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Abstract. Less invasive methods for treating dental caries are strongly desired. However, conventional dental lasers do not always selectively remove caries or ensure good bonding to the composite resin. According to our previous study, demineralized dentin might be removed by a nanosecond pulsed laser operating at wavelengths of around 5.8 μm. The present study investigated the irradiation effect of the light on carious human dentin classified into "remove," "not remove," and "unclear" categories. Under 5.85-μm laser pulses, at average power densities of 30 W/cm^2 and irradiation time of 2 s, the ablation depth of "remove" and "not remove," and also the ablation depth of "unclear" and "not remove," were significantly different ($p < 0.01$). The ablation depth was correlated with both Vickers hardness and Ca content. Thus, a nanosecond pulsed laser operating at 5.85 μm proved an effective less-invasive caries treatment. [DOI: 10.1117/1.JBO.20.5.051023]

Keywords: 5.85-μm wavelength; nanosecond pulsed laser; carious human dentin; selective removal; Vickers hardness; Ca content.

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1 Introduction

Dentistry aspires to ultraconservative management concept, described as minimally invasive treatment and teeth preservation, also referred to as minimal intervention (MI).[1] Available clinical technologies for excavating teeth and removing caries include burs, excavators, air-abrasives, chemo-mechanical agents, ultrasonics, and lasers. Most of these techniques help to create cavities, but they are not self-selective for caries-infected dentin.[2,3] Therefore, to realize MI, new techniques of minimally invasive and selective treatment of caries are required.

Er:YAG and Er, Cr:YSGG lasers have already been applied in a water absorption and rapid evaporation technique for tooth ablation.[4–7] However, to efficiently harness the laser energies, this technique requires strict control by the dentist. In addition, the quantity of excavation is less easily controlled by an Er:YAG laser than by conventional mechanical excavation methods,[8] and the irradiated dentin surface may not strongly bond to the composite resin.[9,10] A recent systematic review concluded that laser treatments are not yet viable as a general dental practice option for caries excavation.[11,12] However, because lasers introduce no noise or vibration, viable laser treatments would increase the comfort levels.[13,14] If the excavation selectivity and tensile bonding strength of laser techniques could be improved, lasers could become an essential instrument of excavation.

In general, there are three sets of absorption bands at the wavelengths of about 3, 6, and 9 μm in dentin. The 6 μm absorption band is attributable to the dominant organic materials of carious dentin, which is the so-called as the amide 1 and amide 2 bands.[15] In our previous studies, we investigated the fundamental ablation properties of bovine dentin under a wavelength-tunable nanosecond pulsed laser operating at 5.6 to 6.6 μm.[16–18] We found that selective removal of demineralized dentin, due to a difference in the amount of ablation for sound and demineralized dentin, could be achieved with less damage to sound dentin, especially at the wavelengths of around 5.8 μm.[15] However, real carious human dentin varies according to the type and progression of caries. Subsequently, the ablation properties of carious human dentin were investigated at the wavelengths of around 5.8 μm.[19] The wavelength of the highest selective removal of carious dentin was then refined to 5.85 μm; however, the ablation selectivity was highly variable, and appeared to depend on the hardness rather than the absorption.[20]

In most cases, removal of grossly softened caries-infected dentin is recommended.[21] Thus, it is important to investigate the relationship between the hardness and ablation characteristics of carious dentin. The present study exploits the hardness difference between human carious and sound dentin to selectively remove the former by laser irradiation at the wavelength of 5.85 μm. Laser ablations were carried out at the optimally absorbed wavelength of 5.85 μm and the strongly absorbed wavelength of 6.00 μm. The relationships between ablation depth and hardness of carious human dentin (quantified by the Vickers hardness and the Ca content) were then evaluated. The dentin-resin bonding strength can potentially be improved by shortening the pulse width of a conventional dental Er:YAG laser to 1 ns. To determine whether this approach is compatible with dental adhesive procedures, we also evaluated the tensile

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bonding strength between the irradiated human sound dentin surface and a composite resin.

