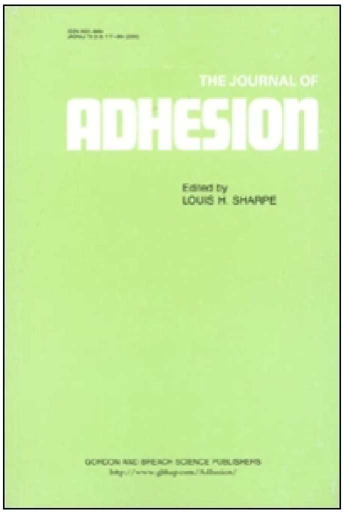

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Evaluation of Laser Treatment on Reline-Base Composites

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The effects of different laser treatments on some mechanical properties of acrylic resin and soft liner were investigated. A total of 60 test specimens were fabricated according to test requirements. The specimens were roughened with Potassium-ticanyl-Phosphate (KTP), Er:YAG, and Nd:YAG lasers before application of soft liner. The flexural, peel, and tensile bond strengths were measured using a universal testing machine. Fourier transform infrared spectroscopy with attenuated total

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reflectance (FTIR-ATR) spectra of surfaces were also obtained to evaluate changes on the lased surfaces. No significant difference was apparent between the tensile bond strength values of the groups. Although peel strength values obtained for each of the laser types were lower than those of the control group, flexural strength values were higher than those of the control specimens. The spectra of specimens showed that lasing led to some chemical changes on the resin surfaces. Physical changes on the treated surfaces were visualized by scanning electron microscopy (SEM) analysis. Results of this study suggest that such treatments may be warranted because of the increase in flexural strength.

Keywords: Flexural strength; Infrared spectroscopy; Interfacial adhesion; Laser applications; Peel strength; Poly (methyl methacrylate); Silicon-based soft liner; Surface treatment; Tensile bond strength

INTRODUCTION

Soft lining materials are often applied to line the denture base of patients with lesions on the mucosal surface, congenital or acquired defects of the palate, and areas of severe undercuts [1–5]. These materials provide a better distribution of the functional load on the denture-bearing areas, thus avoiding local stress concentrations. Soft lining materials may cause several problems associated with their use, such as loss of softness, plaque and calculus accumulation, colonization of microorganisms and porosity. However, one of the most serious problems is the debonding of the soft liner material from the denture base material [6–11].

Many researchers have measured the bond strength between the soft liners and the denture base materials by using peel, shear, and/or or tensilebond tests. It has been shown that the measured bond strength of soft liners to poly (methyl methacrylate) (PMMA) is dependent on the type of testing method used [12–14]. The peel test processing simulates a reline procedure more accurately with an even distribution of force over the bonding area and has been considered to be more clinically relevant [15]. The tensile test does not simulate the forces to which the lining material is clinically exposed; however, this test is a good method of investigating the bond strength of soft lining materials, because it gives information on the strength of the bond in comparison with the tensile strength of the material [12]. On the other hand, the three-point bending test has been widely used by the investigators to determine the bond strength between different materials as well as the flexural strength of the acrylic resin itself. This test evaluates a combination of properties, such as tensile and compressive strength and modulus of elasticity [7].

Many attempts using peel and/or bond-strength tests were made to improve the bonding between the soft liner and acrylic resin by surface treatments. Although some studies have reported that an improvement on interface strength was gained by roughening the surface denture base before applying of lining material [16,17], others have shown the negative effects of the roughening process on the bonding of the two materials [11,18].

Recently, a laser has been used for surfaces of materials such as metal and ceramics to improve bonding and adhesion capacity between them [19,20]. However, there are few studies using lasers for the same purpose between soft liner materials and denture base [18,21]. In this study, the effects of the different laser types on the flexural strength and bonding properties of a silicone-based soft liner and an acrylic resin were studied. To see the physical changes on the acrylic resin surface after laser irradiation, a scanning electron microscopic examination was performed. A Fourier transform infrared spectrometer with a attenuated total reflectance unit (FTIR-ATR) was used to obtain spectra of surfaces before and after laser treatments to evaluate changes on the surfaces of acrylic specimens.

MATERIAL AND METHODS

The soft liner used in this study was a silicone-based material (Molloplast-B, Detax, Ettlingen, Germany) and the denture base material was a heat-cured polymerized acrylic resin (Meliodent, Bayer Dental, Newbury, UK). Acrylic resin surfaces were roughened by using KTP, Er:YAG, and Nd:YAG lasers (DEKA M.E.L.A. Srl Calenzano, Italy) before the application of Molloplast-B soft liner. A total of 60 test specimens were fabricated (20 for each mechanical test). Acrylic resin specimens of each test were randomly assigned to four groups, each containing five specimens ($n = 5$).

