxide (CO_2), and neodymium: yttrium-aluminum-garnet (Nd: YAG) lasers, which remain the most widely used lasers in medicine.

Dental lasers have been using operative dentistry studies like detection of caries, surface etching, removing decay, removal of defective restoration, and bleaching of teeth. The aim of this study is surface etching. The main advantage of the laser-etched surface is acid resistance. It yields more resistant enamel for caries attack.

The absorption of laser energy by water inside the tissue is the most important for hard tissue applications. As a hard tissue, enamel of teeth includes 4% water. Laser light interacts with water droplets and causes the sudden vaporization of water so that small micro explosions occur for the break-up of the hydroxyapatite structure. Laser power provides local melting and ablation of enamel surface which has the similar topology of acid etched. The depth of the surface roughness depends on the power and the wave length of the laser [46]. During this procedure, pulp cavity is affected by laser energy as a side effect of heating. There are lots of studies about thermal effects of laser on dental tissues. According to the previous studies no histological changes were discernible with an intrapulpal temperature increase of $1,8\,^{\circ}$C. $5,5\,^{\circ}$C was accepted as threshold temperature of pulp damage in most studies. They have also found that an increase in intrapulpal temperature of $11,1\,^{\circ}$C showed abscess formation. That is why $5,5\,^{\circ}$C was accepted as threshold temperature in this study.

Different types of laser such as CO_2, Er: YAG, Nd: YAG, and Er,Cr: YSGG have been used in orthodontics for enamel conditioning to bond brackets [47-51].

Some studies show that enamel etching with Nd:YAG lasers results weaker bonds compared with acid etching. It was suggested that to increase efficieny of the Nd:YAG laser a topical absorber like black ink should be applied to enamel surfaces and low pulse energies should be used[52, 53].

Some CO_2 etching studies suggested that bonging forces are comparable with bonding force on acid etched surface [54]. On the other hand during the irradiation of CO_2 laser, pulp temperature increased above the critical temperature value. To avoid the excessive temperature rise, laser can apply under control [11, 55]. CO_2laser in continuous wave mode may cause cracking, flaking, crater formation, melting and recrystallization of enamel [56].

Er: YAG and Er,Cr: YSGG, was compared with acid etched surface are highly absorbed by enamel and they can also etch dental hard tissue without temperature rise in pulp and without using topical absorber [57, 58]. In the study of Sasaki et al, after the ER:YAG irradiation to a group of teeth, phosphoric acid was applied to dental tissue. Those groups showed strong shear bond strength compare to acid etched and laser etched groups. As a result, there has been a conflict between the research findings about laser etching because of the different type of lasers used for the comparison studies. Some studies showed that acid etched teeth had more bond strength than laser etched teeth whereas other studies indicated that laser etching could result in comparable bond strength with acid etching [59-61]. In this study, 1940

References:	Laser Type:	Laser Parameters:	Results:
Lan W. H. et al.	Er:YAG	300 mJ/pulse, 10 pulse per second (pps), 10 s	The mean bond strength is higher than acid etched group.
Lee B. S. et al.	Er:YAG	300 mJ/pulse, 10 10 pulse per second (pps), 10 seconds	The mean bond strength of the laser group is not significantly different from that of the acid-etched group.
Dostálová T.	Er:YAG	208 mJ 1 seconds.	The mean bond strength of the laser group is not significantly different from that of the acid-etched group.
Obata A.	CO2	3W 3s super and normal pulse	The mean bond strength of the laser group is lower than of the acid-etched group.
L Corpas-Pastor	Nd:YAG	8 seconds at 15 pulses per second/0.75 watts	The mean bond strength of the laser group is lower than the acid-etched group.
Basaran G	Er,Cr:YSGG	1 W or 2 W for 15 seconds	The mean bond strength of the laser group is not significantly different from that of the acid-etched group.
Fraunhofer J.A.	Nd:YAG	1W, 2W 12 s	The mean bond strength of the laser group is lower than the acid-etched group

Figure 2.9 Results of laser etching studies with different type of lasers.

nm Thulium Fiber Laser was used to remove the dental hard tissue because of its compactness, beam quality, and environmental reliability as well as easily controlled modulation.

3. MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 Teeth

Bovine mandibular incisors teeth were used in this study. They were obtained from a local abattoir. These teeth were selected instead of human teeth because the limited availability of human teeth and they have similar physical properties of human teeth. They also do not cause the contamination to the handlers and they are risk free of infectious disease transmission. Using of bovine teeth in etching studies were reported previously [62-65].

Figure 3.1 Extracted bovine incisors.

3.1.2 Laser

The laser used in this study is Thulium Fiber Laser (IPG Laser; GMBH) which has 1940 nm wavelength. It was applied in both continuous and modulated modes. Duty cycles of laser controlled by a computer. The beam was delivered a flexible fiber guide from laser source. The laser spot size was 2 Î$\frac{1}{4}$m. At the beginning of each laser etching experiment, the laser power output was monitored by a power meter (Newport, Model 1918-C) and the delivery system was calibrated. The power meter consists of a sensor which measures the power and a monitor to read value of the power. The laser power was maintained at maximum 3.5 Watts. During this process, the thermal

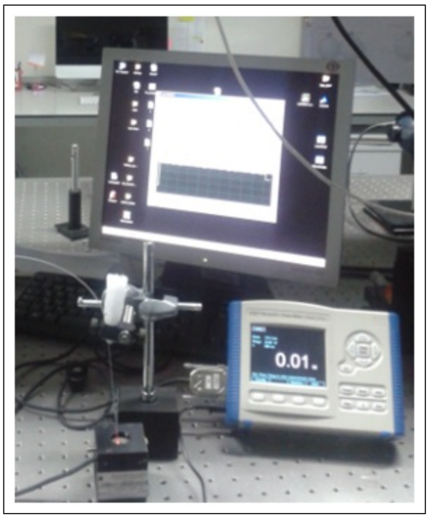

Figure 3.2 Power meter and its sensor.

changes in the tooth pulpa were measured by a K-type thermocouple (OMEGA, OM-CP-0CTTEMP, UK) placed in lingual surface cavity. K-type thermocouple is a material that used in temperature measurement system to measure intra pulpal thermal changes during irradiation.

3.1.3 Cooling Materials

During the laser exposure water is applied to enamel with water spray and air pressure is applied with compressor.

3.1.4 Orthodontic Ceramic Brackets

Polycrystalline ceramic brackets (G&H.US) for maxillary lateral incisors were bonded to the etched bovine incisors. Polycrystalline brackets were used in this study because they are most preferred brackets type because of esthetical reasons.

Figure 3.3 Polycrystalline ceramic bracket samples.

3.1.5 Acid Etchant

For the acid-etching technique, 37% phosphoric acid solution (3M ESPE Scotch bond) was applied to teeth surface with an applicator.

Figure 3.4 Phosphoric acid etchant.

3.1.6 Adhesive

In this study, as a bonding agent chemically curing Bis-GMA resin set (3M Unitek, Unite Bonding Adhesive Set,US) was used to bond brackets to the surface etched by acid or laser.

Figure 3.5 Adhesive, sealant and their applicators.

3.1.7 Debonding Test Machine

A universal testing machine (Lloyd, LF Plus, UK) was used to measure the shear bond strength and detachment time of each specimen. It is consisted of an upper moving section and a rigid base section. A steel jaw was prepared specifically for the base section as a testing frame. To debond the brackets from teeth, the gypsum blocks were placed in this testing frame. Also a steel shearing blade (with 19, 5 cm length) with a square hole was mounted to the upper part of the machine. To control the machine, read, collect and, record data, a computer connected to the testing machine has been utilized.

