School of Dental Science
Dental Materials Unit
Restorative Department

Newcastle University

A study of the interface between Er,Cr:YSGG laser prepared dentine and Glass ionomer cement

Piyanart Ekworapoj

A thesis submitted in fulfilment of the requirement for the degree of DOCTOR OF PHILOSOPHY

I dedicate this work to
my parents in turn and so on.
Piyanart (atom)

Abstract

Lasers have been available for use in dentistry for several years but they are still not widely used in general practice due to expensive technology. The purpose of this research was to study the structure and properties of laser cut tooth surface and the way in which they react with materials to form an effectively bonded and sealed interface. The investigation of laser irradiated dentine showed that the reduction of microhardness by 30-50% compared to baseline dentine. Dentine debris was seen on the surface under SEM microscopy. The interface between the tooth surface prepared by the laser and glass ionomer cement was investigated using confocal microscopy. In addition, the microleakage of cavity prepared by bur and laser was compared.

The adhesion of glass ionomer cement to Er, Cr:YSGG laser cut dentine was measured by means of both microtensile and shear bond strength tests. The mode of failure was assessed by microscopy. The optimal laser parameters were determined. It was found that the bond strength of glass ionomer cement to laser prepared dentine was generally not different from that to conventional cavity preparation. The use of a dentine conditioner had a significant effect on bond strength.

This work suggests that the optimal parameters for use of the Er,Cr:YSGG laser to prepare cavities in dentine are a 4W power setting and a working distance of 1 mm.

Acknowledgements

The author feels grateful to get to know Dr. Sharanbir Sidhu by my teacher in Thailand, Dr. Vidchaya Kanchanavasit. Firstly, I would like to thank him for his introduction to meet my supervisor and his advice about studying in UK. I contacted with Dr. Sidhu via email without seeing her face but I was touched by her generosity via the message from electronic mail. Beyond being taught how to be a good researcher, she is very patient to correct my messy writing. I am lucky enough to have a kind supervisor like Dr. Sidhu. Thank you for being my supervisor. While working as a lecturer in Thailand, I have read a lot about dental materials from many articles both in journals and textbooks. I often came across the name of Prof. McCabe on those publications. His publications inspired me to continue my study at Newcastle University. He is famous professor at the same time as Alan Shearer. His enthusiasm, humour, generosity, and understanding made him an excellent supervisor. I am grateful to have Prof. John McCabe as my supervisor.

I would like to extend huge thanks to my assessors, Prof. Angus Walls and Dr. Paula Waterhouse for their evaluation and direction that has no doubted made me a better researcher. They both are very generous.

I am indebted in their advice and many suggestions throughout my research project.

The author is also grateful to Dr. Mary Anstice, Scientific Affairs Manager of 3M ESPE Dental Product (UK) and Ms. Yukari Endo, R&D Department of GC company (JAPAN), for providing the much-needed materials for this study.

I would also like to thank Dr. Trevor Booth for his guidance with Confocal Microscopy. I am indebted Vivian and Tracy's helps for SEM microscopy.

Special thanks go to Ms Pam Walton for specimen preparation, to Ronnie Allen for his help and kindness in the practical lab (5th floor), to Tom Carrick and Sandra Rusby for the laboratory aspects of this study.

I would like to acknowledge the financial support for my tuition fee and cost of living from the Ministry of University Affairs and Assoc. Dr Tipaporn Wongsurasit (Faculty of Dentistry, Srinakarinwirot University, Thailand) for her supports.

Finally, the author would like to take this opportunity to thank all her friends for their friendly help and suggestions. The author is also greatly indebted to her parents and her family for their support, love and understanding.

Contents

List of Figures

List of Tables

Glossary of Abbreviations

LASER	Light Amplification by Stimulated Emission of Radiation
Nd:YAG	Neodymium:Yttrium-Aluminum-Garnet
Nd:YAP	Neodymium:Yttrium-Aluminum-Perovskite
CO_2	Carbon dioxide
Er:YAG	Erbium:Yttrium-Aluminum-Garnet
Er,Cr:YSGG	Erbium,Chromium:Yttrium-Scandium-Gallium-Garnet
Ho:YAG	Holmium:Yttrium-Aluminum-Garnet
GaAs	Gallium arsenide
AlGaAs	Aluminium gallium arsenide
InGaAlP	Indium Gallium Aluminum Phosphide
XeCl	Xenon chloride
KTP	Potassium titanyl phosphate
GIC	Glass ionomer cement
RMGIC	Resin-modified glass ionomer cement
CLSM	Confocal Laser Scanning Microscopy
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
AFM	Atomic Force Microscopy

CHAPTER 1
INTRODUCTION AND LITERATURE REVIEW

1.1 Introduction

The introduction of lasers in dentistry was inspired by Maiman in 1960 [Maimain, 1960; Ceballos et al., 2001]. The ruby laser was the first system that could ablate dentine and enamel [Stern et al., 1969;Niu et al., 1998]. However, it failed due to the high temperature generated during tooth preparation. The subsequent laser systems, Nd: YAG and CO_2 lasers also showed similar effects on tooth structure [Lin et al., 1999]. The Er:YAG laser was the first laser system used for hard tissue procedures, including caries removal and cavity preparation, and has been approved by the Food and Drug Administration (FDA) in 1997 [Palma Dibb et al., 2002]. During recent years, the Er,Cr:YSGG laser system has been introduced as a multi-purpose laser. The manufacturer claims that this system can minimize the thermal effects on hard tissue and leave no smear layer after preparation [Staninec et al., 2006].

The main problems associated with using a high-speed handpiece for tooth preparation are the noise, vibration and its slow process. These cause the patient discomfort, tooth sensitivity, pain, dental anxiety and dental fear. Most patients require local anaesthesia to reduce pain or

tooth sensitivity during conventional bur preparation. Some patients need tranquilizer drugs (e.g. Valium) or sedation to reduce the anxiety and dental phobia before tooth preparation.

In contrast, laser drilling can reduce vibration and the whining noise generated during tooth preparation due to the non-contacting tip of the laser handpiece. Using lasers instead of rotary drilling is a relatively new alternative for caries removal and cavity preparation.

Over the last few decades, developments in dental adhesives have enabled the dentist to use a variety of materials to bond to dental hard tissue. This has led to improvements in retention and longevity of restorations, as well as, the development of entirely new approaches to treatments. The bonding of materials to tooth structure depends on the specific interactions of adhesive materials at the surface of the prepared tooth. Since virtually all materials have been designed to bond to bur prepared teeth it is uncertain whether the same materials will bond as effectively to laser prepared teeth.

1.2 Basic knowledge of LASER

Based on Eiestein's theory, the photon of different energy is released when the electron at excited stage drops down to the ground stage. If the electron is stimulated to move from the ground stage to excited stage by the energy source, then a second photon will be generated in

the same phase with and propagate in the same direction as - the excitation photon. This is the origin of the term "LASER" which is a short form or acronym of "Light Amplification by Stimulated Emission of Radiation" [Gordon, 1959].

Usually, all the photon particles of the laser light have the same wavelength, when striking a tissue sample the energy may be absorbed, scattered or reflected. This may cause vaporization of liquid within a tissue or carbonization of the tissue. By adjustment of parameters such as the focus point, the pulse mode, frequency, output energy and irradiation time, the laser beam can cut, vapourise or coagulate animal tissue [Miller and Truhe, 1993; Corona et al., 2003].

There are three main components of laser unit: a lasing medium (active medium), energy source and optical resonator [Mercer, 1996; Parker, 2007] as shown in Fig 1.1. The lasing medium can be a solid (e.g. Ruby laser), a liquid (e.g. Dye laser) or a gas (e.g. Argon laser) [Mercer, 1996]. The different types of laser are always named according to the lasing medium. For example, the Nd:YAG laser has a solid lasing medium which is a crystal of Yttrium Aluminum Garnet (YAG), doped with Neodymium (Nd) [Pick, 1993]. When the doping material is changed from Neodymium to Erbium (Er) for the YAG laser, this laser is named the Er:YAG laser [Cavalcantin et al., 2003]. The CO_2 laser has a carbon dioxide gas as the lasing medium [Miller and Truhe, 1993]. In some cases the same lasing medium can emit laser light with more than one

wavelength, the name of this type of laser includes the wavelength of the emission beam together with the lasing medium's name such as 9.3μm CO_2 laser, 9.6μm CO_2 laser, 10.3 μm CO_2 laser, 10.6 μm CO_2 laser [Moshonov et al., 2005]. The atoms or molecules of the lasing medium are required to be excited in order to emit photons of laser light. The energy supply for the laser source may be an electric discharge or a high-powered Xenon flash lamp [Mercer, 1996]. An optical resonator composed of mirrors is used to amplify the light so that the emitted laser light has unique qualities i.e. coherence, collimation and monochromaticity [Mercer, 1996].

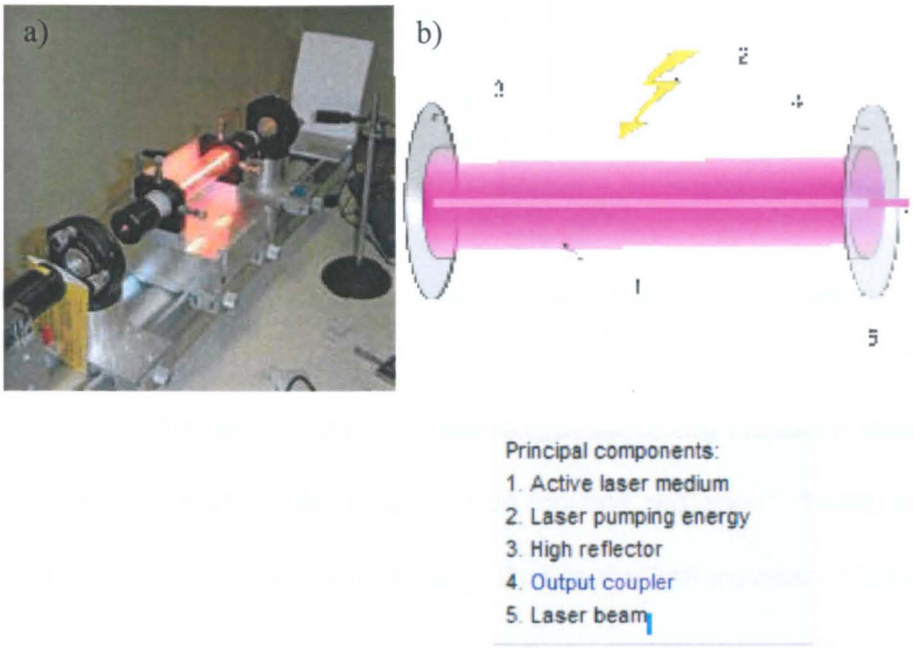


Figure 1.1. a) The prototype of He-Ne laser b) Diagram showing the main component part in laser prototype (retrieved from http//en.wikipedia.org/wiki/laser)

Other parts of a laser unit include the delivery system, cooling system, and control panel. The delivery system may be a quartz fibre optic, a

flexible hollow waveguide, or an articulated arm. The cooling system may be an air or water spray [Parker, 2007].

The primary wavelength of laser radiation for application in dentistry includes invisible ionizing radiation and visible or invisible thermal radiation (Figure 1.2.) [Sulieman, 2005].

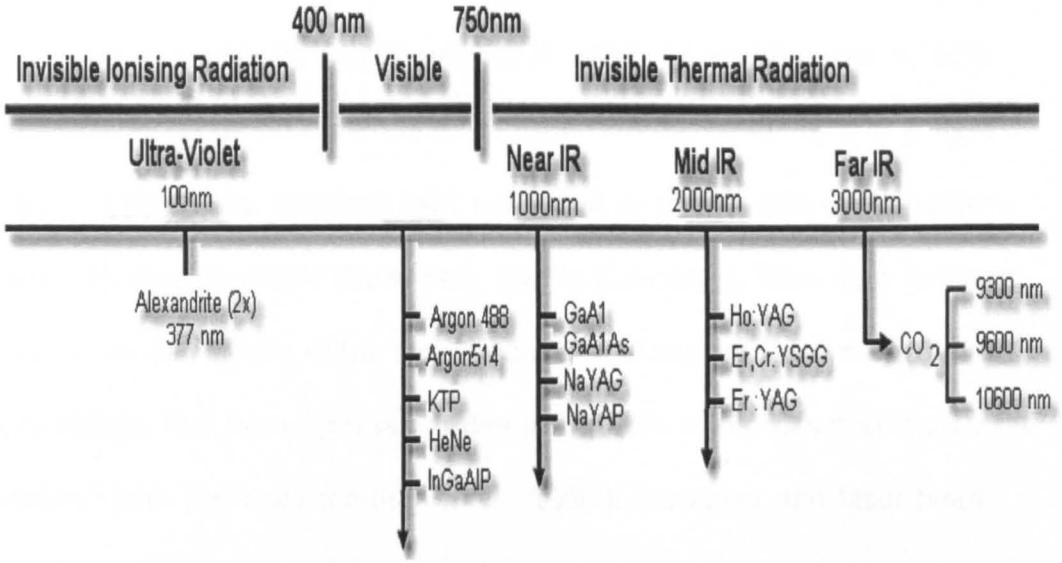


Figure 1.2. The electromagnetic spectrum of laser beam using in dentistry (modified from Sulieman, 2005 and Parker, 2007)

Most dental lasers comprise invisible light since the emission beam is in the infrared portion this is also "non-ionizing radiation". There are also two visible lasers used in dentistry. One is the helium-neon (HeNe) laser used for aiming in conjunction with other invisible lasers. The second is the argon laser, which has a wavelength emission in the blue-green range. More detailed information about the popular dental lasers will be discussed in the following section.

1.2.1 The main characteristic of laser light

Laser light is monochromatic because the photon emitted from a laser source has one particular wavelength [Pick, 1993]. This is opposite to white light which is a combination of all of the different colors in the visible light spectrum [Sulieman, 2005]. This characteristic associated with the energy gap; the transition from excited state to the lower state. The laser is a coherent light source, in which all photons are in both spatial and temporal coherence. In other words, laser light is a light made of physically identical light waves all in phase with one another with identical amplitude [Sulieman, 2005]. Collimated laser light means there is no divergence of the laser beam that is opposite to the light from a flashlight. The laser light is parallel to the axis of the beam since it is emitted from the laser medium [Pick, 1993]. Moreover, the laser beam is often optically focused to a point, at a certain working distance from the tissue being irradiated [Mercer, 1996].

The basic unit of laser energy is the "photon". The relationship of energy with the frequency can be expressed as:

$$E = h\nu \text{ (h=Planck's constant and } \nu = \text{frequency)}$$

Laser light travels through the medium with a constant speed, so the relationship between laser energy and the wavelength of laser light can be defined as:

$$E = hc/\lambda \text{ (derived from the equation: } c=\nu/\lambda)$$

The relationship between laser energy and wavelength is the inverse relationship [Parker, 2007]. Therefore a short wavelength laser (ex. Nd:YAG (λ=1.064 μm)) has a greater laser energy than a long wavelength one (ex.CO_2 (λ=10.6 μm)).

The laser power which is shown on the control panel of laser unit can be defined as the rate at which energy is generated by the laser. For example, a power setting of 1 Watt means that 1 Joule of energy is produced in a second. Therefore, the amount of laser energy delivered depends on the length of time or "pulse duration" that the laser is left on a particular power level [Sulieman, 2005]. The laser energy (Joule) per irradiated area of 1 cm^2 is called "Fluence" (J/cm^2) [Wheeler et al., 2003].

The pulse energy is the relationship between output power level and the pulse duration. For the same output power level, a short pulse must deliver greater pulse energy than for that in a longer pulse. [Sulieman, 2005].

1.2.2 The basic emission modes of dental lasers

There are two basic modes of laser emissions:

1. Continuous wave: in this basic mode the laser emits continuous and constant laser energy as long as the device is activated.

2. Free-running pulse or pulse mode: the laser source emits short bursts of laser photons in the order of a few ten thousands of a second. The

Nd, Ho, Er and Er, Cr:YSGG lasers are examples of instruments operating in this mode. [Coluzzi, 2003]

1.2.3 Laser tissue interaction

The interactions of laser on tissue rely on one of the following interactions: absorption, scattering, transmission, or reflection. The type of the interaction depended on the wavelength of the laser beam, the operation mode of the laser, the amount of energy applied, and tissue characteristic. When the laser light reflects off the surface without penetration or interaction of the light energy with the tissue defined as "Reflection". In case of the laser light is transmitted through the tissue unchanged, as if transparent to the laser beam known as "Transmission". If some laser energy is absorbed into a component of the tissue, this is known as "Absorption". Lastly, when the laser light is scattered within the tissue without producing a noticeable effect on the tissue, this is known as "Scattering" [Sulieman, 2005].

1.2.4 Effect of LASER on soft and hard tissues

Different types of lasers react differently with tissue [Muller and Schaldach, 1989]. It is believed that laser energy causes changes in both the physical and chemical composition of soft and hard tissues. The difference between the effects on soft and hard tissues depends on the amount of laser energy and exposure time (Figure 1.2).

For soft tissue, the desired effect is to cut and coagulate the blood supply at the operative site. Therefore, low-level power lasers are mainly used for soft tissue application. In general, the wavelength of the laser beam used for cutting effectively should be strongly absorbed by that tissue.

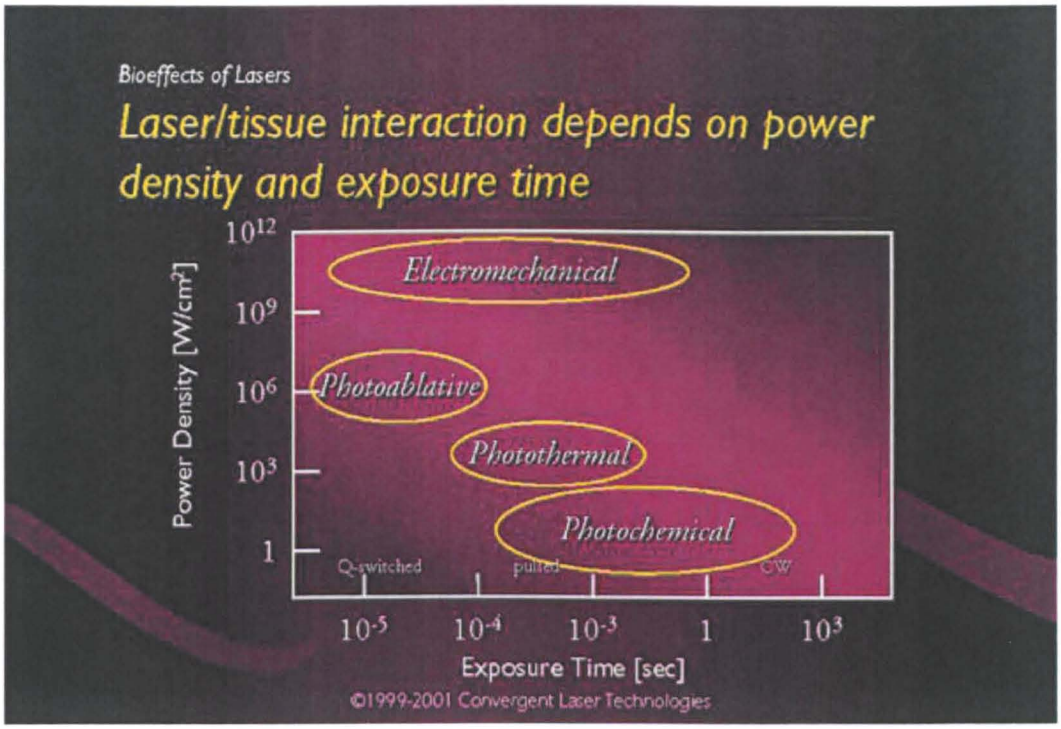


Figure 1.3. The plot between the power density and exposure time which causes the reaction between laser and tissue (retrieved from the website http://www.convergentlaser.com/laser_safety.php?slideno=5)

The photothermal reaction is the primary effect on hard tissue (Fig 1.3). The thermal energy which is converted from the incident laser light plays an important role in causing changes to the hard tissue [Parker, 2007]. The appearance of dental tissue has changed after laser irradiation. For cutting the dental hard tissue, the suitable wavelength of the laser beam

should be maximally absorbed by hydroxyapatite (HA) [Mercer, 1996]. The absorbance peak of HA and water in the tooth tissue is around 2.9 μm (Fig 1.4).

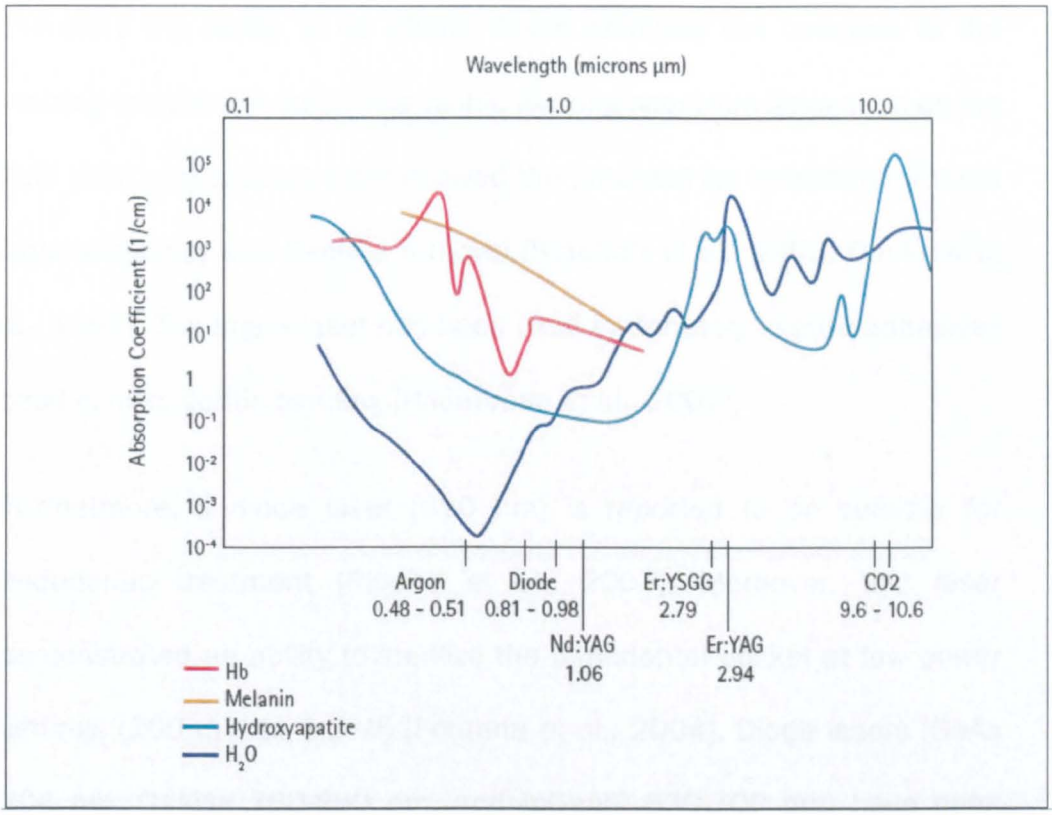


Figure 1.4. The plot between the absorption coefficient of various substance and laser beam using in dentistry (Parker, 2007)

1.2.5 Laser wavelength used in dentistry

A number of investigations have reported various applications of different laser wavelengths in dentistry. The different laser wavelengths have been used in dentistry depending to the interaction between laser light and the biological constituents of dental tissue. In general, the laser machine is not designed for specific application in dentistry. Considering

the surgical area, the proper laser wavelength should have an absorption coefficient closed to the absorption peak of that tissue.

Most of the dental lasers are in the range of non-ionizing wavelength. Therefore the ability to do ablate tissue relatively low compare to the ionizing wavelength laser. The visible ionizing spectrum laser such as the XeCl (308 nm) excimer laser showed the potential for treatment of tooth hypersensitivity and dentine removal [Stabholz et al., 1993; Dankner et al., 1997]. The argon laser has been used in dentistry to cure adhesives used in orthodontic bonding [Hildebrand et al., 2007].

Furthermore, a diode laser (810 nm) is reported to be suitable for endodontic treatment [Ribeiro et al., 2007]. Moreover, this laser demonstrated an ability to sterilize the periodontal pocket at low power settings (200 mW to 1.2 W) [Fontana et al., 2004]. Diode lasers (GaAs 904 nm, GaAlAs 780-890 nm, and InGaAlP 630-700 nm) have been used to treat trauma (bruise tissue). He-Ne (633 nm) laser helps to stimulate and improve regenerative healing. Silvestri et al. (2004) used the long pulse diode laser to prevent third molar agenesis.

In recent years, the other visible laser which low absorptive by dental hard tissue such as a Cu-HyBrID laser (copper laser with Hydrogen Bromide In Discharge with wavelength of 510 and 578 nm) revealed its ability to ablate human enamel [Miyakawa et al., 2007].

The long wavelength with low energy laser like the CO_2 laser demonstrated efficacy in treating peri-implantitis concomitant with soft tissue resection [Deppe et al., 2007]. In this aspect, the CO_2 laser showed the bactericidal effect to the infectious tissue. Low-level laser therapy (LLLT) has been presented with the idea of using the laser light at power levels below that needed to cause tissue change.

1.3 Laser wavelength used for hard tissue application

With the advances in laser dentistry, a wide variety of laser wavelength was available today for dental practitioners. The dental lasers can be used into two areas: soft - and hard - tissue applications [Niu et al., 1998]. The early laser system ruby laser showed the possible used in dentistry specifically to reduce the subsurface demineralization [Stern et al., 1964]. After that, various kinds of laser lights of different wavelengths have been developed and applied for both hard and soft tissue application. Early most of clinicians have used lasers primarily for soft tissue procedure. They used for gingival contouring, gingival troughing which is quick and effective alternative to packing retraction cord with the bloodless gingival sulcus [Wigdor et al., 1995]. Subsequently, the dental lasers have been used mostly for the soft tissue cutting e.g. excisional or incisional surgery (fibroma removal, frenectomy), sulcular debridement and homeostasis. The hard tissue procedures include caries removal, tooth preparation for direct or indirect restoration (inlay/onlay), dentine and enamel etching,

endodontics procedures, and bone cutting, shaving contouring or resection.

The hard tissue application appeared to be caries removal and cavity preparation. With the expansion of the laser application to remove caries and to prepare the cavity for restoration, the investigations on the effect of various laser wavelengths such as CO_2, Nd:YAG and Er:YAG has dramatically increased [Kohara et al., 2002].

The erbium laser systems have been shown to be the suitable candidate for caries removal, cavity preparation, and root canal debridement [van As G et al., 2004; Radatti et al., 2006]. Erbium laser emitted the light at a wavelength in the region of 3 μm which has energy interaction with water at the tissue interface [Botta et al., 2008]. There are now two primary Erbium laser categories available for hard tissue procedures – the Er:YAG (2.94 μm) and the ER,CR:YSGG laser (2.78 μm). The good absorption of the radiation of water is exploited at this wavelength [Apel et al., 2002]. Therefore, these lasers have been indicated for cavity preparation. However, it was reported about their ability to remove oral papilloma [Boj et al., 2007] because the erbium laser light is well absorbed by water inside the tissue. Cutting soft tissue with this laser also provide the precise incisions, bloodless surgical site and perfect hemostasis. Besides the Nd:YAG and CO_2 laser the Er:YAG laser has also been used for periodontal treatment [Ishikawa et al., 2003].

For cavity preparation, pulp damage is a major concern for the clinician when using the laser instead of a high-speed handpiece. The use of the ruby laser was reported to cause pulp damage by heat production during the laser irradiation [Adrian et al., 1971; Wigdor et al., 1995]. However, the Er,Cr:YSGG laser, show a low temperature rise in the pulp chamber because the water spray emitted together with the laser light helps to cool down the heat produced by the laser [Rizoiu et al.,1998; Hadley et al., 2000; Geraldo-Martins et al., 2005]. Earlier investigations demonstrated that the laser beam caused pulpal damage due to the energy output and exposure time [Jeffery et al., 1990; Keller et al., 1991; Allen 1993]. The low-grade intensity of the laser beam has been suggested to reduce the thermal effect to pulp tissue [Serebro et al., 1987]. Therefore, the laser can be considered for replacing or supplementation of the dental drill used under the appropriate energy output and exposure time. However, more thorough investigations on different aspects of dental lasers are essential before they are accepted by patients and dental practitioners. The hard tissue procedure that remains a challenge is crown preparations.

This topic aims to present the potential use of the various wavelength lasers for hard tissue application.

1.3.1 CO_2 laser (Carbon dioxide laser)

The CO_2 laser was one of the earliest gas lasers. This invisible laser with a wavelength of 10.6 μm (10600 nm) was developed by Patel in 1964 and is still used today mainly for soft tissue surgery.

The distinct characteristic of the CO_2 laser is its affinity for wet tissue. The laser can be absorbed by a wet area but there is limited penetration depth (only 0.2 to 0.3 mm). There is no scattering or reflection. Furthermore, tissue pigmentation does not affect the performance of this laser. Thus, it is well suited for oral mucosa, which is pink in colour. The CO_2 laser has been successful in soft tissue surgery such as gingivectomy, frenectomy, and the removal of benign and malignant lesions including excisional and incisional biopsies. Compared to the other lasers for oral use, the CO_2 laser is reported to be the fastest laser for soft tissue removal [Miller and Truhe, 1993]. The CO_2 laser unit with handpiece is shown in Fig 1.5a. The laser beam is emitted through the glass tube containing the CO_2 gas (Fig 1.5b).

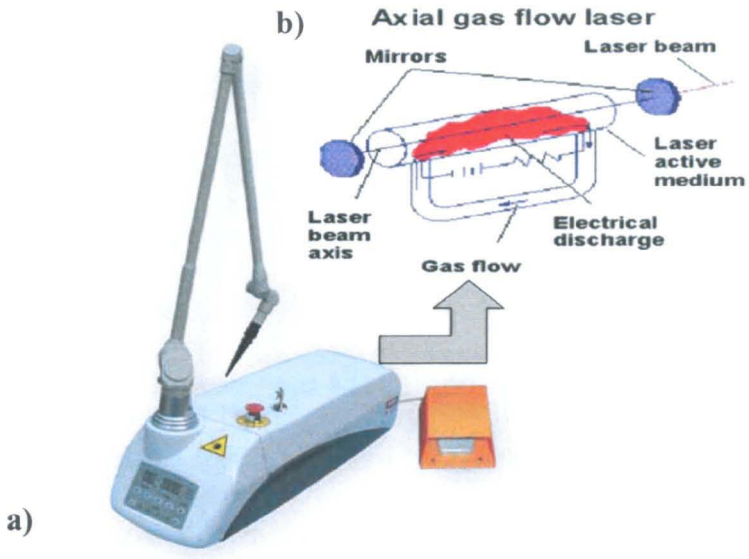


Figure 1.5. a) CO_2 laser unit b) The diagram showing the lasing medium (CO_2 gas), energy resource (Electrical discharge) and the optical resonator (two mirrors) retrieved from http://win.mofcom.gov.cn/en/upphoto/318/10412006510.jpg)

The potential application of the CO_2 laser for dental hard tissues has been studied extensively by researchers [Mullejans et al., 2002; Gonzalez et al., 1999]. The use of this laser for mineralized tissue was first experimented in endodontic situations [Miller and Truhe, 1993]. Later, researchers in France began to use the CO_2 laser with tooth tissue [Melcer et al., 1981; Serebro et al., 1987; Pick, 1993]. When the tooth surface was irradiated with a continuous wave CO_2 laser, the surface was burnt and heat was transferred to the pulp chamber [Wigdor et al., 1995].

Other studies revealed that a pulsed CO_2 laser could elevate the temperature at the tooth surface by up to 1000 °C, which is sufficient to fuse and melt enamel crystals [Featherstone and Nelson, 1987; Wigdor

et al., 1995]. The report by McCormack (1995) similarly showed crystal fusion on CO_2 irradiated enamel surface at low fluences (5 J/cm^2 per pulse). Furthermore, there was no crystal fusion occurring at longer pulses and constant fluence conditions. Malmstrom et al. (2001) experimented with CO_2 lasers for hard tissue surgery to investigate the potential detrimental effect on hard tissue and temperature rise in the pulp chamber (*in vitro*). The SEM and polarized light observations showed the deepest crater formation in the highest exposure time and pulses per second.

Further investigation showed that the CO_2 laser could inhibit the caries lesion up to 85 % which is comparable to a daily application of a sodium fluoride dentifrice [Hadley et al., 2000]. The drawback of the earlier CO_2 laser is the beam carrier. The old CO_2 laser used an articulated arm with an internal mirror and a knuckle joint instead of a fibreoptic carrier. The handpiece was large and the focal distance was considerable. Later, developments for the CO_2 laser include a new flexible fiber that permits convenient access to any part within the mouth [Miller and Truhe, 1993].

The CO_2 laser has been approved by the Food and Drug Administration (FDA) in the United States for soft tissue application in 1976 [Wigdor et al., 1995], as it is beneficial for soft tissue surgery more than hard tissue cutting. There is a great concern about pulpal injury and chars on the CO_2 laser irradiated tooth surface when using inappropriate exposure

energy output and time. The problem of excessive heat production by this laser should be addressed.

In conclusion, the CO_2 laser is acceptable for soft tissue surgery more than hard tissue cutting. This laser needs to be developed further for cavity preparation. However, it can be used in preventative dentistry to enhance the acid resistance of dental hard tissue [Hossain et al., 1999].

1.3.2 Nd:YAG laser (Neodymium: Yttrium-Aluminum-Garnet laser)

At the same time as the development of the CO_2 laser, Geusic improved the Nd:YAG laser in 1964 [Geusic et al., 1964; Pick, 1993]. Meyer applied this laser for use in dentistry in 1984 [Myers et al., 1985]. This laser is composed of YAG, which is a crystal of Yttrium Aluminum Garnet, doped with Neodymium (Nd) [Coluzzi, 2003]. Similar to the CO_2 laser, the Nd:YAG laser cannot be seen by the naked eye because the laser beam is in the infrared range (wavelength of laser light = 1.06 μm). The invisible Nd:YAG beam is indicated by means of a red helium neon laser. Additionally, the Nd:YAG laser is delivered through a fibre optic carrier which makes it practical for use within the oral cavity. In general, it functions in a pulsed mode. Compared to the CO_2 laser, this laser can penetrate through wet tissue more deeply. As a consequence of the affinity for pigmented tissue, the tooth tissue (white in colour) should be marked with black dye before using Nd:YAG irradiation [Pick, 1993]. The

Nd:YAG laser unit can be a combination laser unit of the Nd:YAG and Er:YAG laser system (Fig 1.6.).

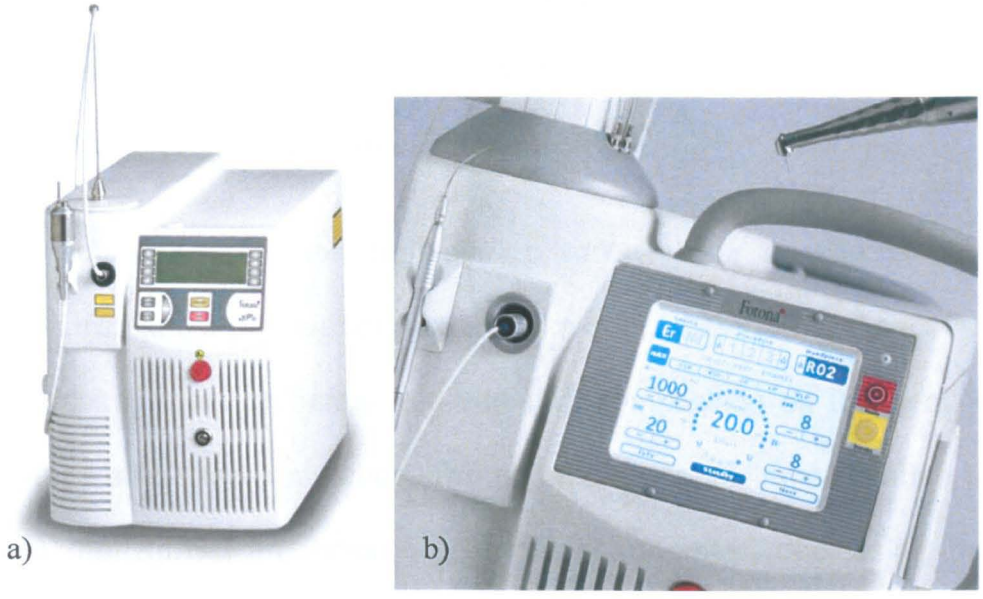


Figure 1.6. (A) The Nd:YAG laser unit (B) Control panel showed two mode system; Nd:YAG or Er:YAG (retrieved from the website www. Medicaservice.org)

Nd:YAG laser irradiation on enamel surface modified the permselectivity (A term used to define the preferential permeation of certain ion species through ion-exchange membrane, www.wikipedia.org) of enamel membrane and microhardness [Marquez et al., 1993]. Knoop microhardness increased after Nd:YAG irradiation which indicated the increase in mineralization or the reorganization of apatite crystal [Turkmen et al., 2006; Gaspirc and Skaleric, 2001]. The permselectivity of enamel membrane became more positive because of the loss of water, carbonate and organic substance, which are responsible for the permeability [Marquez et al., 1993]. These may enhance the acid resistance of the enamel surface.

Nd:YAG irradiation showed the reduction in dentine permeability which is of benefit to endodontic and preventive dentistry [Lee et al., 2002]. A similar report from Ciaramicoli (2003) showed that the Nd:YAG laser was effective in reducing the cervical dentine hypersensitivity by causing partial or total obliteration of dentinal tubules.

The adhesion of composite resin to Nd:YAG laser dentine is poor compared to bur preparation [Ariyaratnam et al., 1999]. This is because of a change in morphology and chemical composition on the dentine surface after laser irradiation. The surface susceptibility of dentine to acid after Nd:YAG irradiation may be reduced therefore decreasing the ability of acid etchant to open dentinal tubules and expose the collagen network [Re et al.,2004].

In conclusion, the Nd:YAG laser is mainly used for soft tissue surgery such as frenectomy, vestibuloplasty, gingivectomy, gingivoplasty, removal of benign tumors and other lesions, periodontal surgery, etc [Re et al., 2004]. The application for hard tissue treatments includes pulpal analgesia, dentinal densitization, enamel etching, caries removal and prevention, etc [Wigdor et al., 1995; Re et al., 2004].

1.3.3 Argon Laser

There are two visible wavelengths of the argon laser; one is 488 nm (blue colour) and the other is 514 nm (blue-green colour). The 488 nm argon laser is useful for activation of light-cured composite resin. The

514 nm argon laser is beneficial for blood coagulation in the case of soft tissue surgery because it interacts well with haemoglobin and melanin [Coluzzi, 2000, Anderson et al., 2002]. Therefore, the argon laser has been used both for soft-tissue surgery and for curing light-cured composite resin. There have been reports that the 488 nm argon laser takes less time to cure the composite resin and enhances the physical properties of composite restoration [Fleming and Maillet, 1999; Powell and Blankenau, 2000]. Hickel et al. (2000) mentioned that the greater depth of cure and degree of polymerization could be obtained from this laser. The 514 nm argon laser provided excellent haemostatic [Kelsey et al., 1991] during surgical operation.

A few studies have focused on its application in hard tissue surgery. Anderson et al. (2002) studied the effect of argon laser irradiation on enamel decalcification. They found that the argon laser was effective in reducing enamel decalcification during orthodontic treatment. Additionally, the use of the argon laser at low energy density can enhance the fluoride retention of the enamel surface [Nammour et al., 2003]. To summarize, the argon laser is used for curing light activated restorative material and also for caries prevention.

1.3.4 Er:YAG laser (Erbium: Yttrium-Aluminum-Garnet laser)

A crystal of Yttrium Aluminium Garnet (YAG) doped with Erbium (Er) emits laser light at a wavelength of 2.94 μm which is in the mid-infrared spectrum. This laser is called the Er:YAG laser and is in the category of

solid state and pulsed lasers [Burkes et al., 1992]. The Er:YAG laser was developed by Zharikov et al. in 1975 for caries treatment. The laser equipment is supplied as a mobile unit with the handpiece and sapphire tip as shown in Fig 1.7. This laser overcame the shortcomings of the ruby laser, CO_2 laser and Nd:YAG laser by causing less pulp tissue damage and heat generation. Moreover the carbonization layer and crack formation was not observed on the dentine surface [Shigetani et al., 2002].

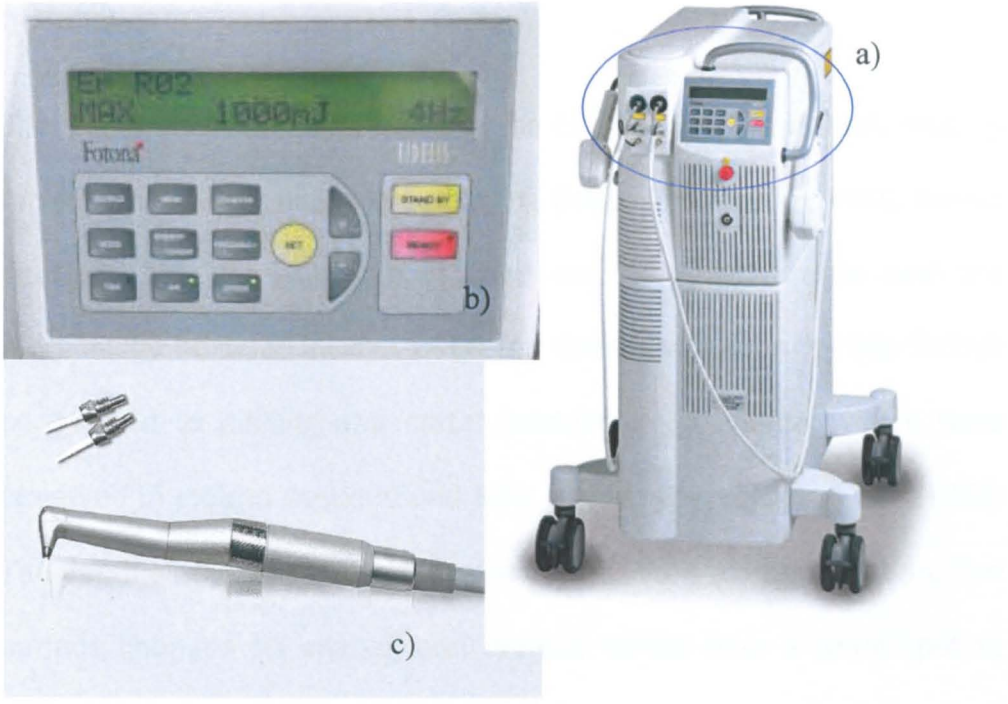


Figure 1.7. a) Er:YAG laser unit b) The laser tip on the handpiece with water spray cooling system c) the control panel showed the energy and repetition rate (retrieved from the website www.ondanet.it/forb/azienda_04.html May 2007)

The emitted wavelength of the Er:YAG laser is close to the major water absorption band. Therefore, the energy generated from this laser is well

absorbed by water within the hydroxyapatite in the tooth and this causes micro-explosion followed by a small portion of tissue being ablated [Burkes et al., 1992]. This is believed to be the major mechanism for dental hard tissue removal. Compared to the other laser systems, the Er:YAG can cut enamel and dentine more effectively [Hibst and Keller, 1989, 1998 ;Ceballos et al., 2002]. In addition, an *in vitro* study disclosed that the Er:YAG laser can remove caries without significant thermal side affects [Miller and Truhe, 1993]. However, the Er:YAG laser does not affect certain restorative materials such as composite filling material.

The erbium doped laser especially the Er:YAG laser was widely studied for the potential of hard tissue cutting [Hibst and Keller, 1989]. Earlier studies on this laser were focused on the ablation rate and the morphology of laser irradiated tissue. Laser irradiation by the Er:YAG contributed to melting and crater production on enamel, which were observed in ground sections and SEM microscopy [Hoke et al., 1990]. The dentine ablation by this laser was better than enamel ablation. The surface changes on enamel and dentine varied from a white spot to charring, fusion, melting, recrystallization, bubble like inclusions, numerous pores, flaking, cracking to crater formation [Serebro et al., 1987; Aminzadeh et al., 1999; Corona et al., 2003; Malmstrom et al., 2001].

Burkes et al. (1992) investigated the enamel ablation by the Er:YAG laser in wet or dry conditions. They tried to assess the thermal side effects during laser preparation using water mist. They found that the available water in enamel had been vapourized and a small amount of enamel ablation occurred, no additional water was available for absorbing energy and no further removal of enamel produced. Not surprisingly, enamel ablation occurred with difficulty, exposing dentine that was ablated even more rapidly. This can be explained by the fact that the water content within dentine is more than the water content in enamel. They concluded that the application of a fine water mist during tooth structure ablation by the Er:YAG laser resulted in a lower temperature rise than using the laser on dry teeth.

The factors affecting the ablation rate when using the Er:YAG have been considered. It has been reported that the ablation rate of the Er:YAG on enamel and dentine is dependent on various factors such as energy per pulse, water spray rate, and frequency (Hz). Increase in energy per pulse results in an increase in ablation rate. Additionally, the ablation rate at low and high energy increases when using the water spray [Kim et al., 2003].

1.3.5 Er,Cr: YSGG laser (Erbium, Chromium: Yttrium-Scandium-Gallium-Garnet laser)

The Er, Cr: YSGG laser uses an Yttrium Scandium Gallium Garnet (YSGG) crystal doped with Chromium and Erbium (Cr, Er) to energize the water

for cutting hard tissue. The emitted wavelength of this laser beam is 2.78 μm [Eversole et al., 1997]. The Er, Cr: YSGG laser delivers the pulsed beam through the contra-angle of the flexible fibre-optic cable with a sapphire tip which is bathed in a mixture of air and water vapour (Figure 1.7b) [Eversole et al., 1997; Hadley et al., 2000].

The Er,Cr:YSGG laser unit is displayed in Fig 1.8a. The term "hydrokinetic system" is used and refers to the manner in which the water microdroplets absorb the laser energy and become the cutting tool. This was believed to be partially responsible for the hard tissue cutting effect [Lin et al., 1999]. In other words, hydrokinetics is the process in which the optimized absorption of Er, Cr: YSGG laser energy atomizes water particles and results in energized microparticles of water capable of cutting tissue precisely [Goldstein, 1999].

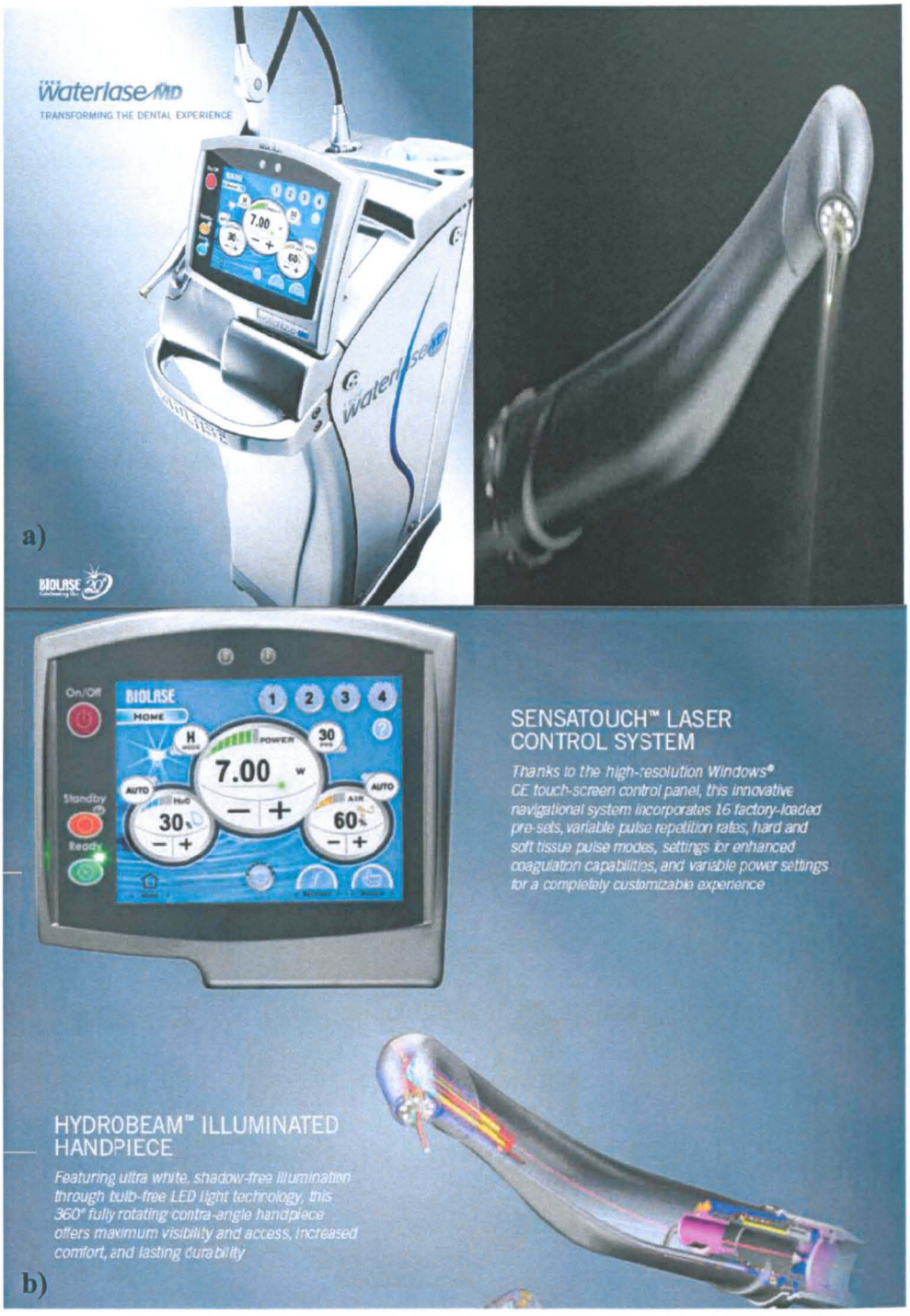


Figure 1.8. a) Er,Cr:YSGG laser or hydrokinetic laser system and laser tip on the handpiece with water spray cooling b) the control panel with hydrobeam illuminated handpiece (retrieved from www.biolase.com).

A laser-powered hydrokinetic system, in the form of the Er, Cr:YSGG laser, has emerged as an innovative laser device for multipurpose. Most published papers about the Er, Cr:YSGG laser have been based on the machine from Biolase Technology Inc.[Eversole et al., 1997;Goldstein, 1999; Lin et al., 1999; Hadley et al., 2000].

Biolase Technology claims that the Er, Cr: YSGG laser has lower thermal effects than the other dental lasers. Eversole et al. (1997) performed animal tests to determine the pulpal response to cavity preparation by Er, Cr:YSGG laser. They reported that no histopathologic evidence of inflammatory cell infiltration occurred in the short-term in teeth prepared by this laser. Intentional pulp exposure by laser did not induce inflammatory cell infiltration because of a hyaline barrier that formed at the laser/tissue interface, which is known as the plasma effect. Hence, it can be postulated that lasers can be used for direct pulp capping. They also suggested that controlled clinical trials using this laser for tooth preparation should be explored to determine whether local anesthetic would be required in conjunction with use of this laser.

Summary

- The CO_2 laser is feasible for soft tissue cutting more than hard tissue cutting.
- Nd:YAG lasers help to seal partial or total the dentinal tube which is benefit for treatment of dentine hypersensitivity especial

cervical abrasion lesion. Thus, they are commonly used for surface treatment and in preventive dentistry to reduce tooth hypersensitivity.

- Erbium lasers (Er:YAG and Er,Cr:YSGG laser) show a capacity for hard tissue cutting because the laser wavelength is close to the absorption peak of water inside the hard tissue. The water plays an important role in tissue ablation.

1.4 Adhesion to laser cut tooth: microleakage and bond strength

Most of infrared lasers have been advocated for hard tissue procedures such as removal of carious tissue, dentine/enamel surface modification (laser etching) and cavity preparation for restoration [Neev et al., 1996, Hossain et al., 2003]. Specifically, the erbium laser has been considered as a potential to substitute high speed air turbine since its high absorption coefficient allows for a large energy/volume deposition and minimal heat diffusion, which improves ablation rates with minimal collateral thermal damage [Colucci et al., 2008, Neev et al., 1996, Fried et al., 1996]. The erbium:yttrium aluminum garnet (Er:YAG) and erbium chromium:yttrium scandium gallium garnet (Er,Cr:YSGG) were two laser devices approved by the Food and Drug Administration (FDA) for use on intraoral hard tissue in 1997 and 2002 respectively [Harashima et al., 2005].

Amalgam restorations were the most affected by using lasers for cavity preparation. It can be explained by the fact that a cavity cut by the laser does not provide sharp margins, which are clearly identifiable, and irregular walls influence the adaptation of amalgam to the prepared cavity. In contrast, a cavity prepared by high-speed bur gave well-defined walls and angles, which meets the requirement for amalgam restoration. Therefore, most of investigations on microleakage and adhesion to laser cut tooth have performed in composite resin restoration.

This section focused on the influence of Erbium laser system on microleakage and adhesion of restorative dental materials.

1.4.1. Microleakage and Bond strength to Er:YAG laser cut tooth

Over the last decade, the use of the Er:YAG laser for cavity preparation and caries removal has increased. Several studies investigated the effect of using this laser to prepare the cavities or modify the tooth for composite restoration. Microleakage, marginal integrity, microtensile strength and patient perception were determined when using this laser [Pelagalli et al., 1997; Stiesch-Scholz and Hannig, 2000; Roebuck et al., 2000; Ceballos et al, 2001; 2002; Shigetani et al., 2002; Kohara et al., 2002; De Munck et al., 2002, Corona et al., 2003].

Studies on using the Er:YAG laser to modified dentine surface as same as acid etching have been reported by Ceballos et al. (2001). Based on

other studies, the lased dentine is rougher than bur prepared dentine [Kohara et al., 2002; Armengol et al. 2003]. Therefore, this morphology may enhance micromechanical retention in the same way as acid etching. Furthermore, lased dentine offers a rough dentine surface without demineralization, opened dentinal tubules without the production of a smear layer and provides sterilization [Ceballos et al., 2001]. For the microleakage evaluation, they concluded that laser irradiation of enamel was not valid alternative to acid-etching pretreatment for resin composite adhesion although no differences were found for microleakage on gingival wall of class V composite resin restoration.

