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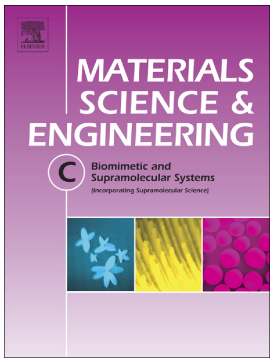

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Femtosecond laser ablation of dentin and enamel for fast and more precise dental cavity preparation

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Abstract

Purpose. The purpose of the present work was to achieve fast and more precise ablation in dentin and enamel by using a commercial femtosecond laser system with high repetition rate, whilst avoiding any collateral irreversible damages in the hard tissue and pulp area.

Methods. We used fluence of the incident laser pulses which was marginally higher than the ablation threshold for dentin and enamel. The study was based on the hypothesis that femtosecond laser operating with a repetition rate in the range of 100 - 500 kHz can

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controllably ablate dental tissue obtaining sufficiently high removal rate whilst avoiding any collateral irreversible damages in the hard tissue and pulp area.

Results. The ablation yielded the formation of 1 mm^3 craters with well-defined precise vertical cavity sides and edges. Advantageous high porosity and numerous interconnected pores were introduced in the ablated zones. Thermal load and hence collateral thermo-mechanical damages were avoided and the crystalline structure of the tooth constituent hydroxyapatite was preserved.

Conclusion. The ultrafast femtosecond laser used in our work hold the promise of a significant drilling ability without collateral thermomechanical effects. It achieves high processing efficiency, overcomes disadvantages of other laser systems reported and can be used to develop an instrument for cavity preparation based on fast and precise ablation. Our further aim is to exceed the speed of traditional drilling instruments and thus to reduce the treatment time which in turn will bring comfort to the patient.

Keywords: femtosecond laser; laser ablation; dentin, enamel; dental cavity preparation.

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1. Introduction

Lasers have been successfully used since the mid-1960s for eye surgery through the precise photocoagulation of the retina [1,2]. Thus, ophthalmologists are the pioneers in developing laser-based applications. Since then, lasers are used in many industrial and scientific applications and continue finding new and innovative areas for broader take up of this technology.

According to the World Health Organization [3], 60–90% of school children and nearly 100% of adults worldwide have dental caries leading to pain and discomfort. Most tooth decay occurs in the enamel (outer surface) and the dentin, which is the region between the enamel and inner region of the tooth containing the nerve endings (pulp) [4]. Mechanical drills are mainly used to remove the dental decay and prepare a cavity in the tooth to receive a filling that replaces the lost structure. These drilling operations are not sufficiently precise [5] and as a consequence of this large amounts of healthy enamel and dentin can be lost while removing the dental decay; in addition, it is an invasive method that causes patient discomfort [6]. The discomfort associated with mechanical drilling can be a psychological barrier for patients to overcome in seeking proper dental care. Due to the vibrations of the drills, it is necessary to use local anesthetic for the majority of dental procedures [6]. A continuous water spray is used in conjunction with the drills to balance the temperature rise due to the friction between the drill and the tooth, and also to wash away the remaining tooth debris. Therefore, the idea of substituting a drill with laser light has several advantages. There is an increased patient acceptance of this technology due to the lack of contact, vibration and noise. In addition, there is little if no pain and discomfort. Furthermore, the dentist may practice a minimal dentistry technique by only needing to remove diseased tissue while keeping sounder tooth tissue untouched.

Lasers are safer compared to rotating instruments due to a very low risk of accidental

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damage to the pulp and soft tissues. Calculus removal can be done with a degree of selectivity, compared to conventional instrumentation. However, those lasers used in dentistry so far have more unwanted than desired effects on the dental tissue [7,8]. Typically, treatment by conventional pulsed laser sources (pulse length of ns to μs) lead to poor surface preparation and relatively high thermal load. This is evident from cracking, melting, charring, fissuring or crazing of tooth surfaces and leads to inefficient and uncontrolled hard tissue removal [7]. The poor absorption of laser radiation by tooth structures requires high powers leading to excessive heat diffusion from the interaction area, with potential for intrapulpal damage [7]. Previous research on the ablation of human hard tissue has been performed using a variety of long-pulsed lasers over a range of wavelengths that can generate a substantial amount of heat in human teeth especially if the pulse duration is too long [8].