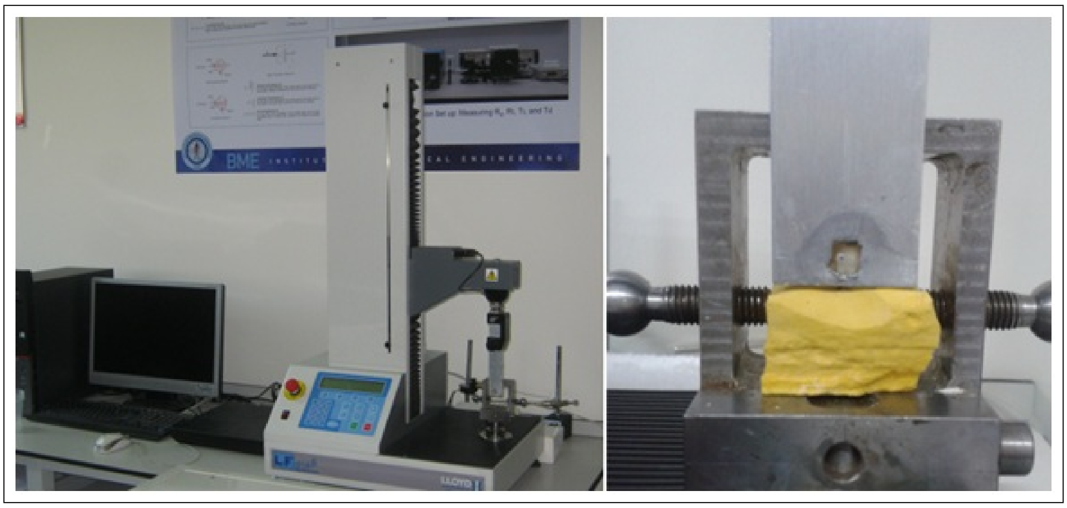

Figure 3.6 Universal testing machine and its computer.

3.2 METHODS

3.2.1 Sample Preparation

Once the teeth extracted, they were cleaned from soft tissue, debris and blood. Teeth were stored in tap water which was selected according to the previous studies [66].The water was changed three times a week to prevent the bacterial formation. Before the etching procedures, each bovine crown was embedded in gypsum blocks. On the lingual surface of the each tooth, a cavity was drilled by a round diamond high speed bur with a 1 mm diameter for the thermal measurements. Silicon thermal paste was filled into the cavity of each tooth to transfer the heat from pulp cavity to thermo probe before thermo couple was placed. The labial surfaces of the teeth were

Figure 3.7 Drilled lingual surface of teeth, a round bur and silicon thermal paste filled cavity.

polished with pumice paste for 15 seconds to remove plaque and the organic pellicle that normally cover the teeth and rinsed with water and dried.

3.2.2 Acid Etching

Acid-etching technique is used to prepare control group. 37% phosphoric acid solution was applied to the bonding surfaces, which are 4x4 mm^2 area, of 8 teeth with applicator for 30 seconds, rinsed thoroughly with water, and dried. The etched enamel showed a uniform, dull, frosty appearance. After then, bonding procedure was carried out.

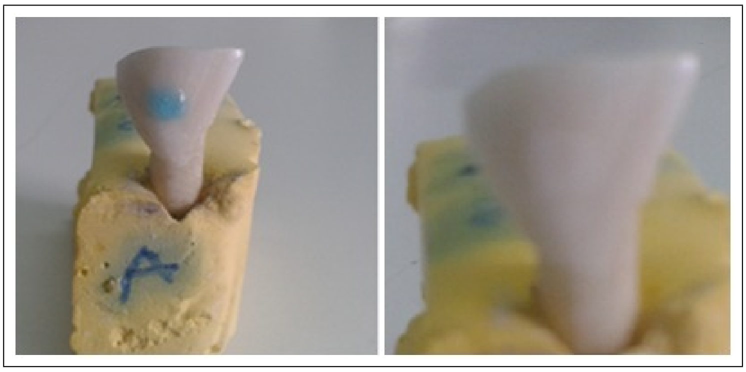

Figure 3.8 (a) Etching enamel with phosphoric acid and (b)Frosty and dull apperance of etched enamel.

3.2.3 Laser Etching

Before the each etching procedure, the laser power output was measured from fiber tip by the power meter to calibrate the laser system. The external diameter of the fiber guide was 2 mm. In order to determine the laser parameters, preliminary experiments were performed in three sections:

1. Determining the laser durations for each lasing mode
2. Finding out the distance between enamel and fiber tip
3. Determining the laser exposure: point shot or scanning the etching area.

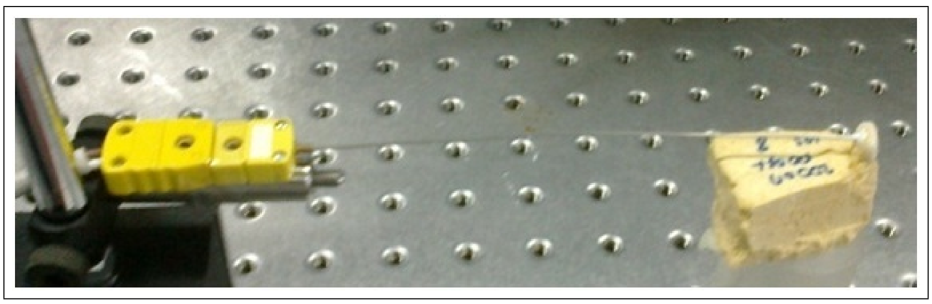

Figure 3.9 Thermocouple probe placed into the teeth cavity.

The main experiments were preceded based on the results acquired from the preliminary experiments. During laser irradiation, thermal changes in pulp of tooth specimen were measured by K-type thermocouple in each part of the experiment. Thermal probe was placed just beneath the roof of the pulp chamber to avoid contacting with the dentinal wall.

In the first section of the preliminary experiments, laser was applied to the enamel surface in seven different modes. At the same time of exposure, intra pulpal temperature increase on tooth samples was measured. Temperature increase was the limitation to determine the exposure time. The limitation of the temperature increase was $5,5\,^{\circ}C$ which causes thermal necrosis in pulp [67]. Fiber tips was placed 1.5cm away from the enamel surface. Laser was applied to same area of sample surface.

Mode1:	**Measured Power: 3.5W ± 0.05**	**Continuous**
Mode2:	**Measured Power: 3W ± 0.05**	**Continuous**
Mode3:	**Measured Power: 3W ± 0.05**	**On time:100(ms) Off time:100(ms)**
Mode4:	**Measured Power: 3W ± 0.05**	**On time:200(ms) Off time:100(ms)**
Mode5:	**Measured Power: 3W ± 0.05**	**On time:150(ms) Off time:50(ms)**
Mode6:	**Measured Power: 3W ± 0.05**	**On time:200(ms) Off time:50(ms)**
Mode7:	**Measured Power: 3W ± 0.05**	**On time:100(ms) Off time:100(ms)**

Figure 3.10 Laser parameters used in the first part of preliminary experiments.

Appropriate parameters were chosen according to the result of time of temperature increase in pulp chamber.

In the second section of the experiments, laser tip was placed in three different distances from enamel surface respectively for determining the optimum distance. Laser was applied on the same area of enamel surface as laser beam spot size (2 mm). Laser was irradiated at same mode and same duration at each distance.

Duty cycle of laser (Measured Power: 3W ± 0.05)	Distance
On time:200 ms Off time:100 ms **Laser duration time: 10.2 s**	1.0cm
	1.5cm
	2.0cm

Figure 3.11 Laser parameters used in the first part of preliminary experiments.

After the etching procedure, ceramic brackets were bonded to etched area of teeth surfaces then debonding forces for each tooth were measured, and compared. In the third section of the preliminary experiments, 4x4 mm^2areas of teeth surfaces, which was the size of the bracket base, were etched with laser by scanning from 2cm distance in two different laser modes. During the laser scanning procedure, temperature increase was controlled. Brackets were bonded to etched surface and were exposed to debonding test.

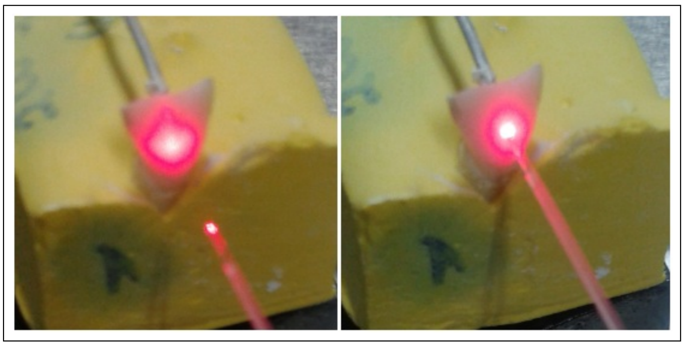

Figure 3.12 Laser application from 2cm distance and contact mode.