For the bonding to ER-YAG laser treated dentine, laser irradiation adversely affects adhesion to dentine and does not constitute an alternative to acid etching [Ceballos et al., 2002]. The rationale for poorer adhesion to Er:YAG laser etching tooth compared to conventional acid etching procedure might be from the laser modified layer. The TEM micrograph showed that the superficial part of the laser modified layer was composed of a scaly surface [Aoki et al., 1998]. Along the basal part of this layer, remnant denatured collagen fibrils were fuse and poorly attached to the underlying dentine substrate. Therefore the interfibrillar spaces were lacking and probably restricted the resin diffusion into the subsurface of intertubular dentine, resulting in lower shear bond strength [Ceballos et al., 2002].

A study on microleakage of class V composite restoration prepared by the Er:YAG laser was investigated by Niu et al. (1998). This study was different from studies of Ceballos et al. (2001). As a laser etching, the working distance of a laser device in Ceballos's study was maintained at 20 mm apart of the tooth surface. They scored the microleakage from individual walls of class V cavity while Niu's study used the average microleakage score. Considering on occlusal margin of class V restoration, acid etching treated cavity showed the least dye penetration. On the gingival wall, statistical analysis did not revealed the differences in microleakage between acid etching and laser etching. Conversely, the results from Niu and coworker concluded that the microleakage in class V cavities prepared by either the Er:YAG laser or air turbine was no different [Niu et al., 1998].

Shigetani et al. (2002) reported that Cl V cavities prepared by the Er:YAG revealed higher marginal leakage in enamel than those prepared by bur. In the dentine, there was no significant differences between cavities prepared by Er:YAG laser or the air turbine handpiece. However, they conclude that cavities prepared using the laser system showed a higher degree of leakage than those prepared by air turbine hand piece [Shigetani et al., 2002]. The crack from laser irradiation on enamel surface may cause poor adaptation of composite filling especially at the enamel margin of class V cavities. Similar to Ceballos et al., the reason for higher degree of leakage in dentine of cavities prepared by Er:YAG

laser for this study may be from the degeneration layer of tooth substance after laser irradiation. By Energy Dispersive Spectroscopy analysis confirmed that dentine degeneration after laser irradiation [Takano, 1997].

Most patients feel more comfortable when using Er:YAG laser to do cavity preparation according to report from Pelagalli et al. (1997). Moreover, the local anesthesia was not needed. This may be beneficial to pediatric patients. In primary human teeth, the degree of microleakage in class V cavities prepared by the Er:YAG laser was less than those of bur preparation [Kohara et al., 2002]. However, the operation time required to finish class V preparation by this laser was three to five times longer than that of the bur treatment. This may be not convenient for child patient.

Similar to previous investigation by Stiesch-Scholz and Hannig (2000), the good adaptation on enamel margin of class V cavity in primary teeth may be from roughening the enamel surface. Therefore, the surface area was enlarged and increase microretentions for composite restoration. In primary teeth, the enamel surface after laser irradiation was similar to the sharply defined etching patterns after conventional acid etching. This is contrary to the laser etching in permanent teeth which resulting poor adaptation and high degree of leakage. The divergent observations after laser irradiation are probably due to the different micromophological

structure of enamel in primary teeth compared to permanent teeth [Silverstone, 1970].

It should be addressed that only one adhesive system and one composite resin was tested at certain laser parameter. The nature of composite and adhesive system also shows the effect on microleakage [Ceballos et al., 2001]. The degree of marginal leakage after filling class V cavities prepared by Er:YAG laser with amalgam, packable composite resin and resin-modified glass ionomer cement higher than those conventionally prepared by air turbine [Corana et al., 2003]. The laser preparation failed to provide the precise and identified margin for amalgam restoration. For the composite restoration, the quality of the marginal seal in non-acid-etching Er:YAG prepared class V cavities depends on the type of resin composite and associated with three-step adhesive system [Delme et al., 2005]. Glass ionomer cements are more sensitive to changes in hard tissue composition than composite resin, which is based on mechanical adhesion [Liberman et al., 1990]. As laser irradiation produced a disorganized, indiscriminate destruction of organic and inorganic components, therefore the adhesion of glass ionomer cement would be compromised [Corona et al., 2003].

Laser parameter also may influence the marginal integrity of dental restoration to laser irradiated tooth. An increase in pulse energy results in deeper crater pattern in tooth surface; this affected the adaptation of the restorative materials to cavity walls [Roeback et al., 2000].

In conclusion, the microleakage of Er:YAG laser treated surfaces to composite is highly variable depend on the choice of laser parameter, inherent characteristics of restorative materials, the type of tested teeth (Permanent or Primary human teeth or bovine teeth) and the use of laser (as etching or preparation).

In term of adhesion to Er:YAG treated teeth, as same as microleakage studies, several investigations were still conflicting [De Moor and Delme, 2006]. In addition, testing method (Tensile or shear bond strength) may affect the value of bond strength to laser treated tooth.

Dunn et al (2005) reported the shear bond strength of composite resin (Z250 and Scotchbond Multipurpose, 3M ESPE, USA) to dentine and enamel etching by laser was significantly lower than that of conventional rotary preparation and acid etching. This study agreed with previous studies from Ceballos et al. (2003). The Er:YAG laser irradiation caused fused collagen fibrils together on the dentine surface resulting in a lack of interfibrillar space for resin penetration. Therefore the shear bond strength of composite resin to laser treated tooth surface was inferior. Also, the evidence from SEM micrograph showed the separation of resin adhesive and laser ablated dentine surface [Dunn et al., 2005]. Laser ablation produced the fissuring and a blending of the distinctive etch pattern. This blending effect likely to prevent the penetration of resin into enamel surface resulting in lower enamel bond strength values [Dunn et al., 2005].

On contrary, Celik et al (2006) concluded their study that Er:YAG laser irradiation did not adversely affect the shear bond strength of Single Bond 2 and Clearfil Protect Bond to dentine, whereas it increased the shear bond strength of Clearfil tri-S Bond. The 7th generation of adhesive system (Clearfil tri-S Bond) achieved bonding to tooth structure by nano interaction zone. The monomer with nanofilled particle may penetrate into the restricted space of fused collagen fibril of destructive dentine layer. Consequently, the shear bond strength of composite to Er:YAG laser prepared dentine when using this adhesive system is higher than those of conventional rotary instrument [Celik et al.,2006]. The study form Bertrand et al (2006) showed the same result as the study of Celik et al (2006). The Er:YAG laser prepared cavities had improved shear bond strength of single-component adhesive system. Although an Er:YAG laser might eliminate the need for acid-etching pretreatment, this step must be maintained for the whole cavity regard of the microleakage values [Bertrand et al., 2006].

Kameyama et al (2000) surveyed the tensile bond strength of 4-META/MMA-TBB resin to bovine dentine prepared by Er:YAG laser. They varied the dentine conditioner applied on the laser treated surface compared to bur cut dentine. The result revealed that ferric chloride containing dentine conditioner was not significantly affecting on bond strength to laser-irradiated dentine. The low bond strength may be from the increase in acid resistance of peritubular dentine after laser

irradiation resulting in diminishing of acid conditioner effect [Kataumi et al., 1998].

1.4.2 Microleakage and Bond strength to Er,Cr:YSGG laser cut tooth

Similar to the Er:YAG laser, researchers investigated the possibility of using the Er,Cr:YSGG laser instead of using acid etching because the tooth surface after laser irradiation is irregular, fissured, and clean without a smear layer. The characteristic of irradiated Er,Cr:YSGG enamel demonstrated an irregular surface, no smear layer with spherical structures which resembles that of a bur-cut and etched surface [Lin et al.,1999]. This is believed to be beneficial for restoration adhesion.

The survey on microleakage of composite fillings in Er,Cr:YSGG laser prepared class II cavities have done by Guknecht et al in 2001. Their results recommended the additional use of etching after Er,Cr:YSGG laser preparation although the statistical analysis revealed no significant difference between Er,Cr:YSGG laser prepared cavity with or without additional acid etching. However, the restorations prepared in the classical manner with a high speed diamond and conditioned by etching showed the least dye penetration. The investigation of the microleakage of class V composite restorations using two different dentine adhesive system and two different modes of cavity preparation: a high speed hand piece and an Er,Cr:YSGG laser was studied by Ergucu et al. (2007). Their resulted showed that self-etch and total etch adhesive systems had

acceptable microleakage scores when used on Er,Cr:YSGG laser-prepared cavities; however, the additional acid etching after Er,Cr:YSGG laser preparation is recommended. On contrast, Shahabi et al. (2008) concluded that based on adhesive materials employed for their study (Total etch and Heliomolar), the higher microleakage occurred with phosphoric acid etching (37% phosphoric acid) of bur- or laser-cut surfaces, than with the surface created by use of the laser alone without additional conditioning for class V cavities.

The Er,Cr:YSGG laser gains an interest for the potential use in orthodontic application in order to substitute of using acid etching before bonding the bracket to enamel surface. Lin et al. (1999) found that the bond strength to Er,Cr:YSGG laser etched enamel relatively lower than that to the bur cut enamel with etching. However, when lased enamel was etched, it revealed similar bond strengths as the etched bur-cut enamel.

A study on laser etching of enamel for direct bonding with an Er,Cr:YSGG hydrokinetic laser system was also performed by Usumez et al. (2002). They compared the shear bond strength between bonding orthodontic brackets with lased and acid etched enamel surfaces. Their results indicated that the enamel surface treated with the Er,Cr:YSGG laser yielded statistically similar but lower and less predictable bond strengths than using acid etching with 37% phosphoric acid for 30 seconds. Nevertheless, it was comparable to the other groups and they reasoned

that this difference was probably from the hand controlled sweeping motion of the laser beam during the conditioning treatment; the motion might cause a weakly standardized etching pattern throughout the irradiated area. They suggested that etching the enamel surface with the Er,Cr:YSGG laser was more practical than with other previous types of lasers because there is no need to mark the enamel surface with ink. Moreover, the orthodontist can control the area for etching with the laser due to the static laser beam. Another advantage is that the laser induces caries resistance [Usumez et al, 2002].

In a subsequent study, Usumez et al. (2003) used the Er,Cr:YSGG to etch the tooth prepared for porcelain laminate veneers and determined the bond strength compared to etching with various kinds of etchants. The result revealed that there is no significant difference in microtensile bond strength between laser etching and various kinds of acid etching. They suggested that using laser etching was still currently too expensive to be cost effective method.

1.5 Glass ionomer cement

1.5.1 General knowledge about glass ionomer cement

Alan Wilson and Brian Kent from the Laboratory of the Government Chemist, London invented glass-ionomer cement in the 1970s [Nicholson, 1998]. Originally, it was developed from a combination of silicate cement and polycarboxylate cement. An attempt of the inventors

to reduce acidity and keep the sustained fluoride release of silicate cement resulted in substituting phosphoric acid, which was used as the liquid in the silicate cement, with polyacrylic acid which is used in the polycarboxylate cement [McLean and Wilson, 1977].

The main compositions of glass-ionomer cement (GIC) are fluoro-aluminosilicate glass and polyacrylic acid. The chemical formulation of the original ion leachable glass is SiO_2-AlO_3-CaF_2-Na_3AlF_6 in which the Al_2O_3 /SiO_2 ratio is approximately 1:2 [Smith, 1998]. As can be seen from the chemical formulation, the powder component of glass-ionomer contains sodium fluoride which acts as a fluoride releasing source. Thus, the crucial characteristic of glass-ionomer is the sustained fluoride release which is similar to that of silicate cement [Wilson and McLean, 1977]. Fluoride release from a restorative material to tooth structure plays an important role in reducing secondary caries incidence [Hickel et al, 2000]. Consequently, glass-ionomer appears to have substantial benefits when used as a restorative material.

Similar to polycarboxylate cement, a polyacrylic acid or polyalkenoic acid is commonly used as the liquid component of a typical glass-ionomer [Kugel, 2000]. The Carboxyl group (-COOH) of polyacrylic acid can form chemical bonding between tooth structure and GIC [McLean and Wilson, 1977]. Glass-ionomer can also be used as a luting agent for crown and bridge restoration [Mount, 1998]. In order to improve the handling

properties, 5-10% tartaric acid is added to the liquid component of the glass-ionomer [Nicholson, 1998].

There have been developments in glass-ionomer resulting in a material which is different from the original glass-ionomer of Wilson and Kent by composition modification. For example, the introduction of dicarboxylic or tricarboxylic acid into the polymer chain can both prevent gelation of the liquid as well as provide greater reactivity between the powder and liquid [Smith, 1998]. A few studies attempted to use other types of glass. Alumino-zinc-silicate glass was developed and used in high-strength hydrolytically stable glass-ionomer cement by Darling and Hill (1994). To enhance the mechanical strength of the glass-ionomer, blending glass particles with metal (e.g. silver) is one way to expand the application of glass-ionomer as a core build-up material [Nicholson, 1998; Guggenberger et al., 1998]. An advanced development of glass ionomer is resin-modified glass ionomer cement [Mitra, 1991; Smith, 1998]. This development was designed to overcome the problems of the insufficient mechanical and physical properties of traditional glass-ionomer cement when used as a posterior restoration.

Resin modified glass ionomer cement (RMGIC) was introduced in 1980s in order to overcome the shortcomings of the conventional materials [Chitnis et al., 2006]. There are several methods for blending the resin component into RMGIC but one common route is through the addition of

2-hydroxylethyl methacrylate (HEMA) during the manufacture of the acid component [Czarnecka and Nicholson, 2006a].

The adhesion of RMGIC to the tooth structure was obtained from a polymerisation reaction and an acid-base reaction. After pre-treatment with polyalkenoic acid, a thin hybrid layer or absorption layer is formed. Later, a chemical bond occurs through the ionic bond between the carboxyl groups of the glass ionomer and the calcium of hydroxyapatite that still remains around the collagen fibre [van Dijken, 2005].

Glass-ionomer is the product resulting from polyalkenoic acid in an aqueous solution attacked by an ion-leachable glass [Wilson et al., 1977]. This is called an "acid-base reaction". The glass is the base whilst the polyalkenoic acid is the acid in the system [Mount, 1998]. The setting mechanism begins with calcium ions preferentially being released from the complex of aluminosilicate glass when the powder and liquid are mixed together. Sequentially, aluminium is released following calcium. The cross-linking of polycarboxylate chains from the reaction between the carboxyl group of polyacrylic acid and a metal ion from glass propagates immediately [Mitra, 1991] as shown in Fig 1.9.

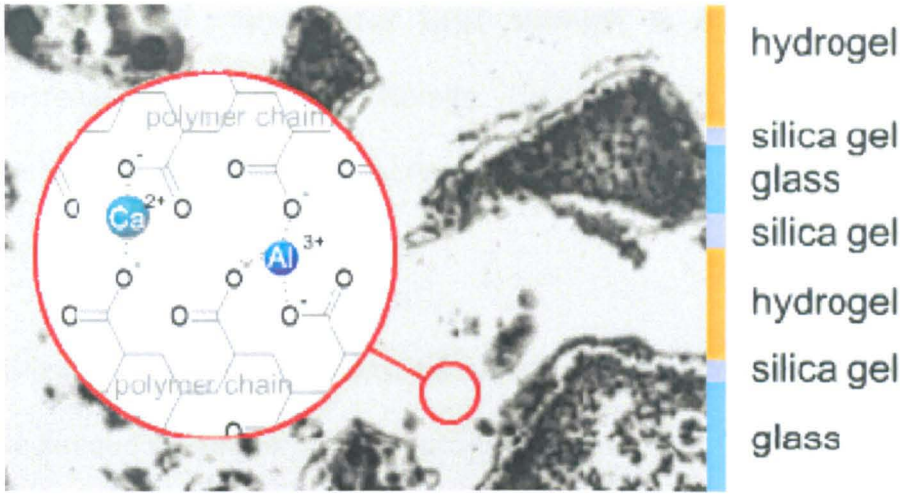


Figure 1.9 Photomicrograph of glass ionomer cement demonstrating the aluminosilicate glass surrounded by silica gel and dispersed in polymer chain matrix (retrieved from http://www.biomaterials.group.shef.ac.uk/biomaterials/gic.php May 2007)

Nicholson (1998) reported that the two metal salt units formed during the setting reaction were calcium acrylate and aluminium acrylate, which can be distinguished between using Fourier-transform infrared spectroscopy. The chemical bonding between glass-ionomer and tooth structure arises following the displacement of phosphate ions from the surface of the tooth with each phosphate ion taking with it a calcium ion to maintain electrolytic balance [Mount, 1998].

Glass ionomer cement used as restorative filling materials (e.g. Ketac Molar, Fuji IX, Glass Ionomer FX-II) was also called highly viscous glass ionomer cement [Irie et al., 2008]. They are commonly used for ART restoration. They can be filled in both class I and II cavities. The survival rate after 24 months was approximately 97.7 % and 69.9 % for class I and class II cavity, respectively [Ersin et al., 2008]. The mechanical

properties of cement and bond strength to dentine and enamel increased after 24 hour storage. The interfacial gap formation also decreases when tested specimen was polished and inspected after 24 hour storage [Irie et al., 2008].

Glass ionomer cement can be used as lining cement for sandwich technique restorations or composite laminated GIC [Opdam et al., 2006; van Dijken et al., 1999]. The purpose to use RMGIC as lining materials before composite placing in cavity is to reduce the polymerization shrinkage of composite resin. In addition, the longevity of restorations may increase due to good marginal seal and continuous fluoride release [Andersson-Wenckert et al., 2004]. Resin-modified glass ionomer cement (Rely X, Luting) also used for cementation purpose. It showed the good marginal adaptation of cast gold inlay restoration comparable to that achieved by zinc phosphate cement [Farrell et al., 2008].

Properties

The mechanical and physical properties of early glass-ionomer cement were not favourable. It was brittle and poorly aesthetic [Kugel, 2000]. The improved glass-ionomer cement demonstrated higher strength and good aesthetics. It was found that composite resin provided the greatest flexural strength, followed by cermet and conventional glass-ionomer respectively [Bapna et al., 2002]. Therefore, the clinical application of

conventional glass-ionomer is limited. It cannot provide sufficient strength for use in high-stress bearing areas [Young, 2002].

The other shortcoming of conventional GIC is moisture sensitivity. During the acid-base reaction, a moist environment is required for the setting step (precipitation, gelation and dehydration). However, the presence of excess water contamination during setting may cause dissolution of the reactant and inhibit the cross-linked matrix formation. Thus, saliva contamination during glass-ionomer setting is considered as a crucial clinical problem [Cho et al., 1995].

It is well known that GIC has anticariogenic properties because the ion-leachable glass particles act as a fluoride-releasing reservoir. Therefore, fluoride is simultaneously released after complete setting and continuously releases fluoride for a long period [Sales et al., 2003]. The remineralization of affected enamel can be activated by the high initial fluoride release. Secondary caries of the glass-ionomer filled tooth can be prevented by long-term fluoride release [Forsten et al., 1998]. Furthermore, GIC can recharge itself when it is exposed to fluoride in the oral cavity such as by dentifrices containing fluoride and fluoride mouthrinse [Kugel, 2000].

Biocompatibility is an important property of the glass-ionomer. There were some reports indicating that resin-based GIC such as Vitrebond (3M ESPE) is cytotoxic when compared with conventional GICs such as

Ketac Fil (Dentsply) [Leyhausen et al., 1998; Schmalz et al., 1994]. In contrast, conventional glass-ionomer demonstrated excellent cytocompatibility. The monomer residue in resin modified glass-ionomer may be the factor causing it to become cytotoxic to cells [Leyhausen et al., 1998].

Glass-ionomer cement comprises COOH groups in the liquid component, which can form hydrogen bonding. This hydrogen bonding is the first stage in obtaining adhesion between glass-ionomer and tooth structure. Glass-ionomer can adhere to enamel, dentine, stainless steel, tin or tin oxide plated with platinum and gold [McLean and Wilson, 1977].

1.5.2 Studies on adhesion of glass ionomer cement to dentine

The chemical bonding of glass ionomer cement to the tooth structure is still unclear. Ionic bonds and Wan der Val force play an important role for adhesion of conventional glass ionomer cement to the tooth structure [Lin et al., 1992]. The polyacrylic acid produces the polyacrylate ions which then form a strong ionic bond with the calcium ion from the tooth structure [Lucas et al., 2003]. It was believed that the polyacrylic acid in the liquid part of glass ionomer set has the ability to decalcify the dentine surface which is similar to the dentine conditioner action [Zimehl and Hannig, 2000]. Therefore, it may not be necessary to apply the dentine conditioner for conventional glass ionomer cement.

It is believed that the conventional GIC bonds to enamel by an ionic interaction with the mineral phase. However, the exact mechanism of adhesion was undefined [Glasspoole et al., 2002]. The chemical bonding of the cement with the tooth structure increases following displacement of the phosphate ion from the surface of the tooth with each phosphate ion taking with it a calcium ion to maintain electrolytic balance. Any contaminants on the surface may interfere with the ion exchange reaction. Thus, to provide a clean tooth surface, it is recommended to use a 10% solution of polyacrylic acid for 10 seconds to remove the smear layer and other contaminants [Mount, 1998].

Several studies have investigated the adhesion of glass ionomer cement to different tooth preparations. The shear bond strength of glass ionomer cement to caries-affected dentine was studied by Palma-Dibb et al. (2003). Their results showed that conventional glass ionomer cement (Fuji IX and Ketac Molar) provided lower bond strength values than the resin-modified glass ionomer cement. Later, de Souza-Zaroni et al. (2006) evaluated the shear bond strength of glass ionomer cement to bovine dentine using the air-abrasion technique for cavity preparation. It was found that Ketac Molar has higher bond strength value in bur prepared cavities whereas Fuji IX has higher bond strength in air-abraded cavities. The shear bond strength of resin modified glass ionomer cements to the Er:YAG laser treated tooth structure was studied by de Souza-Gabriel et al. (2006). They showed that the shear bond

strength of Fuji II LC and Vitremer (3M) using conventional bur preparation has a higher value than those prepared with an Er:YAG laser. It seem to be that the Er:YAG laser adversely affects the shear bond strength of RMGIC for both enamel and dentine. However, the bonding mechanism of GIC to different types of tooth substrate preparations is still not clear.

The investigation of laser treatment on adhesion of glass ionomer cement has rarely been studied. The CO_2 laser etching showed compromised effects on the bond strength of conventional glass ionomer cements (Fuji and Ketac cement) [Melendez, 1992]. The bonding mechanism between laser etching and glass ionomer cement is not clearly stated.

1.6 Summary of literature review

- Laser is effective and useful alternative in soft tissue surgery of oral tissue because it offers the low vibration soft tissue cutting, excellent coagulation ability, and precise incision.
- For the hard tissue application, the erbium laser especially Er,Cr:YSGG laser demonstrated an efficiency capability to prepare enamel, dentine, caries, cementum, and bone.
- The adhesion and microleakage of restorations to laser prepared tooth surface were controversial due to the difference in a type of restorative materials (composite resin, adhesive system) and the

laser parameter (pulse rate, laser energy). In addition, the difference of tooth substrate (permanent/deciduous human tooth) and surface treatment (conditioning, etching) showed different results.

- The bonding mechanism of composite resin to laser cut tooth surface was achieved via micromechanical retention (resin tag of composite resin).
- Few studies investigated the adhesion of glass ionomer to Er,Cr:YSGG laser cut tooth therefore the bonding mechanism still not clearly stated.

CHAPTER 2
OBJECTIVE AND OUTLINE OF STUDY

2.1 Objective

The first purpose of this in vitro study is to examine the morphological characteristics and micromechanical properties of the dentine surface, after being prepared with a Er,Cr:YSGG laser at different laser power settings. This work also aims to determine whether dentine morphology is adversely affected by this parameter. Furthermore, the ablation efficiency was also evaluated. Moreover, the author determined the microtensile and shear bond strength of conventional glass ionomer cement to dentine prepared with Er,Cr:YSGG laser. The influence of dentine conditioner application after laser preparation was additionally evaluated.

2.2 Outline of Study

2.2.1 Surface and Interface studies of laser prepared dentine

The Er,Cr:YSGG laser irradiated dentine surface when varying the laser parameters will be investigated using a microhardness test together with scanning electron microscopy (SEM). In addition, the SEM micrograph of

surface treatment (acid conditioning) on Er,Cr:YSGG laser irradiated dentine will be presented.

Furthermore, confocal fluorescence microscopy (CLSM) will be used initially to examine the glass ionomer cement/tooth interfaces of laser and bur prepared teeth. The images of various parts of Cl V cavity (the enamel-dentine junction, cavity floor, and line angle) will be recorded.

2.2.2 Adhesion studies

This evaluation will take the form of two separate parts:

Adhesion to laser prepared dentine

This is assessed by measuring the microtensile and shear bond strength of conventional glass ionomer cements (GICs) to human dentine prepared by laser or a bur with a high-speed hand piece. In addition, the effect of using dentine conditioner will also be determined.

Optimal laser parameters for adhesion of glass ionomer cement

The optimal laser parameters for adhesion of glass ionomer cement to laser-irradiated dentine will be determined by measuring the effect of varying the laser parameters (e.g. the power setting, the irradiation time, the distance between laser tip and dentine surface) on shear bond strength of GICs.

CHAPTER 3
SURFACE AND INTERFACE STUDIES OF LASER PREPARED DENTINE

3.1 Background

The process of dentine removal with the Er,Cr:YSGG laser is different from that using the conventional high-speed handpiece. The former induces considerable changes on the surface and physical properties of dentine and the thermal effects may cause both de/remineralisation and deproteinization on the dentine surface [Neev et al., 1996]. The non-homogeneous structure of dentine, which consists of peritubular, intertubular and tubular dentine, has been shown to contribute to its mechanical properties. The elastic modulus is also associated with the mineral content as well as porosity of the structure [Barros et al., 2005]. The micromechanical properties such as microhardness and elasticity of dentine play important roles in stress distribution when mastication forces are applied on the filling material of a restored tooth [Jacques et al., 2005].

Few studies have been carried out with the Er,Cr:YSGG laser system to survey the micromechanical properties of the dentine surface [Hossain

Missing page

et al.,1999; Hossain et al., 2003], despite the fact that this may have a profound effect on the abilities of adhesive materials to interact with the surface. The purpose of this research was firstly to study the micromechanical properties and microscopic appearance of Er,Cr:YSGG laser prepared dentine. The second aim was to determine the optimal power settings of an Er,Cr:YSGG laser for cutting human dentine to produce a surface which remains suitable as a foundation on which to build and bond a dental restoration using a glass ionomer cement.

The null hypothesis for this study is the microhardness of dentine surface after Er,Cr:YSGG laser would not be difference from the sound bur prepared dentine.

3.2 Materials and Method

3.2.1 Dentine cutting efficiency of Er,Cr:YSGG laser

The laser used for this study was an Er,Cr:YSGG laser (erbium, chromium, yttrium, scandium, gallium, garnet) [Biolase™, Waterlase™ Millennium®, San Clemente, California, USA], with a pulse duration of 140 μs (Figure 3.1a). The lasing medium was an Yttrium Scandium Gallium Garnet crystal doped with Erbium and Chromium. This medium emits a laser light of wavelength 2.78 μm. The repetition rate was 20 Hz and power output ranges of 0 to 6 watts were used (Figure 3.1 b). The setting of air and water at the control panel were set at 65% and 55%,

respectively. The pulse energy varies from 0 to 300 mJ. A clear sapphire tip of 600μm diameter (G6) was used (Figure 3.1c). A high-speed handpiece (Kavo, Super-Torque 625, Germany) with round diamond bur with diameter of 1 mm (Unodent, BD521, lot 53883, Israel) was used for conventional tooth preparation.

The teeth used in the study were extracted permanent human molars and premolars without caries or defects, stored in an aqueous solution of 0.5 % chloramine T no more than 6 months from time of extraction to inclusion in this study. All teeth were approved by the local ethical committee for use in this experiment.

Figure 3.1 (**a**) The Er,Cr:YSGG laser unit (**b**) Control Panel showing power settings, as well as percent of air and water during the cutting process (**c**) The sapphire tip (1) and zirconia tip (2).

Tooth preparation

One third of the occlusal surface of each of the 20 teeth was trimmed with a high-speed diamond saw (Micro Slice 2, Metals Research Limited, Cambridge, England) using water spray to provide a flat dentine surface.

The surfaces were then cut again to provide 2 mm thick dentine discs. All discs were embedded in a plastic mould and the exposed surface finished using 500 grit SiC paper and subsequently with calcined alumina oxide powder (particle size 3 μm).

Ablation efficiency evaluation

The dentine discs were randomly divided into four groups of five teeth (Groups 1, 2, 3, 4) treated with different laser power settings: 3W (33.9 J/cm^2), 3.5W (39.6 J/cm^2), 4W (45.2 J/cm^2) and 4.5W (50.9 J/cm^2) respectively. Each specimen was placed on a movable stand and a laser handpiece was held with a clamp to fix the distance at 1 mm. The working distance between the end of laser tip and the dentine surface was measured by adjustment of removable stand to suit the thickness of feeler gauge at 1 mm. The repetition line was made free hand by moving the specimen along the path of the laser beam over 60 sec (Figure 3.2 a). All the ablation lesions were duplicated with synthetic rubber replicating compound (Figure 3.2b) (Microset, Microset Products, Ltd, UK) for evaluation using a 3D laser scanning surface profiler [OSP100, Uniscan Instrument, Inc., UK] (Figures 3.2 c and d).

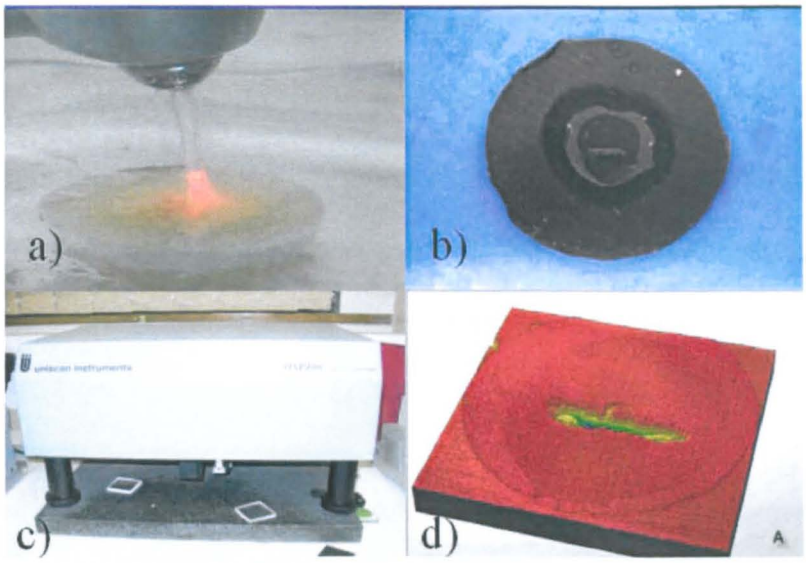


Figure 3.2 a) Each dentine disc was exposed to the laser energy b) Impression of dentine lesion after laser exposure c) The impression was then scanned by a Uniscan profilometer d) The scanned dentine lesion was analysed by computer software.

3.2.2 Structure and properties of laser prepared dentine

Microhardness measurement

The microhardness of the dentine surface before and after laser irradiation was measured; the same specimens from the dentine ablation efficiency study in section 3.2.1 were used for assessment of the microhardness of the dentine surface (Figure 3.3a). Five pyramidal-triangular indentations at a load of 200 g and a dwell time of 20 seconds were made on one half of each disc using a Martens microhardness tester [Zwick/Roell Z2.5, Ulm, Germany] (Figure 3.3b) before and after laser application (Figure 3.3c). By averaging the twenty-five indentations for each group, the mean microhardness value

and elastic modulus before and after irradiation were automatically calculated by the computer software (TestXpert, Germany). The baseline and irradiated dentine microhardness were compared using paired t-tests. The relationship between the microhardness of treated dentine and the laser power setting used was investigated by regression analysis.

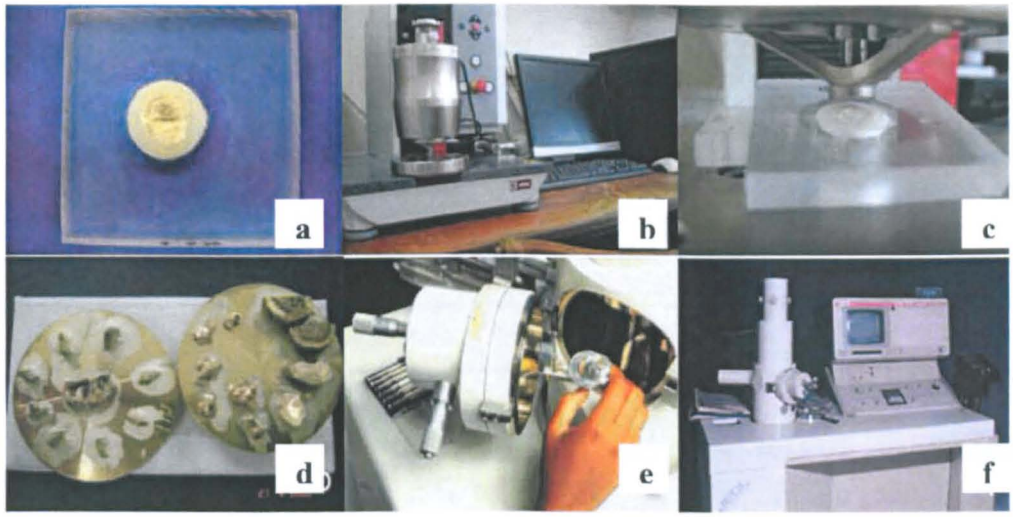


Figure 3.3 **a**) Dentine disc mounted on the plastic mould **b**) the Zwick/Roell Z2.5 microhardness measurement **c**) the indentation on the dentine disc was made by a pyramidal needle (Vicker) **d**) Dentine specimen were mounted on stub **e**) The stub were put into the chamber of Stereoscan S240 **f**) The stereo scan S240 SEM microscopy.

Microscopic Investigation

Representative specimens from each group were cut transversally through the dentine disc and immersed in an ultrasonic cleaner for 1 min. Representative specimens from each group were also treated with two dentine conditioners (Table 3.1) before cutting and immersing in an

ultrasonic cleaner. For the purposes of comparison, a further specimen was prepared in the same way but the surface was treated using a diamond bur on the high-speed handpiece, which was held with a clamp to fix the distance at 1 mm. The repetition line was made free hand by moving the specimen along the path of the laser beam over 60 seconds on the flat dentine. All specimens were then mounted and sputter-coated for examination with a Stereoscan S240 Scanning Electron Microscope (Cambridge, UK) as shown in Figures 3.3 d-f.

Table 3.1: Manufacturers and composition of materials used for this study

Material	Manufacturer	Constituents
Fuji IX GP Capsulated	GC Corp., Japan	Fluoroaluminium silicate glass Polyacrylic acid, Polybasic carboxylic acid
Ketac-Molar Aplicap	3M ESPE, UK	Aluminium-calcium-lanthanum-fluorisilicate glass, 5% spray dried ESPE polycarbonic acid An aqueous solution of polycarbonic acid and tartaric acid
Dentine Conditioner	GC Corp., Japan	10% Polyacrylic acid
Ketac Conditioner	3M ESPE, UK	10% Polyacrylic acid

3.2.3 Interface of laser prepared dentine and glass ionomer cement

Specimen preparation

The teeth used in this study were extracted human molar teeth without caries or defects (cracking or fissuring), stored in an aqueous solution containing 0.5 % chloramine T. Buccal V-shaped Class V cavities with occlusal and gingival margin in enamel and pulpal wall in dentine were prepared by Er,Cr:YSGG laser or bur and each cavity was restored with either Fuji IX or Ketac Molar (Table 3.1, Figures 3.4 a-c). The depth from buccal surface to pulpal wall was approximately 2 mm. To apply the fluorescent rhodamine B dye, two-thirds of each root was removed and then the pulp tissue was removed with a K-file (#20-50) to provide enough access for dye introduction. Each tooth was stood with the crown down in a chamber and rhodamine B dye syringed into the pulp chamber and left for 3 hours. Subsequently they were stored at 37°C and 100% humidity for 24 hours before sectioning.

All teeth were sectioned longitudinally from the crown to the root into two halves with a high-speed diamond saw (Micro Slice 2, Metals Research Limited, Cambridge England) using water spray (Figure 3.4D). The cut surface was finished using precision lapping and a polishing machine (PM2A, Logitech, Materials Technologists Engineers) with

calcined alumina oxide powder particles of size 3 μm. The sections were stored in distilled water at 37°C.

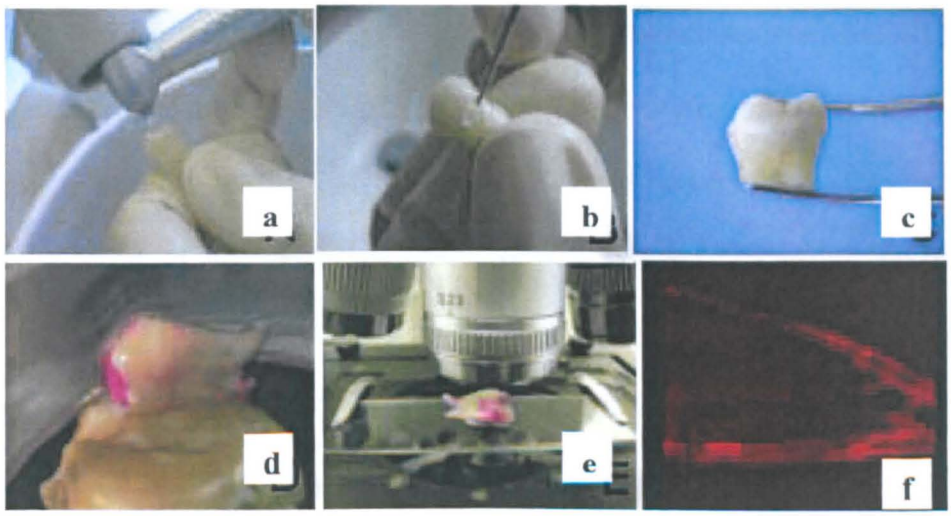


Figure 3.4 **a-f** illustrating the procedure to investigate the GIC/laser prepared dentine interface by using Confocal Laser Scanning Microscopy.

Confocal Microscopy

The sections were visualized by a Leica TCS SP2UV confocal laser scanning microscope (CLSM) (Leica Lasertechnik GmbH, Heidelburg, Germany) with Leica TCS-NT software (version 2). Images were acquired through a 5x objective lens with a numerical aperture (NA) of 0.15, under a 543 nm excitation beam from He/Ne laser and emission collected at 560-620 nm. Images were obtained by X, Y, Z scanning (512x512 pixels).

Montage technique

To investigate the adaptation for the entire cavity by confocal microscopy, a montage technique was employed. As the area was too great to be scanned in one time, the entire cavity was scanned with a 5x lens several times to get the whole image of the cavity. A number of images were taken with a degree of overlap and later composed together. Each section of the image was captured in such a way that visible features identified in the bottom margins of each image could be used as a guide for the next capture, when overlapped with those same features in the top margins of the following image. The captured images were imported into Adobe Photoshop V7.0, cut and pasted onto a suitable canvas (A4) size as separated layers. These were carefully aligned at 100% magnification and adjusted in brightness and contrast to match each other. Once complete, the final image was optimized using the levels control.

Gap measurement

Forty seven CLSM micrographs from six teeth; three teeth from the bur prepared group and three teeth from the laser prepared group were obtained. Each scanned image of the cavity outline was printed on A4 paper and the gap width was measured. The maximum gap width was selected as the representative data for each image. The mean gap width along the Cl V cavity was recorded.

3.3 Results

3.3.1 Ablation efficiency evaluation

The representative scanned images of the ablation lesion on the dentine disc obtained from several power settings are illustrated in Figure 3.5.

The amount of dentine removed by the Er, Cr:YSGG laser with various energy fluencies for a given time is shown in Table 3.1.

Table 3.2: The means (SD) of the amount of dentine ablation using different laser power settings (n=5)

Test group	Average dentine depth (mm)	Average ablated dentine volume (mm^3)	Average ablated dentine volume per sec (mm^3/s)
Group 1 (3 W)	0.15 (0.02)	0.62 (0.25)	0.01
Group 2 (3.5 W)	0.28 (0.10)	1.82 (1.03)	0.03
Group 3 (4 W)	0.33 (0.07)	2.00 (0.69)	0.03
Group 4 (4.5 W)	0.42 (0.14)	2.91 (1.06)	0.04

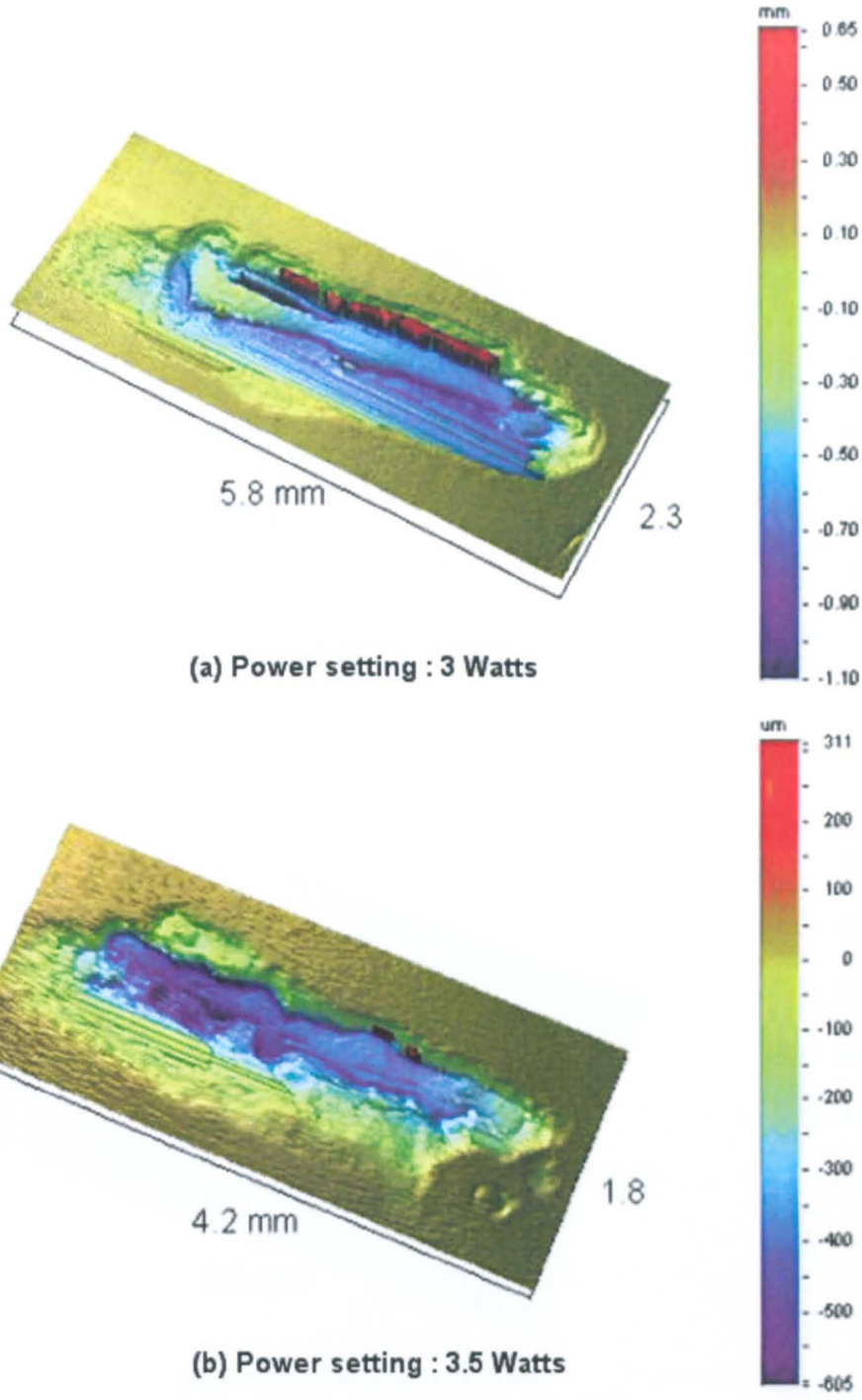
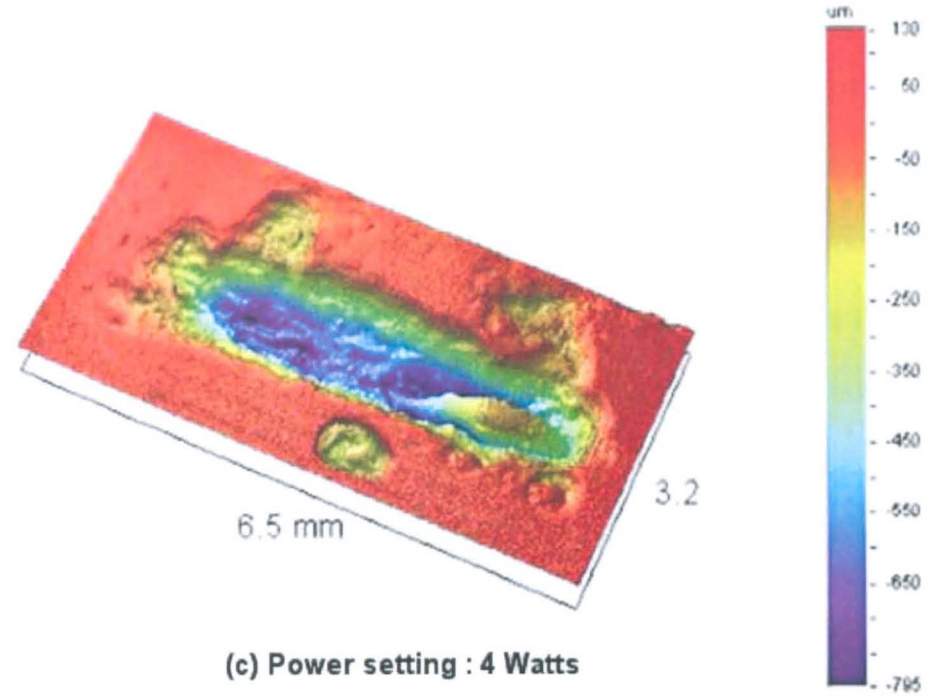
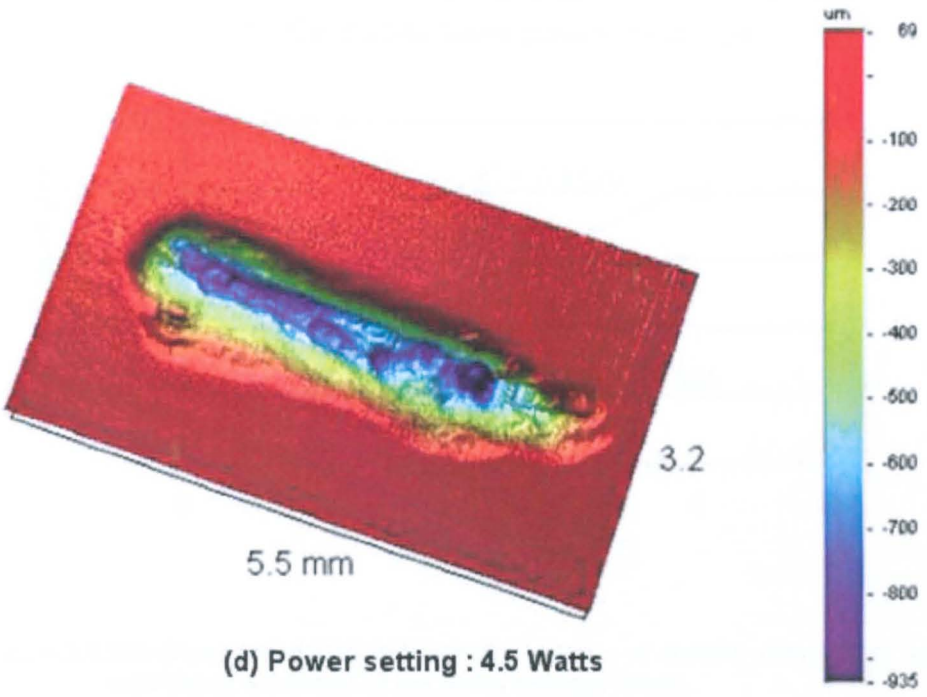


Figure 3.5 Scanned images obtained from laser profilometer showing the dentine lesion at the different power settings; **a**) 3 Watts and **b**) 3.5 Watts



(c) Power setting : 4 Watts



(d) Power setting : 4.5 Watts

Figure 3.5 (Continue) Scanned images obtained from laser profilometer showing the dentine lesion at the different power settings; **c**) 4 Watts and **d**) 4.5 Watts

The mean volume of dentine ablated (mm^3) was calculated by multiplying the surface area of the irradiated dentine lesion with the average ablation depth. The volume of dentine ablated per sec (mm^3/s) was calculated by dividing the volume of dentine ablated over the time (60 seconds). There was an increase in depth of ablated dentine, and dentine ablated volume with an increase in laser output power. The mean ablation efficiency showed a broadly linear correlation with the power output using linear regression analysis (R^2=0.85, $p<0.05$) as shown in Figure 3.6

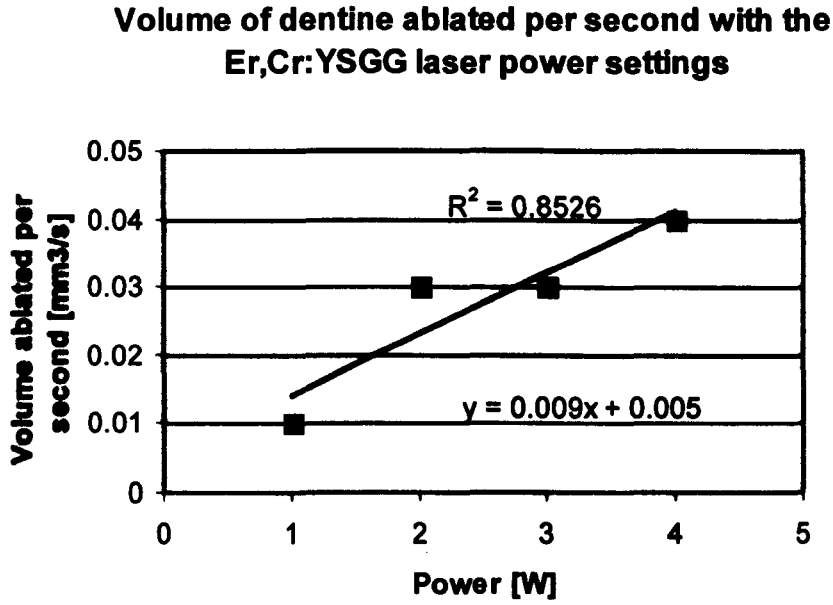


Figure 3.6 The linear correlation between the volume of dentine ablated per second (mm^3/s) as a function of the power settings (Watt).

3.3.2 Microhardness measurement

Values of dentine microhardness (Vicker's Hardness Number, VHN) and elastic modulus for the baseline and post-irradiation are shown in Tables 3.2 and 3.3 as well as in Figure 3.7. Dentine microhardness decreased significantly after laser irradiation (paired t-test, $p<0.05$) at all power settings.

Table 3.3: The means (SD) of Vickers microhardness using different power settings (n=5)

Test group	Baseline	Laser irradiation
Group 1 (3 W)	60.85 (8.39)	42.89 (16.39)
Group 2 (3.5 W)	66.18 (5.12)	30.27 (11.94)
Group 3 (4 W)	62.39 (4.01)	40.41 (19.52)
Group 4 (4.5 W)	64.94 (8.72)	41.27 (9.15)

Table 3.4: The means (SD) of Elastic Modulus (kN/mm^2) of base line and laser irradiated dentine (n=5).

Test group	Baseline	Laser irradiation
Group 1 (3 W)	16.73 (2.53)	17.73 (6.36)
Group 2 (3.5 W)	20.69 (2.85)	18.74 (7.95)
Group 3 (4 W)	17.46 (1.00)	14.65 (3.83)
Group 4 (4.5 W)	18.51 (1.98)	19.92 (6.28)

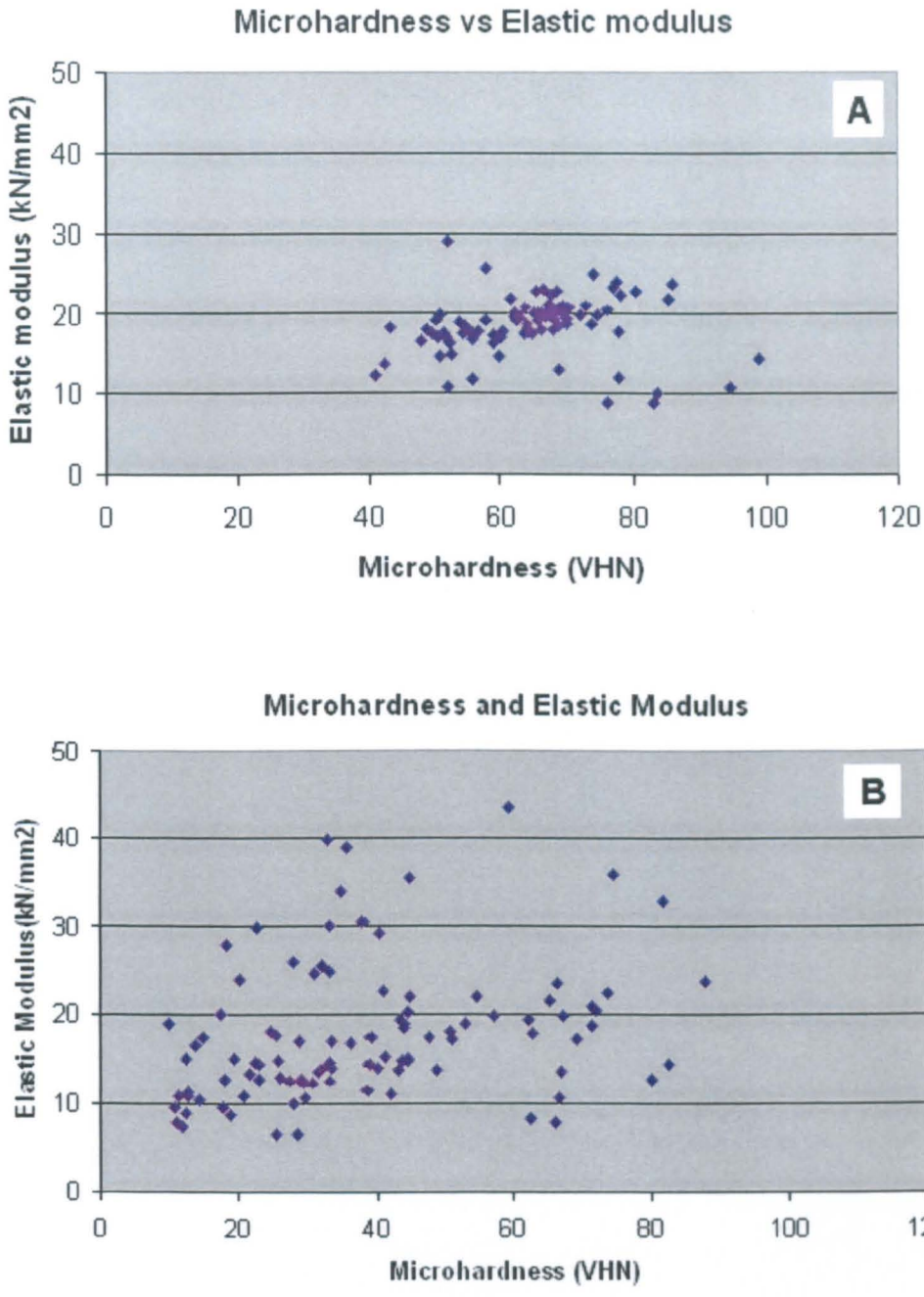


Figure 3.7 The plot between microhardness and elastic modulus **before** laser application (A) and after laser irradiation (B);(A) the variation of elastic modulus and microhardness dentine is in the narrow regime before laser irradiation (B) After laser irradiation, the variation of elastic modulus and microhardness of dentine distributed in a broad regime.