Specimens for peel strength testing were prepared by packing and processing acrylic resin into rectangular strips that measured $75 \times 25 \times 2$ mm. Twenty blank acrylic denture base specimens were constructed in the conventional manner; the polymerization process was carried out in dental flasks in water at 70°C for 1 h followed by boiling in a water bath for 30 min. After polymerization, the acrylic resin strips were deflasked and trimmed away. Surfaces to be bonded with soft liner were smoothed using 240-grit silicon carbide paper, cleaned, and dried. To create a space for soft liner material, the acrylic specimens were reflasked using fresh pink wax material. After the removal of the wax, flasks with acrylic specimens were randomly divided into four groups. Fifteen of the 20 specimens were treated with

TABLE 1 The Conditions of Laser Treatments

Application conditions	Lasers KTP	Er:YAG	Nd:YAG
Wavelength	532 nm	2940 nm	1064 nm
Frequency	30 Hz	20 Hz	30 Hz
Power	3 W	3 W	3 W
Application timing	20 s	20 s	20 s
Transmission system	Optical fibers (300-μm fibre core diameter)	Articulated arm (sapphire tip)	Optical fibers (300-μm fibre core diameter)
Pilot beam	Diode laser (3 mW & 650 nm)	Diode laser (1 mW & 680 nm)	HeNe laser (1 mW & 632.8 nm)
Operation mode	Continuous (CW)	Pulsed	Pulsed
Pulse length	—	230 μs	Enh 3:100 μs
Beam diameter	4 mm	1 mm	4 mm
Maximum pulse energy	—	500 mJ	250 mJ

lasers, and the remaining five were used as control groups. The conditions of the laser treatments are given in Table 1. The focused laser beam was aligned to the polymerized acrylic surface perpendicularly at 1 mm, and the area to be bonded with soft liner (25×25 mm portions of each acrylic resin surface) was treated manually in a sweeping fashion. Primo adhesive of Molloplast-B (Detax, Ettlingen, Germany) was applied onto the laser-treated surfaces and the untreated surface of control specimens. After waiting for 1 h, soft liner material was packed and processed for 2 h in a boiling water bath. The processed flasks were left to cool at room temperature for 20 min and were then kept under running tap water for 10 min.

For testing of the tensile bond strength, acrylic specimens 75 mm long and 12 and 7 mm in diameter (wide and narrow areas, respectively) were prepared. Three-mm sections were cut out from the narrow midsection of specimens using a water-cooled saw (model no. 11-1280-250, Buhler Ltd., Lake Bluff, Ill, USA). Lasers were used to treat the remaining cut surfaces. To provide space for soft liner, a 3-mm-thick Perspex material was placed between acrylic resin strips, and they were reflasked together. After the removal of the Perspex materials, Primo adhesive and soft liner material were applied.

For the flexural test, 20 blank soft liner–acrylic denture base composite specimens ($65 \times 10 \times 2$ mm) were constructed as described previously, and the whole surface of acrylic specimens were treated by lasers.

Mechanical tests were performed on a universal testing machine (Lloyd NK 5, Lloyd Instruments Ltd., Fareham, Hampshire, UK) using a crosshead speed of 10 mm/min for the peel test and 50 mm/min for the flexural and the tensile bond strength tests, respectively. Peel strength was calculated from the equation where the peeling angle was considered 180°:

$$\text{Peel Strength } (\mathrm{Nmm}^{-1}) = \frac{F}{d}\left(\frac{1+\lambda}{2}+1\right)$$

where F is applied force, d is width of the specimen in the peeling area, and λ is extension ratio of the liner (the ratio of stretched to unstretched length).

Tensile bond strength was calculated from the formula:

$$S(\mathrm{MPa}) = \frac{F}{D}$$

where S is tensile bond strength, F is the force, and D is the adhesion surface area.

Flexural strength was measured by the three-point bending test with the surface of the denture base material placed face down for each. The load and deflection curves of the specimens were recorded on a chart recorder. The flexural strength was calculated with the following formula:

$$S(\mathrm{MPa}) = \frac{3FL}{2bd^2}$$

where S is flexural strength, F is applied load, L is span distance, b is width of the specimen, and d is thickness of specimen.

After the collection of data, mean values and standard deviations were calculated with SPSS statistical software program (version 10.0, SPSS Inc., Chicago, USA). The differences of control and laser-treated groups in each test were evaluated by the Kruskall Wallis analysis of variance, and pairwise comparisons within groups for each test were carried out by using the Mann–Whitney U test.