Recently, high average power, high repetition rate, subpicosecond lasers are attractive for developing surgical applications in dentistry due to their precise and highly effective ablation capabilities and minimal thermal and shock wave collateral damages [7-11]. The negative laser effects causing pain to the patient, such as shock wave, vibration and rise in temperature in bulk dental tissue surrounding the treated site are eliminated. This is due to the decrease of the laser pulse durations down to the subpicosecond time range (ps to fs) and a consequent increase of the laser peak power. The ablation mechanism is hence electrostatic and thus the laser light is absorbed in a small volume of material so electrons and then ions are ejected rapidly, reducing the heat that is conducted to the surrounding tissue [9]. The change in ablation mechanism from thermally induced in the conventional long pulse lasers to electrostatic, results in distinct changes in tooth morphology, substantially reduced collateral damage to the tissue, and a decrease in the energy fluence required for significant material removal [11-13]. For laser pulse lengths above 1 ps, ablation is less efficient and surface melting is observed, whereas ablation efficiency can be maximized with pulse durations of 130 fs to 1 ps while the surface shows less evidences of melting [13]. Main applications of

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subpicosecond lasers for hard tissue ablation include mid-ear bone ablation [11], implantation of prosthetic joints [14], dental ablation such as the removal of hard tissue and caries for cavity preparation [8,15-17].

Several studies with subpicosecond pulsed laser systems have been published investigating various process variables in the search for well-suited parameters for dental applications: alternative lasing mediums, wavelengths, pulse durations, repetition rates, cooling supply, etc. [7,18-21]. There was agreement on the benefits of subpicosecond pulsed laser systems such as their ability to ablate various materials (dentin, enamel, composite fillings, metals) or precisely shape tooth cavities and all these with small or no collateral damages (cracks, melting, discoloration, etc.). These studies have used either too low (1 kHz) or too high (500 kHz) laser frequency. For example Fahey and colleagues applied 1.3 ps fiber laser (λ = 1552 nm) with 50 kHz and revealed no collateral damages of the ablated dentin or enamel but thermal damage occurred at 500 kHz [20]. Other study suggested that a repetition rate of about 100 kHz will be the most beneficial for the ablation of hard dental tissue [21]: a fiber laser operated in the femtosecond (fs) regime (500 fs, λ = 1045 nm) at 100 kHz introduced no thermal damages in the dentin and enamel except slight discoloration of the enamel. The authors also compared fs with ps systems working at different wavelengths and higher repetition rate (2 MHz), with or without cooling, and found that the fs laser interaction with the dental tissue was the most precise. On the other side, Rode et al. successfully ablated enamel with ~ 100 fs lasers ($\lambda \sim$ 800 nm) at 1 kHz with no apparent cracking or heat effects, while using a 60 ps Q-switched Nd:YAG laser (λ = 1064 nm) led to the discoloration of the area surrounding the ablated square, melted enamel droplets at the edges and melted floor of the ablated area [7].

In this work, we present experimental data on laser ablation of dentin and enamel using a state-of-the-art, commercially available subpicosecond laser system generating pulses with duration of 310 fs and wavelength λ = 1030 nm, allowing efficient ultrafast controllable

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processing without undesired collateral thermomechanical damages.

2. Materials and Methods

Extracted caries-free human molars from the Birmingham School of Dentistry Tooth Bank (work conducted under ethical approval - REC Ref: 14/EM/1128) were sectioned with a diamond saw into 2 mm slices perpendicularly to their long axis, providing well defined flat areas of enamel and dentin. The slices were polished with a SiC #1200 grinding paper with water irrigation, and subsequently with a diamond suspension (3 µm) to obtain mirror finishing. Ultrasound sonication in distilled water for 15 min was performed to remove any debris. The slices were fixed onto microscope slides with epoxy resin to ensure that the irradiated upper surface was perpendicular to the laser beam. Then the tooth slice with its holder was positioned on an XYZ computer-controlled table (Aerotech, USA) equipped with an optical scan head for delivering the laser beam with a maximum linear velocity of 2000 mm/s (figure 1). The tooth slices were kept in physiological solution before and after experimentation and the remaining moisture before laser irradiation was removed using pressurized air.