2 Materials and Methods

2.1 *Sample Preparations*

Human carious teeth (molar) were extracted and sectioned parallel to the occlusal surface, exposing the carious dentin lesion. The dentin plates were soaked in normal saline solution until required for ablation depth and hardness evaluation (see Sec. 2.4). Prior to experiment, the saline was wiped from the plates. The dentin areas including carious lesions were classified into "remove (carious)," "not remove (sound)," and "unclear (difficult to determine)" categories, based on their optical appearances and palpation by two conservative dentistry specialists with over 8 years of clinical experience. Figure 1 shows the typical absorption spectra of sound and carious human dentin in the mid-infrared range. The absorption spectra were measured by a Fourier transform infrared spectrometer (MB3000, ABB, Switzerland) coupled to an infrared microscope (μMax, Pike Technologies). The spectra were normalized at a peak wavelength of 6 μm.

The tensile bonding strength (see Sec. 2.5) was evaluated on human sound teeth (molar). The teeth were sectioned parallel to the occlusal surface, exposing approximately (10×10) mm^2 of the dentin area, with the dental tubules running perpendicular to the irradiated surface. The surfaces of the plates were ground on a silicon carbide waterproof abrasive paper with 600-grit in the wet condition. The polished plates were soaked in normal saline solution. Prior to experiment, the saline was removed from the plates by a dental air blower.

The human teeth samples were removed at the Department of Oral and Maxillofacial Surgery in the Osaka Dental University Hospital (Osaka, Japan). All samples were anonymized and stored after obtaining informed consent. This study was approved by the ethics committee at the Osaka Dental University (Approved No. 100502).

2.2 *Light Sources*

The nanosecond pulsed laser beam was produced by difference-frequency generation (DFG).[15,22] The DFG laser was jointly developed by RIKEN and Kawasaki Heavy Industries, Ltd. (both based in Japan). The wavelength-tunable range, pulse duration, and repetition rate of the pulse is 5.5 to 10 μm, 5 ns, and 10 Hz, respectively. To achieve a mid-infrared output, two $AgGaS_2$ crystals were inserted between a Q-switched Nd:YAG laser (Tempest 10, New Wave Research Inc.) operating at a wavelength of 1064 nm and a wavelength-tunable Cr:forsterite laser operating at 1150 to 1350 nm. The Cr:forsterite laser was pumped by a Q-switched Nd:YAG laser (Tempest 300, New Wave Research Inc.) operating at 1064 nm and its wavelength was tuned by rotating the rear mirror of an optical resonator.

Caries excavation (Sec. 2.5) was also performed using a conventional dental laser, namely, the Er:YAG laser (Erwin® AdvErL, Morita, Japan). The wavelength, pulse duration, and repetition rate of this laser is 2.94 μm, ~200 μs, and 10 Hz, respectively.

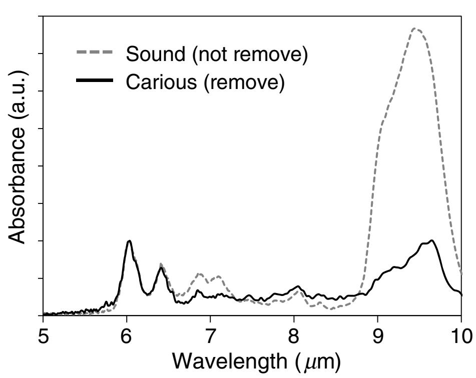

Fig. 1 Absorption spectra of sound and carious human dentins. Both dentins exhibit the characteristic amide 1 and 2 absorption peaks around 6 μm. The absorption peak assigned to the PO vibration (around 9 to 10 μm) is weaker in carious than in sound dentin.

2.3 *Irradiation Conditions*

When evaluating the ablation depth and hardness (Sec. 2.4), the carious human dentin samples were horizontally arranged on an XYZ-stage and the laser beam was irradiated by a parabolic mirror with a focal length of 100 mm. The beam diameters (full width at half maximum values of the beam profile) were determined as 100 to 150 μm by the knife-edge method. The wavelengths were set to 5.85 and 6.00 μm, and the average power density was set to 30 W/cm^2. The irradiation time was set to 2 s using an electric shutter (F77-4, Suruga Seiki, Japan). No water spray was applied.

In the caries excavation (Sec. 2.5), the human sound dentin samples were horizontally arranged on a motorized stage (SG SP 20-20, Sigma Koki, Japan), which was linearly moved at a constant rate. The scanning speed was calculated as the beam size divided by the irradiation time per spot (1 s). The approximate area irradiated by the DFG laser (wavelength = 5.85 μm, average power density = 30 W/cm^2, no water spray) and the Er:YAG laser (pulse energy = 100 mJ, water spray applied at 2 ml/min) was (4.5×4.5) mm^2. The irradiation conditions of the Er:YAG laser were those used in clinical settings.