3.3.3 Microscopic Investigation

SEM observation

The SEM appearance of bur-cut dentine showed a relatively flat topography and the presence of a smear layer (Figures 3.8a and 3.8c). In contrast, the dentine appearance after laser irradiation indicated a corrugated or wavy profile (Figure 3.8b), opened dentinal tubules and the absence of a smear layer (Figure 3.8d and Figure 3.9) at all power settings.

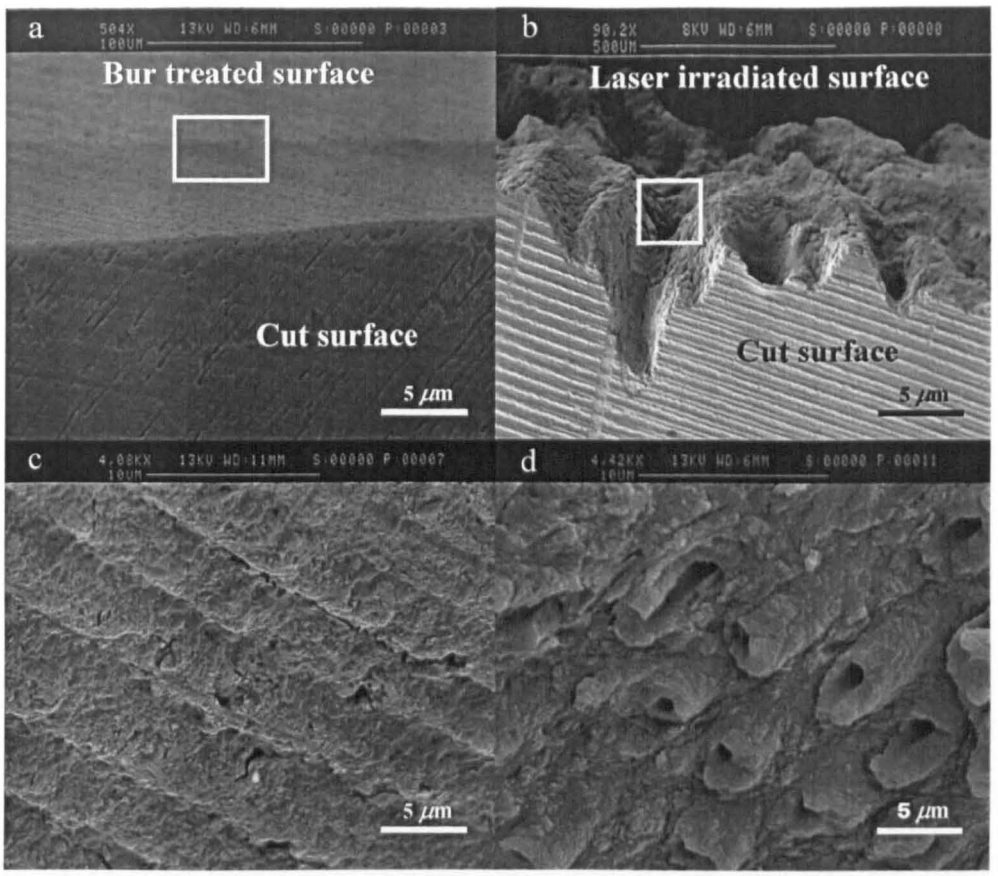


Figure 3.8 SEM micrographs of transversally sectioned dentine disc: **a)** Typical profile of bur-cut dentine showing a flat surface; **b)** Typical profile of laser irradiated dentine at any power setting, showing a corrugated and wavy appearance; **c)** Appearance of the bur-cut surface at high magnification in the boxed area in Figure 3.8a, showing track lines formed due to the rotary instrumentation, and the presence of a smear layer; **d)** Higher magnification of the boxed area in Figure 3.8b showing dentinal tubules opening at different planes and possibly depletion of intertubular dentine after laser application.

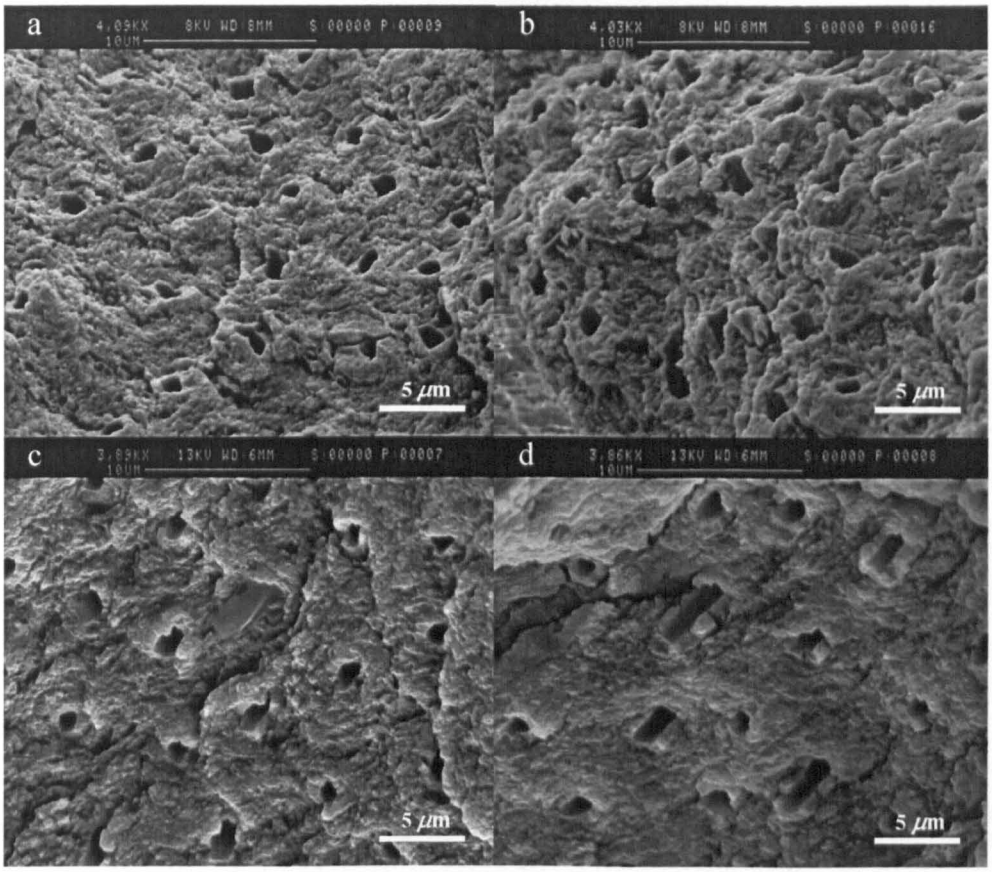


Figure 3.9 SEM micrographs of an irradiated dentine surface with different power settings: **a)** 3 Watts, **b)** 3.5 Watts, **c)** 4 Watts, and **d)** 4.5 Watts, showing opened dentinal tubules and the absence of a smear layer.

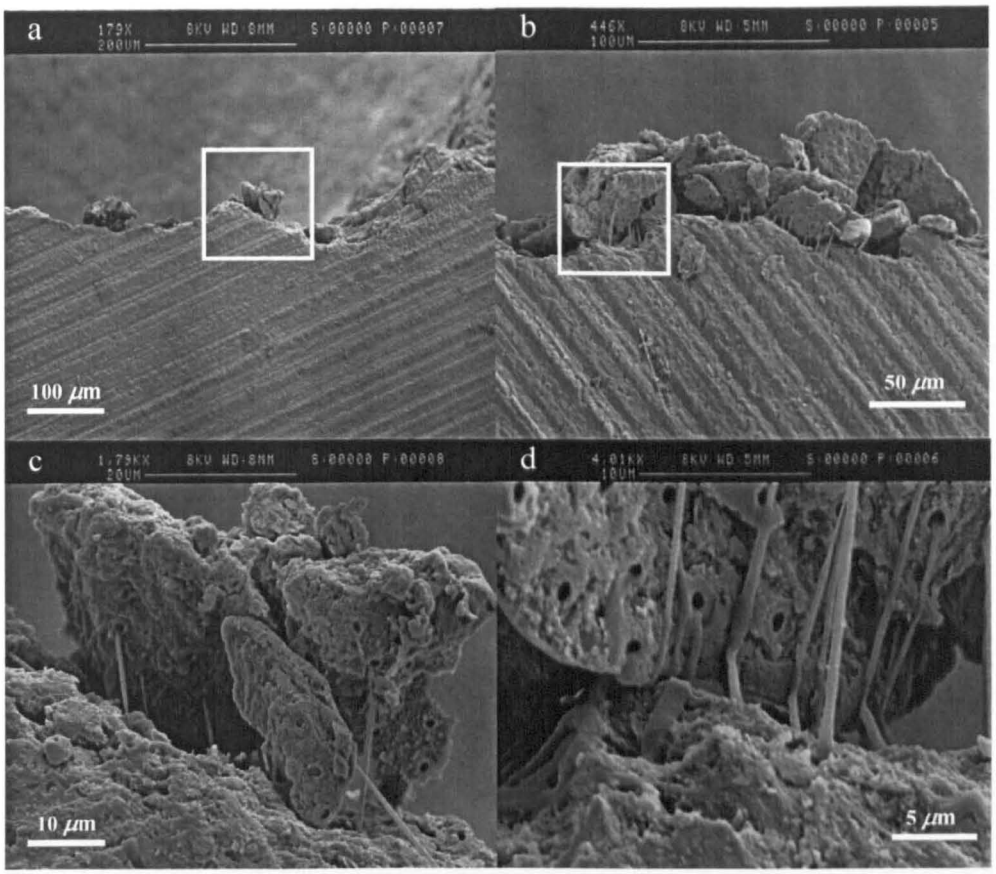


Figure 3.10 SEM micrographs of transversally sectioned dentine using a power setting at 3 Watts. There was irradiated dentine debris along the interface (**a** and **b**). At high magnification, fibre- like tags linked the debris to the underlying dentine (**c** and **d**).

Further examination of the profile at the lowest power setting (3 Watts) revealed the presence of dentine debris with tags attaching it to the underlying lased dentine (Figure 3.10). At 3.5 Watts power setting, there was dentine debris on the surface without any tags (Figure 3.11).

In addition, it appeared that there was a glassy molten substance on the dentine (Figure 3.11b). At the 4 Watts power setting, the irradiated dentine looked more crystalline in nature, without any debris (Figures

3.12a & b). When the highest power setting, 4.5 Watts is applied, a molten appearance was noted on the surface (Figures 3.12c & d). The molten areas were chemically analysed using SEM-EDX (JEOL 5300 – LV, Japan) with an analysis system (Rontec Edwin Energy Dispersive Analysis, Japan) (Figure 3.13). This revealed the presence of Ca, P, C and O peaks coinciding with the molten areas as in the irradiated dentine where there was no molten mass (Figure 3.14).

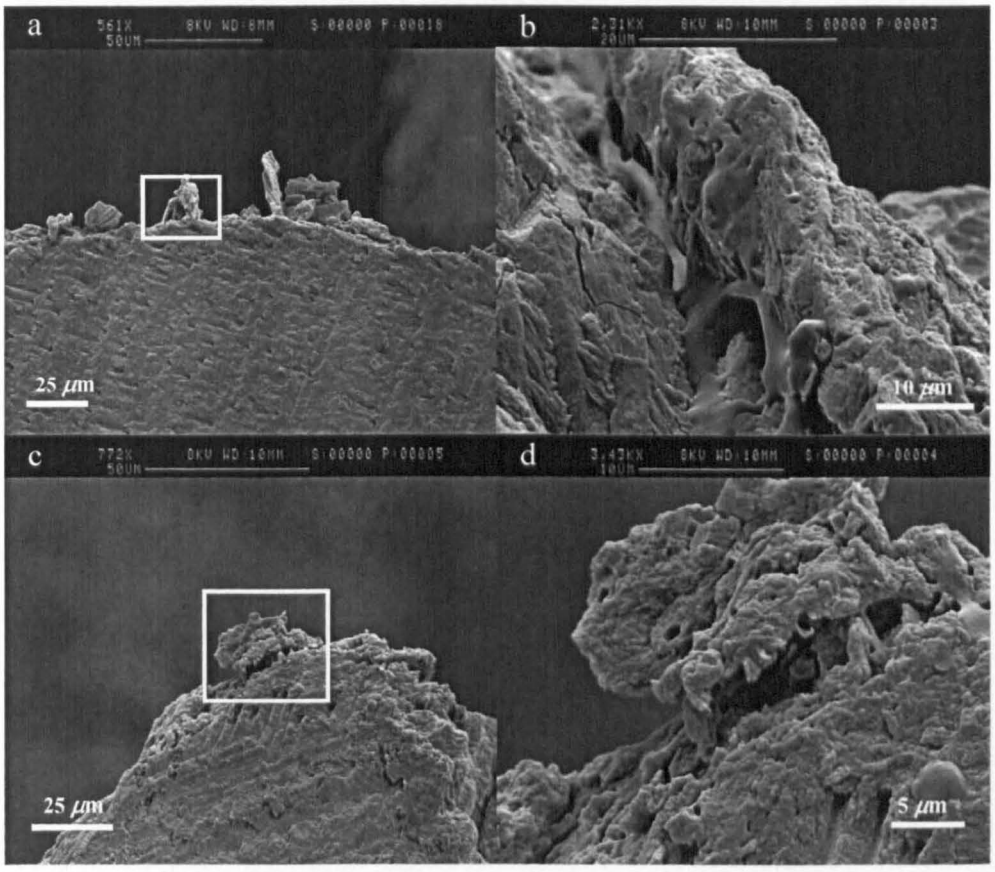


Figure 3.11 SEM micrographs of transverse view of irradiated dentine using a power setting at 3.5 Watts: There were some dentine debris particles (**a, c**) along the interface. At high magnification, a molten - like substance (**b**) Area within the box in Figure 3.11a showing a molten like substance (**d**) Area within the box in Figure 3.11c showing a irregular dentine substance.

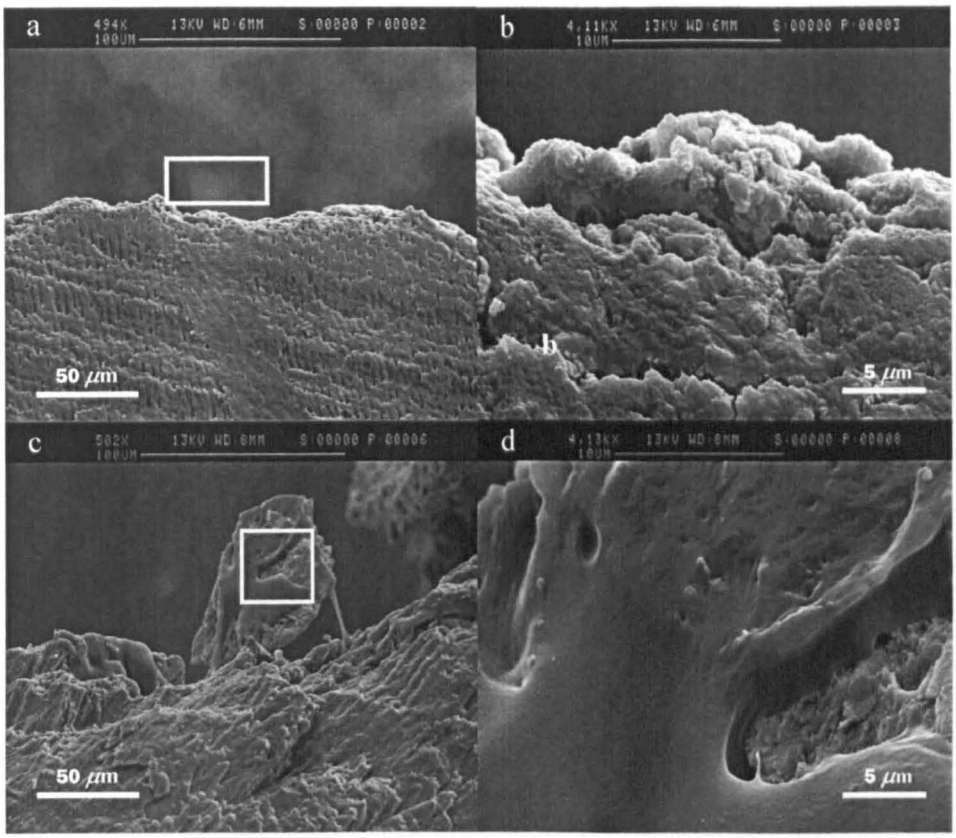


Figure 3.12 SEM micrographs of transverse view of the irradiated dentine surface using a power setting at 4 Watts (**a** and **b**): The dentine surface consists of a crystalline-like substance. At a power setting of 4.5 Watts, there were some dentine debris particles on the surface (**c** and **d**). Figure 3.12**b** is the area in the box of Figure 3.12**a** and Figure 3.12 **d** is the area in the box of Figure 3.12**c** at the high magnification.

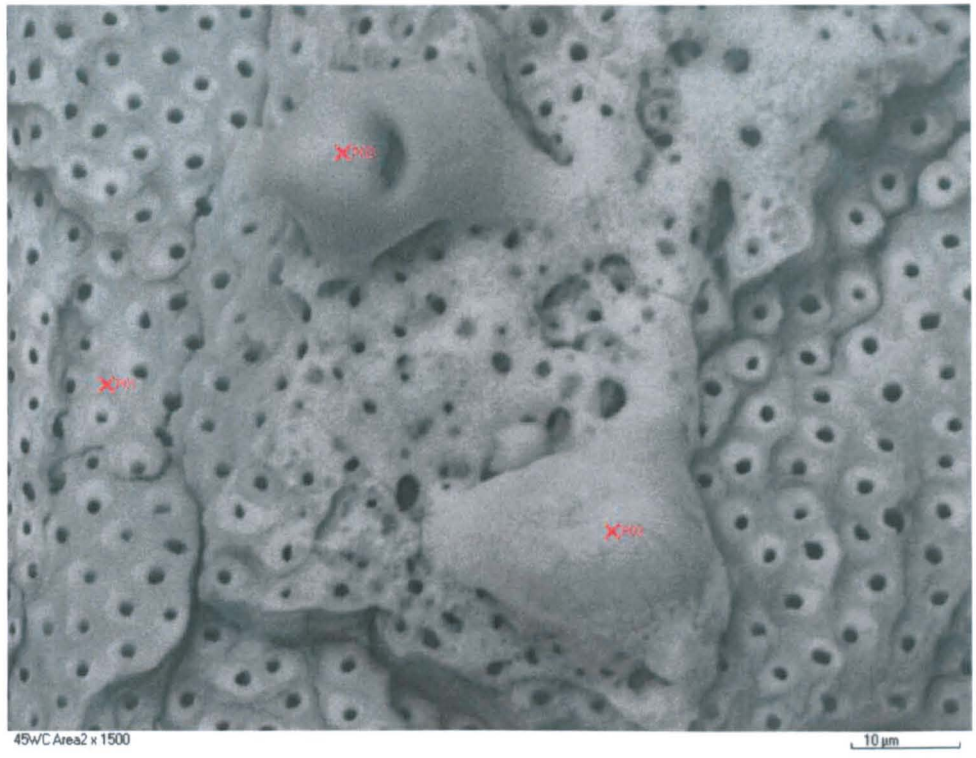


Figure 3.13 SEM-EDX micrograph of the irradiated dentine surface using the power setting of 4.5 W with the three different areas marked in red ink; PO1 is an area of typical dentine, PO2 and PO3 are an area of a molten like substance on the dentine surface.

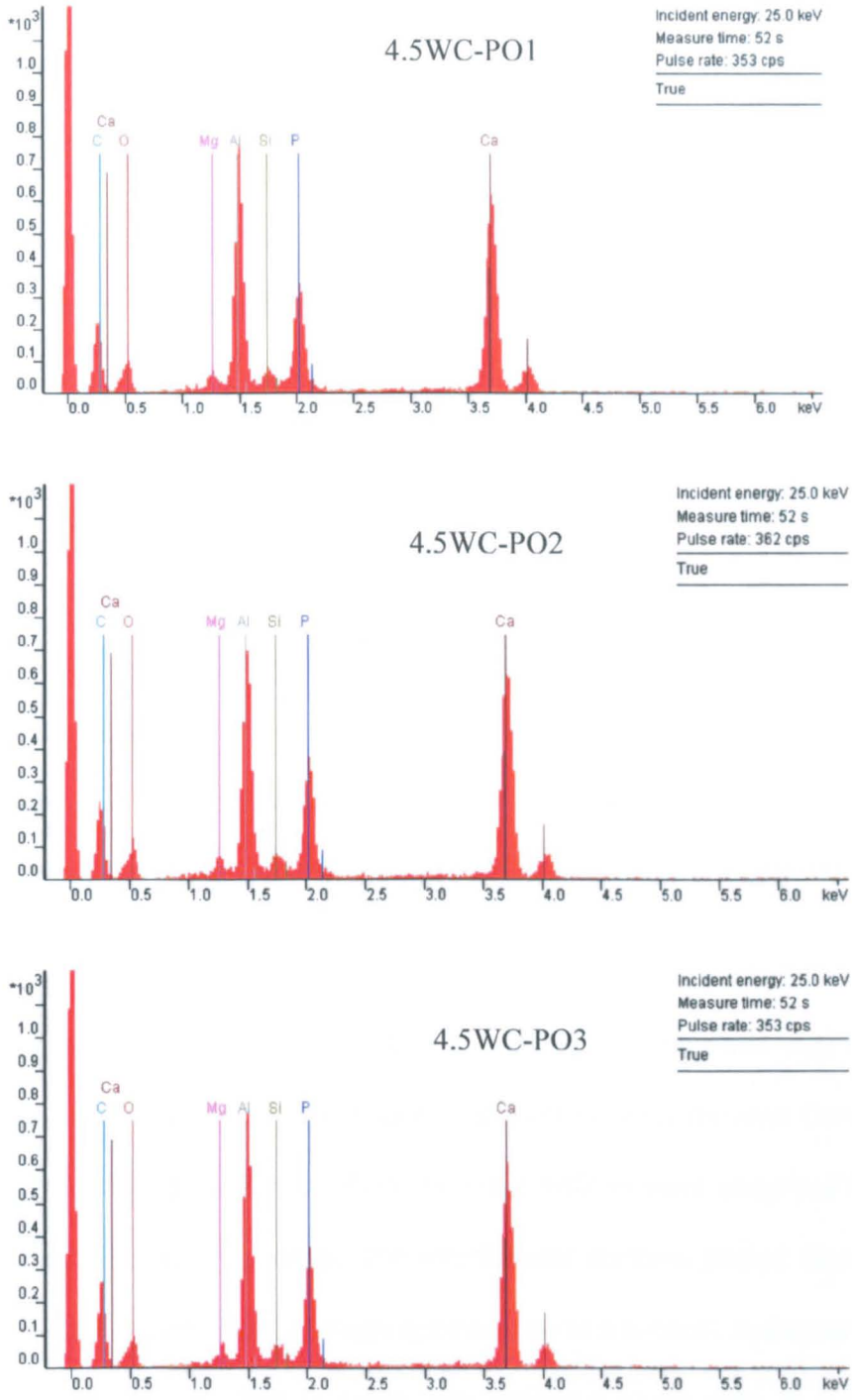


Figure 3.14 SEM-EDX analysis of three different areas coded as in Figure 3.13; showing the same peak of each element such as Ca, P, O.

The representative SEM images of laser-irradiated dentine specimens treated with dentine conditioners (Ketac and Dentine Conditioner) at the different power settings are shown in Figures 3.15-3.18.

At 3 Watts, there is no presence of dentine debris with the linking fibre like tags (Dentine Conditioner specimen). However, there are many molten like dentine patches on the dentine surface (Figure 3.15 a in box 2 and e). The intertubular dentine reduces the degree of roughness, and shows a less scaly appearance (Figure 3.15c). The peritubular dentine appears elevated due to the depletion of intertubular dentine (Figure 3.15c). The dentinal orifices were clearly observed (Figures 3.15b and f) in the Ketac Conditioner specimen. The depletion of intertubular dentine along the peritubular dentine reveals a liquid like appearance (Figure 3.15d). The dissolved intertubular dentine completely covered the orifice of some dentinal tubules (Figure 3.15f, white arrow).

At the 3.5 Watts power setting, a dentine debris plate was also found in the SEM micrographs from laser cut dentine with Dentine Conditioner treatment (Figure 3.16a). Clear dentinal orifices were observed (Figures 3.16c and d). In general, the intertubular dentine (Ketac Conditioner specimen) turns from a rough appearance to a smooth appearance. The scaly peritubular dentine turns from having a sharp edge to a bevelled one (Figure 3.16d).

At the 4 watts power setting, open dentinal tubules were generally found (Figures 3.17 a, b and c). A few dentine debris are found in the representative specimen in which the Ketac Conditioner was applied (Figures 3.17 b and d).

At the 4.5 watts power setting, various types of dentine appearance were observed. In general, the degree of roughness of the dentine surface was reduced (Figures 3.18 a and b). The dissolved intertubular dentine flows to cover the dentinal tubules in the Ketac Conditioner treated specimens (Figure 3.18b). The opening dentinal tubules were more noticeable with the Dentine Conditioner specimen (Figures 3.18 c and e). The collar-like appearance of the peritubular dentine elevated from the intertubular dentine surface was also found in the Ketac conditioner specimen (Figure 3.18d). Loss of depleted intertubular dentine in the crater area is demonstrated in Figure 3.18f.

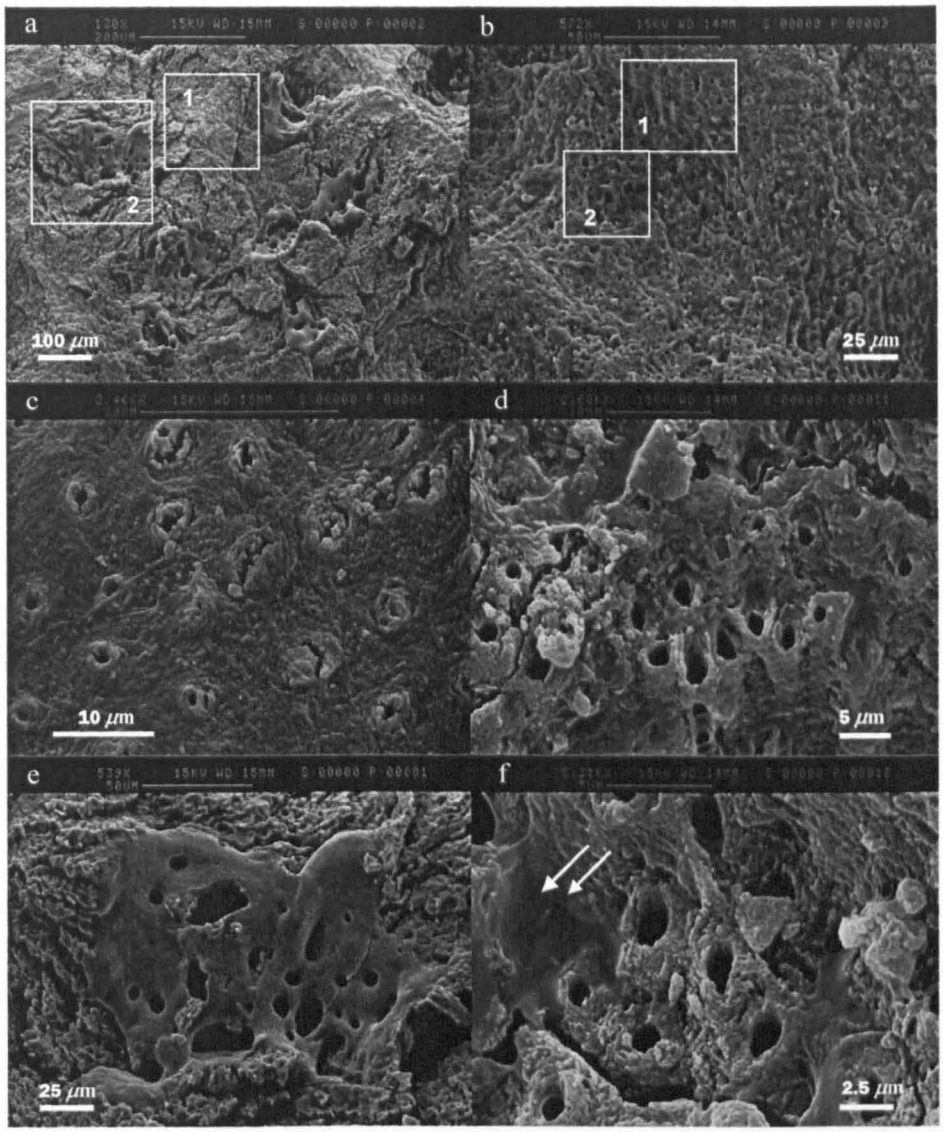


Figure 3.15 Representative SEM micrographs of dentine surface irradiated at the 3 watts power setting and followed by Dentine Conditioner and Ketac Conditioner

a) The irradiated dentine and Dentine Conditioner application revealed the dissolved dentine debris coating on the surface
b) The irradiated dentine and Ketac Conditioner application exhibit a liquid-like layer covering dentine surface.
c) The laser cut dentine (area in the box1 of Figure 3.15a) showed dissolved intertubular dentine and elevation of peritubular dentine around dentinal tubules
d) The laser cut dentine (area in the box1 of Figure 3.15b) demonstrated depletion of intertubular dentine
e) The laser cut dentine (area in the box2 of Figure 3.15a) and f) The laser cut dentine (area in the box2 of Figure 3.15b) showing some dentinal tubules were sealed by dissolved and molten dentine mass.

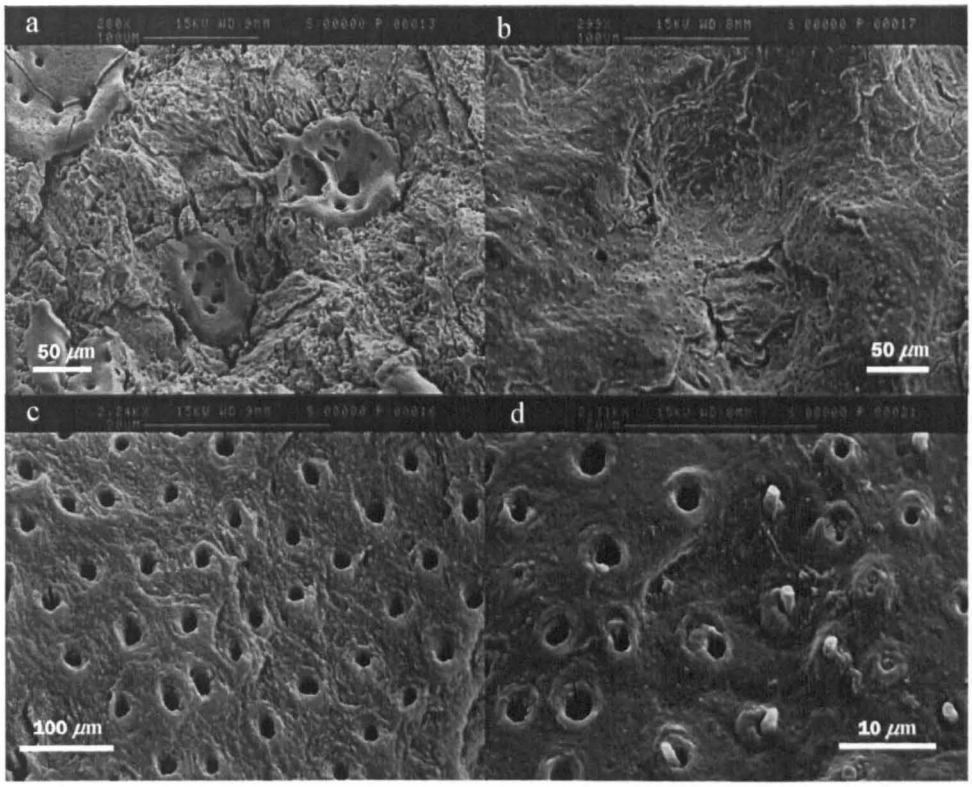


Figure 3.16 Representative SEM micrographs of dentine surface irradiated at the 3 watts power setting followed by Dentine Conditioner (**a and c**) and Ketac Conditioner (**b and d**).

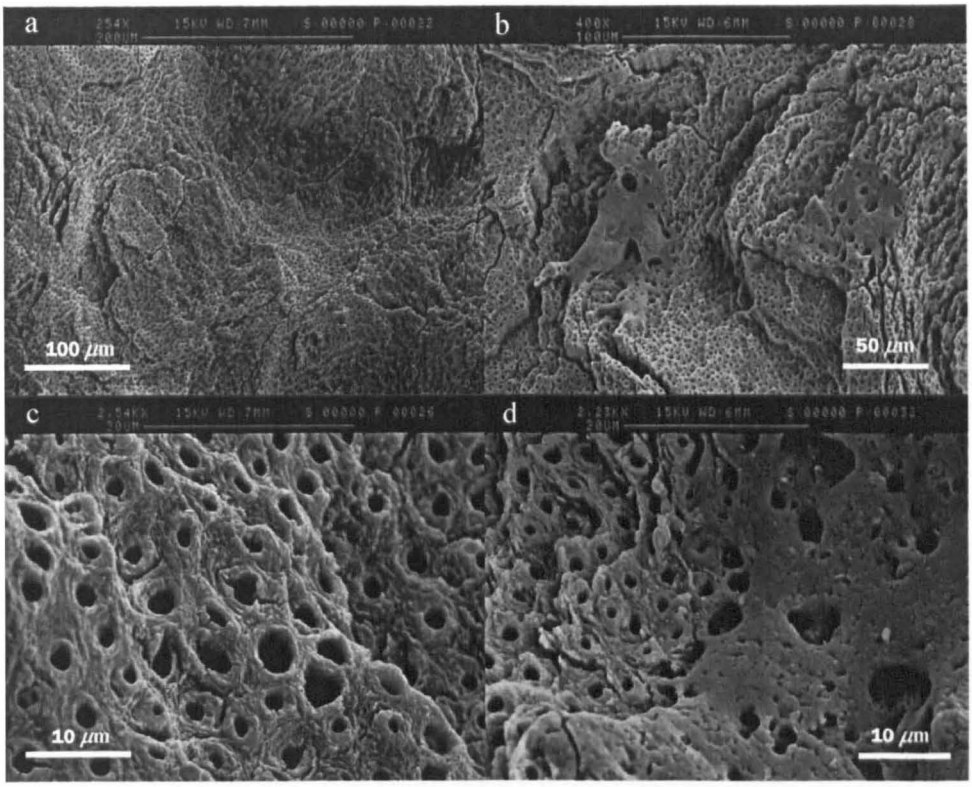


Figure 3.17 Representative SEM micrographs of dentine surface irradiated at power setting of 4 W and followed by Dentine Conditioner (**a** and **c**) and Ketac Conditioner (**b** and **d**); **a)** at low magnification, showing an absence of dentine debris plate **b)** at high magnification, showing the clear wide opening dentinal tubule **c)** at low magnification, showing a presence of plate-like appearance dentine debris **d)** at high magnification, showing a dentine debris plate covering the irradiated surface and opening dentinal tubules.

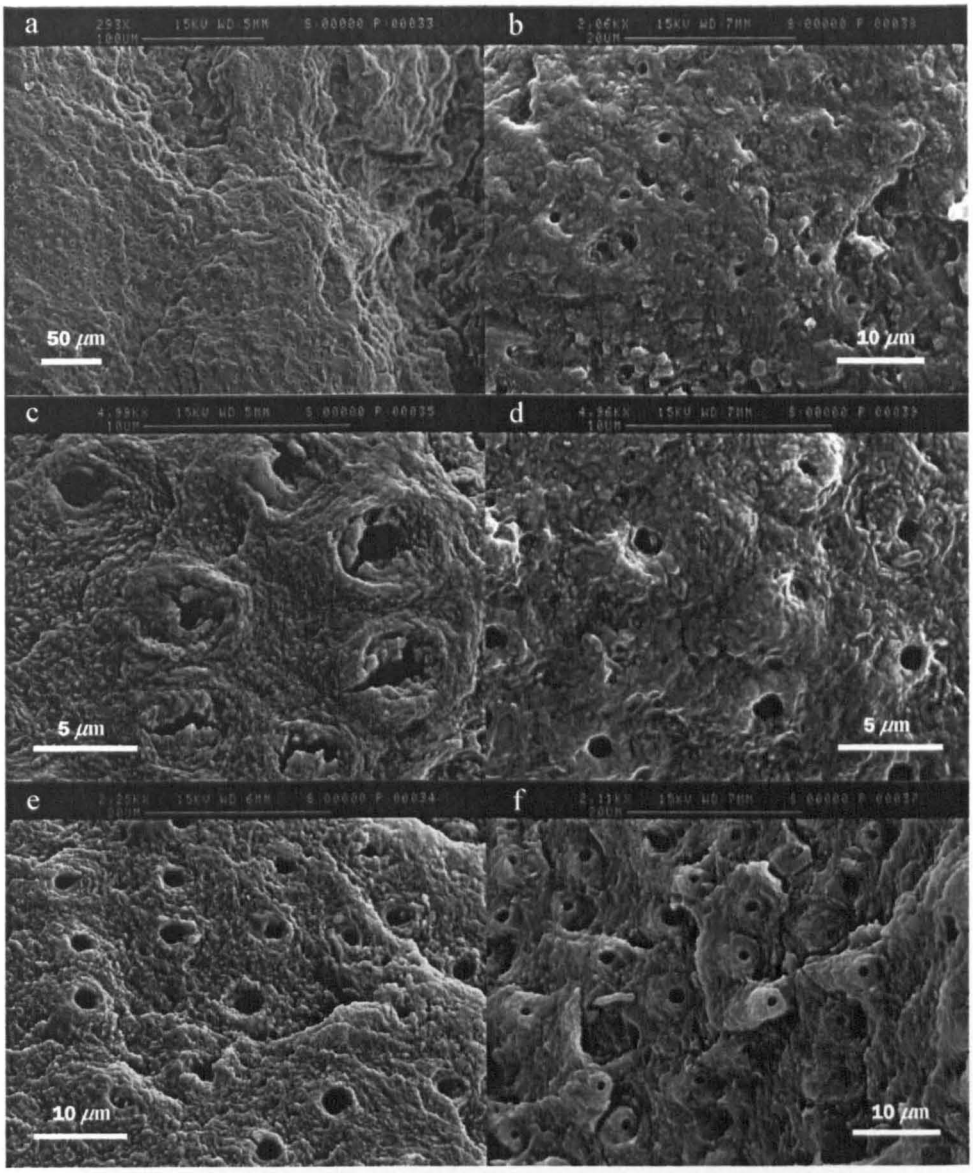


Figure 3.18 Representative SEM micrographs of dentine surface irradiated at the 4.5 watts power setting and followed by Dentine Conditioner (**a,c,e**) and Ketac Conditioner (**b,d,f**); **a**) showing the scaly dentine surface **b**) showing melt intertubular dentine covering the dentinal tubules **c**) the elevated peritubular dentine **d**) cuff like appearance of peritubular dentine **e**) An absence of intertubular dentine **f**) A presence of the depletion of the intertubular dentine.

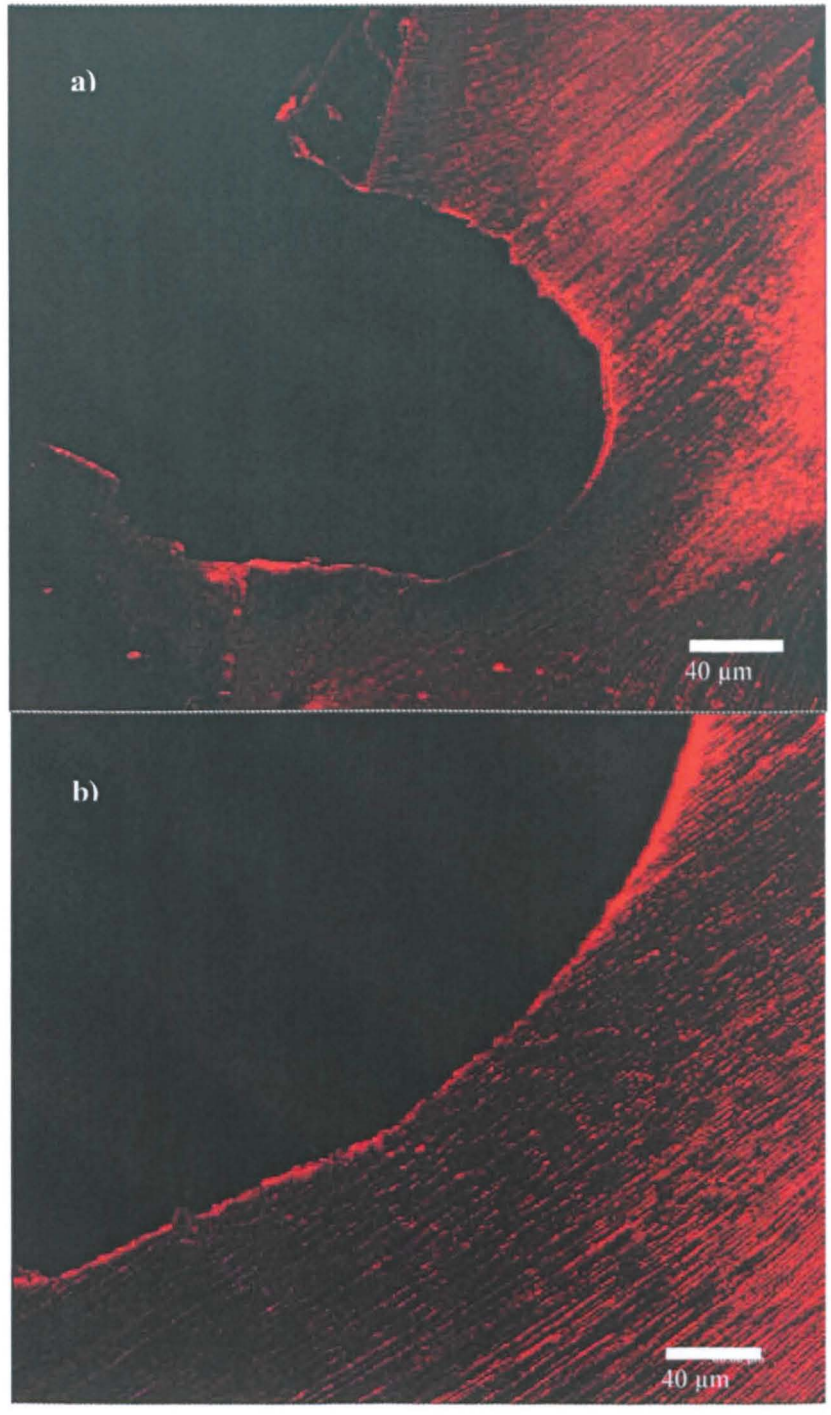


Figure 3.19 CLSM micrograph of Class V bur prepared cavity **a**) x5 lens, **b**) x40 lens/0.85NA.

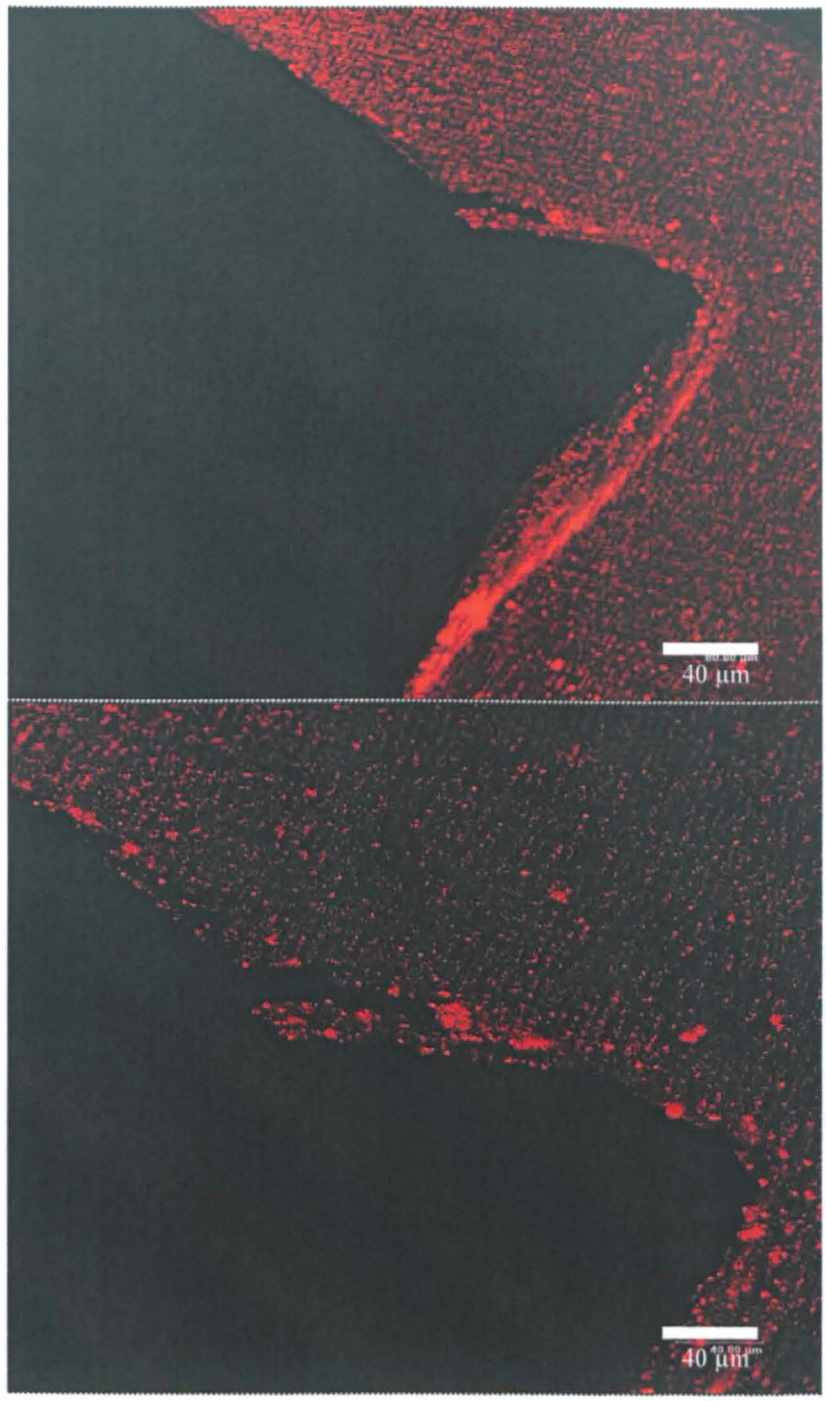
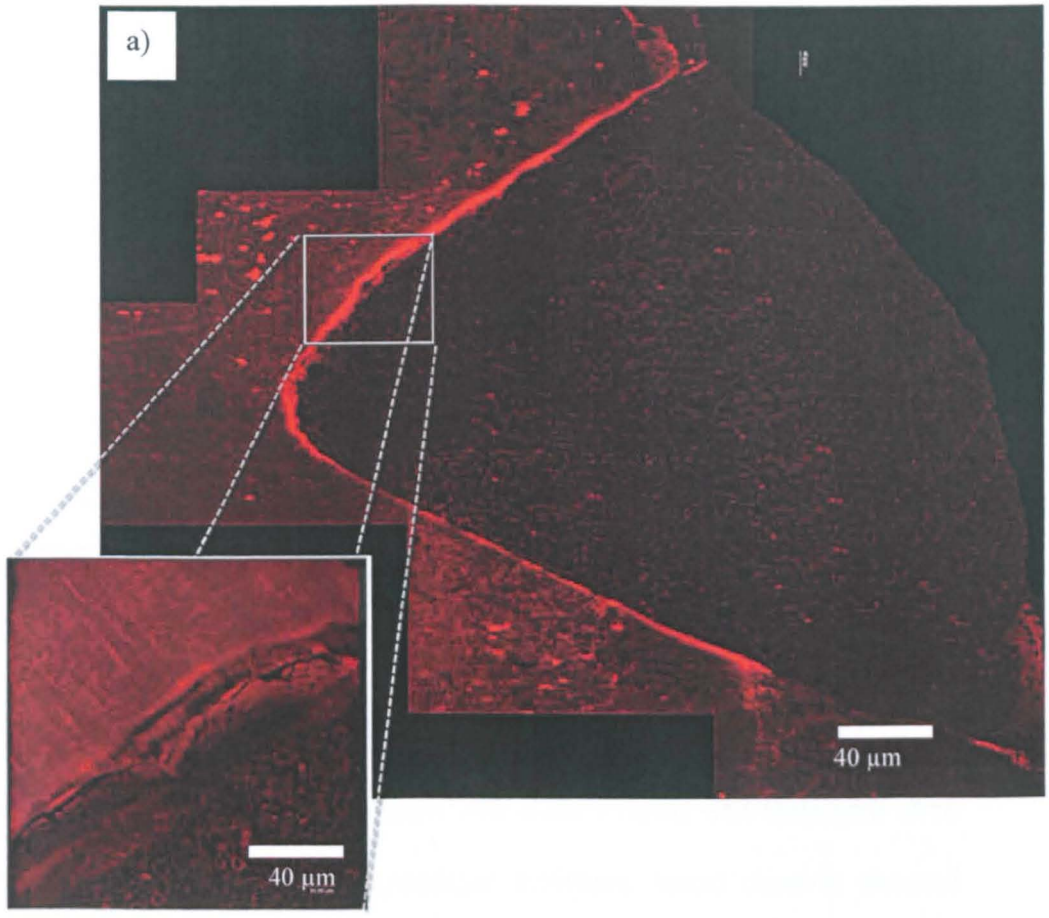


Figure 3.19 CLSM micrograph of Class V laser prepared cavity **c**) x 5 lens, **d**) x40 lens/0.85 NA.

Confocal microscopy

The CLSM micrographs of laser prepared cavities indicated a surface that was corrugated, rough and wavy (Figures 3.19 c and d) compared to bur prepared cavities (Figures 3.19 a and b). In general, the laser prepared surfaces showed more cracking and fissuring of dentine.



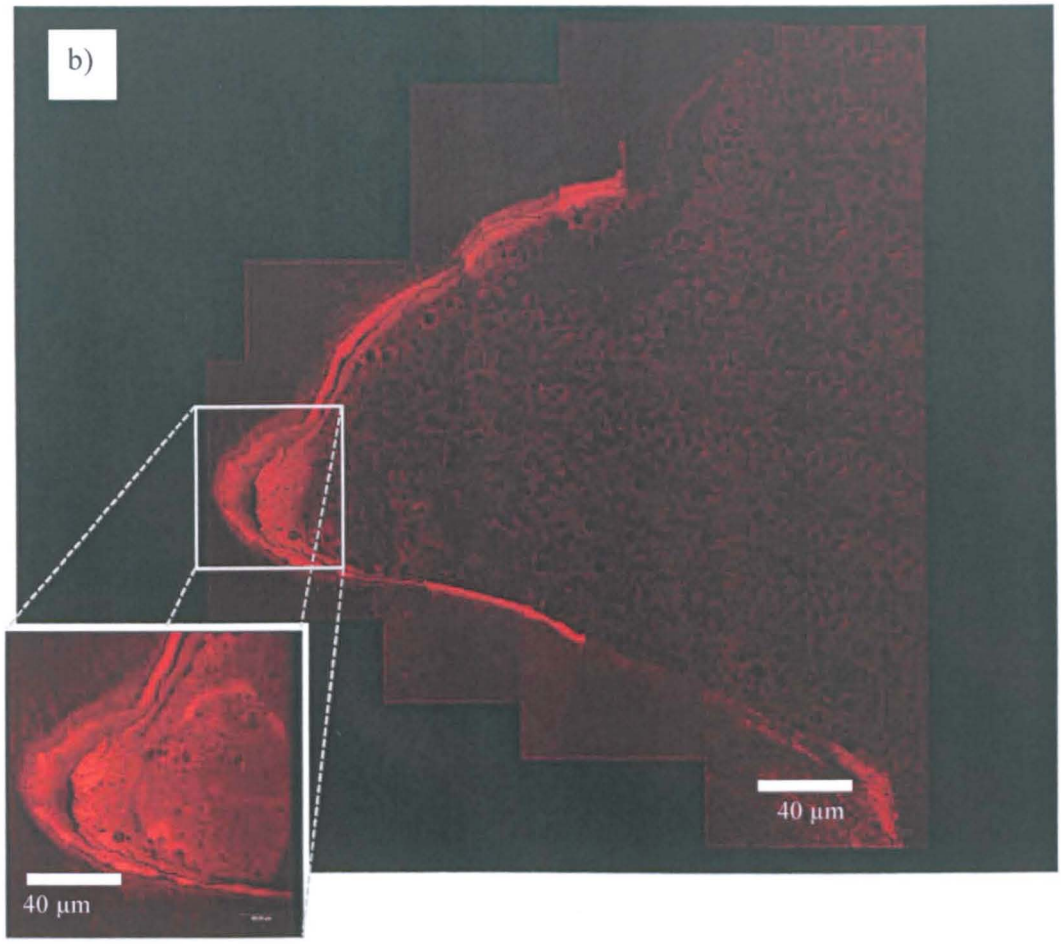


Figure 3.20 CLSM montage of Class V cavity filled with Fuji IX prepared by (A) bur instrumentation and (B) Cr,Er:YSGG laser

Forty-seven CLSM images from six surfaces were surveyed by the montage technique to determine average gap width.