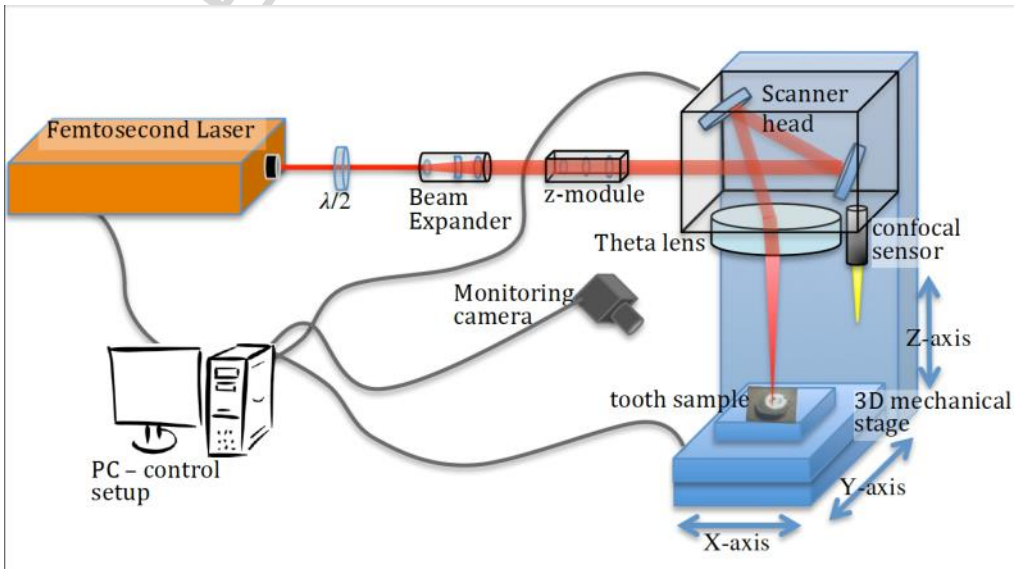

Figure 1. Scheme of the FELS used for the ablation of the tooth slices.

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The study was based on the hypothesis that fs laser operating with a repetition rate in the range of 100 - 500 kHz can controllably ablate dental tissue obtaining sufficiently high removal rate whilst avoiding any collateral irreversible damages in the hard tissue and pulp area. We used mode-locked regenerative amplified Yb:KGW (ytterbium-doped potassium gadolinium tungstate) pulsed laser (Satsuma laser from Amplitude Systems, France) operating at 1030 nm wavelength and having pulse duration of 310 fs. The laser source can deliver a maximum average power of 5 W at pulse repetition rates up to 500 kHz. The laser beam having pulse energy up to 10 µJ was focused on a tooth slice using a 100 mm F-Theta lens to produce a focal spot diameter of 20 µm. Using the scan head coupled with the laser, areas of 1 mm^2 were ablated on the dentin and enamel surface line by line following a hatching design with no overlapping of the lines to produce a square recess. After each scan of the 1 mm^2 area the table was repositioned in Z direction with a step defined by the ablation rate of the dentin and enamel materials and this processing step was repeated until a depth of 1 mm was obtained. The scanning speed was set to 500 mm/sec and repetition rate of 100 kHz was used. A single laser shot per site was applied and overlapping of two consecutive pulses on the tooth surface was avoided. No cooling was used during the irradiation.