Visual comparisons between the surface roughness of control and laser-irradiated group were made using a low-angle scanning electron microscope at 16 × magnification (SEM, Jeol JSM 6400, Noran Instrument, Tokyo, Japan). Additionally, FTIR-ATR spectra of surfaces before and after laser treatments were also taken to evaluate the changes produced. FTIR-ATR unit of a Bruker Vertex 70 spectrometer provided with diamond-protected ATR crystal was used (Bruker Optik GmbH, Ettlingen, Germany). Fifty scans were obtained and averaged to a resolution of $4\,\mathrm{cm}^{-1}$.

RESULTS AND DISCUSSION

Data obtained from the testing experiments were evaluated statistically (Table 2). In the peel test, the highest value (0.34 ± 0.07 Nmm^{-1}) was obtained from the control group. The comparison of the peel-strength results by Kruskall Wallis analysis of variance revealed that difference of the test groups was statistically significant ($p = 0.015$). The pairwise comparison of the groups indicated that although the peel strengths of KTP, Er:YAG, and Nd:YAG laser-treated specimens were different from the control group ($p < 0.05$), they did not seem to be different from one another ($p > 0.05$). This finding was in agreement with the study of Jacobsen *et al.* [18]. They have proposed that lower peel strengths were due to the size of the irregularities created by the laser on acrylic surface that might not be sufficient to allow the flow of the soft lining material [18].

Laser treatments of the acrylic surfaces did not seem to improve the tensile bond strength between the soft liner and acrylic resin (Table 2). Usumez *et al.* [21] have also reported similar results. It can be argued that roughening of the surface might have prevented the formation of a high tensile bond strength because of the stress concentration resulting from the discontinuities on the surface.

Results of the modifications on the laser-treated surfaces were visualized by SEM (Figures 1–3). Figure 4 shows an image of the untreated (control) surface. According to the SEM results, it appears that the KTP laser created randomly distributed larger and deeper pores, without debris (Figure 1); Er:YAG treatments resulted in the formation of numerous smaller, bubble-like inclusions, distributed irregularly (Figure 2); and the Nd:YAG laser caused some meltings

TABLE 2 Results of the Tests Obtained from Each of the Groups

Groups	Peel strength (Nmm^{-1}) ($\bar{x} \pm$ Sd)	Tensile bond strength (MPa) ($\bar{x} \pm$ Sd)	Flexural strength (MPa) ($\bar{x} \pm$ Sd)
KTP treated	0.17 ± 0.06^{a}	11.69 ± 2.79	8.45 ± 1.20^{d}
Er:YAG treated	0.16 ± 0.08^{b}	9.58 ± 5.54	8.66 ± 0.87^{e}
Nd:YAG treated	0.13 ± 0.06^{c}	9.76 ± 0.88	9.30 ± 2.14^{f}
Control	$0.34 \pm 0.07^{a,b,c}$	10.51 ± 0.89	$5.79 \pm 0.95^{d,e,f}$
Kruskal Wallis analysis values	10.49	3.74	9.69
Mann-Whitney U test values	$p = 0.015$ ($p < 0.05$)	$p = 0.291$ ($p > 0.05$)	$p = 0.021$ ($p < 0.05$)

Note: The groups with same superscripted letters are statistically significant by Mann–Whitney U test at the 5% level.

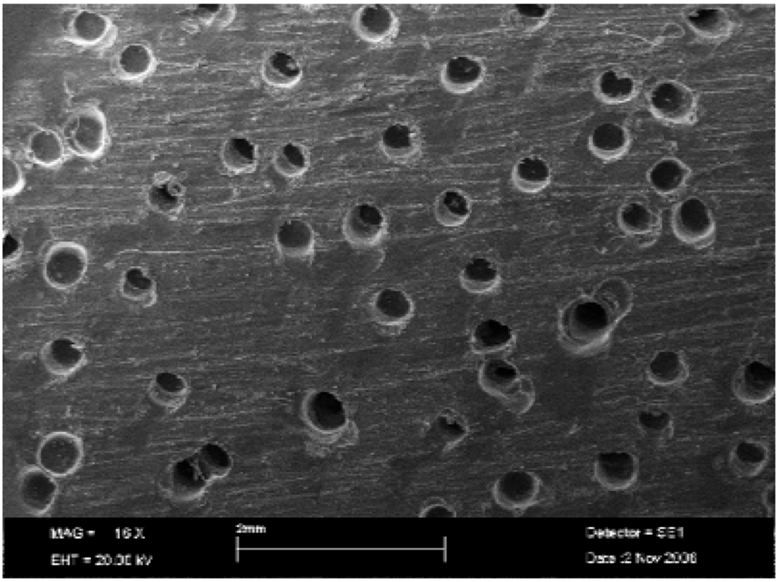

FIGURE 1 SEM view of acrylic surface treated with KTP.