The tasks of main experiment were defined according to debonding results of preliminary experiments. Main experiments were performed in two sections:

1. Lasing without cooling

2. Lasing with cooling

In the first section, bovine enamel was scanned by laser with a direct contact of fiber tip without controlling the temperature increase. According to the whitening of the enamel initiation, application time of laser irradiation was defined for each laser parameter.

Figure 3.13 Enamel whitening after laser exposure.

After the etching of the enamel, brackets were bonded each tooth and debonding forces of them were tested. In order to increase etching duration with scanning by direct contact, enamel surfaces were cooled with water spray and air-pressure in second phase.

In first part of enamel cooling experiment, water spray was applied in different temperature values which were 24 °C (room temperature), 5, 04 °C and 9, 24 °C . Temperature of pulp cavity was 24 °C during the procedure. After the etching procedure, brackets were bonded to teeth surfaces and than debonding forces were measured.

Measured Power	**Laser Mode**	**Duration Time**			
3W ± 0.05	**Continuous**	**3 s**	No cooling	Lateral cooling	Direct cooling
3W ± 0.05	**On time:200 ms Off time:100 ms**	**10 s**	No cooling	Lateral cooling	Direct cooling
2W ± 0.05	**Continuous**	**5 s**	No cooling	Lateral cooling	Direct cooling

Figure 3.14 Lasing modes and cooling directions.

Before the beginning of etching with air-pressure cooling experiments, thickness of each tooth specimen was measured to keep constant distance between fiber tip and thermo couple probe in order to reduce the standard deviation of thermal measurement. Temperature at pulp was 24 °C.

The teeth specimens were exposure to laser with air-pressure cooling directly and laterally, and without cooling in different laser modes.

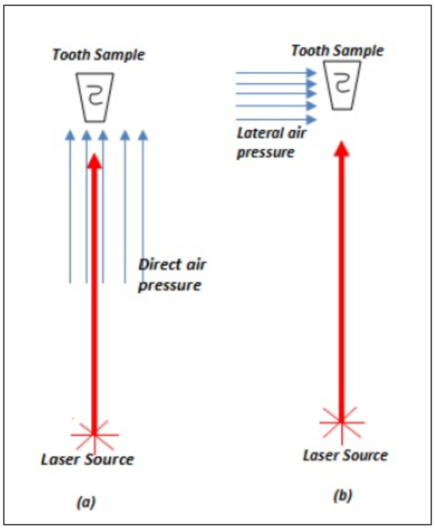

Figure 3.15 Schematic of air pressure application modes (a) direct air pressure cooling (b) lateral air pressure cooling.

At the end of the whole experiments, a tooth sample from each experiment was imaged by SEM (Scanning Electron Microscope) to observe surface roughness.

3.2.4 Bracket Bonding

The surface etched by acid or laser and bracket base was covered with a small amount of bonding sealant (3M Unitek) with a brush. Adhesive was applied to the bracket base over primer. With the adhesive applied, the bracket was seated and positioned in front of the cavity onto the tooth surface immediately and pressed firmly. Excessive sealant and adhesive were removed from the periphery of the bracket base without disturbing bracket position to keep the bond area of each tooth uniform.

Removing excess adhesive is important to prevent periodontal damage and possibility of decalcification [68]. It was waited 4 minutes for curing of adhesive. In order to simulate intra-oral conditions of humidity, all bonded specimens were placed in a beaker with distilled water inside and stored in an incubator at 37 °C for 48 hours in to keep moist and to complete composite polymerization of resin.

Figure 3.16 Bracket bonding procedure.

3.2.5 Debonding Test

The etched teeth specimens were placed in a testing frame before debonding procedure. The tensile load was applied to the specimens and the point where the bracket deattaches from the tooth was recorded in Newton. The results acquired from the tensile strength of the acid etched groups and the laser etched groups were compared.

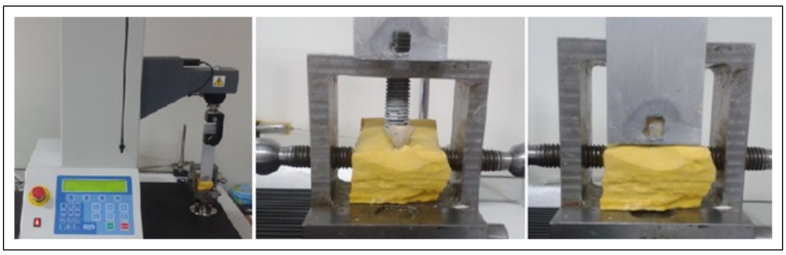

Figure 3.17 Debonding test with Universal Testing Machine

4. RESULTS

4.1 PRELIMINARY EXPERIMENTS

4.1.1 Laser Duration

At the beginning of enamel etching experiments, time of temperature rise in pulp was measured by a thermocouple in order to determine the laser application time for each laser parameters. The limitation of temperature increase is 5,5 °Cwhich is critical for pulp. Fiber tips was placed 1.5 cm away from the enamel surface.

Number of sample :8	3.5CW (a)	3CW (b)	100on-100off (c)	200on-100off (d)	150on-50off (e)	200on-50off (f)	100on-100off (g)
Average :	4.375	9.75	10.75	10.875	7	5.75	10.75
Standard deviation:	2.6692	2.3145	3.10529	4.051013	3.8172	1.66904	3.10529

Figure 4.1 Temperature increase data to critical temperature for different laser parameters.

According to t-test there are significant differences between “a” and “b, c, d” groups and “f” and “b, c, d” groups ($p < 0.05$, student t-test).

4.1.2 Laser Application Distance

200on 100off modulated laser parameters, which provide maximum power and minimum temperature increase, were chosen in order to determine optimum distance between enamel surface and fiber tips. Laser applied to enamel away from 1.0cm, 1.5cm and 2.0cm respectively for 10 second. After the etching procedure ceramic brackets were bonded to etched area of enamel and after that debonding forces for each tooth were measured and compared between each other.

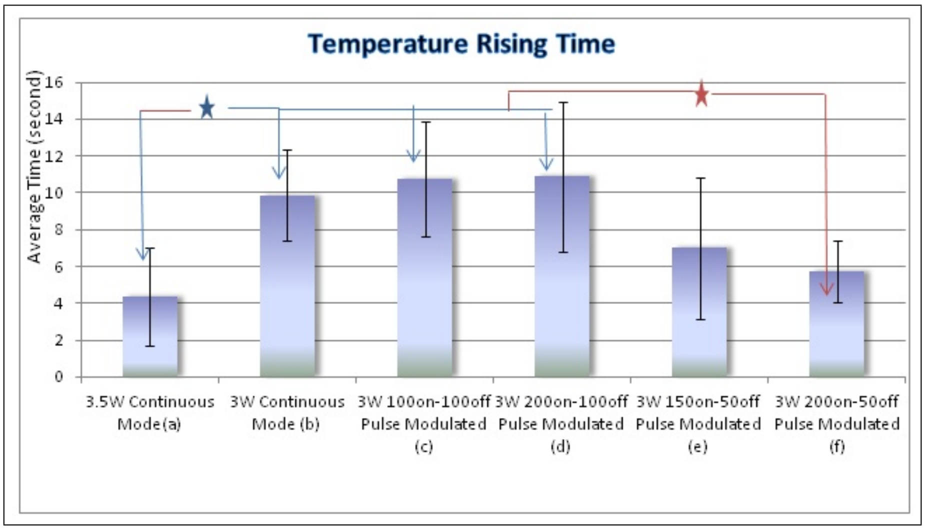

Figure 4.2 Temperature increase to critical temperature graph for different laser parameters. There are significant differences between "a" and "b, c, d" groups and "f" and "b, c, d" groups ($p < 0.05$, student t-test). (Stars show significantly different groups)

Number of sample :8	1.0cm	1.5cm	2.0cm
Average :	2.4175	3.335	3.459375
Standard deviation:	1.751128	1.56553	0.883396

Figure 4.3 Debonding Forces depend on distances between fiber tips and enamel surface.

According to t-test there is not any significant difference between these three groups. After the laser application to enamel surface from different distance, it was observed that debonding forces of brackets are weaker than expected acid debonding value (59.27 ∓ 15.5 Newton).