The laser parameters for effective ablation of the enamel and dentin were identified by varying pulse energy, pulse repetition rate and scanning speed [22]. Satisfactory ablation rates with minimal thermal load and no signs of any collateral damage were considered as effective process settings. The ablation threshold and ablation rate for the single-shot regime in the case of relatively low laser fluence were determined: the ablation threshold was ~ 2.0 J/cm^2 for enamel and ~1.6 J/cm^2 for dentin, respectively, in agreement with other studies [8]. The ablation rate was ~ 600 µm/pulse for enamel and ~ 680 µm/pulse for dentin. Simultaneous measurement of the intrapulpal temperature rise in the tooth was conducted during ablation to avoid irreversible damage of the pulp. A simple set-up was built to measure the temperature on the pulp surface, opposite to the ablated one. The set-up aimed to simulate a condition

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arising when deep dental tissue has to be ablated, and the temperature increase in the pulp adjacent to it is of importance. Teeth used for pulp cavity temperature measurements were cut longitudinally in half with the pulp removed. A thermocouple (Omega, USA) attached to a digital thermometer (measurement step of 0.1°C) was placed in the pulp chamber close to the crown and secured using superglue. The chamber was then sealed using epoxy resin. The laser beam was focused on the crown of the as-prepared tooth half. Temperature recording was initiated some seconds before triggering the laser irradiation and lasted until the laser was stopped. Temperature measurements were taken every 10 ms.

Ablated areas in dentin and enamel were imaged with an optical microscope (OM; Alicona G4 InfiniteFocus, Austria, Measure Suite software) and scanning electron microscope (SEM; Philips XL30 FEG ESEM, Germany, 15 kV, platinum sputtering) coupled with energy dispersive X-ray spectroscope (EDX; Oxford Instruments INCA system). Hard tissue craters with a depth of 200 µm were prepared for the SEM imaging. Structural characterization was performed with a Fourier transform infrared microscope (FTIR; Nicolet iN10 MX, Thermo scientific, ATR mode, spectral range 400-4000 cm^{-1}, resolution 8 cm^{-1}).

Table 1. Approximate composition of enamel and dentin in teeth (volume % of total tissue).

Component	Enamel	Dentin
CO_3-HA	85	47
Water	12	20
Protein and lipid	3	33

3. Results and discussion

Scanning with the laser beam over the dental material instead of hitting in only one point reduces to minimum the laser irradiation of the healthy tissue. If the laser impacts onto the

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dental tissue only in one position, which is equivalent to deep drilling, an excessive heat accumulation occurs. Using the scan head and the motorized XYZ table, the ablation of the 1 mm^3 area in the enamel and dentin zones took ~350 and ~300 sec, respectively, which is equivalent to removal rates of 2.85 mm^3/s and 3.33 mm^3/s. This difference is due to the different ablation rates of the two materials, which comes from their different composition (table 1). The tooth main component is the mineral carbonated hydroxyapatite (CO_3-HA; chemical formula $Ca_{10}(PO_4)_6(OH)_2$). It also contains water and organic components such as proteins and lipids [23,24]. Enamel is built mainly of highly crystalline CO_3-HA and has less water and organic, which makes it much harder than the dentin. On the other side, dentin has more water and organics as constituents, and only 47 vol% CO_3-HA, which is the reason to be more fragile. Laser-tissue interaction depends on the effect of the laser wavelength on the different tissue treated. The interaction of laser light with healthy, demineralized and carious tissues, in primary or permanent teeth will be different due to the different water and organic content, and hence different crystallinity. Carious tissues are demineralized and richer in water in comparison with healthy and/or non-vital teeth. Thus, more energy is needed for enamel and less energy for dentin or carious tissue, depending on laser absorption of different water and organic content in the tooth. Initially all ablated slices were inspected with an OM and no cracks were present in either enamel or dentin zones. A representative image of a mirror polished tooth slice prepared for laser processing is shown in figure 2 where ablated dentin and enamel areas of 1 mm^2 are also included as smaller images. The processed zones in both materials were homogeneously ablated and collateral damage was not present around the craters. This observation was supported with higher resolution SEM images (figure 3). The high magnification images before the laser processing revealed the smoothness of the enamel surface (figure 3 a) and the characteristic tubules distributed throughout the dentin (figure 3 b). The ablated areas had well-defined precise vertical cavity sides and edges typical for laser-tissue interaction of ultrashort pulses (figures 3 c, d). No formation of deep pockets