2.4 *Evaluations of Ablation Depth and Hardness*

Following the irradiation experiments, the ablation depths were measured using a confocal laser microscope (OLS3000, Olympus, Japan). The Vickers hardness around the irradiation spots was measured by a dynamic ultramicrohardness tester (DUH-211, Shimadzu, Japan) with a maximum indentation load and a depth of 196 mN and 3 μm, respectively. Next, the samples were gold-coated by an ion sputtering system (E-1010, Hitachi, Japan), applied for 60 s at 15 mA discharge current. The irradiated regions were then observed using a scanning electron microscope (SEM) (JCM-5700, JEOL, Japan). Additionally, the Ca contents were measured at 10 positions and averaged over the irradiation spots by an energy dispersive x-ray spectrometer (JED-2300, JEOL, Japan) coupled to SEM at an accelerating voltage of 20 kV at 37-fold magnification.

2.5 *Evaluations of Tensile Bonding Strength*

The compatibility of the irradiated normal dentin surface with adhesive restoration procedures was evaluated in tensile bonding strength tests. The preirradiated samples were mounted on a brass jig, leaving an exposed circular region with a diameter of

3 mm for the testing. A self-etching primer (Clearfil Mega Bond, Kuraray Noritake Dental, Japan) was applied to the irradiated dentin surface, and a composite resin (Clearfil AP-X, Kuraray Noritake Dental, Japan) was then bonded to the surface. The samples were retained in water at 37°C for 24 h, and their tensile bonding strengths were then measured by a universal testing machine (IM-20, Intesco, Japan) at a crosshead speed of 0.3 mm/ min until failure occurred. The fractured surfaces of the samples were observed using a laser microscope (VK-X100, Keyence, Japan) at 50-fold magnification to evaluate the failure modes. When cohesive failure accounted for 70% or more of the fractured surface, the mode was regarded as cohesive failure. When cohesive failure rate was <70%, the mode was regarded as mixed failure. When 70% or more of the interface was exposed, the mode was regarded as interfacial (pretesting) failure.

2.6 *Statistical Analysis*

Statistical analysis was performed using the statistical functions of Excel 2013 (Microsoft Office, Microsoft Corporation). Continuous variables were expressed as mean and standard deviation. Fisher's extract tests were used to compare dichotomous variables in two groups and unpaired Student's t tests were used to assess differences in continuous variables. The tests were two-sided and a P-value <0.01 was determined to be statistically significant.

3 Results

3.1 *Selective Removal by 5.85-μm Laser Pulses*

Figure 2 shows the SEM images of the surface morphologies of sound and carious dentin irradiated at 5.85 and 6.00 μm. The 5.85-μm pulses removed considerably more carious dentin than sound dentin. On the other hand, the irradiation spots under 6.00-μm pulses were similarly sized on sound and carious dentin, indicating that demineralized dentin was not selectively removed at this wavelength. Cracking, which is undesirable in restoration treatment, was absent in both dentins.

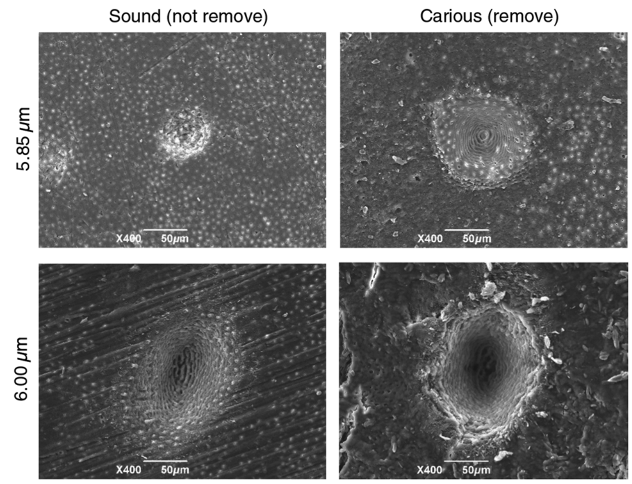

Fig. 2 Scanning electron microscopy images of irradiation spots at 5.85 and 6.00 μm. The 5.85 μm laser light removed a large amount of carious dentin while retaining the sound dentin. The 6.00 μm light was less selective (the irradiation spots in sound and carious dentins are similarly sized).