After combining each image, the entire class V cavity was displayed as in Figure 3.20. At the material/dentine interface, lased dentine showed gap formation at the innermost part of the cavity, while the bur prepared specimens displayed gap formation closer to the dentine/enamel junction.The gaps with lased dentine at the inner surfaces were

continuous and extended over a longer portion of the interface. Gap formations in the bur prepared cavities occurred mostly at the outer part of the cavity and were over a shorter distance. The average maximum gap width between the glass-ionomer and lased dentine was approximately 18.66 (SD:4.38) μm and that of bur prepared dentine was approximately 8.83 (SD: 0.03) μm.

3.4 Discussion

3.4.1 Ablation efficiency evaluation

The ablation efficiency can be defined in terms of the amount of dental tissue removed within a given time [Kim et al., 2005], volume removed per joule of energy incident on the tissue (mm^3/J) [Fried, 2000], and volume ablated per sec (mm^3/s) [Featherstone and Nelson, 1987]. This study reported the ablation efficiency of dentine in terms of volume ablated per sec (mm^3/s). It is in agreement with the work by Rizoiu *et al* (1994). They showed a linear correlation between the volume ablated per sec and the average power of a 2.8 μm erbium doped laser.

The Er,Cr:YSGG laser and the Er:YAG laser both use water mediated ablation [Fried, 2000]. Water plays an important role for dentine ablation. Meister et al (2006) found that in dehydrated moist dentine, the quantity of ablated volume was significantly lower compared to dentine containing water for the Er:YAG laser, while the Er,Cr:YSGG laser

showed superficial melting and a deep carbonisation zone when used without a water spray. Exogenous water has a greater affect than endogenous water on the dentine ablation of the Er,Cr:YSGG laser.

There was no significant relationship between increasing laser output and reduction of dentine hardness (R^2=0.00, $p>0.05$). The stiffness of irradiated dentine was not significantly different from the baseline (paired t-test, $p>0.05$).

3.4.2 Structure and properties of laser prepared dentine

Dentine can be classified as a composite material comprising a collagen matrix (20% by weight) dispersed in an inorganic component (70% by weight) [Mahoney et al., 2000; Anker et al., 2003]. Hydroxyapatite is the main mineral content of the inorganic component, and variants (such as fluoroapatite) provide strength and hardness for the dentine. Elasticity and stiffness are obtained from the organic collagen matrix and water content (10 % by weight). SEM micrographs of the irradiated dentine in this study showed depletion of intertubular dentine (Figure 3.6d) similar to the dentine surface after preparation by the Er:YAG laser and treated with glutaraldehyde [Kameyama et al.,2001]. The peritubular dentine still remained, indicating more resistance to laser energy. This can be explained by the fact that the peritubular dentine has a high mineral content and lacks collagen as an organic matrix while intertubular

dentine has a high content of the collagen matrix (92 % by weight) [Kameyama et al., 2001]. The collagen matrix is rich in water content and laser energy is likely to be absorbed by the dentinal fluid within each tubule and the intertubular dentine more than the peritubular dentine. This study confirms the findings of previous studies that the laser-irradiated dentine was deprived of a smear layer and the orifices of dentinal tubules were opened [Hibst and Keller, 1989; Hossain et al., 2003].

The microhardness of laser-irradiated dentine decreased after laser irradiation by the order of 30-50%. However, the elastic modulus after laser irradiation was not significantly changed (Table 3.3). In contrast, investigations by Hossain et al. (1999) showed that the Knoop hardness of the cavity prepared by a Er,Cr:YSGG laser was not significantly different from bur preparation. Due to the fusion of the enamel surface after CO_2 and Nd:YAG laser irradiation, the Knoop hardness increased [Marquez et al.,1993]. Other studies revealed that higher energy laser irradiation actually decreased the microhardness of dental hard tissue significantly due to the increase in temperature [Kuramoto et al., 2001]. A previous report mentioned that all the lasers that produce some thermal effect could induce alteration in physical and chemical composition [Lee et al., 2003]. It was found that when heating dental hard tissue to a temperature of over 400°C, the mineral decomposes to

form a new mineral phase which enhances the resistance to acid dissolution [Featherstone and Nelson, 1987; Fried, 2000]. Plotting microhardness against elastic modulus after laser application is likely to broaden distribution on both axes. It is similar to the trend lines between microhardness and the elastic modulus of sound dentine and carious dentine [Angker et al., 2005]. This indicated that there is potentially a progressive deterioration of hardness and elastic modulus of dentine after laser irradiation. There is a correlation between the mechanical properties and mineral content in tooth tissue [Senawongse et al., 2006]. The chemical and thermal analysis of laser-irradiated dentine should be studied further.

SEM-EDX (Figures 3.13 and 3.14 a, b, c) confirmed that the glassy molten substance on the surface at higher power settings was composed of the same elements as hydroxyapatite. This phenomenon indicates that some components of dentine may exhibit thermoplastic properties, turning to a liquid on heating and immediately returning to a solid state on cooling with a water spray. Irrespective of the laser system used, the melted and resolidified dentine was a frequent finding when certain energy densities were exceeded [Kreisler et al., 2003].The Er,Cr:YSGG laser radiation is highly absorbed not only by the hydroxyapatite of dentine but also by the protein and lipid in collagen fibres [Fried et al., 1996]. This kind of a pulsed laser causes a rapid

heating and evaporation of the collagen matrix in dentine, firstly, due to the low ablation threshold compared to hydroxyapatite and secondly, due to the gradients of high pressure resulting from evaporation in the ejection of hydroxyapatite [Altshuler et al., 2001].

This is in accordance with previous studies that reported molten and crystalline like structures on irradiated dentine [Serebro et al., 1987; Malmstrom et al., 2001; Aminzadeh et al., 1999].

As widely described in the literature, the morphological of dentine after Er,Cr:YSGG laser irradiation showed a molten lava-like appearance with many micropores [Hossain et al, 1999]. The laser can melt and seal the dentinal tubule [Re et al., 2004]. In present study, the fiber like tag was observed on the irradiated dentine surface. This may be attributed to change in temperature and pressure which is below the melting temperature of dentine composites duet to the low fluence at power setting of 3 Watt.

An understanding of the mechanical and physical properties of prepared dentine after different clinical procedures may help to ascertain the optimal conditions to enhance the adhesion of dental restorations. This study is an initial investigation aimed at presenting the mechanical properties of laser treated dentine when using different laser

parameters. Further studies will focus on the bonding of dental materials to laser irradiated dentine.

3.4.3 Interface of laser prepared dentine and glass ionomer cement

Previous studies have attempted to evaluate cavity adaptation using a dye method for microleakage measurement. Niu et al. (1998) revealed that there was no significant difference in microleakage between the cavities prepared by laser and those prepared by the conventional method. In this study, confocal microscopy provided topographical micrographs of wedge-shaped Cl V cavities prepared by Er,Cr:YSGG laser and a rotary cutting instrument. It was noted that the cavities prepared by this laser showed gap formation in the inner surface of the cavity. Lased enamel provided good adaptation in comparison to lased dentine. These results correlate with an earlier study by Roebuck et al. (2000). They found that dentine leakage was significantly greater than that seen at the enamel margins for all test groups with conventionally prepared or lased cavities. Similarly, the study by Hanig and Stiesch-Scholz (2000) stated that laser treatment did not improve the marginal adaptation of Class II compomer and composite resin restorations placed in primary teeth with proximal margins located in dentine. In the present study, cavities prepared with the Er,Cr:YSGG laser provide good cavity adaptation with glass-ionomer at the enamel interface. However, the GIC/lased dentine interface showed poorer adaptation compared to

dentine prepared by the diamond bur. The investigation of GIC/tooth interface by confocal microscopy has shown that there was no presence of the hybrid-like layer at the interface.

The direction of dentinal tubules at the cervical area is different from those at the occlusal area. Therefore, the depletion of intertubular dentine in the laser prepared cavity was in a diagonal direction (at the bottom of the Cl V cavity). These appear to be undercut and which glass cannot penetrate into to accomplish good adaptation.

In this experiment, the laser produces long steep holes that the cement cannot fill so the gap width increases. Also, the standard deviation of gap width was in the wide range because of the uneven length of long steep holes on the laser irradiated dentine surface.

3.5 Summary

- Based on this study, the depth of the dentine lesion caused by laser irradiation depends on the power setting at a given amount of water and air output. The appearance of laser irradiated dentine at different power settings showed unique characteristics.

- The null hypothesis is rejected. The microhardness of laser irradiated dentine surface reduces by around 30-50% compared

to sound dentine. It did not depend on an increase in power setting.

- The investigation along a Class V cavity prepared by an Er,Cr:YSGG laser demonstrates good adaptation with glass ionomer cement at the enamel surface. However, the glass ionomer cement/laser dentine interface shows poor adaptation compared to dentine prepared by diamond bur.

CHAPTER 4
ADHESION TO LASER PREPARED DENTINE

4.1 Background

Alternative methods of tooth cutting and preparation are now available using laser technology. It is almost a decade since the erbium laser was accepted for tooth cutting in 1997 [Harashima et al., 2005]. Many investigations reported its ability to cut enamel and dentine effectively [Harashima, et al., 2005; Wigdor et al., 1995; Hadley et al., 2000; Featherstone and Nelson, 1987; Hoke et al., 1990; Hibst and Keller, 1989]. Interestingly, the laser irradiated dentine surface was found to be very rough, with irregularities and craters resembling the surface obtained by acid etching [Usumez et al., 2003; Usumez and Aykent, 2003; Silberman et al., 1994]. Hence, these irregularities may encourage the adhesion between restorative materials and the tooth substrate. Therefore, lasers for tooth drilling may be beneficial to enhance the adhesion of restorative material.

The good adhesion between restorative materials and the tooth surface leads to longevity of the restoration. Many dental adhesive filling materials, both salt-based and resin-based, have been developed to interact with the bur-prepared tooth substrate and there is a need to

confirm that such materials will interact in a similar way with laser-prepared tissue. Glass ionomer cement has the ability to bond chemically to the tooth substrate without using an intermediary adhesive system unlike composite resin materials [Hotz et al., 1977].

Using rotary cutting instruments, a debris zone, called the "smear layer", develops on the dentine surface [Eick et al., 1997; Eick et al., 1970; Barros et al., 2005]. This layer obliterates the dentinal tubules resulting in low adhesion efficiency of tooth and restoration. To enhance adhesion to tooth, it is recommended that an acidic conditioner applied on the tooth surface helps to remove the smear layer before tooth restoration [Jacuques et al., 2005; Burrow et al., 2002]. However, using the laser to cut the tooth may promote the bonding of restorative material as the laser-cut dentine provides a clean surface with opened dentinal tubules suitable for chemical and mechanical bonding [Lin et al., 1999].

In general, a common method employed to evaluate the adhesion of a material to a dentine or enamel surface is to determine the tensile or shear stress applied to the bonded specimen. The microtensile test uses a very small surface area, which is believed to have fewer defects occurring at the material/tooth interface. Therefore, this can minimize the variation among samples and provide an accurate method of evaluating the adhesive strength [Burrow et al., 2002].

Numerous studies have been conducted to assess the bond strength of composite resin and adhesive to laser-prepared dentine and enamel [De Munck et al., 2002]. However, there is a lack of studies dealing with glass ionomer in cavities prepared by the Er,Cr:YSGG laser.

This study aims to measure the bond strength of glass ionomer cement with laser irradiated dentine. The null hypothesis was that there was no difference in the bonding to non-lased and lased lased dentine, and that the dentine conditioner does not influence the adhesion of glass ionomer cement to this substrate.

4.2 Microtensile bond strength measurement

4.2.1 Materials and Method

Two conventional glass ionomer cements (GICs) were used Fuji IX (GC Co., Japan) and Ketac Molar (3M ESPE, Germany). The conditioners used were Dentine Conditioner with Fuji IX and Ketac Conditioner with Ketac Molar supplied by the respective companies (Figure 4.1).

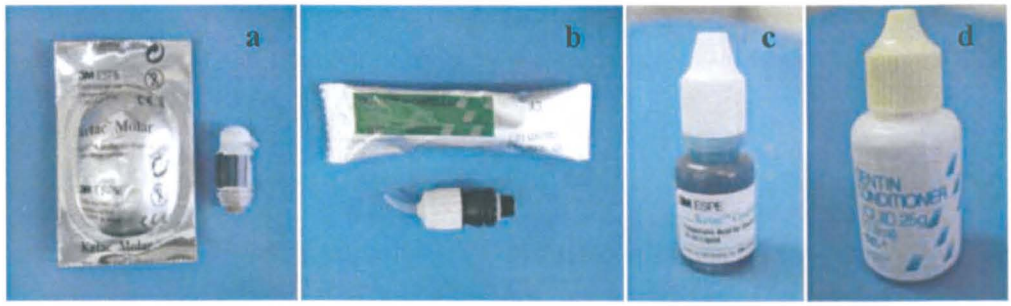


Figure 4.1 Conventional GICs and Dentine conditioner used for this study; a) Ketac Molar (Maxicap, 3M ESPE, UK) b) Fuji IX (GC corp., Japan) c) Ketac Conditioner (3M ESPE, UK) d) Dentine conditioner (GC corp., Japan)

The teeth used in this study were extracted human molars and premolars without caries or defects, stored in an aqueous solution of 0.5% chloramine T no more than 6 months from time of extraction to inclusion in this study.

The laser system and a high speed handpiece used for this study were the same as described in Chapter 3. The repetition rate was 20 Hz and power output was set at 5.5 Watts. The setting of air and water at the control panel were set at 65% and 55% respectively. The pulse energy varies from 0 to 300 mJ. A clear sapphire tip of 600 μm was used.

Specimen preparation

One third of the occlusal surfaces were removed with a high-speed diamond saw (Micro Slice 2, Metals Research Limited, Cambridge England) using water spray, and subsequently ground with 600 grit sand paper (P-800) to provide flat dentine. The cut surface was finished using precision lapping and a polishing machine (PM2A, Logitech, Materials

Technologists Engineers) with calcined alumina oxide powder of 3 μm particle size. A laser handpiece was secured to a clamp to insure that the tooth surfaces were prepared in a standard pattern. The mounted tooth specimen was placed on the removable stand at the working of 1 mm. The dentine surface was irradiated by Er,Cr:YSGG laser by moving the specimen along the part of laser beam and repeating at the same direction throughout the surface. A high speed handpiece was fixed to support as same as laser handpiece. The simple pattern of bur preparation was made by moving specimen up and down over the dentine area. Eight of the trimmed teeth were prepared by laser and the other eight teeth prepared by bur. In half of the laser and bur prepared groups, the relevant dentine conditioner was applied to the dentine surface according to the manufacturer's instructions (application time = 20 seconds, water rinse and dry with air spray). Fuji IX and Ketac Molar capsules were mixed with a triturator (Silamat, Vivadent, Chann, Liechtenstein) and a Tofflemire matrix holder was used to build up the glass-ionomer restoration. The experimental specimens were divided into eight groups.

The experimental groups were defined as following:

Group 1: Bur preparation and Fuji IX restoration

Group 2: Bur preparation and Ketac Molar restoration

Group 3: Bur preparation, Dentine Conditioner application and Fuji IX restoration

Group 4: Bur preparation, Ketac Conditioner application and Ketac Molar restoration

Group 5: Laser preparation and Fuji IX restoration

Group 6: Laser preparation and Ketac Molar restoration

Group 7: Laser preparation, Dentine Conditioner application and Fuji IX restoration

Group 8: Laser preparation, Ketac Conditioner application and Ketac Molar restoration

After 24 hours storage in deionised water at a constant temperature of 37°C, each tooth was sectioned perpendicular to the adhesive interface two times with a high-speed diamond saw with water spray to produce typical microtensile bar shaped specimens (approximately: 1 x 1 mm^2 of adhesive surface). There were 20 specimens in each group.

Microtensile bond strength measurement

The dimensions of the specimens were measured using a digital vernier callipers (Digimatic, Mitutoyo, Japan). The specimens were mounted in a SSP (Single speed pump or SSP Dillon, Compact Gauges 200N, a division of Weigh-Tronix Inc, Fairmont MN, USA) using a cyanoacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA) and tensile forces were applied at a crosshead speed of 1mm min^{-1} until failure occurred (Figure 4.2). The microtensile bond strength was calculated in MPa, the unit is derived from dividing the tensile force (N) at the time of

fracture by the individually measured bond area (mm^2). The mean bond strengths were evaluated by one-way ANOVA and Tukey's comparison, a p value<0.05 was considered to be significant.

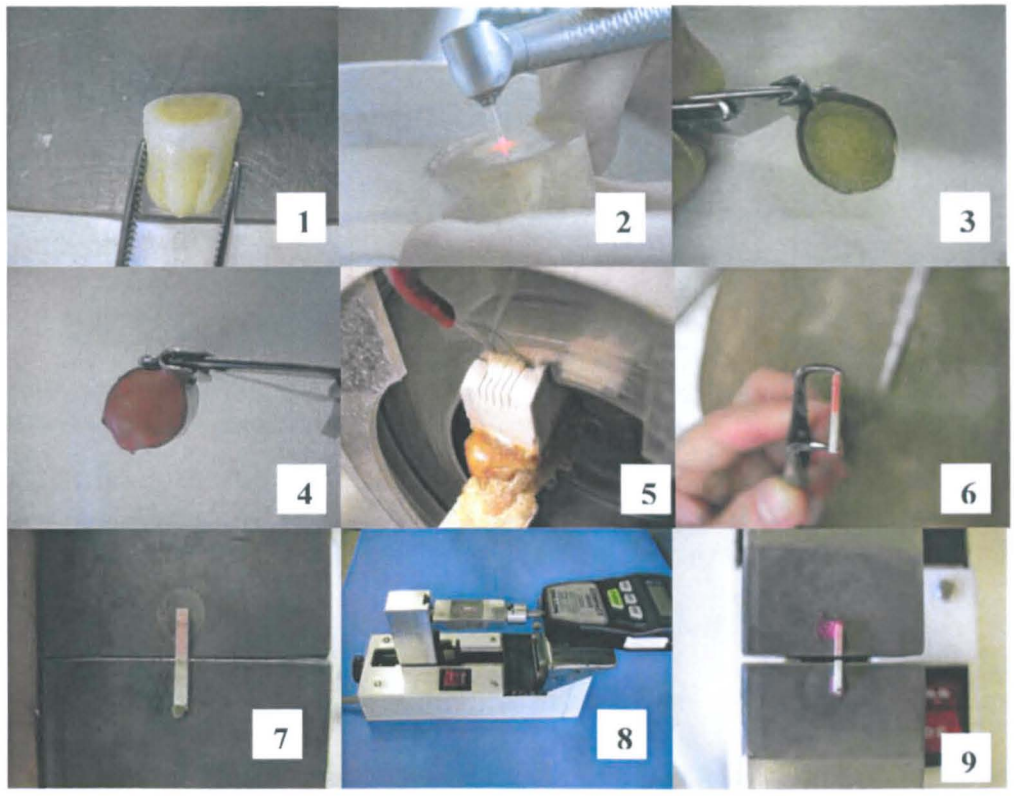


Figure 4.2 Sample preparation for microtensile bond strength test: (1) The occlusal one third of human molars were cut until dentine was exposed; (2) The ground and polished dentine was prepared either by the laser or bur with or without the reverent dentine conditioner; (3) Core build up by using matrix holder; (4) Glass ionomer core build up (5) The restored tooth was embedded in plaster of paris and cut by high speed diamond saw (6) After 24 h, the tooth was mount with plaster of Paris and cut into the stick-shape specimens with dimension 1x1 mm^2; (7) the specimen was attached to the testing device with Zapit glue; (8) the tensile force was applied at a cross head speed of 1mm/min.

SEM analysis of dentine surface

Scanning electron microscopy was used to examine the dentine surface with different preparations in the microtensile bonding study. Four dentine discs were prepared with each of the following surface treatments: (1) bur preparation (2) bur preparation following by Dentine Conditioner (3) Laser preparation (4) Laser preparation following by Dentine Conditioner.

Fracture analysis

After the bond strength tests, all specimens were assessed for the mode of failure using a light microscope (Meiji Techno. Co. Ltd., Saitama, Japan) at a magnification of x22.5. The individual failure modes were noted. Representative specimens from each group were further examined using the Stereoscan S240 Scanning Electron Microscope (Cambridge, UK).

4.2.2 Results

Microtensile bond strength

The results of the microtensile bond strength test are shown in Table 4.1. The mean microtensile bond strength of GIC with bur preparation of dentine ranges from 3.92 - 5.52 MPa. For laser prepared dentine, the mean microtensile bond strength is in the range of 5.61 - 8.62 MPa.

Table 4.1: Microtensile bond strength (MPa); mean values with the same superscript letter were not significantly different (p>0.05).

Conditioner	Fuji IX		Ketac Molar	
	Bur	Laser	Bur	Laser
No conditioner	4.56 (2.19) [a]	5.99 (1.70) [a]	3.92 (1.98) [a]	5.61(1.55) [a]
Dentin Conditioner	5.52 (1.68) [a]	8.62 (3.86) [b]	-	-
Ketac Conditioner	-	-	5.52 (1.52) [a]	8.01(3.26) [b]

Statistical analysis using ANOVA showed that there was no significant difference in microtensile bond strength of GIC between laser-prepared dentine and bur-prepared dentine. However, laser-prepared dentine treated with a dentine conditioner provided significantly higher microtensile bond strengths than bur-prepared dentine with or without using the dentine conditioner ($p<0.05$). Comparing the different GICs, there was no significant difference between using Ketac Molar and Fuji IX without dentine conditioner for filling the prepared tooth by either bur or laser (Figure 4.3).

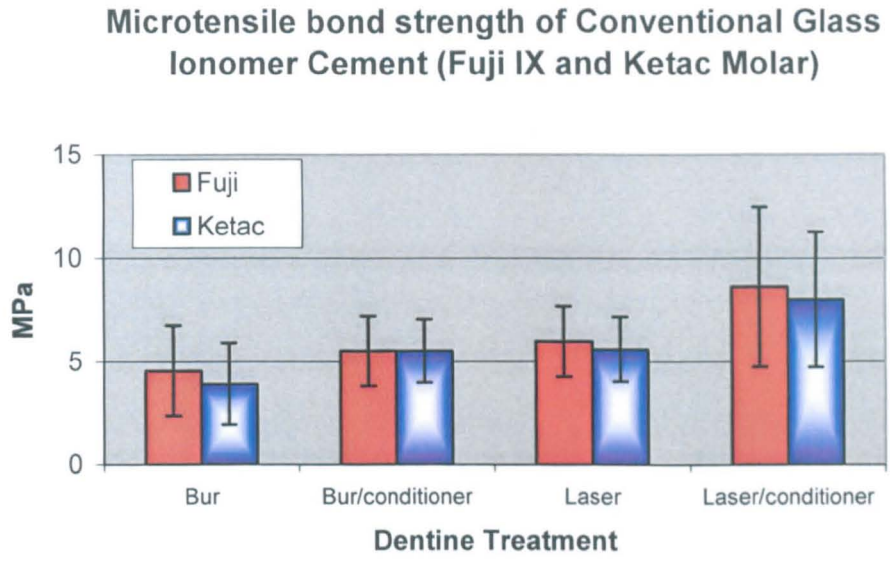


Figure 4.3 The mean values of microtensile bond strength of Fuji IX and Ketac Molar with different dentine treatments

It is showed that the combination of tooth preparation method and surface conditioner did significantly affect the microtensile bond strength. Therefore, the microtensile bond strength of GIC increased when using the laser preparation and the dentine conditioner.

SEM analysis of dentine surface

Dentine surfaces prepared with various preparations are shown in Figure 4.4. The bur preparation produced the smear layer covering the dentine surface (Figures 4.4 a). The laser prepared dentine was rough and with no smear layer (Figure 4.4 c). After applying the dentine conditioner to the laser cut dentine, the dentinal tubules were clearly seen and opened widely (Figure 4.4 d) as comparing to bur cut dentine with conditioner (Figure 4.4 b).

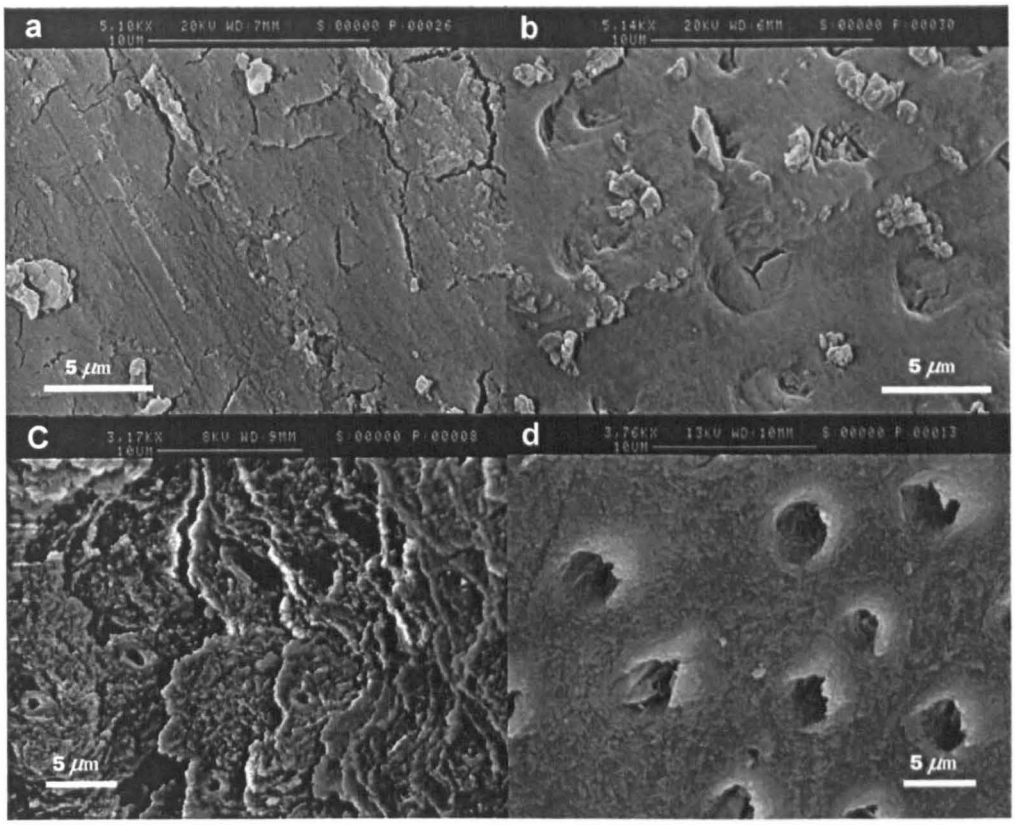


Figure 4.4 a) Bur prepared dentine, b) Bur and dentine conditioner prepared dentine c) Laser prepared dentine d) Laser and conditioner prepared dentine

Fracture analysis

Examination of the fractured specimens under the light microscope revealed different patterns of failure: adhesive, cohesive and a mixed type of failure (Table 4.2, Figures 4.5, 4.6 and 4.7). In bur-prepared dentine without dentine conditioner, the failure mode for all specimens was adhesive failure. In laser-prepared dentine, the mode of failure was cohesive only or a mixed failure.

Table 4.2: Different failure modes of the specimens expressed as a percentage of the total. The actual number of specimens for each mode of failure is in parentheses.

Test group	Adhesive failure	Cohesive failure	Mixed failure
Group 1	100 (20)		
Group 2	100 (20)		
Group 3	25 (5)	40 (8)	35 (7)
Group 4	95 (19)		5 (1)
Group 5		80 (16)	20 (4)
Group 6		80 (16)	20 (4)
Group 7		75 (15)	25 (5)
Group 8		90 (18)	10 (2)

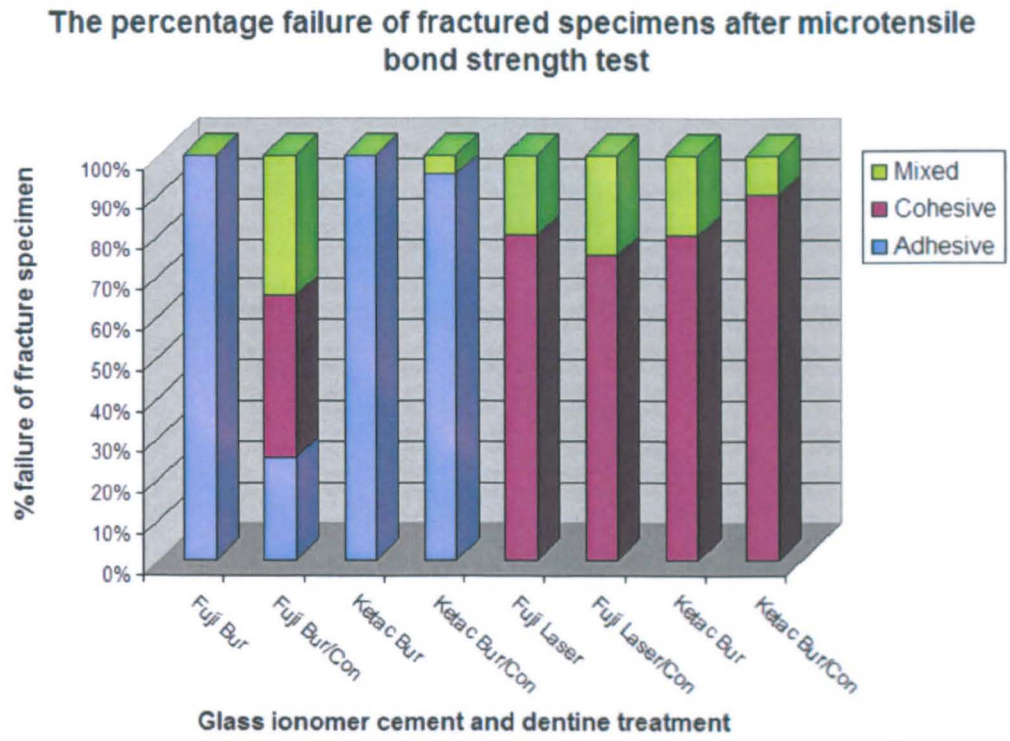


Figure 4.5 Mode of failure noted as percentage failure from microtensile bond strength test of conventional glass ionomer cement to different prepared dentine

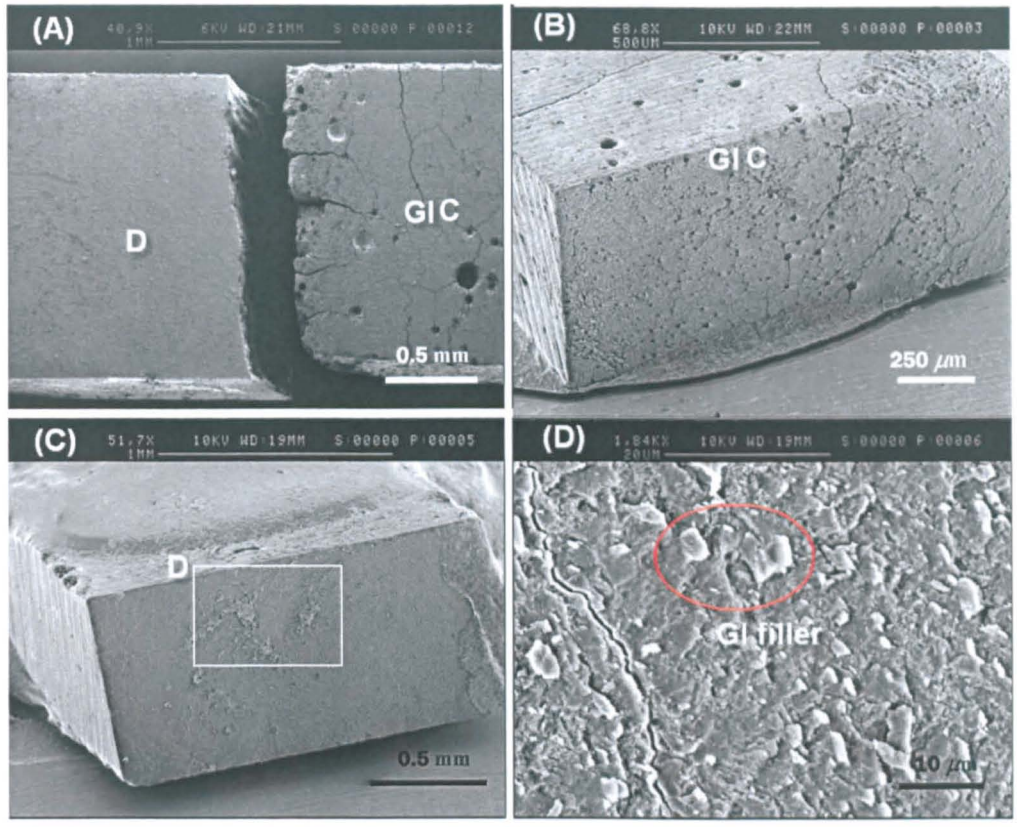


Figure 4.6 Representative SEM micrograph of a fractured specimen of glass ionomer (GIC) on dentine surface (D) prepared by a bur:
(A) A typical fractured specimen exhibiting adhesive failure.
(B) The glass ionomer side of the fractured specimen.
(C) The dentine side of a specimen with mixed failure, with some glass ionomer on the surface.
(D) Higher magnification of the area outlined in (C), showing glass ionomer fillers (GI filler).

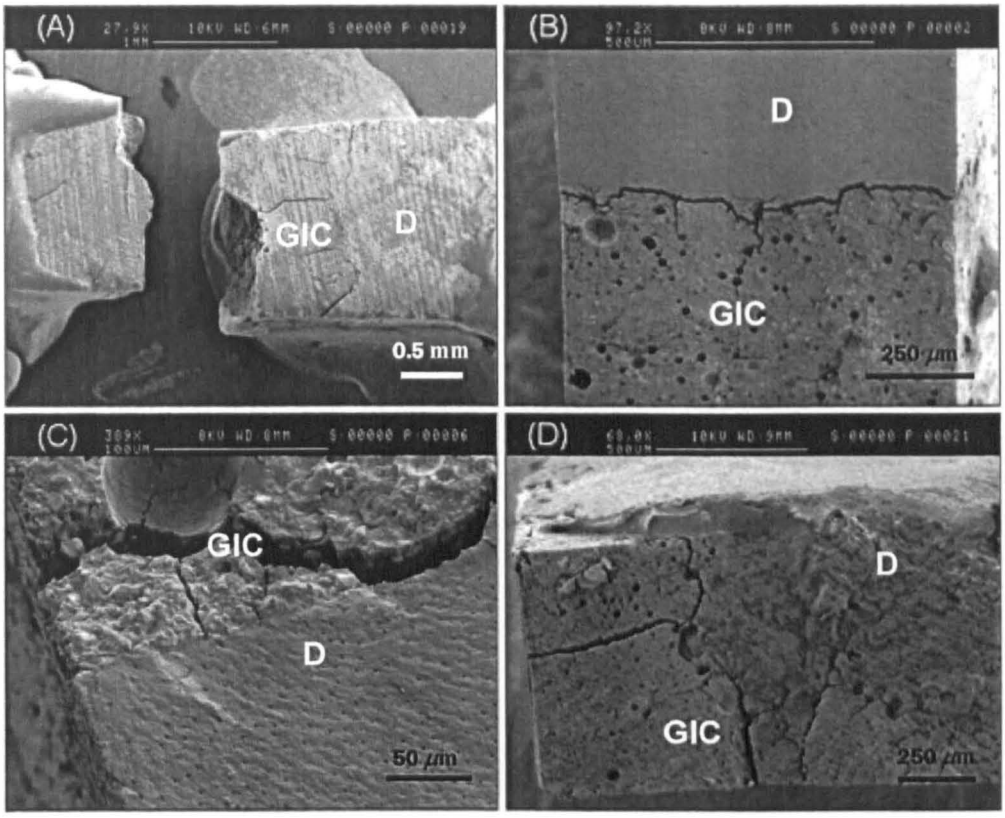


Figure 4.7 Representative SEM micrograph of fractured specimen of glass ionomer cement (GIC) on dentine (D) prepared by Er,Cr:YSGG laser:
(A) With conditioner, showing cohesive failure within the GIC only.
(B) Without conditioner in a specimen with a mixed type of failure.
(C) Higher magnification of a fracture specimen from partially cohesive failure specimen showing remnants of the GIC on the dentine surface in parts and adhesive failure in other areas.
(D) The GIC side of the same fractured specimen as in Figure 4.7(C), with areas displaying fragments of dentine (D).

4.2.3 Discussion

The purpose of using a dentine conditioner is to remove the smear layer and increase the wettability of the tooth substrate [Glasspoole et al., 2002]. The polyacrylic acid provides the carboxyl group for hydrogen bonding and which is later displaced by the stronger interaction of polar and ionic attraction from the GIC setting reaction [Glasspole et al., 2002; Hewlett et al., 1991]. A previous study found that the treatment of cut dentine with 25% polyacrylic acid (Ketac Conditioner) reduced the affinity of the smear layer for the dentine, leaving behind a poorly-attached layer of smear debris, rather than removing it [Hewlett et al., 1991]. Tanumiharja et al. (2000) reported that the use of surface conditioners produced no difference in the bond strength of Fuji IX GP for bur preparation which is in agreement with the results presented here.

In the present study, for the laser-prepared dentine, conditioner application increased the microtensile bond strength in both Fuji IX and Ketac Molar groups. This is probably because laser irradiated dentine has no smear layer and the dentinal tubules were unplugged with dentine debris. Also, the way in which lasers remove the tooth tissue is based on thermomechanical interaction [Meister et al., 2006]. The thermal effect from a laser causes changes in the water content of collagen fibrils [Bachmann et al., 2005]. The thermal stability of collagen was suggested to be a consequence for the dentine surface alteration

[Toledano et al., 2005]. This may increase the dentine surface wettability which is compatible with conventional GICs, resulting in higher bond strength. The water loss in collagen fibrils after laser irradiation may reduce the hydrophilicity of the dentine substrate which is suitable for water sensitive material such as GICs. The matrix of GICs can penetrate well on low hydrophilic laser-cut dentine. The mean microtensile bond strength of GIC to conditioned bur-cut dentine was not significantly different to laser-cut dentine. It can be explained by the fact that bur-cut dentine exhibited clean and visible dentinal tubules after applying dentine conditioner, which was almost similar to the laser-cut dentine [Hossain et al., 2003; Hossain et al., 1999; Ariyaratnam et al., 1999].

It was found that all specimens in bur-prepared dentine failed adhesively and this indicated that the GIC interface was an area of weakness. Cohesive and mixed failures were observed in bur-prepared dentine after the application of dentine conditioner and the bond strength was improved although not significantly different from unconditioned specimens. From SEM imaging on the dentine side of the fractured specimens in mixed failures, there was some GIC residue attached on the dentine surface (Figures 4.7C and 4.7D). From previous work, there was an interdiffusion zone of Fuji IX and conditioned dentine substrate without any tag formation into the dentinal tubules [Ferrari et al., 1997].

In the laser group, the fracture analysis showed that both cohesive and mixed failures occurred within the materials (Figure 4.4). It is likely that porosity within the cement becomes an unpredicted factor. It probably causes a stress concentration for the fracture to occur from this point [Yip et al., 2001]. This implies a tight bond between laser-treated dentine and GIC.

In this study, the number of pre-testing failure was not recorded. All pre-testing failure occurring during specimens' preparation as stick shape with the diamond saw did not included in bond testing. In order to draw the valid result, the pre-testing failure was recommend to be recorded. The microtensile bond strength of glass ionomer cement was relatively low. All of the data in this study relates to short duration storage of sample. It would be considered to extend the storage period before testing until complete setting of glass ionomer cement.

4.3 Shear bond strength

4.3.1 Materials and Method

Specimen preparation

40 occlusal cut teeth were embedded in cylindrical resin moulds (3 cm high; 3 cm diameter) and the end of each resin mould ground with P800 SiC paper, with water irrigation until a complete section of the occlusal dentine was exposed (Figure 4.8 (1)). In each tooth, three

different depths of dentine were used. The superficial dentine depth was prepared just into the dentine after the initial grinding. The middle dentine depth was exposed by a further 0.5 mm grinding, and the deep dentine depth was exposed by a further 0.5 mm grinding. A laser handpiece was secured to a clamp to insure that the tooth surfaces were prepared in a standard pattern. The mounted tooth specimen was placed on the removable stand at the working of 1 mm. The dentine surface was irradiated by Er,Cr:YSGG laser by moving the specimen along the part of laser beam and repeating at the same direction throughout the surface. A high speed handpiece was fixed to a support as same as laser handpiece. The simple pattern of bur preparation was made by moving specimen up and down over the dentine area.

Specimens were made by filling the shorter part of a gelatine capsule (Natural Transparent Hard Gelatine Capsules size 5 [lot No.C207821A], Hertfordshire SG4 9BT, UK) with GIC and the capsule placed carefully on the prepared surface using finger pressure (Figure 4.8 (4)). Any excess GIC was removed carefully. The experimental specimens were divided into the same 8 groups as used in the microtensile testing. After 24 hours storage, the material was left to set in distilled water at a temperature of 37°C and 100% relative humidity, test specimens were removed from the oven and cooled down to the room temperature.

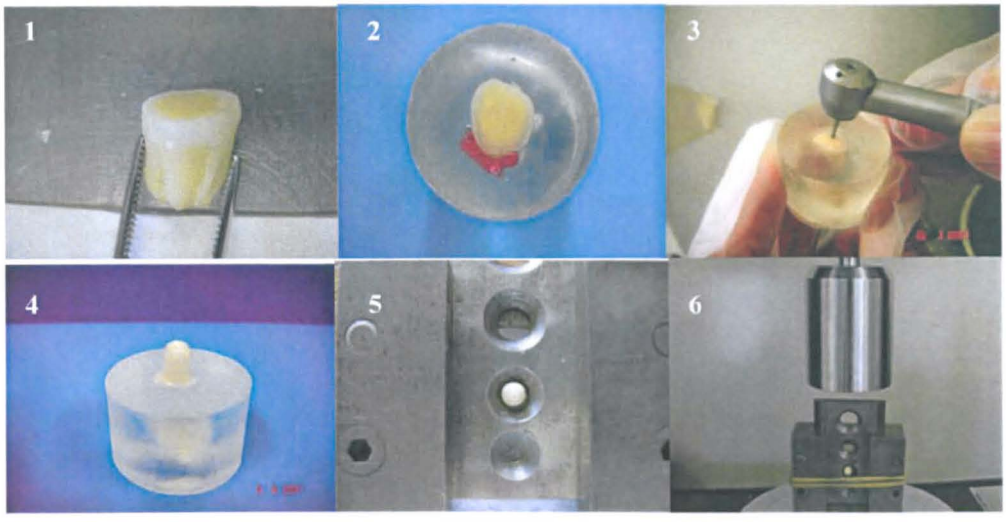


Figure 4.8 Shear bond strength measurement: (1) The occlusal one third was trimmed and (2) mounted on a clear acrylic mould. (3) The exposed dentine was prepared by either laser or bur (4) the glass ionomer cement in gelatine capsule was placed on the specimen (5) then the specimen was placed in a holder and (6) the shear force was applied at a cross head speed of 1 mm/min.

Shear bond strength testing

Each specimen was placed on the specimen holder of the universal testing machine (Instron 5567 series IX; Instron Corporation, London, UK). Compressive force was applied at a cross head speed of 1 mm/min until specimen failure occurred (Figure 4.8 (5-6)). The mean bond strengths were evaluated by one-way ANOVA and Tukey's comparison, a p value<0.05 was considered to be significant.

SEM analysis of dentine surface

Scanning electron microscopy was used to examine the dentine surface with different preparations in the shear bonding study. Six dentine discs were prepared with each of the following surface treatments:

(1) Bur preparation

(2) Bur preparation with Ketac Conditioner

(3) Bur preparation with Dentine Conditioner

(4) Laser preparation

(5) Laser preparation with Ketac Conditioner

(6) Laser preparation with Dentine Conditioner.

Fracture analysis

After the bond strength tests, all specimens were assessed for the mode of failure using the light microscope (Meiji Techno. Co. Ltd., Saitama, Japan) at a magnification of x22.5. The individual failure modes were noted. Representative specimens from each group were further examined using the Stereoscan S240 Scanning Electron Microscope (Cambridge, UK).

4.3.2 Results

Shear bond strength measurement

The results of the shear bond strength test are shown in Table 4.3, Figures 4.9 and 4.10. The combined mean shear bond strength of GIC with bur preparation of dentine ranged from 7.7- 11.8 MPa. For laser

prepared dentine, the mean shear bond strength was in the range of 9.5 - 11.3 MPa.

Table 4.3 Mean (SD) and combined mean (SD) shear bond strength (MPa) of 8 experimental groups at different dentine depths (S = Superficial dentine, M=Middle depth of dentine and D = Deep dentine)

Group	Dentine level	Shear bond strength (MPa)	
		Mean (SD)	Combined Mean (SD)
1	S	9.67 (4.44)	9.17 (3.46)
	M	8.50 (2.53)	
	D	9.35 (0.86)	
2	S	12.94 (2.92)	11.78 (5.49)
	M	10.22 (4.22)	
	D	12.17 (8.62)	
3	S	16.78(5.16)	9.54 (6.24)
	M	4.16 (1.47)	
	D	7.67 (1.32)	
4	S	13.99 (1.71)	11.28 (4.19)
	M	7.37 (1.31)	
	D	12.43 (5.18)	
5	S	6.73 (1.52)	7.71 (2.48)
	M	9.00 (3.45)	
	D	7.40 (1.96)	
4	S	5.44 (1.39)	9.25 (4.00)
	M	12.28 (4.20)	
	D	10.04 (2.45)	
6	S	6.81(1.88)	9.65 (4.55)
	M	13.00 (5.85)	
	D	9.15 (3.90)	
8	S	6.94 (1.41)	10.01 (3.46)
	M	11.11(4.56)	
	D	11.98 (1.97)	

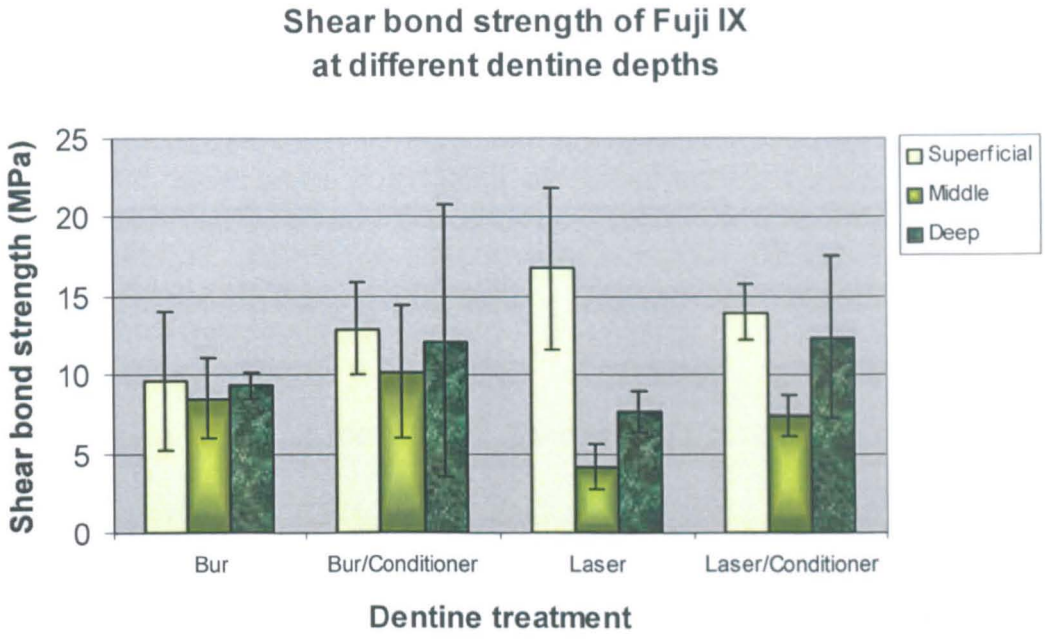


Figure 4.9 The mean values of shear bond strength of Fuji IX at different dentine depths

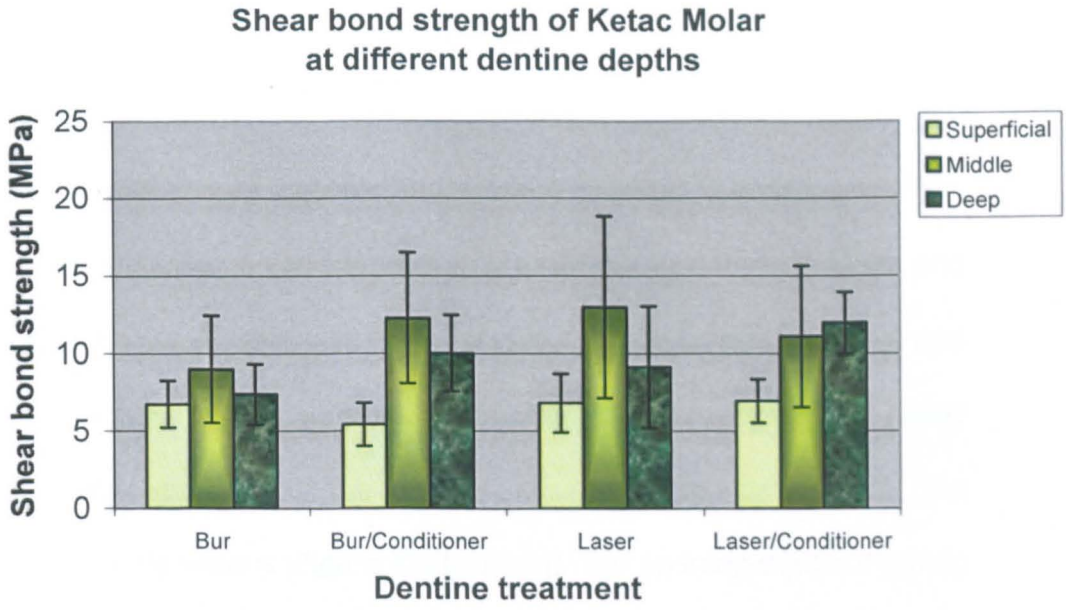


Figure 4.10 The mean values of shear bond strength of Ketac Molar at different dentine depths

Statistical analysis using ANOVA showed that there was no significant difference in combined shear bond strength of GIC between laser-prepared dentine and bur-prepared dentine. In comparison of the different GICs, there was no significant difference between using Ketac Molar and Fuji IX without the dentine conditioner for filling the tooth prepared by both methods.

The statistical analysis of mean shear bond strength among the depths of dentine showed that there was a significant difference in mean shear bond strength in laser prepared dentine both with and without conditioning for Fuji IX. In Ketac Molar restoration, there was significant difference in both laser and bur prepared dentine in the conditioned group.

SEM analysis of dentine surface

The Er,Cr:YSGG laser cut dentine surface showed a rough and flaky appearance compared to the normal bur cut dentine (Figure 4.11a and d). Furthermore, it revealed the depletion of intertubular dentine and cuffing of peritubular dentine. After Dentine Conditioner or Ketac Conditioner application, the degree of roughness decreased. The dentinal tubule widens (Figure 4.11e and f). The opening dentinal tubule of bur prepared dentine after Ketac Conditioner application (Figure 4.11b) was seen clearer than when using Dentine Conditioner (Figure 4.11c).

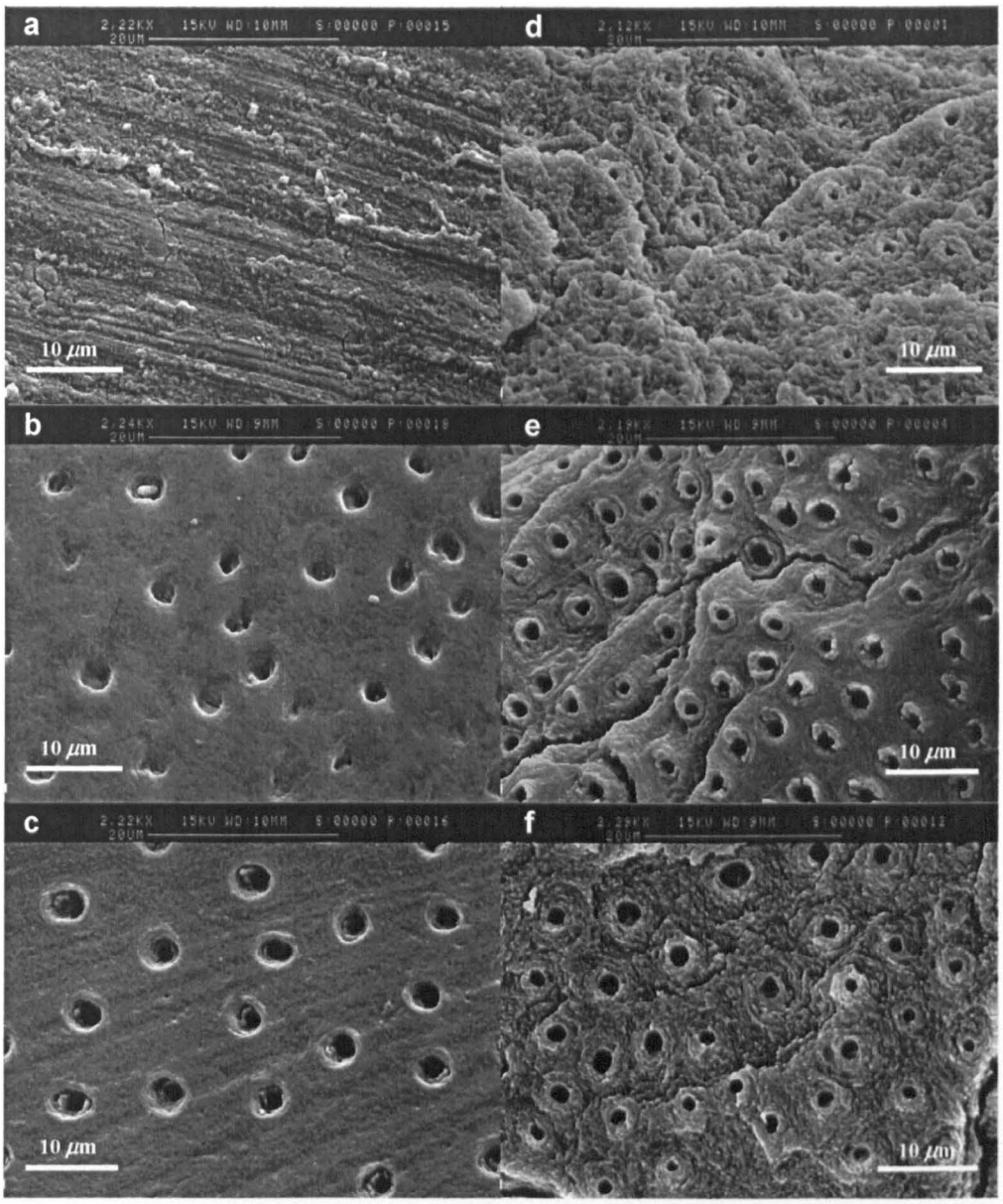


Figure 4.11 SEM micrograph of dentine (a) bur preparation (b) bur preparation and Dentine conditioner application (c) bur preparation and Ketac conditioner application (d) laser preparation (5.5 W, 95%air, 80% water) (e) laser preparation and Dentine conditioner application (f) laser preparation and Ketac conditioner application.