According the debonding force results for laser applications in different distance, 2cm distance is chosen because the average debonding forces is highest value and its standard deviation is least one.

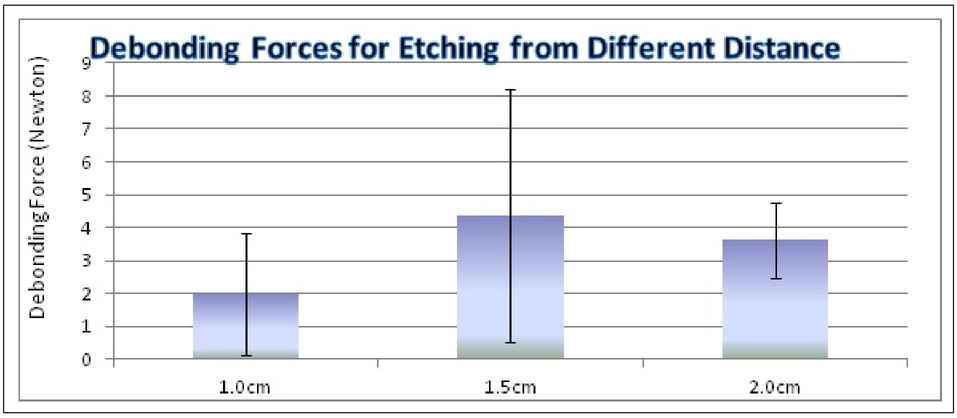

Figure 4.4 Debonding Forces depend on distances between fiber tips and enamel surface. There is no significant difference between debonding forces these three groups (p >0.05, student t-test).

4.1.3 Lasing with Surface Scanning

Rresults of all debonding experiments, mentioned above, showed less debonding forces than the acid etched ones. During these experiments laser applied on same area of enamel surface as laser beam spot size (2 mm). Hence laser scanning of enamel surface were decided to etch enamel as same as size of bracket base. In order to compare the laser parameters, 3W irradiation in CW mode and 200 on 100 off irradiation in pulse mode were applied from 2cm distance to enamel surfaces. During the laser scanning procedure temperature increase was measured. Initial average temperature of pulp was $24 \mp 0,05\,°C$.

Number of sample:8	Temperature Rise(°C) 3W Continuous t=5s	Temperature Rise(°C) 3W200on 100off t=10.2s
Average:	6.5525	8.06
Standard deviation:	1.447280701	1.499066376

Figure 4.5 Temperature rise data of pulpas of teeth when laser applied enamel surfaces with scanning from 2cm distance

According to t-test there is a significant difference between these two groups (student t-test, $p<0.05$).

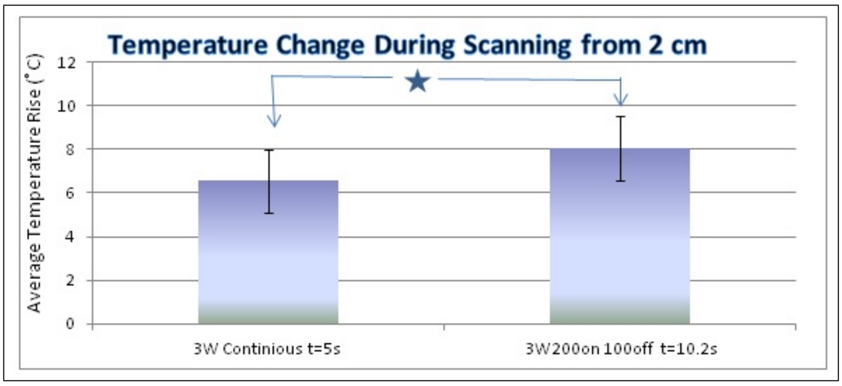

Figure 4.6 Temperature increase of pulpas of teeth when laser applied enamel surfaces with scanning from 2cm distance. There is a significant difference between these two groups ($p<0.05$, student t-test). (Stars show significantly different groups)

Brackets were debonded to these etched enamel surfaces. Debonding forces are shown below:

Number of sample :	3W Continuous t=5s Debonding Forces(N)	3W200on 100off t=10.2s Debonding Forces(N)
Average:	1.681666667	1.911666667
Standart deviation:	1.36565613	1.918774783

Figure 4.7 Debonding force data of brackets debonded laser etched surfaces with scanning from 2cm distance.

According to t-test there is not any significant difference debonding forces between 3W continuous laser and 200 on 100 off modulated groups.

4.2 MAIN EXPERIMENTS

4.2.1 Scanning in Contact Mode

Enamel surfaces were scanned with fiber tips in contact without controlling temperature rise. In order to prevent carbonization of the enamel surface, laser duration of each laser parameter was chosen according to color changing of the surface. During

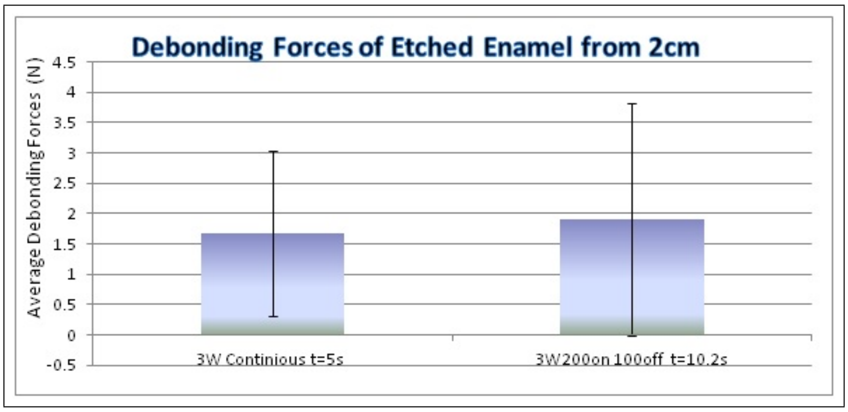

Figure 4.8 Debonding forces of brackets bonded enamels that were etched with laser scanning from 2 cm distance. There is no significant difference between these two groups ($p > 0.05$, student t-test).

the etching procedure with a 3 Watt power and 200 on 100 off modulated pulse in 15 and 10 seconds and 200 on 50 off modulated pulse in 15 seconds and 3W continuous laser in 3 seconds were applied in enamel surface.

Debonding forces of bonded brackets after the laser etching are shown below:

Sample Number:10	**3W 200on 100 off t=15s**	**3W 200on 100 off t=10s**	**3W 200on 50 off t=15s**	**3W continuous t=3s**
Average:	4.356666667	2.467	2.927	1.486666667
Standard deviation:	4.346150508	2.330288728	1.734474304	1.082142936

Figure 4.9 Debonding force data of brackets debonded laser etched surfaces with scanning in contact mode.

4.2.2 Lasing with Cooling

In order to reduce temperature rise in pulpa, water spray and air-pressure were applied to cool enamel surface during the laser applications and laser power were chosen in high value to have a short time laser application.

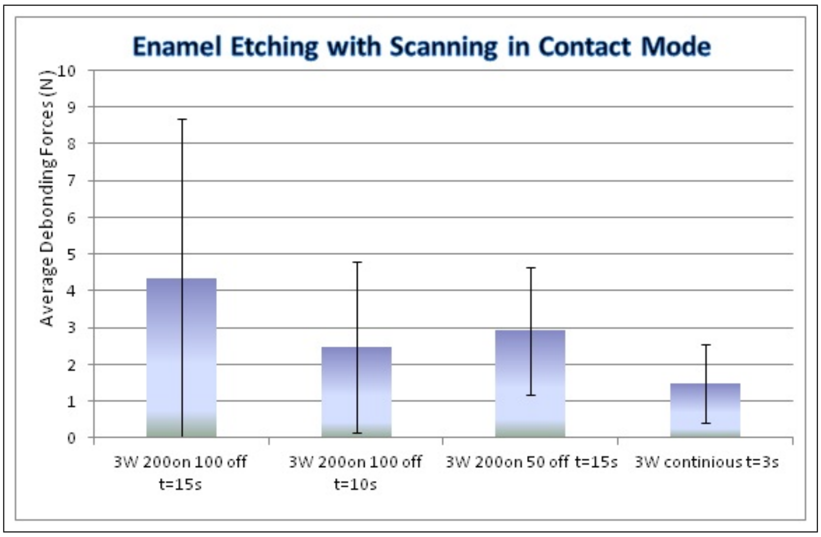

Figure 4.10 Debonding force graph of brackets debonded laser etched surfaces with scanning in contact. There is no significant difference between these two grouos (p >0.05 , student t-test).