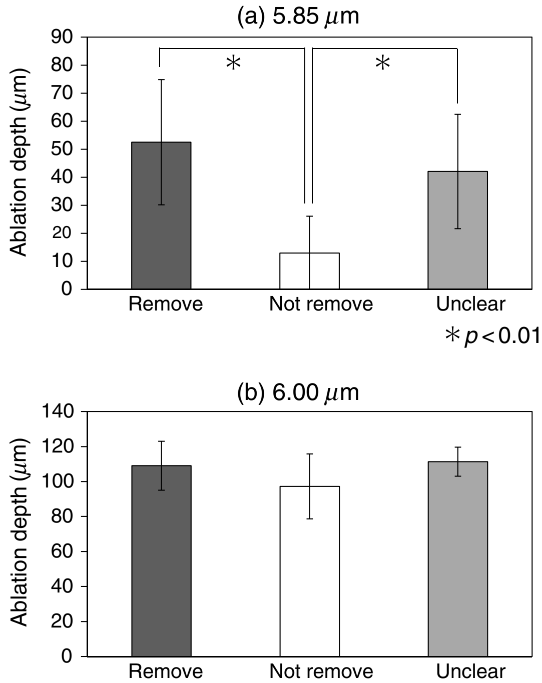

Fig. 3 Differences in ablation depth among three categories of carious lesions, "remove," "not remove," and "unclear" at (a) 5.85 μm and (b) 6.00 μm. At 5.85 μm, the ablation depths significantly differed between "remove" and "not remove," and between "unclear" and "not remove."

Figure 3 shows the averaged differences in ablation depth for the dentin categories "remove," "not remove," and "unclear," irradiated at 5.85 and 6.00 μm. Under 5.85 μm irradiation, the number of sample spots classified as "remove," "not remove," and "unclear" were 121, 88, and 46, respectively [Fig. 3(a)]; under 6.00 μm irradiation, they were 46, 72, and 25, respectively [Fig. 3(b)]. In Fig. 3(a), the ablation depth of "remove" and "not remove," and also the ablation depth of "unclear" and "not remove," were significantly different ($p < 0.01$). The ablation depths showed a large standard deviation (approximately 40 μm) in the "remove" and "not remove" categories. By contrast, in Fig. 3(b), the ablation depths were not significantly different among the three dentin categories.

3.2 *Relationship Between Ablation Depth and Dentin Hardness*

Figure 4 plots the relationships between ablation depth and Vickers hardness of carious dentins. Under 5.85 μm irradiation, the numbers of sample spots classified as "remove," "not remove," and "unclear" were 22, 18, and 16, respectively [Fig. 4(a)]; under 6.00 μm irradiation, they were 14, 15, and 14, respectively [Fig. 4(b)]. Figure 4(a) shows a clear negative correlation between ablation depth and Vickers hardness. Specifically, ablation was reduced (enhanced) at high (low) Vickers hardness. The coefficient of determination in a linear approximation (R^2) was 0.38. However, the ablation depth-hardness correlation disappeared under 6.00 μm irradiation [$R^2 = 0.09$; see Fig. 4(b)]. The Vickers hardness threshold at which the category switched from "remove" to "not remove" was around 30 to 40.

Figure 5 plots the relationships between ablation depth and Ca content of carious dentins. In Fig. 5(a) (5.85 μm), the sample

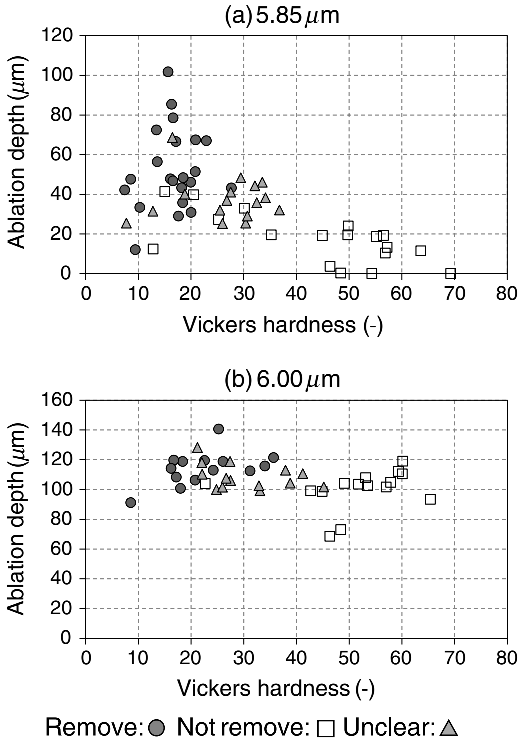

Fig. 4 Relationships between Vickers hardness and ablation depth at (a) 5.85 μm and (b) 6.00 μm. The Vickers hardness is correlated with ablation depth at 5.85 μm, but not at 6.00 μm. In (a), large ablation is associated with low Vickers hardness.