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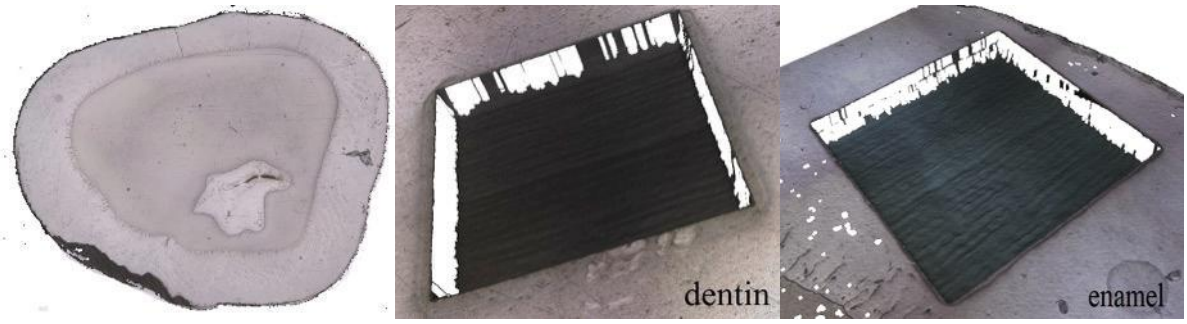

Figure 2. Optical microscopy imaging revealed no cracks or collateral damages after the ablation of dentin and enamel. Left: mirror-polished tooth slice prepared for ablation. Right: ablated enamel and dentin zones.

at the crater edges, as when scanning a meanders design, which arise due to the inertia of the scanning mirrors at the turning points was observed [25]. Detailed images of the edge between the intact and ablated enamel and dentin (figures 3 e, f respectively) also showed no discoloration, melting, carbonization or cracks as a result of the laser interaction with the dental tissue. Figures 3 g, h revealed morphological changes in the ablated enamel and dentin. In contrast to the smooth intact materials (figures 3 a, b) the laser interaction yielded high porosity of enamel and dentin and introduced numerous interconnected pores. The porosity was estimated in percentages using a threshold method of an 8-bit image in ImageJ software for image analysis, which shows the dentin or enamel as white pixels and the pores as black pixels [16,17]. The threshold method showed 56.3 and 55.7% porosity of enamel and dentin, respectively. The increase of tooth surface roughness critically affects the bonding of dental fillings to the tooth cavity [26]. Thus, dental tissue with higher roughness may be advantageous with its bigger surface area and hence improved adhesion strength of the filling, after the clinician has prepared the cavity with the fs laser instrument. The tubules in the intact dentin were not seen after the laser ablation, implying that they were most likely occluded with ablated tissue. Sealing of the dentinal tubules is advantageous for the prevention and treatment of early-stage carious lesions and for making the tissue more