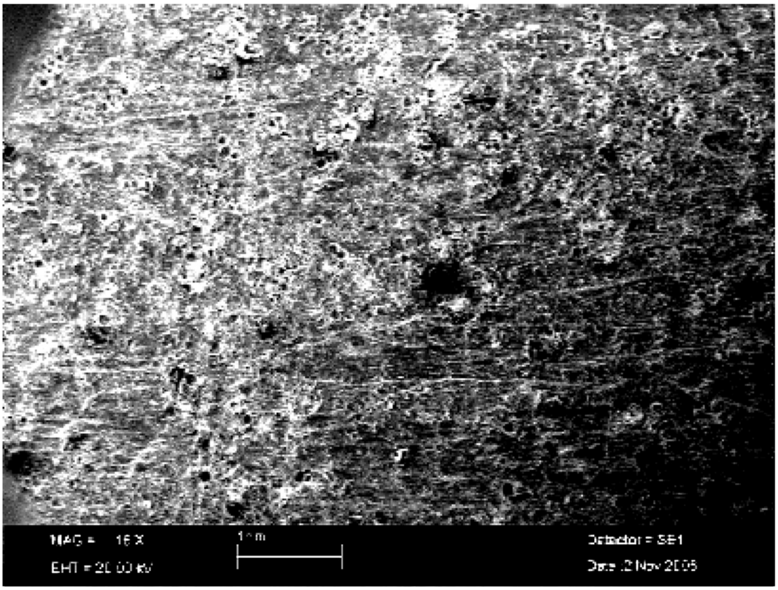

FIGURE 2 SEM view of acrylic surface treated with Er:YAG.

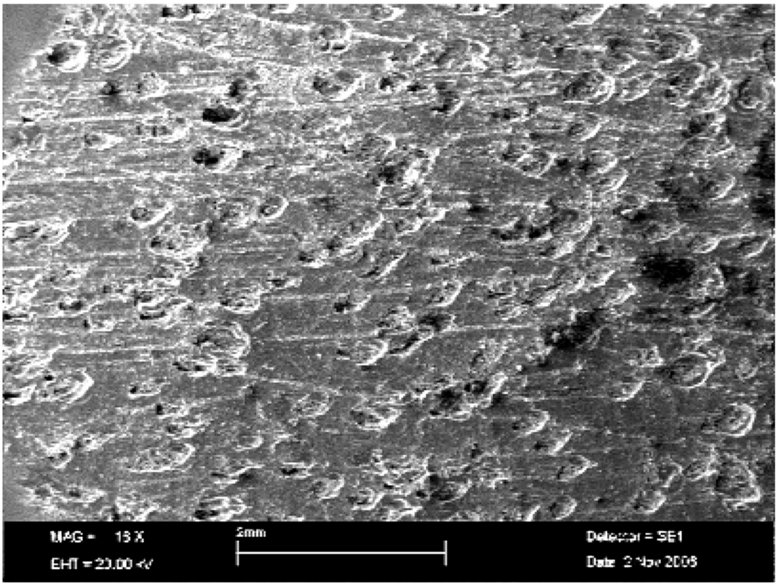

FIGURE 3 SEM view of acrylic surface treated with Nd:YAG.

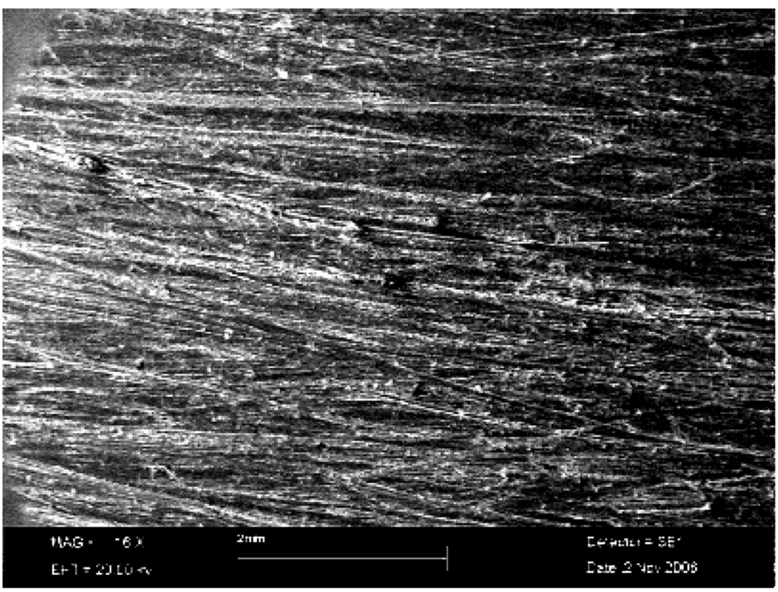

FIGURE 4 SEM view of untreated acrylic surface.