1. Lasing with Water-Spray Cooling In the first part of enamel cooling experiments water was applied in different temperature values which are 24 °C (room temperature), 5, 04 °C and 9, 24 °C. Average pulp temperature was 24 ∓ 0, 05 °C during the procedure.

Sample Number:8	3W Continuous Water temperature=24ºC t=10s	3W Continuous Water temperature=5.04ºC t=20s	3W Continuous Water temperature=9.24ºC t=20s
Average:	7.528571429	-9.642	-3.742857143
Standard deviation:	1.532029	-2.53644	-1.92169

Figure 4.11 Data of temperature change in pulp during etching procedure with water cooling

Brackets were bonded on enamel teeth surfaces. After incubation, debonding forces were measured. Results of the measurements are shown:

2. Lasing with Water-Spray Cooling

Before air pressure cooling experiments, the thickness from enamel to pulp was measured as 1.41 ∓ 0.43 cm for each tooth in order to keep similar distance

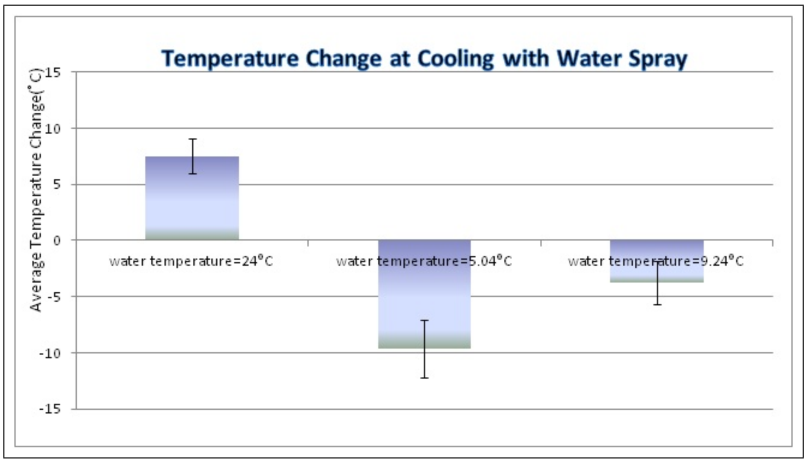

Figure 4.12 Temperature change in pulp during etching procedure with water cooling: (a) Temperature increases above the pulp temperature (b) and (c) Temperature decreases below the pulp temperature.

Sample Number:8	3W Continuous Water temperature=24°C t=10s	3W Continuous Water temperature=5.04°C t=20s	3W Continuous Water temperature=9.24°C t=20s
Average:	3.38625	0.526	1.472857
Standard deviation:	2.329272955	0.423355642	1.847789

Figure 4.13 Debonding force data of brackets from etched enamel surface that cooled with water

between thermo probe and fiber tip. Then, laser was applied for etching with air-pressure cooling in two ways. During the etching procedure air-pressure was applied laterally and directly to enamel surface. According to temperature results of measurements, ceramic brackets were bonded to the group of etched enamel with lateral air pressure cooling. Debonding forces of brackets were shown below:

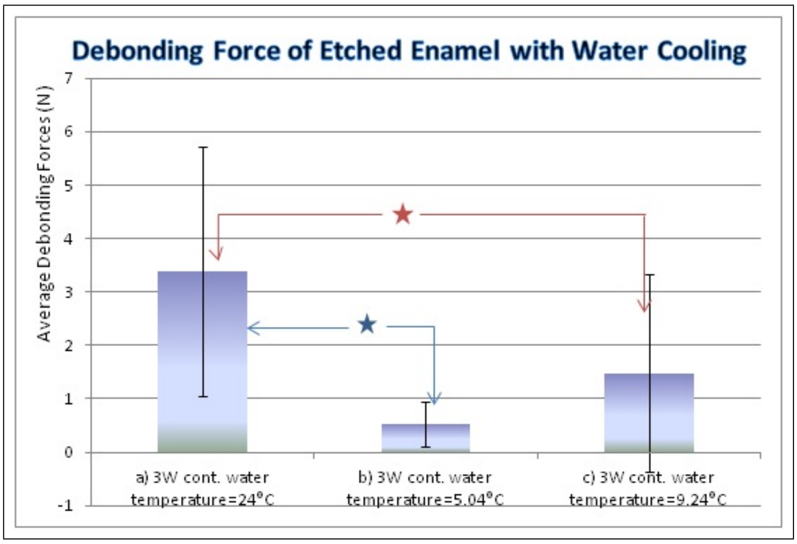

Figure 4.14 Debonding force data of brackets from etched enamel surfaces that cooled with water. There are significant differences between “a” and “b” groups and “a” and“c” groups ($p < 0.05$, student t-test). (Stars show significantly different groups)

4.3 SEM IMAGES

The twelve specimens were examined using a scanning electron microscope (SEM) to determine whether enamel etched or not. SEM images showed that enamel surfaces were melted partially.

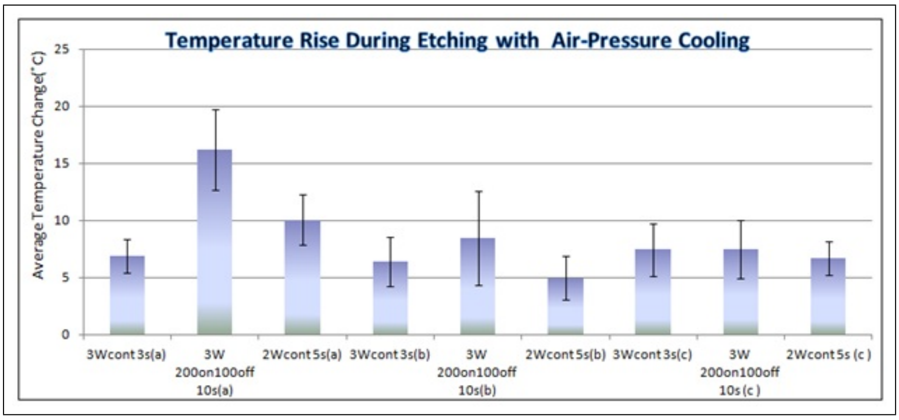

Figure 4.15 Temperature change in pulp during etching procedure with air-pressure cooling: (a) no cooling (b) air-pressure cooling directly and (c) air-pressure cooling laterally. There are no significant differences between these groups ($p>0.05$, student t-test)

Sample Number:8	3Wcont t=3s	3W 200on100off t=10s	2Wcont t=5s
Average:	1.015	0.75	1.03875
Standard deviation:	0.890184572	0.664293395	0.611892556

Figure 4.16 Debonding forces of brackets from etched enamel surfaces that were cooled with air pressure laterally.