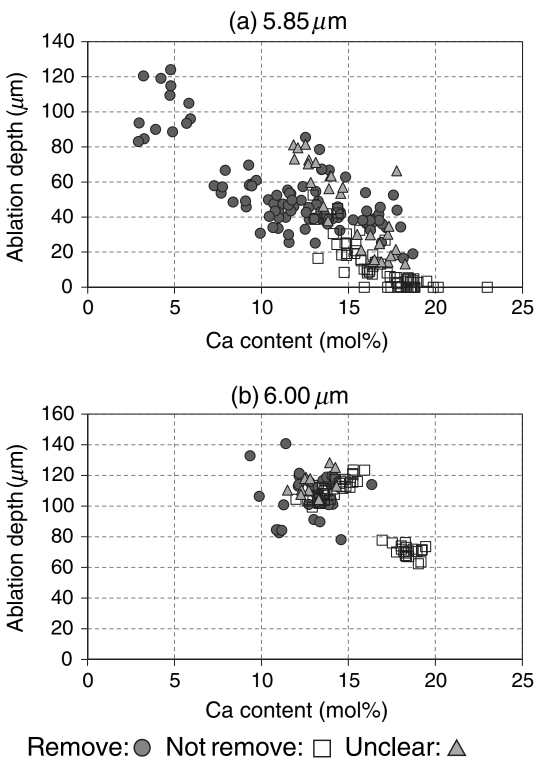

Fig. 5 Relationships between Ca content and ablation depth at (a) 5.85 μm and (b) 6.00 μm. The Ca content is correlated with ablation depth at 5.85 μm, but not at 6.00 μm. In (a), large ablation is associated with low Ca content.

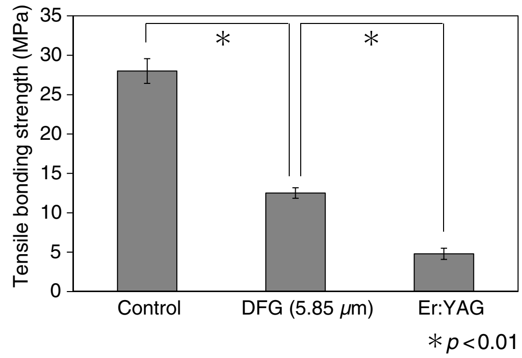

Fig. 6 Tensile bonding strengths under different irradiation conditions. The bonding strength was much higher under 5.85-μm DFG laser irradiation than under Er:YAG laser irradiation.

spots classified as "remove," "not remove," and "unclear" numbered 99, 70, and 30, respectively; in Fig. 5(b) (6.00 μm), they numbered 32, 56, and 11, respectively. The results of this analysis approximately mirror those of the Vickers hardness, but no clear threshold separates the "remove" and "not remove" categories.

3.3 *Tensile Bonding Strength Measurements*

Figure 6 shows the tensile bonding strengths of nonirradiated samples, samples irradiated by the 5.85 μm-DFG laser, and samples irradiated by the Er:YAG laser. In all cases, five samples were irradiated. The tensile bonding strength was significantly higher under the optimized DFG laser conditions than that under standard Er:YAG laser conditions ($p < 0.01$). The failure modes of nonirradiated conditions, DFG laser conditions, and Er:YAG laser conditions were evaluated as cohesion failure, mixed failure, and interfacial failure, respectively.