and resolidifications on the acrylic resin surface (Figure 3). Such different effects could be accounted for by the properties inherent in each of the laser types (Table 1).

Although laser application was not found to have a positive influence on tensile and peel strengths, their use provided an increase in flexural strength of PMMA denture base resin lined with soft material. The highest flexural strength value was recorded for the Nd:YAG group (Table 2). Comparison of the flexural strength values among the groups yielded statistically important differences ($p = 0.021$). Although the flexural strength values of the KTP, Er:YAG, and Nd:YAG laser-treated groups did not differ significantly from one another ($p > 0.05$), all the laser-treated groups showed higher strength values than that of the control group ($p < 0.05$). It was not possible to compare these results with the literature because no relevant reports have been available. However, FTIR-ATR analysis of the laser-treated acrylic surfaces indicated that cyclic anhydride or lactone structures might reduce the flexibility of the acrylic backbone. Süske *et al.* [22] have stated that laser applications led to chemical changes on acrylic films, and they have claimed that such changes bring about shortening of the chain length and cross-linking of the

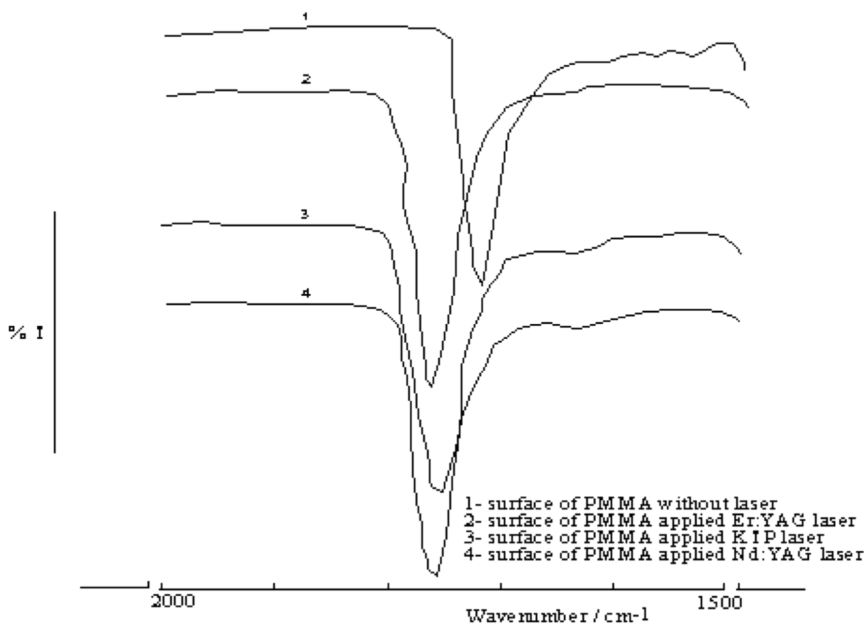

FIGURE 5 FTIR-ATR spectra of each group.

chains. It might be argued that these events could be responsible for the observed increases in the flexural strength values.

FTIR-ATR spectra of laser-treated and acrylic surfaces are shown in Figure 5. The band seen at $1727\,cm^{-1}$ indicates the main carbonyl group (C=O) of the control specimen. The band at $1750\,cm^{-1}$ indicates the anhydride or lactone structures within the laser-treated acrylic specimens. During the course of laser application, some of the acrylic chains may have been cleaved and rejoined because of the thermal degradation of the acrylic resin. These results suggest that laser treatments caused some chemical changes on the acrylic surfaces.

CONCLUSION

The FTIR results suggest that chemical changes occur on the acrylic surfaces treated with KTP, Er:YAG and Nd:YAG lasers. The physical outcomes of such chemical changes were also evidenced by the SEM micrographs. Laser treatment provides an improvement in the flexural strength of the acrylic denture base resin relined with Molloplast-B. However, it did not have beneficial effects on peel and tensile bond strengths between the two materials.

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