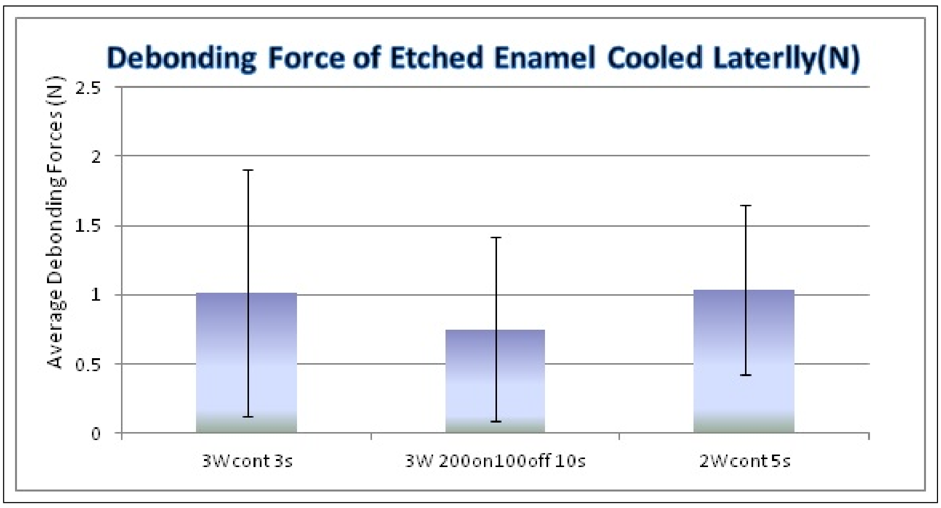

Figure 4.17 Debonding forces of brackets from etched enamel surfaces that were cooled with air pressure laterally. There are no significant differences between these groups ($p>0.05$, student t-test)

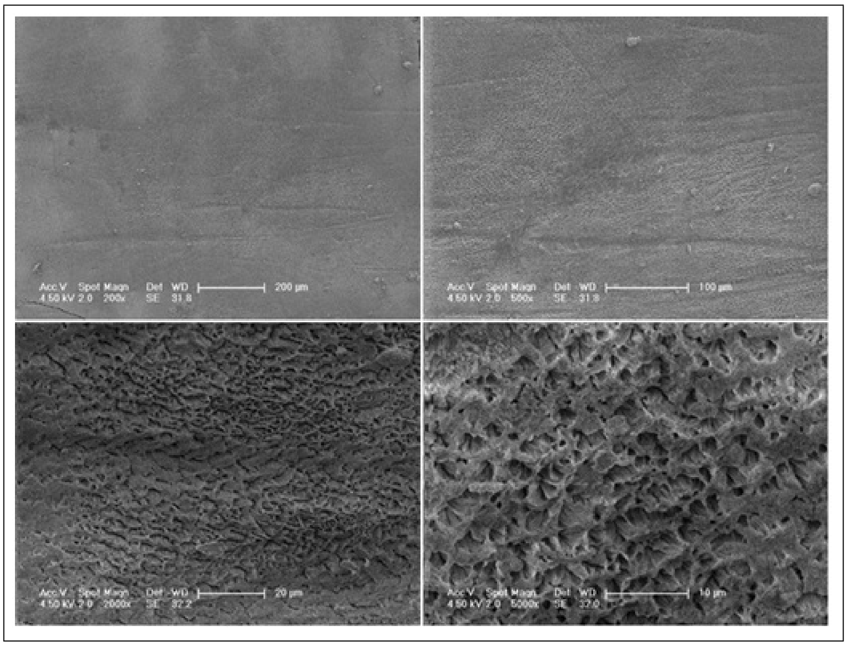

Figure 4.18 Conventional Acid Etching Method.

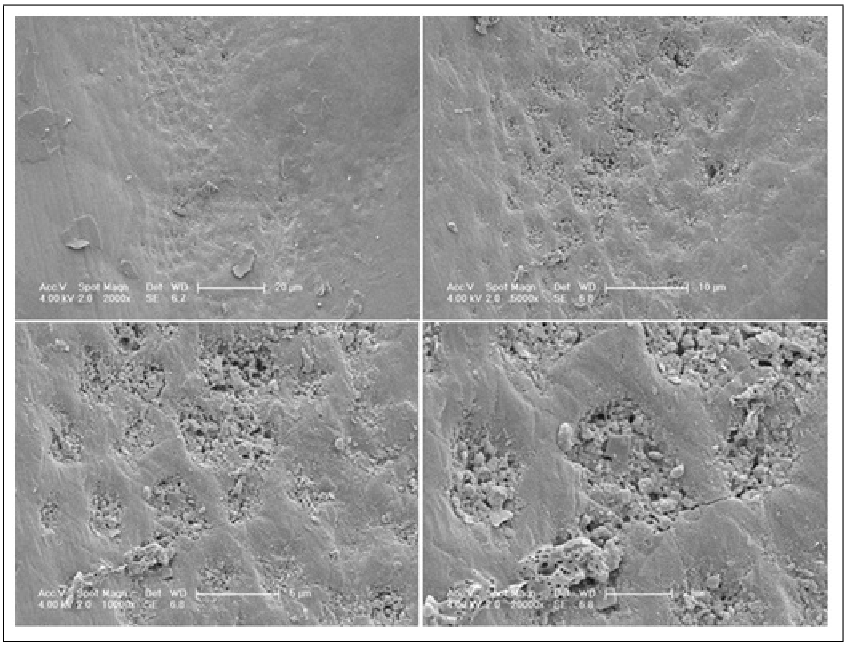

Figure 4.19 3W continuous laser mode, exposure time is 3 second.

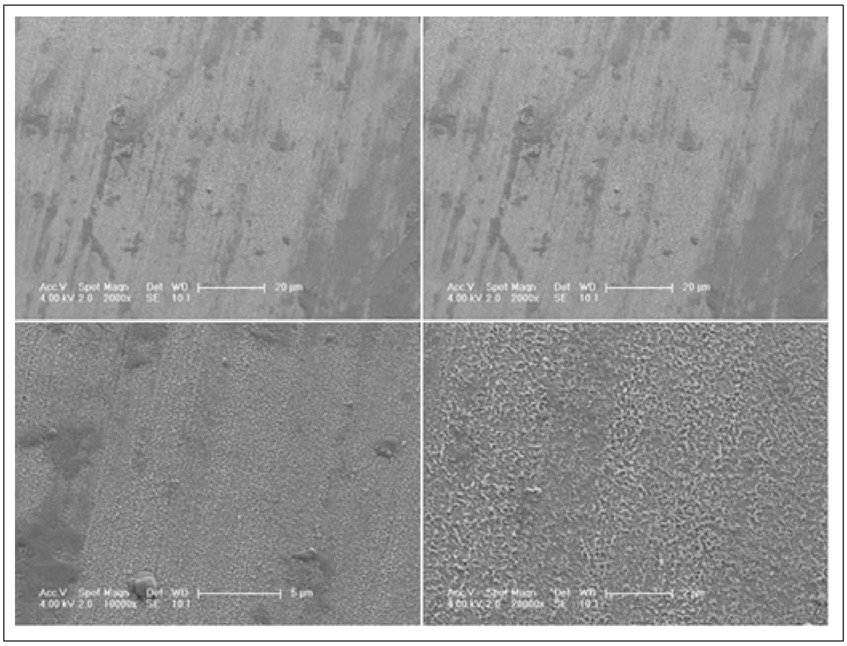

Figure 4.20 200 on 100 off pulse modulated laser, exposure time is 10 second.