Failure Analysis

The modes of failure for the specimens of each group are presented in Table 4.4 and Figure 4.12. For each group, the majority of failures were mixed failures (Figure 4.13). There was no cohesive failure within the restorative materials or within dentine alone. The mixed failure occurred in laser prepared dentine more than in bur prepared dentine but was not significantly different for both Fuji IX and Ketac Molar. At the superficial dentine depth, laser prepared dentine with or without dentine conditioner in Fuji IX showed only a mixed failure mode (0% in adhesive failure mode). At the middle level, it showed more adhesive failure both in bur and laser prepared dentine. At the deep dentine depth, the laser prepared dentine with dentine conditioner in Ketac Molar showed a mixed failure mode. No additional pre-testing failure was produced during the specimen preparation for shear bond strength testing.

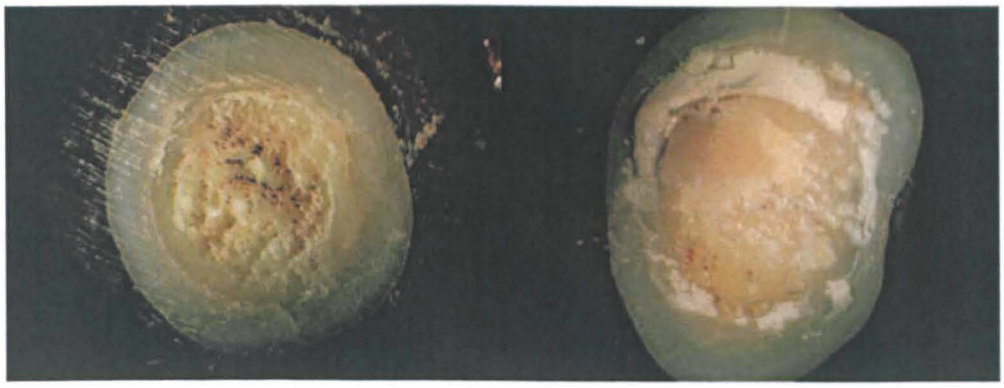


Figure 4.12 Light microscopy showing two types of failure a) Adhesive failure and b) Mixed failure

Table 4.4 Different failure modes of the specimens expressed as a percentage of the total. The actual number of specimens for each mode of failure is in parentheses.

Group	Preparation	Conditioner	Adhesive failure	Mixed failure
Fuji IX				
1	Bur	None	33% (5)	67% (10)
2	Bur	Conditioner	54% (8)	46% (7)
3	Laser	None	40% (6)	60% (9)
4	Laser	Conditioner	33% (5)	67% (10)
Ketac Molar				
5	Bur	None	40% (6)	60% (9)
6	Bur	Conditioner	54% (8)	46% (7)
7	Laser	None	40% (6)	60% (9)
8	Laser	Conditioner	13% (2)	87% (13)

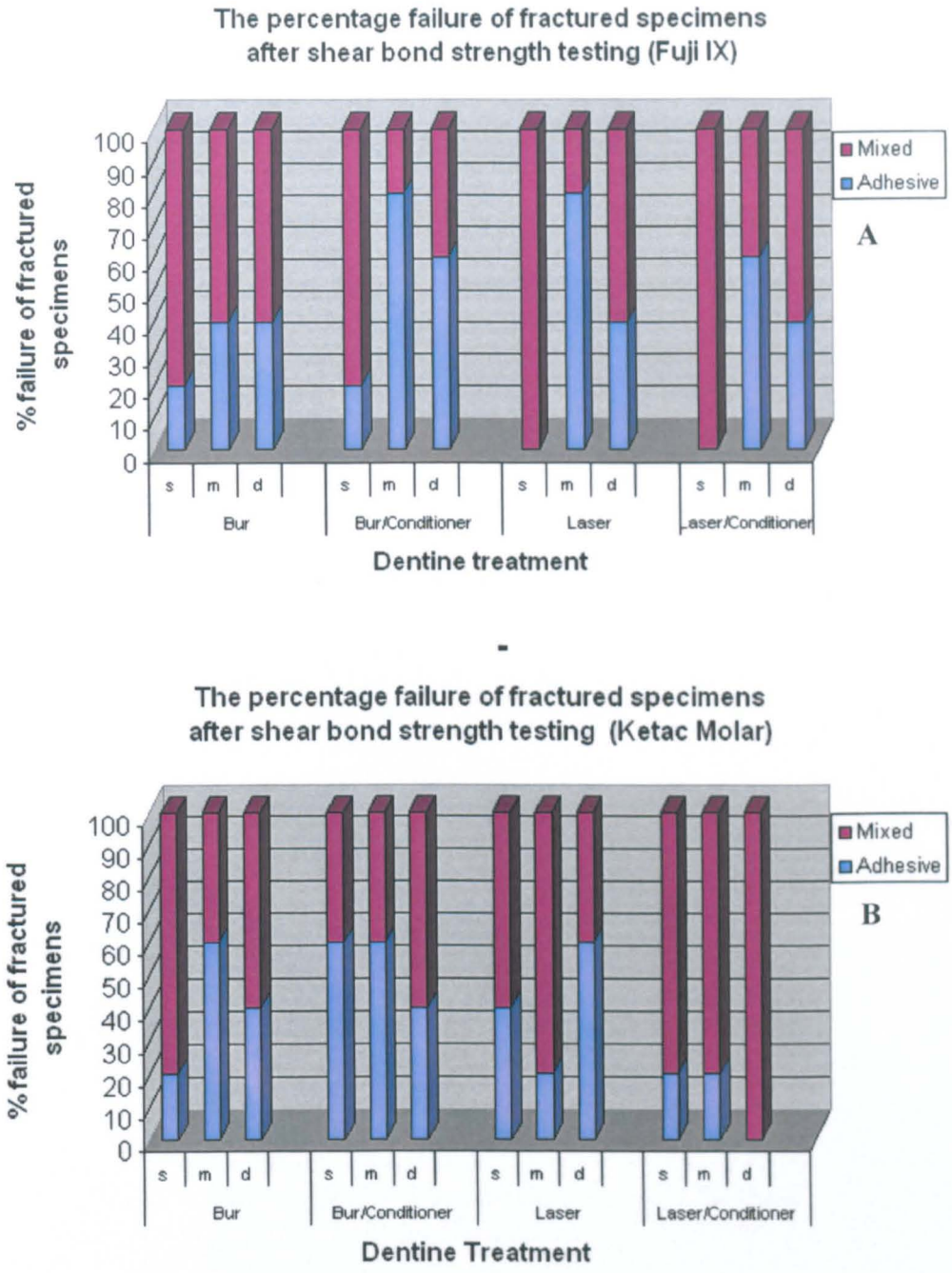


Figure 4.13 Graph demonstrating the percent failure of fractured specimens after shear bond strength testing according to the different dentine depths in superficial (s), middle (m) and deep (d) dentine in (A) Fuji IX and (B) Ketac Molar

SEM analysis of fractured specimen

SEM micrographs of laser prepared dentine, with dentine conditioner (Figure 4.14) showed the mixed mode of failure with partially attached GIC on the laser irradiated dentine. SEM micrographs of laser cut dentine, Figures 4.15 a, c and e, and with dentine conditioner, Figures 4.15 b, d and f, showed adhesive failure. The high magnification of the fractured specimen on the dentine side revealed penetration of GICs into the dentinal tubules (Figure 4.15d).

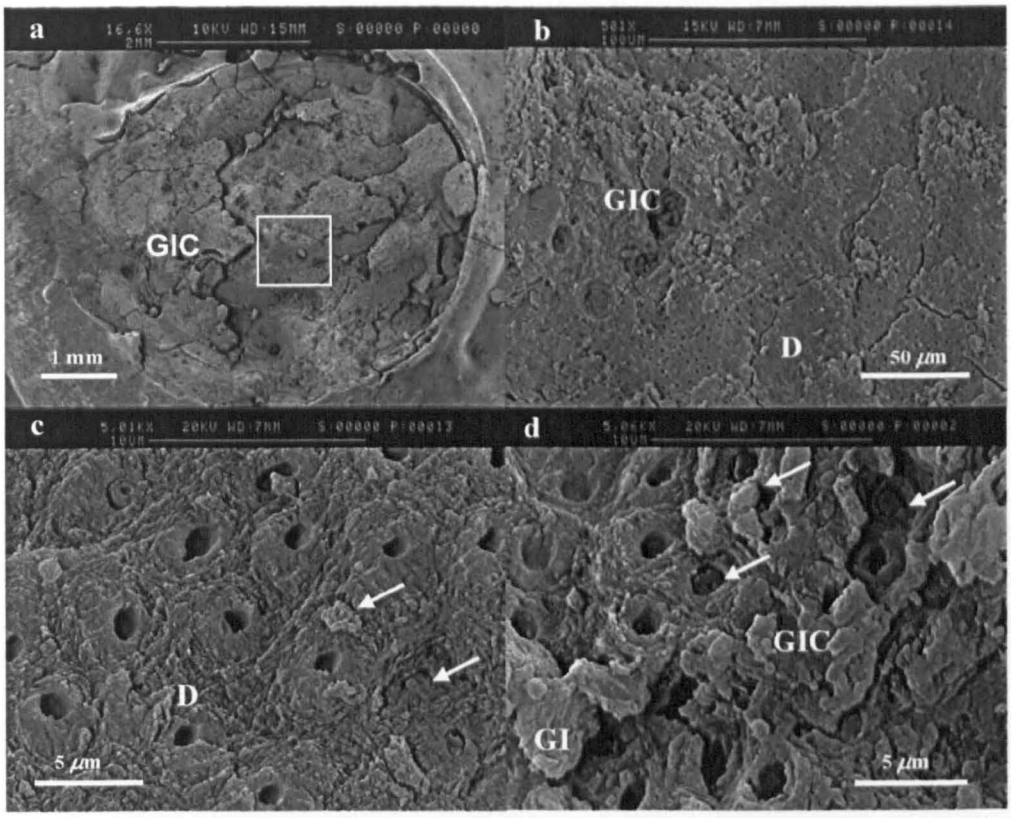


Figure 4.14 Representative SEM micrographs of fractured specimens from the dentine side (from the laser prepared group) **a**) A typical fractured specimen exhibiting partially cohesive failure **b**) The glass ionomer cement fragment on the dentine side in the box area (Figure 4.14a) **c**) The laser cut dentine with glass ionomer plugs in the dentinal tubules (arrowed). **d**) The conditioned laser cut dentine showing both a bulky mass of glass ionomer cement attachment and plugs into dentinal tubules.

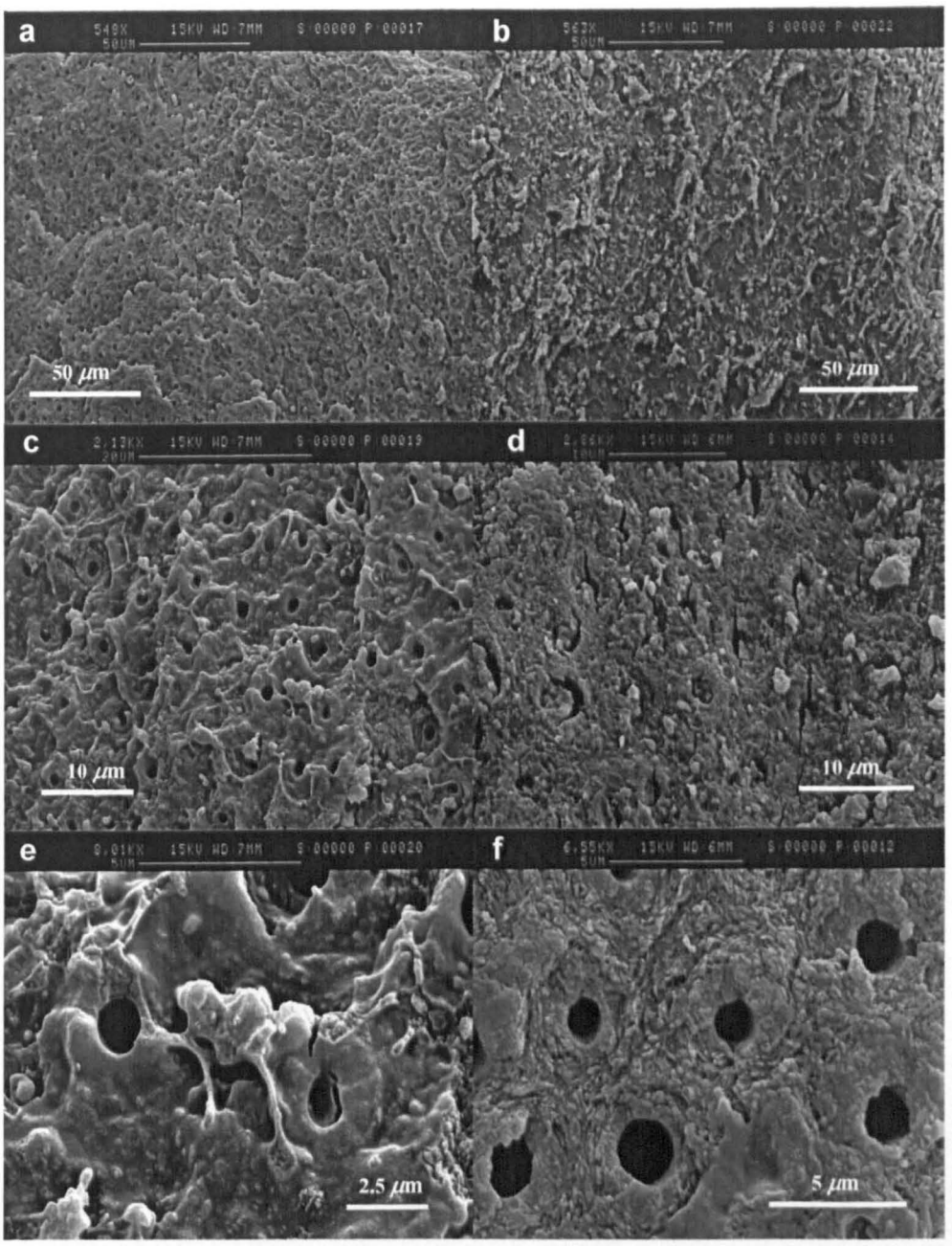


Figure 4.15 Representative scanning electron microscopy (SEM) micrographs of fractured specimens of glass ionomer cement on dentine prepared by Er,Cr:YSGG laser. a) A typical fractured specimen from the laser cut dentine group exhibiting adhesive failure. b) A typical fractured specimen from the laser cut dentine followed by dentine conditioner exhibiting adhesive failure. c) The laser cut dentin showing melting of intertubular dentine and opening of dentinal tubule. d) The laser cut dentine following dentine conditioner application revealing the glass ionomer on the surface. Some GIC fragments plug into the tubules. e) At high magnification, showing the intertubular dentine covering by the dentine debris. f) At high magnification, showing the open dentinal tubules after applying the dentine conditioner. There are some dentinal tubules that are still covered with molten intertubular dentine.

SEM micrographs of bur prepared dentine, with and without dentine conditioner (Figure 4.16) showed the adhesive failure and adhesive failure with GIC fragments on the surface.

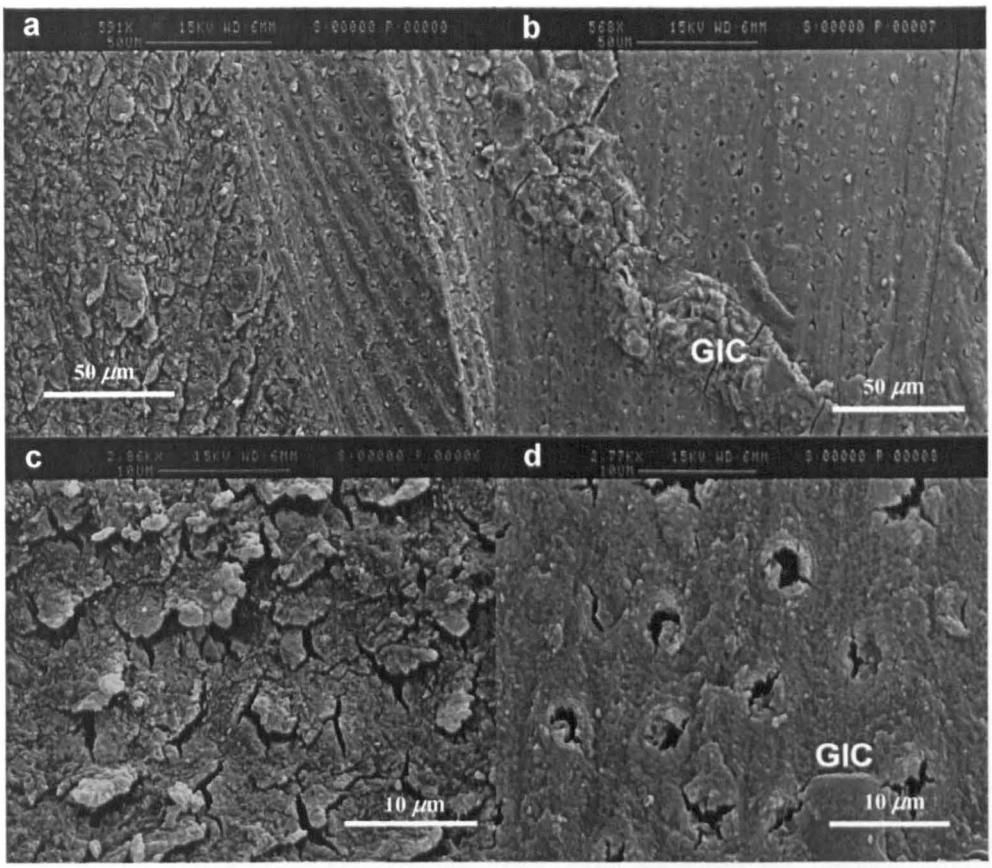


Figure 4.16 Representative scanning electron microscopy (SEM) micrographs of fractured specimens of glass ionomer cement (GIC) on dentin (D) prepared by bur. a) At low magnification, a typical fractured specimen from the bur cut dentin group exhibiting adhesive failure. b) At low magnification, a typical fractured specimen from the bur cut dentine followed by a dentine conditioner exhibiting mixed failure. c) At high magnification, the bur cut dentine showed there was GIC filler attached on the dentine surface and the dentinal tubules were not clear. d) At high magnification, the bur cut dentine following dentine conditioner application revealed some glass ionomer penetrating into the opened dentinal tubules.

4.3.3 Discussion

Data on the bond strength of a GIC to laser cut dentine is rare especially the bond strength of conventional GICs [De Moor et al., 2006]. It is hard to determine the bond strength of GICs as these are brittle materials and have low bond strengths in sound dentine [Czarnecka et al., 2007]. The mean shear bond strength from this study was measured from different depths of dentine. In the bur preparation, there was no significant difference in shear bond strength at different dentine depths. Ceballos et al. (2002) reported that shear bond strength of composite resin to the Er:YAG laser preparation was not influenced by dentine depth. They found that the bond strength to deep dentine was higher than that in superficial dentine but this was not significant. The present study is comparable to this study in that the dentine depth did not show any effect on shear bond strength. The dentine conditioner and interaction between laser cut dentine and dentine conditioner showed an effect on the shear bond strength of GIC but not significantly. Mixed failure occurred more in conditioned laser cut dentine. This was evidenced by the SEM micrographs which revealed the bulky or plate like appearance of GIC attaching on the laser cut dentine with conditioner (Figures 4.14 b and d). Moreover, a tag-like appearance was observed at high magnification on conditioned laser cut dentine (Figures 4.14 d and 4.15 d). This may be explained by the fact that the dentine conditioner helps to widen the dentinal tubules on laser cut dentine. The dentinal orifices

are then accessible for GIC cement penetration. The effect of the conditioner was not apparent for all parts of dentine and hence the shear bond strength increased but not significantly. There were some parts which the dentine conditioner failed to clear the dentine orifices because the molten intertubular dentine completely covered the orifices. The molten intertubular dentine was not uniform on the dentine surface. This may be due to the pulsed nature of the energy of the laser machine.

The studies of bond strengths of restorative materials to laser cut dentine are still controversial. This is because the morphological appearance of laser cut dentine is different from conventional bur cut dentine and hence the adhesion to laser cut dentine would be different. The thermo-mechanical process alters both the chemical structure and the morphological structure of dentine when laser energy is applied to the dentine surface [Rosa et al., 2004]. Re et al. (2004) reported that the shear bond strength of different adhesive systems to Nd:YAG cut dentine was significantly lower than the bur cut dentine. The cut dentinal tubules with this laser could be sealed rather than left open. Therefore, this helps to prevent microorganism invasion into tubules which leads to a possible reduction in postoperative sensitivity [Whitterst et al., 1995]. The Nd:YAG laser is suitable for the treatment of tooth hypersensitivity more than cavity preparation [Mercer, 1996]. The Nd:YAG laser cut dentine shows less susceptibility to an acid solution due to the change in

its Ca:P ratio and chemical structure . This may reduce the efficiency of acid etching in bonding procedures for adhesive-dependent restorative materials like composite resin [Re et al., 2004]

In contrast, numerous researchers have reported a rough, smear layer free dentine, and opening dentinal tubules of the Erbium laser cut dentine which could help to promote the retention of restorative materials. The use of the laser for an effect similar to acid etching was thus introduced. Previous reports encourage the use of the erbium laser to enhance the bond strength of restorations in the same way as acid treatment. The advantages are the enlargement of the adhesive area due to the scaly and flaky structure after laser irradiation [Keller and Hibst, 1989; Visuri et al., 1996b]. The report from Usumez et al. (2003) indicates that dentine surface conditioning with the Er,Cr:YSGG laser yields statistically similar microtensile bond strengths to acid etching. In contrast, Ceballo et al (2002) showed that etching dentine with the low level power of the Er:YAG laser adversely affected adhesion to dentine and did not constitute an alternative to acid etching.

Many investigations have been focused on the bonding of composite materials and adhesive systems to laser cut dentine [Dunn et al., 2005; Celik et al., 2006]. Laser irradiation may produce an unfavourable dentine appearance. The laser melted intertubular dentine and sealed the dentinal tubules. Therefore, the bond strength to laser cut dentine

was relatively low compared to conventional bur cut dentine. On the other hand, laser irradiation produces a smear layer-free surface with wide orifices of dentinal tubules. The bond strength to laser cut dentine is improved. It is possible that the energy per pulse may not be constant during laser irradiation. The variability of dentine tissue is possibly another cause of the different appearance upon laser application.

Comparison between shear bond strength testing and microtensile bond strength testing, the shear bond strength was higher than microtensile bond strength for all experimental group except group 8. Although, it appeared that the microtensile test was better able to differentiate between experimental groups but number of the pre-testing failure sample is higher than the shear bond strength testing. Therefore in the next experiment, the shear bond test was selected because there was no any pre-testing failure during the specimen preparation.

4.4 Summary

The use of a dentine conditioner showed an effect on laser irradiated dentine and helped to promote the bond strength of glass ionomer to laser prepared dentine, thereby rejecting the null hypothesis.

CHAPTER 5
OPTIMAL LASER PARAMETERS FOR ADHESION OF GLASS IONOMER CEMENT

5.1 Background

Laser preparation produces a dentine surface which has different characteristics from those prepared with the conventional bur instruments. The rough surface, clean and open dentinal tubules is the dominant characteristic when using laser preparation (Er:YAG and Er,Cr:YSGG laser). The peritubular dentine protruding from the surrounding intertubular dentine was observed at the microscopic level [Aranha et al., 2007]. The peritubular dentine is less susceptible to laser energies; this is possibly due to the high mineral content and low water content. These dentine characteristics may be favourable for good bonding of dental restorative materials. However, many studies still report the contrary regarding bonding to laser prepared dentine. Most of the in vitro investigations showed that laser preparation had the neutral or adverse effect to bond strength of restorative materials [Lin et al., 1999; Ceballos et al., 2002; Donadio-Moura et al., 2005]. The adverse effects on dentine adhesion with a laser are possibly due to the fact that the laser cannot selectively remove hydroxyapatite crystallites without

affecting the collagen fibre network [Sheth et al., 2004]. The unwanted side effect of laser irradiation would be necrosis, carbonisation, leaving some residual zones of debris and microcracks on the dentine tissue [Koort and Frentzen, 1992]. In addition, the side effect of pulsed infrared laser types was mainly influenced by the physical and chemical properties of the dental tissues, not only by the selected laser parameters. Dentine tissue property variation depends on many factors such as region (the distance from pulp tissue, the alignment of dentinal tubule), aging, etc [Pereira et al., 2000]. Laser energy alters the physical chemical and mechanical properties of dentine tissue. [de Carvalho et al., 2007]. Tonami et al. (2005) showed the effects of Laser irradiation on tensile bond strength of bovine dentine. They found that tensile bond strength of bovine dentine decreases after laser irradiation.

To bond to laser prepared dentine, the restorative materials used should be chosen depending on the laser energies applied to the tooth surface [Roebuck et al., 2000]. Ribeiro et al. (2005) investigated the different laser energy densities of the Nd:YAG laser (1.4 Watts;120 mJ/pulses, 1.2 Watts; 140 mJ/pulses and 1.6 Watts; 160 mJ/pulses) on the marginal leakage in class V cavities (composite resin). They found no significant difference in marginal leakage score between the three different laser energies applied to the dentine surface. However, the marginal leakage decreases after laser conditioning when compared to a

controlled group. The low score of marginal leakage was observed when using high laser energy densities (140 mJ and 160 mJ). Similarly, Roebuck et al. (2000) showed that three different laser energy (200,240,300 mJ) of the Er:YAG laser did not adversely influence microleakage of class V compomer restoration. They suggested that the optimum laser energy which was the best to seal enamel and dentine margin when using Compoglass was 240 mJ. This is the laser parameter which best enhances the marginal seal or bond strength of individual restorative material.

Due to differences in dentine appearance and micromechanical properties after laser irradiation at various power settings, it was important to assess the optimal laser parameters required to maximize the adhesion of glass ionomer cement to the dentine surface.

This chapter aims to identify the optimal laser power parameters for good adhesion of glass ionomer cement to Er,Cr:YSGG laser prepared dentine based on the shear bond strength test and SEM investigation of the fractured specimens after testing the shear bond strength. The null hypothesis tested was the working distance, the power setting and the laser application time did not influence the mean shear bond strength between glass ionomer cement and laser irradiated dentine.

5.2 Shear bond strength measurement

5.2.1 Materials and method

Specimen preparation

In this section, the same materials and shear bond strength testing methods as were described in the previous chapter are applied. Two hundred teeth were used and the specimens were prepared in the same way. The laser power setting, distance between the laser tip and the dentine surface and the exposure time was varied in order to optimise the laser parameters, to achieve the highest shear bond strength between conventional glass ionomer cement and dentine.

Power-Setting

Eighty specimens were randomly divided into four groups of twenty teeth according to the different laser power settings: 3 Watts (33.9 J/cm^2), 3.5 Watts (39.6 J/cm^2), 4 Watts (45.2 J/cm^2) and 4.5 Watts (50.9 J/cm^2) respectively. Each specimen was placed on a movable stand and the laser hand piece was held with a clamp to fix its distance at 1mm. The repetition line was made freehand, by moving the specimen along the path of the laser beam over a period of 60 seconds.

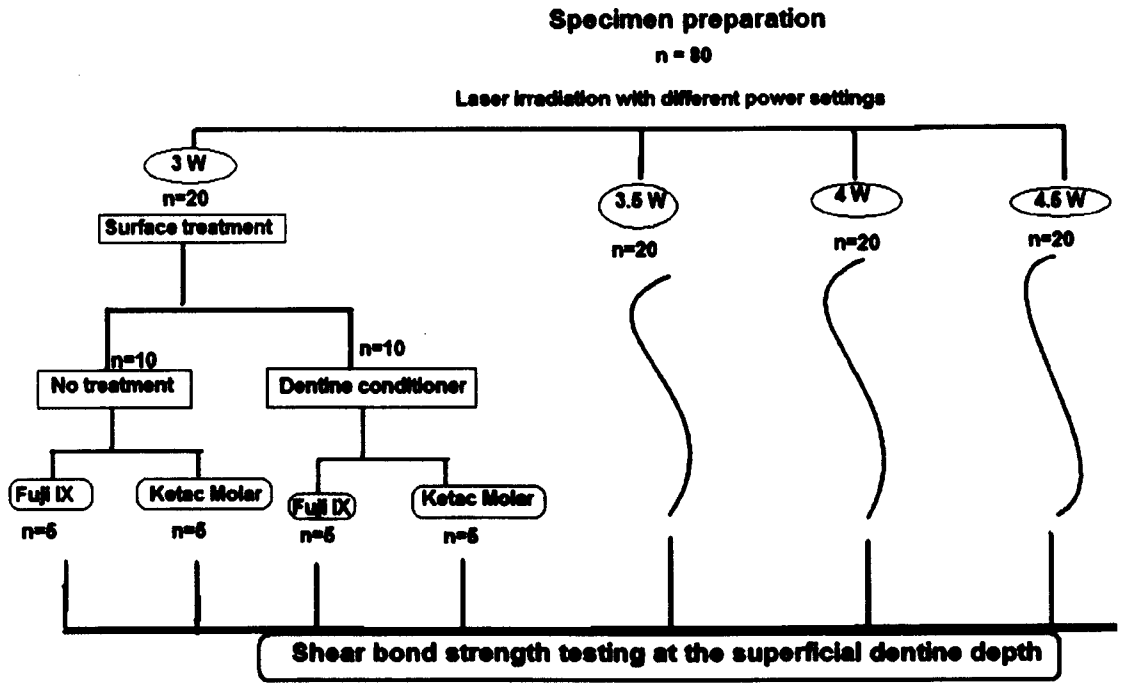


Figure 5.1 Schematic showing the experimental group when varying power settings of laser

The distance between laser tip and dentine surface

Sixty specimens were randomly divided into three groups of twenty teeth according to the different distances between the laser tip and the dentine surface: 1, 1.5, and 2 mm respectively. Each specimen was placed on a movable stand and the laser handpiece was held with a clamp. A feeler gauge was used to measure the working distance. The repetition line was made freehand by moving the specimen along the path of the laser beam over a 60 second period. The power setting was fixed at 4 Watts.

The exposure Time

Sixty specimens were randomly divided into three groups of twenty teeth according to different laser application times: 30, 60, and 90 seconds respectively. Each specimen was placed on a movable stand and a laser handpiece was held with a clamp at a fixed distance of 1 mm. The repetition line was made freehand by moving the specimen along the path of the laser beam over a 60 second period. The power setting was fixed at 4 Watts.

After 24 hours storage in distilled water with a constant temperature of 37°C and 100% relative humidity, test specimens were removed from the incubator and cooled down to room temperature.

Shear bond strength measurement

The method was the same as described in chapter 4.

Failure investigation

The method was the same as described in chapter 4.

Statistical Analysis

The mean values and standard deviations were analysed using three-way analysis of variance (ANOVA) and Tukey's Test ($p<0.05$). The materials the laser parameters (power setting, distance, and application time), the dentine conditioner and the depth of dentine were the three factors for each material (Ketac Molar and Fuji IX). In addition, the

interaction of laser parameters was evaluated using General Linear Model analysis ($p<0.05$).

5.2.2 Results

Shear bond strength measurement

Power setting

The effect of power settings on shear bond strength of Ketac molar and Fuji IX are shown in Tables 5.1 and 5.2, respectively (Figure 5.2). The mean shear bond strength of Ketac Molar when varying the power settings ranged from 4.67–6.59 MPa. The maximum combined shear bond strength was obtained from the specimens prepared using the power setting of 3.5 Watts and after the dentine was conditioned with Ketac Conditioner. The minimum combined shear bond strength was obtained from the specimens prepared using the power setting of 3 Watts without conditioner application. Three ways ANOVA (three factors; power setting, conditioner and dentine level) showed that varying the power setting, conditioner and dentine level produced no significant effect on shear bond strength for Ketac Molar ($p>0.05$) (Table 5.3). However, the shear bond strength to conditioned laser prepared dentine was higher than that for the unconditioned group except at a power setting of 4 Watts.

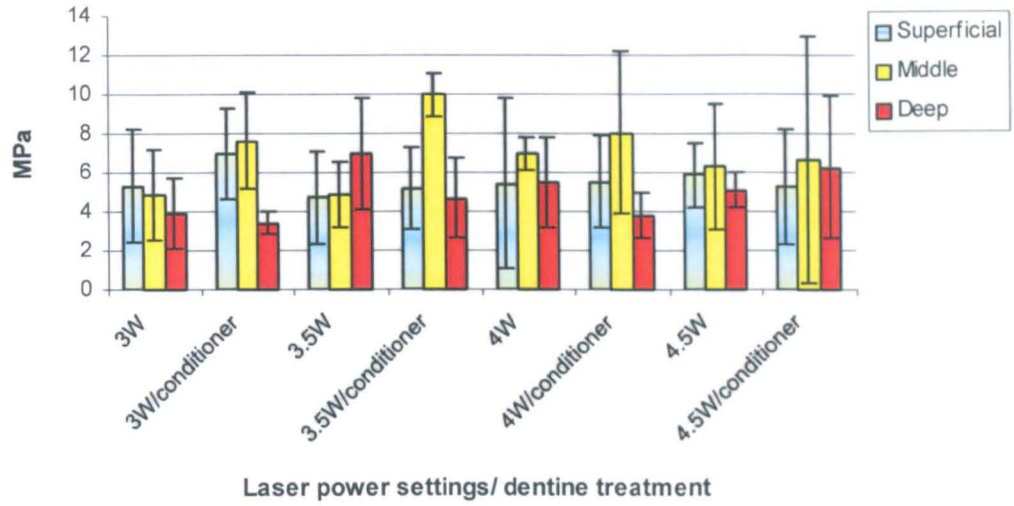
Table 5.1 Mean shear bond strengths of **Ketac Molar** as function of Power setting obtained from superficial (**S**), middle (**M**) and deep (**D**) dentine. Standard deviation is given in parentheses.

Group	Conditioner used	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
3W	no	S	5.31 (2.87)	4.67 (2.29)
		M	4.85 (2.34)	
		D	3.86 (1.79)	
	yes	S	6.96 (2.29)	6.00 (2.64)
		M	7.59 (2.47)	
		D	3.42 (0.60)	
3.5W	no	S	4.70 (2.34)	5.50 (2.40)
		M	4.84 (1.66)	
		D	6.94 (2.82)	
	yes	S	5.13 (2.11)	6.59 (3.00)
		M	9.96 (1.14)	
		D	4.66 (2.05)	
4W	no	S	5.42 (4.35)	5.97 (2.78)
		M	6.99 (0.84)	
		D	5.49 (2.33)	
	yes	S	5.51 (2.36)	5.79 (3.19)
		M	8.03 (4.16)	
		D	3.83 (1.17)	
4.5W	no	S	5.89 (1.63)	5.76 (2.06)
		M	6.28 (3.23)	
		D	5.10 (0.92)	
	yes	S	5.26 (2.97)	6.05 (4.26)
		M	6.64 (6.35)	
		D	6.24 (3.61)	

Table 5.2 Mean shear bond strengths of Fuji IX as function of Power setting obtained from superficial (S), middle (M) and deep (D) dentine. Standard deviation is given in parentheses.

Group	Conditioner	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
3W	no	S	1.66 (0.42)	2.88 (1.63)
		M	4.33 (1.93)	
		D	2.56 (0.82)	
	yes	S	5.78 (3.21)	5.31 (2.56)
		M	4.32 (2.94)	
		D	5.83 (1.46)	
3.5W	no	S	3.16 (0.83)	3.03 (1.95)
		M	3.91 (2.80)	
		D	2.00 (1.57)	
	yes	S	5.24 (4.00)	4.63 (2.90)
		M	4.05 (2.08)	
		D	4.60 (2.87)	
4W	no	S	2.88 (1.68)	3.78 (1.70)
		M	4.15 (2.07)	
		D	4.29 (1.21)	
	yes	S	5.63 (3.20)	5.21 (2.47)
		M	5.22 (2.82)	
		D	4.77 (1.62)	
4.5W	no	S	4.84 (3.61)	3.15 (2.56)
		M	3.01 (1.47)	
		D	1.59 (0.45)	
	yes	S	7.13 (3.66)	5.97 (2.72)
		M	5.01 (2.60)	
		D	5.76 (1.67)	

(a) SBS of Ketac Molar as a function of laser power settings and different dentine depths



(b) SBS of Fuji IX as a functions of power settings and different dentine depths

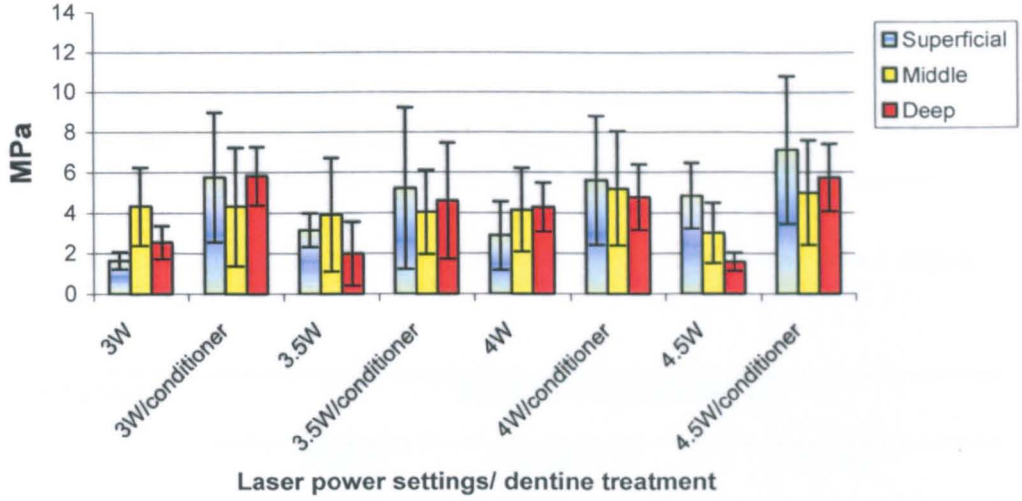


Figure 5.2 Shear bond strength (SBS) of Ketac Molar (A) and Fuji IX (B) **as a function of power settings**/ dentine treatment at different dentine depths.

The combined shear bond strength of Fuji IX when varying the power setting ranged from 2.88-5.97 MPa. The maximum combined shear bond strength was recorded at a power setting of 4.5 Watts after conditioning with dentine conditioner while the minimum was recorded for the group prepared using the 3 Watts power setting and no dentine conditioner treatment. Three ways ANOVA (three factors; power setting, conditioner and dentine level) showed that varying the power setting, conditioner and dentine level produced no significant effect on shear bond strength for Fuji IX ($p>0.05$) (Table 5.4).

Table 5.3 Statistical analysis of shear bond strength of Ketac Molar (p-value) when varying laser power settings.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (3W)	4.67 (2.29)	6.00 (2.64)	0.154
Group 2 (3.5W)	5.50 (2.40)	6.59 (3.00)	0.280
Group 3 (4W)	5.97 (2.78)	5.79 (3.19)	0.873
Group 4 (4.5W)	5.76 (2.06)	6.05 (4.26)	0.815

Table 5.4 Statistical analysis of shear bond strength of Fuji IX (p-value) when varying laser power settings.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (3W)	2.88 (1.63)	5.31 (2.56)	0.004
Group 2 (3.5W)	3.03 (1.95)	4.63 (2.90)	0.086
Group 3 (4W)	3.78 (1.70)	5.21 (2.47)	0.075
Group 4 (4.5W)	3.15 (2.56)	5.97 (2.72)	0.007

Four ways General linear model (four factors; materials, power setting, conditioner and dentine level) revealed that the type of restorative material, dentine conditioner and the dentine level significantly affects the shear bond strength to laser prepared dentine (Figure 5.3).

General Linear Model: Shear bond strength (C1) versus Material (C2), Power setting (C3), conditioner (C4), and depth of dentine (C5)

Factor	Type	Levels	Values
C2	fixed	2	1, 2
C3	fixed	4	1, 2, 3, 4
C4	fixed	2	1, 2
C5	fixed	3	1, 2, 3

Analysis of Variance for C1, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
C2	1	143.995	143.995	143.995	21.24	0.000
C3	3	10.627	10.627	3.542	0.52	0.667
C4	1	110.248	110.248	110.248	16.26	0.000
C5	2	52.133	52.133	26.067	3.85	0.023
Error	232	1572.707	1572.707	6.779		
Total	239	1889.711				

S = 2.60363 R-Sq = 16.78% R-Sq(adj) = 14.26%

Figure 5.3 General Linear Model analysis showed the effect of **power settings** to shear bond strength with the interaction between materials (Fuji IX and Ketac Molar), surface treatment (conditioning), and dentine depth (superficial, middle, and deep).

The distance between laser tip and dentine surface

The effect of distance between the laser tip and dentine surface on the shear bond strength of Ketac Molar and Fuji IX is shown in Tables 5.5 and 5.6, respectively (Figure 5.4).

Table 5.5 Mean shear bond strengths of **Ketac Molar** as function of **the distance between the laser tip and the dentine surface** obtained from superficial (S), middle (M) and deep (D) dentine. Standard deviation is given in parentheses.

Group	Conditioner	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
1 mm	no	S	5.42 (4.35)	5.97 (2.78)
		M	6.99 (0.84)	
		D	5.49 (2.33)	
	yes	S	5.51 (2.36)	5.79 (3.19)
		M	8.03 (4.16)	
		D	3.83 (1.17)	
1.5 mm	no	S	2.61 (1.69)	4.30 (2.31)
		M	5.83 (2.12)	
		D	4.45 (2.17)	
	yes	S	5.44 (2.95)	5.14 (3.58)
		M	2.94 (1.73)	
		D	7.22 (4.87)	
2 mm	no	S	3.87 (2.65)	4.45 (2.26)
		M	5.20 (1.86)	
		D	4.26 (2.47)	
	yes	S	4.99 (3.63)	3.58 (2.93)
		M	1.72 (0.47)	
		D	4.01 (3.11)	

The combined shear bond strength for Ketac Molar when varying the distance between the laser tip and the dentine surface ranged from 3.58-5.97 MPa. The maximum combined shear bond strength was obtained from specimens prepared at a distance of 1 mm and without Ketac Conditioner treatment while the minimum strength was gained from at a distance of 2 mm with Ketac Conditioner treatment.

The combined shear bond strength of Fuji IX when varying the working distance ranged from 2.90-5.21 MPa. The maximum combined shear bond strength was obtained from specimens prepared at a distance of 1 mm with Dentine Conditioner treatment while the minimum strength was measured at a distance of 2 mm without Dentine Conditioner treatment.

Table 5.6 Mean shear bond strengths of **Fuji IX** as function of **the distance between the laser tip and the dentine surface** obtained from superficial (S), middle (M) and deep (D) dentine. Standard deviation is given in parentheses.

Group	Conditioner	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
1 mm	no	S	2.88 (1.68)	3.78 (1.70)
		M	4.15 (2.07)	
		D	4.29 (1.21)	
	conditioner	S	5.63 (3.20)	5.21 (2.47)
		M	5.22 (2.82)	
		D	4.77 (1.62)	
1.5 mm	no	S	3.81 (1.36)	4.12 (3.55)
		M	6.10 (5.45)	
		D	2.44 (1.71)	
	conditioner	S	5.94 (1.74)	4.73 (2.49)
		M	4.28 (3.12)	
		D	3.94 (2.44)	
2 mm	no	S	3.56 (2.39)	2.90 (1.85)
		M	2.15 (0.87)	
		D	2.99 (2.05)	
	conditioner	S	3.25 (1.69)	3.70 (1.92)
		M	3.00 (1.59)	
		D	4.84 (2.22)	

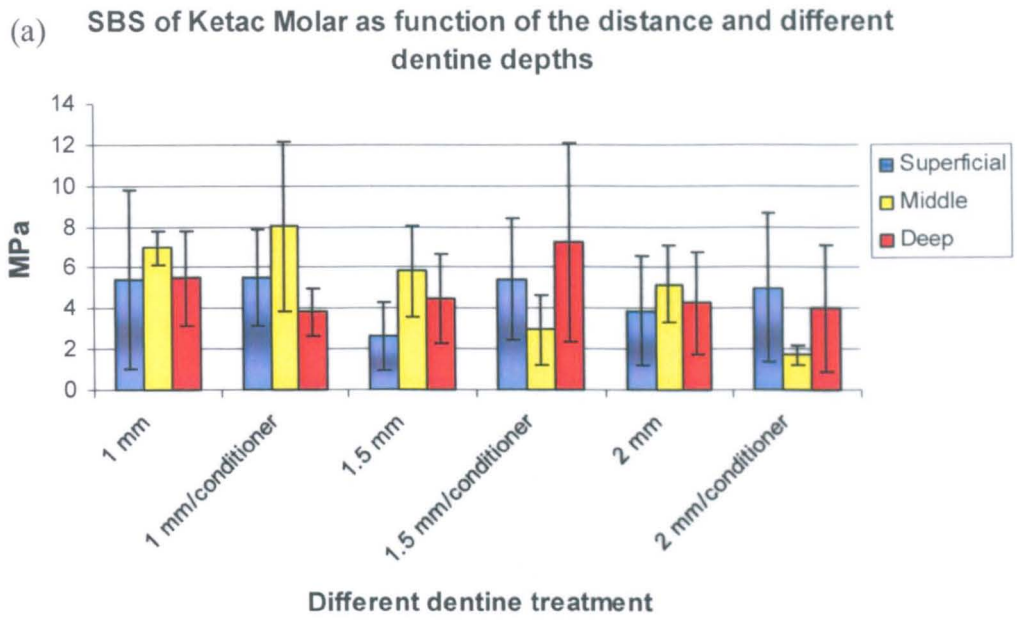
Four ways - General linear model analysis (four factors; materials, distance, conditioner and dentine level) showed that the effect of the distance between laser tip and dentine surface on shear bond strength ($p<0.05$) was significant (Figure 5.5). In addition, the material type also shows an effect on shear bond strength ($p<0.05$) (Figure 5.6). However, no significant affect on shear bond strength of Ketac Molar or Fuji IX (Three way ANOVA, $p>0.05$) is observed with or without dentine conditioner or for different dentine levels as shown in Table 5.7 and 5.8.

Table 5.7 Statistical analysis of shear bond strength of Ketac Molar (p-value) when varying the distance between laser tip and dentine surface.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (1 mm)	**5.97 (2.78)**	**5.79 (3.19)**	**0.873**
Group 2 (1.5 mm)	**4.30 (2.31)**	**5.14 (3.58)**	**0.450**
Group 3 (2 mm)	**4.45 (2.26)**	**3.58 (2.93)**	**0.372**

Table 5.8 Statistical analysis of shear bond strength of Fuji IX (p-value) when varying the distance between laser tip and dentine surface.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (1 mm)	**3.78 (1.70)**	**5.21 (2.47)**	**0.075**
Group 2 (1.5 mm)	**4.12 (3.55)**	**4.73 (2.49)**	**0.594**
Group 3 (2 mm)	**2.90 (1.85)**	**3.70 (1.92)**	**0.255**



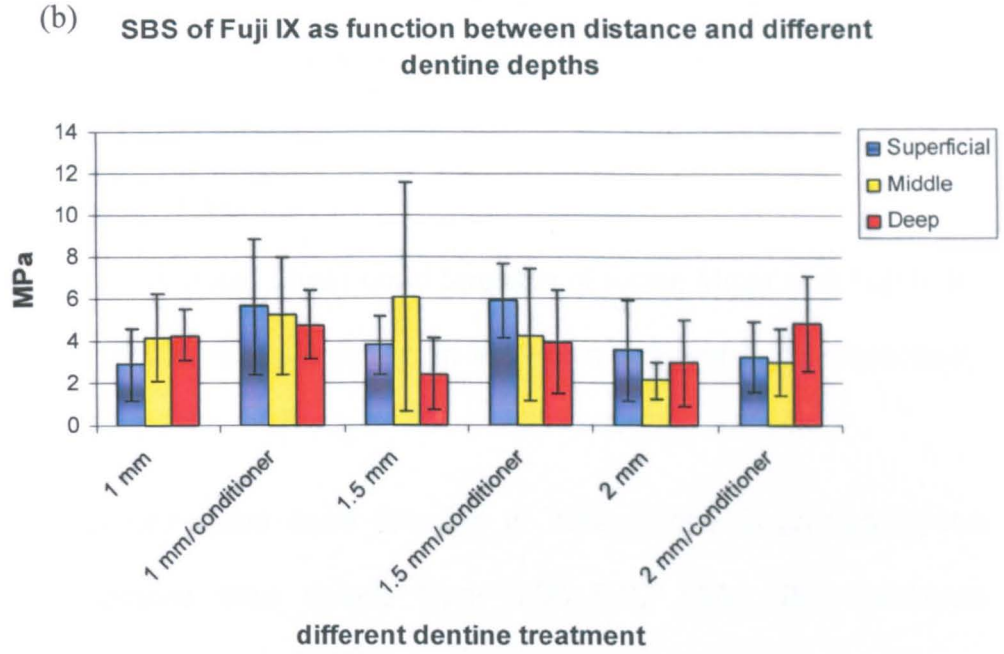


Figure 5.4 Shear bond strength (SBS) of Ketac Molar a) and Fuji IX b) as function of the distance between laser tip and dentine surface/dentine treatment at different dentine depths.

General Linear Model: Shear bond strength (C1) versus Material (C2), The distance between laser tip and dentine surface (C3), conditioner (C4), Different dentine depth (C5)

Factor	Type	Levels	Values
C2	fixed	2	1, 2
C3	fixed	3	1, 2, 3
C4	fixed	2	1, 2
C5	fixed	3	1, 2, 3

Analysis of Variance for C1, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
C2	1	28.688	28.688	28.688	4.08	0.045
C3	2	71.286	71.286	35.643	5.07	0.007
C4	1	8.672	8.672	8.672	1.23	0.268
C5	2	2.484	2.484	1.242	0.18	0.838
Error	173	1216.994	1216.994	7.035		
Total	179	1328.124				

S = 2.65229 R-Sq = 8.37% R-Sq(adj) = 5.19%

Figure 5.5 General Linear Model analysis showed the effect of **the distance between laser tip and dentine surface** to shear bond strength with the interaction between materials (Fuji IX and Ketac Molar), surface treatment (conditioning), and dentine depth (superficial, middle, and deep).

The exposure time

The combined mean shear bond strength of Ketac Molar and Fuji IX for different exposure times is shown in Tables 5.9 and 5.10 respectively (Figure 5.6).

The combined shear bond strength of Ketac Molar when varying the laser exposure time ranges from 3.35–5.97 MPa. The maximum combined shear bond strength was determined from the specimens using an exposure time of 60 seconds without Ketac Conditioner

pretreatment while the minimum value was obtained from specimens using an exposure time of 30 seconds without Ketac Conditioner.

Table 5.9 Mean shear bond strengths of **Ketac Molar** as function of **laser exposure time** obtained from superficial (**S**), middle (**M**) and deep (**D**) dentine. Standard deviation is given in parentheses.

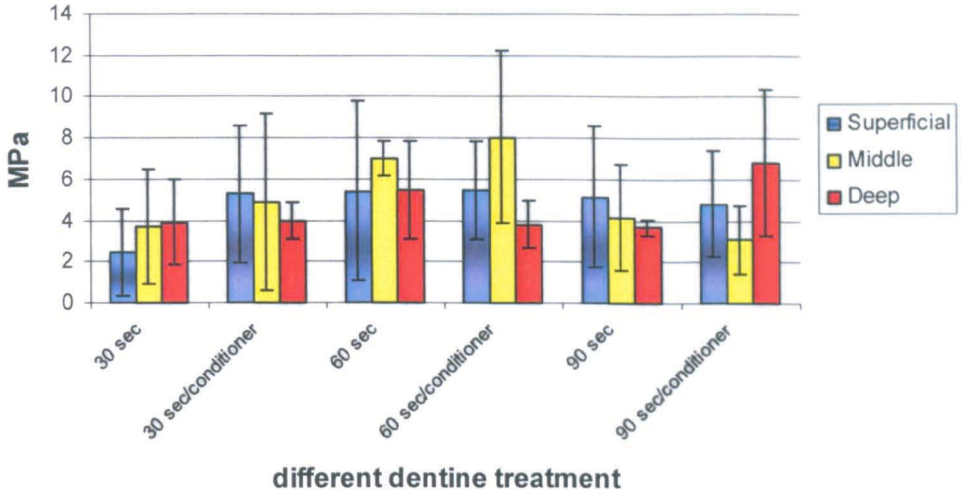
Group	Conditioner	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
30 s	no	S	2.45 (2.12)	3.35 (2.27)
		M	3.69 (2.78)	
		D	3.91 (2.05)	
	yes	S	5.28 (3.32)	4.73 (2.99)
		M	4.91 (4.28)	
		D	3.97 (0.89)	
60 s	no	S	5.42 (4.35)	5.97 (2.78)
		M	6.99 (0.84)	
		D	5.49 (2.33)	
	yes	S	5.51 (2.36)	5.79 (3.19)
		M	8.03 (4.16)	
		D	3.83 (1.17)	
90 s	no	S	5.15 (3.42)	4.34 (2.39)
		M	4.16 (2.59)	
		D	3.69 (0.36)	
	yes	S	4.83 (2.55)	4.93 (2.94)
		M	3.10 (1.63)	
		D	6.84 (3.52)	

The combined shear bond strength of Fuji IX when varying the laser exposure time ranges from 3.71 - 5.61 MPa. The maximum combined shear bond strength was obtained from the specimen using a laser exposure time of 90 seconds without Dentine Conditioner while the minimum was obtained from the group using an exposure time of 30 seconds with Dentine Conditioner application.