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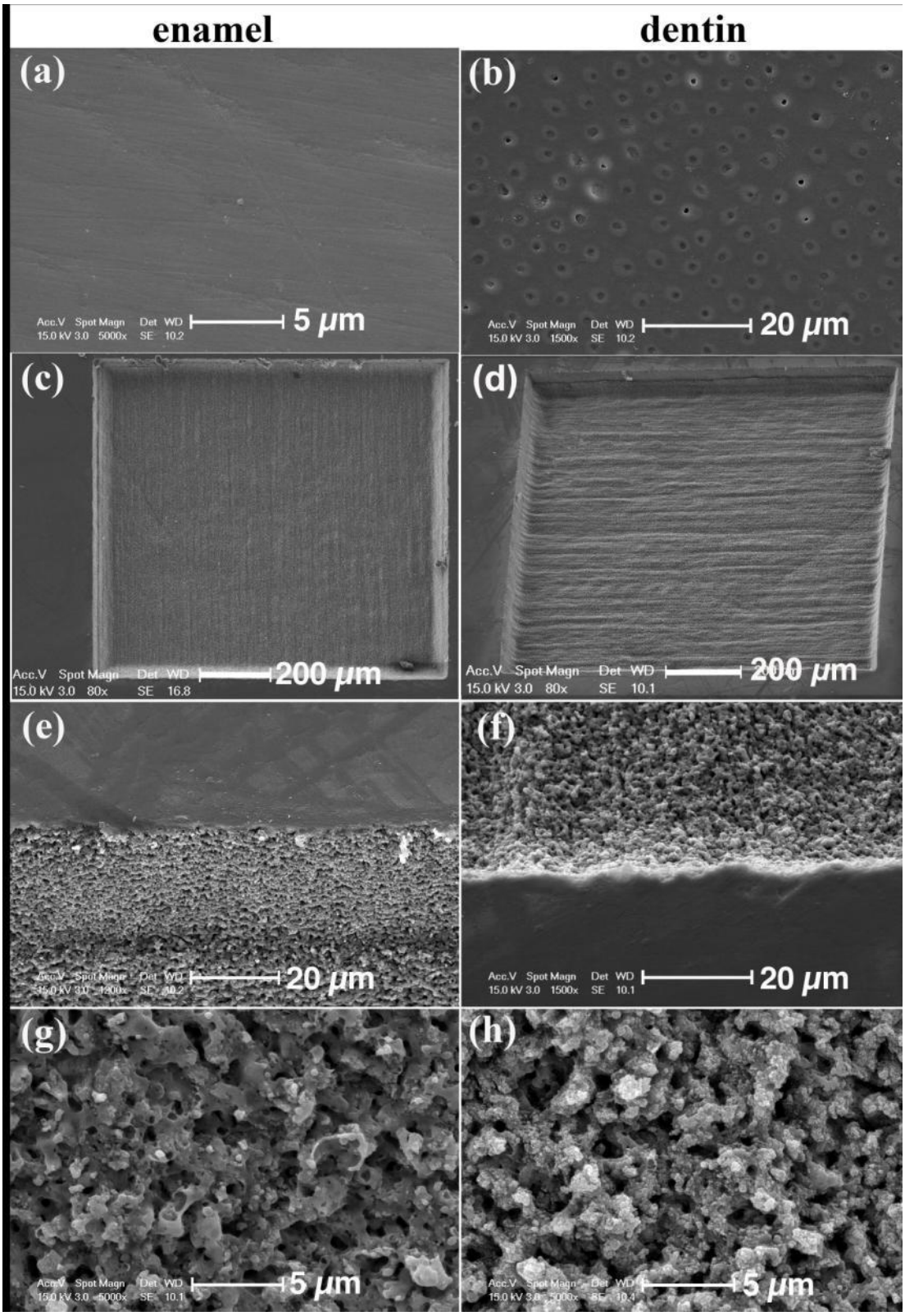

Figure 3. SEM imaging confirmed the absence of collateral damages after the ablation: (a, b) mirror polished enamel and dentin before laser ablation; (c, d) ablated enamel and dentin craters; (e, f) the crater edge between intact and ablated enamel and dentin; (g, h) morphological changes in the ablated enamel and dentin: advantageous increase of surface roughness was obtained for both materials.

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resistant to development of new caries [27].

To use the laser as a tool in dentistry, it is compulsory to minimize the amount of heat diffusion to the surrounding tooth. The human tooth is extremely sensitive to temperature variation: a patient can sense local temperature change at the pulp of $\pm 4^0$C [28]. To protect the tissue in the pulp from irreversible damage, the temperature rise cannot exceed 10^0C [8,29]. Therefore, in this experiment we performed simultaneous measurement of the temperature change in close vicinity of the area at which ablation occurred using a thermocouple attached both to the tooth cavity and a digital thermometer. The temperature increased by only 5^0C and remained stable during the ablation process. No temperature jumps were observed at the start or after the laser irradiation. Dental enamel and dentin can withstand higher temperature levels as long as thermal or mechanical damage (melting or cracking) is not induced and it is evident that such levels were not reached in our experiment. The ablation occurred during a period shorter than the time required for the heat and shock waves to affect the material surrounding the laser-matter interaction area. Therefore, there was no collateral damage due to any heating or mechanical effects on the tooth slices. Thus, the calcified tissue in the close vicinity to the interaction area was not affected.