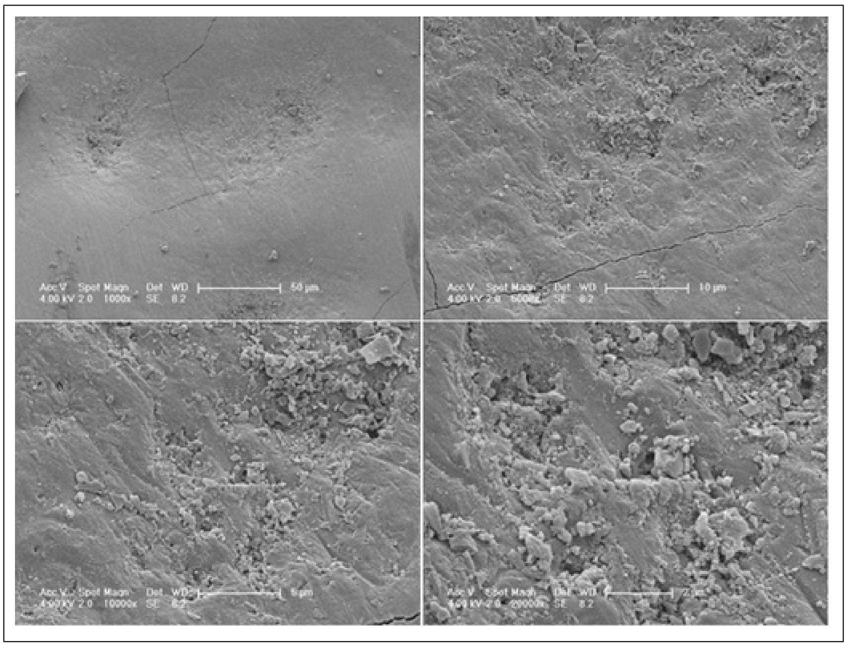

Figure 4.21 200 on 100 off laser, exposure time is 15 second

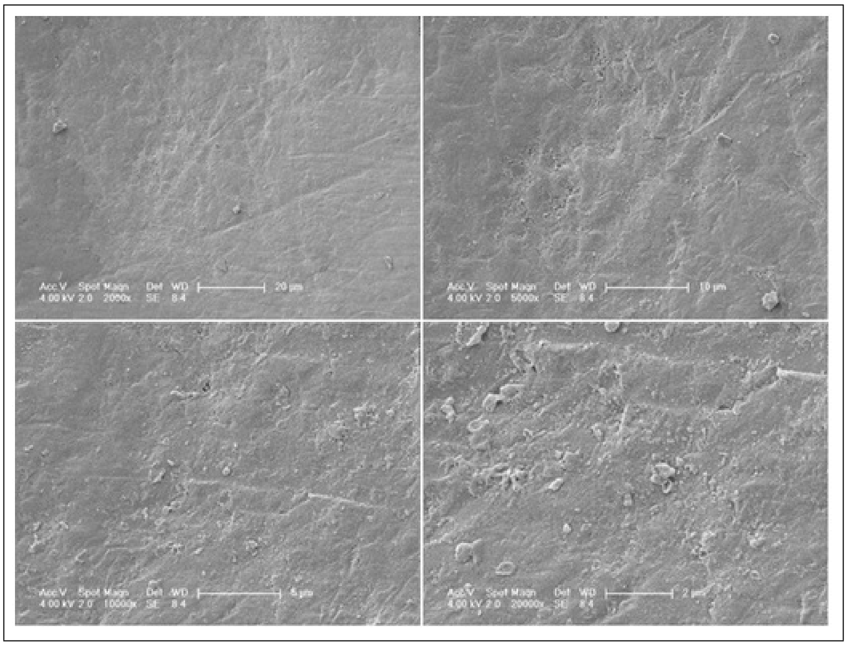

Figure 4.22 250 on 50 off laser, exposure time is 15 second

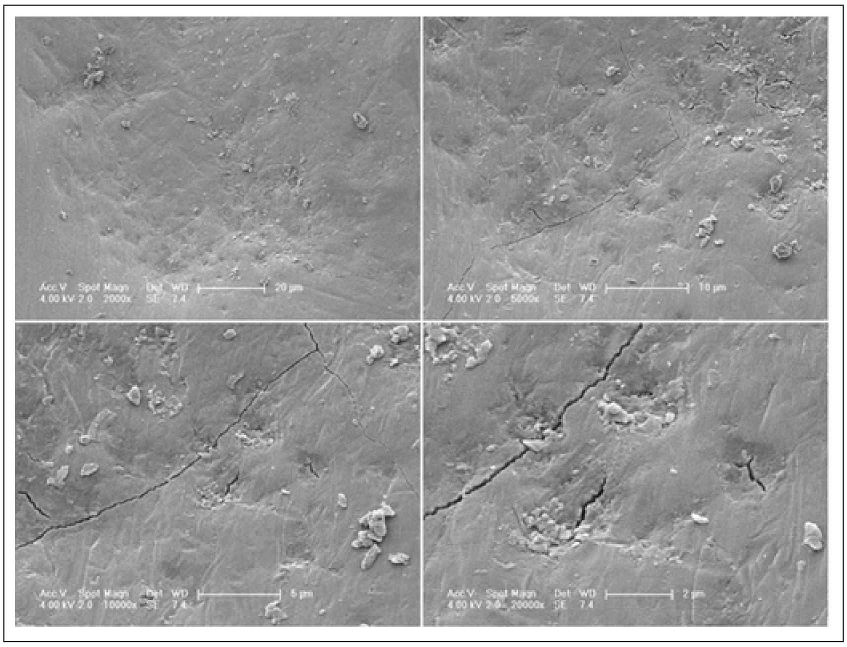

Figure 4.23 3W continuous laser, water temperature is 24 °C , exposure time is 10 second

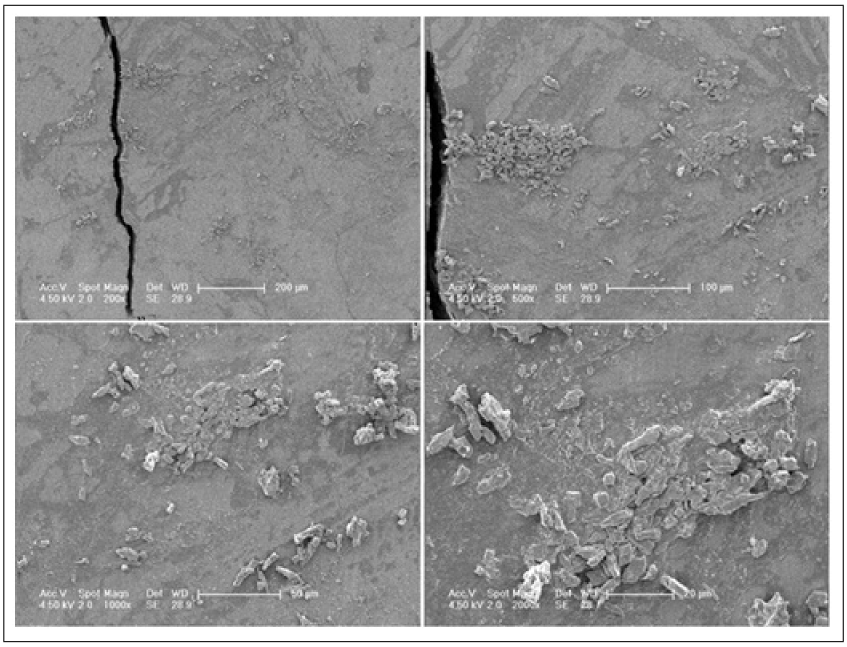

Figure 4.24 3W continuous laser, water temperature is $5,04\,^{\circ}\mathrm{C}$ exposure time is 20 second

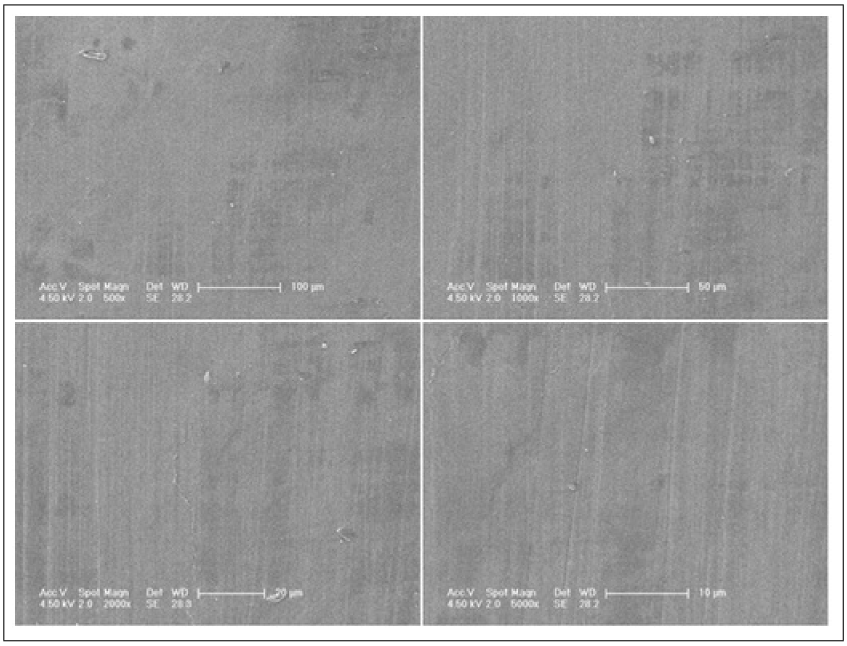

Figure 4.25 3W continuous laser, water temperature is $9,24\,^{\circ}\mathrm{C}$ exposure time is 20 second