Table 5.10 Mean shear bond strengths of Fuji IX as function of laser exposure time obtained from superficial (S), middle (M) and deep (D) dentine. Standard deviation is given in parentheses.

Group	Conditioner	Dentine level	Shear bond strength (MPa)	
			Mean (SD)	Combined Mean (SD)
30 s	no	S	4.42 (1.72)	3.89 (1.66)
		M	3.74 (1.94)	
		D	3.49 (1.51)	
	yes	S	4.68 (1.53)	3.71 (1.45)
		M	2.72 (1.23)	
		D	3.71 (1.00)	
60 s	no	S	2.88 (1.68)	3.78 (1.70)
		M	4.15 (2.07)	
		D	4.29 (1.21)	
	yes	S	5.63 (3.20)	5.21 (2.47)
		M	5.22 (2.82)	
		D	4.77 (1.62)	
90 s	no	S	6.28 (4.50)	5.61 (3.28)
		M	5.81 (1.90)	
		D	4.72 (3.48)	
	yes	S	5.45 (2.19)	4.93 (1.83)
		M	4.74 (2.43)	
		D	4.57 (0.64)	

(a) SBS of Ketac Molar as function between exposure time and different dentine depths



(b) SBS of Fuji IX as function between exposure time and different dentine depths

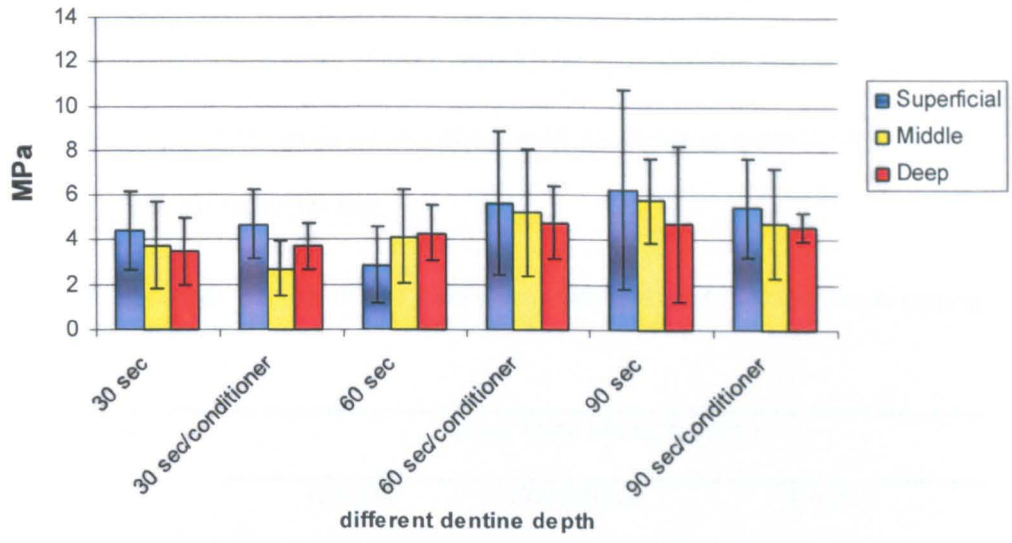


Figure 5.6 Shear bond strength (SBS) of Ketac Molar (a) and Fuji IX (b) as function of the distance between laser tip and dentine surface/dentine treatment at different dentine depth.

There was no significant difference in shear bond strength between conditioned and unconditioned Ketac Molar laser prepared dentine at any given exposure time (Three way ANOVA, $p > 0.05$)(Table 5.11).

Table 5.11 Statistical analysis of shear bond strength of Ketac Molar (p-value) when varying the exposure time.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (30 s)	**3.35 (2.27)**	**4.73 (2.99)**	**0.168**
Group 2 (60 s)	**5.97 (2.78)**	**5.79 (3.19)**	**0.873**
Group 3 (90 s)	**4.34 (2.39)**	**4.93 (2.94)**	**0.550**

For Fuji IX, there was also no significant difference in shear bond strength between conditioned and unconditioned laser prepared dentine at any given exposure time (Three way ANOVA, $p>0.05$) (Table 5.12). The shear bond strength tends to increase with increasing exposure time but was not significantly different.

Table 5.12 Statistical analysis of shear bond strength of Fuji IX (p-value) when varying the exposure time.

Test group	Shear bond strength (MPa)		
	Control	Conditioner	P-value
Group 1 (30 s)	**3.35 (2.27)**	**4.73 (2.99)**	**0.755**
Group 2 (60 s)	**3.78 (1.70)**	**5.21 (2.47)**	**0.075**
Group 3 (90 s)	**4.34 (2.39)**	**4.93 (2.94)**	**0.487**

Statistical analysis using four way General linear model analysis (four factors; materials, exposure time, conditioner and dentine level) revealed that the only significant correlation is between exposure time and shear bond strength for the two glass ionomer cements ($p<0.05$). The other factors show no significant affect on the shear bond strength ($p>0.05$), see figure 5.7.

General Linear Model: Shear bond strength (C1) versus Material (C2), application time (C3), conditioner (C4), different dentine depth (C5)

Factor	Type	Levels	Values
C2	fixed	2	1, 2
C3	fixed	3	1, 2, 3
C4	fixed	2	1, 2
C5	fixed	3	1, 2, 3

Analysis of Variance for C1, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
C2	1	4.949	4.949	4.949	0.78	0.378
C3	2	54.653	54.653	27.327	4.32	0.015
C4	1	6.949	6.949	6.949	1.10	0.296
C5	2	5.354	5.354	2.677	0.42	0.656
Error	173	1095.132	1095.132	6.330		
Total	179	1167.037				

S = 2.51600 R-Sq = 6.16% R-Sq(adj) = 2.91%

Figure 5.7 General Linear Model analysis showed the effect of laser parameter to shear bond strength with the interaction between materials (Fuji IX and Ketac Molar), surface treatment (conditioning), and dentine depth (superficial, middle, and deep).

Fracture analysis

Examination of the fractured specimens under the light microscope revealed two different patterns of failure: adhesive and mixed failure. The modes of failure for Ketac Molar and Fuji IX are presented in Tables 5.13 and 5.14. The majority of fractured specimens both from Ketac Molar and Fuji IX exhibited adhesive failure. At a working distance of 1 mm, Ketac Molar showed approximately 60% of mixed failure. The fracture mode of Ketac Molar with an application time of 60 sec revealed a 60% mixed failure. The power setting did not show any effect on the fracture mode that correlates to the shear bond strength. Furthermore, the dentine conditioner and dentine depth show no effect on the mode of failure. For Fuji IX specimens, the fracture modes were uncorrelated to the laser parameters, dentine conditioner, and dentine depth.

Table 5.13 Different failure modes of fractured specimens (Ketac Molar) expressed as a percentage of the total. The number of specimens for each mode of failure is in parentheses.

Parameter	Failure Pattern			
	No conditioner		Conditioner	
	Adhesive failure	Mixed failure	Adhesive failure	Mixed failure
Power setting				
3W	80% (12)	20% (3)	73% (11)	27% (4)
3.5W	73% (11)	27% (4)	60% (9)	40% (6)
4W	40% (6)	60% (9)	80% (12)	20% (3)
4.5W	40% (6)	60% (9)	87% (13)	13% (2)
Distance				
1 mm	40% (6)	60% (9)	80% (12)	20% (3)
1.5 mm	80% (12)	20% (3)	100% (15)	0% (0)
2 mm	93% (14)	7% (1)	100% (15)	0% (0)
Application time				
30 s	93% (14)	7% (1)	93% (14)	7% (1)
60 s	40% (6)	60% (9)	80% (12)	20% (3)
90 s	93% (14)	7% (1)	93% (14)	7% (1)

Table 5.14 Different failure modes of fractured specimens (Fuji IX) expressed as a percentage of the total. The number of specimens for each mode of failure is in parentheses.

Parameter	Failure Pattern			
	No conditioner		Conditioner	
	Adhesive failure	Mixed failure	Adhesive failure	Mixed failure
Power setting				
3W	100% (15)	0% (0)	100% (15)	0% (0)
3.5W	87% (13)	13% (2)	87% (13)	13% (2)
4W	80% (12)	20% (3)	80% (12)	20% (3)
4.5W	87% (13)	13% (2)	80% (12)	20% (3)
Distance				
1 mm	80% (12)	20% (3)	80% (12)	20% (3)
1.5 mm	100% (15)	0% (0)	100% (15)	0% (0)
2 mm	100% (15)	0% (0)	100% (15)	0% (0)
Application time				
30 s	100% (15)	0% (0)	100% (15)	0% (0)
60 s	80% (12)	20% (3)	80% (12)	20% (3)
90 s	80% (12)	20% (3)	93% (14)	7% (1)

SEM microscopy

Small fragments of glass ionomer cement were observed on the unconditioned dentine at any power setting (Figure 5.8 a-d). In conditioned laser prepared dentine, tags of glass ionomer cement are seen penetrating into the dentinal tubules (Figure 5.9 a-d). Notably, it was seen that the molten irradiated dentine surface at a power setting of 4.5 Watts was elongated. The debonded area of laser prepared dentine when using a power setting of 4.5 Watts has two different characteristics (Figure 5.10 a-d). It shows the smooth intertubular dentine surrounding the peritubular dentine (Figure 5.10a and c) or a scaly, rough, cuff-like appearance of intertubular dentine around the peritubular dentine (Figure 5.10 b and d).

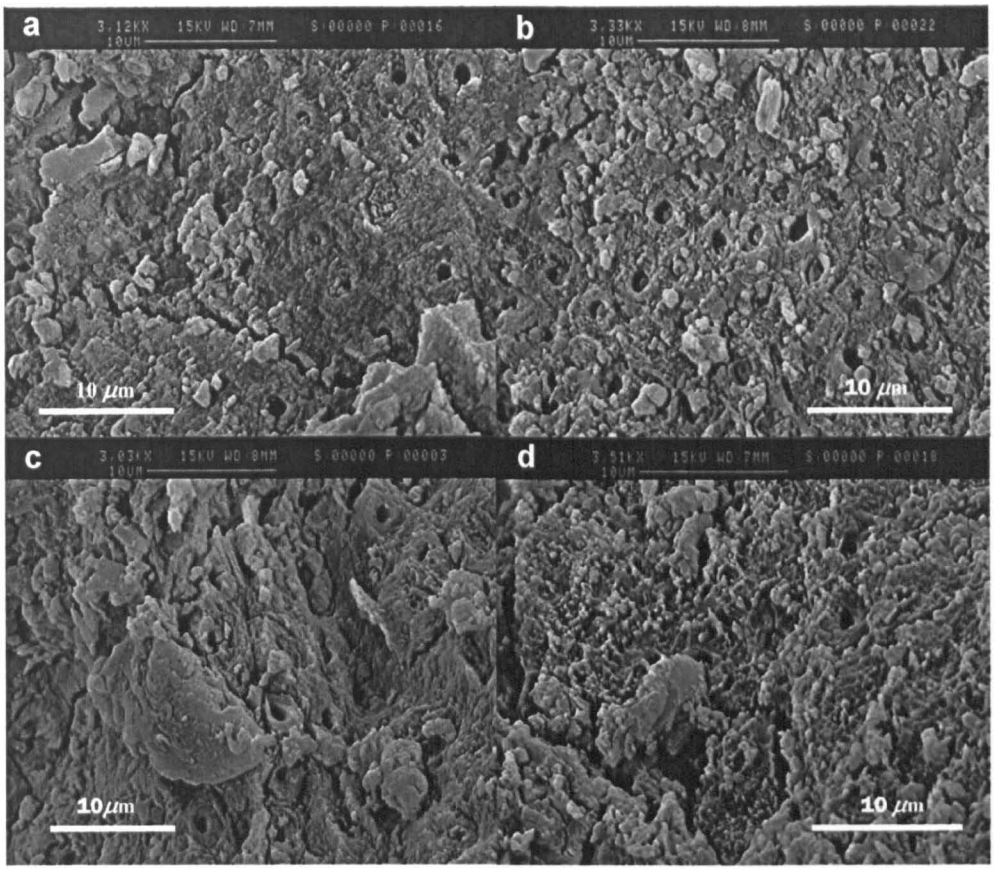


Figure 5.8 Representative SEM micrographs of a fractured specimens of glass ionomer cement (GIC) on dentine (D) prepared by Er,Cr:YSGG laser. All these micrographs are from adhesive failure group.

a) At a power setting of 3 Watts, the fractured dentine surface exhibited some small fragment of GIC attached on the surface.
b) At a power setting of 3.5 Watts, the fractured dentine surface showed some fragment of GIC and some cement plugs into the dentinal tubules.
c) At the power setting of 4Watts, the laser cut dentine showed bulky fragments of GIC attached on dentine surface. The dentine surface appeared distorted due to the shear force.
d) At a power setting of 4.5Watts, the fractured dentine surface revealed molten dentine mass mixed with fragments of GIC and filler attached on the surface.

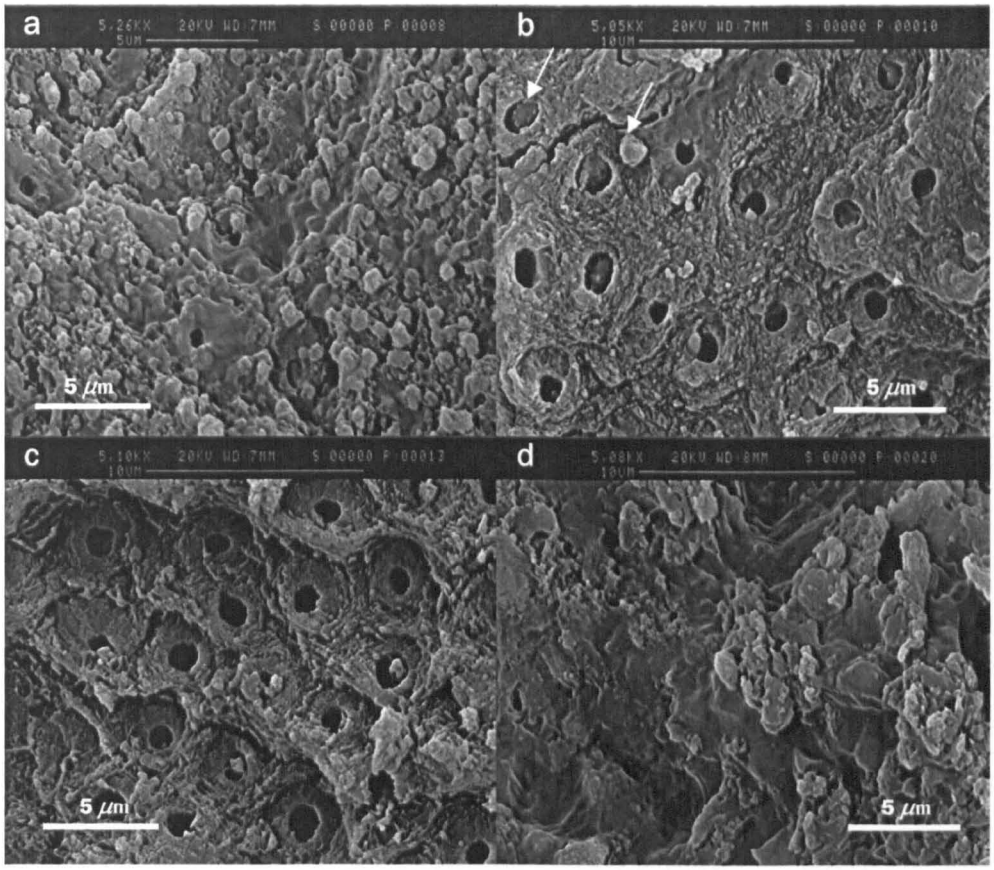


Figure 5.9 Representative SEM micrographs of a fractured specimens of glass ionomer cement (GIC) on dentine (D) prepared by Er,Cr:YSGG laser following dentine conditioner on the dentine side. All these micrograph are from adhesive failure group.

a) At a power setting of 3Watts, the fractured dentine surface exhibited some small inclusions of GIC attached on the surface and molten appearance of dentine surface was observed.
b) At a power setting of 3.5Watts, the fractured dentine surface showed GIC cement plugged into the dentinal tubules.
c) At a the power setting of 4 Watts, the laser cut dentine showed the edge of peritubular dentine and some GIC fragment plugged into the dentinal tubules
d) At a power setting of 4.5 Watts, the fractured dentine surface revealed a molten dentine mass mixed with fragments of GIC and filler attached on the surface.

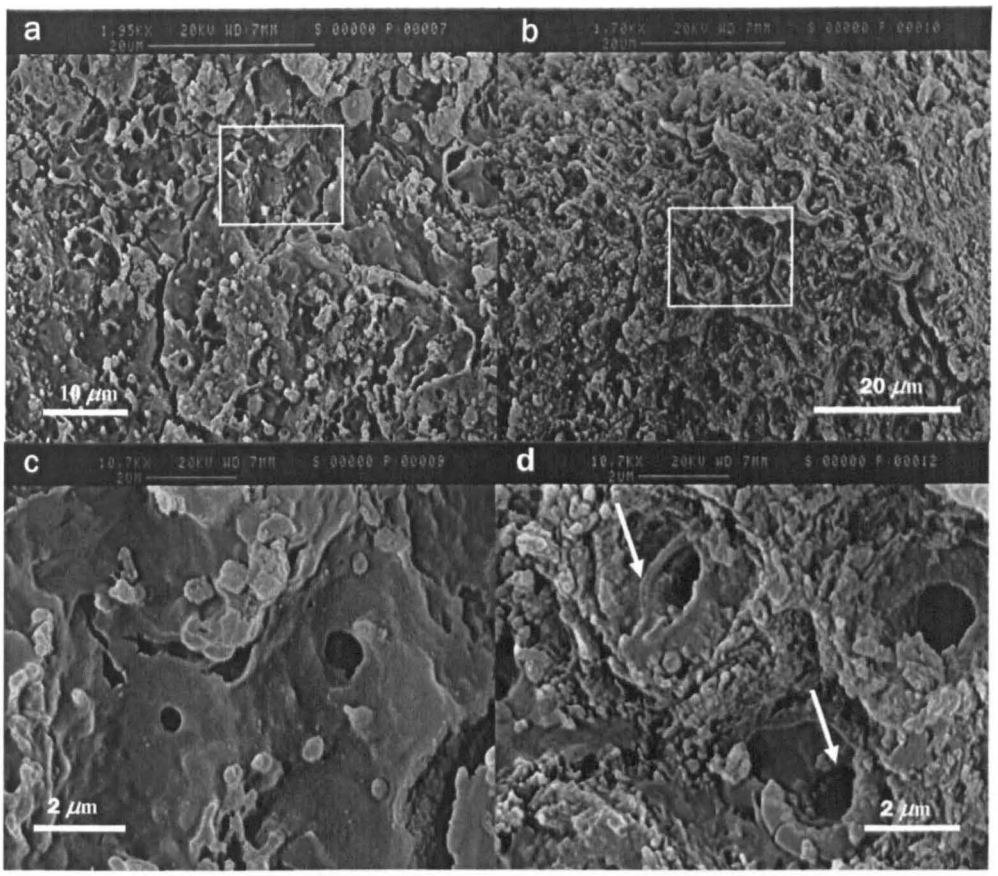


Figure 5.10Representative SEM micrographs of fractured specimens of GIC on dentine (D) prepared by Er,Cr:YSGG laser **following dentine conditioner** on the dentine side. All these micrographs are from the adhesive failure group and the power setting of 4.5 Watts.

a) Dentine appearance of fracture specimen, showing the small fragments of GIC attached on the surface
b) At power setting of 4.5 Watts, the fractured specimen exhibited a collar-like appearance of the laser-irradiated dentine (depletion of intertubular dentine).
c) At high magnification, the molten intertubular dentine coated the surface and reduced the size of dentinal orifices.
d) At higher magnification, the fractured dentine surface revealed the depletion of intertubular dentine and molten mass of peritubular dentine.

5.2.3 Discussion

Adhesion of glass ionomer cement to sound dentine or conventional bur preparation is weak compared to composite resin. Shear bond strength of glass ionomer cement to dentine was reported from 1-3 MPa, and rarely exceed 5 MPa [Czarnecka et al., 2007]. In the present study, the shear bond strength of conventional glass ionomer cement to laser prepared dentine was in the range of 3-7 MPa. In this respect, it seems to be that laser preparation improves the adhesion of the glass ionomer cement to the dentine surface. The adhesion mechanism of conventional glass ionomer is based on mechanical theories, adsorption theories and diffusion theories. The hydrogen ions from the glass ionomer cement and phosphate ions from hydroxyapatite crystals play an important role for glass ionomer bonding. The inherent wetness of the dentine interface may affect the bond strength [Watson et al., 1998]. Some literatures have reported the reduction of the collagen component and recrystallization of hydroxyapatite on the dentine surface after Er:YAG laser irradiation [Carmerlingo et al., 2004; Armengol et al.,2002]. Therefore, the interaction between glass ionomer and laser prepared dentine would be different from the conventional preparation.

Of critical concern, the shear bond strength value which measured from this study was approximately half the value found in the previous chapter. This is because of the different laser parameter. Previous

chapter have used the power setting 5 Watt in order to get high dentine ablation efficiency. This laser output was over the acceptable limits for dentine cutting.

In the present study, the combined shear bond strength of Ketac Molar was low when using a power setting of 3 Watts (33.9 J/cm^2). From the previous chapter, a dentine debris layer was seen on the surface under SEM observations at this power setting. This is comparable to the report from Ishizaka et al. (2002). They found a thin layer after Er:YAG irradiation using the laser energy densities ranged from 20.8 J/cm^2 to 36.5 J/cm^2. In chapter 3, the microhardness after laser irradiation decreased therefore this thin layer may be the organic part of the dentine (protein in collagen fibril) covering the dentine surface. The heat production which cannot be eliminated by the cooling system may cause the protein denature (in collagen fibril) [Kayano et al., 1991]. This is not favourable for adhesion to lased dentine. It is speculated that the intermechanical locking is not achieved and hence does not promote bond strength at this power setting.

The maximum shear bond strength of Ketac Molar is obtained when using a power setting of 3.5 Watts (40 J/cm^2) with dentine conditioner. This demonstrates that the combination of treatments between increasing laser energies and the effect of Ketac Conditioner is successful in removing the debris layer on the irradiated dentine

surface. The results from previous chapter showed that the microtensile bond strength of the glass ionomer cement (Fuji IX and Ketac Molar) to the laser prepared dentine was improved when using the Dentine conditioner or Ketac conditioner. The shear bond strength increases in the conditioned group in comparison to the unconditioned group at every power setting except at 4 Watts. The possible reason for this may be the excessive demineralisation reaction of the dentine conditioner to the dentine surface irradiated at a power setting of 4 Watts. From previous chapter, the SEM micrograph of the laser irradiated dentine surface at a power setting of 4 Watts has the recrystallized like substance without dentine debris on the surface. Therefore, the excessive demineralisation from dentine conditioner application may destroy the recrystallization of hydroxyapatite which is in fungi form appearance and helps to contribute to the micromechanical retention or possible chemical adhesion of glass ionomer cement to the dentine surface [Cooper et al., 1988; Melendez et al., 1992]. This demonstrates that the dentine conditioner helps to remove the debris layer that is seen in irradiated dentine at 3, 3.5 and 4.5 Watts and is comparable to the SEM images in Chapter 3, where it was found that dentine debris occurs in every power setting except 4 Watts. Moreover, it is in agreement with the previous study by de Carvalho et al. (2005). As evidenced by their SEM micrographs, the irradiated dentine surface showed occluded dentinal tubule and intact smear layer when irradiating at low energy densities (18.1 and 17.1 J/

cm^2). This is possibly due to the temperature for evaporation not being fully realised and only partial ablation occurs [Keller and Hibst, 1990]. The Ketac Conditioner shows an adverse affect on shear bond strength of Ketac Molar when using the laser output of 4 Watts. This was comparable with study from Melendez et al. (1992). They concluded that shear bond strength of Ketac Cement (Ketac Fil) was adversely affected by laser etching. On the contrary, the Fuji material (Fuji II) has improved shear bond strength after laser etching. The different brand of glass ionomer cement, the delivery system and the inherent properties of materials could be related to the adverse affects when using the laser for tooth preparation [Melendez et al., 1992]. The report from Czarnecka et al. (2007) provides supportive information in terms of the delivery system. The shear bond strength of Fuji IX caps and Ketac molar Aplicap is higher than that with hand mixed type.

For Fuji IX, the Dentine Conditioner seems to be successful in removing the dentine debris layer at a power setting of 4.5 Watts because the shear bond strength achieved is at its maximum. The lowest shear bond strength is obtained for a power setting of 3 Watts with unconditioned dentine and then increases significantly when dentine conditioner is applied. This indicates that the dentine debris layer reduces the ability to bond between Fuji IX and irradiated dentine and the dentine conditioner improves the surface characteristics to promote higher bond strength.

At all power settings, the conditioned dentine surface has higher bond strength than for the unconditioned groups. There was a significant difference in shear bond strength between the conditioned and unconditioned groups particularly in the 3 and 4.5 Watt groups (Three ways ANOVA, $p < 0.05$). These data demonstrates that the shear bond strength of Fuji IX to laser irradiated dentine improves by using Dentine Conditioner. It is possible that the dentine debris layer is completely removed by the conditioner.

The optimal power for good adhesion of glass ionomer cement to laser prepared dentine is achieved when no dentine debris layer is produced. Therefore, the optimal setting from these results should be 4 Watts. However, the ability to remove the dentine at a power setting of 4Watts is lower than that at the power setting of 4.5 Watts. Therefore, it is possible to use the power setting of 4.5 Watts followed by use of the dentine conditioner.

The results indicate that the optimal working distance for this laser is 1 mm when working on the dentine surface of a molar tooth (mean surface area = 3.14 mm^2) as this is the distance at which the maximum shear bond strength for both Ketac Molar and Fuji IX was achieved. The lowest shear bond strength was obtained at a working distance of 2 mm, it is conjectured that this is due to the decrease in the energy density at

this distance. Therefore the dentine surface may not be different from ground dentine. It was observed that when the distance between the laser tip and the dentine surface is less than 1 mm or too close to the tooth surface, there was sparkling (infrared indicator light) occurring on the tooth surface. The dentine was burnt and also the end of the laser tip was damaged. The Er,Cr:YSGG laser only operates in a non-contact mode. The position of the handpiece of the laser should be placed perpendicular to the tooth surface. The Er:YAG laser has two operating modes; contact and non-contact type. Previous studies have reported that the beam incidence angle has a strong influence on the amount of dentine substance removal when using the Er:YAG laser. The studies showed that the optimal angle of incidence is perpendicular to the tooth [de Carvalho et al., 2005].

The shear bond strength of Ketac molar is highest when using the medium range of application time (60 sec). The longer exposure time (such as 90 sec) or an insufficient exposure time (e.g. 30 sec) is not seen to be beneficial for adhesion of Ketac Molar on the laser prepared dentine. On the other hand, the adhesion of Fuji IX to laser prepared dentine was improved when using a longer application time such as 90 sec. An application time of 30 sec is inadequate to provide a favourable dentine surface for Fuji IX restoration.

The SEM investigation of fractured specimens showed the glass ionomer cement tag penetration into clear opened dentinal tubules. The small inclusion of glass ionomer cement also was seen. This is matched with the SEM micrograph from Yip et al. (2001). They reported that the agglomerates particle composed of a granular cluster on the dentine side of the fracture specimen after microtensile testing of Fuji IX to sound dentine. These agglomerates were remnants of GIC corresponding with their TEM micrographs. The particle of glass is trapped within the fine granular hydrogel during the setting reaction of the GIC. The remnant GIC shows that there was mechanical retention between glass ionomer and laser prepared dentine. Also, it appears that the glass-ionomer cement bonding characteristic to laser prepared dentine was no different to that obtained in bonding to sound dentine.

Adhesive or Cohesive failure has been used as the indicator of mechanical retention between restorative resin material and the dentine surface. In the other words, it is implied that high bond strength shows more cohesive failure than low bond strength [Burrow et al., 2002]. In this study, the conditioned dentine did not showed more mixed failure which implies that mechanical interlocking did not play an important role in the adhesion between glass ionomer and laser prepared dentine.

5.3 Summary

From the studies in this chapter, the following conclusions can be drawn:

- The power setting has no significant effect on the shear bond strength of GIC. However, the distance between the laser tip and dentine surface did affect the shear bond strength of the GIC.

- The optimal power setting of the Er,Cr:YSGG laser for dentine preparation and conventional glass ionomer restoration without dentine pre-treatment is 4 Watts. The advisable distance between laser tip and dentine surface is 1 mm. Time required to completely irradiate the dentine surface for a molar tooth is approximately one minute.

- The in-vitro testing is limited. In vivo, bond strengths may be affected by the ooze of dentinal fluid from the dentine tubules. Therefore, the interaction between GIC and different laser parameters requires further investigation *in vivo*.

CHAPTER 6
Conclusion

6.1 Conclusion

Based on the results of all investigations, the following conclusion can be made:

- The ablation efficiency of the laser is still not as well accepted as bur preparation.
- The microhardness of dentine after Er,Cr:YSGG irradiation is decreased.
- Cavities prepared with the Er,Cr:YSGG laser provide the good cavity adaptation with glass ionomer cement at enamel surface.
- The glass ionomer/lased dentine interface showed poorer adaptation compared to dentine prepared by dental bur.
- The bond strength to laser prepared dentine could be enhanced with dentine conditioner application.

- The Er,Cr:YSGG laser irradiation at 4 W output power in associated with 55% air, 65% water and the working distance of 1 mm (perpendicular to tooth surface) would be the optimal parameter to prepare dentine for glass ionomer restoration.

- The burnt scars and carbonized dentine would be appeared with the higher power settings.

6.2 Further work

- The standard variation of microhardness of laser prepared dentine is in a wide range. Further work is recommended to measure nanohardness of laser prepared dentine in order to get to the actual hardness.

- The hardness of dentine surface after laser irradiation is lower than the normal dentine. The softening effect is likely to be only superficial. Further work is needed to determine the extent of any softening.

- The Er,Cr:YSGG laser is not a device used only dentine. It would be recommended to survey the effect of Er,Cr:YSGG laser on enamel surface and the interaction of glass ionomer cement with this prepared enamel surface.

- There is still much to be investigated on the effect of Er,Cr:YSGG laser on tooth surface, especially the microstructure of the adhesive interface and alterations in substrate compounds, under different laser parameter.

- Further in vivo researches on the interaction of glass ionomer cement to lasing tooth structure are mandatory to determine whether glass ionomer cement is proper materials for lased cavity and access the longevity of restoration under real oral conditions.

- Most restorative materials aim to formulate for adhesion to conventional tooth preparation. In the future, it is suggested that a new class of restorative material is designed specifically for use with laser prepared teeth.

References

Adrian JC, Bernier JL, Sprague WG. Laser and the dental pulp. *JADA* 1971; 83: 113-117.

Allen DJ. Thermal effects associated with the Nd/YAG dental laser. *Angle Orthod* 1993; 63: 299-303.

Altshuler GB, Belikov AV, Sinelnik YA. A Laser-Abrasive Method for the Cutting of Enamel and Dentin. *Lasers Surg Med* 2001; 28: 435-444.

Andersson-Wenckert IE, van Dijken JW, Kieri C. Durability of extensive class II open-sandwich restorations with resin-modified glass ionomer cement after 6 years. *Am J Dent* 2004; 17: 43-50.

Anderson AM, Kao E, Gladwin M, et al. The effects of argon laser irradiation on enamel decalcification: An in vivo study. *Am J Orthodo Dentofacial Orthop* 2002; 122: 251-259.

Aminzadeh A, Shahabi S, Walsh LJ. Raman spectroscopic studies of CO_2 laser-irradiated human dental enamel. *Spectrochimica Acta Part A* 1999; 55: 1303-1308.

Angker L, Swain MV, Kilpatrick N. Micro-mechanical characterisation of the properties of primary tooth dentine. *J Dent* 2003; 31: 261-267.

Angker L, Swain MV, Kilpatrick N. Characterising the micro-mechanical behavior of the carious dentine of primary teeth using nano-indentation. *J Biomech* 2005; 38: 1535-1542.

Apel C, Gutknecht N. Demineralization of Er:YAG and Er,Cr:YSGG laser-prepared enamel cavities in vitro. *Caries Res* 2003; 37: 34-37.

Apel C, Meister J, Schmitt N, et al. Calcium Solubility of Dental Enamel Following Sub-Ablative Er:YAG and Er:YSGG Laser Irradition In Vitro. *Lasers Med Sci* 2002; 30: 337-341.

Ariyaratnam MA, Wilson AS, Blinkhorn AS. An analysis of surface roughness, surface morphology and composite/dentin bond strength of human dentin following the application of the Nd:YAG laser. *Dent Mater* 1999; 15: 223-228.

Armengol V, Rohanizadeh R, Hamel H. Scanning electron microscopic analysis of diseased and healthy dental hard tissues after Er:YAG laser irradiation: in vitro study. *J Endod.* 1999; 25: 543-6.

Armengol V, Jean A, Enkel B, Assoumou M, et al. Microleakage of Class V Composite Restorations Following Er:YAG and Nd:YAP Laser Irradiation Compared to Acid-etch: an In Vitro Study. *Lasers Med Sci* 2002; 17: 93-100.

Aranha ACC, De Paula Eduardo C, Gutknecht N, et al. Analysis of the Interfacial Micromorphology of Adhesive Systems in Cavities Prepared With Er,Cr:YSGG, Er:YAG Laser and Bur. *Micros Res Tech* 2007; 1: 1-7.

Awliya WY, Akpata ES. Effect of fluorosis on shear bond strength of glss ionomer-based restorative materials to dentin. *J Prosthet Dent* 1999; 81: 290-293.

Baschog W, Suetterlin R, Hefti A, et al. Confocal laser scanning microscopy and scanning electron microscopy of Ti-implant interfaces. *Micron* 2001; 32: 33-41.

Bachmann L, Diebolder R, Hibst R, Zezell DM. Changes in chemical composition and collagen structure of dentin tissue after erbium laser irradiation. *Spec Acta Part A* 2005; 61: 263-269.

Barceleiro Mde O, de Mello JB, de Mello GS, et al. Hybrid layer thickness and morphology: the influence of cavity preparation with Er:YAG laser. *Oper Dent.* 2005; 30: 304-310.

Barros JA, Myaki SI, Nor JE, and Peters MC. Effects of bur type and conditioning on the surface and interface of denitn. *J Oral Rehabil* 2005; 32: 849-856.

Bapna MS, Gadia CM, Drummond JL. Effects of aging and cyclic loading on the mechanical properties of glass ionomer cements. *Eur J Oral Sci* 2002; 110: 330-334.

Bertrand MF, Semez G, Leforestier E, et al. Er:YAG Laser Cavity Preparation and Composite Resin Bonding With a Single-Component Adhesive System: Relationship Between Shear Bond Strength and Microleakage. *Lasers Surg Med* 2006; 38: 615-623.

Boj JR, Hernandez M, Espasa E, Poirier C. Laser treatment of an oral papilloma in the pediatric dental office: a case report. *Quintessence Inter* 2007; 38: 307-312.

Botelho MG. The microtensile bond strength of Fuji IX glass ionomer cement to antibacterial conditioned dentin. *Oper Dent.* 2005; 30: 311-317.

Bouillaguet S, Ciucchi B, Jacoby T, et al. Bonding characteristics to dentin walls of class II cavities, in vitro. *Dent Mater* 2001; 7: 316-321.

Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1995; 34: 849-853.

Burke Jr JE, Hoke J, Gomes E, et al. Wet versus dry enamel ablation by Er:YAG laser. *J Prosthet Dent* 1992; 67: 847-851.

Burrow MF, Nopnakeepong U, and Phrukkanon S. A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentin. *Dent Mater* 2002; 18: 239-245.

Carmerlingo C, Lepore M, Gaeta GM, et al. Er:YAG laser treatments on dentine surface: micro-Raman spectroscopy and SEM analysis. *J Dent* 2004; 32: 399-405.

Cavalcanti BN, Lage-Marques JL, Rode SM. Pulpal temperature increases with Er:YAG laser and high-speed handpieces. *J Prosthet Dent* 2003; 90: 447-451.

Ceballos L, Osorio R, Toledano M, et al. Microleakage of composite restorations after acid or Er-YAG laser cavity treatment. *Dent Mater* 2001; 17: 340-346.

Ceballos L, Toledano M, Osorio R, et al. Bonding to Er-YAG-laser treated Dentine. *J Dent Res* 2002; 81: 119-122.

Cecchini RCM, Zezell DM, de Oliveira E. Effect of Er:YAG Laser on Enamel Acid Resistance: Morphological and Atomic Spectrometry Analysis. *Lasers Surg Med* 2005; 37: 366-372.

Cehreli SB, Gungor HC, Karabulut E. Er,Cr:YSGG Laser Pretreatment of Primary Teeth for Bonded Fissure Sealant Application: A Quantitative Microleakage study. *J Adhes Dent* 2006; 8: 381-386.

Celik EU, Ergucu Z, Turkun LS, et al. Shear bond strength of different adhesives to Er:YAG laser-prepared dentin. *J Adhes Dent.* 2006; 8: 319-25.

Chitnis D, Dunn WJ, Gonzales DA. Comparison of in-vitro bond strengths between resin-modified glass ionomer, polyacid-modified composite resin, and giomer adhesive systems. *Am J Orthod Dentofacial Orthop* 2006; 129: 330.e11-16.

Cho E, Kopel H, White SN. Moisture susceptibility of resin-modified glass-ionomer materials. *Quintessence Inter* 1995; 26: 351-358

Ciaramicoli MT, Carvalho RCR, Eduardo CP. Treatment of Cervical Dentin Hypersensitivity Using Neodymium:Yttrium-Aluminum-Garnet Laser:Clinical Evaluation. *Lasers Surg Med* 2003; 33: 358-362

Coluzzi DJ. Lasers in dentistry-wonderful instruments or expensive toys? *International Congress Series* 2003; 1248: 83-90

Compton AM, Meyers CE Jr, Hondrum SO, et al. Comparison of the shear bond strength of a light-cured glass ionomer and a chemically cured glass ionomer for use as an orthodontic bonding agent. *Am J Orthod Dentofacial Orthop* 1992; 101: 138-44.

Corona SAM, Borsatto MC, Pecora JD, et al. Assessing microleakage of different class V restoration after Er:YAG laser and bur preparation. *J Oral Rehab* 2003; 30: 1008-1014

Coutinho E, Yoshida Y, Inoue S, et al. Gel phase formation at resin-modified glass-ionomer/tooth interface. *J Dent Res* 2007; 86: 656-61.

Czarnecka B, Nicholson JW. Ion release by resin-modified glass-ionomer cements into water and lactic acid solutions. *J Dent.* 2006; 34: 539-43.

Czarnecka B, Nicholson JW. A preliminary study of the interaction of glass ionomer dental cements with amino acids. *Dent Mater* 2006; 22: 133-137.

Czarnecka B, Nosowicz PD, Saw HL, et al. Shear bond strengths of glass ionomer cements to sound and to prepared carious dentine. *J Mater Sci: Mater Med* 2007; 18: 845-849.

Dailey M, Marrs G, Satz J, et al. Concepts in Imaging and Microscopy: Exploring Biological Structure and Function with Confocal Microscopy. *Biol Bull* 1999; 197: 115-122.

Darling M, Hill R. Novel polylkenoate (glass-ionomer) dental cements based on zinc silicate glasses. *Biomaterials* 1994; 15: 299-306.

Dankner E, Neev J, Stabholz A, Rotstein I. Effect of XeCl-308 excimer laser on the mineral content of human dentin. *Endod Dent Traumatol* 1997; 13: 234-237.

Da Costa Ribeiro A, Norqueira GE, Antoniazzi JH, et al. Effect of diode (810 nm) laser irradiation to root canal walls: thermographic and morphological studies. *J Endodon* 2007; 33: 252-255.

Davidson CL and Mjor IA. Advance in Glass-Ionomer Cements (1999). Quintessence Publishing.

De Carvalho RCR, de Freitas PM, Otsuki M, et al. Influence of Er:YAG Laser Beam Angle, Working Distance, and Energy Density on Dentin Morphology: An SEM Investigation. *J Oral laser Applications* 2005; 5: 237-243.

De Carvalho RCR, de Freitas PM, Otsuki M, et al. Micro-shear bond strength of Er:YAG-laser-treated dentin. *Lasers Med Sci* 2008; 23: 117-124.

De Moor RJG and Delme KIM. Erbium Lasers and Adhesion to Tooth Structure. *J Oral Laser Appli* 2006; 6: 7-21.

De Munck J, Van Meerbeek B, Inoue S, et al. Micro-tensile bond strengths of two adhesives to Erbium:YAG-lased vs bur-cut enamel and dentin. *Am J Dent* 2002; 15: 322-329.

de Souza-Gabriel AE, Chinelatti MA, Borsatto MC, et al. Effect of Er:YAG laser irradiation distance on superficial dentin morphology. *Am J Dent.* 2006; 19: 217-221.

de Souza-Zaroni WC, Nhani VT, Ciccone-Nogueira JC,et al. Shear bond strength of glass-ionomer cements to air-abraded dentin. *J Adhes Dent.* 2006 Aug; 8: 233-237.

Delme KI, Deman PJ, De moor RJ. Microleakage of class V resin composite restoration after conventional and Er:YAG laser preparation. *Oral Rehabil* 2005; 32: 676-85.

Deppe H, Horch HH, Neff A. Conventional versus CO2 laser-assisted treatment of peri-implant defects with the concomitant use of pure-phase beta tricalcium phosphate: a 5 year clinical report. *Int J Oral Maxillofac Implants* 2007; 22: 79-86.

Donadio-Moura J, Gouw-Soares S, de Freitas PM, et al. Tensile bond strength of a flowable composite resin to Er:YAG laser treated dentin. *Laser Surg Med* 2005; 36: 351-355.

Dostalova T, Jelinkova H, Krejsa O, et al. Evaluation of the surface changes in enamel and dentin due to possibility of thermal overheating induced by Erbium:YAG laser radiation. *Scanning Micros* 1996; 10: 285-290.

Dunn WJ, Davis JT, Bush AC. Shear bond strength and SEM evaluation of composite bonded to Er:YAG laser-prepared dentin and enamel. *Dent Mater* 2005; 21: 616-624.

Eick JD, Wilko RA, Anderson CH, et al. Scanning electron microscopy of cut tooth surfaces and identification of debris by use of the electron microprobe. *J Dent Res* 1970; 49: 1359-1368.

Eick JD, Gwinnett AJ, Pashley DH, et al. Current concepts on adhesion to dentin. *Crit Rev Oral Biol Med.* 1997; 8: 306-35.

Ergucu Z, Celik EU, Turkun M. Microleakage study of different adhesive systems in class V cavities prepared by Er,Cr:YSGG laser and bur preparation. *Gen Dent* 2007; 55: 27-32.

Ersin NK, Aykut A, Candan I, et al. The effect of a chlorhexidine containing cavity disinfectant on the clinical performance of high-viscosity glass-ionomer cement following ART: 24-month results. *Am J Dent* 2008; 21: 39-43.

Eversole LR, Kimmel AI. Pulpal response to cavity preparation by an erbium, chromium:YSGG laser-powered hydrokinetic system. *J Am Dent Assoc* 1997; 128 : 1099-1106.

Eugenio S, Sivakumar M, Vilar R, et al. Characterisation of dentin surface processed with KrF excimer laser radiation. *Biomaterials* 2005; 26: 6780-6787.

Fano L, Fano V, Ma WY, et al. Structure of dental glass-ionomer cements by confocal fluorescence microscopy and stereomicroscopy. *Biomaterials* 2001; 22: 2353 - 2358.

Farrell CV, Johnson GH, Oswald MT, et al. Effect of cement selection and finishing technique on marginal opening of cast gold inlays. *J Prosthet Dent* 2008; 99: 287-292.

Ferrari M. and Davidson CL. Interdiffusion of a traditional glass ionomer cement into conditioned dentin. *Am J Dent* 1997; 10: 295-297.

Featherstone JDB, Nelson DGA. Laser effects on dental hard tissue. *Adv Dent Res* 1987; 1: 21-26.

Fleming MG and Maillet WA. Photopolymerization of composite resin using the argon laser. *J Can Dent Assoc* 1999 Sep; 65: 447-50.

Fontana CR, Kurachi C, Mendoca CR, et al. Microbial Reduction in Periodontal Pockets under Exposition of a Medium Power Diode Laser: An Experimental Study in Rats. *Lasers Surg Med* 2004; 35: 263-268.

Freiberg RJ and Cozean C. Pulsed erbium laser ablation of hard dental tissue: the effects of atomized water spray vs water surface film. *Proc SPIE* 2002; 1605-7422: 74-79.

Fried D. IR laser ablation of dental enamel. *Proc SPIE* 2000; 3910: 136-138.

Fried D, Ashouri N, Breunig T, et al. Mechanism of water augmentation during IR laser ablation of dental enamel. *Lasers Surg Med* 2002; 31: 186-193.

Fuentes V, Ceballos L, Osorio R, et al. Tensile strength and microhardness of treated human dentin. *Dent Mater* 2004; 20: 522-529.

Gaspirc B and Skaleric U. Morphology, chemical structure and diffusion processes of root surface after Er:YAG and Nd:YAG laser irradiation. *J Clin Periodontol* 2001; 28: 508-516.

Glasspoole EA, Erickson RL, Davidson CL. Effect of surface treatment on the bond strength of glass ionomers to enamel. *Dent Mater* 2002; 18: 454-462.

Geraldo-Martins VR, Tanji EY, Wetter NU, et al. Intrapulpal temperature during preparation with the Er:YAG laser: an in vitro study. *Photomed Laser Surg* 2005; 23: 182-6.

Geusic JE, Marcos HW, Van Uitert LG. Laser oscillations in Nd-doped yttrium aluminum, yttrium gallium, and gadolinium garnets. *Appl Phys Lett* 1964; 4: 182.

Goldstein RE. Multipurpose Laser Offers Unique Approach to Restorative Dentistry. *Dent Today* 1999; 18: 1-4.

Gonzale M, Banderas JA, Rodriguez V, et al. Particle induced X-ray emisión and scanning electrón microscopic analyses of the effects of CO_2 laser irradiation on dentinal structure. *J Dent* 1999 Nov; 27: 595-600.

Grgurevic J, Grgurevic L, Meletic I, et al. In Vitro Study of the Variable Square Pulse Er:YAG Laser Cutting Efficacy for Apicectomy. *Lasers Surg Med* 2005; 36: 347-350.

Guggenberger R, May R, Stefan KP. New trends in glass-ionomer chemistry. *Biomaterials* 1998; 19: 479-483.

Hadley J, Eversole LR, Gornbein JA. A laser-powered hydrokinetic system for caries removal and cavity preparation. *J Am Dent Assoc* 2000; 131: 777-85.

Hashimoto M, Ohono H, Kaga M, et al. Fractured surface characterization: wet versus dry bonding. *Dent Mater* 2002; 18: 95-102.

Harashima T, Kinoshita J, Kimura Y, et al. Morphological Comparative Study on Ablation of Dental Hard Tissue at Cavity Preparation by Er:YAG and Er,Cr:YSGG lasers. *Photomed. Laser Surg.* 2005; 23: 52-55.

Hewlett ER, Caputo AA, and Wrobel CD. Glass ionomer bond strength and treatment of dentin with Polyacrylic acid. *J Prosthet Dent* 1991; 66: 767-772.

Hickel R, Garcia-Godoy F. Clinical results and new developments of direct posterior restorations. *Am Dent* 2000; 13: 41-54.

Hibst R, Keller U. Experimental Studies of the Application of the Er:YAG laser on Dental Hard Substances: I Measurement of the Ablation Rate. *Lasers Surg Med* 1989; 9: 338-344.

Hildebrand NK, Raboud DW, Heo G, et al. Argon laser vs conventional visible light cured orthodontic bracket bonding: An in-vivo and vitro study. *Am J Orthood Dentofacial Orthop* 2007; 131: 530-536.

Hoke JA, Burkes EJ Jr, Gomes ED, et al. Erbium:YAG (2.94μm) Laser Effects on Dental Tissues. *J Laser Appl* 1990; Summer/Fall; 2: 61-65.

Hossain M, Nakamura Y, Yamada Y, et al. Ablation depths and morphological changes in human enamel and dentin after Er:YAG laser irradiation with or without water mist. *J Clin Laser Med Surg* 1999; 17: 105-9.

Hossain M, Nakamura Y, Kimura Y, et al. Acquired acid resistance of dental hard tissue by CO_2 laser irradiation. *J Clin Laser Med Surg* 1999; 17: 223-6.

Hossain M, Nakamura Y, Yamada Y, et al. Effects of Er,Cr:YSGG laser irradiation in human enamel and dentin: ablation and morphological studies. *J Clin Laser Med Surg* 1999; 17: 155-9.

Hossain M, Yamada Y, Murakami Y, et al. Compositional and structural changes of human dentin following caries removal by Er,Cr:YSGG laser irradiation in primary teeth. *J Clin Pediatr Dent* 2002; 26: 377-82.

Hossain M, Nakamura Y, Tamaki Y, et al. Atomic analysis and knoop hardness measurement of the cavity floor prepared by Er,Cr:YSGG laser irradiation in vitro. *J Oral Rehab* 2003; 30: 515-521.

Irie M, Maruo Y, Nishgawa G, et al. Class I Gap-formation in highly-viscous glass ionomer restorations delayed vs immediate polishing. Oper Dent 2008; 33: 196-202.

Ishikawa I, Sasaki KM, Aoki A, et al. Effects of Er:YAG on periodontal therapy. *J Int Acad Periodontol.* 2003; 5: 23-28.

Ishizaka Y, Eguro T, Maeda T, et al. Effects of Er:YAG Laser Irradiation on Human Dentin: Polarizing Microscopic, Light Microscopic and Microradiographic Observations, and FT-IR Analysis. *Lasers Surg Med* 2002; 31: 171-176.

Jacuques P and Hebling J. Effect of dentin conditioners on the microtensile bond strengths of self-etching primer adhesive. *Dent Mater* 2005; 21:103-109.

Jaffery IW, Lawrenson B, Saunders EM, et al. Dentinal temperature transients cause by exposure CO_2 laser irradiation and possible pulp damage. *J Dent* 1990; 18: 31-6.

Itota T, Carrick TE, Yoshiyama M, et al. Fluoride release and recharge in giomer, compomer, and resin composite. *Dent Mater* 2004; 20: 789-795.

Kameyama A, Kawada E, Takizawa M, et al. Influence of Different Acid Conditioners on the Tensile Bond Strength of 4-META/MMA-TBB Resin to Er:YAG Laser-Irradiated Bovine Dentin. *J Adhesive Dent* 2000; 2: 297-304.

Kataumi M, Nakajima M, Yamada T, et al. Tensile bond strength and SEM evaluation of Er:YAG laser irradiation dentin using dentin adhesive. *Dent Mater* 1998; 17: 125-138.

Kayano T, Ochiai S, Kiyono K, et al. Effects of Er:YAG laser irradiation on human extracted teeth. *J Clin Laser Med Surg* 1991; 9: 147-150.

Keller U, Hibst R, Geurtsen W, et al. Erbium:YAG laser application in caries therapy. Evaluation of patient perception and acceptance. *J Dent* 1998; 26: 649-656.

Keller U, Hibst R. Ultrastructural changes of enamel and dentin following Er:YAG laser irradiation on teeth. *SPIE* 1990; 1200: 408-415.

Kelsey WP 3rd, Blankenau RJ, Powell GL. Application of the argon laser to dentistry. *Lasers Surg Med.* 1991; 11: 495-8.

Kim K-S. Advances of laser dentistry in Korea and various factors of Er:YAG laser effecting the ablation rate of dental hard tissue. *International Congress Series* 2003; 1248: 45-50.

Kim KS, Kim ME, Sin EJ. Irradiation Time and Ablation Rate of Enamel in Contact and Non-contact Irradiation with Er:YAG laser. *Photomed Laser Surg* 2005; 23: 216-218.

Knight GM, McIntyre JM, Craig GG, et al. Electron probe microanalysis of ion exchange of selected elements between dentine and adhesive restorative materials. *Aust Dent J.* 2007; 52: 128-32.

Kohara KE, Hossaine M, Kimura Y, et al. Morphological and Microleakage Studies of the Cavities Prepared by Er:YAG Laser Irradiation in Primary Teeth. *J Clin Laser Med Surg* 2002; 20: 141-147.

Konishi N, Fried D, Staninec M, et al. Artificial caries removal and inhibition of artificial secondary caries by pulsed CO2 laser irradiation. *Am J Dent* 1999; 12: 213.

Koort HJ and Frentzen M. YAG-Lasers in Restorative Dentistry A histological investigation. SPIE 1992; 1643: 403-411.

Kreisler M, Kohnen W, Beck M, et al. Efficacy of $NaOCl/H_2O_2$ Irrigation and GaAlAs Laser in Decontamination of Root canals In Vitro. *Laser Surg Med* 2003; 32: 189-196.

Kugel G. Direct and indirect adhesive restorative materials: A review. *Am J Dent* 2000; 13: 35-40.

Lalh MS, Titley KC, Torneck CD, et al. Scanning Electron Microscopic Study of the Interface of Glass Ionomer Cement Sealer and Condition Bovine Dentin. *J Endodon* 1999; 25: 743-746.

Lee BS, Lin CP, Lin FH, et al. Ultrastructural Changes of Human Dentin After Irradiation by Nd:YAG Laser. *Lasers Surg Med* 2002; 30: 246-252.

Lee BS, Lin CP, Lin FH, et al. Effect of Nd:YAG laser irradiation on hardness and elastic modulus of human dentin. *J Clin Laser Med Surg* 2003; 21: 41-46.

Leyhausen G, Abtahi M, Karbakhsch M, et al. Biocompatibility of various light-curing and one conventional glass-ionomer cement. *Biomaterials* 1998; 19: 559-564.

Liberman R, Eli I, Imber S, et al. Glass ionomer cement restorations: the effects of lasing the cavity walls on marginal microleakage. *Clin Prevent Dent* 1990; 12: 5-8.

Lin A, McIntyre NS, Davidson RD. Studies on the adhesion of glass-ionomer cements to dentin. *J Dent Res.* 1992 Nov; 71: 1836-1841.

Lin S, Caputo AA, Eversole LR, et al. Topographical characteristics and shear bond strength of tooth surface cut with a laser-powered hydrokinetic system. *J Prosthet Dent* 1999; 82: 451-455.