4 Discussion

Since Stern first reported laser ablation of dental hard tissue approximately 50 years ago,[23,24] tooth excavation by laser has been trialed at numerous wavelengths. The first lasers approved for hard dental tissue use were the Er:YAG and Er, Cr:YSGG lasers operating at 2.94 and 2.79 μm, respectively.[4–7] Excellent high-speed precise ablation of dental caries has been achieved by pulsed CO_2 lasers with the wavelengths of 9.6 and 9.3 μm.[25–31] Dental target selectivity using CO_2 lasers, Nd:YAG laser, and lasers with the wavelengths of 355, 377, and 400 nm has also been reported.[32–36]

The present study of selective laser-based ablation for carious dentin is entirely novel. The mid-infrared wavelength of 5.85 μm has not been previously considered for medical applications. Laser light of 5.85 μm applied to demineralized or carious dentin without water spray is mainly absorbed by collagen. The absorption coefficient (μ_a) of carious dentin is similar to that of collagen and demineralized dentin, and is estimated as 500 to 1000 cm^{-1} at 5.85 μm.[15,37] The thermal relaxation time τ_{therm}[38] and stress relaxation time τ_{stress}[39] are given by

$$\tau_{therm} = \frac{\delta_p^2}{4\alpha} = \frac{1}{4\alpha\mu_a^2}, \tag{1}$$

$$\tau_{stress} = \frac{\delta_p}{c_s} = \frac{1}{\mu_a c_s}, \tag{2}$$

where δ_p is the optical penetration depth, α is the thermal diffusivity, and c_s is the speed of sound. The α of dentin was assumed as 1.83×10^{-3},[40] while c_s of demineralized dentin was taken as 1.60×10^5 cm/s.[41] Using these parameters, the τ_{therm} and τ_{stress} of carious dentin at 5.85 μm were calculated as 0.14 to 0.55 ms and 6.3 to 12.5 ns, respectively. In the present study, the DFG laser was pulsed at 5 ns, sufficiently shorter than τ_{therm}. Since the interval (0.1 s) exceeds the τ_{therm} of carious dentin, the interaction time τ_{int} corresponds to the pulse duration. The degree of thermal damage can be assessed by considering the thermal confinement condition ($\tau_{int} < \tau_{therm}$). In the present study, thermal confinement was satisfied and photomechanical interactions were induced because τ_{int} and τ_{stress} were comparable. Indeed, the ablation craters were extremely clean and appeared to be free of cracks induced by thermally induced stresses.

Thermal damage to the irradiated dentin surface is thought to contribute to reduced tensile bonding strength.[42,43] In this study, the tensile bonding strength between the composite resin and dentin surface was significantly higher under the DFG laser irradiation (12.5 MPa) than under irradiation by the standard dental Er:YAG laser (4.8 MPa). Different failure modes were also observed between the DFG laser and the Er:YAG laser. The interfacial failure by the Er:YAG laser is likely to be due to a smear layer caused by the thermal damage.[44] By contrast, the mixed failure by the DFG laser indicates less smear layer. Therefore, the advantages of short laser pulses are twofold; less thermal damage to the dentin surface and enhanced tensile bonding. On the other hand, the tensile bonding strength was significantly lower under the DFG laser irradiation than under nonirradiation (28.0 MPa). However, reportedly, bonding strength higher than 10 MPa is considered clinically sufficient.[45,46] Therefore, the tensile bonding strength under the DFG laser irradiation, satisfied the clinical demand, is not thought to be a severe problem.

Water absorbs over a broad wavelength range around 6 μm, attributed to its OH bending vibration mode.[47] The shoulder of this band is located at 5.85 μm. The μ_a and optical penetration depth δ_p of water are approximately 540 cm^{-1} and 18.5 μm, respectively. Thus, the light absorption at 5.85 μm is affected by the oral hydric environment and the amount of water spray, and the selectivity and accuracy of laser absorptions are inconsistent. In clinical applications at 5.85 μm, no or less water spray is preferred for stable and selective ablation. In the mid-infrared range, specifically at wavelengths that are strongly absorbed by dental hard tissue, microsecond pulses likely require less water cooling to prevent peripheral thermal damage.[31] The pulse width applied in the present study (5 ns) is significantly shorter than the thermal diffusion time of carious dentin, so it is expected to incur minimal thermal damage and allow less water spray.

Laser caries treatment should also account for the thermal effects on dental pulp vitality. Short pulses and the high absorption coefficient of the dentin reduce the accumulation of heat in the dentin. Because the present study satisfies the thermal confinement condition, the temperature rise can be limited to the optical penetration region of the dentin, preventing thermal damage to wider areas. The δ_p of carious dentin at 5.85 μm is approximately 10 to 20 μm; that is, the light penetrates only the dentin surface and remains far from the dental pulp. Based on these optical tissue properties, no thermal effects on dental pulp vitality are expected. However, this hypothesis requires verifying in further experiments accounting for pathological or biochemical effects.