Table 2. Elemental analysis of intact and ablated enamel and dentin obtained by EDX spectroscopy.

Element (at%)	Enamel	Enamel ablated	Dentin	Dentin ablated
C	13.9	11.4	25.9	16.2
O	53.3	58.1	49.9	55.7
Ca	18.5	17.7	13.7	15.7
P	12.9	11.8	9.6	11.1
Ca:P ratio	1.43	1.50	1.43	1.41

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The chemical analysis of the enamel and dentin before and after the laser ablation carried out with the EDX spectrometer is shown in table 2. There was a decrease of the carbon and a corresponding increase of the oxygen concentrations after the laser processing, which was more significant for the ablated dentin ($p < 0.05$). The calcium and phosphorus concentration did not vary significantly ($p > 0.05$) and the Ca:P ratio was stable before and after the laser-matter interaction for both materials. The decrease of the carbon content was attributed to the cleaning effect of the laser ablation, which removed the surface contamination.

FTIR spectroscopy data of intact dentin and enamel showed characteristic peaks of CO_3-HA dominating the spectral region of 750-2000 cm^{-1} (spectra 1 and 2 in figure 4). The most intensive peaks at 997 and 1066, and a shoulder at 960 cm^{-1} were due to ν_1 and ν_3 P-O stretching vibrations in phosphate (PO_4^{3-}) group of HA. C-O vibrations in the CO_3^{2-} group were presented by a shoulder at 870 and a double peak at 1410/1450 cm^{-1} related to the ν_2 C-O bending and ν_3 C-O stretching modes, respectively. Despite the low abundance of proteins in enamel, they play essential roles in controlling the nucleation, growth, and organization of HA crystals. The spectra data confirmed that enamel consists mainly of inorganic compounds

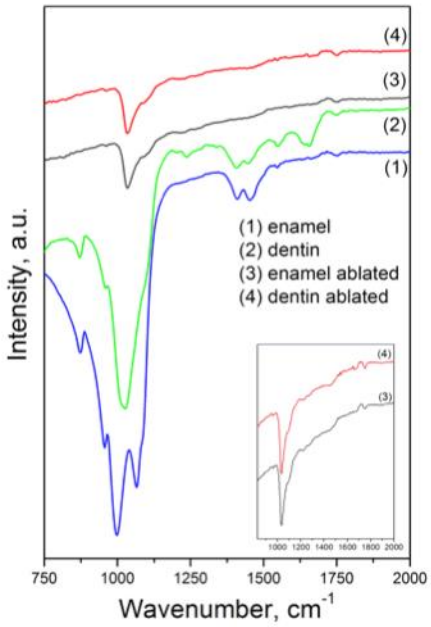

Figure 4. FTIR spectroscopy data for the intact and ablated enamel and dentin revealed no change in the mineral structure due to overheating effect of the laser during ablation.

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due to the low intensity of the amide bands of proteins in the region 1200-1650 cm^{-1} (spectrum 1). Peaks due to the amide I, II and III proteins were found at 1650, 1550 and 1237 cm^{-1}, respectively [30]. The amide II band confirmed that the organic part of enamel consists of collagen proteins. Stretching vibration of C=O bond in carbonyl groups originating from collageneous proteins was observed at 1750 cm^{-1}. On the other side, dentin with its high organic content is less crystalline (spectrum 2: the doublet structure of the PO_4 group is missing) and hence more porous and acid soluble [31,32]. The protein peaks in the region 1200-1750 cm^{-1} were also observed in the dentin spectrum with higher intensity which is equivalent to a higher concentration due to the superior organic content in the dentin tissue (table 1). Spectra of both dentin and enamel after the ablation (spectra 3 and 4) preserved the characteristic features of the inorganic HA with weak peaks of the collagen proteins as detailed in the inset. The lower peak intensity occurs because the ablated crater is deep (1 mm) and the signal coming back to the detector is weaker. However, it is certain that the more crystalline intact dentin and enamel have been transformed to more amorphous materials because the well-resolved HA peaks disappeared and only an envelope of the PO_4 group of HA was visible in the spectra. In conclusion, FTIR spectroscopy showed the lack of phase changes of the HA mineral which means no overheating was attained with the laser ablation.