Liu J-F, Liu Y, Stephen HC. Optimal Er:YAG laser energy for preventing enamel demineralisation. *J Dent 2006*; 34: 62-66.

Lukac M, Marincek M, Grad L. Super VSP Er:YAG Pulses for Fast and Precise Cavity Preparation. *J Oral Laser Appli* 2004; 4: 171-173.

Mahoney E, Holt A, Swain M, et al. The hardness and modulus of elasticity of primary molar teeth:an ultra-micro-indentation study. *J Dent* 2000; 20: 589-594.

Maiman TH. Stimulated optical radiation by ruby. *Nature* 1960; 187: 493-494.

Malmstrom HS, McCormack SM, Fried D, et al. Effect of CO_2 laser on pulpal temperature and surface morphology: an in vitro study. *J Dent* 2001; 29: 521-529.

Marquez F, Quintana E, Roca I, et al. Physical-mechanical effects of Nd:YAG laser of sound dental enamel. *Biomaterials* 1993; 14: 313.

Matsumoto K, Hossain MM, Kawano H, Kimura Y. Clinical assessment of Er,Cr:YSGG laser application for cavity preparation. *J Clin Laser Med Surg* 2002; 20: 17-21.

Matsumoto K, Mazeki K, Kimura Y. Clinical dental application of Er:YAG laser for Class V cavity preparation. *J Clin Laser Med Surg* 1996; 14: 123-7.

McCabe JF. Resin-modified glass-ionomers. *Biomaterials* 1998; 19: 521-527.

McCormack SM, Fried D, Featherstone JDB, Glena RE, Seka W. Scanning Electron Microscope Observations of CO_2 Laser Effects on Dental Enamel. *J Dent Res* 1995; 74:1702-1708.

McDonough WG, Antonucci JM, He J, et al. A microshear test to measure bond strengths of dentin-polymer interfaces. *Biomaterials* 2002; 23: 3603-3608.

McLean JW ,Wilson AD. The clinical development of the glass-ionomer cements. I Formulation and properties. *Aus Dent J* 1977; 22: 31-36.

Mehl A, Kremers L, Salzmann K, et al. 3D volume-ablation rate and thermal side effects with the Er:YAG and Nd:YAG laser. *Dent Mater* 1997; 13: 246-451.

Meister J, Franzen R, Forner K, et al. Influence of the water content in dental enamel and dentin on ablation with erbium YAG and erbium YSGG lasers. *J Bio Optic* 2006; 11: 034030-1-7.

Melcer J, Melcer F, Dejardin J, et al. Apport du laser a CO_2 dans le traitement des foyers infectieux d'origine dentaire. *Isced News* 1981; 20: 39-43.

Melendez EJ, Arcoria CJ, Dewald JP, et al. Effect of laser etch on bond strengths of glass ionomers. *J Prosthet Dent* 1992; 67: 307-12.

Mercer C. Lasers in Dentistry: A Review. Part 1. *Dental Update* 1996: 74-78.

Messias DCF, de Souza-Gabriel AE, Palma-Dibb, et al. Efficiency and Effectiveness of Er:YAG Laser on Carious Tissue Removal. *J Oral Laser Appli* 2006; 6: 181-186.

Miller M, Truhe T. Lasers in Dentistry: An Overview. *JADA* 1993; 124: 32-35.

Mitra SB. In vitro Fluoride Release from a Light-cured Glass-ionomer Liner/Base. *J Dent Res* 1991; 70: 75-78.

Mitra SB. Adhesion to Dentin and Physical Properties of a light-cured Glass-ionomer Liner/Base. *J Dent Res* 1991; 70: 72-74.

Miyakawa W, Pizzo AM, Salvadori MCBS, et al. Cavity generation in dental enamel using a copper-HyBrID laser. *J Mater Sci* 2007; 27: epublic ahead of print.

Mount GJ. Glass ionomer cement and future research. *Am J Dent* 1994; 7: 286-292.

Mount GJ. Clinical performance of glass-ionomers. *Biomaterials* 1998; 19: 573-579.

Moshonov J, Stabholz A, Bar-Hiel R, et al. Chemical analysis and surface morphology of enamel and dentin following 9.6μ CO_2 laser irradiation versus high speed drilling. *J Dent* 2005; 33: 427-432.

Mullejans R, Eyrich G, Raab WH, et al. Cavity preparation using a superpulsed 9.6-micron CO_2 laser—a histological investigation. *Lasers Surg Med* 2002; 30(5): 331-6.

Myers TD, Myers WD. In vivo caries removal utilizing the YAG laser. *J Mich Dent Assoc* 1985; 67:66-69

Nakabayashi N, Kojima K, Yasuda N. Hybrid layer as a dentin-bonding mechanism. *J Esthet Dent* 1982; 3: 133-138.

Nammour S, Demortier G, Florio P, et al. Increase of Enamel Fluoride Retention by Low Fluence Argon Laser In Vivo. *Lasers Surg Med* 2003; 33: 260-263.

Nair PNR, Baltensperger MM, Luder HU, et al. Pulpal Response to Er:YAG Drilling of Dentine in Healthy Human Third Molars. *Lasers Surg Med* 2003; 32: 203-209.

Nagai K, Kinoshita JI, Kimura Y, et al. Morphological and Compositional Changes in Human Teeth following 9.6 μm CO2 Laser Irradiation In Vitro. *J Oral Laser Appli* 2006; 6: 265-276.

Ngo HC, Mount G, Mc Intyre J, et al. Chemical exchange between glass-ionomer restorations and residual carious dentine in permanent molars: an in vivo study. *J Dent* 2006; 34: 608-13.

Nicholson JW. Chemistry of glass-ionomer cements: a review. *Biomaterials* 1998; 19: 485-494.

Niu W, Eto JN, Kimura Y, et al. A study on microleakage after resin filling of class V cavities prepared by Er:YAG laser. *J Clin Laser Med Surg* 1998; 16: 227.

Opdam NJ, Bronkhorst EM, Roeters JM, et al. Longevity and reasons for failure of sandwich and total etch posterior composite restorations. *J Adhes Dent* 2007; 9: 469-475.

Palma-Dibb RG, de Castro CG, Ramos RP, et al. Bond strength of glass-ionomer cements to caries-affected dentin. *J Adhes Dent.* 2003; 5: 57-62.

Papagiannoulis L, and Eliades G. *In vivo* vs *in vitro* anticariogenic behavior of glass-ionomer and resin composite restorative materials. *Dent Mater* 2002; 18: 561-569.

Parker S. Introduction, history of lasers and laser light production. *Br Dent J* 2007; 1: 21-31.

Parker S. Laser-tissue interaction. *Br Dent J* 2007; 2: 73-80.

Parker S. Low level laser use in dentistry. *Br Dent j* 2007; 3: 131-138.

Patel CKN. Continuous-wave laser action on vibrational-rotational transition of CO_2 laser. *Phys. Rev. A* 1964;136: 1187-93.

Pashley DH, Sano H, Ciucchi B, et al. Adhesión testing of dentine bonding agents: A review. *Dent Mater* 1995; 11: 117-125.

Pelagalli J, Gimbel CG, Hansen RT, et al. Investigational study of the use of Er:YAG laser versus dental drill for caries removal and cavity preparation–phase I. *J Clin Laser Med Surg.* 1997; 15: 109-115.

Pereira PNR, Sano H, Ogata M, et al. Effect of region and dentin perfusion on bond strength of resin-modified glass ionomer cements. *J Dent.* 2000; 28: 347-354.

Piwowarczyk A, Lauer HC, Sorensen JA. In vitro shear bond strength of cementing agents to fixed prosthodontic restorative materials. *J Prosthet Dent.* 2004; 92: 265-73.

Pick RM. Using lasers in clinical in clinical dental practice. *JADA* 1993; 124: 37-47.

Pioch T, Staehle HJ, Duschner H. Applications of Confocal Laser Scanning Microscopy to Dental Bonding. *Adv. Dent Res* 1997; 14: 453-461.

Phillips RW, Drexell AB, and Healey HJ. Clinical observations on amalgam with known physical properties: final report. *Journal of the American Medical Association* 1945; 32: 325-330.

Powell GL and Blankenau RJ. Laser curing Dental Materials. *Dent Clin North Am* 2000; 44: 923-30.

Quo BC, Drummond JL, Koerbe A, et al. Glass ionomer microleakage from preparation by an Er/YAG laser or a high-speed handpiece. *J Dent* 2002; 30: 141-146.

Radatti DA, Baumgartner JC, Marshall JG. A comparison of the efficacy of Er,Cr:YSGG laser and rotary instrumentation in root canal debridement. *J Am Dent Assoc* 2006; 137: 1261-6.

Randall RC, Wilson NHF. Clinical testing of restorative materials: some historical landmarks. *J Dent* 1999; 27: 543-550.

Rosa AD, Sarma AV, Le CQ, et al. Peripheral Thermal and Mechanical Damage to Dentin With Microsecond and Sub-Microsecond 9.6 μm, 2.79 μm, and 0.355 μm Laser Pulses. *Lasers Surg Med* 2004; 35:214-228.

Re D, Augusti D, Salina S, Cerutti A, et al. In vitro Bonding to Nd:YAG Laser-treated Dentin. *J Oral Laser Appli* 2004; 4: 83-88.

Ribeiro CF, Anido AA, Pauscher FC. Marginal Leakage in Class V cavities Pretreated with Different Laser Energy Densities. *Photomed Laser Surg* 2005; 23: 313-6.

Rigby PJ, Goldie RG. Confocal Microscopy in Biomedical Research. *Croatian Medical Journal* 1999; 40: 346-352.

Rizoiu IM, DeShazer LG. New Laser-Matter Interaction Concept to Enhance Hard Tissue Cutting Efficiency. *Laser Int V* 1994; 2134: 309-317.

Rizoiu IM, Kohanghadosh F, Kimmel AI, et al. Pulpal thermal responses to an erbium, chromium:YSGG pulsed laser hydrokinetic system. *Oral Surg Oral Med Oral Patho* 1998; 86: 220-223.

Roebuck EM, Saunders WP, Whitters CJ. Influence of Erbium:YAG laser energies on the microleakage of Class V resin-based composite restorations. *Am J Dent* 2000; 13: 280-284.

Sales D, Sae-Lee D, Matsuya S, et al. Short-term fluoride and cations release from polyacid-modified composites in a distilled water, and acidic lactate buffer. *Biomaterials* 2003; 24: 1687-1696.

Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength - Evaluation of a micro-tensile bond test. *Dent Mater* 1994; 10: 236-240.

Sassi JF, Chimello DT, Borsatto MC, et al. Comparative study of the dentin/adhesive systems interface after treatment with Er:YAG laser and acid etching using scanning electron microscope. *Lasers Surg Med* 2004; 34: 385-390.

Schein MT, Bocangel JS, Eduardo G, et al. SEM evaluation of the interaction pattern between dentin and resin after cavity preparation using Er:YAG laser. *J Dent* 2003; 31: 127-135.

Schmalz G, Hiller K-A, Dorter-Aslan F. New developments in the filter test system for cytotoxicity. *J Mater Sci Mater Med* 1994; 5: 43.

Serebro L, Segal T, Nordenberg D, et al. Examination of Tooth Pulp Following Laser Beam Irradiation. *Lasers Surg Med* 1987; 7: 236-239.

Shahabi S, Ebrahimpour L, Walsh LJ. Microleakage of composite resin restorations in cervical cavities prepared by Er,Cr:YSGG laser radiation. *Aust Dent J* 2008; 53: 172-175.

Sheth KK, Staninec M, Sarma AV, et al. Selective Targeting of Protein, Water, and Mineral in Dentin Using UV and IR Pulse Lasers: The Effect on the Bond Strength to Composite Restorative Materials. *Lasers Surg Med* 2004; 35: 245-253.

Shigetani Y, Tate Y, Okamoto A, et al. A study of cavity preparation by Er:YAG laser-effect on the marginal leakage of composite resin restoration. *Dent Mater* 2002; 21: 238-249.

Silberman JJ, Dederich DN, Vergas M, et al. SEM Comparison of Acid-Etched, CO2 Laser-Irradiated, and Combined Treatment on Dentin Surface. *Laser Surg. Med.* 1994; 15: 269-276.

Smith DC. Development of glass-ionomer cement systems. *Biomaterials* 1998; 19: 467-478.

Sidhu SK. Interfacial characteristics of resin-modified glass-ionomer materials: a study on fluid permeability using confocal fluorescence microscopy. *J Dent Res.*1998; 77: 1749-59.

Sidhu SK, Watson TF. Failure of resin-modified glass-ionomers subjected to shear loading. *J Dent* 1999; 27: 373-381.

Silverstone LM. The histopathology of early approximal caries in the enamel of primary teeth. *J Dent Child* 1970; 37: 201-210.

Silvestri Jr AR, Mirkov MG, Connolly RJ. Prevention of Third Molar Tooth Development in Neonate Rat With a Long Pulse Diode Laser. *Lasers Surg Med* 2004; 35: 385-391.

Stabholz A, Neev J, Liaw LH, et al. Sealing of human dentinal tubules by XeCL 308 nm excimer laser *Lasers Surg Med* 1993; 19: 267-71.

Steel J. ART for treating root caries in older people. *J Dent Res.* 2006; 85: 929-32.

Stiesch-Scholz M, Hannig M. In vitro study of enamel and dentin marginal integrity of composite and compomer restorations placed in primary teeth after diamond or Er:YAG laser cavity preparation. *J Adhesive Dent* 2000; 2: 213-222.

Stern RH, Renger HI, Howell FV. Laser effects on vital pulps. *Br Dent J* 1969; 127:26

Sulieman M. An Overview of the Use of Lasers in General Dental Practice: 1. Laser Physics and Tissue Interactions. *Dent Update* 2005; 32: 228-236.

Sulieman M. An Overview of the Use of Lasers in General Dental Practice: 2. Laser Wavelengths, Soft and Hard Tissue Clinical Applications. *Dent Update* 2005; 32: 286-296.

Takano T. A Study on morphological and component change of toth surface irradiated by Er:YAG laser. *Japan J Conserv Dent* 1997; 40: 908-919.

Tnaumiharja M, Burrow MF, and Tyas MJ. Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentin using four dentioners. *J Dent* 2000; 28: 361-366.

Toledano M, Osorio R, Osorio E,Prati C, Carvalho RM. Microhardness of acid-treated and resin infliltrated human dentin. *J Dent* 2005; 33: 349-354.

Turkmen C, Sazak H, Gunday M. Effect of the Nd:YAG laser, air-abrasion, and acid-etchant on filling materials. *J Oral Rehabil* 2006; 33: 64-69.

Ususmez S, Orhan M, Uzumez A. Laser etching of enamel for direct bonding with an Er, Cr:YSGG hydrokinetic laser system. *Am J Orthod Dentofacial Orthopod* 2002; 122: 649-656

Usumez A, Aykent F. Bond strengths of porcelain laminate veneers to surface prepared with acid and Er,Cr:YSGG laser etching. *J Prosthet Dent* 2003; 90: 24-30

Van As G. Erbium lasers in dentistry. *Dent Clin North Am* 2004; 48(4):1017-59.

Van Dijken JWV, Kieri C, Carlen M. Longevity of Extensive class II Open-Sandwich Restoration with a Resin-modified glass ionomer cement. *J Dent Res* 1999;78: 1319-1325.

Van Dijken JW Retention of a resin-modified glass ionomer adhesive in non-carious cervical lesions. A 6-year follow-up. *J Dent.* 2005 Aug; 33: 541-7. Epub 2005 Feb 8.

Van Meerbeek B, Dhem A, Goret-Nicaise M, et al. Comparative SEM and TEM Examination of the Ultrastructure of the Resin-Dentin Interdiffusion Zone. *J Dent Res* 1993; 72: 495-501.

Van Meerbeek B, Inokoshi S, Breaem M, et al. Morphological Aspects of the Resin-Dentin Interdiffusion Zone with Different Dentin Adhesive Systems. *J Dent Res* 1992; 71: 1530-1540.

Van Meerbeek B, Vargar M, Inoue S, et al. Microscopy investigations. Techniques, results, limitations. *Am J Dent* 2000; 13(Special issue): 3-18.

Visuri SR, Gilbert JL, Wrigth DD, et al. Shear strength of composite bonded to Er:YAG laser-prepared dentin. *J Dent Res* 1996; 75: 599-605.

Visuri SR, Walsh JT, Wigdor HA. Erbium Laser Ablation of Dental Hard Tissue. *Lasers Surg Med* 1996; 18: 294-300.

Watson TF, Pagliari D, Sidhu SK, et al. Confocal microscopic observation of structural changes in glass-ionomer cements and tooth surfaces. *Biomaterials* 1998; 19: 581-588.

Wheeler CR, Fried D, Featherstone JDB, et al. Irradiation of Dental Enamel with Q-switched λ=355-nm Laser Pulses: Surface Morphology, Fluoride Adsorption, and Adhesion to Composite Resin. *Lasers Surg Med.* 2003; 32: 310-317.

Wilson AD. Characterization of glass-ionomer cements 4. Effect of molecular weight on physical properties. *J Dent* 1977; 5: 117-120.

Wigdor HA, Walsh JT, Featherstone JDB, et al. Lasers in Dentistry. *Lasers Surg Med* 1995; 16: 103-133

Yip HK, Tay FR, Ngo HC, et al. Bonding of contemporary glass ionomer cements to dentin. *Dent Mater* 2001; 17: 456-470.

Young AM. FTIR investigation of polymerization and polyacid neutralization kinetics in resin-modified glass-ionomer dental cements. *Biomaterials* 2002; 23:3289-3295.

Yiu CK, Tay FR, King NM, et al. Interaction of resin-modified glass-ionomer cements with moist dentine. *J Dent.* 2004 ; 32: 521-30.

Yu DG, Kimura Y, Kinoshita J, et al. Morphological and atomic analytical studies on enamel and dentin irradiated by an erbium, chromium:YSGG laser. *J Clin Laser Med Surg* 2000; 18: 139-43.

Xia D, Brantley WA, Culberton BM, et al. Mechanical properties and microstructures of glass-ionomer cements. *Dent Mater* 2000; 16: 129-138.

Zharikov EV, Zhekov VI, Kulevskii LR, et al. Stimulated emission from Er^{3+} ions in yttrium aluminium garnet crystals at λ = 2.94 μm. *Sov J Quant Elect* 1975; 4: 1039-1044.

Zimehl R, Hannig M. Non metallic restorative materials based on glass ionomer cements–recent trends and developments. *Colloids and Surface A:Physicochemical and Engineering Aspects* 2000; 163: 55-62.

Publication list

Publications in International Journals

- Ekworapoj P, Sidhu SK, McCabe JF. **Effect of different power parameter of Er,Cr:YSGG laser on human dentine.** *Lasers Med Sci.*2007 Sep; 22 (3): 175 -82. [Epub ahead from print 2007 Jan 23]

- Ekworapoj P, Sidhu SK, McCabe JF. **Effect of Surface Conditioning on Adhesion of Glass Ionomer Cement to Er,Cr:YSGG-Laser-Irradiated Human Dentin.** *Photomed Laser Surg* 2007 April; 25(2): 118-23.

Publications in Proceeding of Conference (Abstract)

- Ekworapoj P, Sidhu SK, McCabe JF. (2008) **Optimal laser energy for adhesion of glass ionomer cement.** The Academy of Laser Dentistry's 15th Annual Conference and Exhibition 2008; 9th -12th April, 2008 at San Diego, USA.

- Ekworapoj P, Sidhu SK, McCabe JF. (2007) **Microtensile bond strength of different types of glass ionomer cements to Er,Cr:YSGG laser prepared dentin.** International Dental Materials Congress 2007, 21st–24th November, 2007 at Bangkok, Thailand, paper# 0015

- Ekworapoj P, Sidhu SK, McCabe JF. (2007) **Shear bond strength of glass ionomer cement to laser prepared dentin.** BSDR/NOF Joint Scientific Meeting 2007, 3rd-5th April, 2007 at Durham, UK, paper# 0216

- Ekworapoj P, Sidhu SK, McCabe JF. (2006) **Er,Cr:YSGG–dentin interaction: Micromechanical and Microscopic characterisation.** IADR Pan European Federation 2006, 13th–16th September, 2006 at Trinity College, Dublin, Ireland, paper #200

- Ekworapoj P, Sidhu SK, McCabe JF. (2006) **Microtensile bond strength of glass ionomer cement with laser prepared dentin.** 84th General Session and Exhibition of the International Association for Dental Research, 28th June – 1st July, 2006 at Brisbane, Australia, paper# 45322

- Ekworapoj P, Sidhu SK, McCabe JF. (2006) **Laser in Dentistry.** Yorkshire and North East Hub Poster Competition Networking Event, 3rd May 2006 at Northumbria University, Newcastle upon Tyne, UK

- Ekworapoj P, Sidhu SK, McCabe JF. (2004) **The glass-ionomer interface with laser and bur prepared dentine.** 82nd General Session and Exhibition of the International Association for Dental Research, 10th–13th March, 2004 at Honolulu, Hawaii, USA, paper# 45322

Photomedicine and Laser Surgery
Volume 25, Number 2, 2007

Pp. 000–000
DOI: 10.1089/pho.2006.2004

Brief Report

Effect of Surface Conditioning on Adhesion of Glass Ionomer Cement to Er,Cr:YSGG-Laser–Irradiated Human Dentin

PIYANART EKWORAPOJ, D.D.S., M.Sc., SHARANBIR K. SIDHU, B.D.S., Ph.D., and JOHN F. McCABE, Ph.D., D.Sc.

ABSTRACT

Objective: **This study aimed to show the effect of dentin conditioner on the bond strength of glass ionomer cement to Er,Cr:YSGG-laser-irradiated dentin compared to conventional bur-prepared dentin.** ***Background Data:*** **Glass ionomer cement bonds to tooth structure via direct chemical bonding without using any adhesive system. To improve the adhesion of this material, pretreating the dentin surface with a conditioner is recommended. Recently, lasers for tooth drilling have been used for cavity preparation, especially the Er,Cr:YSGG laser. However, there is a lack of research on the bond strength of glass ionomer cement to conditioned Er,Cr:YSGG-laser-irradiated dentin.** ***Methods:*** **Extracted human teeth were divided into eight experiemental groups and prepared by using either Er,Cr:YSGG laser or a conventional bur. In half of these two groups, the relevant conditioner was applied on the dentin before building up with glass-ionomer cement. After 24 h, all teeth were sectioned, and the microtensile bond strength was determined. The mode of failure was observed, and the fractured surfaces were examined by scanning electron microscopy (SEM).** ***Results:*** **The use of conditioner was found to significantly affect bond strength for laser-prepared samples (analysis of variance [ANOVA],** $p < 0.05$**). Failure analysis showed no adhesive failures in the laser-prepared teeth, and this suggests an effective bond between glass ionomer cement and Er,Cr:YSGG-laser-cut dentin.** ***Conclusion:*** **To obtain the maximum retention of a glass ionomer restoration to Er,Cr:YSGG-laser-irradiated dentin, the results of this study confirm that pretreatment of the laser-prepared dentin with dentin conditioner is advantageous.**

INTRODUCTION

ALTERNATIVE METHODS of tooth cutting and preparation using laser technology are now available. It is now a decade since the erbium laser was first accepted for tooth drilling in 1997.[1] Many investigations have reported its ability to cut enamel and dentin effectively.[1–6] Interestingly, laser-irradiated dentin surface was very rough, with irregularities and craters resembling the surface obtained by acid etching.[7–9] Hence, these irregularities may encourage adhesion between filling materials and the tooth substrate. Therefore, lasers for tooth drilling may be beneficial in enhancing the adhesion of filling material. After cavity preparation, filling materials play a major role for successful tooth restoration. Additionally, good adhesion between filling materials and the tooth surface leads to longevity of the restoration. Many dental adhesive filling materials—both salt-based and resin-based—have been developed to interact with bur-prepared tooth substrate, and there is a need to confirm that such materials will interact in a similar way with laser-prepared tissue. Glass ionomer cement is the material of choice for laser-cut dentin based on the fact that it has the ability to bond chemically to the tooth substrate without using an intermediary adhesive system unlike the composite resin materials.[10]

School of Dental Sciences, University of Newcastle, Newcastle upon Tyne, United Kingdom.

When using rotary cutting instruments, a debris zone, called the "smear layer," develops on the dentin surface.[11-13] This covering layer obliterates the dentinal tubules, resulting in low adhesion efficiency of tooth and restoration. To enhance adhesion to tooth, it is recommended that an acidic conditioner be applied on the tooth surface to remove the smear layer before tooth restoration.[14-15] However, using the laser to cut the tooth may help to promote the bonding of filling material as the laser-cut dentin provides a clean surface with opened dentinal tubules suitable for chemical and mechanical bonding.[16]

A common method to evaluate adhesion to a dentin or enamel surface is to determine the tensile or shear stress applied to a bonded specimen. The microtensile test uses a very small surface area and thus measures fewer defects occurring at the material/tooth interface. This test minimizes the variation among samples and provides an accurate method of evaluating adhesive strength.[15]

Numerous studies have been conducted to assess the bond strength of composite resin and adhesive to laser-prepared dentin and enamel.[17,18] However, there is a lack of studies dealing with glass ionomer in cavities prepared by Er,Cr:YSGG laser. This study aimed to measure the bond strength of glass ionomer cement with laser-irradiated dentin. The research hypothesis was that the laser-prepared cavities would improve the bond strength of glass ionomer cement to dentin.

METHODS

Two conventional glass ionomer cements (GICs) were used: Fuji IX (GC Co., Tokyo, Japan) and Ketac Molar (3M ESPE, Seefeld, Germany). The conditioners used were Dentin Conditioner with Fuji IX and Ketac Conditioner with Ketac Molar (supplied by the respective companies). The laser used for this study was a hydrokinetic laser (Waterlase laser), specifically a Cr,Er:YSGG laser (Biolase Inc., San Clemente, CA), with a pulse duration of 140 μsec. The lasing medium is Yttrium-Scandium-Gallium-Garnet crystal doped with Chromium and Erbium. This medium emitted a laser beam with a wavelength of 2.78 μm. The repetition rate was 20 Hz, and power output was 0–6 watts. Pulse energy varied from 0 to 300 mJ/pulse. The laser tip was a G-type with a long shank (6 mm). A high-speed handpiece (Kavo, Super-Torque 625, Biberach, Germany) and round diamond bur (Unodent, BD521, lot 53883, Israel) were used for conventional tooth preparation.

The 16 teeth used in this study were sound extracted human molar teeth without caries or any defects (i.e., cracking or fissuring), previously stored in an aqueous solution containing 0.5% chloramine, according to institutional protocols.

Specimen preparation

The occlusal surfaces of the teeth were trimmed with a high-speed diamond saw (Micro Slice 2, Metals Research Limited, Cambridge, UK) using water spray to remove one third of the crown, and subsequently ground with 600-grit sandpaper (P-800) to provide flat dentin. The cut surfaces were finished using precision lapping and a polishing machine (PM2A, Logitech, Materials Technologists Engineers, Alexandria, Scotland) with calcined alumina oxide powder of particle size 3 μm. Eight of the trimmed teeth were prepared by laser and the other eight teeth were prepared by bur. In half of each of the laser-prepared, and bur-prepared groups, the relevant dentin conditioner was applied to the dentin surface according to the manufacturer's instructions. A Tofflemire matrix holder was used to build up the glass ionomer restoration. Fuji IX and Ketac Molar capsules were then mixed with a triturator (Silamat, Vivadent, Chann, Liechtenstein) and placed on the respective dentin surfaces to a height of approximately 5 mm.

The experimental groups (two teeth each) were hence defined as follows:

Group 1: Bur preparation and Fuji IX
Group 2: Bur preparation and Ketac Molar
Group 3: Bur preparation, Dentin Conditioner application, and Fuji IX
Group 4: Bur preparation, Ketac Conditioner application, and Ketac Molar
Group 5: Laser preparation and Fuji IX
Group 6: Laser preparation and Ketac Molar
Group 7: Laser preparation, Dentin Conditioner application, and Fuji IX
Group 8: Laser preparation, Ketac Conditioner application, and Ketac Molar

After 24-h storage in deionized water at 37°C, each tooth was sectioned perpendicularly to the adhesive interface with a high-speed diamond saw with water spray to produce the typical microtensile bar-shaped specimens (approximately 1 × 1 mm^2 of adhesive surface). There were 20 specimens in each group.

Microtensile bond strength test

The dimensions of the specimens were measured by using digital Vernier Callipers (Digimatic, Mitutoyo, Tokyo, Japan). The specimens were then mounted in an SSP apparatus (single speed pump; SSP Dillon, Compact Gauges 200N, division of Weigh-Tronix Inc., Fairmont, MN) by using cyanoacrylate glue (Zapit, Dental Ventures of America Inc., Corona, CA), and tensile forces were applied at a crosshead speed of 1 mm min^{-1} until failure occurred. The microtensile bond strength was calculated in MPa derived from dividing the tensile force (N) at the time of fracture by the individually measured bond area (mm^2).

Statistical analysis

The mean bond strengths were evaluated by one-way analysis of variance (ANOVA) and Tukey's comparison, and a p value < 0.05 was considered to be significant.

Fracture analysis by light microscopy and scanning electron microscopy

After the bond strength tests, all specimens were assessed for mode of failure by using a light microscope (Meiji Techno. Co. Ltd., Saitama, Japan) at a magnification of ×22.5. The individual failure modes were noted. Representative specimens from each group were further examined by using scanning electron microscopy (SEM) via a Stereoscan S40 (Cambridge, UK).

TABLE 1. MICROTENSILE BOND STRENGTH (MPA)

	Fuji IX		*Ketac Molar*	
Conditioner	*Bur*	*Laser*	*Bur*	*Laser*
No conditioner	4.56 (2.19)[a]	5.99 (1.70)[a]	3.92 (1.98)[a]	5.61 (1.55)[a]
Dentin conditioner	5.52 (1.68)[a]	8.62 (3.86)[b]	—	—
Ketac conditioner	—	—	5.52 (1.52)[a]	8.01 (3.26)[b]

Mean values with the same superscript letter were not significantly different ($p > 0.05$).

RESULTS

Microtensile bond strength

The results of the microtensile bond strength test are shown in Table 1. The mean microtensile bond strength of glass ionomer cement (GIC) with bur preparation of dentin was 3.92–5.52 MPa. For laser-prepared dentin, the mean microtensile bond strength was in the range of 5.61–8.62 MPa. Statistical analysis using ANOVA showed that there was no significant difference in microtensile bond strength of GIC between laser-prepared dentin and bur-prepared dentin. However, laser-prepared dentin treated with dentin conditioner provided significantly higher microtensile bond strengths than bur-prepared dentin with or without dentin conditioner ($p < 0.05$). Comparing the different GICs, there was no significant difference between using Ketac Molar and Fuji IX without dentin conditioner for filling a tooth prepared by either bur or laser (Fig. 1). The results showed that the combination of tooth preparation method and surface conditioner did not significantly affect the microtensile bond strength. The microtensile bond strength of GIC increased when using laser preparation and dentin conditioner.

Fracture analysis

Examination of the fractured specimens under the light microscope revealed different patterns of failure: adhesive, cohesive, and a mixed type of failure (Table 2, and Figs. 2 and 3). In bur-prepared dentin without dentin conditioner, the failure mode for all specimens was adhesive failure. In laser-prepared dentin, the mode of failure was cohesive only or a mixed failure.

DISCUSSION

The purpose of using a dentin conditioner is to remove the smear layer and increase the wettability of the tooth substrate.[19] Dentin Conditioner supplied with Fuji IX is composed of aqueous polyacrylic acid with aluminium chloride. It is believed that aluminium chloride acts as a wetting promoter, while polyacrylic acid provides the carboxyl group for hydrogen bonding, which is later displaced by the stronger interaction of polar and ionic attraction from the GIC setting reaction.[19,20] A previous study found that the treatment of cut dentin with 25% polyacrylic acid (Ketac Conditioner) reduced the affinity of the smear layer for the dentin, leaving behind a poorly attached layer of smear debris, rather than removing it.[20] Tanumiharja et al.[21] reported that the use of surface conditioners produced no difference in bond strength of Fuji IX GP for bur preparation, which is in agreement with the results presented here.

In our study, for the laser-prepared dentin, conditioner application increased the microtensile bond strength in both Fuji IX

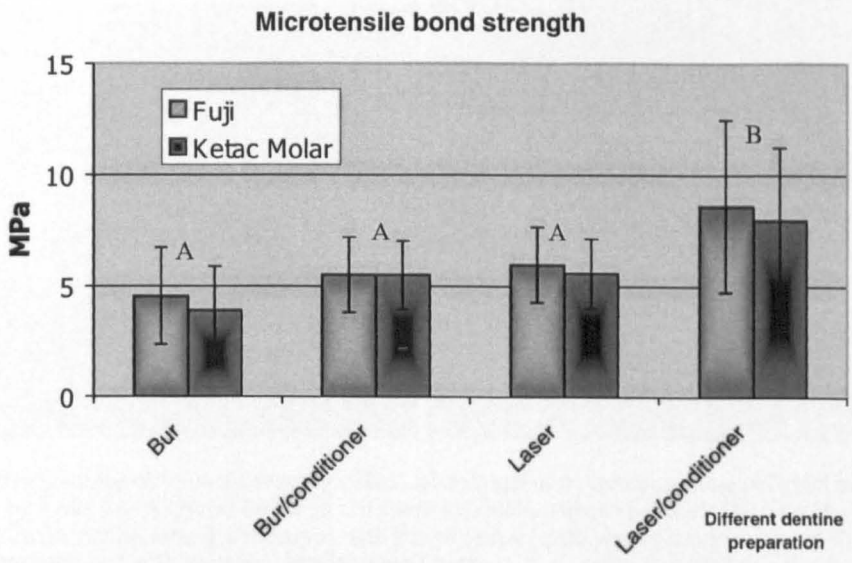


FIG. 1. The mean values of microtensile bond strength of Fuji IX and Ketac Molar with different dentin treatments.

TABLE 2. DIFFERENT FAILURE MODES OF THE SPECIMENS EXPRESSED AS A PERCENTAGE OF THE TOTAL

Test group	*Adhesive failure*	*Cohesive failure*	*Mixed failure*
Group 1	100 (20)		
Group 2	100 (20)		
Group 3	25 (5)	40 (8)	35 (7)
Group 4	95 (19)		5 (1)
Group 5		80 (16)	20 (4)
Group 6		80 (16)	20 (4)
Group 7		75 (15)	25 (5)
Group 8		90 (18)	10 (2)

The number of specimens for each mode of failure is in parentheses.

and Ketac Molar groups. This is probably because laser-irradiated dentin has no smear layer, and the dentinal tubules were unplugged with dentin debris. The other factor is thermomechanical interaction—that is, the way that lasers remove tooth tissue.[22] The thermal effect from a laser causes changes in the water content of collagen fibrils.[23] It is suggested that the thermal stability of collagen is a consequence of dentin surface alteration.[24] This may increase the dentin surface wettability, which is compatible with conventional GICs. This results in higher bond strength. The water loss in collagen fibrils after laser irradiation may reduce the hydrophilicity of the dentin substrate, which is suitable for hydrophobic polymeric materials such as the GICs. The matrix of GICs can penetrate well on low hydrophilic laser-cut dentin.

The mean microtensile bond strength of GIC to conditioned bur-cut dentin was not significantly different from that of laser-cut dentin. This can be explained by the fact that the bur-cut dentin exhibited clean and visible dentinal tubules after applying dentin conditioner, which were almost similar to the laser-cut dentin.[25–27]

All specimens in bur-prepared dentin failed adhesively, and so the interface between GIC was an area of weakness. Cohesive and mixed failures were observed in bur-prepared dentin after dentin conditioner application and the bond strength was improved, although not significantly different from unconditioned specimens. From SEM imaging on the dentin side of the fractured specimens in mixed failures, there was some GIC residue attached on the dentin surface (Fig. 2C,D).

Previous work showed that there was an interdiffusion zone of Fuji IX and conditioned dentin substrate without any tag for-

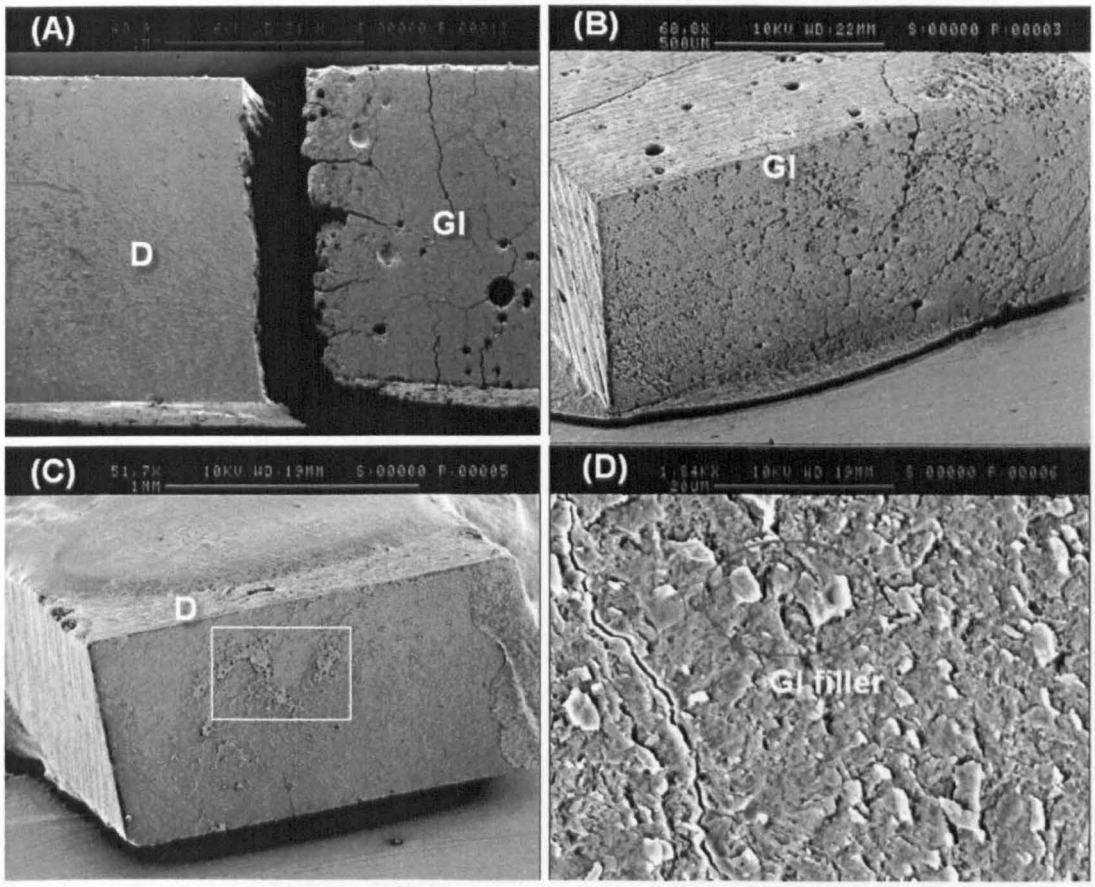


FIG. 2. Representative scanning electron microscopy (SEM) micrograph of fractured specimen of glass ionomer (GI) on dentin surface (D) prepared by a bur. (**A**) A typical fractured specimen exhibiting adhesive failure. (**B**) The glass ionomer side of the fractured specimen. (**C**) The dentin side of a specimen with mixed failure, with some glass ionomer on the surface. (**D**) Higher magnification of the area outlined in C, showing glass ionomer fillers.

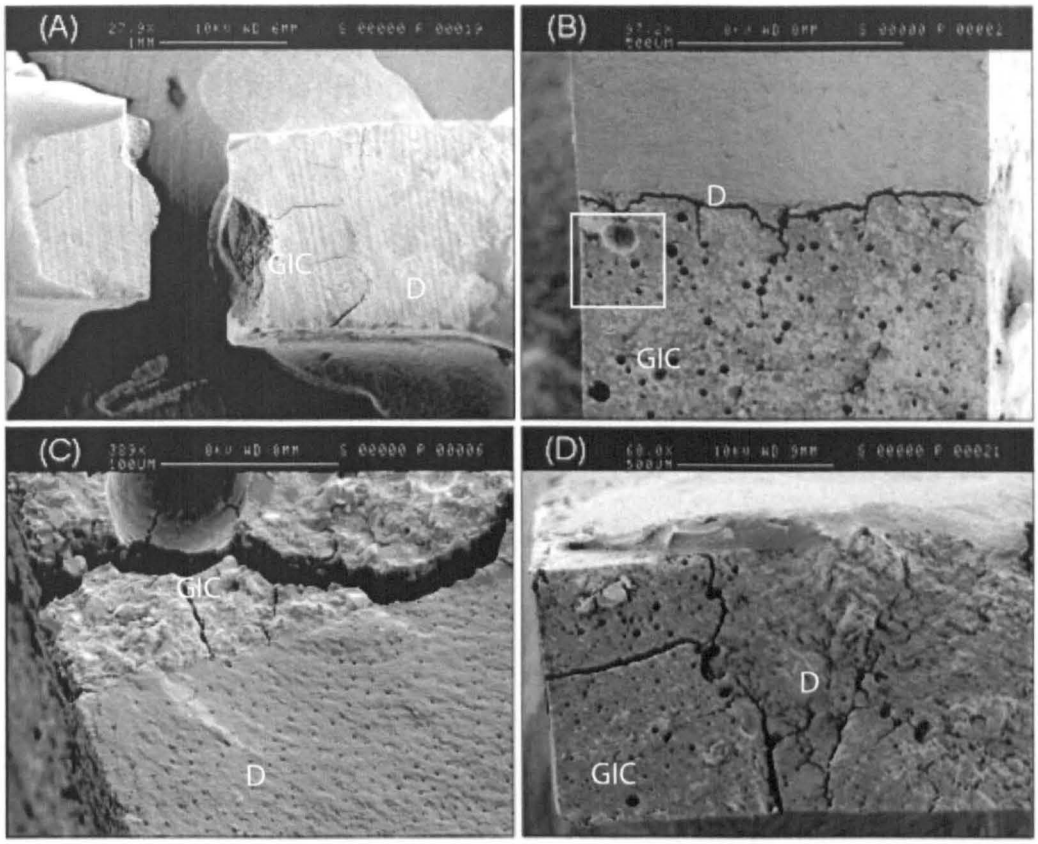


FIG. 3. Representative scanning electron microscopy (SEM) micrograph of fractured specimen of glass ionomer cement (GIC) on dentin (D) prepared by Er,Cr:YSGG laser. (**A**) With conditioner, showing cohesive failure within the GIC only. (**B**) Without conditioner in a specimen with a mixed type of failure. (**C**) Higher magnification of the area outlined in B, showing remnants of the GIC on the dentin surface in parts and adhesive failure in other areas. (**D**) The GIC side of the same fractured specimen, with areas displaying fragments of dentin (D).

mation into the dentinal tubules.[28] In the laser group, the fracture analsis showed that both cohesive and mixed failures occurred within the materials (Fig. 3). It is likely that porosity within the cement is an unpredicted factor. It probably causes a stress concentration, which allows the fracture to occur from this point.[29] This implies a tight bond between laser-treated dentin and GIC.

CONCLUSION

It may be concluded that laser preparation alone did not improve the bond strength between GIC and dentin. However, laser-irradiated dentin in combination with the application of a conditioner may improve the bond strength of glass ionomer restoration.

REFERENCES

1. Harashima, T., Kinoshita, J., Kimura, Y., et al. (2005). morphological comparative study on ablation of dental hard tissue at cavity preparation by Er:YAG and Er,Cr:YSGG lasers. Photomed. Laser Surg. 23, 52–55.
2. Wigdor, H.A., Walsh, J.T., Featherstone, J.D.B., et al. (1995). Lasers in dentistry. Laser Surg. Med. 16, 103–133.
3. Hadley, J., Eversole, L.R., and Gornbein, J.A. (2000). A laser-powered hydrokinetic system for caries removal and cavity preparation. J. Am. Dent. Assoc. 131, 777–785.
4. Featherstone, J.D.B., and Nelson, D.G.A. (1987). Laser effects on dental hard tissue. Adv. Dent. Res. 1, 21–26.
5. Hoke, J.A., Burkes, E.J., Jr., Gomes, E.D., et al. (1990). Erbium:YAG (2.94 μm) laser effects on dental tissues. J. Laser Appl. 2, 61–65.
6. Hibst, R., and Keller, U. (1989). Experimental studies of the application of the Er:YAG laser on dental hard substances: I. Measurement of the ablation rate. Lasers Surg. Med. 9, 338–344.
7. Usumez, S., Orhan, M., and Usumez, A. (2002). Laser etching of enamel for direct bonding with Er,Cr:YSGG hydrokinetic laser system. Am. J. Orthod. Dentofacial Orthop. 122, 649–656.
8. Usumez, A., and Aykent, F. (2003). Bond strength of porcelain laminate veneers to tooth surfaces prepared with acid and Er,Cr:YSGG laser etching. J. Prosthet. Dent. 90, 24–30.
9. Siberman, J.J., Dederich, D.N., Vergas, M., et al. (1994). SEM comparison of acid-etched, CO_2 laser-irradiated, and combined treatment on dentin surfaces. Laser Surg. Med. 15, 269–276.
10. Hotz, P., McLean, J.W., Sced, I., et al. (1977). The bonding of glass ionomer cements to metal and tooth substrate. Br. Dent. J. 142, 41–47.
11. Eick, J.D., Gwinnett, A.J., Pashley, D.H., et al. (1997). Current concepts on adhesion to dentin. Crit. Rev. Oral Biol. Med. 8, 306–335.
12. Eick, J.D., Wilko, R.A., and Anderson, C.H. (1970). Scanning electron microscopy of cut tooth surface and identification of debris by use of electron probe. J. Dent. Res. 49, 1359–1368.

13. Barros, J.A., Myaki, S.I., Nor, J.E., et al. (2005). Effect of bur type and conditioning on the surface and interface of dentin. J. Oral Rehabil. 32, 849–856.
14. Jacuques, P., and Hebling, J. (2005). Effect of dentin conditioners on the microtensile bond strength of a conventional and self-etching primer adhesive. Dent. Mater. 21, 103–109.
15. Burrow, M.F., Nopnakeepong, U., and Phrukkanon, S. (2002). A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentin. Dent. Mater. 18, 239–245.
16. Lin, S., Caputo, A.A., Eversole, L.R., et al. (1999). Topographical characteristics and shear bond strength of tooth surfaces cut with a laser-powered hydrokinetic system. J. Prosthet. Dent. 82, 451–455.
17. De Munck, J., Van Meerbeek, B., Yudhira, R., et al. (2002). Microtensile bond strengths of two adhesives to erbium:YAG-lased vs. bur-cut enamel and dentin. Eur. J. Oral Sci. 15, 322–329.
18. Dunn, W.J., Davis, J.T., and Bush, A.C. (2005). Shear bond strength and SEM evaluation of composite bonded to Er:YAG laser-prepared dentin and enamel. Dent. Mater. 21, 616–624.
19. Glasspoole, E.A., Erickson, R.L., and Davidson, C.L. (2002). Effect of surface treatment on the bond strength of glass ionomers to enamel. Dent. Mater. 18, 454–462.
20. Hewlett, E.R., Caputo A.A., and Wrobel, C.D. (1991). Glass ionomer bond strength and treatment of dentin with polyacrylic acid. J. Prosthet. Dent. 66, 767–772.
21. Tanumiharja, M., Burrow, M.F., and Tyas, M.J. (2000). Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentin using four conditioners. J. Dent. 28, 361–366.
22. Meister, J., Franzen, R., Forner, K., et al. (2006). Influence of the water content in dental enamel and dentin on ablation with erbium:YAG and erbium:YSGG lasers. J. Biomed. Opt. 11, 34030.
23 Bachmann, L., Diebolder, R., Hibst, R., et al. (2005). Changes in chemical composition and collagen structure of dentine tissue after erbium laser irradiation. Spectrochim. Acta A Mol. Biomol. Spectrosc. 61, 263–2639.
24. Toledano, M., Osorio, R., Osorio, E., et al. (2005). Microhardness of acid-treated and resin infiltrated human dentin. J. Dent. 33, 349–354.
25. Hossain, M., Nakamura, Y., Tamaki, Y., et al. (2003). Atomic analysis and Knoop hardness measurement of the cavity floor prepared by Er,Cr:YSGG laser irradiation *in vitro*. J. Oral Rehabil. 30, 515–521.
26. Hossain, M., Nakamura, Y., Yamada, Y., et al. (1999). Effects of Er,Cr:YSGG laser irradiation in human enamel and dentin: ablation and morphological studies. J. Clin. Laser Med. Surg. 17, 155–9.
27. Ariyaratnam, M.A., Wilson, A.S., and Blinkhorn, A.S. (1999). An analysis of surface roughness, surface morphology and composite/dentin bond strength of human dentin following the application of the Nd:YAG laser. Dent. Mater. 15, 223–228.
28. Ferrari, M., and Davidson, C.L. (1997). Interdiffusion of a traditional glass ionomer cement into conditioned dentin. Am. J. Dent. 10, 295–297.
29. Yip, H.K., Tay, F.R., Ngo, H.C., et al. (2001). Bonding of contemporary glass ionomer cements to dentin. Dent. Mater. 17, 456–470.

Address reprint requests to:
Dr. John F. McCabe
School of Dental Sciences
University of Newcastle
Framlington Place
Newcastle upon Tyne, NE2 4BW, UK

E-mail: j.f.mccabe@ncl.ac.uk

AQ1: with respect to "microtensile test" is editing correct, where text now reads "and thus measures fewer defects."

Lasers Med Sci
DOI 10.1007/s10103-006-0426-6

ORIGINAL ARTICLE

Effect of different power parameters of Er,Cr:YSGG laser on human dentine

Piyanart Ekworapoj · Sharanbir K. Sidhu · John F. McCabe

Received: 20 June 2006 / Revised: 4 August 2006 / Accepted: 16 November 2006

Abstract The aim of this work was to determine the optimal power setting of an Er,Cr:YSGG laser for cutting human dentine to produce a surface that remains suitable as a foundation on which to build and bond a dental restoration. The cutting efficiency and resulting microhardness of the dentine were evaluated for various laser power settings, and representative samples were examined by SEM. The microhardness of the dentine was significantly reduced by 30–50% ($p<0.05$, paired t test) after laser irradiation, irrespective of the power setting used. The mean ablation efficiency increased in proportion to the power setting of the laser. Although the laser power setting did not affect the extent of reduction in microhardness, it did affect the microstructure of human dentine.

Keywords Er,Cr:YSGG laser · Microhardness · Laser irradiated dentine · Laser ablation Dentine morphology

Introduction

There is currently an increased interest in the use of Er,Cr:YSGG lasers for hard tissue removal in dentistry, but many practitioners remain to be convinced of the advantages of the laser-cutting instrument. This instrument allows a nearly pain-free cutting without the need for a local anaesthetic [1] and, therefore, has the potential to be a major innovation in dentistry.

The ruby laser was the first laser system that could ablate the dentine and enamel [2, 3]. Subsequent improvements such as the Er:YAG and CO_2 lasers have also shown an ability to ablate dental hard tissue [1]. A recent tooth-cutting tool is the Er,Cr:YSGG laser. This laser is a kind of erbium laser (2.69–2.94 μm) similar to Er:YAG (2.94), Er:YLF (2.81), Er:YAP (2.73), and CTE:YAG (2.69), and emits light in the mid-infrared region at 2.79 μm [4]. The mechanism of dentine removal by this laser is called a "thermomechanical process" in which the emission laser light is absorbed by the water within the hydroxyapatite of a dental hard tissue [5, 6]. The water is then heated and evaporated, resulting in a high pressure of steam that causes a microexplosion of tooth tissue below the melting point of tooth tissue (approximately 1,200°C) [7]. Tooth surface ablation may vary from a white spot to charring, fusion, roughening, melting, recrystallization, bubble-like inclusions, numerous pores, flaking, and cracking to crater formation [8–11]. The irregularities and crater-shaped appearance of ablated dentine is comparable to the dentine surface after acid etching. This may promote the micromechanical interlocking between dental restorative materials and tooth surface.

The process of dentine removal by the laser is different from the conventional high-speed handpiece; the former induces considerable changes in the surface morphology and physical properties of the dentine. On the other hand, the thermal effect may cause both de/remineralization and deproteinization on the dentine surface [12]. The non-homogeneous structure of the dentine that consists of peritubular, intertubular, and tubular dentine has been

P. Ekworapoj (✉) · S. K. Sidhu · J. F. McCabe
School of Dental Sciences, Newcastle University,
Framlington Place,
Newcastle upon Tyne NE2 4BW, UK
e-mail: piyanart.ekworapoj@ncl.ac.uk

S. K. Sidhu
e-mail: s.k.sidhu@ncl.ac.uk

J. F. McCabe
e-mail: j.f.mccabe@ncl.ac.uk

shown to contribute to its hardness. The elastic modulus is also associated with the mineral content as well as the porosity of the structure [13]. The micromechanical properties, such as the microhardness and elasticity of the dentine, play important roles in stress distribution when the mastication forces are applied on the filling material of a restored tooth [14].

Few studies have been carried out with this laser system to survey the micromechanical properties of the dentine surface [12, 15] despite the fact that this may have a profound effect on the abilities of adhesive materials to interact with the surface. The purpose of this research was to study the micromechanical properties and microscopic appearance of the Er,Cr:YSGG-laser-prepared dentine.

Materials and methods

The laser used for this study was a Cr,Er:YSGG laser (Biolase™, Waterlase™, Millennium®, San Clemente, CA, USA) with a pulse duration of 140 μs. The lasing medium was an yttrium scandium gallium garnet crystal exchanged with erbium and chromium. This medium emits a laser light of 2.78 μm. The repetition rate was 20 Hz and a power output range of 0 to 6 W. The pulse energy varies from 0 to 300 mJ. A G6 clear sapphire tip of 600-μm diameter was used. The teeth used in the study were extracted human molars and premolars without caries or defects, stored in an aqueous solution of 0.5% chloramine T.

Tooth preparation

One third of the occlusal surfaces of 20 teeth were trimmed with a high-speed diamond saw (Micro Slice 2, Metals Research, Cambridge, England) using water spray to provide a flat dentine surface. The surfaces were then cut again to provide 2-mm-thick dentine disks. All disks were embedded in a plastic mould and the exposed surface finished using 500 grit SiC paper and, later, with calcined alumina oxide powder (particle size 3 μm).