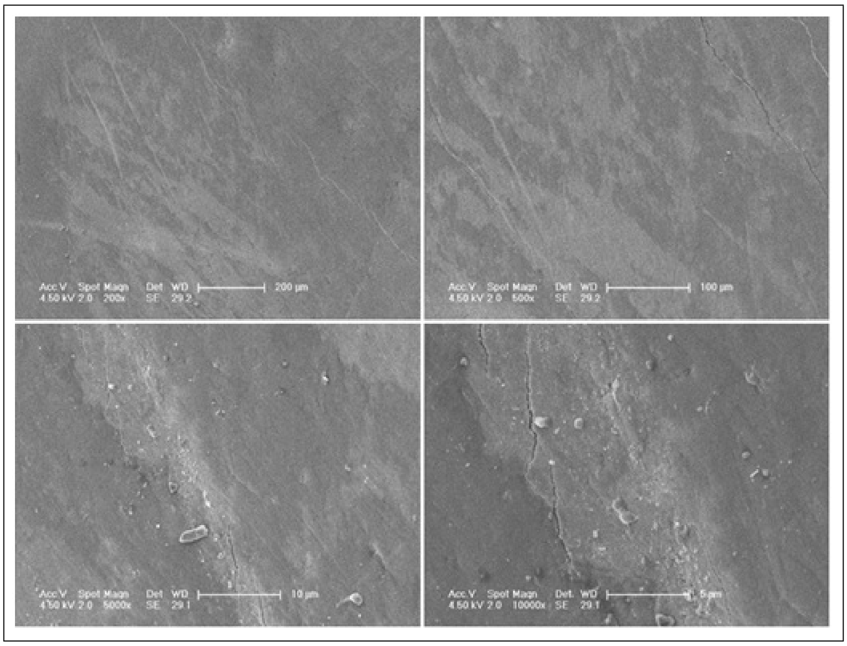

Figure 4.26 200 on 100 off laser with air pressure exposure time is 10 second

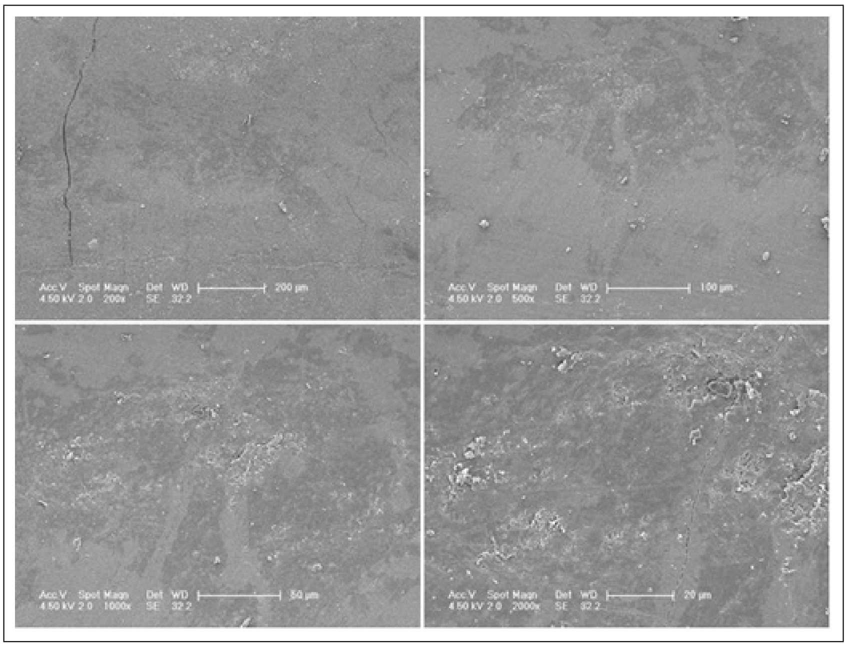

Figure 4.27 2W continuous laser with air pressure exposure time is 5 second.

5. DISCUSSION

Bovine mandibular incisors teeth were used in this study since the availability of human teeth is limited and bovine teeth have similar physical properties to human teeth and do not transmit infectious disease.

In this study, debonding forces from the enamel surfaces which were treated conventional acid etching and laser etching were compared. Thulium Fiber Laser has been used in this experiment.

According to the literature, enamel etching was performed using CO_2, Nd: YAG and Er: YAG lasers in several experiments. According to those experiments, it can be concluded that the dental hard substances can be removed by pulsed Er:YAG laser radiation. Conversely, CO_2 and Nd:YAG lasers are unable to etch effectively but they cause some surface changes in enamel like fissuring, cracking, or melting [11-69-71].

The wavelength of Thulium Fiber Laser which was used for this study (infrared wavelength: 2940 nm) is also between wavelength of Nd:YAG (infrared wavelength: 1064 nm) and ER:YAG (infrared wavelength: 2940 nm).

For the preliminary experiment, to define the laser exposure time for each parameters, time of temperature rise in pulp cavity was measured to limit the cavity temperature to $5,5\,^{\circ}$C. Then, laser was irradiated as point shot from different distances to determine the optimum space between fiber tip and tooth surface. However, the results of debonding forces of brackets attached the etched enamel under this conditions were significantly different ($p<0.05$, student t-test) than debonding force of acid etched groups. This finding suggested choosing the laser parameters independent from the distance and the temperature for the main experiment.

In the main experiments, the enamel surface was scanned with laser as much as bracket base size since it was thought that adequate area could not be etched by point shot lasing. The laser etching was carried out by two different approaches: Lasing without cooling and with cooling.

For the lasing without cooling, the bonding strength of the braces were analyzed: it was observed that the mean bond strength of the laser etched group with 200 on 100 off pulse mode (4.35 ∓ 4.34 N) applied 15s , the mean bond strength (2.46 ∓ 2.33N) of 200 on 100 off pulse mode applied 10 second, the mean bond strength (2.92 ∓ 1.73N) of 250 on 50 off pulse mode applied 15 second, and the mean bond strength (1.48 ∓ 1.08N) of 3W CW mode were significantly different from than the bond strength of acid-etched group ($P<0.05$).

SEM images for lasing without cooling groups showed that 3W CW irradiation in 3 second etched partially but it did not provide sufficient bonding strength. 200 on 100 off pulse modulated irradiation in 10 second and 250 on 50 off irradiation in 15 second caused the surface melting, for 200 on 100 off pulse modulated irradiation in 15 second images showed a lot of flake and several cracks. But during this procedure, excessive temperature rise in pulp was observed.

For the second approach, to increase lasing time and decrease temperature rise in pulp, water spray cooling was applied at the same time with laser irradiation in the first part of cooling experiment. Bonding strength of brackets from water cooled and etched surface were really lower than acid etched values. According to the literature, water can focus the beams of some laser types; it can reduce the power for the other laser types absorbing the energy [4]. SEM images of water cooled surfaces show that there are no morphological changes. Due to our findings from both debonding forces and SEM images of etched surface with cooling water, it can be said that water may absorbed the energy of Thulium laser.

In the second part of cooling experiment, teeth specimens were exposed to laser with air-pressure cooling laterally and directly. Bonding strengths of brackets for two different applications were significantly different from the ones from control group. In SEM images, deep fissures and micro cracks were displayed different from the acid etched surface images. In conclusion, similar results were found to the ones reported by Fraunhofer et al. and Obata et al. In the study conducted by Fraunhofer et al., Nd:YAG laser in 2W continuous, 12 second was used and in the study of Obata et al., CO_2 laser in 3W continuous, 3second was utilized .They found out that the mean bond strength was lower than acid etched control group. In our study, we applied laser for both 2W 5s and 3W 3s and controlled the temperature increase. We also found out that the mean bond strength of laser etched group was lower than acid etched control group [11].

In contrast, in the study by Basaran et al. and DostÃ¡lovÃ¡, they used Er,Cr:YSGG and ER:YAG lasers with lower power and found out that the mean bond strength of the laser etched groups were similar with their acid etched control groups.

6. CONCLUSION AND FUTURE WORK

Experimental conditions were consistent with the protocols for the safety of tooth pulp. Therefore, the laser power levels and the irradiation times were set as low as possible but temperature rise was not prevented in pulp cavity even though with cooling. Besides, mean tensile strength values for the bonded brackets were higher for the acid etched group than for the laser etched group.

In conclusion, laser etching of the enamel with Thulium Fiber laser (1940 nm) did not provide the sufficient bond strength like conventional acid etching technique. Further investigations is necessary to find the suitable laser parameters to improve the etching procedures e.g. to apply ink to the enamel surface or to combine laser with acid

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