Because demineralization elutes hydroxyapatite from the dentin, carious dentin chiefly comprises organic material. Reportedly, the constituent ratio of organic to mineral material continuously alters as the caries progresses, with corresponding changes in hardness.[48–51] In this study, carious human dentins were diagnosed by their optical appearances and palpation by dentists. The three categories of carious lesions "remove," "not remove," and "unclear" were categorized by the Vickers hardness. The threshold of Vickers hardness separating the "remove" and "not remove" categories was around 30 to 40, suggesting that the Vickers hardness provides an index parameter for selective removal of carious dentin. In fact, some studies have reported a relationship between caries progression and hardness, and the effectiveness of hardness in caries diagnosis.[52,53] In the ablation experiment undertaken at 5.85 μm, areas categorized as "remove" and "unclear" were removed more rapidly than the areas categorized as "not remove"; that is, the technique preferentially ablated soft carious dentin. These findings are likely explained by the relationship between the counteracting force of photomechanical ablation and the hardness of carious dentin. High absorption energy density increases the counteracting force. High μ_a of the target increases the absorption energy density. As the organic material maximally absorbs at 6.00 μm, consequently, the counteracting force is higher at 6.00 μm than at 5.85 μm. The increased counteracting force is likely responsible for the nonselective excavation at 6.00 μm. On the other hand, the counteracting force at 5.85 μm was below the mechanical strength of hard carious dentin but exceeded that of soft carious dentin. Consequently, soft carious dentin was ablated while the hard dentin remained intact.

The relationship between the mechanical strength of carious dentin and the counteracting force of laser irradiation is crucial in selective ablation of carious dentin by mid-infrared laser pulses. The irradiation conditions of this study (wavelength = 5.85 μm, average power density = 30 W/cm^2, and irradiation time = 2 s) provided the appropriate counteracting force for selective removal. Conversely, the irradiation condition for selective removal is underspecified if the mechanical strength versus irradiation force relationship is met. In our previous works, we selectively removed demineralized bovine at several combinations of wavelengths and energies.[15,17] Selective removal was achieved by either high absorption wavelengths and low average power densities or low absorption wavelengths and high average power densities. Therefore, provided that μ_a is not altered much from its value at 5.85 μm, carious dentin should be selectively removable at other wavelength ranges, such as 3 μm and 6 to 10 μm. However, extremely-high or extremely-low absorption ranges are inadvisable because low-power irradiation reduces the ablation rate, whereas high-power irradiation reduces the selectivity. In addition, wavelengths that are strongly absorbed by water should be avoided because the ablation properties would then strongly depend on the wetness conditions. Consequently, the suitable wavelength range is limited in practice.

Despite the difficulty of high-power oscillations or generations at 5.85 μm, a compact dental laser device could be constructed using nonlinear optical techniques and quantum cascade laser (QCL) techniques under low-repetition and short-pulse control, which are currently available. However, these techniques are problematic. The low repetition rate of the DFG laser caused a low ablation rate in our current experiment. To increase the ablation rate, we must increase either the peak power or the repetition rate. Nonlinear optical techniques with Q-switched pulsed lasers can generate short pulses at high peak power, but current repetition rates are regulated only at tens of hertz. Conversely, QCL techniques realize high repetition operation, but are unsuited for high peak power operation. Therefore, if QCL techniques are to be useful in dental treatment, they require higher average power oscillation and pulse control techniques. Short laser pulses at 5.85 μm can also be delivered by the optical hollow fiber technique,[54] which has already been applied in a dental Er:YAG laser device. Further innovation is required to render the tip of the optical hollow fiber nontoxic and highly transmitting.

Although selective removal of caries-infected dentin is ultimately required in the MI approach to caries treatment, hardness may not reliably indicate infection. Thus, to ensure that carious dentin ablation complies with the MI concept, the relationship between the degree of infection and ablation selectivity must be investigated in further study.

5 Conclusion

Carious human dentin was selectively removed by applying nanosecond pulsed laser light at 5.85 μm. This treatment caused minimal damage to sound dentin, whereas material with low Vickers hardness and low Ca content was largely removed. Future study will investigate the relationship between degree of infection and ablation selectivity, attempt to improve the ablation rate, and develop a compact device for clinical application.

Acknowledgments

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