Ablation efficiency evaluation

The dentine disks were randomly divided into four groups of five teeth (groups 1, 2, 3, 4) treated with different laser power settings: 3 W (33.9 J/cm^2), 3.5 W (39.6 J/cm^2), 4 W (45.2 J/cm^2), and 4.5 W (50.9 J/cm^2), respectively. Each specimen was placed on a movable stand, and a laser handpiece was held with a clamp to fix the distance at 1 mm. The repetition line was made free-hand by moving the specimen along the path of the laser beam in more than 60 s. All the ablation lesions were duplicated with a synthetic rubber replicating compound (Microset, Microset Products, UK) for evaluation using a 3D-laser scanning surface profiler (OSP100, Uniscan Instrument, UK).

Table 1 The means (SD) of the amount of dentine ablation using different laser power settings (n=5)

Test group	Average dentine depth (mm)	Average ablated dentine volume (mm^3)	Average ablated dentine volume per sec (mm^3/s)
Group 1 (3W)	0.15 (0.02)	0.62 (0.25)	0.01
Group 2 (3.5W)	0.28 (0.10)	1.82 (1.03)	0.03
Group 3 (4W)	0.33 (0.07)	2.00 (0.69)	0.03
Group 4 (4.5W)	0.42 (0.14)	2.91 (1.06)	0.04

Microhardness measurement

Five pyramidal–triangular indentations at a load of 200 g and a dwell time of 20 s were made on one half of each disk using a Martens microhardness tester (Zwick/Roell Z2.5, Ulm, Germany) before and after the laser application. By averaging the 25 indentations for each group, the mean microhardness value and elastic modulus before and after irradiation were automatically calculated by the computer software (TestXpert, Germany). The baseline and irradiated dentine hardness were compared using paired t tests. The relationship between the microhardness of the treated dentine and the laser power setting used was investigated by regression analysis.

Microscopic investigation

Representative specimens from each group were cut transversally through the dentine disks and immersed in an ultrasonic cleaner for 1 min. For the purposes of comparison, a further specimen was prepared in the same way but

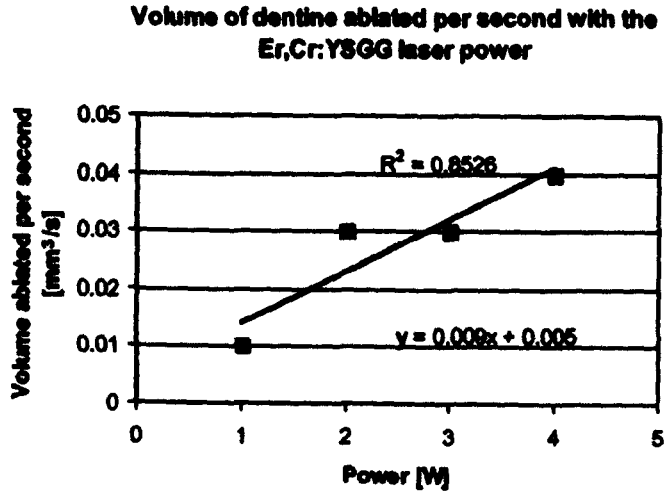


Fig. 1 The linear correlation between the volume of laser-ablated dentine per second (mm^3/s) as a function of power setting (W)

Table 2 The means (SD) of Vicker's micro-hardness using different power settings (n=5)

Test group	Baseline	Laser irradiation
Group 1 (3W)	60.85 (8.39)	42.89 (16.39)
Group 2 (3.5W)	66.18 (5.12)	30.27 (11.94)
Group 3 (4W)	62.39 (4.01)	40.41 (19.52)
Group 4 (4.5W)	64.94 (8.72)	41.27 (9.15)

surface treated using a diamond bur on the flat dentine. All specimens were mounted and sputter-coated for examination with a Stereoscan S240 scanning electron microscope (Cambridge, UK).

Results

Ablation depth evaluation

At the pulse duration of 140 μs and a pulse repetition rate of 20 Hz, the amount of dentine removed by Er,Cr:YSGG laser with various energy fluence for a given time is shown in Table 1. The mean dentine-ablated volume (mm^3) was calculated by multiplying the surface area of the irradiated dentine lesion with the ablation depth. The dentine-ablated volume per sec (mm^3/s) was calculated by dividing the dentine-ablated volume with a given time (60 s). There was an increase in the depth of ablated dentine and in the dentine-ablated volume with an increase in laser output power. The ablation efficiency showed a significant linear correlation with the power output using linear regression analysis (R^2=0.61, p<0.05) as shown in Fig. 1.

Microhardness measurement

The values of dentine hardness (Vicker's hardness number) and elastic modulus for the baseline and post-irradiation are shown in Tables 2 and 3, as well as in Figs. 2 and 3. The dentine hardness decreased significantly after laser irradiation (paired t test, p<0.05). There was no significant relationship between the increasing laser output and the reduction of dentine hardness (R^2=0.00, p>0.05). The stiffness of the irradiated dentine was not significantly different from the baseline (paired t test, p>0.05).

Table 3 The means (SD) of the elastic modulus (kN/mm^2) of the baseline and laser-irradiated dentine (n=5)

Test group	Baseline	Laser irradiation
Group 1 (3W)	16.73 (2.53)	17.73 (6.36)
Group 2 (3.5W)	20.69 (2.85)	18.74 (7.95)
Group 3 (4W)	17.46 (1.00)	14.65 (3.83)
Group 4 (4.5W)	18.51 (1.98)	19.92 (6.28)

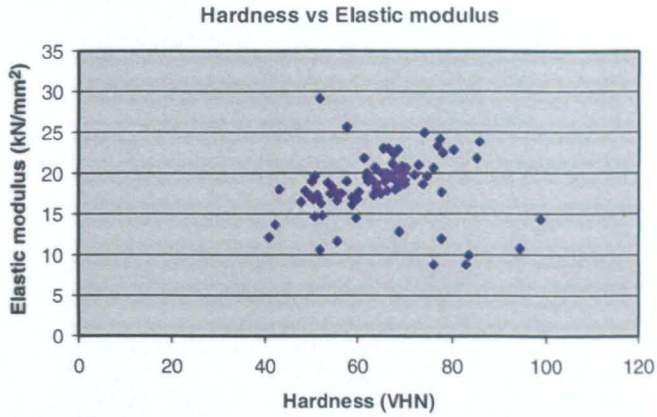


Fig. 2 The plot between hardness and elastic modulus before laser application

Microscopic investigation

The SEM appearance of bur-cut dentine showed a relatively flat topography and a smear layer (Fig. 4a and c). In contrast, the dentine appearance after the laser irradiation indicated a corrugated or wavy profile (Fig. 4b), opened dentinal tubules, and the absence of a smear layer (Figs. 4d and 5) at all power settings. Further examination of the profile at a low power setting (3 W) revealed the presence of dentine debris with tags attaching it to the underlying lased dentine (Fig. 6). At 3.5 W, there was a dentine debris on the surface without any tags (Fig. 7). In addition, it appeared that there was a glassy molten substance on the dentine (Fig. 7b). At 4 W, the irradiated dentine looked more crystalline in nature without any debris (Fig. 8a and b). When the highest setting (4.5 W) was used, a molten appearance was often noted on the surface (Fig. 8c and d).

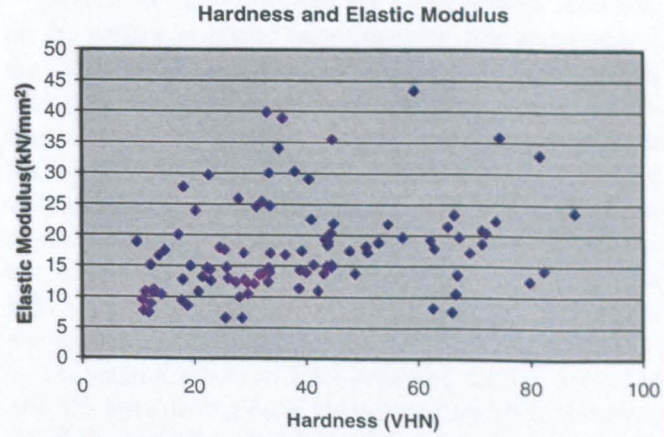


Fig. 3 The plot between hardness and elastic modulus after laser application

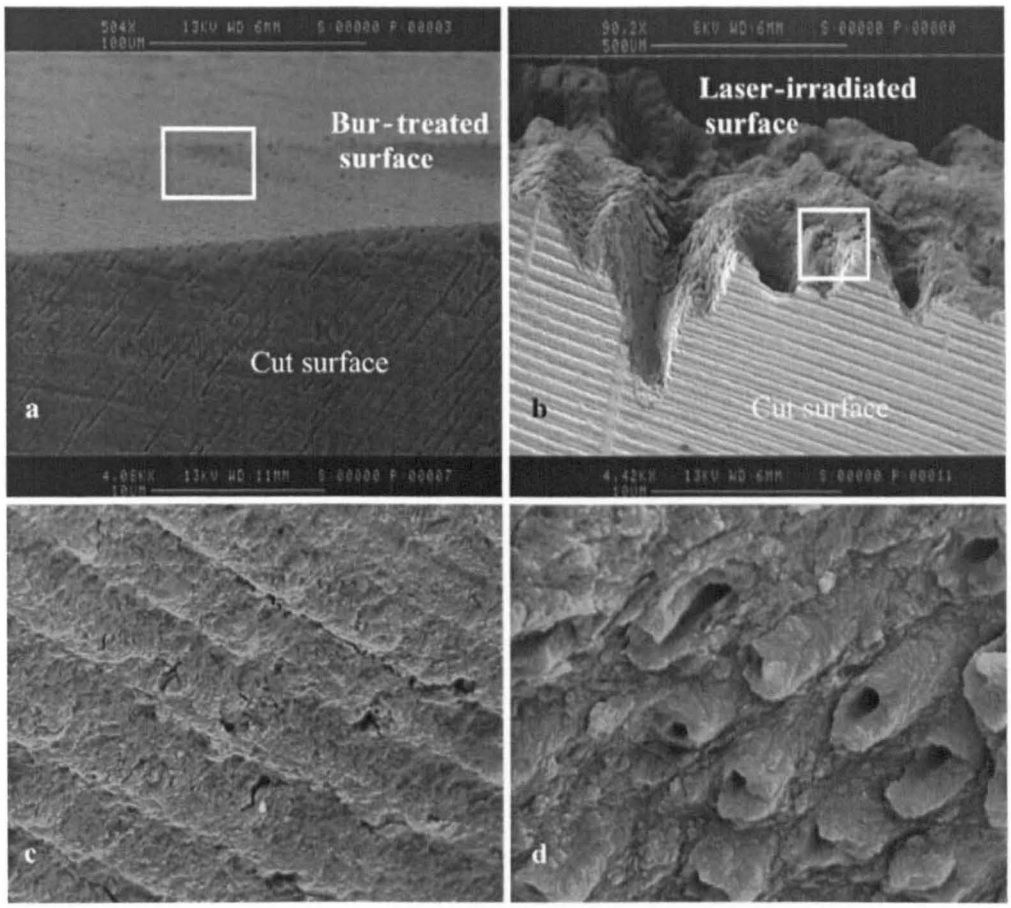


Fig. 4 SEM micrographs of transversally sectioned dentine disks: **a** typical profile of bur-cut dentine showing a flat surface; **b** typical profile of laser irradiated dentine at any power setting, showing a corrugated and wavy appearance; **c** appearance of the bur-cut surface at high magnification in the *boxed area* in Fig. 1a, showing track lines formed due to the rotary instrumentation, and presence of a smear layer; **d** higher magnification of *boxed area* in **b** showing dentinal tubules opening at different planes and possibly the depletion of intertubular dentine after laser application

The molten areas were chemically analysed using SEM–EDX (EDX, energy-dispersive x-ray; JEOL 5300-LV, Japan) with an analysis system (Rontec Edwin energy dispersive analysis, Japan). This revealed the presence of Ca, P, C, and O peaks coinciding with the molten areas as in the irradiated dentine where there was no molten mass.

Discussion

Dentine can be classified as a composite material comprising of a collagen matrix (20% by weight) dispersed in an inorganic component (70% by weight) [16, 17]. Hydroxyapatite is the main mineral content of the inorganic component, and variants (such as fluoroapatite) provide the strength and hardness for the dentine. Its elasticity and stiffness are obtained from the organic collagen matrix and water content (10% by weight). The SEM micrographs of the irradiated dentine in this study showed the depletion of the intertubular dentine (Fig. 4d), similar to the dentine surface after the preparation by Er:YAG laser and treatment with glutaraldehyde [18]. The peritubular dentine still remained, indicating more resistance to laser energy. This can be explained by the fact that the peritubular dentine has a high mineral content and lacks collagen as an organic matrix. Contrary to the intertubular dentine, it makes up 92% of the collagen matrix [19–21]. The collagen matrix is rich in water content, and laser energy is likely to be absorbed more than the peritubular dentine by the dentinal fluid within each tubule and the intertubular dentine. This study confirms the findings of previous studies that the laser-irradiated dentine was deprived of a smear layer, and the orifices of dentinal tubules were opened [11, 12].

SEM–EDX confirmed that the glassy molten substance on the surface at higher power settings was composed of the same elements as hydroxyapatite. This phenomenon indicates that some components of dentine may exhibit thermoplastic properties, whereby they turn liquid on heating and immediately return to a solid state on cooling with a water spray. This is because Er,Cr:YSGG radiation is highly absorbed not only by the hydroxyapatite of dentine but also by the protein and lipid in collagen fibre [22]. This is in accordance with previous studies that reported molten and crystalline like structures on irradiated dentine [7–9].

The microhardness of laser-irradiated dentine decreased after the laser irradiation in the order of 30–50%. However, the elastic modulus after laser irradiation was not significantly changed (Table 3). In contrast, investigations by

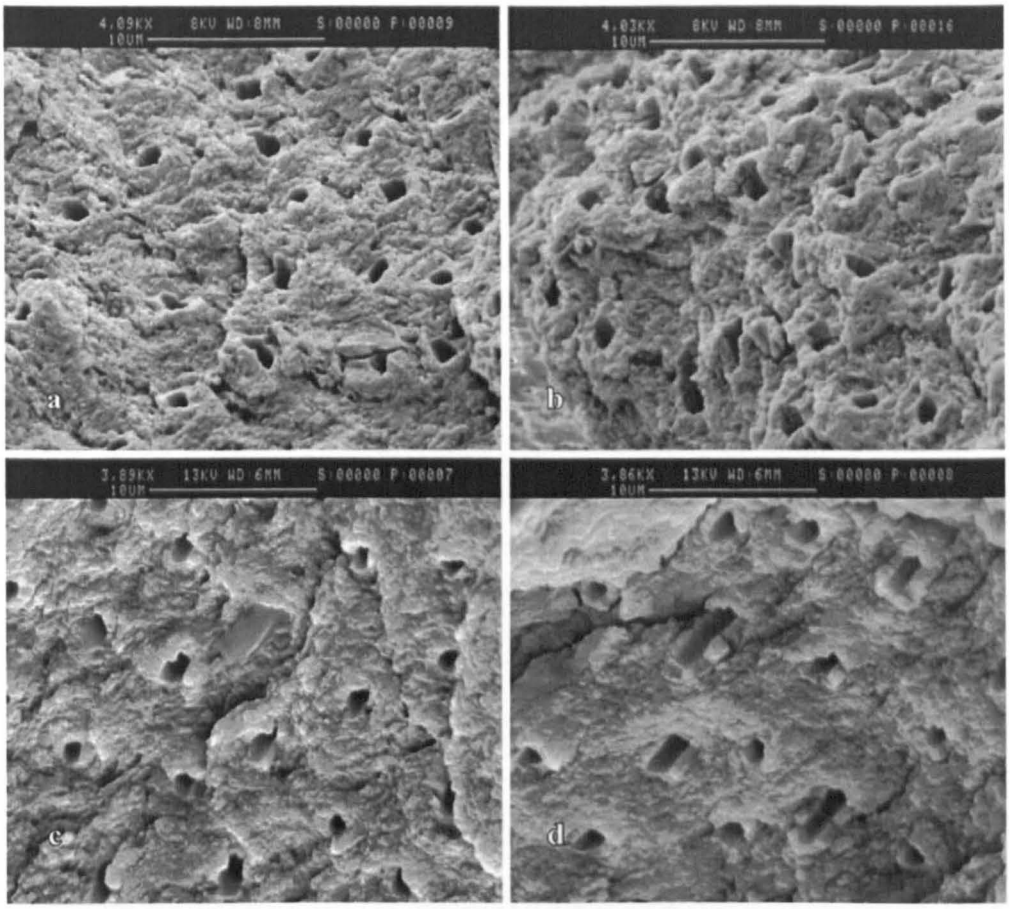


Fig. 5 SEM micrographs of irradiated dentine surface with different power settings: **a** 3 W, **b** 3.5 W, **c** 4 W, and **d** 4.5 W, showing opened dentinal tubules and the absence of a smear layer

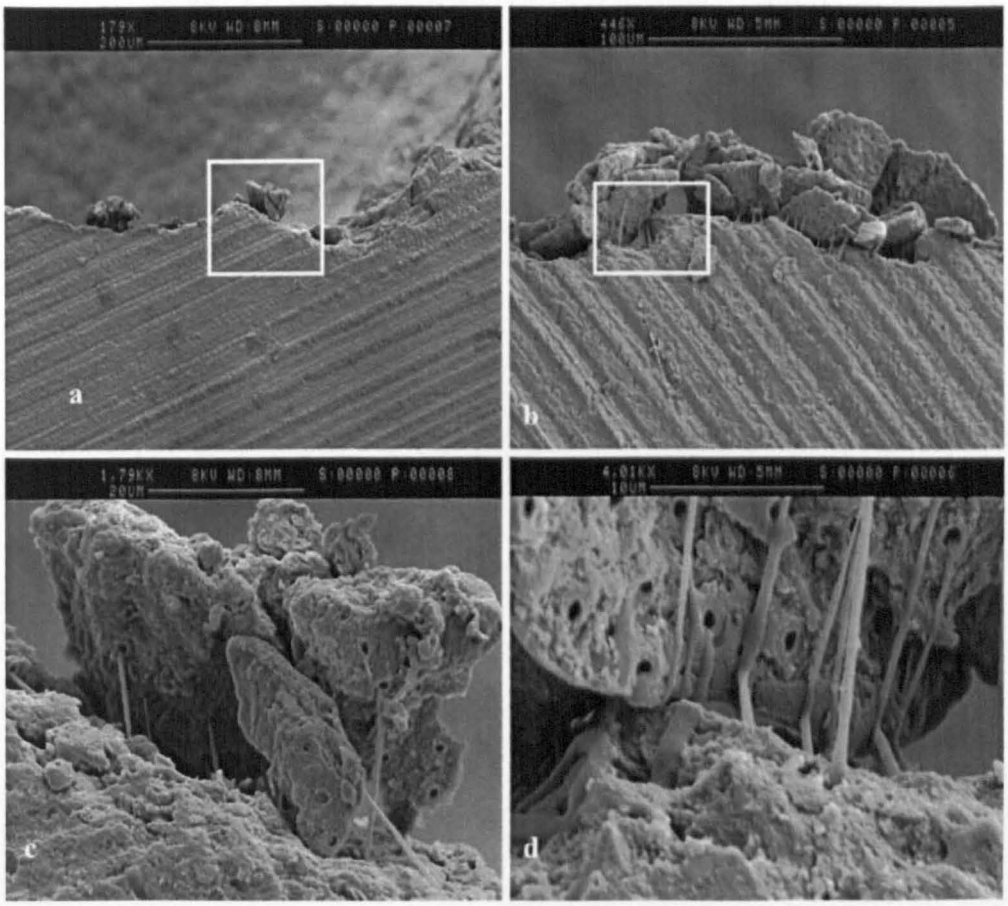


Fig. 6 SEM micrographs of the transversally sectioned dentine disk of the irradiated dentine surface using a power setting of 3 W. There was a dentine debris along the interface (**a** and **b**). At high magnification, fibre-like tags linked the debris to the underlying dentine **c** and **d**

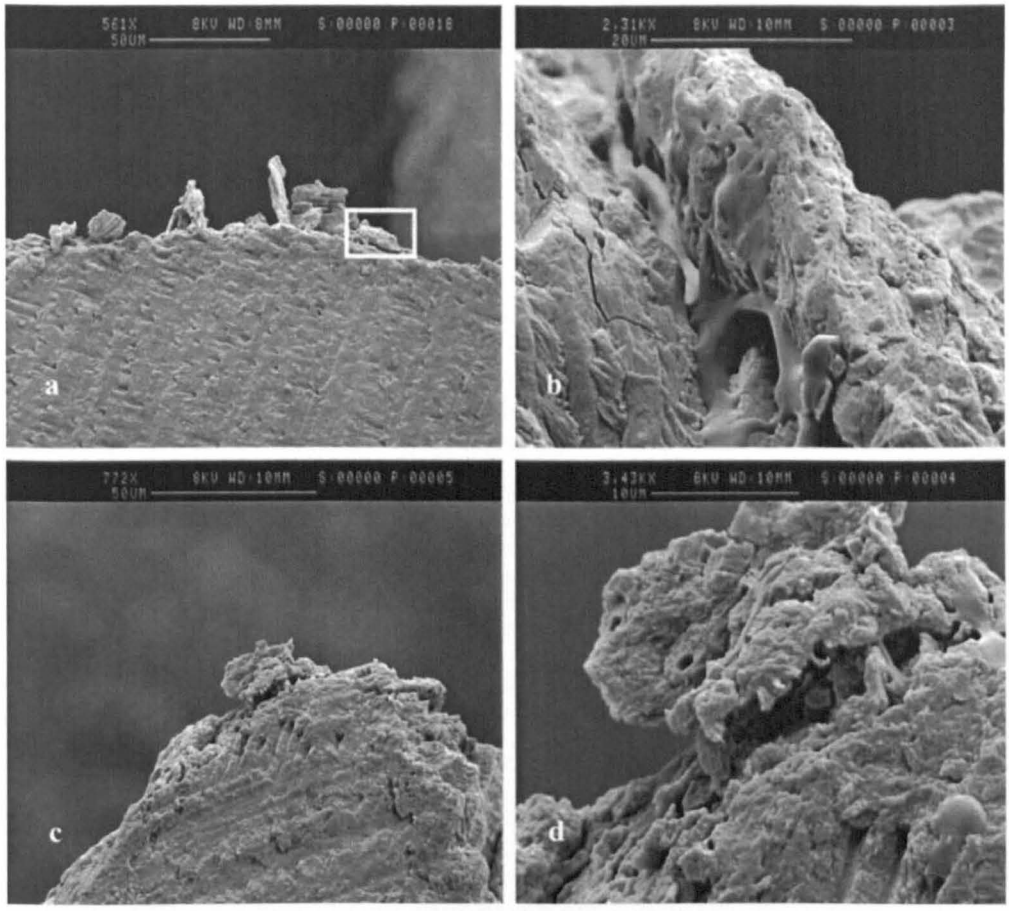


Fig. 7 SEM micrographs of transverse view of irradiated dentine using a power setting of 3.5 W: There were some dentine debris particles (**a**, **c**) along the interface. At high magnification, a molten-like substance (**b**) and malformation dentine substance (**d**) is seen

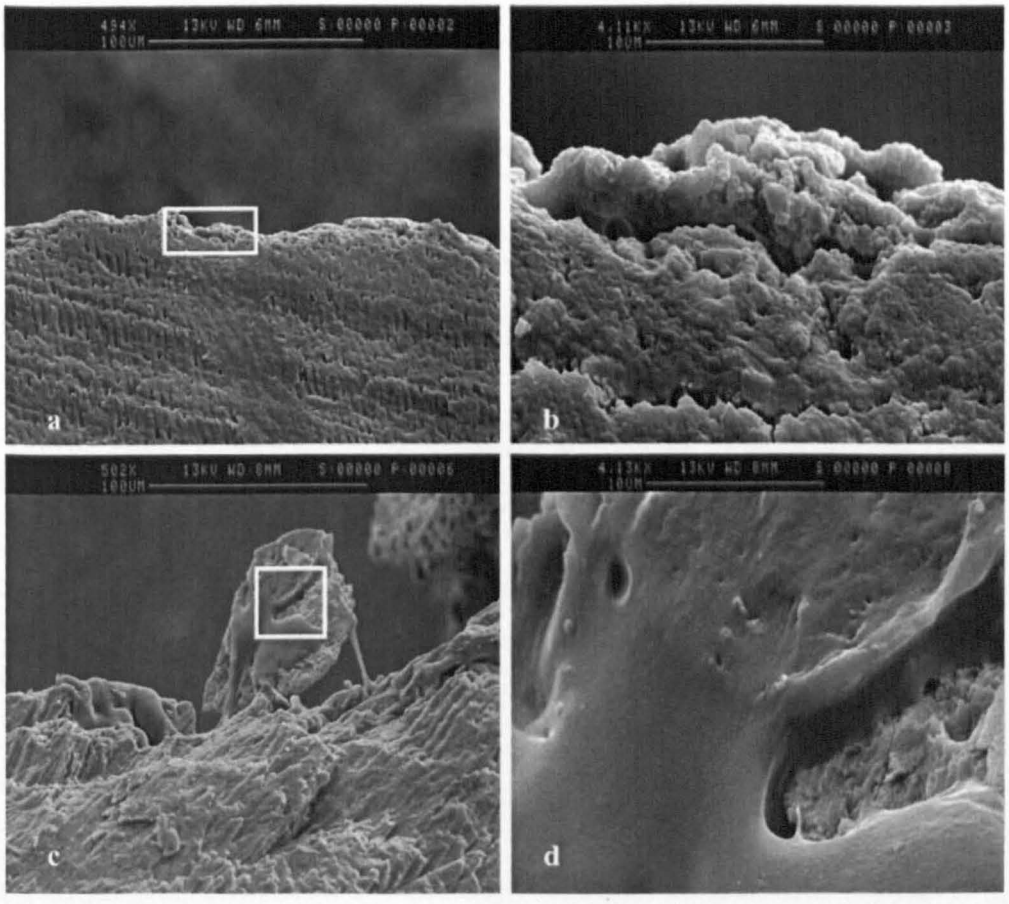


Fig. 8 SEM micrographs of a transverse view of the irradiated dentine surface using a power setting of 4 W (**a** and **b**): The dentine surface consisted of a crystalline-like substance. At a power setting of 4.5 W, there were some dentine debris particles on the surface (**c** and **d**)

Hossain et al. [23] showed that the Knoop hardness of the cavity prepared by the Er,Cr:YSGG laser was not significantly different from bur preparation. Due to the fusion of the dental hard tissues after CO_2 and Nd:YAG laser irradiation, the Knoop hardness increased [24, 25]. Other studies revealed that higher energy laser irradiation actually decreased the microhardness of dental hard tissue significantly due to the increase in temperature [26]. A previous report mentioned that all the lasers that produce some thermal effect could induce an alteration in physical and chemical composition [27]. It was found that when heating dental hard tissue to a temperature of more than 400°C, the mineral decomposes to form a new mineral phase that enhances the resistance to acid dissolution [4, 28]. Plotting the hardness against the elastic modulus after the laser application is likely to broaden the distribution both axes. It is similar to the trend line between the hardness and the elastic modulus of sound dentine and carious dentine [13]. This indicated that there is potentially a progressive deterioration of the hardness and the elastic modulus of the dentine after the laser irradiation. There is a correlation between the mechanical properties and the mineral content in tooth tissue [19]. The chemical analysis and thermal analysis of the laser-irradiated dentine should be studied further.

The ablation efficiency can be defined in terms of the amount of dental tissue within the given time [6], the volume removed per joule of energy incident on the tissue (mm^3/J) [4], and the volume ablated per second (mm^3/s) [28]. This study reported the ablation efficiency of dentine in terms of the volume ablated per second (mm^3/s). It is in agreed with the work by Rizoiu and DeShazer [29]. They showed a linear correlation between the volume ablated per second and the average power of a 2.8-μm erbium laser.

Both the Er,Cr:YSGG laser or the Er:YAG laser uses the water-mediated ablation [4]. The water plays an important role for dentine ablation. Meister et al. [5] found that, in dehydrated moist dentine, the quantity of ablate volume was significantly lower compared to the dentine containing water for the Er:YAG laser, whereas the Er,Cr:YSGG laser showed superficial melting and a deep carbonization zone when used without water spray. The exogenous water has a greater affect more than endogenous water on the dentine ablation of Er,Cr:YSGG laser.

An understanding of the mechanical and physical properties of prepared dentine after different clinical procedures may help to find the optimized conditions to enhance the adhesion of dental restoration. This study is an initial part aimed to present the mechanical properties of laser-treated dentine when using a different parameter of laser. Further studies will focus on the bonding of dental materials on irradiated dentine.

References

1. Lin S, Caputo AA, Eversole LR, Rizoiu I (1999) Topographical characteristics and shear bond strength of tooth surface cut with a laser-powered hydrokinetic system. J Prosthet Dent 82:451–455
2. Maiman TH (1960) Stimulated optical radiation in ruby. Nature 187:493–494
3. Ceballos L, Osorio R, Toledano M, Marshall GW (2001) Microleakage of composite restorations after acid or Er-YAG laser cavity treatment. Dent Mater 17:340–346
4. Fried D (2000) IR laser ablation of dental enamel. Proc SPIE 3910:136–138
5. Meister J, Franzen R, Forner K, Grebe H (2006) Influence of the water content in dental enamel and dentin on ablation with erbium YAG and erbium YSGG lasers. J Biomed Opt 11:0340301–0340307
6. Kim KS, Kim ME, Shin EJ (2005) Irradiation time and ablation rate of enamel in contact and non-contact irradiation with Er:YAG laser. Photomed Laser Surg 23:216–218
7. Serebo L, Segal T, Nordenberg D, Gorfil C, Bar-Lev M (1987) Examination of tooth pulp following laser beam irradiation. Lasers Surg Med 7:236–239
8. Malmstrom HS, McCormack SM, Fried D, Featherstone JD (2001) Effect of CO_2 laser on pulpal temperature and surface morphology: an in vitro study. J Dent 29:521–529
9. Aminzadeh A, Shahabi S, Walsh LJ (1999) Raman spectroscopic studies of CO_2 laser-irradiated human dental enamel. Spectrochim Acta Part A Mol Biomol Spectrosc 55:1303–1308
10. Corona SAM, Borsatto MC, Pecora JD, De SA Rocha RA, Ramos TS, Palma-Dibb RG (2003) Assessing microleakage of different class V restoration after Er:YAG laser and our preparation. J Oral Rehabil 30:1008–1014
11. Hibst R, Keller U (1989) Experimental studies of the application of the Er:YAG laser on dental hard substances: I. Measurement of the ablation rate. Laser Surg Med 9:338–344
12. Hossain M, Nakamura Y, Tamaki Y, Yamada Y, Murakami Y, Matsumoto K (2003) Atomic analysis and Knoop hardness measurement of the cavity floor prepared by Er,Cr:YSGG laser irradiation in vitro. J Oral Rehabil 30:515–521
13. Angker L, Swain MV, Kilpatrick N (2005) Characterising the micro-mechanical behavior of the carious dentine of primary teeth using nano-indentation. J Biomech 38:1535–1542
14. Fuentes V, Ceballos L, Osorio R, Toledano M, Carvalho RM, Pashley D (2004) Tensile strength and microhardness of treated human dentin. Dent Mater 20:522–529
15. Harashima T, Knoshita J, Kimura Y, Brugnera A Jr, Zanin F, Pecora JD, Matsumoto K (2005) Morphological comparative study on ablation of dental hard tissues at cavity preparation by Er:YAG and Er,Cr:YSGG lasers. Photomed Laser Surg 23:52–55
16. Mahoney E, Holt A, Swain M, Kilpatrick N (2000) The hardness and modulus of elasticity of primary molar teeth: an ultra-micro-indentation study. J Dent 20:589–594
17. Angker L, Swain MV, Kilpatrick N (2003) Micro-mechanical characterisation of the properties of primary tooth dentine. J Dent 31:261–267
18. Kameyama A, Oda Y, Kawada E, Takizawa M (2001) Resin bonding to Er:YAG laser-irradiated dentin: combined effects of pre-treatment with citric and glutaraldehyde. Eur J Oral Sci 109:354–360
19. Senawongse P, Otsuki M, Tagami J, Mjor I (2006) Age-related changes in hardness and modulus of elasticity of dentine. Arch Oral Biol 51:457–463
20. Bachmann L, Diebolder R, Hibst R, Zezell DM (2005) Changes in chemical composition and collagen structure of dentine tissue after erbium laser irradiation. Spectrochim Acta Part A Mol Biomol Spectrosc 61:2634–2639

21. Chng HK, Ramli HN, Yap AUJ, Lim CT (2005) Effect of hydrogen peroxide on intertubular dentine. J Dent 33:363–369
22. Fried D, Visuri SR, Featherstone JDB, Walsh JT, Seka W, Glena RE, McCormack SM, Wigdor HA (1996) Infrared radiometry of dental enamel during Er:YAG and Er:YSGG laser irradiation. J Biomed Opt 1:455–465
23. Hossain M, Nakamura Y, Yamada Y, Kimura Y, Matsumoto N, Matsumoto K (1999) Effects of Er, Cr:YSGG laser irradiation in human enamel and dentine: ablation and morphological studies. J Clin Laser Med Surg 17:155–159
24. Marquez F, Quintana E, Roca I, Salgado J (1993) Physical–mechanical effects of Nd:YAG laser of sound dentine and enamel. Biomaterials 14:313
25. Konishi N, Fried D, Staninec M, Featherstone JD (1999) Artificial caries removal and inhibition of artificial secondary caries by pulsed CO_2 laser irradiation. Am J Dent 12:213
26. Kuramoto M, Matson E, Turbino ML, Marques RA (2001) Microhardness of Nd:YAG laser irradiated enamel surface. Braz Dent J 12:31–33
27. Lee BS, Lin CP, Lin FH, Li UM, Lan WH (2003) Effect of Nd:YAG laser irradiation on hardness and elastic modulus of human dentin. J Clin Laser Med Surg 21:41–46
28. Featherstone JDB, Nelson DGA (1987) Laser effects on dental hard tissues. Adv Dent Res 1:21–26
29. Rizoiu IM, DeShazer LG (1994) New laser–matter interaction concept to enhance hard tissue cutting efficiency. Laser Interact V 2134:309–317

ALD
Academy of Laser Dentistry

Via Email

TO: Dr. Piyanart Ekworapaj
School of Dental Science, New Castle University
Framingham Place
Newcastle upon Tyne
NE3 4BW
Piyanart.ekworapoj@ucl.ac.uk

CC: Dr. Ana Triliouris, Chair Student Scholarship, anadds@aol.com
Dr. Peter Rechmann, 2008 Conference Chair rechmannp@dentistry.ucsf.edu

Mentor: Professor J F McCabe j.f.mccabe@ncl.ac.uk

FROM: Gail Siminovsky, CAE, Executive Director
Academy of Laser Dentistry
3300 University Drive, 704
Coral Springs, FL 33065
Telephone: 954-346-3776, Fax 954-757-2598
Email siminovsky@laserdentistry.org

RE: Acceptance of Dr. Eugene Seidner Student Scholarship

December 12, 2007

Dear Dr. Ekworapaj:

Congratulations! On behalf of ALD's Board of Directors and the University and Academia Relations Committee with the subcommittee on Student Scholarship it is my pleasure to invite you to present your scientific dental laser research at ALD 2008, the Academy of Laser Dentistry's 15th Annual Conference and Exhibition. The meeting will be held at the Loews Coronado Bay Resort in San Diego.

The Academy is delighted to accept your abstract for oral presentation as part of the General & Scientific Session as follows:
Title: **Optimal laser energy for adhesion of glass ionomer cement**
Date **Thursday, April 10, 2008, 10 minute lecture plus 5 minutes Q. & A.**
Time: **General Session 7: 30am – 1:00 pm, Specific time To Be Determined**

As one of three recipients of the Dr. Eugene Seidner Student Scholarship this year your presentation will be judged by members of the Student Scholarship and University and Academia Relations Committees. Scholarship recipients will be ranked first, second or third and a small cash gift associated with each place.

On Saturday evening April 12, 7:00-8:00 pm, your work will be acknowledged as we present you with a certificate of successful participation during the Academy's Awards Ceremony. Plus, we look forward to your attendance at the Standard Proficiency Course on Wednesday, April 9 the sessions of the annual conference April 10 – 12 as well as Saturday's Presidents Awards Ceremony Reception and Dinner Dance.

The Academy is pleased to provide round trip coach class air transportation for you, 5 nights complimentary hotel stay at the Loews Coronado Bay Resort beginning Tuesday April 8 departing Sunday April 13, complimentary conference registration including meals provided according to the registration schedule and a 2008 student membership. We will be in touch regarding your air travel arrangements.

The Scientific Sessions committee is responsible for the general and scientific program and will be in touch with further speaker information. Please visit the conference website for complete information at http://laserdentistry.org/ald2008.

Please confirm by return email your acceptance of this honorable invitation.

Congratulations again on achieving this successful review of your abstract. We all look forward to welcoming you in San Diego next April.

Feel free to contact me at any time.

Sincerely,

Gail S. Siminovsky, CAE
Executive Director

CC: Dr. Craig Gimbel, President
Dr. Peter Rechmann, President Elect 08, Conference Chair
Dr. Ana Triliouris, Student Scholarship Chair

3300 University Drive, Suite 704, Coral Springs, Fl 33065
Telephone (954) 346-3776 Fax (954) 757-2598 Email laserexec@laserdentistry.org
www.laserdentistry.org

Optimal laser energy for adhesion of glass ionomer cement

P. Ekworapoj, DDS, MSc (presenter), Dr. Sharanbir Sidhu, BDS, MSc, PhD, Prof. J.F. McCabe, BSc, PhD, DSc

Newcastle upon Tyne University- Newcastle upon Tyne, UK

Abstract

Introduction: Besides laser characteristic (wavelength specificity, energy density and pulse repetition rate), the parameter used for laser irradiation influence the chemical and morphology alternations of tooth tissue resulting different wettability and adaptability to restorative dental materials. The purpose of this study is to assess the optimal laser energy of Er,Cr:YSGG laser required to achieve the maximum bond strength of glass ionomer cement to dentin.

Materials and Methods: Eighty extracted human molar teeth with an exposed flat occlusal dentine surface were prepared by different laser power settings (3,3.5,4,4.5 W) of Er, Cr: YSGG laser [Biolase Inc., San Clemente, USA]. The power settings were varying with fixed repetition rate of 20 Hz and pulse duration of 140 μs. The working distance and irradiation time were 1 mm and 60 sec respectively. The teeth in each of these four different power setting groups were further subdivided into two equal groups for restoration with one of two glass ionomer cements: Fuji IX (GC) or Ketac Molar (3M ESPE). Half of the specimens restored with each material were treated with the relevant dentin conditioner. (Dentin Conditioner, GC; Ketac Conditioner, 3M ESPE) prior to bonding using gelatin capsules filled with the materials and perpendicular to the dentin surface. Shear bond strength was tested after 24 h storage in distilled water by Instron 5567 [Instron Corp, UK] at cross head speed of 1 mm min^{-1} until failure occurred. Subsequently, mode of failure was observed and the fracture surfaces were examined by using SEM [Stereoscan 240, Cambridge, UK].

Results: Shear bond strength (MPa)(standard deviations in parentheses (n=15); mean values with the same superscript were not significantly different (p>.05)

Power settings	Shear bond strength (MPa)			
	Ketac Molar		Fuji IX	
	No Conditioner	Conditioner	No Conditioner	Conditioner
3W	4.67 (2.29)	6.00 (2.64)	2.88 (1.63)	5.31 (2.56)
3.5W	5.50 (2.40)	6.59 (3.00)	3.03 (1.95)	4.63 (2.90)
4W	5.97 (2.78)	5.79 (3.19)	3.78 (1.70)	5.21 (2.47)
4.5W	5.76 (2.66)	6.05 (4.26)	3.15 (2.56)	5.97 (2.72)

From three ways ANOVA analysis showed that the power setting did not affected shear bond strength of glass ionomer cement (p>0.05). Four ways General Linear Model analysis revealed that glass ionomer, dentin treatment and dentin depth has influence on shear bond strength (p<0.05). Most of test specimens showed adhesive failure more than mixed failure. SEM observations showed the fragment of the cement plugged into dentinal tubule and cement's filler attached on the fracture specimens from 4 W and 4.5 W groups.

Discussion and Conclusion: There was an increase in shear bond strength of glass ionomer cement to non-conditioned dentin when increasing the power setting but not significantly different then decreasing at power setting of 4.5W. This possibly concluded

that the maximum shear bond strength of glass ionomer cement (both Ketac Molar and Fuji IX) to untreated dentin surface was obtained when using power setting at 4W. For conditioned dentin group, the maximum shear bond strength (MSBS) of Ketac Molar was obtained at low power setting (3.5W) while MSBS of Fuji IX was obtained at highest power setting (4.5 W). Thus, more consideration is needed with regard to selection the laser parameter for different clinical situation, in order to ensure optimum adhesion of restorative dental materials to tooth prepared by laser.

Disclosure: All researchers (Dr. P.Ekworapoj, Dr. S.K.Sidhu and Prof. J.F. McCabe) have no relationships with any dental laser manufacturer. There are no conflicts of interest with any of these devices used in the study

Microtensile bond strength of different types of Glass ionomer cements to Er,Cr:YSGG laser prepared dentin

***Ekworapoj P, Sidhu SK, McCabe JF**

Newcastle University, Newcastle upon Tyne, United Kingdom

Introduction:

The introduction of dental laser for cavity preparation has been existed for many years since 1960s. The Er,Cr:YSGG has used for cavity preparation as alternative method instead of using high speed hand piece. Many investigations focused on bond strength of composite and adhesive system to dentine prepared by these lasers system. There are a few reports about bond strength of glass ionomer cement to laser prepared dentin. The purpose of this study is to determine microtensile bond strength of three different glass ionomer cement to Er,Cr:YSGG laser prepared dentin.

Materials and Methods:

Two conventional type II Restorative glass ionomers were use: Fuji IX (GC Co., Japan and Ketac Molar (3M ESPE, Germany) as well as two conventional Type I Luting glass ionomers: Fuji I and Ketac Cem and a resin modified glass ionomer cement (Photac Fil).

The occlusal third of fifteen extracted human premolars was removed and the cut surfaces were prepared with Er,Cr:YSGG laser (Waterlase ™). The experimental specimens were divided into five groups according to the various kinds of glass ionomers and built up with glass ionomers.

After 24 hours storage in deionised water, all teeth were sectioned to produce specimens of the "slab" configuration for microtensile testing; there were 20 specimens per group. Microtensile bond strength was measured by Instron 5567 (Instron Corporation, UK) with crosshead speed of 1mm min^{-1}.The mean bond strength were evaluated by one-way ANOVA and Tukey's comparison. The mode of failure was observed and the fractured surfaces were examined by using SEM

Results: The mean microtensile bond strength (MPa)(standard deviation in parentheses) of various types of glass ionomer cement (n=20); mean value with the same superscript were not significant different ($p>0.05$).

Glass Ionomer cements	Mean microtensile bond strength (MPa)(SD)
Fuji I	1.36 (0.98) [a]
Ketac Cem	1.06 (0.87) [a]
Fuji IX	1.84 (1.53) [a,b]
Ketac Molar	3.12 (1.62) [b]
Photac Fil	6.15 (3.38) [c]

Microtensile bond strength of Photac Fil to laser prepared dentin was significantly different from Fuji IX and Ketac Molar. Most of the fractured specimens from Photac Fil revealed the highest percentage of cohesive and mixed failures.

Discussion:

Tensile bond strength of conventional glass ionomer cement to laser prepared dentin ranged from 1-3 MPa which was not different from that to conventional prepared dentin [1]. Similarly to conventional prepared dentin, tensile bond strength of RMGIC to laser prepared dentine was higher than that of conventional glass ionomer.

Conclusions:

Resin modified glass ionomer cement has a greater microtensile bond strength to laser prepared dentine than conventional glass ionomer cement.

References:

[1] Czarnecka B, Deregowska-Nosowicz P, Nicholson JW. Shear bond strengths of glass ionomer cements to sounds and to prepared carious dentine. J Mater Sci:Mater Med 2007;18:845-849.

Microtensile bond strength of glass ionomer cement with laser prepared dentine

P.Ekworapoj, S.K. Sidhu, J.F. McCabe

Objectives: This study compared microtensile bond strength of glass-ionomer to dentine prepared with Er,Cr:YSGG laser or bur.

Methods: Eight experimental groups were prepared from sixteen extracted human molar teeth with an exposed flat occlusal surface by using either Er, Cr: YSGG laser [Biolase Inc., San Clemente, USA] or bur cutting. In half of the bur and laser prepared groups, the relevant conditioner was applied on the dentine surface before building up with glass-ionomer cement [Fuji IX (GC, Japan) or Ketac Molar (3M ESPE, UK)]. After 24 hours storage in deionised water, all teeth were sectioned to produce specimens of the "slab" configuration for microtensile testing; there were 20 specimens per group. Microtensile bond strength was measured by a Dillon Quantrol™ microtensile machine [Weight Tronix Inc., Fairmont, MN, USA]. The mode of failure was observed and the fractured surfaces were examined by using SEM.

Results: Microtensile bond strength (MPa)(standard deviations in parentheses (n=20); mean values with the same superscript were not significantly different (p>.05)

Conditioner	*Fuji IX*		*Ketac Molar*	
	Bur	***Laser***	***Bur***	***Laser***
No conditioner	4.56 (2.19) [a]	5.99 (1.70) [a]	3.92 (1.98) [a]	5.61(1.55) [a]
Dentine conditioner	5.52 (1.68) [a]	8.62 (3.86) [b]	-	-
Ketac conditioner	-	-	5.52 (1.52) [a]	8.01(3.26) [b]

The use of conditioner was found to significantly affect bond strength for laser prepared samples ($p < .05$, ANOVA) but not bur prepared samples. There was no significant difference between the microtensile bond strengths of the two materials. All specimens prepared with a bur but no conditioner showed adhesive failure while the other groups showed either cohesive or adhesive failures.

Conclusions: Laser preparation alone would not improve the bond strength between glass-ionomer cement and dentine.

Er,Cr:YSGG laser-dentine interaction: micromechanical and microscopic characterization

P.Ekworapoj, S.K. Sidhu, J.F.McCabe

School of Dental Science, Framlington Place, Newcastle Upon Tyne

Objectives: This study determined the micro-mechanical properties and microstructure of dentine irradiated with a Er,Cr:YSGG laser.

Methods: Twenty extracted human premolar teeth with an exposed flat occlusal surface were cut transversally to prepare twenty dentine disks with 2 mm thickness. After mounting on a plastic support, all specimens were wet polished by using 500 grit SiC paper. They were divided into four experimental groups according to the power setting of Er,Cr:YSGG laser applied to the dentine surface; namely 3W, 3.5W, 4W and 4.5W. The focus length between the laser tip and dentine surface was 1 mm and the exposure time was 1 min. The depth of dentine removed by laser application was investigated by using a 3D laser scanning surface profiler. The microhardness values were measured by using a Martens microhardness tester before and after laser irradiation. Representative specimens were examined microscopically by SEM.

Results: The mean ablation depth (SD) of each experimental group was 0.15 (0.02), 0.28(0.10), 0.33(0.07) and 0.42(0.14) respectively. The mean value of irradiated dentine hardness (SD) was 42.89(16.39), 30.27(11.94), 40.41(19.52), 41.27(9.15) and which was significantly lower than the baseline dentine hardness which ranged from 60.85(8.39) to 66.18 (5.12). Linear regression analysis showed a significant relationship between increasing power setting of the laser and the ablation depth of the irradiated dentine (R^2 = 0.61, $p < 0.05$). There was no relationship between power setting and hardness. The dentine hardness decreased significantly when treated with Er,Cr:YSGG laser (Pair t-test, $p<.05$). SEM examinations revealed the differences in dentinal tubule morphology with different power settings.

Conclusions: Increasing the power setting resulted in increasing ablation depth. Laser irradiation may cause a reduction in the hardness of the dentine and changes of dentine structure.

UNIVERSITY OF
NEWCASTLE UPON TYNE

Laser in Dentistry

P.Ekworapoj, S.K. Sidhu, J.F. McCabe
For promoting your research to public, 3rd of May

Fear of the dentist's drill is one factor which makes a visit to the dentist something to be approached with anxiety for many patients. The fear may be associated with an anticipation of pain or sensitivity or may be associated with the noise and vibration of the procedure. For some patients it is the fear of the local anaesthetic which causes their anxiety. Nowadays an alternative method of tooth cutting and preparation is available using powerful laser technology. One laser system employs a jet of laser energised water which has sufficient energy to cut dental hard tissue without overheating the adjacent tissues. The instrument allows pain-free cutting without the need for a local anaesthetic and therefore has the potential to be a major innovation for dentistry.

The Waterlase (Biolase Inc) is currently not widely used in general dental practice for various reasons. Firstly, the equipment is relatively expensive and many practitioners are likely to wait until prices fall as the technology becomes more widely accepted. Secondly, many practitioners remain to be convinced of the advantages of the laser cutting instruments. One area of concern is the nature of the surface of the dental hard tissues after laser cutting. Many dental adhesive systems, both salt based and resin based have been developed to interact with bur-cut dentine and enamel and there is a need to confirm that such materials will interact in a similar way with laser-cut tissues. The purpose of this research was to study the structure and properties of laser cut tooth surfaces and the way in which they interact with materials to form an effectively bonded and sealed interface.

Paper #45322

The glass-ionomer interface with laser and bur prepared dentine

P. EKWORAPOJ, S. SIDHU, and J.F. MCCABE, School of Dental Sciences, University of Newcastle upon Tyne, United Kingdom

Objectives: The glass-ionomer interface with laser prepared enamel and dentine has not been assessed. This study compared the glass-ionomer interface between laser and bur prepared dentine.

Methods: Class V (wedge-shaped) cavities were prepared in human sound molars by using either Cr, Er: YGSS laser (Waterlase YSGG, Biolase Technology, Inc.,San Clemente, USA) or diamond bur. All cavities were restored with Fuji IX glass-ionomer (GC, Japan). The teeth were stored moist at 37 °C for 24 h before removing the root tips for introduction of rhodamine B dye into the pulp chamber. All teeth were then sectioned longitudinally with a high-speed diamond saw. One half of each tooth was examined using a confocal laser-scanning microscope (Leica TCS SP2UV, Heidelberg, Germany). The glass-ionomer interface with enamel and dentine was assessed for adaptation and presence of gaps.

Results: All specimens showed good adaptation at the enamel/glass-ionomer interface. At the material/dentine interface, lased dentine showed gap formation at the innermost (pulpal wall) part of the cavity, while the bur prepared specimens displayed gap formation closer to the dentine-enamel junction. The gaps with lased dentine occurred at the pulpal wall, were continuous and extended over a longer portion of the interface. Gap formation in the bur prepared cavities occurred mostly at the occlusal wall and were over a shorter distance. The average maximum gap width between the glass-ionomer and lased dentine was approximately 18.66 (SD: 4.38) ∎m and that of bur prepared dentine was approximately 8.83 (SD: 0.03) ∎m.

Conclusions: Cavities prepared with the Cr, Er: YGSS laser provided good cavity adaptation with glass-ionomer at the enamel interface. However, the glass-ionomer/lased dentine interface showed poorer adaptation compared to dentine prepared by a diamond bur.

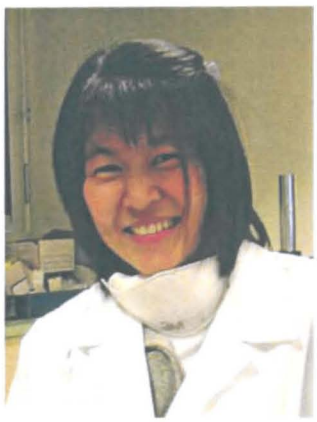
Abstract ID#: 45322
Password: 767498
Submitter's or presenter's Email: piyanart.ekworapoj@ncl.ac.uk
Preferred Presentation Mode: Poster
Audio/Visual Selection: Not Applicable
The presenter is a Post-graduate student whose faculty advisor's email address is s.k.sidhu@ncl.ac.uk.

Keywords: Dental materials, Glass ionomers, lasers, interfaces

First Author

Presenting
Piyanart Ekworapoj, MSc
Dental Materials
School of Dental Sciences, University of Newcastle upon Tyne
Framlington place, Newcastle upon Tyne, NE2 4BW,UK
Newcastle upon Tyne, NE2 4BW
United Kingdom

CURRICULUM VITAE



DR. PIYANART EKWORAPOJ

School of Dental Science,
Dental Materials Laboratory
Newcastle University,
Framlington Place, Newcastle upon Tyne,
NE2 4BW
Email: piyanart.ekworapoj@ncl.ac.uk

Education

Present: PhD student (writing up stage)
Newcastle University, Newcastle upon Tyne, UK

2002: MSc (Polymer Science- Dental Nanocomposites),
Chulalongkorn University, Bangkok, Thailand

1997: Doctor of Dental Surgery,
Mahidol University, Bangkok, Thailand

Profession Experience

1997- 2003 Lecturer (General Dentistry Department)

- Teach undergraduate students
- Class preparation such as handout, grading and aiding student
- Supervise and guide undergraduate student's thesis work (6th year dental student)
- Design and developed undergraduate course in dental materials for 3rd and 4th year dental student
- Served as student advisor
- Developed and present seminar (dental materials)
- Supervise and manage undergraduate dental laboratory
- Design and implemented testing model in dental laboratory
- Conduct the own research project
- Participate in all phases of the research process, including research design, data collection, and data analysis both in student's thesis project and own project.
- Provide both inpatient and outpatient care
- Handled for emergency dental patients

Clinical Expertise & Skills

- Restorative Dentistry (Operative, Endodontic, Periodontic, and Prosthodontic)
- Lasers Dentistry (Hard tissue application)
- Composite and Glass ionomer restoration

Research Skills& Expertise

- Lasers in dentistry
- Lasers tissue interaction
- Safety issue for using laser in dentistry
- Microscopy (CLSM, SEM)
- Bond strength testing (Microtensile, Shear bond strength testing)
- Nanocomposite synthesis for dental composite
- Chemical Analysis with XRD, FTIR, DTS instrument and Rheometer.
- Microscopic survey by using SEM, TEM and CLSM microscopy.
- Mechanical Testing for material by using Universal Testing Machine (Instron) and Vicker hardness testing machine
- Utilized SPSS and MINITAB statistical programme

Computer& Software Skills

- Microsoft word, excel
- Photoshop, Adobe, PageMaker
- Power point presentation
- E publication searching (Medline, Pubmed)