Cavity preparation and treatment of caries in restorative dentistry has been performed so far mainly by the family of erbium lasers (Er; pulse durations in the range of 50 - 400 μs) which cut permanent teeth at similar rates to a dental drill. However, the hard tissue ablation with these lasers is based on the transfer of energy to the tissue, which causes different rapidly occurring phenomena, leading to undesired thermal and thermal-mechanical effects. This means that once the water absorbs the energy, the latter is converted into heat, causing superheating and vaporization. The increased steam pressure produces micro-explosive expansion within the hard tissue itself, causing the tissue to blast away. The fast shock wave created by Er laser absorption causes a massive disruptive volume expansion and enormous

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subsurface pressures. As a result the surrounding mineral matrix is blasted away and the fractured particles of tooth are ejected as part of the tooth ablation. That is why the Er laser family requires replacement by subpicosecond lasers, which can be used for cavity preparation. In this study we used a state-of the art and commercially available fs laser system to achieve high ablation rates due to subpicosecond pulses, high repetition frequency and ability to scan a wide area instead of using a stationary laser beam. Fast ablation resulted in laser-matter interaction with minimal side effects and reduced temperature increase. This overcomes the disadvantage of the laser systems reported in the literature, i.e. the unacceptable local tissue damages. Additional benefits to the resulting cavity included: lack of contaminants, absence of smear layer, as well as disinfection of the dentin and enamel surface prior to the filling. Further goal of the teeth ablation by using our fs laser system is to exceed the speed of drilling of the traditional rotary instruments and Er lasers, and thus to reduce the treatment time which in turn will bring comfort to the patient.

4. Conclusions

A state-of-the-art, high repetition rate subpicosecond laser was used in this work for fast and precise ablation of dentin and enamel hard tissue. Imaging revealed the formation of craters with well-defined precise vertical cavity sides and edges. There was no discoloration, melting, carbonization or cracks as a result of the laser interaction with the dental tissue. The fs laser ablation led to an advantageous roughness increase and the removal of contaminants. Structural characterization showed the lack of phase changes of the HA mineral indicating that no overheating occurred as a result of the laser ablation.

The contemporary high repetition rate, subpicosecond-pulsed lasers hold the promise of significant drilling capabilities. A laser instrument for use in restorative dentistry eliminates the vibration of the dental drill, which also eliminates the risk of causing microfractures in the

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tooth. The laser used in this work achieves high processing efficiency and there is a potential for developing an instrument integrating a high dynamics subpicosecond pulsed laser for cavity preparation.

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Compliance with Ethical Standards

Authors Todor Petrov, Emilia Pecheva, Anthony D. Walmsley and Stefan Dimov declare that:

- There are no conflicts of interest;
- The work was supported by the following funding bodies acknowledged in the manuscript as well:
  - Engineering and Physical Sciences Research Council of the United Kingdom (grant № EP/J014060/01) ;
  - H2020 European Training Network ("European ERSs Network on Short Pulsed Laser Micro/Nanostructuring of Surfaces for Improved Functional Applications", Laser4Fun;
- Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors. Extracted teeth used in the study were received from the Birmingham School of Dentistry Tooth Bank and the work was conducted under ethical approval - REC Ref: 14/EM/1128.
- Informed consent was obtained from all individual participants included in the study.

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Highlights

1. Fast and precise ablation of dentin and enamel was attained with high repetition rate subpicosecond laser.
2. Craters with well-defined precise vertical cavity sides and edges and no collateral damage were obtained.
3. Advantageous roughness increase of the dental tissue and removal of contaminants were additional achievements with this type of laser.
4. No phase changes of the dental tissue were found after the laser ablation.
5. The contemporary high repetition rate, subpicosecond-pulsed laser hold the promise of significant drilling capabilities.

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